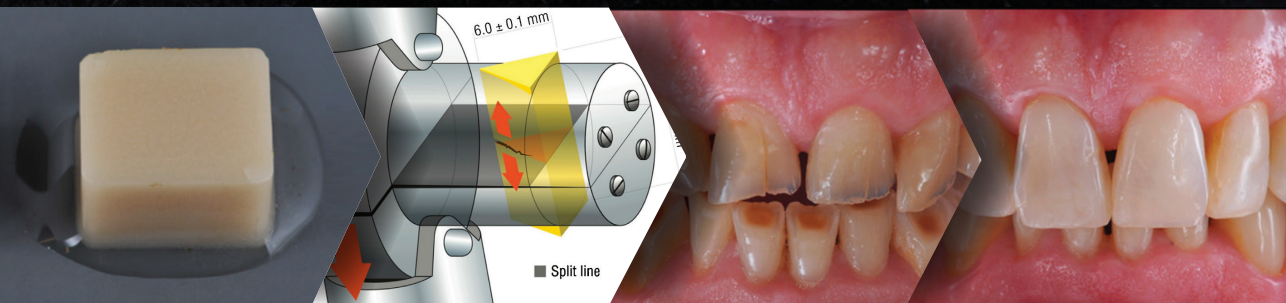




PROPERTIES AND APPLICATIONS OF POLYMER-INFILTRATED CERAMIC NETWORK MATERIALS



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Thesis submitted to obtain the degree
of Doctor in Dental Science
of the University of Liège, Belgium

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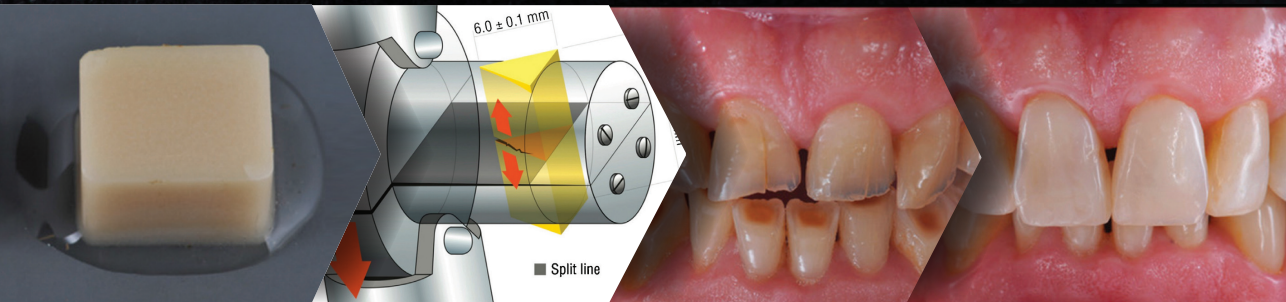
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ABSTRACT

Computer-aided design and manufacturing (CAD-CAM) materials are gaining popularity in the field of restorative dentistry. Among recently developed materials are polymer-infiltrated ceramic network (PICN) materials, a sub-class of CAD-CAM composites, comprised of 75 vol% sintered glass-ceramic network that is secondarily infiltrated with monomers and polymerized under high-temperature and pressure; whereas the other sub-class of CAD-CAM composites, dispersed fillers (DF), consist of inorganic fillers classically incorporated by mixing in an organic matrix that is secondarily polymerized under high-temperature.

The first objective of this work was to use fracture mechanics, particularly the notchless triangular prism (NTP) specimen fracture toughness test to: 1) evaluate the interfacial fracture toughness (IFT) of a resin composite luting agent (RCLA) with PICNs, represented by an experimental and a commercial PICN (Vita Enamic), versus DF materials. Lithium disilicate glass-ceramic (IPS e.max CAD, EMX) was also tested as a gold standard for comparison. Samples were pretreated with hydrofluoric acid (HF) or gritblasting (GR), and the results were correlated with the developed interfacial area ratio (Sdr) of representative samples subjected to the same pretreatment procedures. 2) Evaluate the influence of silane on the IFT of RCLA with PICN and DF after HF and GR, and correlate the results with the Sdr and surface wettability of representative samples. The results showed that the IFT of PICNs was significantly superior to DF, and IFT of etched experimental PICN was significantly higher than EMX. In addition, there was a strong correlation between the IFT and the Sdr of the representative samples, PICNs demonstrating significantly higher surface roughness than DF when pretreated. These results highlighted the importance of material microstructure and then class (DF vs PICN) on the bonding interface performance. Etching of PICN led to the selective dissolution of the glass-ceramic at the surface, creating an original polymer-based honeycomb structure that promoted the micromechanical retention of RCLA. This micromechanical retention is enhanced by the application of silane, which allows RCLA penetration in surface roughness. On the other hand, IFT of DF was not influenced by silane, regardless of the surface pretreatment, which was less effective in creating surface roughness.

The second objective was to introduce of a functionally-graded (FG) PICN block as a biomimetic material for CAD-CAM prostheses. FG-PICN is characterized by a gradient of mechanical and optical properties, in which the surface properties resemble the hardness and modulus of enamel, while the deeper layers resemble those of dentin. The flexural strength values at the dentin-like layer was similar to glass-ceramic EMX and flexural load energy was significantly higher than EMX and monolithic zirconia. These properties could

promote occlusal stress absorbance in treatment of patients with bruxism, such as worn dentition cases, and on implant restorations.

Finally, the third objective was to participate in two clinical studies performed following new treatment protocols developed with commercial PICN; a minimally invasive approach of worn dentition treatment with Vita Enamic bonded restorations; the “One-step No-prep” protocol and a novel approach for restoring a missing posterior tooth with immediate loading of an implant and a final crown made of PICN in a single visit; the “One-tooth One-time, 1T1T” protocol. The restorations success rate after 2 years was high (93.75% for the former and 90% for the latter), highlighting some edge chipping in the first study, and debonding from the ti-base in the second.

In conclusion, PICNs show a promising potential to be used in a variety of restorative applications, especially in minimally invasive dentistry, such as demonstrated in novel clinical applications of Vita Enamic. Indeed, *in vitro* studies highlighted the superiority of PICN, particularly the new PICN generation, to DF and lithium disilicate glass-ceramic in terms of IFT with RCLA. In addition, the original gradient of mechanical properties of the FG-PICN, opens the door for restorations with an improved bio-mechanical behavior compared to other CAD-CAM materials available on the market. Future perspectives include fatigue tests and clinical studies with experimental PICN materials.

KEYWORDS

Polymer-infiltrated ceramic network; dispersed fillers; resin cement; CAD-CAM; high-temperature high-pressure; notchless triangular prism test; interfacial fracture toughness; biomimetic; minimally-invasive dentistry; worn dentition; implant dentistry.

RESUME

Les matériaux de conception et de fabrication assistés par ordinateur (CFAO) gagnent en popularité dans le domaine de la dentisterie restauratrice. Parmi les matériaux récemment développés, on distingue deux sous-classes de composites CFAO : les PICNs (polymer-infiltrated ceramic network : réseau de céramique infiltré par un polymère) composés d'un réseau de vitrocéramique fritté (75% en volume), secondement infiltré par du monomère polymérisé sous haute température et haute pression ; et les composites à charges dispersées (DFs), lesquels sont constitués de charges inorganiques classiquement incorporées par mélange dans une matrice organique, qui est secondairement polymérisée sous haute température.

Le premier objectif de cette étude était d'utiliser la mécanique de la rupture et en particulier le test NTP (« notchless triangular prism ») de manière à : 1) mesurer la tenacité de l'interface adhésive (IFT : « interfacial fracture toughness ») d'un composite de collage à base de résine (RCLA : « resin composite luting agent ») avec les PICNs, à savoir un PICN expérimental et un PICN commercialisé (Vita Enamic), par opposition aux charges dispersées. La vitrocéramique au disilicate de lithium (IPS e.max CAD, EMX) a également été testée en tant que gold-standard afin d'être utilisée comme contrôle positif. Les échantillons ont été prétraités soit à l'acide fluorhydrique (HF) soit par micro-sablage (GR), et les résultats ont été corrélés au Sdr (« developed interfacial area ratio »), qui caractérise la rugosité de surface développée après pré-traitement, d'échantillons représentatifs soumis aux mêmes procédures de prétraitement 2) Évaluer l'influence du silane sur l'IFT du RCLA avec le PICN et le composite à charges dispersées, après prétraitement à l'acide fluorhydrique ou par micro-sablage, puis de corréler ces résultats avec le Sdr et la mouillabilité de surface d'échantillons représentatifs. Les résultats ont montré que l'IFT des PICNs était significativement supérieure à celle des DF et que l'IFT du PICN expérimental était significativement supérieur à celle de l'EMX. En outre, il y avait une forte corrélation entre l'IFT et le Sdr des échantillons représentatifs, les PICNs présentant une rugosité de surface significativement supérieure à celle des DFs après prétraitement. Ces résultats soulignent l'importance de la microstructure des matériaux, ainsi que celle de leur classe (PICN vs DF), sur la performance de l'interface adhésive. Le mordantage des PICNs a conduit à la dissolution sélective de la vitrocéramique en surface, créant une structure originale en nid d'abeilles formée par le réseau de polymère, favorisant ainsi la rétention micromécanique du RCLA. Cette rétention micromécanique est favorisée par l'application de silane, qui permet la pénétration du RCLA dans la rugosité de surface. D'autre part, le silane n'a pas montré d'influence sur l'IFT des DFs, quel que soit le prétraitement de surface, qui a été moins efficace pour créer de la rugosité de surface qu'au niveau des PICNs.

Le deuxième objectif était d'introduire un bloc de PICN expérimental biomimétique à gradient fonctionnel (FG-PICN) pour les restaurations CFAO. Ce FG-PICN est caractérisé

par un gradient de propriétés mécaniques et optiques : les propriétés de la surface du bloc ressemblent à l'émail en termes de dureté et de module d'élasticité, tandis que les couches plus profondes sont plus similaires aux propriétés de la dentine. Les valeurs de résistance en flexion de la couche simulant la dentine étaient similaires à celles de l'EMX et l'énergie de rupture en flexion était nettement supérieure à celle de l'EMX et de la zircono monolithique. Ces propriétés pourraient favoriser l'absorption du stress occlusal dans le cadre du traitement des patients bruxeurs, notamment dans les cas d'usure dentaire, et pour les restaurations sur implants.

Enfin, le troisième objectif était de participer à deux études cliniques réalisées selon deux nouveaux protocoles de traitement mis au point avec le PICN commercialisé : une approche minimalement invasive du traitement des cas avancés d'usure dentaire à l'aide de restaurations collées en Vita Enamic, le protocole «One-Step No-Prep», ainsi qu'une nouvelle approche pour réhabiliter un édentement postérieur unitaire en une seule visite via la mise en charge immédiate de l'implant avec une couronne définitive en PICN, le protocole «One-Tooth One-Time, 1T1T». Le taux de succès des restaurations en PICN après deux ans était élevé (93,75% pour la première étude et 90% pour la deuxième), soulignant quelques écaillages mineurs dans la première étude et un décollement du pilier en titane dans la seconde.

En conclusion, les PICNs présentent un potentiel prometteur pour être utilisés dans une grande variété d'applications en dentisterie restauratrice, en particulier dans le cadre d'une dentisterie minimalement invasive, comme démontré dans les approches cliniques innovantes avec le Vita Enamic. En effet, les études *in vitro* ont mis en évidence la supériorité des PICNs, en particulier de la nouvelle génération de PICNs, en termes de ténacité interfaciale (IFT) avec le composite de collage (RCLA), en comparaison aux charges dispersées et à la vitrocéramique renforcée au disilicate de lithium. En outre, le gradient original de propriétés mécaniques au sein du FG-PICN ouvre la voie à la réalisation de restaurations présentant un comportement biomécanique amélioré par rapport aux autres matériaux CFAO disponibles sur le marché. Les perspectives futures comprennent des tests en fatigue ainsi que des études cliniques incluant les PICNs expérimentaux.

MOTS CLEFS

Polymer-infiltrated ceramic network; composites à charges dispersés ; colles ; CFAO : haute-température haute-pression ; notchless triangular prism test ; ténacité interfaciale ; biomimétique ; dentisterie minimalement invasive ; usure dentaire ; implantologie.

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GLOSSARY

This glossary presents the different scientific expressions used throughout the manuscript and their abbreviations. Scientific expressions could change from one chapter to another due to journals' requirements; however, they carry the same meaning

CAD-CAM: Computer-aided design and computer-aided manufacturing

PICN: Polymer-infiltrated ceramic network; Polymer-infiltrated ceramic

DF: Dispersed fillers

RCLA: Resin composite luting agent; composite cement; resin cement

HT-HP: High-temperature and high-pressure

RC: Resin composites; composite resins

MTBS: Microtensile bond strength test

K_{IC} : Fracture toughness

IFT: Interfacial fracture toughness

NTP: Notchless triangular prism specimen fracture toughness test

Sdr: Developed interfacial area ratio

HF: Hydrofluoric acid etching

GR or AB: Gritblasting; airborne-particle abrasion; sandblasting

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INTRODUCTION

Chapter 1

1 | 1. The translational research approach

Dental clinicians use restorative materials in their daily practice that have been on the market usually long enough with a reputation of good quality, but also very often, they use materials recently launched and not yet sufficiently tested. Typically, a series of *in vitro* and, sometimes, *in vivo* studies are performed on materials for efficacy and/or comparison with other similar products, and normally, *in vitro* tests precede *in vivo* tests, both being important in evaluating a material's performance (Garcia-Godoy et al. 2012). While some aspects of the restorative materials are tested by companies *in vitro* prior to launching, many other aspects are being evaluated by dental researchers once the product is on the market already. *In vitro* studies provide controlled conditions for testing specific aspects of dental materials; however, it is difficult to reproduce the oral conditions exactly as they are with the masticatory forces, fluids and other variables (Faggion 2012). Van Meerbeek et al. (2010) pointed out the advantages of *in vitro* testing by being relatively fast, easy, cheap, specific parameters could be controlled and different materials could be compared to each other or to a well-performing "gold-standard". Those criteria tested *in vitro* would in turn give an idea about the clinical performance of the material (Van Meerbeek et al. 2010). Concurrently, researchers perform clinical trials on the new products to evaluate their performance and longevity; however, follow-up periods are often short and the sample size is usually small (Belli et al. 2016). Clinical research is important in providing a better insight on the performance of the materials, in which some failures could be demonstrated clinically but were not shown in *in vitro* tests (Kokich 2013), which creates a dynamic flow of input from clinicians to researchers and vice versa, supplying the manufacturers with feedback on the properties and aspects of the materials that need to be improved or adapted to better suit the oral environment. This shift from laboratory to clinical research is called translational research, which can be defined as the translation or movement of basic science or research from the laboratory to the clinical practice (Rubio et al. 2010). According to the National Institutes of Health (NIH), translational research is the process of moving forward with research from the pre-clinical studies to human trials, i.e. from the bench to the patient or the clinic, or in other words; from fundamental research to a macro-level. The main aim of shifting towards clinical research is to provide a better perception on the products with an overall goal of improving patients' health (Rubio et al. 2010; Hostiuc et al. 2016). Translational research is not uni-directional i.e. it can move in both ways providing a dynamic flow of information with the aim of improving the product (Hostiuc et al. 2016; van der Scheer et al. 2017). In fact, *in vitro* and *in vivo* research are both needed to build scientific evidence which is important to determine the quality of a restoration; however, it is not mandatory. The pyramid of

scientific evidence begins with *in vitro* studies at the base of the pyramid, which represent the first line of mechanical and biocompatibility research on a product (Varoni et al. 2015). The following blocks of the pyramid involve pre-clinical *in vivo* studies in which inclusion of animals to test the local tissue response for any possible cytotoxic or systemic responses can take place (Murray et al. 2007). Once all the biocompatibility parameters have been satisfied, the product can move to clinical trials on patients and the last step, the apex of the pyramid, involves systematic reviews and meta-analysis. Building evidence on dental materials is a long process that develops throughout the years by different research teams and it is the social responsibility of the personnel in the dental field, whether they are clinicians or technicians, towards their patients to gather all the available information on the different products and assess which best suits each clinical case. This dynamic and rapid evolution in the field of dentistry also necessitates dental clinicians to continuously develop their skills to the updated knowledge and to be aware of the most recent advances in the dental field, which will in turn benefit the people in concern, in that case the society (Slavkin 2017).

1 | 2. Evolution of restorative dentistry

Nowadays, three aspects of restorative dentistry evolution influence materials development and new treatment strategies: 1) the development of minimally invasive dentistry, 2) the development of CAD-CAM processes, particularly chair-side, as the trend to restore tooth tissues with 3) biomimetic materials.

2.1 Minimally invasive dentistry

Minimally invasive dentistry is an approach of restoring the health of teeth by means of early detection of caries, remineralization of demineralized dental hard tissues, minimally invasive intervention and repairing rather than replacing restorations (Frencken 2017); whereas the definition of minimally invasive dentistry in the field of teeth restoration can be simply explained by the removal of as minimal amount of tooth tissue as possible and placing small fillings (Ericson et al. 2003). According to Miles Markley, one of the pioneers in preventive dentistry: “the loss of even a part of a human tooth should be considered a serious injury, and that dentistry’s goal should be to preserve healthy, natural tooth structure”. These words are being appreciated decades after they were written in 1951 (Murdoch-Kinch and McLean 2003). Nowadays, minimally invasive intervention is made possible with the evolution of adhesive dentistry, for example in the treatment of worn dentition.

2.2 CAD-CAM processes

Dental computer-aided design/computer-aided manufacturing (CAD-CAM) system was introduced in 1971 by Francois Duret, who fabricated the first crown using an optical impression and a numerically-controlled milling system, known later on as the Sopha system (Priest 2005; Miyazaki et al. 2009). In the early 80s, Mormann sought help from Brandestini to develop a chair-side digitization system for the production of inlays and together they developed the CEREC system which was produced by Siemens (now known as Sirona) (Mormann 2006). Since then, various CAD-CAM systems were produced and released on the market under different brand names, and modifications in the hardware and software allow for the 3-dimensional fabrication of a wide range of restorations ranging from inlays, onlays, crowns, veneers to implant abutments, short-span bridges and partial denture frameworks. The CAD-CAM digital workflow may involve three steps: 1) digitization of tooth surface; 2) digital data treatment and digital design of the component and 3) digital component manufacturing. Dental CAD-CAM systems pave the way for the use of a variety of high strength ceramics and composites, in addition to metals for fixed partial dentures (Liu 2005; Beuer et al. 2008).

In CAD-CAM systems, an image is taken either mechanically via a touch-probe (earlier generations, Procera system), or via non-contact methods using an optical laser or white light reflected from the tooth to a camera that records a point cloud and translates it into a geometric figure by triangulation technology (data treatment) (Persson et al. 2006; Miyazaki et al. 2009). Further development of the triangulation technology led to the development of a 3D capture technique called the Multiscan technology. This technology uses 10 intraoral cameras and 5 projectors for a more precise image. Data capturing could also be done following the principles of confocal microscopy video sequence. It gives a 3D calculation of the scanned object by backscattering of the light rays at different depth-of-field levels. Other technologies are also available, for example, the wavefront technology, which utilizes multiple cameras simultaneously in a similar manner to the human eye, and the stereophotogrammetric video (Zimmermann et al. 2015).

Since the surface of the tooth is complex and varies from one person to another, the accuracy of the virtual 3-D image will depend on the distribution of an adequate number of points, known as the point density, to record the anatomy of the tooth from cusps to fossae, grooves, embrasures and undercuts, as well as the finish line (Vlaar and van der Zel 2006). Once the image is saved in the software, computer-aided design (CAD) of the restoration takes place. Design of the restoration could be performed either in the office or in the laboratory once the optical impression is received. Afterwards, a model of the restoration is formed and the restoration is either designed on the computer, or a wax-up is formed on the stone model, which is further scanned and a restoration is designed by combining the images done before and after the wax-up (double scan) (Davidowitz and Kotick 2011). The last step in manufacturing CAD-CAM

restorations is the computer-aided manufacturing (CAM) process. Most often, a block of the material of choice is fixed in the milling machine and with the aid of burs the restoration is fabricated. This method of manufacturing is considered as a subtractive method, since an amount of material is removed from the block to shape the final restoration. Although subtractive manufacturing is considered a rapid method of fabrication of restorations, it results in a waste of even a larger amount of material than what is used or the final restoration (van Noort 2012; Alholm et al. 2018). Another method of manufacturing of CAD-CAM prostheses is the additive method, which is used, for example, to produce metal frameworks, such as with laser melting (van Noort 2012). In this method, layer-by-layer deposition of the material on top of each other, in which one layer, represented by a cross-sectional slice in the 3-D image designed in the computer, solidifies then the next one is deposited on top of it until the final 3-D shape is obtained. A major advantage of this method is that only the needed amount of material is utilized and there is no waste of material such as in the subtractive method; however, it is time consuming compared to milling, making it not well-adapted to chair-side systems (van Noort 2012). Another disadvantage when considering ceramic and composite materials is that these materials produced by addition (for example with 3D printing) show a certain degree of porosity and less density (van Noort 2012).

The CAD-CAM technology is considered the future of restorative dentistry for many reasons, notably economical purposes. Most importantly, CAD-CAM processes have allowed the introduction of high-performance ceramic and composite materials which are industrially fabricated, therefore more homogenous, with minimal flaws. Moreover, industrial processes allow for the use of high performance polymerization processes for composite materials, improving the degree of conversion and mechanical properties (Mainjot et al. 2016). Regarding ceramic materials, CAD-CAM processes are the only method which allow the fabrication of high-strength ceramics such as alumina and zirconia materials, being able to manage high sintering shrinkage. Furthermore, the milling process is standardized resulting in high reproducibility (Brenes 2016; Ahlholm et al 2018). In addition, digital scanning impressions allow for record saving of all preparations for potential future need (Brenes 2016). While some people argue that optical impressions are more accurate than traditional impressions, which may suffer from inaccuracies related to the impression material such as polymerization shrinkage, incorporation of bubbles and the inability to record undercuts (Alghazzawi 2016), others contradict these claims simply by pointing out the difficulties encountered while taking optical impressions by structures like the tongue, cheeks and gingiva, as well as moisture contamination (Miyazaki et al. 2009). In fact, the process of optical scanning and designing of the restoration requires extensive training for one to be able to master it. CAD-CAM echnology allows for the fabrication of dental restorations chair-side with minimal, or without, dental laboratory involvement, therefore saving time for both the clinician and the patient (Miyazaki et al. 2009; Santos et al. 2013; Alghazzawi 2016). However, there

are some limitations, for example, not all dentists can afford to have a chair-side CAD-CAM system in their dental practice due to its high cost. Moreover, although the process of milling is done by a computer-controlled system, it does not accurately reproduce the desired tooth shape, especially the occlusal surface due to the limitation of the axes of cutting and milling (Karl et al. 2012). Furthermore, the pre-fabricated blocks offer limited color variations, even with the availability of multilayer blocks, unlike laboratory-fabricated restorations in which the color and shade can be tinted to better match the adjacent teeth (Santos et al. 2013). Yet, regarding the marginal fit of the restorations, studies have shown that CAD-CAM processes produce a marginal fit within the clinically acceptable range (up to 120 μm) (Boitelle et al. 2014; Tsiogiannis et al. 2016; Papadiochou and Pissiotis 2018).

Clinical studies comparing CAD-CAM ceramics and their hot-pressed versions are scarce. However, Belli et al. (2016) estimated a lower failure rate for the IPS Empress CAD inlays (9.5 %) and onlays (10.5 %) as compared to the hot-pressed version (IPS Empress) (12.5 %) over a period of 12 years. Moreover, clinical studies were conducted to compare the accuracy of conventional versus digital impressions. Some studies favored the conventional techniques over the digital ones for single crowns, fixed partial dentures and implant supported restorations, while others found out that digital impressions were more accurate in such cases. In conclusion, there was no difference in accuracy of the impression between conventional and digital impression techniques for single or short-span restorations; however, for full-arch fixed partial dentures, conventional methods were found to be more precise and reliable than digital impressions due to the higher local deviations produced by digital impressions (Ahlholm et al. 2018).

2.3 Biomimetics

Biomimetics in restorative dentistry refers to the restoration of the lost tooth substance by a material that would mimic the original tooth structure (Slavkin 1996; Mann 1997; Magne and Douglas 1999). This concept opens innovative aspects in terms of restoring the biomechanical and esthetic properties in a way that resembles the natural dental hard tissues. In order to be able to mimic a tooth, one must understand its complex structure. Enamel, the hard tissue at the surface, covers the coronal part of the tooth and protects the underlying soft dentin, whereas enamel itself is a non-homogenous structure showing a decrease in mechanical properties from the surface to the enamel-dentin junction that separates it from dentin. Although the structure of enamel is strong, it is brittle and cracks may propagate within it; however, the presence of the enamel-dentin junction offers a smooth transition throughout the two surfaces and hinders crack propagation (Magne and Douglas 1999). Similarly, an ideal restoration should provide characteristics resembling those of enamel and dentin for protection of the underlying tissues and the opposing dentition as well. Indeed, a restoration should be

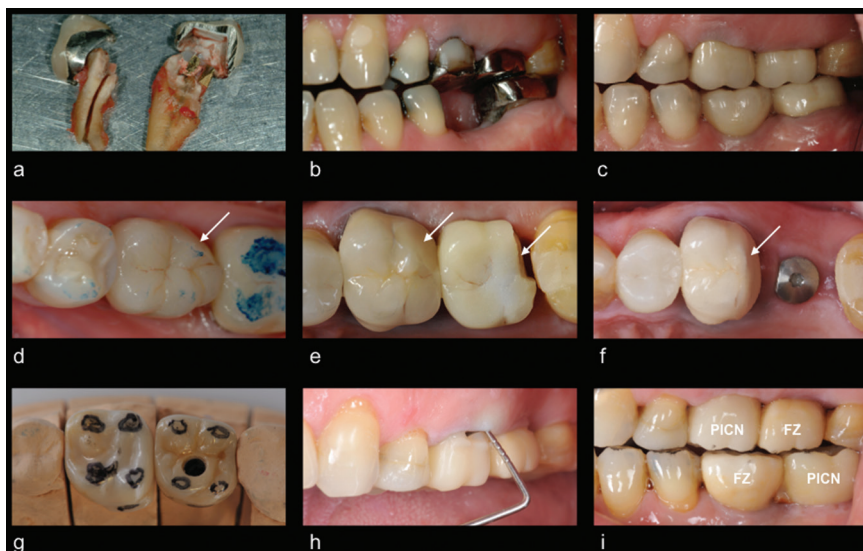


Figure 1. presents the history of a patient who showed unfavorable occlusal relationships and bruxism for over 12 years (a and b) Patient with root fracture #36 and extraction. (c) Realization of veneered zirconia crowns on teeth #26, #27, and #37 and implant #36. (d and e) Chipping (arrowed) on #26 and #36 after 6 months and on #27 after 3 years. (f) Root fracture #27 after 8 years. (g) Monolithic zirconia crowns #26, #27, and #36. (h) Root fracture on tooth #26 after 1 year. (i) Use of polymer-infiltrated ceramic network (PICN) material for crowns on implant #26 and implant #37 at a 2-year follow-up. This clinical case shows that improving material's resistance does not necessarily result in a better treatment performance (Clinical case, A. Mainjot).

strong enough to withstand masticatory forces, but in some cases such as bruxism, high strength restorations can engender complications resulting from concentration of stresses on the weakest link, resulting in root fracture as shown in Fig 1. In addition, an ideal restorative material should wear at the same rate as natural teeth to allow for the natural evolution of occlusion. For example glass-ceramics, with their heterogenous microstructure and high hardness, present a hazard on antagonistic teeth, while, on the contrary, composites usually wear at a rate higher than natural teeth (Swain et al. 2016).

1 | 3. Materials

3.1 CAD-CAM materials for restorative dentistry

3.1.1 Ceramics

All ceramic restorations are gaining popularity and are replacing porcelain-fused-to-metal restorations mainly because of their better esthetics and biocompatibility (Kelly 2008). The development of all ceramic restorations started in the 1980s and 90s when successful all ceramic restorations started to be produced by manufacturers, such as In Ceram (Vita Zahnfabrik , Bad Sackingen, Germany), Dicor (Dentsply-Sirona, York, USA) and Empress (Ivoclar Vivadent). Nowadays, all ceramic CAD-CAM restorations can be divided into 1- glass ceramics; 2- infiltrated ceramics and 3- polycrystalline ceramics (Fig.2).

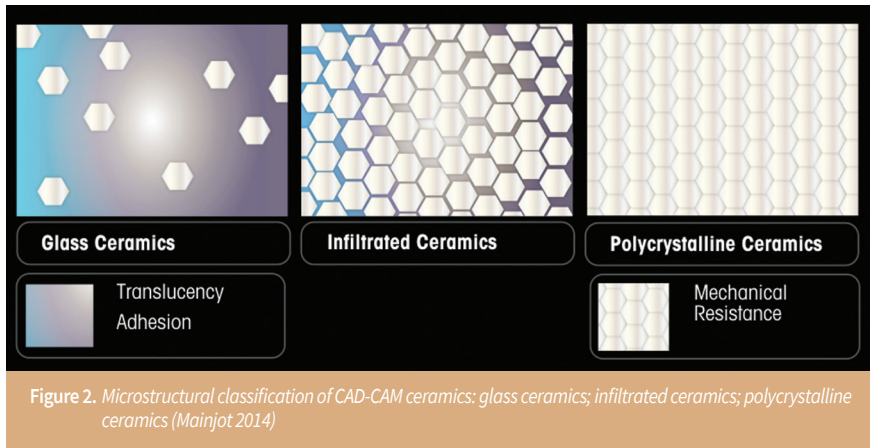


Figure 2. Microstructural classification of CAD-CAM ceramics: glass ceramics; infiltrated ceramics; polycrystalline ceramics (Mainjot 2014)

1- Glass-ceramics

Glass ceramics can be sub-divided into feldspathic ceramics, leucite-reinforced and lithium disilicate-reinforced glass-ceramics. The first CAD-CAM ceramic block was a feldspathic Vita Mark I block produced by Vita Zahnfabrik (Bad Sackingen, Germany) in 1985 for inlays. In 1991, the Vita Mark I blocks were substituted with the more resistant Vita Mark II which are still being marketed to date. Vita Mark II is formed by heating at a high plasticizing temperature and pressing to blocks, then sintering after drying slowly over a period of several days. Feldspathic ceramic, also known as aluminosilicate ceramic, contains ~40 % high melting glass such as albite making it highly translucent and biocompatible; however, its flexural strength, ranging between (100-160) MPa contraindicates its use in posterior load-bearing areas (Kelly and Benetti 2011; Li et al. 2014; Lambert et al. 2017). Leucite-reinforced glass-ceramic, on the other hand, contains (40-50) % leucite fillers, with a refractive index matching that of feldspathic glass, which makes it also highly translucent with superior esthetics. Its flexural strength is around 160 MPa and therefore recommended for single unit restorations in non-load bearing areas. It consists of (40-50) % leucite crystals (1-5 μm) which is known for its selective etching, therefore resulting in micro-irregularities at the surface that enhance the micromechanical interlocking with resin composite luting agents. Commercially known as Empress CAD (Ivoclar Vivadent, Schaan, Liechtenstein), which was produced in 2006 to replace its predecessor ProCAD, it is available in high and low translucencies and polychromatic blocks for single unit restorations. Another available leucite-reinforced glass-ceramic is Paradigm C (3M Oral Care, St. Paul, MN, USA) which has similar properties to Empress CAD (Kelly and Benetti 2011; Li et al. 2014; Lambert et al. 2017). The third class of CAD-CAM glass-ceramics is the lithium disilicate-reinforced, which contains ~70 % elongated-shaped crystals, offering additional resistance against fracture. Lithium disilicate-reinforced IPS emax CAD (Ivoclar Vivadent), also marketed in 2006, is supplied in a pre-crystallized blue state of low strength for the ease of machinability.

lity. These blue blocks, which are made of metasilicates and lithium disilicate nuclei, can be milled to the desired shape of the dental restoration. Afterwards, the milled dental restorations are placed in a dedicated furnace at a temperature above 850°C for further crystallization and dissolution of the metasilicates. This crucial step can lead to not only the elevated strength of ~360 MPa, but to also the final shade and translucency. They are recommended for single or multiple up to 3 units anterior and posterior (limited to premolars) restorations and implant abutments (Kelly and Benetti 2011; Li et al. 2014; Lambert et al. 2017). In addition to the CAD-CAM blocks, leucite and lithium disilicate reinforced glass-ceramics are also available as pressing ingots. However, the latter would require an extra step of making wax-patterns, which would require more time and laboratory involvement in the process (Brenes 2016). Glass-ceramics should be etched and silanized in order to achieve high bonding performance with resin composite luting agents (Nagai et al. 2005; Kalavacharla et al. 2015). This is considered as the gold standard method for bonding these restorations (El-Damanhoury and Gaintantzopoulou 2018). Later on when the patent went public, others manufacturers, such as Vita Zahnfabrik and Dentsply-Sirona (York, USA) introduced variations of CAD-CAM glass-ceramics consisting of lithium silicate/phosphate, marketed as Vita Suprinity and Celtra Duo, respectively. Both blocks are similar in microstructure with the dispersion of 10 % zirconia particles in the glass matrix, which turned out to not have a significant strengthening effect on the material in comparison with lithium disilicate IPS e.max CAD (Spitznagel et al. 2018a). There are many other glass-ceramic products available in the market now under different commercial names. Examples of those glass-ceramic commercial products include Obsidian (Glidewell Dental Laboratories, Newport Beach, USA), which is also a lithium silicate/phosphate reinforced glass ceramic and GC Initial LRF Block (GC, Tokyo, Japan), which is a leucite-reinforced glass-ceramic.

2- Infiltrated ceramics

Infiltrated ceramics were invented in 1987 by Sadoun, marketed by Vita Zahnfabrik until the early 2000s then gradually discontinued with the evolution of zirconia and CAD-CAM processes. They consist of a matrix of sintered polycrystalline ceramic, which is secondarily infiltrated by glass. Three forms existed, namely In-Ceram Alumina, Spinnell and Zirconia with a crystal content of 75 vol% at the expense of glass.

3- Polycrystalline ceramics

Polycrystalline, alumina and zirconia, are CAD-CAM ceramics composed entirely of densely packed crystals, which makes them stronger and tougher than glass-ceramics, but resistant to etching. The solid phase sintering process is accompanied by shrinkage which must be accommodated for while designing the restoration, thus they cannot be processed by any means other than CAD-CAM processes (Kelly and Benetti 2011;

Li et al. 2014). Regarding Alumina, it has been available in the market since the 1990s as full alumina frameworks for anterior crowns and bridges up to 3 units. However, even though it has a lower refractive index, and so showing better optical properties, it is currently being replaced with zirconia restorations (Mainjot 2014). Zirconia, on the other hand, consists of more than 90 % zirconium oxide with other impurities such as titanium, silicon and iron oxides (Santander S. 2010). The zirconium oxide which comprises the majority of zirconia exists in 3 crystal structures: 1) monoclinic at room temperature; 2) tetragonal above 1130°C and 3) cubic above 2370°C. Upon cooling, phase transformation, accompanied by a 3-4 % volume expansion, occurs from transformation of zirconia from tetragonal to monoclinic phase. The addition of dopants, such as yttrium oxide, stabilizes the tetragonal phase at room temperature; however, yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) can change into monoclinic phase under induced stresses resulting in a phenomenon called transformation toughening. The accompanying expansion of crystals from the tetragonal to monoclinic transformation leads to the development of compressive stresses which in turn closes the formed cracks and enhances the fracture toughness, thus zirconia is considered as the toughest dental ceramic (Santander S. 2010; Li et al. 2014; Mainjot 2014; Lambert et al. 2014). This tetragonal to monoclinic transformation can also occur in the absence of stresses in the oral environment enhanced by prolonged exposure to moisture at low temperatures resulting in a phenomenon called low-temperature degradation which, on the contrary, results in a deleterious decrease in strength (Chevalier et al. 2011; Sanon et al. 2013; Mainjot 2014). Compared to other ceramics, polycrystalline ceramics, especially the first generations of zirconia restorations (3Y-TZP), are considered the strongest and toughest restorative materials (flexural strength: 0.9-1.5 GPa, toughness: 3.5-4.5 MPa.m^{1/2}); however, this comes at the expense of esthetics and translucency, therefore they are used mainly for frameworks. Increasing the yttria content in the next generation of zirconia restorations (4Y-TZP) showed higher translucency at the expense of the mechanical properties (flexural strength: 0.6-1.0 GPa, toughness: 2.5-3.5 MPa.m^{1/2}), while the third generation (5Y-TZP) showed further improvements in esthetics at the expense of the mechanical properties (flexural strength: 0.5-0.9 GPa, toughness: 2.2-2.7 MPa.m^{1/2}). Both the second and third generations can be used in monolithic restorations (Zhang and Lawn 2018).

3.1.2 Resin composites

Resin composites (RC) are considered one of the most important and widely used materials in dental restoratives applications. They consist of inorganic glass-fillers embedded in an organic matrix. Among their applications are restoration of teeth and cementation of restorations; the former composites have high viscosity while the latter are flowable. The main difference between both types is the filler content, which is

higher in packable composites used in restorations to withstand high masticatory forces, while cementing composites should flow easily within the microroughness created on the tooth and the restoration for proper bonding (Ferracane 2011). Silica fillers are added to the resin matrix to enhance the mechanical properties and reduce the polymerization shrinkage. These fillers are coated with a silane coupling agent, which would allow free-radical addition polymerization reaction to occur, resulting in chemical bonding between the inorganic fillers and the organic matrix. The organic matrix is composed of highly viscous monomers such as bis-GMA and urethane dimethacrylate (UDMA), so diluents such as tri-ethylene-glycol-dimethacrylate (TEGDMA) and 2-hydroxyethyl methacrylate (HEMA) are sometimes added. The polymerization reaction of RC can be activated by blue light in the presence of a photo-initiator such as camphorquinone. Alternatively, this reaction can be activated chemically through an organic amine supplied as a separate catalyst paste. The polymerization reaction is well known to be accompanied by polymerization shrinkage that may subsequently lead to microleakage and failure at the margins of the restoration (Ferracane 2005; 2011). According to Cramer et al. (2011), polymerization shrinkage is considered to be the primary cause of failure of direct RC as a result of internal stresses and microleakage. The amount of C=C double bonds converted to C-C single bonds determines the degree of conversion of RC (Ribeiro et al. 2012), and light curing of direct RC could result in a degree of conversion ranging from (35-77) %, depending on several extrinsic parameters such as the efficacy of the LED used and the irradiation time, as well as intrinsic parameters such as viscosity of the organic matrix and the fillers load, size and refractive index (Leprince et al. 2013). This creates a variation in the mechanical and the optical properties of the restoration. Although the amount of unreacted monomer cannot induce an adverse systemic effect, it was found that small amounts of the diluent materials TEGDMA and HEMA might cause soft tissue irritation, trigger an allergic reaction upon contacting the gingiva and mucosa, induce bacterial growth, impair the repairing process of secondary and reparative dentin and induce pulpal inflammation, in addition to the possibility of causing genotoxic and cytotoxic effects (Krifka et al. 2013).

Classification of resin composites

According to the filler size, RC could be classified into: traditional RC with large filler particles having a rough surface and poor esthetics, microfilled RC with smaller filler particles having a smoother surface but lower mechanical properties and hybrid RC with different sizes of filler particles combining the properties of both (Ferracane 2011). According to the classification proposed by Willems et al. (1992), five categories of commercial RC were divided according to their filler content into: Densified Composites; Microfine Composites; Miscellaneous Composites; Traditional Composites and Fiber-Reinforced Composites. While Microfine Composites contain fillers with a small mean

particle size ($\sim 0.04 \mu\text{m}$) and a low volume fraction, Densified composites have larger fillers with a higher volume fraction. Densified composites are further subdivided into Midway-Filled ($<60 \text{ vol}\%$) and Compact-Filled ($>60 \text{ vol}\%$), in which each of the 2 sub-classes is further subdivided according to the mean particle size into Ultrafine ($<3 \mu\text{m}$) and Fine ($>3 \mu\text{m}$) (Willems et al. 1992). Nguyen et al. (2013) found that increasing the filler volume fraction increases the mechanical properties within limits, in which incorporation of fillers over the limit did not have a significant influence on the flexural strength, hardness or fracture toughness. This might be due to difficulties encountered with a high volume fraction of fillers that led to the incorporation of porosities.

The broader and more general classification of RC is direct and indirect composites. As the name suggests, direct RC are applied directly into the cavity and light cured. Since they provide a degree of conversion ranging between (35-77) % (Leprince et al. 2013), which is considered relatively low, they must be applied into the cavity in small increments that are light-cured separately to minimize the effect of polymerization shrinkage. Larger cavities such as inlays, onlays and veneers can be restored by indirect composite restorations which are fabricated and cured outside the mouth after taking an impression, then cemented into the cavity. Indirect RC can be classified into artisanal and CAD-CAM composites (Mainjot et al. 2016). Artisanal composites are basically direct composites that are built up and photo-polymerized in increments extra-orally, which eliminates the negative effect of polymerization shrinkage on the tooth. Governed by several operator-dependent variables such as the light-curing unit, distance from the source and time of exposure, artisanal composites offer limited degree of conversion, which also varies from the surface to the depth of the restoration, influencing the mechanical properties and resulting in a non-homogenous restoration from the surface to the depth and between one restoration and another of the same composite material (Mainjot et al. 2016). Post-curing of artisanal composites results in only a limited increase in the degree of conversion due to the limited diffusion and reactivity of free monomers after light-curing (Leprince et al. 2013). CAD-CAM composites, on the other hand, are industrially fabricated under controlled environmental conditions resulting in a more homogenous block that is secondarily milled into the desired shape (Giordano 2006; Mainjot et al. 2016). CAD-CAM composites show superior mechanical properties than artisanal composites due to; 1) the ability to incorporate a larger volume percentage of fillers than artisanal composites, which need to be plastic enough to be shaped into the desired restoration (Mainjot et al. 2016); 2) the mode of polymerization of CAD-CAM composites which involves high temperature (HT) ($>100^\circ\text{C}$) which is sometimes accompanied by high pressure (HT-HP) ($>300 \text{ MPa}$) (Nguyen et al. 2014). HT-HP was shown to offer a high degree of conversion ($>96 \%$) (Phan et al. 2014) which in turn enhances the mechanical properties and results in homogenous blocks with less porosities and flaws. The application of HT-HP to 4 commercially available direct

light-cure (LC) composites was found to significantly increase their flexural strength, hardness and fracture toughness. The accompanying increase in density of the RC subjected to HT-HP in comparison to their LC versions suggested that an increase in the density of the organic matrix occurred due to a decrease in the flaws which, in turn, enhanced the mechanical properties (Nguyen et al. 2012). CAD-CAM composites can be sub-divided according to their microstructure into dispersed fillers (DF) and polymer-infiltrated ceramic network (PICN) materials. DF composites consist of silica-based glass dispersed in UDMA and TEGDMA, which are then polymerized under high-temperature ($>100^{\circ}\text{C}$). On the other hand, PICNs are made of pre-sintered glass-ceramic blocks infiltrated with UDMA and TEGDMA, which are polymerized under HT-HP (180°C- 300 MPa) (Fig 3 and 4). The idea of infiltrating a ceramic scaffold with

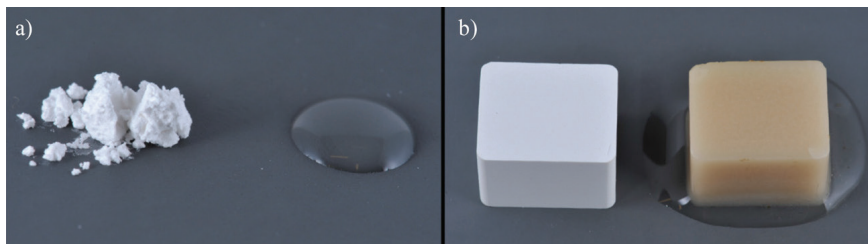


Fig. 3 a) Fillers that are incorporated by mixing in a monomer mixture to produce a dispersed filler (DF) composite block.
 b) A pre-sintered glass-ceramic scaffold infiltrated with monomer, which is secondarily polymerized under high-temperature and high-pressure to produce a polymer-infiltrated ceramic network (PICN) block. In PICN, glass-ceramic particles are interconnected creating a double-network material (which has a lower content of organic phase than DF).

Figure 4. Schematic illustration of the microstructure of a composite with dispersed fillers (DF) incorporated by mixing versus a polymer-infiltrated ceramic network (PICN) material. In PICN, the ceramic network (white color) constitutes a 3-dimensional scaffold of sintered ceramic particles, which form a real skeleton (Mainjot et al. 2016)

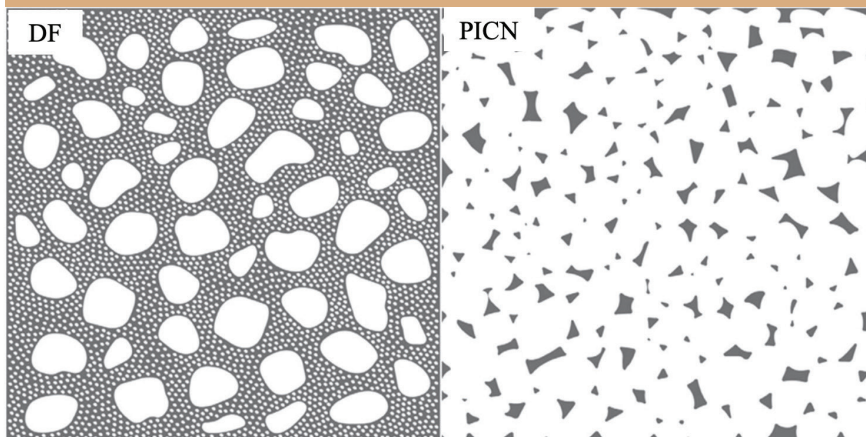


Table 1 Some of the commercially available CAD-CAM composites in the market and their organic matrix and filler compositions. Data were completed according to the manufacturers information when possible and completed following (Alamouh et al. 2018; Hampe et al. 2018; Lucsanzsky and Ruse 2019; Mainjot et al. 2016). bis-GMA: bisphenol A glycidylmethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylglycol dimethacrylate; bis-EMA: Ethoxylated bisphenol A dimethacrylate.

Commercial name	Manufacturer	Organic matrix	Inorganic fillers
Paradigm MZ100	3M ESPE (St. Paul, MN, USA)	bis-GMA + TEGDMA (light-cured)	Silica (0.6 μm) + zirconia (0.6 μm) (85 wt%)
Lava Ultimate	3M Oral Care (St. Paul, MN, USA)	UDMA	Silica (20 nm) + zirconia (4-11 nm) + zirconia-silica clusters (0.6-10 μm) (79 wt%)
Cerasmart	GC (Tokyo, Japan)	UDMA + other DMA	Silica-based glass + silica (20 and 300 (71wt%))
Shofu block	Shofu (Kyoto, Japan)	UDMA + TEGDMA	Silica-based glass + silica (61 wt%)
Brilliant Crios	Coltene (Göppingen, Germany)	bis-GMA + bis-EMA + TEGDMA	Glass + amorphous silica (70 wt%)
Tetric CAD	Ivoclar Vivadent (Schaan, Liechtenstein)	bis-GMA + bis-EMA + TEGDMA + UDMA	Barium glass (<1 μm) + SiO ₂ (<20 nm) total fillers volume 51 vol%
Grandio	Voco GmbH (Cuxhaven, Germany)	UDMA + other DMA	Nanohybrid fillers (86 wt %)
Katana Avencia	Kuraray Noritake (Tokyo, Japan)	UDMA + TEGDMA	Al ₂ O ₃ (20 nm) + SiO ₂ (40 nm) (62 wt%)
KZR-CAD-HR2	Yamakin Co., Ltd. (Osaka, Japan)	UDMA + TEGDMA	SiO ₂ (20 nm) + aggregated SiO ₂ -Al ₂ O ₃ -ZrO ₂ (20-60 μm) cluster (1-6 μm) + fluoride particles (700 nm)
CAMouflage NOW	Glidewell Dental Laboratories (Newport Beach, USA)	No available data	No available data
Mazic Duro	Vericom Co., Ltd. (Gyeonggi-do, Korea)	No available data	80 wt% nano-particle sized ceramic fillers (zirconia, silicate)
Vita Enamic	Vita Zahnfabrik (BadSäckingen, Germany)	UDMA + TEGDMA	Glass-ceramic sintered network (86 wt% - 75 vol%)

organic monomers was inspired by Giordano from infiltrated ceramics (In-Ceram) in 1997, replacing the glass with monomers, therefore creating a skeleton-like structure that can distribute stresses in all directions. The main drawback of monomer infiltration into ceramics is usually due to polymerization shrinkage, which would lead to the development of cracks and reduced mechanical properties. To avoid this drawback, Sadoun (2011) developed a new method of polymerization in which HT is accompanied

by HP to compensate for the shrinkage. It was found that increasing the pressure, within limits, decreases the intermolecular distances between free monomers and brings them closer together, in addition to the influence on the degree of crosslinking which might have also occurred (Nguyen et al. 2012).

Vita Enamic (Vita Zahfabrik, BadSäckingen, Germany) is the only marketed PICN, while other composite DF blocks are marketed under different commercial names. Some examples of commercially available DF are listed in *Table 1*.

3.1.3. Ceramic versus resin composite materials for CAD-CAM restorations

Monolithic esthetic CAD-CAM restorations can be either glass-ceramics or RC, and recently translucent zirconia restorations have been introduced. They differ in their mechanical and optical properties, which is why each case should be treated separately according to different factors evaluated by the dentist to select the material most suitable for a given situation. Indeed, the mechanical properties of glass ceramics, represented by flexural strength, modulus and hardness, are higher than those of RC, with superior optical properties, as well (Lawson et al. 2016; Maunula et al. 2017; Wendler et al. 2017). Additionally, glass-ceramics offer high levels of esthetics, especially with their high translucency and high surface gloss after glazing. Unlike RC restorations, this highly glossed surface can be attained for long periods with high resistance to abrasive methods, such as the use of tooth brushes (Mormann et al. 2013). However, glass-ceramics are brittle materials with relatively low fracture toughness and may fail in the presence of flaws, therefore not recommended to be milled in thin sections, especially in the cases of minimally invasive treatment of worn dentition (Chen et al. 2014). Moreover, ceramics need an extra step following milling, in that case crystallization for glass-ceramics and sintering for zirconia, thus increasing chair-time and cost. However, PICN do not require that extra step and can be placed directly as a restoration (Mainjot et al. 2016). Despite the superiority of glass-ceramics over CAD-CAM composites in terms of mechanical and optical properties, yet composites have a lower elastic modulus, in particular PICN which demonstrates elastic modulus values between those of enamel and dentin, allowing them to readily absorb shocks arising from masticatory forces (Ruse and Sadoun 2014; Awada and Nathanson 2015; Goujat et al 2018). Since they are RC based ceramics, they can be easily adjusted in the mouth, as well as they allow the addition of direct composite onto their surfaces if required. Furthermore, their low hardness values cause no harm to the opposing enamel, unlike glass-ceramics which can cause wear to opposing enamel especially in cases of bruxism (Mormann et al. 2013; El Zhawi et al. 2016; Naumova et al. 2017). Another important aspect of CAD-CAM composites is their edge chipping resistance which is higher than that of ceramics, even though glass-ceramics have higher values of flexural strength, hardness and fracture toughness than composites. This

could be attributed to the higher brittleness of ceramics and the lower elastic modulus achieved via polymer infiltration, which in turn will absorb the cracks introduced by the bur while milling and form a plastic zone at the crack tip. This property indicates that CAD-CAM composites can be milled at a faster rate than ceramics, and most importantly they offer smoother margins especially in thin restorations such as in the treatment of worn dentition (Chavali et al. 2017; Curran et al. 2017; Pfeilschifter et al. 2018). More specifically, Coldea et al. (2015) tested the damage tolerance of PICNs and found that they have the ability to tolerate adjustments by burs with low strength reductions compared to the initial strength. It was also shown that the damage tolerance of PICN was higher than feldspathic and glass-ceramics.

3.2 Resin composite luting agents

The evolution towards partial bonded restorations necessitated the evolution of adhesive cements for the retention of restorations. Adhesive cements evolved since the early 1900s from zinc phosphate cements, to zinc polycarboxylate cements in the 60s and glass ionomer cements in the 70s (Diaz-Arnold et al. 1999; Baig and Fleming 2015). Later on, the use of resin composite luting agents (RCLA) started to gain popularity in the field of adhesive dentistry. While bonding of RCLA to enamel is simple and consistent, bonding to dentin is a challenging process due to several factors which include the high amount of organic collagen, irregularity of the inorganic hydroxyapatite crystals and the projection of dentinal tubules which pump fluids with a relatively high intrapulpal pressure, thus creating a smear layer (0.5-5.0 μm thick) when cutting through dentin that may interfere with the adhesive resin (Swift et al. 1995). Micromechanical interlocking occurs after the penetration and polymerization of the RCLA into the microporosities created by etching the surfaces of enamel and dentin by a process termed hybridization (Van Meerbeek et al. 2003; Rosa et al. 2015). RCLA could be classified into etch-and-rinse, e.g., Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein); Calibra (Dentsply Caulk, York, Pennsylvania, USA); Nexus (Kerr, Detroit, Michigan, USA), or self-etch, e.g. Panavia 21, Panavia F and Panavia F 2.0 (Kuraray Noritake, Tokyo, Japan); Multilink (Ivoclar Vivadent, Schaan, Liechtenstein). A third sub-class of RCLA was developed later on, the self-adhesives, e.g. RelyX Unicem (3M Oral Care, St. Paul, MN, USA) (Van Meerbeek et al. 2003; Radovic et al 2008). The etch-and-rinse adhesives involve etching of the tooth with phosphoric acid or a conditioner which dissolves the inorganic hydroxyapatite crystals at the dentin surface leaving a collagen network. A primer is then applied, followed by an adhesive, and the resulting bond is micromechanical between the exposed collagen network and the polymerized RCLA; therefore, it is a 3-step procedure which is sometimes simplified into 2 steps by combining the primer and the adhesive into one single bottle, followed by application of RCLA (Van Meerbeek et al. 2003; Breschi et al. 2018). The application of the self-etch involves dissolving of

the hydroxyapatite by acidic functional monomers and the infiltration of those monomers into the microroughness at the same time. According to their degree of acidity, self-etch adhesives could be further sub-divided into strong (pH <1) and mild (pH ~2) self-etch adhesives. When bonding to dentin, strong self-etch adhesives result in complete dissolution of the hydroxyapatite crystals resulting in a similar bonding mechanism to etch-and-rinse, i.e. hybridization with organic collagen. On the other hand, mild self-etch adhesives partially demineralize exposed dentin, leaving behind residual hydroxyapatite crystals within the porous collagen network, which may enhance a chemical bonding mechanism with the functional monomers (for example phosphate-based monomers) in addition to the micromechanical bond (Van Meerbeek et al. 2003; Takamizawa et al. 2018). While the etch-and-rinse adhesives could be considered as highly effective with enamel, the mild self-etch adhesives are believed to result in more effective bonding with dentin than the etch-and-rinse because of the added value chemical bonding might bring, in addition to the protection provided by the hydroxyapatite crystals against hydrolysis of the collagen (Van Meerbeek et al. 2003). Alternatively, self-adhesive cements are applied directly on the tooth without prior pretreatment, therefore it is a simple procedure and saves time for the dentist with less room for mistakes. It is a cement with a low pH and high hydrophilicity in its early stages, with a limited self-etch effect and a superficial interaction with the smear layer. As the reaction progresses, the pH increases by the reaction of the acidic functional monomers with the smear layer covering the dentin surface and with the metal oxides in the fillers, leading to neutralization of the cement and a change from hydrophilic to hydrophobic nature, which is beneficial to minimize water sorption, hygroscopic expansion and hydrolytic degradation (Radovic et al. 2008; Ferracane et al. 2011; Manso and Carvalho 2017; Shim et al. 2017).

1 | 4. CAD-CAM materials mechanical and bonding properties *in vitro* testing

4.1 Testing of the mechanical properties of CAD-CAM materials

Testing of the mechanical properties of dental restorative materials can be performed *in vitro* by a number of methods, to evaluate various physical properties of the materials.

4.1.1. Flexural strength

A very common method for testing ceramics and composites is the 3-point flexural strength test due to its simplicity. This test is recommended by the Academy of Dental

Materials, and is utilized by manufacturers prior to launching restorative materials. In this method, samples are cut into bars, which are polished on the tension side and supported on 2 rollers with a specified span distance in a universal testing machine (Quinn 1992; Ilie et al. 2017). Force is applied on the top of the samples at its center (Fig 5) and the load is recorded at failure using a computer controlled software in which the flexural strength (σ_f) (MPa) is derived from the formula:

$$\sigma_f = \frac{3FL}{2hc^2},$$

where F (Newton) is the load at fracture, L (mm) the span, h (mm) the specimen width, and c (mm) the specimen height

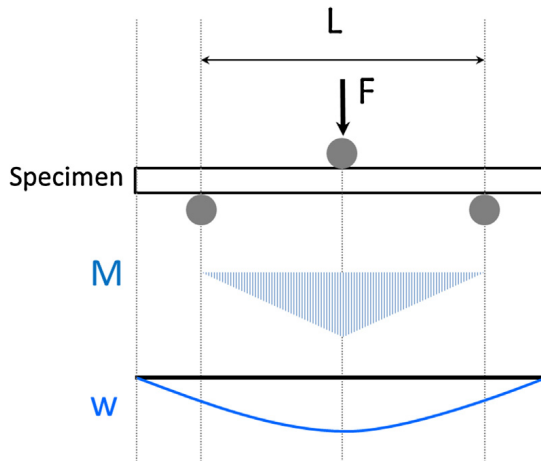


Figure 5. Schematic representation of the 3-point bending test. The figure was reprinted from Ilie et al. (2017) with permission from Elsevier

Beside flexural strength, 3-point bending configuration is considered the most common method for the determination of the flexural modulus, which is the ability of a material to withstand deformation under load (Ilie et al. 2017). This is done by attaching an extensometer to the 3-point bending device to avoid the compliance and computing the flexural modulus (E) (GPa) using the formula:

$$E = \frac{FL^3}{4hc^3d},$$

where d (mm) is the deflection corresponding to load F (Newton) at the end of the curve, in which part of the curve behaves in an elastic manner (linear portion) and the rest of the curve behaves in a plastic manner.

Another property obtained via the 3-point flexural test is the flexural load energy (U_r), which can be calculated using the formula:

$$U_r = \frac{F\Delta}{2},$$

where Δ is the maximum deflection at fracture (mm)

Flexural load energy is the maximum amount of stresses a material can withstand before it fractures, which gives an idea about the material's shock absorbing or damping effect.

4.1.2. Hardness

Hardness is not an intrinsic property of dental materials; it is a property of the surface that is governed by the type of test, indentation load and time (10-15 seconds). Hardness testing differs according to the type of indenter used, such as Vicker's, Rockwell, Knoop and Brinell (Fig 6). An indenter is loaded for a specific amount of time at the material's surface after polishing, then the hardness value is calculated from the size or depth of the print or the indentation created after the removal of the indenter (Ilie et al. 2017).

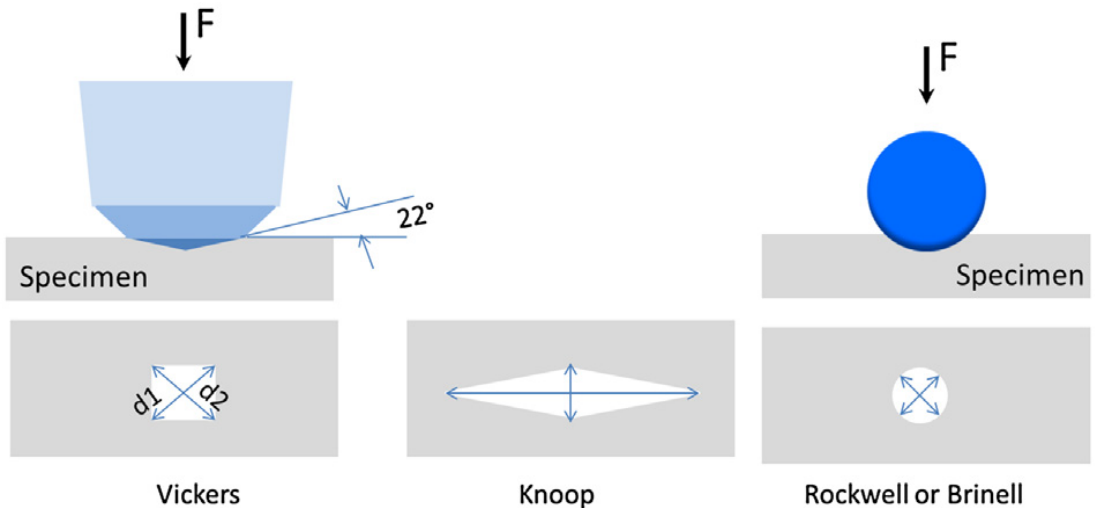


Figure 6. Different indentation techniques producing a print or an indentation on the surface of samples. The figure was reprinted from Ilie et al. (2017) with permission from Elsevier

4.1.3. Fatigue

The failure of dental restorations occurs through cyclic loading over a period of time at relatively low (sub-critical) loads causing sub-critical crack propagation. These sub-critical cracks are also enhanced by chemical and biological stresses over time and fracture often results from all factors acting together towards catastrophic failure. In that case, fatigue is considered the most important clinical criteria of mechanical properties testing; thus, it is important to determine the fatigue limit of a material in relation with its use, which is the stress below which the speed of sub-critical crack propagation is very low in relation to life expectancy. However, fatigue testing of dental materials is time consuming and costly, therefore is not widely utilized. There are 2 currently known methods of fatigue testing, namely fatigue limit and staircase method. In the fatigue limit, a sample is loaded to failure at a stress close to its static tensile or flexural strength, then the stress is reduced in the next sample. This goes on until a sample does not break at a given number of cycles e.g.; (105-106) cycles and this load at which the material does not fail is then identified as the fatigue or endurance limit (ASTM D7774-12 2012; ASTM D7791-12 2012; Kelly et al. 2012). The staircase method, on the other hand, is less time consuming than the fatigue limit and is more commonly used by dental researchers. Yet, the insight it provides on the fatigue properties of materials is not as wide as that of the classic fatigue limit approach (Kelly et al. 2012). The staircase method for fatigue testing of dental materials was introduced by Braem et al. (1994) to study the *in vitro* flexural fatigue behavior of RC. The maximum amount of cycles is set, for example 10,000 cycles, and the first sample is loaded at a given amount of stress, which is usually (50-60) % of its static flexural strength. If the first sample does not fracture, a second sample is loaded with a higher stress, but if it fails, then the stress is reduced. This process continues for the given amount of samples to be tested and a plot is drawn to identify the range of fatigue strength of the material (Braem et al. 1994; Garcia-Godoy et al. 2012; Belli et al. 2014; Ilie et al. 2017).

4.1.4. Wear resistance

For RC restorative materials, the mechanical properties such as flexural strength, elastic modulus, fracture toughness and hardness influence the wear resistance of the restoration, as well as the antagonistic natural teeth. To date, there is no standardized *in vitro* method to qualify wear measurement in dental restorations. However, researchers in the dental field develop their own chewing simulators, but their results cannot be comparable to one another due to the difference in force, frequency, direction of movement, number of cycles, as well as other variables such as soaking medium and temperature (Ilie et al. 2017). The analysis of wear is usually performed using digital profilometry to produce a

cloud of points of the surface scanned. At different time intervals, the data obtained are superimposed with the baseline to calculate the amount of wear using special matching softwares. However, differences among studies exist in terms of procedures for replication of teeth, scanning and the superimposition matching softwares, which are also limited in terms of accuracy and precision to a maximum of 30 μm *in vitro* (Wulfman et al. 2018).

4.1.5. Fracture toughness

Measuring the fracture toughness is important in evaluating the mechanical properties of dental materials, in which the critical stress intensity factor (K_{IC}) is a measure of the amount of stresses concentrated at a tip of a crack that initiate catastrophic failure. Fracture toughness is an inherent property of a material and since all materials contain flaws with varying degrees, the resistance of propagation of cracks from such flaws is known as the fracture toughness (K_{IC}), which is stress at which catastrophic failure or irreversible damage of the material occurs. For ceramic dental materials, which are considered brittle materials, failure occurs in an opening mode or a tensile manner, which is designated the subscript (I) in laboratory testing, thus the unit of measurement of the fracture toughness of dental materials is always given as K_{IC} (Cesar et al. 2017; Ilie et al. 2017).

Different methods for evaluating the fracture toughness

Several testing methods have been implemented for determining the fracture toughness of dental materials. Among these tests are i) the indentation fracture test; ii) the single edge “V” notch beam; iii) the surface crack in flexure; iv) the compact tension and v) the chevron notch beam which was modified to several versions including the notchless triangular prism (NTP) specimen fracture toughness K_{IC} test (Ruse et al. 1996; Sehgal and Ito 1998; Cesar et al. 2017; Ilie et al. 2017).

- i) The indentation fracture test is considered the simplest method in evaluating the fracture toughness of ceramics. It involves polishing of one surface of the material then introducing an indentation using the Vicker’s indenter (Fig 7). It must be noted that, cracks should be measured as soon as possible to avoid subcritical crack growth which may occur due to humidity (Sehgal and Ito 1998; Cesar et al. 2017; Ilie et al. 2017).
- ii) The single edge “V” notch beam (SEVNB) test is a common method for testing dental ceramic and composite materials, which is widely used due to its simplicity. Samples are cut into bars and a notch is introduced on the polished side of the bar, in the tension part. The sample is placed in the flexural testing device to open the notch and the K_{IC} is calculated (Fig 8). The main limitation of this test is the difficulty of positioning the notch in the same straight line with the applied load, which might introduce cracks further



Figure 7. A Vicker's indentation with projecting cracks performed on a sample of a ceramic. The figure was reprinted from Sehgal and Ito (1998) with permission from John Wiley & Sons-Books

away from the created notch. A modification of this test is the single edge pre-cracked beam, in which Vicker's indentations are introduced on the polished side, then the sample is loaded in a special pre-cracking device to introduce an initiation crack or a notch, then fracture toughness is calculated afterwards from the failure load. There are criteria for the required sample size, in which the samples must have certain dimensions for the test to be valid (ASTM E1304-9 2014; Cesar et al. 2017; Ilie et al. 2017).

- iii) Surface crack in flexure combines the flexure testing and indentation methods, in which the initiation crack is introduced via Knoop indentation to control the surface flaws (Cesar et al. 2017).

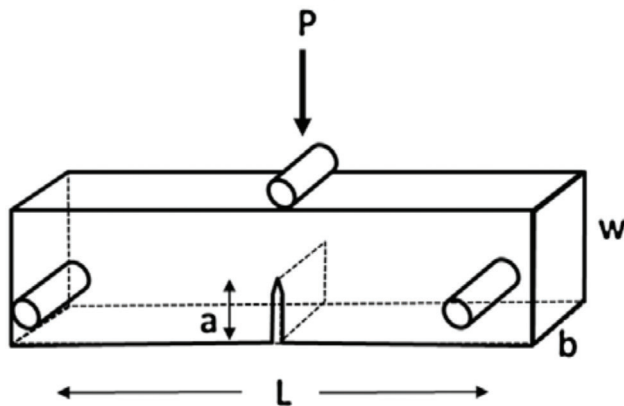


Figure 8. A bar loaded in flexion with force (P) applied on the opposite side of a pre-formed notch. The figure was reprinted from Ilie et al. (2017) with permission from Elsevier

- iv) In the compact tension test, samples are in the form of beams with a notch in the center and 2 holes on the top and bottom of the notch where a tensile force is applied to open the notch and the fracture toughness is calculated (Fig 9). Similar to the SEVNB, there are criteria for the required sample size, in which the samples must have certain dimensions for the test to be valid (ASTM D5045-14 2014; Ilie et al. 2017).

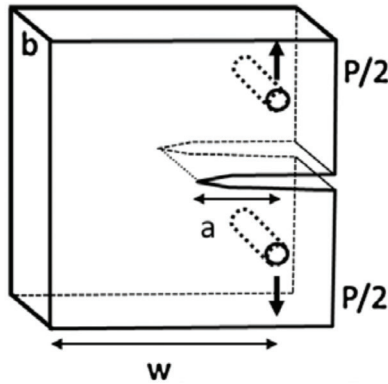


Figure 9. A sample loaded in compact tension, with arrows pointing in the opposite directions from the notch. The figure was reprinted from Ilie et al. (2017) with permission from Elsevier

- v) The chevron notch beam test is a popular method for evaluating the fracture toughness of dental materials which requires small specimens; thus, more economic in terms of material consumption. The cylindrical specimens are introduced with a "V" shape chevron notch from which the crack will propagate till catastrophic failure (Fig 10) (ASTM E1304-9 2014; Cesar et al. 2017; Ilie et al. 2017).

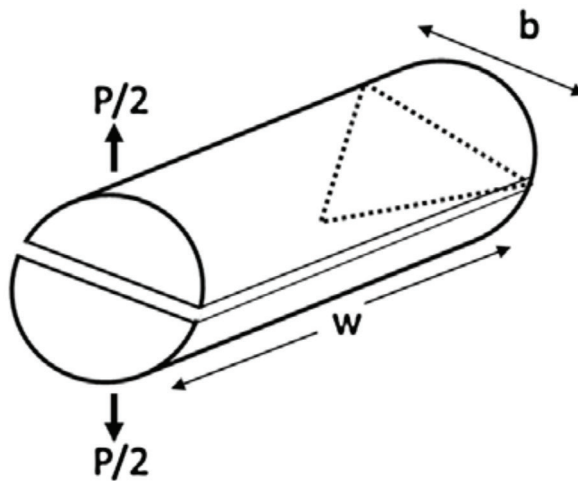


Figure 10. A sample loaded in the chevron notch beam test with arrows indicating force (P) application in a tensile mode. The figure was reprinted from Ilie et al. (2017) with permission from Elsevier

One of the modifications of the chevron notch beam is the notchless triangular prism (NTP) specimen K_{IC} test. This modification was validated through finite element analysis for measuring the fracture toughness and adhesive interfaces of dental materials. The samples are presented in the form of 6-mm a side, 12-mm long triangular prisms, representing the chevron shape of the chevron notch beam. The NTP test is considered a simple method because it eliminates the need to create a notch; hence the term notchless. Only a crack initiation point is introduced from which the crack will propagate and which was shown, via finite element analysis, to concentrate the maximum amount of stresses allowing for stable crack growth at a low cross-speed. It is noteworthy to mention that the size of the samples used for the NTP specimen K_{IC} test enable the application to small, dentally relevant structures (Ruse et al. 1996); however, the initial cost of the materials for manufacturing and testing of the prisms is relatively high and their preparation needs low tolerance (Ilie and Ruse 2019).

4.2 Testing of the bonding properties of resin composite luting agents to CAD-CAM materials

Bond strength testing of dental materials is a complicated process due to the large number of variables encountered while performing the testing. Not only does this large number of variables complicate the process of bond strength testing, but it also makes comparing the results of different studies almost impossible. Those variables include, but not limited to, the tooth structure, type of restorative, pre-treatment conditions, post-bonding storage or aging procedure and the type of bond strength test to be performed (Armstrong et al. 2010; Roeder et al. 2011). While pre-treatment methods include physical and chemical procedures, micromechanical interlocking induced by etching or sandblasting is considered the most important means of bonding for dental materials. Etching or gritblasting create microroughness for the penetration of the cement before polymerizing and hardening into a solid mass within the substrate. Photo-polymerization of samples while testing *in vitro* should also follow some guidelines, in which the light-curing unit should be efficient enough to ensure a high degree of polymerization of the resin composite luting agent and it should be held perpendicularly and in close proximity to the curing unit to avoid overlapping light curing (Armstrong et al. 2017).

Post-bonding conditions such as water storage and thermocycling also affect the outcome of bond strength testing. It is important to subject the samples to some form of aging prior to testing in order to simulate mouth conditions. Besides, those harsh

conditions may lead to spontaneous de-bonding of some samples, which should then be recorded as pre-test failures and given a zero value. According to the Academy of Dental Materials guidance, the recommendations of aging of bonded samples require a minimum of 10,000 cycles in aqueous media between 5 and 55°C, starting after 1-7 days of storage in aqueous media at 36-37°C. The exposure in each bath should be at least 20 seconds and the transfer time between baths should be as short as possible (5-10 seconds) (Roeder et al. 2011; Armstrong et al. 2017). Certainly, a strong bond between the tooth and the restoration is crucial for the survival of prosthetic restorations, which is why a reliable bond strength test is needed for the verification of the adhesive assembly. In the past, bond strength testing was performed using the macro test methods, shear and tensile mainly. Later on, micro test methods were developed to overcome the increased numbers of cohesive failures which do not contribute to the actual bond strength of the interface (Braga et al. 2010). Additionally, the internal flaws introduced at the adhesive interface are reduced with the smaller samples of microtensile bond strength test (Neves Ade et al. 2008). Although numerous studies have been performed using the shear and microtensile bond strength tests, yet there has not been an established standardized protocol (Armstrong et al. 2010), which would be very complicated to establish with respect to the different methodological parameters. However, comparisons between different materials and mutual ranking of bond strength provide relevant and meaningful data (Sudsangiam and van Noort 1999).

4.2.1. Shear bond strength test

Shear bond strength test (SBS) was the most commonly used method for bond strength and is still used by manufacturers for gathering information about the bonding performance of adhesives because it is easy and quick. It is a simple test and the samples are easy to prepare and require no further modifications after bonding. However, the high amounts of cohesive failures in dentin associated with it, and the fact that stresses are not uniformly distributed at the adhesive-tooth interface, raise doubts regarding the validity of its results (Della Bona and van Noort 1995; Van Meerbeek et al. 2010). In an attempt to reduce the amounts of cohesive failures and make this test more reliable, the micro-SBS was introduced which turned out to be a worse rather than a better representation of the existing model. Placido et al. (2007) proved via finite element analysis the presence of non-uniform stress distribution in both types of SBS. Those non-uniform stresses resulted in a predominance of tensile over shear stresses, which were more apparent in the micro-shear rather than the conventional SBS test due to the relatively higher thickness of the adhesive (Armstrong et al. 2010) which renders it a less popular type of test.

4.2.2. Microtensile bond strength test

The microtensile bond strength test (MTBS) was first developed for metal alloys, which was then modified for several other applications in the medical field before being adapted by Sano et al. (1994) for dental use. Since then, dental researchers have been using it widely for its efficacy when compared to conventional SBS. MTBS represents a more accurate modification of the tensile bond strength test (TBS), in which the adhesive interface is smaller, leading to less flaws while preparing the samples with a more homogenous stress distribution. This was interpreted by the higher values obtained by MTBS when compared to TBS (Sano et al. 1994; Scherrer et al. 2010). Moreover, MTBS results in less cohesive failures than TBS (Abdalla 2004). In addition, it is more economic, in which multiple specimens can be prepared from the same tooth (Van Meerbeek et al. 2010), and also allowing for comparison of bond strength with different regions of the tooth e.g. occlusal and cervical parts (Pashley et al. 1999). The MTBS was developed to overcome inaccuracies related to cohesive failures with SBS. Indeed, it provides more accurate measurement of the bond interface with dentin (Scherrer et al. 2010). Variations in the MTBS design include the gripping devices, specimen design and the crosshead speed of testing.

Regarding the gripping device, several modifications have been introduced to limit the non-uniformity in stress distribution resulting from mal-alignment of specimens. Gripping devices could be broadly classified as active and passive. Active gripping devices attach to the specimens mechanically, or most commonly, by gluing, which might encounter some inaccuracies such as contamination and drying of the samples, as well as deformation or fracture of the glue interface before the adhesive interface. Passive gripping devices were developed to overcome the inaccuracies encountered by the active ones in which fixation of the specimens occurs via stainless steel pins guided into custom-designed aluminum parts fixed within the gripping device (Armstrong et al. 2010).

Specimens for MTBS testing originally took the form of an hourglass (Sano et al. 1994), which was later on modified into rectangular, stick and dumbbell shapes (Fig 11). The hourglass samples were found to experience inaccuracies, thus the dumbbell shape was proposed (Neves Ade et al. 2008; Soares et al. 2008). Inaccuracies may be a result of mal-alignment of specimens and/or the introduction of micro-flaws during trimming of the small samples, which can be overcome by sufficient training and careful handling of the prepared specimens, especially while fixing them in the testing machine (Abdalla 2004; Scherrer et al. 2010). The stick-shaped samples also provide reliable measurements due to the simplicity in preparing the samples, which require no further trimming (Neves Ade et al. 2008). According to finite element analysis, it was found that the

geometry of dumbbell and stick-shaped specimens are preferred for MTBS due to the absence of stress concentration (Neves Ade et al. 2008; Soares et al. 2008). MTBS provides reliable bond strength evaluation as long as the criteria of preparation, bonding, storage, aging and testing are performed correctly (Armstrong et al. 2017).

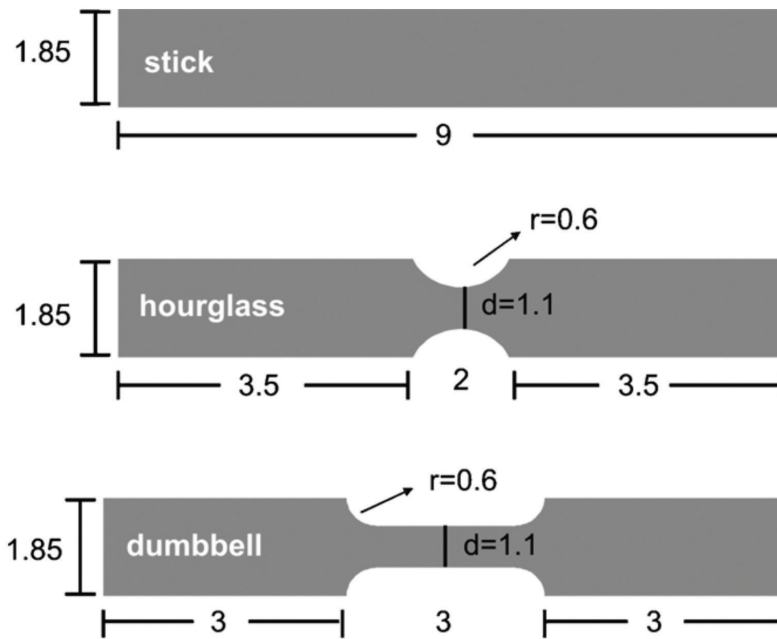


Figure 11. Different shapes of MTBS samples. The figure was reprinted from Neves Ade et al. (2008) with permission from Elsevier

4.2.3. Interfacial fracture toughness for measurement of adhesive interfaces

Inaccuracies encountered by SBS and MTBS led to the suggestion of introducing fracture mechanics for evaluating the adhesive interfaces through stably initiating a crack and measuring the adhesive's ability to resist the crack in a tensile or a peeling manner (Scherrer et al. 2010). Yet, to date, only a few studies evaluated the bond strength using fracture mechanics (De Munck et al. 2013; De Munck et al. 2015; Pongprueksa et al. 2016a; Pongprueksa et al. 2016b; Paes et al. 2017; Samimi et al. 2018). All of those studies, except Paes et al. (2017) who used the compact tension test, utilized a modification of the chevron notched beam to test different adhesive systems to dentin (De Munck et al. 2013; De Munck et al. 2015; Pongprueksa et al. 2016b; Samimi et al. 2018) or enamel (Pongprueksa et al. 2016a) (Fig 12). This mini-IFT, as the authors opted to call it, was

proven to be a reliable method in comparison to the MTBS in which only the adhesive interfacial properties were tested due to the accurate placement of the notch. On the other hand, the increased numbers of cohesive failures associated with the MTBS suggest that the strength of the whole bonded assembly is tested rather than the properties of the adhesive interface (Scherrer et al. 2010; Pongprueksa et al. 2016b). In addition, the mini-IFT is not test-dependent with less flaws introduced into the samples, which allows for comparison of results from different studies (De Munck et al. 2013). However, this test requires training and skills to accurately present and align the notch.

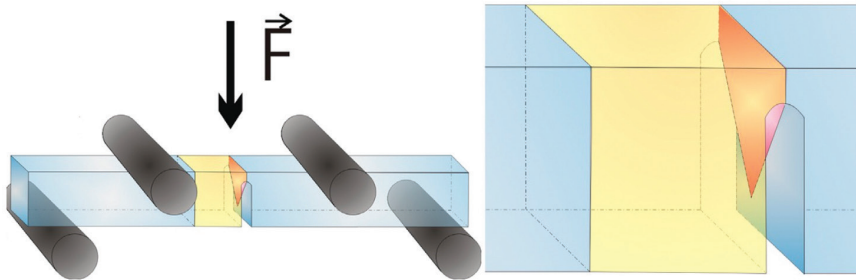


Figure 12. A schematic illustration of the mini-IFT. To the left, 4-point testing of a bonded sample; to the right, the introduction of a notch at the adhesive interface. The figure was reprinted from De Munck et al. (2013) with permission from Elsevier

Other studies (Tantbirojn et al. 2000; Moharamzadeh et al. 2008; Hooshmand et al. 2012) modified the conventional SBS into an IFT test by altering the bonding area into a chevron-shaped area and testing it in the same manner as the SBS to measure the critical energy G_{IC} . Although this method is more accurate than SBS with no premature failures recorded, the formula used to derive the G_{IC} is not always suitable for dental materials due to their plastic behavior.

Another method was developed for evaluation of the adhesive interfaces using the notchless triangular prism (NTP) specimen K_{IC} test, which has been discussed earlier as a modification of the chevron notched beam for the evaluation of the IFT of dental materials. It has been used by Far and Ruse (2003) and later on by Mesmar and Ruse (2019) for the evaluation of IFT of CAD-CAM composites. NTP compared to other IFT tests is a simple method since it requires no notch, only a crack initiation point, thus no bias related to introducing a notch. Samples are prepared in the shape of prisms which are then split into two and each half prism is bonded to its counterpart. A crack initiation point is introduced at the adhesive interface and IFT or resistance to crack propagation is measured in tension by opening the 2 halves of the prism

4.2.4 Fatigue testing of bonded interfaces

In order to provide more clinically relevant data on the bond durability, bond strength tests have been adapted to evaluate the fatigue behavior of the bonding systems. Ruse et al. (1995) used the SBS test to evaluate the fatigue behavior of a direct RC with enamel. The frequency was set at 1 Hz and the bonded samples were loaded at different pre-set stress values until failure or a maximum of 10^5 cycles. This, in turn, initiated micro-cracks at the interface and as the test progressed, crack growth and propagation occurred. In a similar manner, SBS was also used by Drummond et al. (1996) to evaluate the fatigue behavior of a direct RC with dentin using the staircase approach. A modification of the MTBS was also realized by De Munck et al. (2005) to determine the fatigue resistance of 2 adhesives with enamel and dentin. The test provided better long-term evaluation of the performance of the adhesives with enamel and dentin surfaces. In general, fatigue studies provide more clinically relevant insight on the bonding performance; however, the procedure is time-consuming, rendering it less popular (Arola 2017).

1 | 5. Research background on PICN

5.1 *In vitro* research background with PICN materials

Different properties of PICN materials have been tested *in vitro*. Prior to launching Vita Enamic (ENA) (Vita Zahnfabrik, BadSäckingen, Germany) on the market, Coldea et al. (2013) tested the flexural strength, elastic modulus and hardness of 4 experimental PICNs at 59, 63, 68 and 72 vol% ceramic and found that increasing the ceramic content had a positive effect on the elastic modulus and hardness, while it decreased the flexural strength. Among the tested experimental PICNs, the 72 vol% was the closest the commercial version ENA (75 vol%) and it showed values of 131 MPa for flexural strength, an elastic modulus of 28 GPa and hardness value of 2.1 GPa. On the other hand, numerous studies (Albero et al. 2015; Awada and Nathanson 2015; Coldea et al. 2015; Stawarczyk et al. 2015; Argyrou et al. 2016; Lawson et al. 2016; Goujat et al. 2018; Choi et al. 2019; Lucsaszky and Ruse 2019; Porto et al. 2019) compared the mechanical properties of the commercial product ENA to other ceramic and dispersed fillers (DF) materials. Flexural strength was (124-202) MPa, higher than feldspathic ceramics but lower than glass-ceramics and some DF; however, the Weibull modulus was higher than glass-ceramics and DF which gives an indication on the homogeneity of PICN (Albero et al. 2015; Awada and Nathanson 2015; Coldea et al. 2015; Stawarczyk et al. 2015; Argyrou et al. 2016; Lawson et al. 2016; Goujat et al. 2018; Choi et al. 2019; Lucsaszky and Ruse 2019; Porto et al. 2019). The modulus of ENA was (22-35) GPa,

which is an intermediary value, higher than DF and lower than ceramics (Awada and Nathanson 2015; Coldea et al. 2015; Lawson et al. 2016; Goujat et al. 2018; Lucsanzsky and Ruse 2019; Porto et al. 2019). Hardness values were also higher than those of DF and lower than ceramics with values ranging between (1.5-2.4) GPa (Albero et al. 2015; Coldea et al. 2015; Lawson et al. 2016; Goujat et al. 2018), while the fracture toughness, measured by single edge “V” notch beam test (SEVNB), was shown to be (1.1-1.4) MPa.m^{1/2} (Della Bona et al. 2014; Coldea et al. 2015; Goujat et al. 2018). Continuous developments have led to the augmentation of the mechanical properties of experimental PICNs, in particular the flexural strength which has increased two-fold reaching values ~305 MPa (Nguyen et al. 2014).

Regarding the mechanical properties of DF, there are many available DF composites on the market and it is difficult to assume that they all behave in a similar manner since they differ in the amount and composition of fillers, as well as the organic matrix composition as shown in *Table 1*. Some of those materials have been tested *in vitro*, while other materials, notably the recent generation which has been marketed later, have not yet been sufficiently tested. Indeed, the mechanical properties of DF differ from PICNs, especially the modulus and hardness which are lower than dentin. For example, Cerasmart (CRT) showed a modulus of (8-12) GPa, Shofu blocks ~10 GPa, Mazic Duro ~11 GPa, KZR-CAD-HR2 (KZR) ~11 GPa, CAMouflage NOW (CMN) ~13 GPa and Lava Ultimate (LVA) was slightly higher (11-16) GPa (Awada and Nathanson 2015; Argyrou et al. 2016; Lawson et al. 2016; Choi et al. 2019; Lucsanzsky and Ruse 2019; Porto et al. 2019), and the hardness values ranged between 0.6-0.7 GPa for CRT to 0.95-1.2 GPa for LVA (Albero et al. 2015; Lawson et al. 2016; Goujat et al. 2018). While, on the other hand, flexural strength values of CRT and LVA were higher than ENA with values of (165-234) MPa and (159-248) MPa, respectively, while the values of Mazic Duro was ~145, CMN was ~174 MPa, KZR was ~197 MPa and Shofu blocks flexural strength values were (170-180) MPa (Awada and Nathanson 2015; Argyrou et al. 2016; Lawson et al. 2016; Choi et al. 2019; Lucsanzsky and Ruse 2019; Porto et al. 2019). The fracture toughness was 1.2 MPa.m^{1/2} for CRT and 1.6 MPa.m^{1/2} for LVA by SEVNB (Goujat et al. 2018), and 0.68 for CMN MPa.m^{1/2} and 1.37 for KZR MPa.m^{1/2} by NTP specimen K_{IC} test (Lucsanzsky and Ruse 2019).

Bonding properties of PICNs are also of great importance, and have been evaluated by different researchers using either MTBS (Elsaka 2014; Frankenberger et al. 2015; Campos et al. 2016; Peumans et al. 2016; Lise et al. 2017), micro-SBS (Cekic-Nagas et al. 2016), and recently by fracture mechanics using the notchless triangular prism (NTP) specimen K_{IC} test (Mesmar and Ruse 2019). Hydrofluoric acid etching and silanization resulted in good bonding performance with resin composite luting agents (RCLA) and was found to be the most reliable pre-treatment method for bonding with RCLA.

Etching of PICN leads to selective dissolution of the feldspathic ceramic leaving micro-porosities that would enhance micromechanical retention. This effect of partial dissolution of the glass-ceramic surface was also observed with Vita Mark II samples, which is the main constituent of the ceramic skeleton of PICN (Hu et al. 2016). In comparison to other CAD-CAM materials, etched ENA showed higher MTBS values than etched and gritblasted LVA and higher micro-SBS values than both CRT and LVA (Elsaka 2014; Frankenberger et al. 2015; Cekic-Nagas et al. 2016). The NTP specimen K_{IC} test has been recently used to measure the interfacial fracture toughness (IFT) by Mesmar and Ruse (2019) of ENA and LVA bonded with RCLA, with and without water storage for 60 days. For the non-aged samples, LVA showed significantly higher IFT than ENA; however, after aging for 60 days, the IFT of LVA was reduced significantly compared to before aging, while ENA was not affected by aging in water. The authors explain this significant decrease in IFT of LVA with RCLA by the possibility of the swelling of the organic matrix from the slow diffusion of water leading to its softening and the subsequent degradation with the inorganic fillers. ENA, on the other hand, was etched, leading to the creation of a very rough surface that might have protected the adhesive interface.

In addition to the mechanical and bonding properties, PICNs were found to be biocompatible with the oral tissues, in particular gingival fibroblast and keratinocytes, as well as dental pulp and stem cells, with no monomer release (Phan et al. 2014; Grenade et al. 2016; Tassin et al. 2016; Grenade et al. 2017).

5.2 Clinical research background with PICN materials

5.2.1. Results of clinical studies with Vita Enamic material

In contrast to *in vitro* studies on Vita Enamic, only a few clinical studies were performed to evaluate the performance of the material. Clinical studies were performed either prospectively (Lu et al. 2018; Spitznagel et al. 2018b) or retrospectively (Chirumamilla et al. 2016; Belleflamme et al. 2017). Among the clinical studies on Vita Enamic is the prospective study performed by Spitznagel et al. (2018b), in which 103 Vita Enamic posterior restorations (58 partial coverage restorations and 45 inlays) were manufactured and bonded to the prepared teeth of 47 patients. Clinical evaluation was performed by 2 individual investigators at 6, 12, 24 and 36 months following the United States Public Health Service (USPHS) criteria. Within the included patients, none experienced parafunctional habits such as bruxism. The 36-month follow-up period witnessed the fracture of 2 partial coverage restorations and 1 inlay which all needed replacements, in addition to some minor chippings in the partial coverage restorations that were within

the acceptable range. The overall estimated Kaplan-Meier survival probability was 96.4%. Another prospective study was performed by Lu et al. (2018) to compare the performance of CAD-CAM onlays Vita Enamic and Vita Mark II on endodontically treated posterior teeth. Vita Enamic restorations (67) were placed in 31 patients, while 34 Vita Mark II restorations were placed in 32 patients and evaluated after 3 years by 2 individual examiners following the USPHS criteria. Two debondings with Vita Enamic and 3 with Vita Mark II were reported with an overall estimated Kaplan-Meier survival probability of 97 % for the former and 90.7 % for the latter. In addition to the prospective studies on Vita Enamic, Chirumamilla et al. (2016) conducted a retrospective study on 29 patients having 36 crowns after 2 years of placing them. The evaluation was conducted by one examiner and showed the debonding of a crown cemented with resin-modified glass-ionomer cement, which also had secondary caries, while a second failure resulted from a tooth that was cracked prior to cementation of the crown with a resin composite luting agent and was regarded as a failure. The overall estimated Kaplan-Meier survival probability was 96.8 % and 92.9 % for Vita Enamic crowns cemented with the resin composite luting agent and resin-modified glass-ionomer, respectively. Similarly, Belleflamme et al. (2017) also conducted a retrospective study, but this time the restorations were endocrowns; however, among the restorations examined in this study, only 12 Vita Enamic restorations were evaluated by 2 individual examiners following the FDI criteria, showing a Kaplan-Meier survival probability of 89.9 % after 44.7 months.

5.3 Contribution of PICN materials to new treatment strategies development

Two new treatment protocols have been recently proposed, which were based on the specific *in vitro* properties of PICN materials. Those approaches are related to the field of worn dentition treatment and implant dentistry.

5.3.1. Treatment of worn dentition

Tooth wear is a result of attrition, abrasion, erosion and most probably a combination of one or more of these factors (Bartlett 2016), in which there are some predisposing factors that aggravate wear in a much faster rate such as bruxism and clenching mechanically and the consumption of acidic drinks, sodas and gastroesophageal reflux chemically. In some cases, worn teeth are left untreated (Loomans et al. 2017), whereas in other cases, restorative intervention is indicated due to complications arising from the loss of dental hard tissue such as poor esthetics, pain, temporo-mandibular joint and muscular discomfort (Gillborg et al. 2017). Ideally, the lost dental tissues should be restored in a minimally invasive manner to avoid the loss of more tissues. Worn dentition could be restored either by direct resin composites, which are the most common type

of restorations in such cases, or by indirect restorations such as ceramics, and the selection of one material over the other remains in the hands of the clinician (Mesko et al. 2016). Although direct resin composites bond to the tooth and are considered minimally invasive, they are operator-dependent and the material properties are limited, especially in the posterior region, needing regular maintenance. The intervals of maintenance may vary from one patient to another, and for some patients could be regarded as an added cost (Bartlett 2016). In that sense, indirect restorations could be considered as materials of choice for such patients. However, more dental hard tissue needs to be removed to accommodate for the thickness of ceramics to avoid its chipping at the margins. Chen et al. (2014) found that there is a direct correlation between the thickness of lithium disilicate glass-ceramic IPS e.max CAD (Ivoclar Vivadent) and its fracture resistance, in which a restoration less than 0.5 mm would be prone to chipping, while for the CAD-CAM composite Lava Ultimate (3M Oral Care), this correlation did not exist. Recently, a non-invasive restoration of worn dentition protocol has been proposed by Mainjot (2018) using very low thickness PICN restorations (0.2 mm) for full-mouth rehabilitation. This protocol presents a minimally-invasive approach for worn dentition, supported by 3 pilot cases of full-mouth rehabilitation, which paves the way for the use of PICNs for the treatment of worn teeth without the need to further remove enamel or dentin to accommodate for the bulk of the restoration. PICNs offer 1) low thickness restorations due to their less edge chipping than ceramics when milled; 2) can be easily adjusted in the mouth prior to cementation and easily repaired; 3) less brittle than ceramics with a higher elastic modulus than light-cure composites; 4) better biological properties than direct composites with a higher degree of conversion and lower monomer release and 5) good bonding properties with resin composite luting agents when etched and silanized.

5.3.2. Implant dentistry

Recent advances in implant dentistry led to the development of implant immediate loading protocols, to spare the patients from the repeated surgical interventions and to provide an immediate alternative to the non-esthetic edentulous area. Due to the increased forces of mastication, provisional crowns are usually placed out of occlusion to allow osseointegration around the placed implant without interruption from masticatory forces, especially with high risk patients such as patients with bruxism, medically compromised patients and smokers (Vogl et al. 2015; Moraschini and Porto Barabozza 2016). A novel protocol has been proposed by Lambert & Mainjot (2017) to replace the edentulous posterior area with the implant and the final restoration in a single visit without the need for a provisional restoration. This One-tooth One-time (1-T 1-T) proof of concept, supported by 2 pilot cases with successful outcomes, provides the patient with the final restoration of a CAD-CAM fabricated PICN on the same day,

therefore the cost and time are reduced with an overall patient satisfaction increase. The main idea is that PICNs, with their polymeric phase and the associated low elastic modulus and high flexural load energy, are materials able to absorb stress, offering a damping effect and allowing normal osseo-integration without interference from masticatory forces.

1 | 6. References

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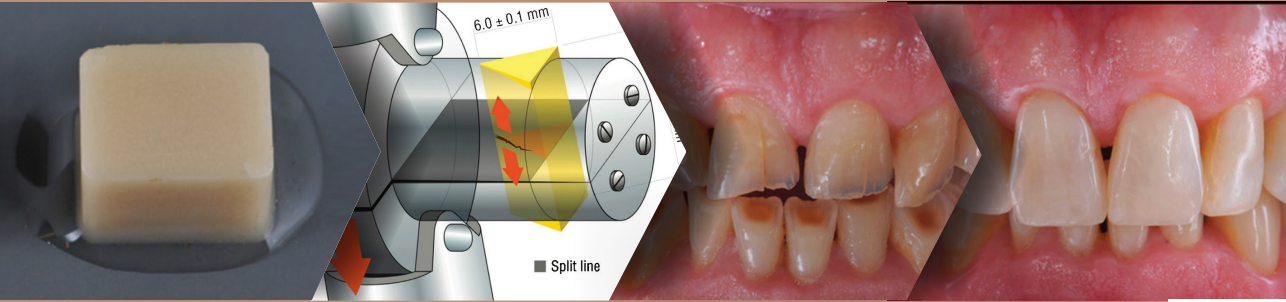
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OBJECTIVES




Chapter 2

The *in vitro* studies constitute the bulk of this thesis. The objectives of this section was:

- To study the bonding properties of PICN materials by
 - evaluating the interfacial fracture toughness (IFT) of a resin composite luting agent (RCLA) with dispersed fillers (DF) versus polymer-infiltrated ceramic network (PICN) CAD-CAM composite blocks, studying the influence of surface etching and gritblasting pretreatment. Moreover, to correlate IFT results with the developed interfacial area ratio (Sdr). The first null hypothesis was that material class and surface pre-treatment do not influence IFT of RCLA with CAD-CAM composites. The second null hypothesis was that there is no correlation between IFT and Sdr.
 - evaluating silane influence on the interfacial fracture toughness (IFT) of a resin composite luting agent with the two sub-classes of CAD-CAM composites. In addition, to correlate IFT results with Sdr and surface wettability. The null hypotheses were that material class, surface texture after pre-treatment and silane do not influence IFT.

- To introduce a functionally graded PICN block characterized by a gradient of mechanical and optical properties throughout the entire thickness, as a biomimetic material for CAD-CAM prostheses.

- On the other hand, this thesis also aimed to contribute in translational research on PICN materials. Therefore, clinical applications of PICN materials were explored in the second section of this thesis, participating to two clinical studies related to new treatment protocols developed with PICN materials, particularly:
 - A minimally invasive approach of worn dentition treatment: the “One-step No-prep” protocol.
 - A novel approach for restoring a missing posterior tooth with immediate loading of an implant and a final crown made of PICN in a single visit: the “One-tooth One-time, 1T1T” protocol.



BONDING TO CAD-CAM COMPOSITES: AN INTERFACIAL FRACTURE TOUGHNESS APPROACH

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3 | 1. Abstract

The objective of this study was to evaluate the interfacial fracture toughness (IFT) of composite cement with dispersed filler (DF) versus polymer-infiltrated ceramic network (PICN) computer-aided design and computer-aided manufacturing (CAD-CAM) composite blocks after 2 different surface pretreatments using the notchless triangular prism (NTP) test. Two DFs (Cerasmart [CRT] and Lava Ultimate [LVA]), 2 PICNs (Enamic [ENA] and experimental PICN [EXP]), and e.max CAD lithium disilicate glass-ceramic (EMX, control) prism samples were bonded to their counterparts with Variolink Esthetic DC composite cement after either hydrofluoric acid etching (HF) or gritblasting (GR). Both procedures were followed by silanization. All samples ($n = 30$ per group) were thermocycled (10,000 cycles) and tested for their IFT in a water bath at 36°C.

Moreover, representative samples from each group were subjected to a developed interfacial area ratio (Sdr) measurement by profilometry and scanning electron microscopy (SEM) characterization. EXP-HF gave the highest IFT ($1.85 \pm 0.39 \text{ MPa}\cdot\text{m}^{1/2}$), followed by EMX-HF and ENA-HF, while CRT-HF gave the lowest ($0.15 \pm 0.22 \text{ MPa}\cdot\text{m}^{1/2}$). PICNs gave significantly better results with HF, and DF showed better results with GR. A 2-way analysis of variance indicated that there were significantly higher IFT and Sdr for PICNs than for DF. A positive correlation ($r^2 = 0.872$) was found between IFT and Sdr. SEM characterization showed the specific microstructure of the surface of etched PICNs, indicating the presence of a retentive polymerbased honeycomb structure.

Etching of the typical double-network microstructure of PICNs causes an important increase in the Sdr and IFT, while DF should be gritblasted. DF exhibited significantly lower Sdr and IFT values than PICNs. The present results show the important influence of the material class and surface texture, and consequently the micromechanical bond, on the adhesive interface performance of CAD-CAM composites.

Keywords: dental materials, dental prosthesis retention, resin cement, surface properties, ceramics, dental adhesives.

3 | 2. Introduction

Computer-aided design and computer-aided manufacturing (CAD-CAM) composites are gaining popularity in the field of restorative materials, competing with glass-ceramics for single-unit restorations. These composites are notable for their better machinability, higher resilience, and lower elastic modulus, hardness, and brittleness (Albero et al. 2015; Coldea et al. 2015). Specifically, their ability to be milled to a very low thickness offers interesting perspectives for use as minimally invasive bonded restorations, such as so-called occlusal tabletops. However, there is still a lack of data regarding their bonding properties compared with the better known glass-ceramic materials (Spitznagel et al. 2014); in particular, the effect of the CAD-CAM composite microstructure on its performance is unknown. Indeed, the CAD-CAM composite blocks can be divided into 2 distinct classes based on their microstructure: dispersed filler (DF) and polymer-infiltrated ceramic network (PICN) materials (Coldea et al. 2013; Mainjot et al. 2016). In DF materials, the fillers are classically incorporated by mixing them in a matrix composed of dimethacrylates, such as urethane dimethacrylate (UDMA) and triethylene glycol dimethacrylate (TEGDMA), which are polymerized under high temperature (>100°C). The available products contain varying amounts, sizes, and compositions of filler, according to technical data released by the respective manufacturers. On the other hand, PICNs are composed of a sintered glass-ceramic block (75% of the solid volume) secondarily infiltrated with UDMA and TEGDMA, which are polymerized under high temperature and high pressure (HT-HP) (180°C, 300 MPa) (Nguyen et al. 2014). The specific polymerization mode of CAD-CAM composite blocks, particularly the patented HT-HP polymerization mode (Sadoun 2011), which has been shown to increase the degree of conversion up to 96% (Phan et al. 2015), may decrease the chemical co-polymerization of any remaining free monomers of the CAD-CAM block with the monomers of the composite cement. In general, manufacturers recommend the same bonding protocol for CAD-CAM composite blocks as for indirect composite filling materials (i.e., the use of alumina air-abrasion or tribochemical silica-coating procedures to roughen the material surface), followed by the application of a silane layer (Spitznagel et al. 2014). For Vita Enamic (ENA; Vita Zahnfabrik), the only PICN on the market, hydrofluoric acid (HF) etching can be used as an alternative to gritblasting (GR), which has also been proposed for Cerasmart (GC), a DF material.

Several previous studies of the bonding properties of composite cement to CAD-CAM composite blocks evaluated the microtensile bond strength (μ TBS) or (micro)shear bond strength according to the type of pretreatment, composite cement, or material (Elsaka

2014; Frankenberger et al. 2015; Campos et al. 2016; Cekic-Nagas et al. 2016; Peumans et al. 2016; Schwenter et al. 2016; Lise et al. 2017). However, despite the popularity of the μ TBS and its reliability compared with shear bond testing, its use results in inaccuracies in bond strength evaluation, mainly due to the sample preparation after bonding, which leads to an increased number of pretest failures and problems related to nonuniform stress distribution (Scherrer et al. 2010; Van Meerbeek et al. 2010). Therefore, some authors have recently recommended the introduction of alternative and more reliable methods for evaluating adhesive interfaces, particularly fracture mechanics, which allows the measurement of the interfacial fracture toughness (IFT) (K_{IC}) of the adhesive layer (Armstrong et al. 2010; Scherrer et al. 2010; Soderholm 2010; Van Meerbeek et al. 2010; De Munck et al. 2013; Pongprueksa et al. 2016). The goal is to stably initiate and propagate a crack through the bonded interface and to measure the crack-propagation resistance or peeling resistance from the substrate (Scherrer et al. 2010). The IFT is advantageous because it evaluates the interface properties rather than the mechanical strength of the whole assembly (Pongprueksa et al. 2016). Various types of tests have already been used with dental materials, including the single-edge notched beam (Toparli and Aksoy 1998) and the chevron notch short rod or beam test (Armstrong et al. 1998), which has been modified into various versions adapted to dental samples (Ruse et al. 1996; De Munck et al. 2013; Pongprueksa et al. 2016). One variation is the notchless triangular prism (NTP) test introduced by Ruse et al. (1996), which was reported to be a simple method (Soderholm 2010). This method was previously proposed to evaluate both the fracture toughness of materials and the fracture toughness of bonded interfaces (Ruse et al. 1996; Far and Ruse 2003). The authors validated the stress distribution by a finite element analysis, showing that the maximum tensile stress was concentrated at the tip of the crack (Ruse et al. 1996). A significant advantage of the NTP test is the good level of control and stable crack growth at a low cross-speed (0.05 mm/min), which allows for reliable measurements and the possibility of performing a fatigue process study, controlling crack propagation for a given K_I . Moreover, the NTP test avoids bias related to the creation of a notch, which is particularly challenging due to the low interface thickness.

The objectives of this study were to use the NTP test to evaluate the IFT at mouth temperature of composite cement with 2 classes of CAD-CAM composite blocks, DF and PICN, after 2 different surface pretreatments and upon thermocycling aging.

3 | 3. Materials and Methods

Samples Preparation (Appendix Fig. 1)

Prism Manufacturing. Two DF composite materials, Lava Ultimate (LVA; 3M ESPE) and Cerasmart (CRT; GC), and 2 PICN materials, ENA and an experimental PICN (EXP; MaJEB), were tested. EXP was composed of 73.8 vol.% slip-casted and sintered Vita Mark II glass-ceramic powder (Vita Zahnfabrik, 2.6- μm D50 grain-size distribution), which was infiltrated by HT-HP (180°C, 300 MPa), UDMA (Esstech), and di-tert-amyl peroxide (Sigma-Aldrich) (initiator), following the procedure described by Nguyen et al. (2014). In addition, a lithium disilicate-reinforced glass-ceramic, IPS e.max CAD (EMX; Ivoclar Vivadent), was used as a positive control. CAD-CAM blocks were cut using a low-speed saw (IsoMet; Buehler) under continuous water irrigation at an angle of 60° to produce 4 samples per block. The samples ($n = 60$ per material) were then ground into the desired 14.0 \pm 0.1-mm-long triangular prisms with a 6.0 \pm 0.1-mm side width using 220-grit silicon carbide (SiC) paper, at 300 rpm under water cooling (Struers) using a custom-built specimen holder. EMX samples were manufactured in the crystalline intermediary stage and then fired in a dedicated furnace (Programat; Ivoclar Vivadent) at 820°C for 10 min (90°C/min), followed by 840°C for 7 min (30°C/min), according to the manufacturer's recommendations. All prisms were then split into 2 with the NTP test (Ruse et al. 1996) to produce half prisms and to measure material fracture toughness for future research.

Surface Pretreatment. The bonding surfaces were polished with 1,000-grit SiC paper under water cooling to obtain 6.0 \pm 0.1-mm-long prisms. Samples were ultrasonically cleaned in 90% ethanol for 3 min (Vita Sonic II; Vita Zahnfabrik), dried with oil-free air for 10 s, and then randomly distributed to either the HF or GR pretreatment group ($n = 30$ per material in each group). For the HF group, 5% hydrofluoric acid (Vita ceramics etch; Vita Zahnfabrik) was applied and left for 60 s, and then the samples were washed under running water for another 60 s and air dried, as recommended by the manufacturers of ENA and CRT. The samples were then additionally cleaned ultrasonically in ethanol for 3 min and air dried for 10 s. A silane layer (Monobond Plus; Ivoclar Vivadent) was applied using a microbrush and left for 60 s, and then samples were air dried for 10 s. The positive control (EMX) was treated following the same procedure as the other samples except that the etching was performed with 4.5% hydrofluoric acid (IPS Ceramic; Ivoclar Vivadent) for 20 s, as specified in the manufacturer's recommendations. For the GR group, gritblasting was performed using 50- μm Al₂O₃ particles (Danville) for 5 s in a perpendicular direction at a distance 1 cm from the sample (Basic Professional 942; Renfert). The pressure varied according to the manufacturers' instructions for each material: ENA and EXP were subjected to a 1-bar gritblasting pressure, while 1.5

bars and 2 bars were used for CRT and LVA, respectively. Then, ultrasonic cleaning and silanization were performed in the same way as in the HF group.

Bonding Procedure. Half prisms were bonded to their counterparts using a custom-designed alignment system into which they were fixed (*Appendix Fig. 2*). A dual-cure composite cement (Variolink Esthetic DC, neutral shade; Ivoclar Vivadent) was applied on each surface with the mixing syringe, and half prisms were put into contact. Three 20-s light applications (Bluephase 20i; Ivoclar Vivadent) were performed at high power (1,200 mW/cm²) at close proximity to each side of the alignment apparatus. An extra 40 s of curing was applied at a distance of 2 mm on each side after removal from the alignment apparatus to ensure optimal curing. The samples were left in water for 24 h at 36°C, and then the excess composite cement was removed by polishing the prisms with 1,000-grit SiC paper under water cooling.

Aging. Samples were submitted to thermocycling for 10,000 cycles (5/55°C, 30 s in each bath) (Armstrong et al. 2017).

IFT Measurement with the NTP Test

The IFT was measured using the NTP test (*Fig. 1*), following the procedure described by Ruse et al. (1996). Samples ($n = 30$ per group) were fixed into 1 half of the NTP specimen holder, and a crack initiation point (~0.1 mm) was made at the bond interface under a light microscope (Light Highlight 3001; Olympus) at a magnification of $\times 20$ using a sharp scalpel (size 11 surgical blade; Swann-Morton). After securing the other half of the specimens, the samples were mounted on the computer-controlled (Bluehill; Instron Canada) universal testing machine (Instron model 5565) within a water bath at 36°C, at a cross-head speed of 0.05 mm/min. The strain values were recorded at failure arrest in a tensile mode, and the IFT was calculated using the formula $K_{IC} = Y^*_{min} P_{max} / DW^{1/2}$, where P_{max} is the maximum load at failure, D is the NTP specimen diameter (12.0 mm), W is the NTP specimen length (10.5 mm), and Y_{min} is the dimensionless stress intensity factor coefficient minimum (28) as given by Ruse et al. (1996).

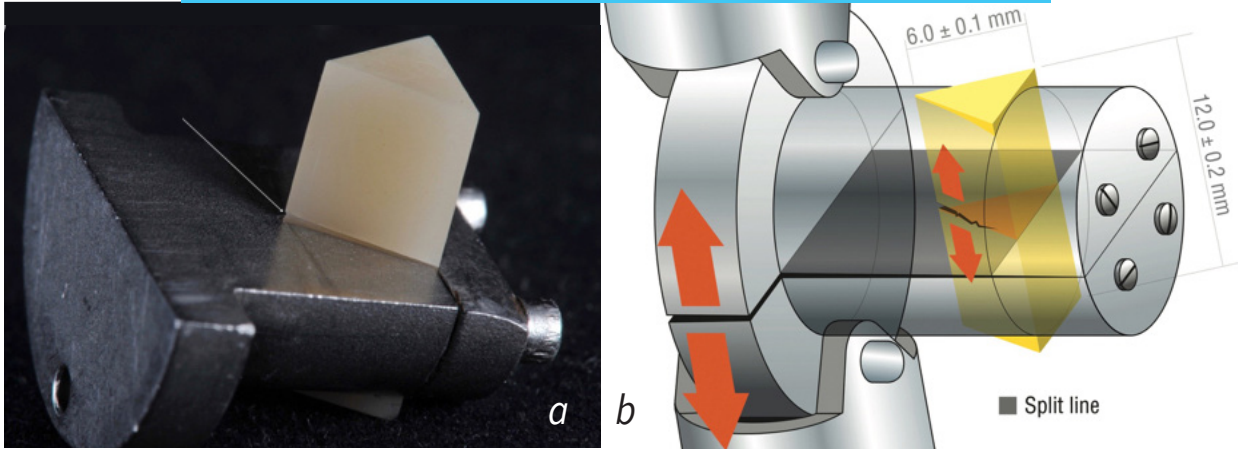
Each sample was examined under a light microscope following each test to identify the failure mode as adhesive, mixed, or cohesive, as described by Scherrer et al. (2010) and illustrated in *Appendix Figure 3*.

Surface Characterization

Three rectangular samples (dimensions 14.0 × 12.0 × 2.0 mm³) of each material investigated were manufactured by cutting the CAD-CAM blocks using a low-speed IsoMet saw and were polished with 1,000-grit SiC paper. Two of the samples were

Figure 1. Notchless Triangular Prism (NTP) test.

- a) A bonded prism fixed to 1 half of the cylindrical mounting block, with an arrow pointing at the crack initiation point.
- b) A schematic illustration of the NTP apparatus in motion, with a bonded prism fixed within. The arrows pointing in opposite directions indicate the application of tensile force on the bonded interface, marked by the split line, with crack propagation starting from the tip of the prism at the crack initiation point until fracture or crack arrest.



etched and gritblasted following the previously described procedures. The third sample was used as a control.

Profilometry. Profilometry was carried out on samples to measure the developed interfacial area ratio (Sdr), which is expressed as the percentage of additional surface area contributed by the texture compared to an ideal plane the size of the measurement region. The Sdr is obtained by calculating the topographical area with respect to this ideal plane and gives the surface enlargement induced by the different pretreatments. Measurements were performed using a 3-dimensional optical microscope (Contour GTI; Bruker) in high-resolution vertical scanning interferometry (VXI) mode at < 1 nm, independent of the objective. Five measurements were taken at the center of each sample with a $\times 115$ objective (image size: 0.06×0.04 mm²; optical lateral resolution: 0.33 μ m) with no filtering and removal of the tilt and cylinder terms. The values of the Sdr were obtained by calculating the mean of 5 values measured for each sample.

Scanning Electron Microscopy. The samples were cleaned ultrasonically in 90% ethanol for 3 min and subsequently gold-coated to be analyzed by scanning electron microscopy (SEM) (S-3000N; Hitachi).

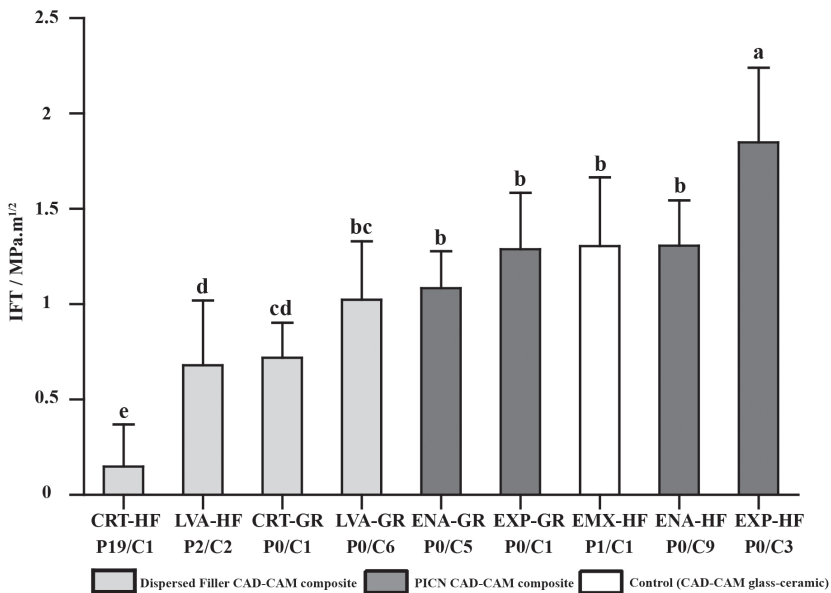
Statistical Analysis

The results were analyzed by 1- and 2-way analysis of variance (ANOVA), followed, if warranted, by Scheffé's multiple mean comparisons ($\alpha = 0.05$), using PASW Statistics 18 (SPSS, Inc.). Pearson's tests were used to verify the presence of statistically significant correlations between the IFT and Sdr ($\alpha = 0.05$).

3 | 4. Results

IFT

The means and standard deviations of the IFT measurements for each group of samples, along with the statistical analysis, are provided in *Figure 2a* and *Tables 1* and *2*. Some samples experienced debonding prior to IFT testing. The samples showing pretest failures were included as zero values, while samples that fractured in a cohesive mode were excluded from the data analysis. The numbers of pretest and cohesive failures for each group of samples are provided in detail in *Figure 2a*. EXP-HF had a significantly higher IFT than other samples ($1.85 \pm 0.39 \text{ MPa}\cdot\text{m}^{1/2}$), followed by EMX-HF and ENA-HF, while CRT-HF had the lowest value ($0.15 \pm 0.22 \text{ MPa}\cdot\text{m}^{1/2}$). A 2-way ANOVA revealed that there was a significantly higher IFT for PICNs than for DF, and this finding was independent of the surface pretreatment performed ($P < 0.05$) (*Appendix Table*). PICNs gave significantly higher results with HF and DF with GR.



a

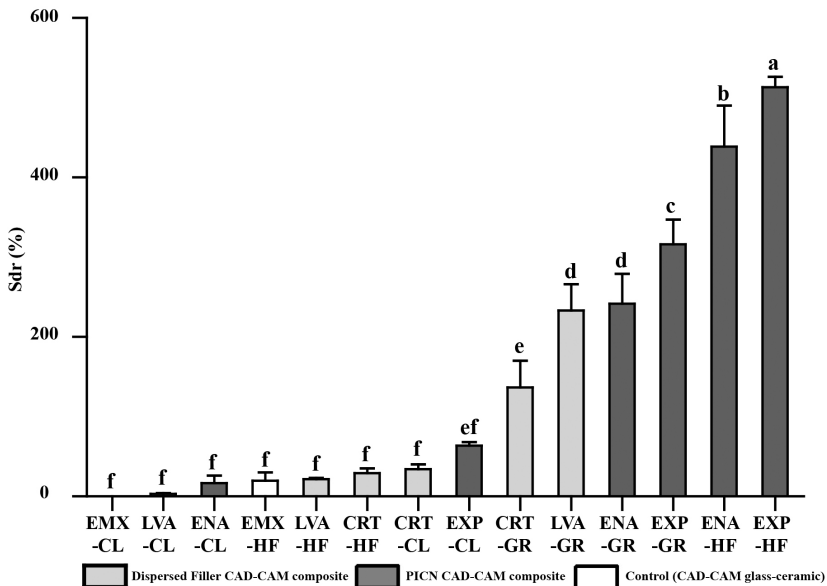
Figure 2. Results of interfacial fracture toughness (IFT) and developed interfacial area ratio (Sdr) measurements.
 a) A bar graph of the IFT measurement. The letters P and C under the columns represent pretest failures (P) and cohesive failures (C) among the initial number of tested samples in each group of materials. The (C) samples were not included in the IFT calculation.
 b) A bar graph of the Sdr measurement. The results are expressed as the means \pm SD. Superscript letters indicate statistically homogeneous subgroups within a material category (1-way analysis of variance)

Developed Interfacial Area Ratio (Sdr)

The mean and standard deviations of the Sdr measurements for each group of samples, along with the results of the statistical analysis, are summarized in *Figure 2b* and *Tables 1* and *2*. EXP-HF had a significantly higher Sdr than the other groups ($513.0\% \pm 13.1\%$), followed by ENA-HF and EXP-GR, while EMX-CL had the lowest value ($0.2\% \pm 0.1\%$). A 2-way ANOVA revealed that there was a significantly higher Sdr for PICNs than for DF, independent of the surface pretreatment performed ($P < 0.05$) (*Appendix Table*). PICNs gave significantly higher results with HF and DF with GR.

Correlation between the IFT and Sdr

The statistical analysis using Pearson's correlation coefficient revealed that there was a strong ($r^2 = 0.8723$) and significant ($P < 0.05$) correlation between the IFT and Sdr



b

followed by Scheffé test, $\alpha = 0.05$). The same superscript letters demonstrate that there were no significant differences for each factor. CRT-CL, Cerasmart control; CRT-GR, gritblasted Cerasmart; CRT-HF, etched Cerasmart; EMX-CL, IPS e.max CAD control; EMX-HF, etched IPS e.max CAD; ENA-CL, Enamic control; ENA-GR, gritblasted Enamic; ENA-HF, etched Enamic; EXP-CL, experimental polymer-infiltrated ceramic network (PICN) control; EXP-GR, gritblasted experimental PICN; EXP-HF, etched experimental PICN; LVA-CL, Lava Ultimate control; LVA-GR, gritblasted Lava Ultimate; LVA-HF, etched Lava Ultimate.

Table 1. Representative Means and Standard Deviations of the Interfacial Fracture Toughness (IFT).

	Failure Mode, n					
	IFT, MPa.m ^{1/2}	Pretest	Adhesive	Mixed	Cohesive	Sdr, %
EMX-CL						0.20 ± 0.10 ^f
EMX-HF	1.31 ± 0.36 ^b	1	26	2	1	19.60 ± 10.80 ^f
CRT-CL						34.30 ± 5.80 ^f
CRT-GR	0.72 ± 0.18 ^{c,d}	0	27	2	1	136.60 ± 33.6 ^e
CRT-HF	0.15 ± 0.22 ^e	19	10	0	1	29.40 ± 5.90 ^f
LVA-CL						3.50 ± 0.60 ^f
LVA-GR	1.02 ± 0.31 ^{b,c}	0	18	6	6	233.10 ± 33.00 ^d
LVA-HF	0.68 ± 0.34 ^d	2	26	0	2	21.70 ± 1.70 ^f
ENA-CL						16.70 ± 9.40 ^f
ENA-GR	1.08 ± 0.19 ^b	0	15	10	5	241.70 ± 37.60 ^d
ENA-HF	1.31 ± 0.24 ^b	0	14	7	9	438.90 ± 51.20 ^b
EXP-CL						63.60 ± 4.50 ^{e,f}
EXP-GR	1.19 ± 0.30 ^b	0	28	1	1	316.20 ± 31.00 ^c
EXP-HF	1.85 ± 0.39 ^a	0	25	2	3	513.00 ± 13.10 ^a

Values are expressed as the MPa.m^{1/2} ± SD, with the number of samples that failed before the test and in an adhesive, mixed, and cohesive mode, respectively (the samples showing pretest failures were included as zero values, while samples that fractured in a cohesive mode were excluded from the data analysis). Also shown are the means and standard deviations of the Sdr (developed interfacial area ratio), expressed as a percentage. Superscript letters indicate statistically homogeneous subgroups within a material category (1-way analysis of variance followed by Scheffé test, α = 0.05). The same superscript letters demonstrate that there were no significant differences for each group. CRT-CL, Cerasmart control; CRT-GR, gritblasted Cerasmart; CRT-HF, etched Cerasmart; EMX-CL, IPS e.max CAD control; EMX-HF, etched IPS e.max CAD; ENA-CL, Enamic control; ENA-GR, gritblasted Enamic; ENA-HF, etched Enamic; EXP-CL, experimental polymer-infiltrated ceramic network (PICN) control; EXP-GR, gritblasted experimental PICN; EXP-HF, etched experimental PICN; LVA-CL, Lava Ultimate control; LVA-GR, gritblasted Lava Ultimate; LVA-HF, etched Lava Ultimate.

Table 2. Results of a 1-Way Analysis of Variance for the IFT and Sdr.

	Sum of Squares	Degrees of Freedom	Mean Squares	F Statistic	Significance
IFT					
Between groups	52.678	8	7	77.799	^a
Within groups	19.636	232	0.085		
Total	72.314	240			
Sdr					
Between groups	1,944,352.102	13	149,565.392	272.377	^a
Within groups	30,750.278	56	549.112		
Total	1,975,100.380	69			

IFT, interfacial fracture toughness; Sdr, developed interfacial area ratio.

^aA significant difference was detected (P < 0.05).

for all groups of CAD-CAM composite blocks. This positive correlation suggests that, under the conditions of this study, over 87% of the variation in the IFT was explained by the Sdr (*Appendix Fig. 4*). When EMX was included, the correlation was weaker ($r^2 = 0.5697$).

SEM

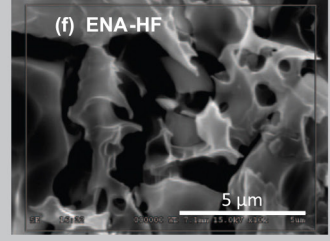
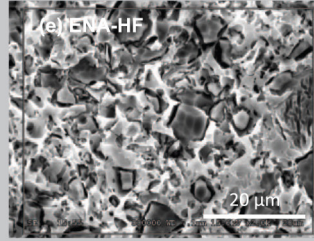
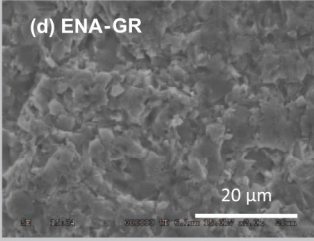
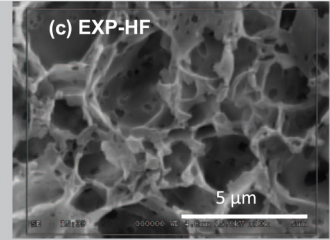
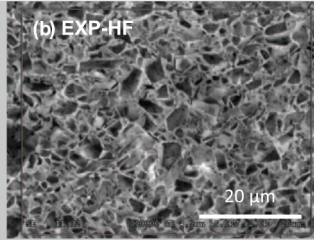
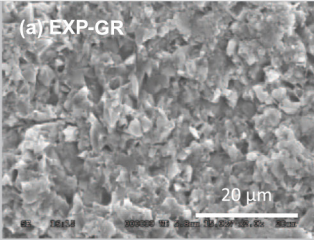
SEM characterization showed the specific microstructure of the surface of etched PICNs, characterized by dissolution of the glass-ceramic network and the presence of a typical polymer-based honeycomb structure, including micro- and nanoporosities. HF had the highest impact on the EXP, with a higher glass-ceramic phase dissolution. For ENA, the impact was slightly lower; the glass-ceramic network was not totally dissolved and the microporosities were larger. With LVA, etching resulted in the partial dissolution of the filler particles and in a significantly smoother surface than PICNs. HF created very minor porosities on the surface of CRT, which appeared to have the smoothest surface among the etched composites. Finally, EMX showed a slight dissolution of glass, with a smoother surface than PICNs. GR created roughness on the surface of all samples, with PICNs showing a finer roughness than DF. CRT showed more roughness when gritblasted than when etched. Finally, gritblasting PICN did not result in the creation of a honeycomb structure, as did etching (*Fig. 3*).

SANDBLASTED X2K

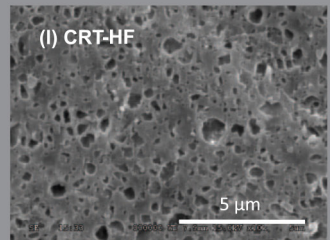
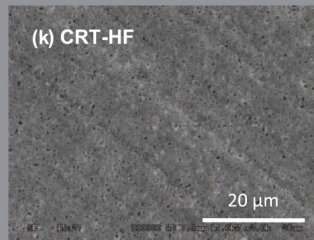
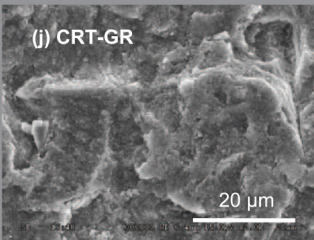
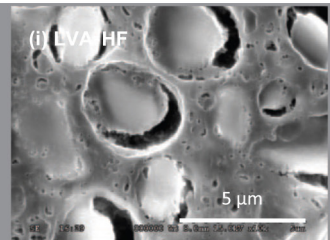
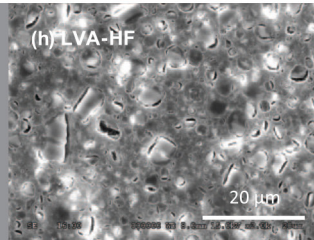
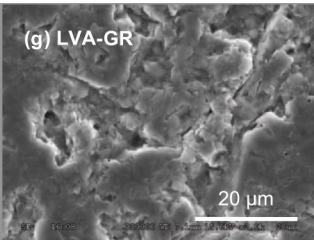
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PICNs



Dispersed fillers



Glass-ceramic

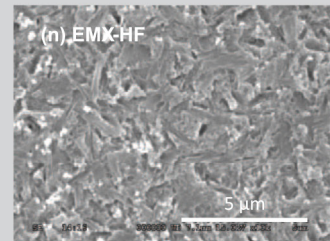
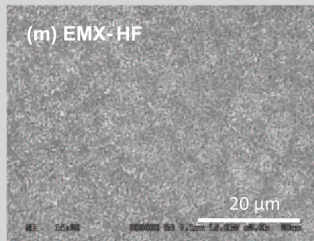


Fig. 3 Scanning electron microscopy images of the samples following different surface pretreatments:
 a) gritblasted experimental polymer-infiltrated ceramic network (PICN) at $\times 2,000$ magnification (EXP-GR);
 b) etched experimental PICN at $\times 2,000$ magnification (EXP-HF);
 c) etched experimental PICN at $\times 10,000$ magnification (EXP-HF);
 d) gritblasted Enamic at $\times 2,000$ magnification (ENA-GR);
 e) etched Enamic at $\times 2,000$ magnification (ENA-HF);
 f) etched Enamic at $\times 10,000$ magnification (ENA-HF);
 g) gritblasted Lava Ultimate at $\times 2,000$ magnification (LVA-GR);
 h) etched Lava Ultimate at $\times 2,000$ magnification (LVA-HF);
 i) etched Lava Ultimate at $\times 10,000$ magnification (LVA-HF);
 j) gritblasted Cerasmart at $\times 2,000$ magnification (CRT-GR);
 k) etched Cerasmart at $\times 2,000$ magnification (CRT-HF);
 l) etched Cerasmart at $\times 10,000$ magnification (CRT-HF);
 m) etched IPS e.max CAD at $\times 2,000$ magnification (EMX-HF); and
 n) etched IPS e.max CAD at $\times 10,000$ magnification (EMX-HF).

3 | 5. Discussion

The NTP test was successfully used to evaluate adhesive interfaces, yet the alignment system for half-prism bonding could be improved to accurately control the cement thickness. In the present study, samples were tested in a water bath set at 36°C to eliminate any bias resulting from differences in temperature (Soderholm 2010) and to provide a close approximation of the conditions in the mouth. Cohesive failures can be explained by the fact that the IFT is close to or higher than the reported fracture toughness of the different CAD-CAM materials (LVA, $0.91 \text{ MPa}\cdot\text{m}^{1/2}$; ENA, $0.88 \text{ MPa}\cdot\text{m}^{1/2}$; EXP, $2.7 \text{ MPa}\cdot\text{m}^{1/2}$; EMX, $1.79 \text{ MPa}\cdot\text{m}^{1/2}$) (Nguyen et al. 2014; Ruse and Sadoun 2014).

Our results indicated that the material class (DF vs. PICN) and its interaction with different surface pretreatment (HF or GR) influence both IFT and Sdr. PICNs showed significantly higher IFT values than DF. In previous studies, etched Enamic gave better results than Lava, regardless of the type of pretreatment (Elsaka 2014; Frankenberger et al. 2015; Cekic-Nagas et al. 2016), while Cerasmart had poorer results when etched (Cekic-Nagas et al. 2016) but similar results when gritblasted, contrary to the present findings (Lise et al. 2017). The difference observed between the 2 classes of materials can be attributed to the surface texture after pretreatment, as shown by the correlation found between the IFT and Sdr, which corresponds to the additional surface area created by acid etching or gritblasting. Indeed, the effectiveness of bonding was suggested to be related to 2 important parameters, the micromechanical bond or interlocking, which is promoted by an increased material surface roughness, and the chemical bond between the composite cement and the specific material, which is promoted by a silane application (Tian et al. 2014). Increasing the surface roughness was previously reported to be more important than chemical conditioning to improve the bonding properties of most indirect composite filling materials (Spitznagel et al. 2014). For those materials, which are all DF, gritblasting was shown to be more effective than etching

(Spitznagel et al. 2014), as was shown for DF CAD-CAM composites in the present study. In contrast, for PICNs, etching gave significantly better results than gritblasting, with other studies using μ TBS showing also better results with HF (Elsaka 2014; Frankenberger et al. 2015). The influence of micromechanical interlocking on the performance of the adhesive interface of CAD-CAM composites is particularly important, since a high degree of conversion of polymers decreases the potential for chemical co-polymerization of any remaining free monomers with the monomers of the composite cement. This micromechanical interlocking is also influenced by roughness induced by milling, which varies with bur grit size (Lebon et al. 2015).

The significantly higher Sdr and subsequent micromechanical bonding potential of PICNs compared to DF are related to their specific microstructure when etched. As seen in SEM images, surface etching results in the creation of a honeycomb polymer-based structure. The presence of micro- and even nanoporosities for EXP increases the surface texture and consequently promotes interlocking with the composite cement. EXP-HF had a significantly higher IFT than all of the other materials. Indeed, the Sdr of EXP-HF, which was even underestimated due to the lateral resolution of the profilometer (approximately 0.5 μ m), was significantly higher than that of other groups, including ENA-HF, which can be explained by the smaller glass-ceramic grain size of experimental PICN compared to Enamic.

In line with the study by Frankenberger et al. (2015), ENA-HF showed findings similar to those of etched lithium-disilicate glass-ceramic (EMX-HF), which was used as a positive control due to its good bonding properties (Tian et al. 2014). Moreover, in the present study, EXP-HF gave significantly better results than EMX-HF. Of note, the Sdr values for EMX-HF were low in comparison with PICNs: the absence of a correlation with the IFT suggests that there may be differences in the bonding mechanisms between glass-ceramics and composite materials. While micromechanical interlocking seems to be fundamental for composites (Spitznagel et al. 2014), the chemical bond seems to be more important for glass-ceramics, as mentioned by Tian et al. (2014) in a recent review.

In the present work, CRT gave the lowest IFT values, and SEM characterization showed the lack of effectiveness of HF on CRT, although the manufacturer recommends this procedure as an alternative to gritblasting.

3 | 6. Conclusion

There is a growing variety of CAD-CAM composite blocks on the market, and recent breakthroughs have led to confusion about their specific properties. Among these properties, the microstructure is a critical factor, which has an important influence on the bonding properties of CAD-CAM composites. Indeed, PICNs, with their typical double-network microstructure resulting in a honeycomb polymer-based structure when etched, were shown to exhibit significantly better IFT values than CAD-CAM composites with dispersed fillers. The correlation found between the IFT and CAD-CAM composite surface enlargement after pretreatment shows the importance of the micro-mechanical bond on adhesive interface performance. In that view, the present results suggest that PICNs should be etched while DF should be sandblasted. The good results of PICNs compared to a gold standard such as lithium-disilicate glass-ceramic will need to be confirmed by further research, particularly via *in vitro* fatigue tests of the interface and clinical studies in the framework of partial bonded restorations.

3 | 7. Author contributions

M. Eldafrawy, contributed to design, data acquisition, analysis, and interpretation, drafted and critically revised the manuscript; M.G. Ebroin, P.A. Gailly, contributed to acquisition and analysis, critically revised the manuscript; J.-F. Nguyen, contributed to data analysis and interpretation, drafted the manuscript; M.J. Sadoun, contributed to conception, design, data analysis, and interpretation, critically revised the manuscript; A.K. Mainjot, contributed to conception, design, data analysis, and interpretation, drafted and critically revised the manuscript. All authors gave final approval and agree to be accountable for all aspects of the work.

3 | 8. Acknowledgements

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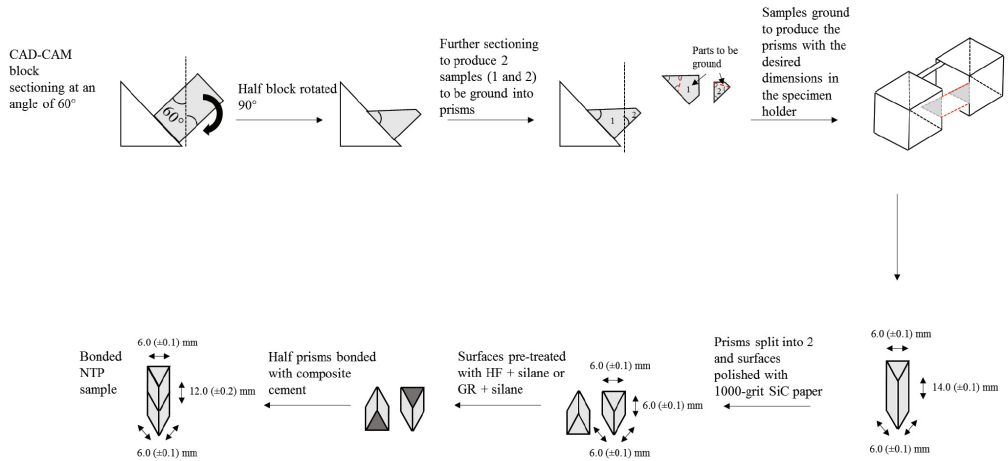
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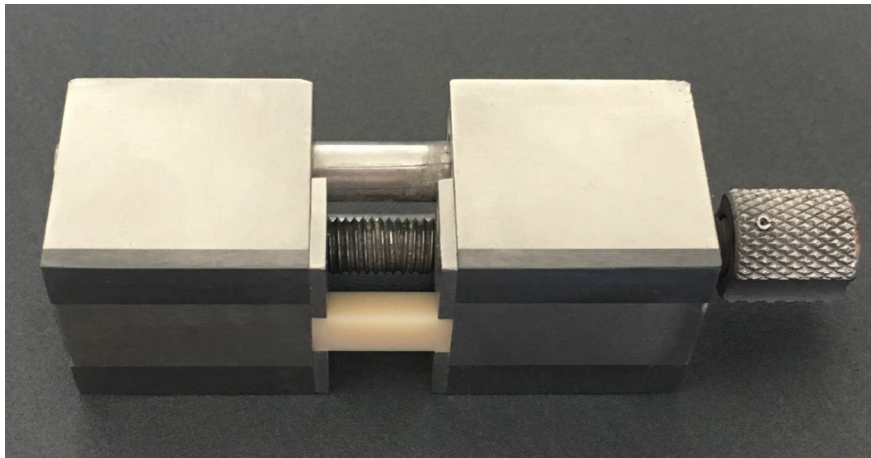
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3 | 10. Appendix

a

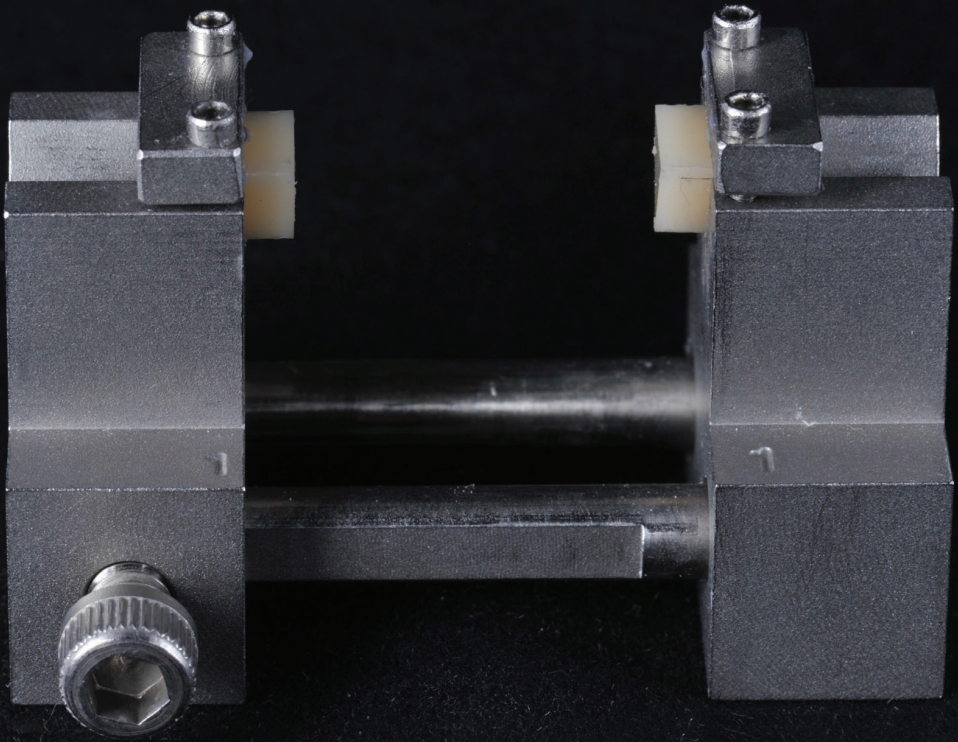


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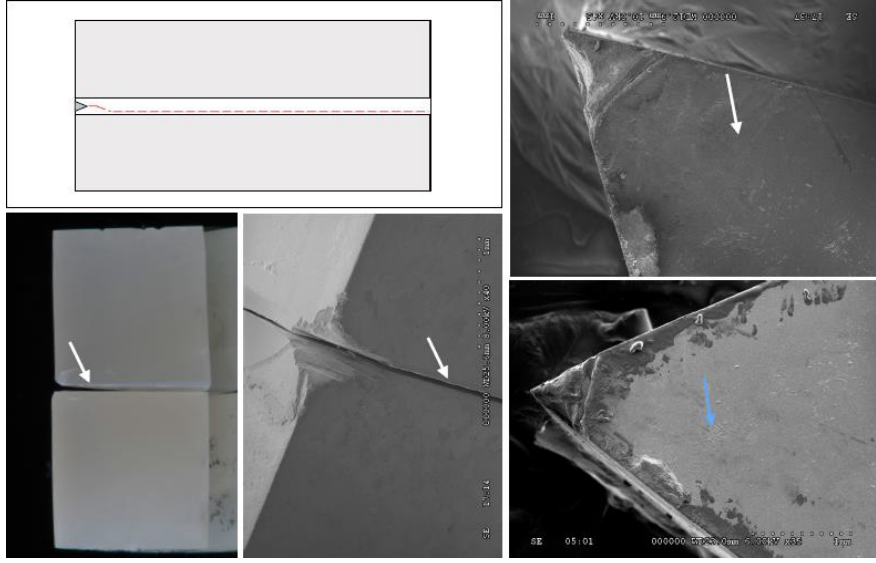
Appendix Figure 1 a) Schematic representation of the preparation of the NTP specimens for IFT measurement. b) Specimen holder with the prepared prism inside.

3

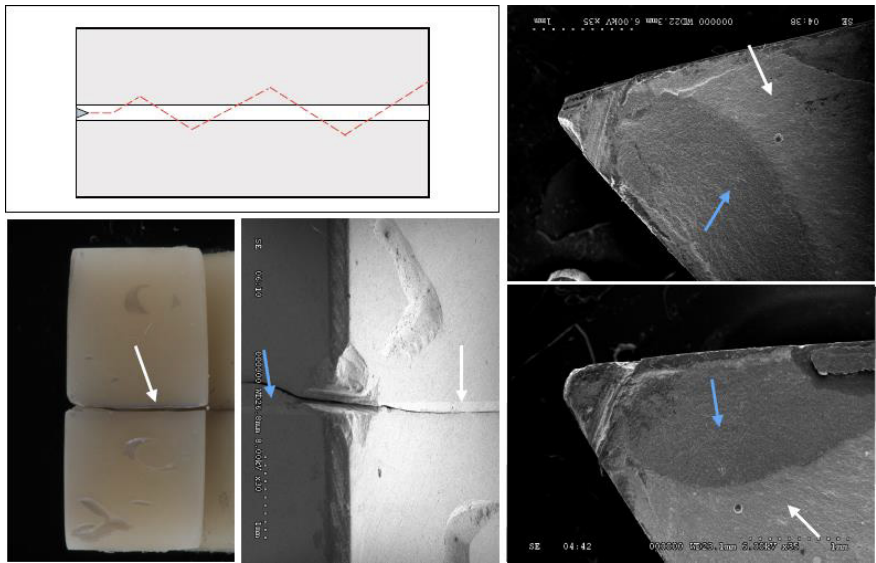


Appendix Figure 2 Custom-designed alignment system. A dual-cure composite cement (Variolink Esthetic DC, neutral shade, Ivoclar Vivadent, Schaan, Liechtenstein) was applied on each surface with the mixing syringe, then half prisms were put into contact and secured, which gave a cement thickness comprised between 70 and 100 μm (as measured on bonded samples by SEM).

a

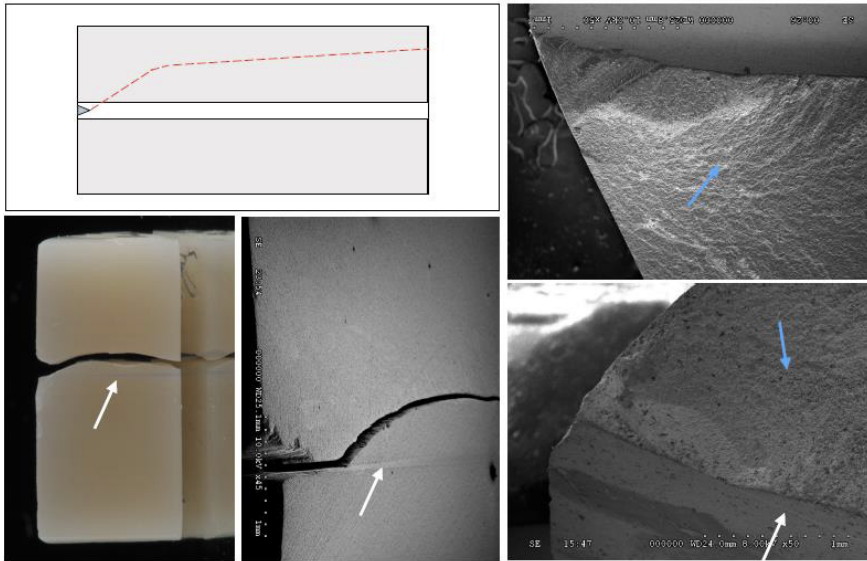


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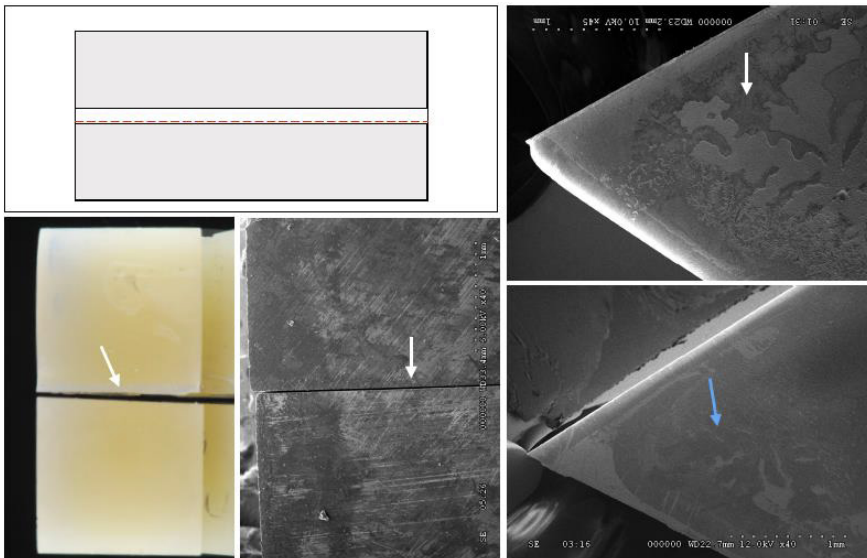


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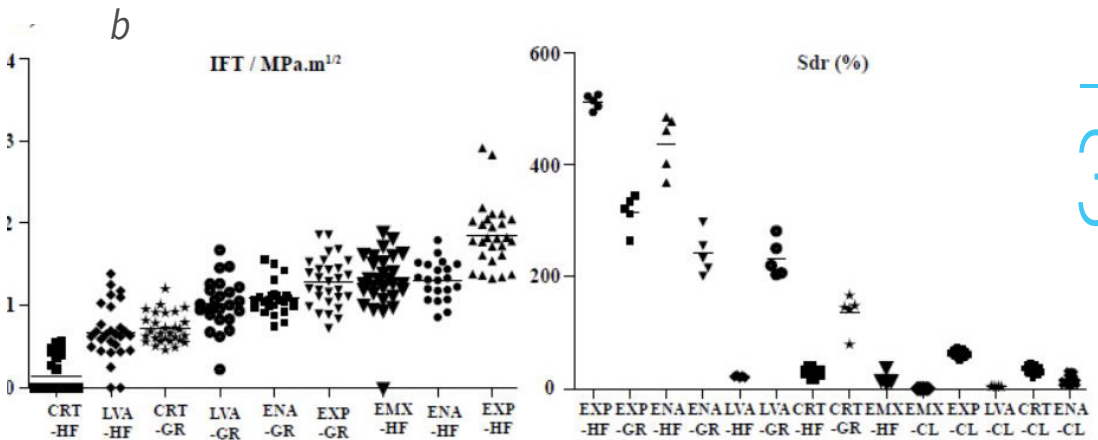
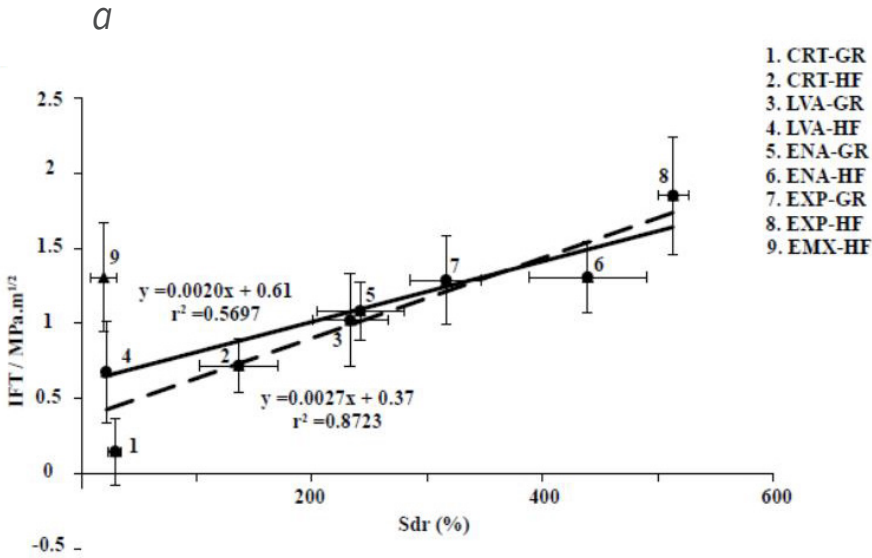


d



Appendix Figure 3 Typical failure modes encountered with the NTP test, illustrated with figures, pictures and SEM images of tested samples (white arrows indicate the composite cement layer and the blue CAD-CAM composite material).

- a) Adhesive failure (the crack propagated at the interface) in EXP-HF sample.
- b) Mixed failure (the crack propagated at the interface but with small portions of CAD-CAM composite or cement in the fracture surface) in ENA-HF sample.
- c) Cohesive failure (the crack propagated in the CAD-CAM composite) in ENA-HF sample.
- d) Premature failure in CRT-HF sample: the sample debonded at the interface with thermo-cycling.



Appendix Figure 4 a) IFT vs Sdr and their correlation curves with EMX-HF ($r^2=0.5697$) and without EMX-HF ($r^2=0.8723$). Mean and standard deviation for IFT and Sdr were obtained from measurements of each specimen. Note that the Sdr value cannot be negative and that a surface with an Sdr value equal to zero (ideal plane) can allow bonding and then be associated to an IFT value superior to zero.

b) Scatter Plot of IFT and Sdr. EXP-HF is etched experimental PICN, EXP-GR is gritblasted experimental PICN, EXP-CL is experimental PICN control, EMX-HF is etched IPS e.max CAD, EMX-CL is IPS e.max CAD control, ENA-HF is etched Enamic, ENA-GR is gritblasted Enamic, ENA-CL is Enamic control, LVA-HF is etched Lava Ultimate, LVA-GR is gritblasted Lava Ultimate, LVA-CL is Lava Ultimate control, CRT-HF is etched Cerasmart, CRT-GR is gritblasted Cerasmart, CRT-CL is Cerasmart control.

IFT / MPa.m ^{1/2}								
Factor		Mean ± SD	df	Type III sum of squares	Mean squares	F	Significance	
M: material class	PICN	1.39±0.41 ^a	1	19.627	19.627	186.554	*	
	Dispersed Filler	0.63±0.41 ^b						
S: surface treatment	HF	0.96±0.73 ^a	1	0.516	0.516	4.904	-	
	GR	1.03±0.33 ^a						
M*S	PICN	HF	1	4.583	4.583	43.564	*	
		GR						1.19±0.27 ^b
	Dispersed Filler	HF						0.41±0.39 ^d
		GR						0.86±0.29 ^c

Sdr (in %)								
Factor		Mean ± SD	df	Type III sum of squares	Mean squares	F	Significance	
M: material class	PICN	377.5±112.9 ^a	1	741435.323	741435.323	330.601	*	
	Dispersed Filler	105.2±91.6 ^b						
S: surface treatment	HF	250.8±233.9 ^a	1	3554.662	3554.662	1.585	-	
	GR	231.9±72.5 ^a						
M*S	PICN	HF	1	317312.909	317312.909	141.488	*	
		GR						279.0±51.0 ^b
	Dispersed Filler	HF						25.6±5.8 ^d
		GR						184.8±59.8 ^c

Appendix Table The results of a two-way analysis of variance (ANOVA 2) for the Interfacial Fracture Toughness (IFT) and Sdr (developed interfacial area ratio) of CAD-CAM composites. HF is hydrofluoric acid etching and GR is gritblasting. A significant difference was detected ($p < 0.05$). Superscript letters indicate statistically homogeneous subgroups within a material category (Scheffé's test, $\alpha = 0.05$). The same superscript letters demonstrate that there were no significant differences for each factor. DF indicates samples of Dispersed Filler (LVA, CRT) and PICN indicates Polymer-Infiltrated Ceramic Network materials (EXP, ENA). Df is the degrees of freedom. The F-statistic column is labeled "F".

SILANE INFLUENCE ON BONDING TO CAD-CAM COMPOSITES; AN INTERFACIAL FRACTURE TOUGHNESS STUDY

Chapter 4

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The Influence of Silane on the Interfacial Fracture Toughness of PICNs with Resin Cement. Eldafrawy M, Greimers L, Nguyen J-F, Sadoun M, Mainjot A. Congress of the French Society of Dental Biomaterials (SFBD), Paris, July 2017. Oral session.

4 | Abstract

Objectives: To evaluate silane influence on the interfacial fracture toughness (IFT) of composite cement, with the two sub-classes of CAD-CAM composites, polymer-infiltrated ceramic networks (PICN) and dispersed fillers (DF), after hydrofluoric acid etching (HF) or airborne-particle abrasion (AB). A secondary objective was to correlate results with developed interfacial area ratio (Sdr) and surface wettability.

Methods: Experimental PICN and DF blocks were cut into equilateral half-prisms, which were treated with HF or AB, then treated with an experimental silane or not and bonded to their counterparts with an experimental light-cure resin cement. After thermocycling, samples (n = 30 per group) were tested for IFT using the notchless triangular prism test in a water bath at 36°C. Moreover, profilometry and contact angle measurement were performed on rectangular samples of each group. Finally, bonding interface was analysed by SEM.

Results: PICN-HF treated with silane showed the highest IFT significantly. Three-way ANOVA revealed the influence of silane, material class and surface pre-treatment (HF or AB) on IFT ($p < 0.05$). When silane was used, IFT was correlated with Sdr, while surface wettability was increased. Silane application significantly increased IFT for PICN but not for DF, while PICN performed better with HF and DF with AB.

Significance: Silane increases IFT of composite cement with PICNs, but not with DF materials. Results suggest that silane increases the micromechanical bond by promoting resin cement spreading and penetration in surface roughness. This roughness is significantly higher for pre-treated PICNs than for DF due to their specific honeycomb microstructure when etched, which explains their better bonding properties.

Keywords: Dental materials, Prosthetic dentistry, Dental prosthesis retention, Polymer-infiltrated ceramic network, Composite cement, High-temperature high-pressure, polymerization, Fracture mechanics, Notchless triangular prism test, Surface roughness, Biomaterials.

4 | 1. Introduction

New advances in CAD-CAM processes and related materials allow for the realization of bonded restorations in high-performance composite materials, which exhibit better machinability than ceramics and are able to be milled at very low thickness, offering interesting perspectives in terms of minimally-invasive treatments [1–3]. However, there is a need to study their bonding properties [4] and the mechanism of composite cement adhesion to the material's surface, depending on material composition and microstructure [4].

According to the microstructure, CAD-CAM composites can be sub-classified into dispersed fillers (DF) and polymer-infiltrated ceramic networks (PICN). While DF consist of inorganic fillers, mainly silica-based glass mixed with an organic matrix, most often of urethane di-methacrylate (UDMA), eventually combined with tri-ethylene glycol di-methacrylate (TEGDMA), polymerized at high temperature (>100°C), PICNs (also called "hybrid ceramics") consist of monomers secondarily infiltrated into a pre-sintered glass-ceramic scaffold and polymerized under high temperature and pressure (HT-HP) (180°C–300 MPa) [5] (Fig. 1). This patented HT-HP polymerization process [6] provides a high degree of conversion of PICNs, which is advantageous in terms of improving the mechanical properties and monomer release [7–9], but raises some doubts regarding the chemical bonding between the free monomers and the resin cement [5]. Examples of commercially available DF include Lava Ultimate (3M ESPE, St. Paul, MN, USA), Cerasmart (GC, Tokyo, Japan), Shofu blocks (Shofu Inc., Kyoto, Japan), Tetric CAD (Ivoclar Vivadent, Schaan, Liechtenstein), Katana Avencia (Kuraray Noritake, Tokyo, Japan), Brilliant Crios (Coltene, Göppingen, Germany) and Grandio blocs (Voco, Cuxhaven, Germany), which are different in the amount, size and composition of fillers, as well as the organic matrix, while Vita Enamic (Vita Zahnfabrik, Bad Säckingen, Germany) is the only marketed PICN with 75 vol.%(86 wt.%) glass-ceramic.

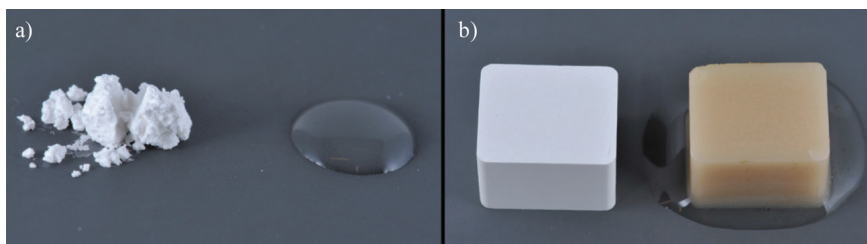


Fig. 1 a) Fillers that are incorporated by mixing in a monomer mixture to produce a dispersed filler (DF) composite block.
b) A pre-sintered glass-ceramic scaffold infiltrated with monomer, which is secondarily polymerized under high-temperature and high-pressure to produce a polymer-infiltrated ceramic network (PICN) block. In PICN, glass-ceramic particles are interconnected creating a double-network material (which has a lower content of organic phase than DF).

The effectiveness of composite cement bonding to materials was suggested to be related to two important parameters, the micromechanical interlocking, promoted by surface roughness, and the chemical bond, promoted by silane application [10]. Regarding classical light-cured indirect composites, increasing surface roughness by airborne-particle abrasion was shown to have a greater effect on bond strength than chemical bonding [4]. For CAD-CAM composite materials, the high degree of conversion of the polymers could be suspected to decrease the potential for a chemical co-polymerization with the resin cement, therefore micromechanical interlocking might be of even greater influence on the adhesive interface performance. Regarding DF CAD-CAM composites, airborne-particle abrasion was previously shown to be more effective than etching in increasing the surface roughness and consequently improving the bonding properties [4, 11, 12], while on the contrary, PICNs showed significantly higher bond strength results when etched than when treated with airborne-particle abrasion [11, 13, 14]. Indeed, previous work using fracture mechanics highlighted the influence of CAD-CAM composite microstructure on surface roughness and bonding properties [11]. PICNs have a typical double-network microstructure which results in a honeycomb polymer-based structure after etching, which enhance micro-mechanical retention with composite cement and yields significantly higher IFT values than DF [11, 15], with a direct correlation between IFT and surface roughness (expressed as the developed interfacial area ratio, Sdr) having been highlighted.

On the other hand, silane pre-treatment was shown to have a significant positive effect on glass-ceramics bonding [16] and is recommended for all CAD-CAM composites. At low pH, condensated silane molecules react with each other forming dimers, which then condensate to form siloxane oligomers. Subsequently, a further reaction between silane oligomers leads to the formation of strong siloxane bonds ($-\text{Si}-\text{O}-\text{Si}-$) on the fitting surface of the substrate [17, 18]. Another theory suggested that the mechanism of bond enhancement by silanes is that they improve the wettability of ceramic surfaces and reduce the contact angle of resin cements with ceramics. This could allow the resin cement to penetrate into the micro-roughness created on the surface after etching, resulting in increased micromechanical retention [19].

The bonding properties of CAD-CAM composites have been evaluated in a few studies using microtensile or micro(shear) bond strength tests to measure the influence of the type of pre-treatment, composite cement or material [13, 14, 20–24]. However, bond strength testing suffers from several drawbacks which may result in inaccuracies in the evaluation of the bond strength as a result of sample preparation after bonding, which leads to an increased number of pre-test failures, as well as problems relating to non-uniform stress distributions. A four-point bending test has been recently introduced to

reduce those issues [25–27]. The need for a different and more reliable method for the evaluation of the adhesive interfaces drove some authors to introduce fracture mechanics for evaluating the interfacial fracture toughness (IFT) (K_{IC}) of the adhesive layer [25, 26, 28–32]. IFT allows for the evaluation of the interfacial properties by stably initiating and propagating a crack through the bonded interface, and measuring the crack-propagation resistance or peeling resistance from the substrate, unlike bond strength testing which evaluates the mechanical strength of the whole assembly [25, 30]. Several types of fracture toughness tests have already been implemented with dental materials, such as the indentation fracture; single-edge notched beam; simple edge pre-cracked beam; surface crack in flexure; and the chevron notch short rod or beam test [32–35]. The latter has been modified into various versions adapted to dental samples [29,30,36]. Among these versions, the notchless triangular prism (NTP) test, introduced by Ruse et al. [36], was reported to be a simple method [31] and was previously proposed to evaluate both the fracture toughness of a material and the fracture toughness of bonded interfaces [36, 37]. Finite element analysis showed that the maximum tensile stress was concentrated at the tip of the crack [36], providing a good level of control and stable crack growth at a low cross-speed (0.05 mm/min); thus, allowing for reliable measurements and the possibility of performing a fatigue process study, controlling crack propagation for a given K_I . In addition, the NTP does not require a notch prior to testing, only a crack initiation point, therefore minimizing the bias related to creation of the notch, especially with the low adhesive interface thickness.

Consequently, the main objective of this study was to evaluate the influence of silane on the interfacial fracture toughness (IFT) of composite cement with two sub-classes of CAD-CAM composites, polymer-infiltrated ceramic network (PICN) and dispersed filler (DF), after etching or airborne-particle abrasion. A secondary objective was to correlate the results with developed interfacial area ratio (Sdr) and surface wettability. The null hypotheses are that (1) material class; (2) surface texture after pre-treatment; (3) silane, do not influence IFT.

4 | 2. Materials and methods

Experimental blocks of the two classes of CAD-CAM composites, PICN and DF, were manufactured and tested with experimental resin cement and silane. The details of the compositions of all the materials are described below.

2.1. Experimental material compositions

2.1.1. Experimental PICN

The process of producing the porous ceramic network is described by Nguyen et al. [8]. After sintering, the porous network consisted of 73.8 vol.% slip casted Vita Mark II glass-ceramic powder (Vita Zahnfabrik, 2.6 μm D50 grain size distribution); which were then infiltrated by UDMA and *di-tert-amyl* peroxide (initiator), and polymerized under HT-HP (180°C–300 MPa) to give the final PICN blocks of 85 wt.% glass-ceramic network and 15 wt.% organic component (Fig. 1).

2.1.2. Dispersed filler resin blocks

The dispersed filler resin composite blocks were composed of 2:1 (weight) UDMA and TEGDMA, with the addition of 1 wt.% benzoyl peroxide (Aldrich, Darmstadt, Germany) to form the organic part. The mixture was left for 24 h to stabilize.

67 wt.% silanated barium borosilicate glass (0.7 μm) (Esschem Europe, Seaham, England) and 3 wt.% synthetic amorphous organosilane-treated silica (40 nm) (Evonik Industries, Essen, Germany) were added to 30 wt.% of the organic mixture and polymerized under HT-HP, as was done for PICN.

2.1.3. Experimental composite cement

The light-cure experimental composite cement was composed of 2:1 (weight) UDMA and TEGDMA with the addition of 1 wt.% 4 N, N-trimethylanilin and 0.5 wt.% camphorquinone (Aldrich, Darmstadt, Germany) to initiate the photo-polymerization. After mixing, it was left for 24 h to stabilize. Silanated barium borosilicate (0.7 μm) was added at 22 parts of 100% of the organic mixture and amorphous organosilane-treated silica nano-fillers (40 nm) was added at 13 parts.

2.1.4. Silane

The experimental silane solution of 3-(trimethoxysilyl) propylmethacrylate (Aldrich, Darmstadt, Germany) (2 wt.%) was dissolved in 92.8 wt.% absolute ethanol, 5 wt.% deionized water and 0.2 wt.% acetic acid.

2.2. Sample preparation

2.2.1. Prism manufacturing

CAD-CAM blocks were cut at an angle of 60° with a low-speed saw (Isomet; Buehler, Lake Bluff, IL, USA) under continuous water irrigation to produce the prism-shaped samples, which were then wet-ground into the desired 14 ± 0.1 mm long triangular prisms, 6 ± 0.1 mm on a side with 220 grit silicon carbide (SiC) paper, at 300 rpm (Struers, Ballerup, Denmark) using a custom-built specimen holder. A total of 240 prisms were manufactured (120 PICN + 120 DF) which were split into two to produce the half prisms.

2.2.2. Surface pre-treatment

Bonded surfaces were polished with 1000 grit SiC paper to produce the equilateral 6 ± 0.1 mm long prisms. All samples were sonically cleaned in ethanol for 3 min then dried with oil-free dry air for 10 s. PICN and DF samples were then divided into two groups ($n = 60$ samples per group).

For the first group, 5% hydrofluoric acid (HF) (Vita ceramics etch, Vita Zahnfabrik, Bad Säckingen, Germany) was applied and left for 60 s, washed under running water for another 60 s, air dried, then sonically cleaned in ethanol for 3 min and again air dried.

For the second group, airborne-particle abrasion (AB) was performed using $50 \mu\text{m}$ Al_2O_3 particles (Danville, Zürich, Switzerland) for 5 s in a perpendicular direction at a distance of 1 cm from the sample at a pressure of 1 bar for PICN and 1.5 bar for DF. Samples were then sonically cleaned in ethanol for 3 min and air dried.

Each group was further divided into 2 sub-groups ($n = 30$ per sub-group); one sub-group receiving silane treatment (S), which was applied with a plastic brush and left for 60 s then dried with a stream of hot air (100°C) for 30 s. The other sub-group was bonded immediately following surface pretreatment.

2.2.3. Bonding

Half prisms were fixed into a custom-designed computer-controlled (SMC 100, Newport Corporation, California, USA) motorized alignment system (Newport Motion Controller) which controls the cement thickness at a precision of $0.1 \mu\text{m}$, and the resin cement was applied on each of the pre-treated surfaces using a plastic spatula. Prisms were put into contact and the cement thickness was set to $50 \mu\text{m}$. High power ($1200 \text{ mW}/\text{cm}^2$) light applications were performed on each of the three sides of the prisms at close proximity (Bluephase 20i; Ivoclar Vivadent, Schaan, Liechtenstein) and an additional 40 s of curing was applied at a distance of 2 mm on each side after removal from the alignment apparatus to ensure optimal curing.

2.2.4. Aging

Samples were left for 24 h at 36°C, 90% humidity (VCN 100, Vötsch Industrietechnik GmbH, Balingen, Germany), after which they were polished to remove excess resin cement using 1000 grit SiC, then submitted to thermocycling for 10,000 cycles at 5–55°C, with a dwelling time of 30 s in each bath.

2.3. Interfacial fracture toughness testing

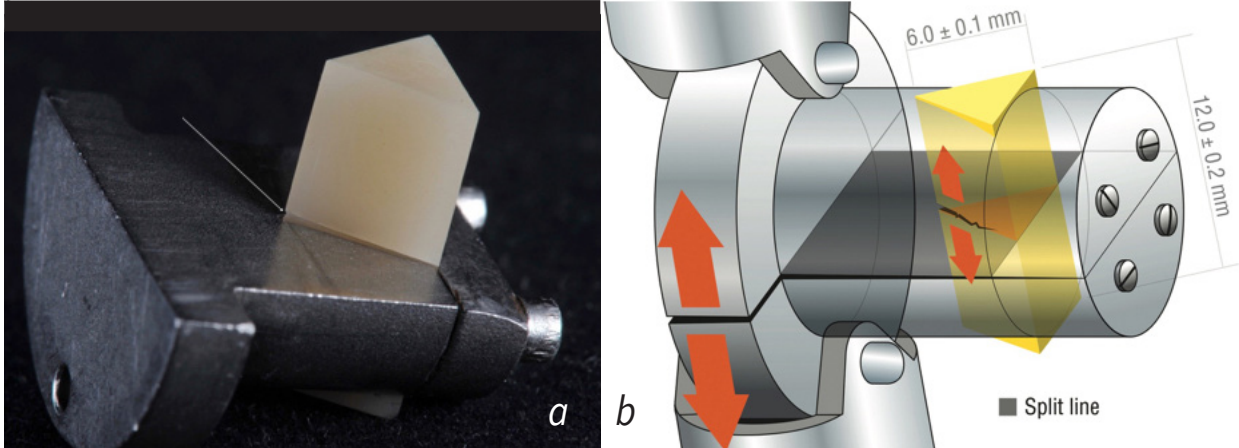
IFT was measured using the NTP test following the procedure described by Ruse et al. [36], in which samples were fixed into one half of the specimen holder and a crack initiation point (~0.1 mm) was made using a sharp scalpel under a light microscope at a magnification of ×20. After securing the other half of the specimens, samples were mounted on the computer-controlled (Bluehill, Instron Canada Inc., Burlington, ON) universal testing machine (Instron model 5565) within a water bath at 36°C, at a cross-head speed of 0.05 mm/min (Fig. 2). The values of load were recorded at failure arrest in a tensile mode and IFT was calculated using the formula $K_{IC} = Y_{min}^* P_{max} / DW^{1/2}$, where P_{max} is the maximum load at failure, D is the NTP specimen diameter (12 mm), W is the NTP specimen length (10.5 mm) and Y_{min} is the dimensionless stress intensity factor coefficient minimum (28) as given by Ruse et al. [36]. All prisms were examined under the light microscope to determine the mode of failure. Adhesive failures are those with a crack propagating at the interface between the resin cement and the composite material, while in mixed failures the crack propagates at the interface but with small portions of composite material or resin cement in the fracture surface. In cohesive failures, the crack propagates only within the composite material and samples that fracture in a cohesive mode are excluded from the data analysis [11, 25].

2.4. Contact angle measurement

Fifteen rectangular samples (17.5 × 14.0 × 4.0 mm) of PICN and DF, in addition to 10 samples of UDMA polymerized under HT-HP and a pure glass-ceramic represented by Vita Mark II (Vita Zahnfabrik), were manufactured and polished in the same way as described before. For PICN and DF, 5 samples were treated with HF, 5 with AB, following the previously described procedures of bonding, and 5 were not submitted to a surface treatment. For UDMA and Vita Mark II, only untreated and etched samples were analyzed ($n = 5$ per group). Samples were cleaned sonically in alcohol, air-dried and analyzed before and after silanization by measuring the contact angle of 3 drops of deionized water on the surface after 60 s (DSA-30, Kruss, Hamburg, Germany). An identical set of samples were tested for contact angle measurement with a drop of TEGDMA instead of water.

Fig. 2 Notchless triangular prism (NTP) test.

- a) A bonded prism fixed to one half of the cylindrical mounting block, with an arrow pointing at the crack initiation point.
- b) A schematic illustration of the NTP apparatus in motion, with a bonded prism fixed within. The arrows pointing in opposite directions indicate the application of tensile force on the bonded interface, marked by the split line, with crack propagation starting from the tip of the prism at the crack initiation point until fracture or crack arrest. Figures were reprinted by permission of SAGE Publications Inc. from Eldafrawy et al. [11].



2.5. Developed interfacial area ratio

Three rectangular samples of DF and PICN of the same dimensions as the ones used for contact angle measurement were manufactured and polished with 1000-grit SiC paper. Two of the samples were treated with HF and airborne-particle abrasion following the previously described procedures. The third sample was left untreated. Profilometry was carried out on the samples to measure the developed interfacial area ratio (Sdr), which is expressed as the percentage of additional surface area contributed by the texture compared to an ideal plane the size of the measurement region. The Sdr is obtained by calculating the topographical area with respect to this ideal plane and gives the surface enlargement induced by the different pre-treatments. Measurements were performed using an optical profiler (Contour GT-I; Bruker) in high-resolution vertical scanning interferometry (VXI) mode at <1 nm, independent of the objective. Five measurements were taken at the centre of each sample with a $\times 115$ objective (image size: 0.06×0.04 mm²; optical lateral resolution: 0.33 μ m) with no filtering and removal of the tilt and cylinder terms. The values of the Sdr were obtained by calculating the mean of 5 values measured for each sample.

2.6. SEM surface characterization

Rectangular samples of PICN, DF, UDMA and Vita Mark II were manufactured and polished. For PICN and DF, one sample was treated with HF, one with airborne-particle

abrasion and the third sample was left untreated. For UDMA and Vita Mark II, only etched and untreated samples were prepared. Immediately following surface pre-treatment, each sample was cleaned sonically in ethanol for 3 min and subsequently gold-coated to be analyzed by scanning electron microscopy (SEM) (S-3000 N; Hitachi, Tokyo Japan).

2.7. SEM interface analysis

Four rectangular samples of each of PICN and DF, and 2 samples of Vita Mark II were manufactured and polished the same way described before. For PICN and DF, surface pre-treatments (HF and AB), with and without silane application, were carried out using the same protocol described for bonding. For Vita Mark II, only HF was applied. On each sample, a drop of experimental composite cement devoid of fillers was applied perpendicular to the surface and left for 60 s, then light-cured for another 60 s. Samples were then embedded in epoxy resin which was left to set for 24 h. The samples were then cut using a low-speed saw through the material/composite cement interface. They were then polished with 600 and 1000 grit SiC paper, then with a diamond pad and scanned by the SEM.

2.8. Statistical analysis

IFT and Sdr results were analyzed by one-way ANOVA followed, if warranted, by Scheffé multiple mean comparisons ($\alpha = 0.05$), using PASW Statistics 18 (SPSS, USA). Weibull statistics parameters were calculated for the IFT data using the Weibull statistics option in Excel (Microsoft, USA). The Weibull distribution was described in previous studies [38,39]. Two-way ANOVA was used to evaluate the influence of microstructure, surface pre-treatment and both combined on the Sdr, while 3-way ANOVA was used to evaluate the influence of microstructure, surface pre-treatment, silane and all three combined on the IFT. Pearson's tests were used to verify the presence of statistically significant correlations between the IFT and Sdr ($\alpha = 0.05$).

4 | 3. Results

3.1. IFT

The IFT values (mean and standard deviations) of the different groups, along with the statistical analysis, are summarized in Fig. 3. PICN-HF-S showed the highest IFT ($2.02 \pm 0.37 \text{ MPa}\cdot\text{m}^{1/2}$) significantly followed by PICN-AB-S ($1.6 \pm 0.48 \text{ MPa}\cdot\text{m}^{1/2}$). The lowest IFT values were shown by DF-HF with and without silane ($0.8 \pm 0.21 \text{ MPa}\cdot\text{m}^{1/2}$ and

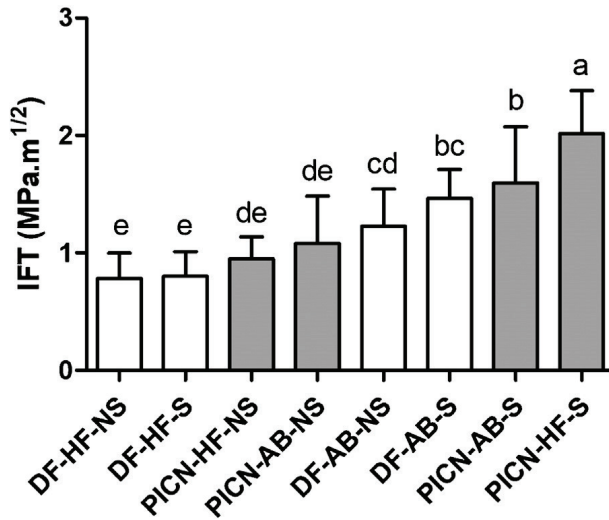


Fig. 3 A bar graph of the interfacial fracture toughness (IFT) measurement. Results are expressed as the means \pm SD. Superscript letters indicate statistically homogeneous subgroups within a material category (1-way ANOVA followed by Scheffé test, $\alpha = 0.05$). The same superscript letters demonstrate that there were no significant differences for each factor. PICN-HF-S is etched silanized PICN; PICN-AB-S is airborne-particle abraded silanized PICN; DF-AB-S is airborne-particle abraded silanized dispersed fillers; DF-AB-NS is airborne-particle abraded non-silanized dispersed fillers; PICN-AB-NS is airborne-particle abraded non-silanized PICN; PICN-HF-NS is etched non-silanized PICN; DF-HF-S is etched silanized dispersed fillers; DF-HF-NS is etched non-silanized dispersed fillers.

$0.78 \pm 0.22 \text{ MPa.m}^{1/2}$, respectively). Silane application was shown to have a significant effect on IFT for PICN but not for DF materials. All the samples failed in an adhesive or a mixed manner, no cohesive or pre-test failures were observed in any of the tested groups. The amount of adhesive (A) and mixed (M) failures were: DF-HF-NS (A/30, M/0); DF-HF-S (A/29, M/1); PICN-HF-NS (A/30, M/0); PICN-AB-NS (A/29, M/1); DF-AB-NS (A/30, M/0); DF-AB-S (A/27, M/3); PICN-AB-S (A/27, M/3); PICN-HF-S (A/26, M/4). Weibull analysis of the PICNs was higher when treated with HF than when treated with airborne-particle abrasion. In contrast to this, Weibull analysis of the DFs was higher when treated with airborne-particle abrasion than when treated with HF (Fig. 4). Three-way ANOVA highlighted the significant effect of material class, surface pre-treatment and silanization, as well as the significant effect of all three combined on the IFT with resin cement (Table 1).

3.2. Sdr

The mean and standard deviations of the Sdr measurements for each group of samples, along with the results of the statistical analysis, are summarized in Fig. 5. PICN-HF showed the highest Sdr, significantly followed by PICN-AB, while there was no

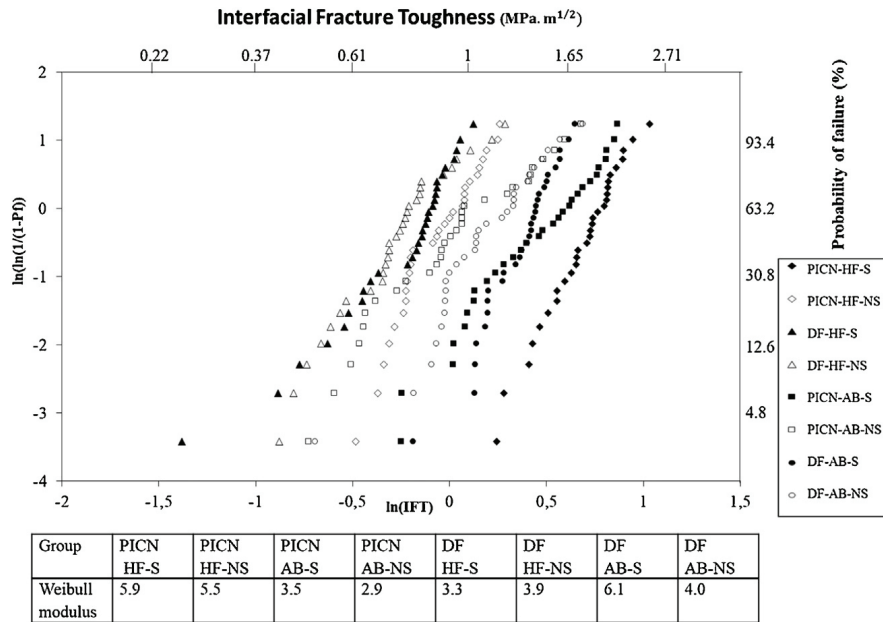


Fig. 4 Weibull plots of the interfacial fracture toughness (IFT) results. PICN-HF-S is etched silanized PICN; PICN-AB-S is airborne-particle abraded silanized PICN; DF-AB-S is airborne-particle abraded silanized dispersed fillers; DF-AB-NS is airborne-particle abraded non-silanized dispersed fillers; PICN-AB-NS is airborne-particle abraded non-silanized PICN; PICN-HF-NS is etched non-silanized PICN; DF-HF-S is etched silanized dispersed fillers; DF-HF-NS is etched non-silanized dispersed fillers.

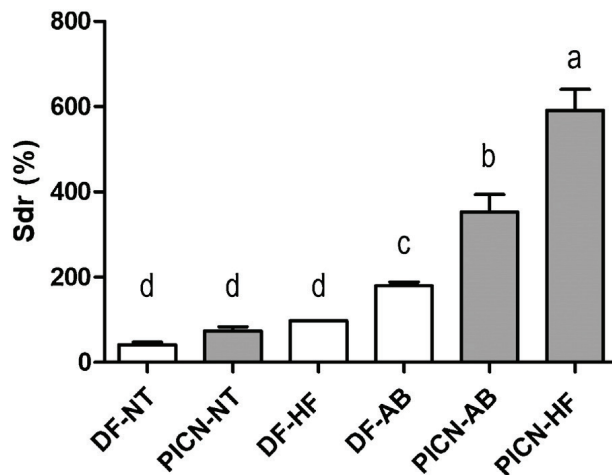


Fig 5 A bar graph of the developed interfacial area ratio (Sdr) measurement. Results are expressed as the means \pm SD. Superscript letters indicate statistically homogeneous subgroups within a material category (1-way ANOVA followed by Scheffé test, $\alpha = 0.05$). The same superscript letters demonstrate that there were no significant differences for each factor. PICN-HF is etched PICN; PICN-AB is airborne-particle abraded PICN; DF-AB is airborne-particle abraded dispersed fillers; DF-HF is etched dispersed fillers; PICN-NT is PICN with no treatment; DF-NT is dispersed fillers with no treatment.

IFT (MPa.m ^{1/2})									
Factor				Mean ± SD (95% confidence interval)	df	Type III sum of squares	Mean square	F	Significance
M: material class	PICN			1.40±0.58 ^a (1.34-1.46) 1.07±0.21 ^b (1.01-1.13)	1	6.507	6.507	61.905	*
ST: surface treatment	HF	AB		1.14±0.57 ^b (1.08-1.20) 1.33±0.44 ^a (1.27-1.39)	1	2.205	2.205	20.971	*
Si: Silane	Silane			1.47±0.55 ^a (1.41-1.53) 1.00±0.34 ^b (0.94-1.05)	1	13.431	13.431	127.765	*
M × ST PICN	HF			1.48±0.61 ^a (1.40-1.57) 1.31±0.54 ^a (1.23-1.39)	1	7.896	7.896	75.117	*
	DF	AB		0.79±0.21 ^b (0.71-0.87) 1.35±0.31 ^a (1.26-1.43)					
M × Si PICN	Silane			1.81±0.47 ^a (1.72-1.89) 0.99±0.33 ^b (0.91-1.07)	1	7.103	7.103	67.571	*
	DF	Silane		1.13±0.40 ^b (1.05-1.22) 1.00±0.35 ^b (0.92-1.09)					
ST × SiHF	Silane			1.41±0.68 ^a (1.33-1.49) 0.86±0.22 ^c (0.78-0.95)	1	0.304	0.304	2.892	-
	AB	Silane		1.53±0.38 ^a (1.45-1.61) 1.13±0.39 ^b (1.05-1.21)					
M × ST × Si	PICN	HF	Silane	2.02±0.37 ^a (1.90-2.13) 0.95±0.19 ^{de} (0.83-1.07)	1	1.918	1.918	18.245	*
		AB	Silane	1.60±0.48 ^b (1.48-1.71) 1.03±0.43 ^{de} (0.91-1.15)					
	DF	HF	Silane	0.80±0.21 ^e (0.69-0.92) 0.78±0.22 ^e (0.66-0.90)					
		AB	Silane	1.46±0.25 ^{bc} (1.35-1.58) 1.23±0.32 ^{cd} (1.11-1.34)					

Table 1 The results of a 3-way ANOVA of the interfacial fracture toughness (IFT). * indicates that a significant difference was detected ($p < 0.05$). Superscript letters indicate statistically homogenous sub-groups within a material category (Scheffé test, $\alpha = 0.05$). The same superscript letters demonstrate that there were no significant differences for each factor. The F-statistic column is labeled "F". Number of samples (n) = 30.

significant difference between DF-HF, PICN-NT and DF-NT. Images of profilometry are shown in Fig. 6.

Two-way ANOVA highlighted the significant effect of material class, surface pre-treatment and both combined on the Sdr (Table 2).

				Sdr (%)						
Factor				Mean \pm SD	df	Type III sum of squares	Mean square	F	Significance	
M: material class	PICN			471.7 \pm 132.5	1	522235,281	522235,281	465,157	*	
		DF		143.2 \pm 43.8						
ST: surface treatment	HF			371.5 \pm 262.4	1	28503,800	28503,800	25,388	*	
		AB		266.3 \pm 95.3						
M * ST	PICN	HF		590.7 \pm 49.9	1	120689,794	120689,794	107,499	*	
			AB							352.8 \pm 40.6
		DF	HF							97.5 \pm 1.1
			AB							179.8 \pm 8.6

Table 2 The results of a 2-way ANOVA of the developed interfacial area ratio (Sdr). * indicates that a significant difference was detected ($p < 0.05$). Scheffé test, $\alpha = 0.05$. The F-statistic column is labeled "F". Number of samples (n) = 30.

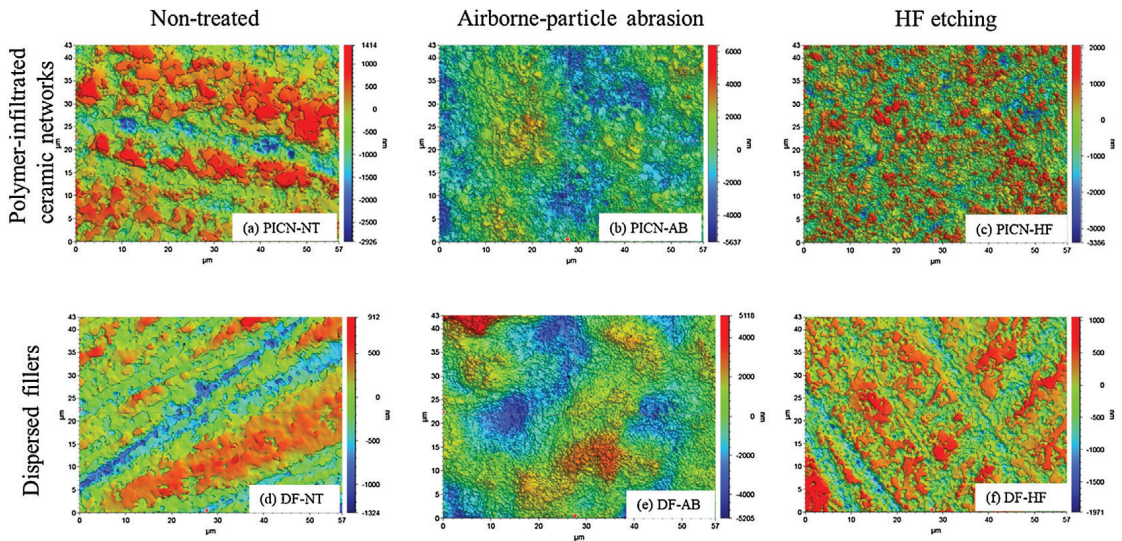


Fig. 6 Profilometry images of the samples following different surface pre-treatments. The blue areas represent negative values, while the red areas represent positive values. (a) Non-treated PICN; (b) airborne-particle abraded PICN; (c) etched PICN; (d) Non-treated DF; (e) airborne-particle abraded DF; (f) etched DF.

3.3. Correlation between the IFT and Sdr

Statistical analysis using Pearson's correlation coefficient revealed that there was a strong ($r^2= 0.8411$) and significant ($p < 0.05$) correlation between the IFT of PICN and DF, when pre-treated with silane, and the Sdr. This positive correlation suggests that, under the conditions of this study, over 84% of the variation in the IFT was explained by the Sdr (Fig. 7). The correlation was found to be weaker when the non-silanized samples were included ($r^2= 0.5918$).

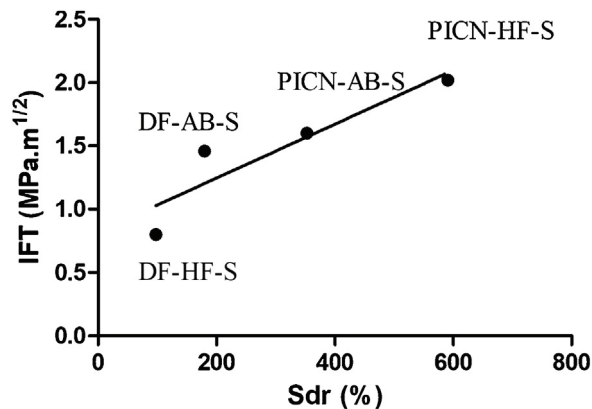


Fig. 7 Interfacial fracture toughness (IFT) versus developed interfacial area ratio (Sdr) and their correlation curve ($r^2= 0.8411$). Results are expressed as the means \pm SD of IFT and Sdr for the tested samples of polymer infiltrated ceramic network (PICN) materials and dispersed filler (DF). PICN-AB-S is airborne-particle abraded silanized PICN; PICN-HF-S is etched silanized PICN; DF-AB-S is airborne-particle abraded silanized dispersed fillers; DF-HF-S is etched silanized dispersed fillers.

3.4. Contact angle

Contact angle measurements with water are displayed in Fig. 8. For DF, PICN and UDMA, surface pre-treatment with AB or HF increased the contact angle, i.e. lowered the wettability. On the other hand, following silane application, the contact angle was lowered, i.e. the wettability was increased. Conversely, Vita Mark II samples showed increased wettability after etching, and subsequent silane application decreased it. For contact angle measurement using TEGDMA, all the samples gave a zero value.

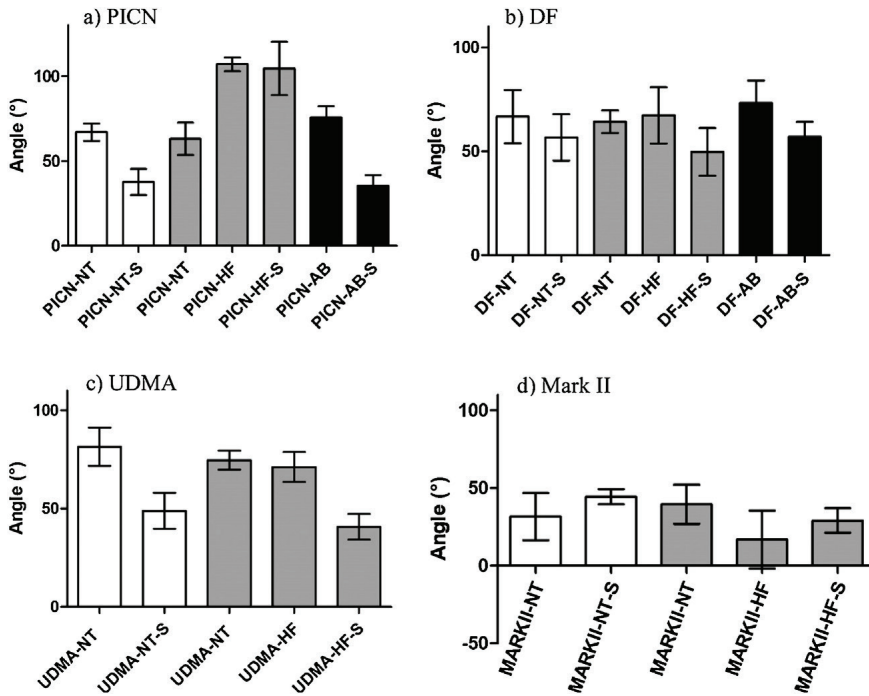


Fig. 8 Bar graphs of the results of contact angle measurements. (a) PICN; (b) DF; (c) UDMA; (d) Vita Mark II. Bars with the same color represent the same sample. Results are expressed as the means \pm SD.

3.5. SEM surface characterization

SEM images of non-treated and etched PICN, DF, Vita Mark II and pure UDMA, as well as PICN and DF with airborne-particle abrasion are displayed in Fig. 9. The difference in microstructure between PICN and DF is shown in the non-treated samples, in which PICN, with its ceramic network, has a higher glass-ceramic density compared to DF, however sintering necks are difficult to highlight due to the presence of the polymer. Etching of PICN resulted in a specific microstructure of the surface, characterized by dissolution of the glass-ceramic network and the presence of a typical polymer-based honeycomb structure (Fig. 9b), including micro- and nano-porosities. On the other hand, HF did not create as much roughness on the surface of the DF as it did with PICN, while Vita Mark II showed marked dissolution of the glass-ceramic at the surface and there was no observable change on the surface of UDMA after etching. The effect of AB on both classes, PICN and DF, was very slight in comparison with HF.

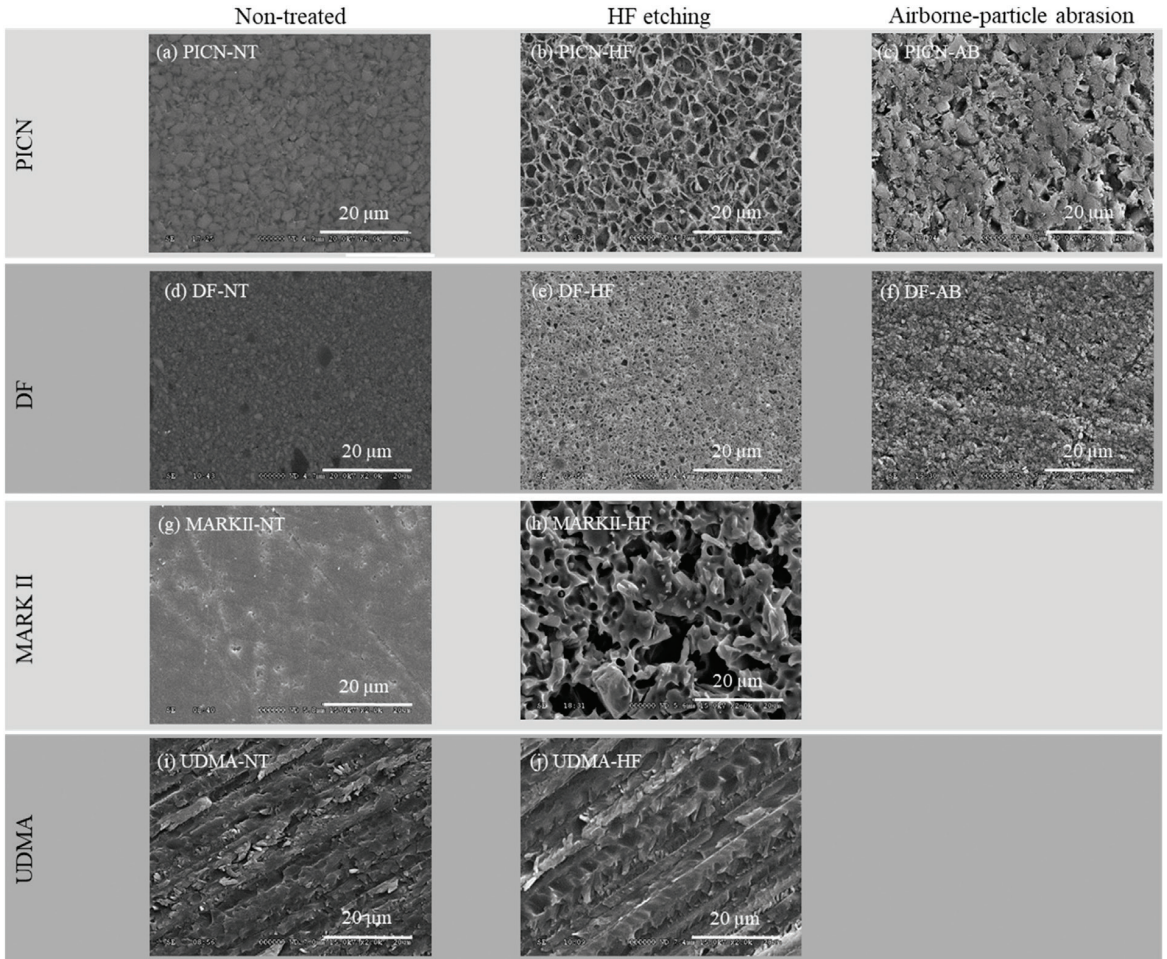


Fig. 9 Scanning electron microscopy characterization of the samples following different surface pre-treatments. a) Non-treated PICN; b) etched PICN; c) airborne-particle abraded PICN; d) non-treated DF; e) etched DF; f) airborne-particle abraded DF; g) non-treated Mark II; h) etched Mark II; i) non-treated UDMA; j) etched UDMA.

3.6. SEM interface analysis

Fig. 10 shows the SEM images of the interface, highlighting the presence of gaps between glass-ceramic and composite cement on PICN-HF, PICN-AB, and etched Vita Mark II samples in comparison to their silanized counterparts. In contrast, such gaps were not highlighted on DF samples, whatever the type of pre-treatment.

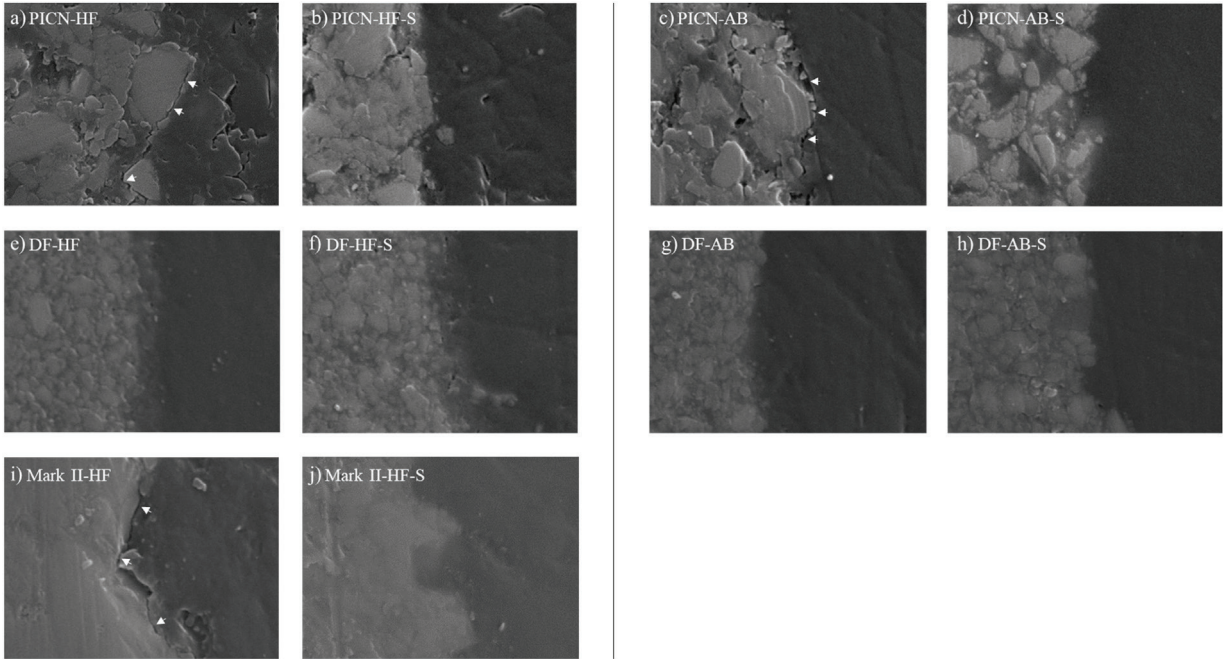


Fig. 10 Scanning electron microscopy of the cement interface with PICN, DF and Vita Mark II. The arrows are pointing at areas of incomplete wettability.

- a) Etched PICN;
- b) etched silanized PICN;
- c) airborne-particle abraded PICN;
- d) airborne-particle abraded silanized PICN;
- e) etched DF;
- f) etched silanized DF;
- g) airborne-particle abraded DF;
- h) airborne-particle abraded silanized DF;
- i) etched Vita Mark II;
- j) etched silanized Vita Mark II.

4 | 4. Discussion

The NTP test was successfully used to evaluate adhesive interfaces and the obtained results were shown to be within the same range of values compared to previous work using the same method to evaluate IFT of composite cement with PICN and DF CAD-CAM composites [11, 15]. In the present work, the alignment system for half-prism bonding was improved to accurately control the cement thickness, which was set at 50 μm to be close to clinical situations [40, 41]. AB pressure varied between the 2 materials because the authors followed the instructions of the commercially available products, Vita Enamic (Vita Zahnfabrik) for PICN and Cerasmart (GC) for DF. Thermo cycling was performed following recent guidelines [42], with aging having an important influence on the reliability of adhesive interfaces due to hydrolytic degradation of the bonding interface [43]. The samples were tested in a water bath set at 36°C to provide a close approximation of the in-mouth conditions and to eliminate any bias resulting

from differences in temperature [31]. Finally, the use of experimental materials was intended to control material composition and microstructure.

According to this study, the material microstructure (PICN or DF) and the surface pre-treatment (HF or AB) were confirmed to significantly influence IFT with composite cement, hence the first and second null hypotheses were rejected [11]. PICNs were shown to perform significantly better than DF, with higher reliability as confirmed by Weibull analysis (Fig. 4), and showed higher IFT when etched, while DF should be pre-treated with airborne-particle abrasion, confirming previous results [11]. Material microstructure was previously shown to be a critical factor, which has an important influence on the bonding properties of CAD-CAM composites. The typical honeycomb polymer-based structure of PICN, when etched, was shown to exhibit significantly higher surface enlargement and consequently an increased chance of micromechanical bond (Figs. 5 and 9b). Silane application was shown to have a significant effect on IFT for PICN, but not for DF materials. A strong correlation between Sdr and IFT was found only when silane was applied, thus the third null hypothesis was rejected for PICN, but not for DF. One explanation is that, as described in literature [19], silane increases material surface wettability, which can promote composite cement spreading into the high surface roughness of PICN and consequently micromechanical bond. Indeed, contact angle measurements showed that if, as expected, surface HF or AB treatment decreased wettability with water of PICN, DF and UDMA, silane application increased it again in a significant way for all materials except for Vita Mark II. It must be noted that TEGDMA, with its low viscosity, was shown to spread readily on all the surfaces irrespective of the material, pre-treatment or silane application. The interface SEM observations (Fig. 10) support this explanation, highlighting the presence of gaps between glass-ceramic and composite cement on non-silanized PICN and glass-ceramic (Vita Mark II) samples. However, findings also support the second reported effect of silane [16,19], which is the creation of a chemical bond with glass-ceramics, also explaining the presence of gaps after composite cement polymerization and retraction. The slight decrease of wettability with silane application on the etched Vita Mark II sample surfaces, also promotes the hypothesis of the chemical bonding to explain silane positive effect on glass-ceramics [16, 19]. In fact, the mode of action of silane is unclear and seems very complex. Its amphiphilic nature can explain its affinity for both the inorganic and organic parts of PICN and DF materials, as for water and TEGDMA, which could promote the energy increase of all surfaces.

Until now, few studies have evaluated the influence of silane pre-treatment on bonding properties of CAD-CAM composites with resin cement. DF and PICN represented by Lava Ultimate and Vita Enamic respectively, were tested for MTBS with 2 different

composite resins, with etching and airborne-particle abrasion, with and without silane, for each type of pre-treatment [14]. When silane was applied, HF-PICN also gave better results than AB-DF or HF-DF, while there was no significant effect of silane on Lava Ultimate, as observed with DF in the present study. However, in a recent study on different DF CAD-CAM composites [44] the shear bond strength was significantly higher with sandblasting and silanization than with sandblasting alone, except for one material, which was attributed to damages induced by the sandblasting procedure. The positive influence of silane on the bond strength for PICN was also highlighted in a previous study conducted by Elsaka [13], in which the MTBS values, after aging in water for 30 days, were significantly higher when silane was applied than when the material was etched and bonded directly.

4 | 5. Conclusion

Silane increases bonding effectiveness of resin cement to PICNs but not to DF materials. Results of the present study highlight the importance of material microstructure and consequently surface properties on bonding effectiveness to composite cement. They suggest that silane improves micromechanical interlocking by promoting surface wettability and composite cement penetration in PICN high surface roughness when pre-treated. Indeed, PICNs, with their specific honeycomb microstructure when etched, exhibited significantly higher surface roughness, as shown by the Sdr values, and then IFT with composite cement than DF materials. Within the limitations of the present study, particularly slight differences in composition of experimental compared to marketed materials, results suggest that PICNs should perform better in terms of bonding properties than DF, and should be etched and silanized before resin cement application, while DF should be sandblasted and silanized, even if silanization is not as effective as with PICNs. Further research is needed to determine the exact effect silane has on the surface properties of CAD-CAM composite materials.

4 | 6. Acknowledgement

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z (mm)

0.6

Layer 1

Enamel-like

2.1

Layer 2

3.6

Layer 3

5.1

Layer 4

6.6

Layer 5

8.1

Layer 6

9.6

Layer 7

A FUNCTIONALLY GRADED
PICN MATERIAL FOR BIOMIMETIC
CAD-CAM BLOCKS

11.1

Layer 8

12.6

Layer 9

Dentin-like

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Chapter 5

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5 | Abstract

The objective of this study was to introduce a functionally graded (FG) polymer-infiltrated ceramic network (PICN) block, characterized by a gradient of mechanical properties, as a biomimetic material for computer-aided design and manufacturing (CAD-CAM) prostheses. FG-PICN blocks were manufactured from a slurry of glass-ceramic powder, which was subsequently centrifuged and sintered. The ceramic network was infiltrated with urethane dimethacrylate and polymerized under high temperature-pressure. Blocks were sectioned into 9 layers, and each layer was subsequently cut into 3 samples. Samples were loaded into a 3-point bending device and tested for flexural strength, flexural load energy, and flexural modulus. The volume percentage of glass-ceramic, hardness, and brittleness index were also measured and scanning electron microscopy (SEM) observations were performed. Katana translucent zirconia (HT-ZIR) and e.max-CAD (EMX) were tested for comparison. Flexural strength, flexural load energy, and Weibull modulus of FG-PICN were shown to increase from the first (enamel-like zone) to the ninth layer (dentin-like zone), while, on the contrary, flexural modulus, hardness, brittleness index, and ceramic volume percentage decreased. SEM characterization highlighted a higher porosity in layer 9 than in layer 1.

Flexural strength of the dentin-like zone (372.7 ± 27.8 MPa) was similar to EMX and lower than HT-ZIR. Flexural modulus was shown to vary from 41.9 ± 5.1 to 28.6 ± 2.0 GPa from surface to depth. Flexural load energy in the dentin-like zone (27.1 ± 4.9 mJ) was significantly superior to EMX and HT-ZIR. Hardness gradient was shown to be close to tooth tissues. This work introduces FG-PICN blocks, with a gradient of mechanical and optical properties through the entire thickness of the block designed to mimic dental tissues. FG-PICN demonstrated a favorable gradient of flexural strength, elastic modulus, and, most of all, flexural load energy and hardness compared to other CAD-CAM materials, which can promote the biomechanical behavior of single-unit restorations on teeth and implants.

Keywords: prosthetic dentistry/prostodontics, composite materials, bioengineering, ceramics, biomaterial(s), materials science(s).

5 | 1. Introduction

Biomimetics aims to use artificial processes to synthesize materials that are similar in structure to those that are biologically produced by natural processes. Ideally, materials used for dental prostheses should imitate the tissues to be replaced (i.e., enamel and dentin). Like many other biological structures, enamel and dentin are graded materials. The structure of human enamel is nonhomogeneous in composition and microstructure with a decrease in mechanical properties from the surface (cusp tip or incisal edge) toward the dentin-enamel junction (DEJ). The modulus of elasticity (E) and hardness of enamel at the occlusal surface were reported to be ~94 GPa and ~3.6 GPa (Xu et al. 1998), and in a more recent publication ~108 GPa and ~5.8 GPa (Elfallah et al. 2015), and decrease to ~64 GPa and ~3.5 GPa near the DEJ, respectively (Marshall et al. 2001). Those values drop substantially in the less brittle structure of dentin (~20 GPa for E and ~0.8 GPa for hardness) (Xu et al. 1998; Marshall et al. 2001).

One of the main causes of failure of materials that interface with one another is at the contact between them, particularly if the transition is sharp, as this creates stresses at the interface, which leads to failure (Mahamood et al. 2012; Madfa and Yue 2016). However, the DEJ creates a buffer zone between the enamel and dentin, delivering a gradual decrease in elastic modulus, thus avoiding the creation of subsurface cracks that may lead to fracture of the tooth under long-term masticatory forces (Marshall et al. 2001; Mahamood et al. 2012; Madfa and Yue 2016).

Unfortunately, most dental restorations offer no gradual transition within the tooth restoration assembly. In bilayered prostheses, the E of the restoration is ~70 GPa at the ceramic veneer, fused, for example, to a stiff zirconia or metal core with a higher E of ~200 GPa, then dropping to 2 to 13 GPa at the composite cement interface, which is bonded to dentin with an E of ~20 GPa (Niu et al. 2009; Rahbar and Soboyejo 2011; Madfa and Yue 2016). This mismatch in E leads to stress concentration and promotes crack initiation (Rahbar and Soboyejo 2011; Zhang et al. 2012) and fracture, which constitutes a major clinical complication of all-ceramic restorations (Conrad et al. 2007).

Nowadays, recent advances, especially in computer-aided design and manufacturing (CAD-CAM) processes, have shifted the tendency toward monolithic restorations, either ceramics or composites, with uniform mechanical properties, showing either higher values than enamel (ceramics) or lower than dentin (composites), except for

polymer-infiltrated ceramic network (PICN) CAD-CAM composites, which were shown to exhibit intermediary values (Awada and Nathanson 2015; Lawson et al. 2016; Mainjot et al. 2016; Swain et al. 2016). Monolithic ceramics include glass-ceramics and polycrystalline ceramics. IPS e.max CAD (Ivoclar Vivadent) is a popular lithium disilicate-reinforced glass-ceramic, with E and hardness values close to those of enamel (flexural strength ~340 to 380 MPa, E ~60 to 80 GPa, hardness ~4.5 to 6.0 GPa) (Stawarczyk et al. 2015; Elsaka and Elnaghy 2016; Lawson et al. 2016; Swain et al. 2016), while 3 mol% yttria-stabilized zirconia (3Y-TZP) is a polycrystalline ceramic known for its high strength (flexural strength ~900 to 1,500 MPa, E ~200 to 210 GPa), hardness (~12.9 GPa), and toughness (~3.5 to 4.5 MPa.m^{1/2}) due to the phase transformation it can exhibit, but at the same time, its optical properties limit its use as monolithic restorations (Tong et al. 2016; Zhang and Lawn 2018). Second and third generations of zirconia have progressively led to translucency improvement but at the expense of strength and toughness (dropping to ~400 to 900 MPa and ~2.2 MPa.m^{1/2}, respectively), without affecting E , while hardness was increased (~13.1 GPa) (Tong et al. 2016; Zhang and Lawn 2018). CAD-CAM composites are also used as monolithic restorations and can be divided into 2 subclasses according to their microstructure: dispersed fillers and PICNs (Mainjot et al. 2016). Dispersed fillers are composed of inorganic fillers of different sizes, amounts, and compositions according to each manufacturer; dispersed in a matrix of urethane dimethacrylate (UDMA), either with or without triethylene glycol dimethacrylate (TEGDMA); and polymerized under high temperature (HT). Their E is lower, while their hardness is similar to dentin (~8 to 16 GPa and ~0.6 to 1 GPa, respectively) (Awada and Nathanson 2015; Lawson et al. 2016). PICNs, on the other hand, are composed of a presintered glass-ceramic scaffold (86 wt.%) infiltrated with urethane dimethacrylate (UDMA), which is secondarily polymerized under high temperature and pressure (HT-HP), offering a high degree of conversion of monomers (~95%). The unique microstructure of PICNs increases the E and hardness to values ranging between those of dentin and enamel (~22 to 32 GPa and ~1.5 to 2.4 GPa, respectively) (Awada and Nathanson 2015; Lawson et al. 2016; Mainjot et al. 2016; Swain et al. 2016). Flexural strength of commercially available PICNs (Vita Enamic; Vita Zahnfabrik) was shown to be similar to dispersed filler CAD-CAM composites (~137 to 248 MPa) (Albero et al. 2015; Awada and Nathanson 2015; Stawarczyk et al. 2015; Lawson et al. 2016; Swain et al. 2016), while some experimental PICNs demonstrated higher values (305 MPa), close to that of lithium-disilicate glass-ceramic (Nguyen et al. 2014). Regarding bonding properties with resin cements, PICNs exhibited high performance when etched with 5% hydrofluoric acid (Eldafrawy et al. 2018).

On the other hand, engineers in materials science have been developing functionally graded materials (FGMs) since the 1980s. These form a class of advanced materials that

are characterized by engineered gradients in composition and structure over volume, resulting in corresponding changes in the properties of the material, which are overall unique and different from any of the individual materials that they are formed from (Mahamood et al. 2012; Madfa and Yue 2016). There is a wide range of manufacturing processes and applications for FGMs, including the centrifugal method, which leads to the creation of a gradient from powders of materials with different densities (Mahamood et al. 2012). In the dental field, some researchers have developed experimental FGMs for prosthodontics, but none of them have reproduced the properties of enamel and dentin combined (Madfa and Yue 2016). Zhang and Kim (2009) infiltrated silicate glass on the top and bottom surfaces of 3Y-TZP to produce a glass-zirconia-glass restoration with graded E to improve esthetics, reduce radial cracking at the cementation surface, and promote bond strength (Zhang and Lawn 2018). The graded material showed an increase in E from the surface toward the core from 125 to 240 GPa (Zhang et al. 2010). Dorthé and Zhang (2012) also infiltrated alumina with silicate glass in the same way to manufacture graded glass-alumina-glass, with a resulting E gradient from 110 to 419 GPa. In an attempt to mimic the gradual decrease in E from enamel to dentin via the DEJ, Huang et al. (2007) synthesized an experimental functionally graded layer (FGL) on the micro-scale to offer a smooth transition between the tooth and the restoration, in the same way as the DEJ, and to reduce stresses induced by the E mismatch, as demonstrated by finite element simulation. FGL is a composite material composed of layers with varying amounts of alumina and zirconia to control the E (from ~190 GPa to ~35 GPa) toward dentin (Huang et al. 2007; Niu et al. 2009; Rahbar and Soboyejo 2011; Du et al. 2013).

The objective of this work is to introduce a functionally graded PICN block characterized by a gradient of mechanical and optical properties throughout the entire thickness, as a biomimetic material for CAD-CAM prostheses.

5 | 2. Materials and Methods

Manufacturing of CAD-CAM Blocks

Functionally graded PICN (FG-PICN) blocks were produced by preparing a slurry by mixing 2 albite glass-ceramic powders: enamel powder (25 vol.%, 2.6 μm D50 grain size, incisal optical properties) and D2M2 (25 vol.%, 4.5 μm D50 grain size, dentin optical properties) (Vita Zahnfabrik) with deionized water (50 vol.%) for 20 min in a planetary mixer (ARE-250; Thinky). The slurry was then centrifuged at 200 g for 15 min in a 20 mm square-shaped container, dried, and sintered at 860°C for

2 h to form the glass-ceramic porous network. Silanization was done using prehydrolyzed 3-(Trimethoxysilyl) propyl methacrylate (Sigma- Aldrich); then, after drying under vacuum at 130°C for 1 h, infiltration with UDMA (Esstech) monomer with (0.5 wt.%) di-tert-amyl peroxide (Sigma-Aldrich) initiator was done under vacuum. Blocks were polymerized under HT-HP (180°C– 300 MPa) following the procedure described by Nguyen et al. (2014), resulting in FG-PICN blocks with dimensions of $(13.2 \pm 0.1) \text{ mm} \times (14.6 \pm 0.1) \text{ mm} \times (18 \pm 0.1) \text{ mm}$.

Commercial blocks ($n = 4$) of IPS e.max CAD lithium disilicate glass-ceramic (EMX) (Ivoclar Vivadent) as well as a disc of high translucent zirconia (HT-ZIR) (Katana Zirconia; Kuraray Noritake) were also used in this study for comparison.

Flexural Strength, Flexural Load Energy, and Flexural Modulus

For each block, 9 layers of $(1.2 \pm 0.1) \text{ mm}$ thickness were cut using a low-speed saw (thickness 0.3 mm) (Isomet; Buehler) under continuous water irrigation starting from the surface (enamel zone) to the bottom (dentin zone) (Fig. 1). The layers were then cut to the desired $(1.2 \pm 0.1) \text{ mm} \times (4.0 \pm 0.1) \text{ mm} \times (18 \pm 0.1) \text{ mm}$ bars ($n = 30$ per layer), then polished with a diamond pad ($10 \mu\text{m}$) on one side, at 150 rpm under water (Struers).



Fig. 1 A functionally graded polymer-infiltrated ceramic network (FG-PICN) block with, on the left part, a schematic illustration of the cutting into 9 layers from the top (enamel zone) to the bottom (dentin zone). The black spaces between the layers represent the 0.3 mm of material lost during cutting (saw thickness). The thickness of each layer is $(1.2 \pm 0.1) \text{ mm}$; measurements refer to the center of each layer (dashed line).

EMX samples ($n = 30$) were manufactured in the crystalline intermediary stage with the same dimensions and were polished with 320-, 600-, and 1,000-grit silicon carbide (SiC) paper, then with the diamond pad. Crystallization firing of EMX was performed in a dedicated furnace (Programat; Ivoclar Vivadent) at 820°C for 10 min (90°C/min), followed by 840°C for 7 min (30°C/min), according to the manufacturer's recommendations. HT-ZIR samples ($n = 30$) were prepared with larger dimensions, $(1.6 \pm 0.1) \text{ mm} \times (5.5 \pm 0.1) \text{ mm} \times (22 \pm 0.1) \text{ mm}$, to compensate for shrinkage after sintering, and then were polished carefully on the diamond pad. HT-ZIR samples were sintered following the manufacturer's recommendations by heating at 1,550°C for 2 h (10°C/min) and subsequent cooling at the same rate to room temperature (Zyrcomat furnace; Vita Zahnfabrik).

Bars were tested in a 3-point bending device (15 mm span width), with the polished surface in tension, on a computer-controlled (Bluehill; Instron) universal testing machine (Instron model 4301, with an extensometer) at a cross-head speed of 1 mm/min. Flexural strength, σ_f , was calculated according to the following formula:

$$\sigma_f = \frac{3FL}{2hc^2},$$

where F is the load at fracture, L the span, h the specimen width, and c the specimen height. The values of h and c were measured immediately prior to testing of each sample using a digital caliper (Mitutoyo).

Flexural load energy, U_r , was calculated according to the following formula:

$$U_r = \frac{F\Delta}{2},$$

where Δ is the maximum deflection.

The flexural modulus (modulus of elasticity), E , was calculated according to the following formula:

$$E = \frac{FL^3}{4hc^3d},$$

where d is the deflection corresponding to load F at a point in the straight-line portion of the trace.

Hardness

The microhardness gradient was measured using the Vickers indenter (Zwick-Rowell) with integrated software (Indentec) on 5 additional FG-PICN blocks with surfaces polished in the same manner, using a 25-N loading and 10-s dwell time. On each block, 3 indentations were made every 0.25 mm following straight lines parallel to the surface (gradient lines, $n = 15$ per gradient line group); starting from the enamel zone to the dentin zone, each indentation was separated from the next by 2 mm to avoid overlap. For HT-ZIR, a representative sample (12 ± 0.1) mm \times (4 ± 0.1) mm \times (20 ± 0.1) mm was used, in which 15 indentations were made using a 10-N load and 10-s dwell time.

Brittleness Index

Brittleness index (B) was measured on 2 additional FG-PICN blocks, as described in the Appendix.

Volume Percentage Glass-Ceramic

The volume percentage of ceramic of each layer was measured from all fractured samples by weighing the samples (Mettler Toledo) before and after firing at 900°C for 2 h until all the polymer was burned and calculating the vol.% of glass-ceramic, assuming that the densities of the glass-ceramic and UDMA are 2.42 and 1.206, respectively.

Scanning Electron Microscopy

Samples of the first and the last layers (from the volumetric percentage glass-ceramic experimentation) were gold-coated and examined by scanning electron microscopy (SEM)(S-3000N; Hitachi).

Statistical Analysis

The results were analyzed by 1-way analysis of variance (ANOVA), followed, if warranted, by Scheffé's multiple mean comparisons ($\alpha = 0.05$), using PASW Statistics 18 (SPSS, Inc.). Weibull statistical parameters were calculated for the flexural strength of each layer using the Weibull statistics option in Excel (Microsoft).

5 | 3. Results

Results for flexural strength, flexural load energy, flexural modulus, Weibull modulus, brittleness index, and volume percentage of glass-ceramic are presented in the Table, along with the associated statistical analysis. Hardness results are summarized in *Figure 2*. Flexural strength, flexural load energy, and Weibull modulus were shown to increase from the first (enamel-like zone) to the ninth layer (dentin-like zone), while, on the contrary, flexural modulus, hardness, brittleness index, and volume percentage of glass-ceramic decreased (*Figs. 2 and 3, Appendix Fig.*). SEM porous network characterization highlighted a higher porosity in layer 9 than in layer 1 (*Fig. 4*).

Table. Mechanical Properties and Weibull Modulus of the 9 Layers of the Functionally Graded Polymer-Infiltrated Ceramic Network (FG-PICN) Blocks, HT-ZIR and EMX, and the Brittleness Index and the Glass-Ceramic Vol.% of the 9 Layers of the FG-PICN Blocks.

Layer	Flexural Strength, σ_f (MPa)	Flexural Load Energy, U_r (mJ)	Elastic Modulus, E (GPa)	Weibull Modulus	Brittleness Index, B ($\mu\text{m}^{-1/2}$)	Vol.% Glass-Ceramic
1	175.8 ± 30.8 ^f	2.9 ± 1.3 ^g	41.9 ± 5.1 ^a	6.4	1.8 ± 0.4 ^a	82.4
2	198.8 ± 26.3 ^{ef}	4.2 ± 1.2 ^{fg}	40.0 ± 3.5 ^a	7.8	1.4 ± 0.1 ^b	80.2
3	210.6 ± 25.8 ^{de}	5.1 ± 1.6 ^{efg}	36.8 ± 3.8 ^b	8.9	1.2 ± 0.1 ^{bc}	78.2
4	217.2 ± 22.6 ^{de}	6.0 ± 1.7 ^{ef}	33.9 ± 2.5 ^b	10.7	1.1 ± 0.1 ^{bcd}	77.0
5	231.7 ± 26.5 ^d	7.3 ± 1.7 ^{de}	30.9 ± 2.1 ^c	9.4	1.0 ± 0.1 ^{cd}	74.7
6	263.3 ± 20.8 ^c	10.1 ± 2.4 ^d	29.8 ± 1.6 ^{cd}	13.2	0.8 ± 0.1 ^{de}	73.5
7	301.0 ± 25.2 ^b	14.0 ± 2.6 ^c	28.8 ± 1.2 ^{cd}	13.1	0.6 ± 0.3 ^{ef}	72.6
8	327.9 ± 34.0 ^b	20.5 ± 4.9 ^b	27.2 ± 2.1 ^d	10.2	0.4 ± 0.4 ^{fg}	72.4
9	372.7 ± 27.8 ^a	27.1 ± 4.9 ^a	28.6 ± 2.0 ^{cd}	14.6	0.1 ± 0.3 ^g	72.4
EMX	369.0 ± 55.6	6.5 ± 2.0	92.5 ± 14.0	7.1		
HT-ZIR	673.77 ± 170.0	12.0 ± 4.9	214.8 ± 19.8	4.1		

EMX, IPS e.max CAD; HT-ZIR, translucent zirconia.

The results are expressed as the means ± SD. Materials with similar letters in each column were not statistically different (1-way analysis of variance followed by Scheffé test, $\alpha = 0.05$).

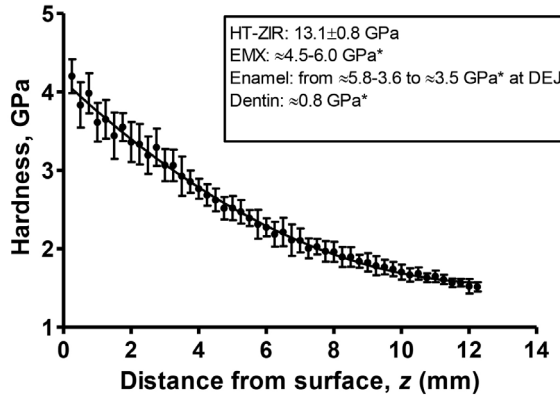


Fig. 2 Gradient of Vicker's microhardness in GPa across the functionally graded polymer-infiltrated ceramic network (FG-PICN) blocks. Indentations were done every 0.25 mm following straight lines parallel to the surface, starting from the enamel zone to the dentin zone. For each gradient line, 15 indentations were taken. The results are expressed as the means \pm SD of 15 indentations per gradient line. Hardness value of translucent zirconia (HT-ZIR) is noted separately on the upper right corner of the graph ($n = 15$). Average values of microhardness of IPS e.max CAD (EMX), enamel, and dentin are also presented for comparison. *Data from Xu et al. (1998), Marshall et al. (2001), Elfallah et al. (2015), Elsaka and Elnaghy (2016), Lawson et al. (2016), Swain et al. (2016), and Madfa and Yue (2016).

5 | 4. Discussion

Composite materials exhibit several advantages in regard to producing CAD-CAM monolithic restorations. They show better machinability than ceramics, characterized by a faster milling process, less edge chipping, and the possibility to be milled to a very low thickness to produce minimally invasive restorations (Coldea et al. 2015; Lebon et al. 2015; Chavali et al. 2017). They are particularly adapted to chair-side systems since they do not require any firing procedures, as is the case with many ceramic materials. They are also easy to adjust and repair intraorally, and their optical properties allow for the manufacturing of restorations with various degrees of translucency. Among CAD-CAM composites, PICNs, due to their specific microstructure and manufacturing process, also showed bonding properties better than lithium-disilicate glass-ceramics (Eldafrawy et al. 2018) and good biocompatibility properties toward human gingival fibroblasts and keratinocytes, with the absence of monomer release (Phan et al. 2014; Grenade et al. 2016; Grenade et al. 2017).

The larger elastic modulus of PICNs with a glass-ceramic network infiltrated by polymer, compared to dispersed filler composites, can be explained by its underlying microstructure (Awada and Nathanson 2015; Lawson et al. 2016; Mainjot et al. 2016; Swain

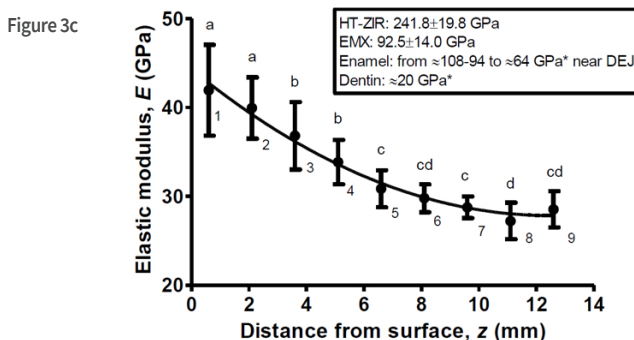
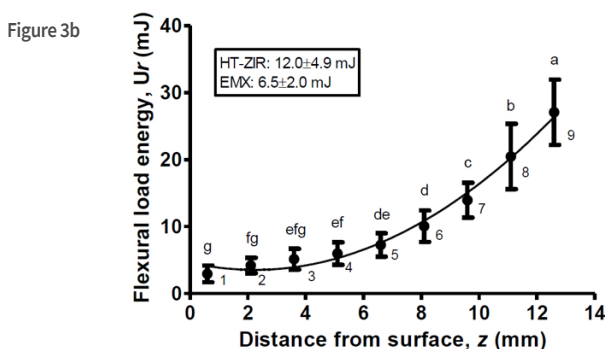
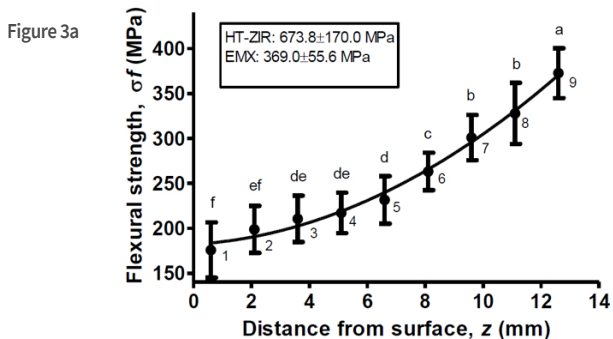


Fig. 3 Flexural properties.

- Flexural strength (σ_f) in MPa of the 9 layers of the functionally graded polymer-infiltrated ceramic network (FG-PICN) blocks ($n=30$ per layer). Values of translucent zirconia (HT-ZIR) and IPS e.max CAD (EMX) are noted separately on the upper left corner of the graph ($n=30$ per material).
- Flexural load energy (U_r) in mJ of the 9 layers of the FG-PICN blocks ($n=30$ per layer). Values of HT-ZIR and EMX are noted separately on the upper left corner of the graph ($n=30$ per material).
- Modulus of elasticity (E) in GPa of the 9 layers of the FG-PICN blocks ($n=30$ per layer). Values of HT-ZIR and EMX are noted separately on the upper right corner of the graph ($n=30$ per material). Average values of E of enamel and dentin are also presented for comparison. *Data from Xu et al. (1998), Marshall et al. (2001), Elfallah et al. (2015), and Madfa and Yue (2016). The numbers (1 to 9) underneath the data points represent the corresponding layer within the FG-PICN blocks. The results are expressed as the means \pm SD. Superscript letters indicate statistically homogeneous subgroups within a material category (1-way analysis of variance followed by Scheffé test, $\alpha = 0.05$). The same superscript letters demonstrate that there were no significant differences for each factor.

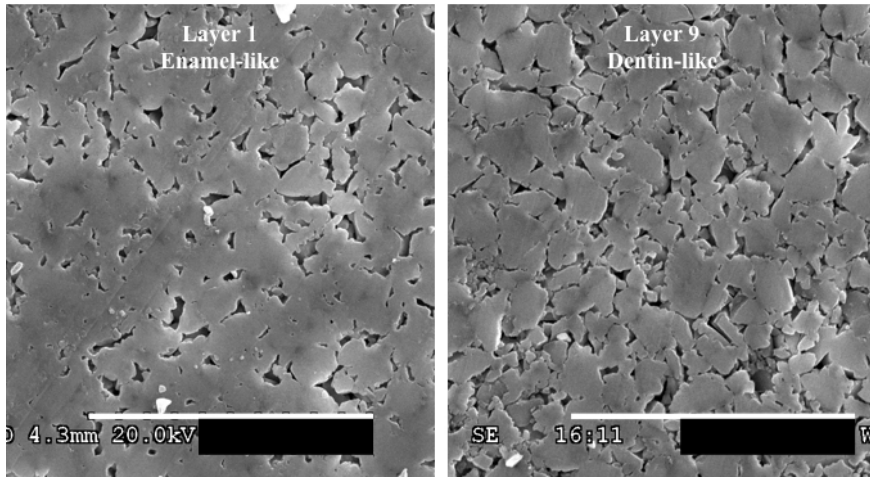


Fig. 4 Scanning electron microscopy images of the first (enamel-like) layer (to the left) and the last (dentin-like) layer (to the right) at $\times 2,000$ magnification.

et al. 2016). The wet manufacturing process of the glass-ceramic network used in experimental PICNs, associated with the HT-HP polymerization process of polymer, was previously shown to increase material flexural strength up to ~ 305 MPa (Nguyen et al. 2014). In the present work, the addition of a centrifugation processing of the glass-ceramic powder before the sintering procedure allowed the obtaining, after sintering, of a gradient of glass-ceramic powder density, as demonstrated by the decrease of glass-ceramic volume percentage with depth (around 10 vol.%). The centrifugation of 2 different glass-ceramic powders with respective grain sizes (enamel powder, $2.6 \mu\text{m}$ D50 grain size, incisal optical properties, and D2M2, $4.5 \mu\text{m}$ D50 grain size, dentin optical properties) allowed for the creation of a gradient in the distribution of the different grains: large grains will concentrate in the external part of the centrifuged block and the smaller ones inside. Due to their respective grain size, the partial sintering of the enamel powder will give rise to higher shrinkage and a reduced ratio of open porosities for monomer infiltration than dentin powder, as shown in the SEM images. Consequently, the resulting FG-PICN is characterized by a gradient in microstructure over the thickness of the sample, resulting in progressive and significant variations in the material optical properties, on one hand, and in the mechanical properties, on the other hand, as demonstrated by flexural strength, E , flexural load energy, and hardness measurements.

Regarding flexural strength, the results cannot be compared to enamel and dentin values due to the absence of data in the literature related to the difficulty in measurement (Coldea et al. 2013). Compared to other CAD-CAM materials, the flexural strength of FG-PICN in the dentin-like zone (372.7 ± 27.8 MPa) was shown to be significantly

higher than commercial PICN or other CAD-CAM composites (~137 to 248 MPa) (Albero et al. 2015; Awada and Nathanson 2015; Stawarczyk et al. 2015; Lawson et al. 2016; Swain et al. 2016) and similar to lithium-disilicate glass-ceramic, while HT-ZIR showed much higher flexural strength (673.8 ± 170.0 MPa). The dentin-like zone was shown to be more resistant than the enamel-like zone, with the flexural strength increasing from 175.8 ± 30.8 to 372.7 ± 27.8 MPa with increasing polymer fraction (from 17.6 to 27.6 vol.%). In fact, the polymer fraction promotes material viscoelastic behavior, which hinders crack initiation and propagation, similar to dentin (while enamel is brittle, with only 10 vol.% polymer fraction compared to 30 vol.% for dentin) (Berkovitz et al. 2009), as confirmed by the Weibull modulus increase from layer 1 to 9. The brittleness of ceramic materials is an inconvenience compared to composites due to edge chipping during the manufacturing process (Chavali et al. 2017).

The FG-PICN elastic modulus was shown to be, within the surface, around 20 GPa lower than deeper layers of natural enamel and similar to dentin in depth, while EMX is similar to the in-surface layers of natural enamel and HT-ZIR is much too rigid. Actually, glass-ceramics are similar to enamel in terms of stiffness: the more glass-ceramic phase in the FG-PICN, the closer to enamel the E value is, while experimental graded zirconia exhibited a value close to enamel only on the material surface, which was infiltrated with glass (~75 GPa) (Zhang and Ma 2009). In fact, FG-PICN mimics the gradient found in dentin and deeper layers of enamel in terms of stiffness variations. However, it must be noticed that the reported data of the E of enamel and dentin (Xu et al. 1998; Marshall et al. 2001; Elfallah et al. 2015), as well as graded zirconia (Zhang and Ma 2009; Zhang et al. 2010; Dorthe and Zhang 2012), were obtained via indentation, while those of EMX were obtained via flexion (Awada and Nathanson 2015; Elsaka and Elnaghy 2016; Lawson et al. 2016), as in the present study, which could result in some differences. The gradual transition in E , in a similar manner to tooth tissues, suggests that fatigue stresses and restoration failure can be decreased (Rahbar and Soboyejo 2011; Du et al. 2013). Moreover, compared to metal or ceramics, restorative materials with low E , such as composites, are reputed to better distribute stresses around implants (Maminskas et al. 2016) and to show a damping effect (Magne et al. 2013), which can promote implant osseointegration and survival (Lambert and Mainjot 2017).

The most interesting finding are the results of the flexural load energy, which were shown to reach 27.1 ± 4.9 mJ in the dentin-like zone, while EMX and HT-ZIR exhibited only 6.5 ± 2.0 mJ and 12.0 ± 4.9 mJ, respectively. Flexural load energy is the amount of stresses a material can absorb before fracture. This capacity also improves the material's damping effect, which is particularly important in the case of high stress, such as in the presence of bruxism, or with implants. The enamel-like zone showed significantly

higher brittleness compared to the dentin-like zone, as observed with tooth tissues. Hardness showed a biomimetic tendency with values nearly matching those of enamel and dentin. This property is particularly important not only to the wearing processes of the material itself but to antagonistic teeth, as well. Actually, materials should wear at the same rate as tooth tissues: not more, not less, to allow for favorable evolution of occlusion. From that point of view, ceramic materials are not well adapted, with zirconia being not susceptible to self-wear and glass-ceramics causing excessive wear of the opposing teeth (Swain et al. 2016). If commercial PICN was shown to be submitted to more self-wearing than enamel, due to their higher in-surface hardness, it could be expected that the behavior of FG-PICN would be more favorable (hardness ~1.5 to 2.4 GPa for Vita Enamic compared to 4.2 for enamel-like zone of FG-PICN) (Lawson et al. 2016; Swain et al. 2016; Xu et al. 2017). In addition, increasing hardness decreases the machinability of CAD-CAM blocks, with composite materials being more advantageous than zirconia from that point of view.

Finally, the manufacturing process allowed for the creation of a shade gradient through the block. The presence of a higher volume percentage of glass-ceramic in the surface is susceptible to improving material polishing and gloss compared to commercial PICN.

The limitation of FG-PICN is that the gradient is 1-dimensional, while it is 3-dimensional in natural teeth, which should not cause a problem in manufacturing inlays, onlays, tabletops, and palatal and buccal veneers, but is not ideal for crowns.

5 | 5. Conclusion

This work introduces, for the first time, a functionally graded biomimetic material for CAD-CAM prostheses, with a gradient of mechanical and optical properties throughout the entire thickness of the block. The FG-PICN material demonstrated a favorable gradient of 1) flexural strength similar to lithium-disilicate glass-ceramic (EMX), associated with a high Weibull modulus; 2) elastic modulus close to tooth tissues; 3) flexural load energy superior to EMX and translucent zirconia; and 4) hardness similar to enamel and dentin. The graded character of this material can improve the biomechanical behavior of single-unit restorations on teeth and implants. Due to its innovative microstructure, FG-PICN combines the advantages of both CAD-CAM composite and glass-ceramic materials, without their limits or those of zirconia, in terms of mechanical properties, but also in terms of machinability, absence of firing procedure, and optical and bonding properties.

Future perspectives include the study of the fatigue behavior of this innovative material and clinical studies.

5 | 6. Author contributions

M. Eldafrawy, contributed to data acquisition, analysis, and interpretation, drafted the manuscript; J.F. Nguyen, contributed to data analysis and interpretation, critically revised the manuscript; A.K. Mainjot, contributed to data analysis and interpretation, drafted the manuscript; M.J. Sadoun, contributed to conception, design, data acquisition, analysis, and interpretation, critically revised the manuscript. All authors gave final approval and agree to be accountable for all aspects of the work.

5 | 7. Acknowledgements

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The authors declare no other potential conflicts of interest with respect to the authorship and/or publication of this article.

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Brittleness index

Brittleness index (B) of the 9 layers was measured on 2 additional FG-PICN blocks using a Vickers indenter (Zwick-Roell) with integrated software (Indentec), with the surfaces polished with a diamond pad (10 μm), at 150 rpm under water (Struers). On each block, 5 indentations were made for each of the 9 layers, following straight lines parallel to the surface, with a load of 98 N for the first four layers (enamel-like) and 196 N for layers 5 to 9 (dentin-like), to induce cracks. Brittleness index was calculated following the formula given by Sehgal and Ito (1998):

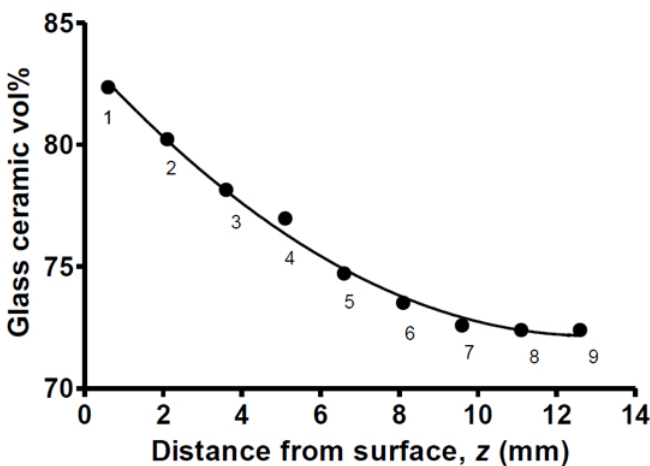
$$B = \gamma P^{-1/4} \left[\frac{C}{a} \right]^{3/2}$$

where P is the indentation load (N) for median cracking, γ is constant and equals to $2.39 \text{ N}^{1/4}/\mu\text{m}^{1/2}$, C is the characteristic crack length and a is the Vickers indentation diagonal length.

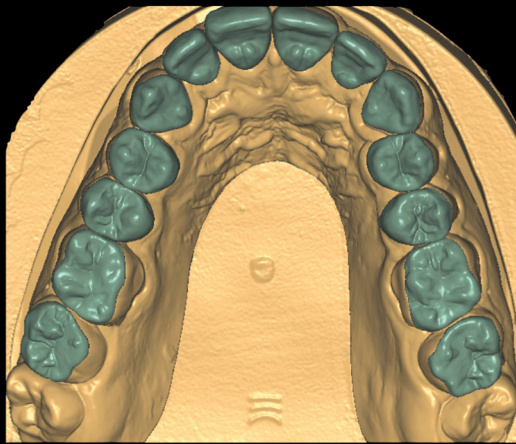
The values of brittleness index decreased significantly from the first layer ($1.8 \pm 0.4 \mu\text{m}^{-1/2}$) to the ninth layer ($0.1 \pm 0.3 \mu\text{m}^{-1/2}$) as shown in *table 1*.

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Appendix Figure: Glass-ceramic volume percentage (vol.%) of the nine layers of the functionally graded PICN blocks (FG-PICN). The numbers (1-9) underneath the data points represent the corresponding layer within the FG-PICN blocks.



THE ONE-STEP NO-PREP APPROACH
FOR FULL-MOUTH REHABILITATION
OF WORN DENTITION USING PICN
CAD-CAM RESTORATIONS:
2-YR RESULTS OF A PROSPECTIVE
CLINICAL STUDY

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Chapter 6

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6 | Abstract

Objectives: To prospectively evaluate the One-step No-prep treatment of full mouth-worn dentition, a minimally invasive and multidisciplinary approach using PICN CAD-CAM composite restorations without provisional phase.

Methods: Seven patients (n=192 restorations) with severe tooth wear were included. Patient data were recorded, and an occlusal analysis and a tissue-guided wax-up were realized. After replacement of old fillings, no-prep Vita Enamic restorations (posterior restorations and palatal veneers) were bonded within 24h. Direct composites were performed to mask the buccal joint on anterior teeth. Maxillo-facial physiotherapy was performed. Restorations were evaluated following World Dental Federation criteria. Treatment influence on Oral-Health-Impact-Profile-49 (OHIP-49) score was assessed.

Results: Tooth wear etiology was related to soft drink consumption and bruxism. Mean VDO increase was 5.09 ± 0.85 mm on the incisal pin. The mean restoration thickness on molars was 0.55 ± 0.21 mm, and the lowest was 0.11mm. 2-year survival rate of restorations was 100 % and success rate was 93.5%, with 11 minor chippings and one debonding. A significant improvement of the global OHIP-49 score was observed.

Conclusions: In this clinical study on high risk patients, PICN restorations, applied in a minimally invasive way, showed high survival and success rates after two years, while minor chipping of very thin occlusal borders constituted the most frequent complication. Moreover, the patient acceptance was good according to OHIP-49 in this multidisciplinary approach.

Clinical significance: The use of PICNs allows the development of no prep and simple treatment protocols of worn dentition. The absence of provisionals did not engender any problem, on the basis of the realization of an occlusal analysis, the support of a maxillo-facial physiotherapist, and the use of an easy-to-adjust restorative material.

Keywords: tooth wear; minimally invasive treatment; CAD-CAM composite; fixed prosthodontics; dental materials..

6 | 1. Introduction

In recent years, a significant increase in the prevalence of tooth wear has been observed, especially in young patients [1–3]. These problems are promoted by changing lifestyles, particularly chemical erosion favored by acidic food/drink and gastro-esophageal reflux, while mechanical wear is often related to the presence of bruxism, which engenders attrition and abfraction [4,5]. Severe dental tissue wear can engender dental pain due to exposure of dentinal tubules [6,7] and a decrease in the masticatory function. Moreover, severe tooth wear leads to an unsightly appearance in most patients, who report a real social handicap [8,9]. In addition to preventive approaches in the absence of symptoms and patient demand [10], current recommendations emphasize the need to develop minimally invasive treatment strategies that preserve as much as possible remaining tooth tissues [9], with the current tendency being to avoid any tooth tissue preparation (“No prep”). However, restoring functional occlusal relationships and aesthetics requires a full-mouth rehabilitation with an increase in the vertical dimension of occlusion (VDO), constituting a complex treatment. Various techniques have been described: direct techniques with light-cured composites, which constitute the most commonly reported treatment [8], indirect techniques; or a mix of the two techniques. The direct technique can involve the estimation of the new VDO in the articulator before light-cured composites placement using silicone bite-stops in the posterior zone (DSO-technique) [11,12], or the realization of a global wax-up with an arbitrarily determined increase of the VDO, followed by the placement of light-cured composites using a transparent silicon key [13,14]. Additionally, some authors have performed the wax-up on the basis of an analysis of occlusal relationships (e.g., using facial bow and a jig) [13], and others have used provisional restorations to test the new VDO before final direct composite realization [15]. The direct technique is particularly minimally invasive and reversible, restorations being easy to repair, while the treatment cost is reported to be reduced [16,17]. However, the technique can result in the need for maintenance care (for example polishing and repair), especially in the presence of bruxism [9] since the prognosis of restorations is patient dependent [18]. Conversely, with indirect, minimally invasive strategies, composite palatal veneers, eventually associated with ceramic buccal veneers, are usually performed to restore the upper incisors and canines (“sandwich technique”), while ceramic or composite occlusal onlays are realized on the premolars and molars [19–21]. These treatments gave multiple steps and require several appointments, involving the realization of mock ups and provisional restorations, which are intended to test the new VDO, to validate the aesthetic results and to guide tooth tissue preparation for indirect restoration [22]. If indirect techniques allow for the use of materials that are more performant than light-cured composites, the restorations are more expensive [18]. However, there is no evidence regarding the best technique (direct or indirect) or material (composite or ceramic) to be used to restore severely

6

worn dentition [8,9], and to the authors' knowledge, there have been no clinical studies examining the performance of indirect restorations used for full-mouth rehabilitation of worn dentition. Recently, a novel technique (One-step No-prep) for full-mouth rehabilitation of worn dentition using CAD-CAM composite restorations was introduced [23]. It is characterized by the absence of tooth tissue preparation and a provisional phase, the use of Polymer-Infiltrated Ceramic Network (PICN) materials, the realization of an occlusal analysis and the collaboration with physiotherapists to treat associated symptoms, such as muscular hypertrophy and pain.

In this "one step" treatment, definitive indirect restorations are bonded within two consecutive days, reducing visit numbers in comparison with other indirect techniques and treatment complexity (particularly regarding very thin provisional restorations management). PICN materials (hybrid ceramics, Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany) were shown to exhibit several advantages compared to ceramics for this indication, such as the ability to be milled to a very low thickness and ease of in-mouth adjustments [24]. Furthermore, they have also shown good mechanical [25] and bonding properties [26]. The One-step No-prep approach was shown to yield successful short-term clinical results in three pilot cases and to be particularly straightforward and minimally invasive. The absence of provisional restorations to test the VDO increase did not engender any inconvenience and the patients' perceptions were very positive. However, those preliminary results must be confirmed with an in-depth analysis of the different facets of this protocol. Moreover, there is a lack of data about the clinical behavior of PICN partial coverage restorations [27–29].

Consequently, the objective of the present study was to prospectively evaluate the "One Step - No Prep" protocol for full-mouth rehabilitation of worn dentition, studying the clinical behavior of PICN restorations and treatment influence on patients' Oral-Health-Related-Quality of Life (OHRQoL) using the Oral-Health-Impact-Profile-49 questionnaire (OHIP-49).

6 | 2. Materials and methods

Study Design

This study was a prospective clinical study evaluating patients treated with the «One-step No-prep» protocol. The patients were treated in the Department of Fixed Prosthodontics of the University Hospital of Liège by four experimented practitioners. They were informed about the objectives of the study, and their consent was obtained before treatment. The study was approved by the Ethics Committee of the University Hospital of Liège (B707201526682).

Patient record registrations

Specific data were collected regarding the following parameters: sex, age, and occlusal relationships were registered (dental class, function, overjet, overbite, crossbite).

Patient selection

Seven patients presenting generalized severe tooth wear with an aesthetic or functional demand were included in the study. The patients were required to have a minimum of 8 teeth, palatine veneers of the canine to canine superior teeth and a minimum of 3 teeth per posterior sextant to restore with an indirect restoration (*Figs. 1a, 2a, b*). The following patients were excluded from the study: smokers and patients with poor oral hygiene, those with periodontal disease or severe osteoarthritis, and patients with crowns, bridges, or implants. Patients with Parkinson disease or spontaneous temporomandibular joint pain associated with a mandibular deflection and an opening limitation (<25 mm) were also excluded.

Wear quantification

To quantify dental wear, the Basic Erosive Wear Examination index (BEWE) [30] was calculated for each patient by the same practitioner.

Chemical erosion assessment

In addition to a thorough clinical examination to detect the presence of dental erosion surfaces (concave, cuneiform or flat lesions), an accurate medical history was recorded including questions about nutrition habits, general diseases, medications, and environmental factors.



Fig. 1a



Fig. 1b

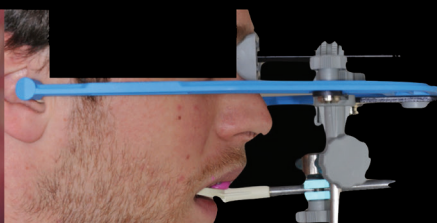


Fig. 1c



Fig. 1d

- Fig. 1** Case 1 (Photo courtesy of A. Mainjot)
- Occlusal views before treatment.
 - Occlusal relationships registered using a resin jig and a double layer of wax (Moyco Beauty Wax).
 - Facebow (Quick facebow).
 - Plaster models placed in the articulator.
 - "Tissue-guided" full-mouth wax-up.
 - Superimposition of the scans of the models and the wax-up with the Ceramill system.
 - Wax CAD-CAM mock-up.
 - Occlusal views of final PICN restorations on plaster models after polishing (in this case, restorations were not stained).
 - A 0.11 mm-thick PICN (Vita Enamic) posterior occlusal table top immediately after milling.

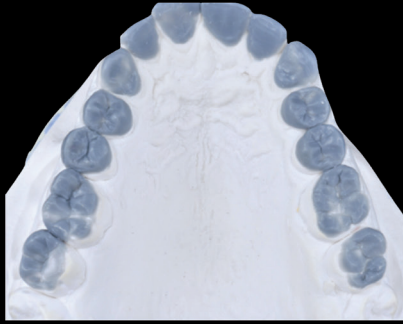


Fig. 1e

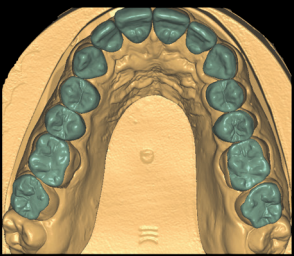


Fig. 1f

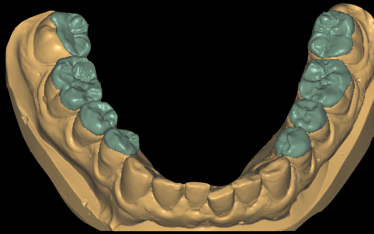


Fig. 1g



Fig. 1h



Fig. 1i

Non instrumental approach of bruxism assessment

A clinical examination was performed to register the presence of clinical signs of bruxism, such as dental attrition, cracks/fractures, masseteric hypertrophy, linea alba, exostoses or crenated tongue [31,32]. The presence of bruxism was recorded if the patient fulfilled at least two criteria: A) reporting of tooth grinding during the night or day; or B) the presence of at least one clinical sign among the following: abnormal attrition wear facets on the teeth; transitory pain or fatigue on waking felt in the jaw muscles; temporal headaches on waking; and jaw locking on waking related to teeth grinding during sleep [31,33]. A complementary clinical examination was performed by an occlusodontist (i.e. a specialist in occlusion and TMDs) to detect the presence of temporomandibular joint (TMJ) disorder. If patients had symptoms of TMJ disorder, they were sent to a maxillo-facial physiotherapist before treatment. Finally, the wearing of an occlusal nightguard before treatment was recorded.

Clinical protocol

The patients were treated according to the previously described “One-step No-prep” protocol [23]. Before including patients in the study, a complete dental check-up with carious and periodontal examinations, X-rays and photographs was performed. Double mix impressions with polyvinyl siloxane (PVS) material (Imprint 4 Heavy and XLV, 3M ESPE, Seefeld, Germany) were realized, and study models were cast (GC Fujirock EP Super Hard Plaster, GC Europe, Leuven, Belgium). Then, an occlusal analysis was performed using a resin jig [34] (Fig. 1b) and a facebow (Fig. 1c) (Quick facebow, Sintec Inc, New Hampshire, USA). The jig was placed for a few minutes to induce muscular relaxation and lower jaw repositioning, and then occlusal relationships were registered with wax (Moyco Beauty Wax, Philadelphia, PA, USA) in double thickness (Fig. 1d). The dental technician started to deposit the wax on the less damaged teeth and was guided by the residual tissues to restore tooth anatomy, resulting in very low wax thickness on some posterior teeth (Fig. 1e). With this “tissue-guided” approach, the estimation of the new VDO was empirical. The full wax-up was shown to the patient for approval. Subsequently, the treatment started with the replacement of amalgam fillings and efficient composite restorations with direct composite restorations (Els composite extra low shrinkage, Saremco Dental, Rebstein, Switzerland). Before impressions, dental tissues were not prepared, but sharp angles were softened and large direct composite fillings, presenting large proximal boxes, were partly removed to be replaced by the indirect restorative material, with cavities filled with provisional composite resin (Telio CS Onlay, Ivoclar Vivadent, Schaan, Lichtenstein). If required, endodontic treatments were performed previously. New double mix impressions and occlusal analysis following the

same protocol as previously were performed after a visit to the physiotherapist, which was intended to equilibrate the posture and muscular chains before final occlusal analysis. It should be emphasized that the patients did not wear any occlusal splints to test the new VDO before treatment. A resin posterior key was realized to validate the occlusal relationships in the articulator. The models and the full wax-ups were scanned and superimposed using a CAD-CAM system (Ceramill system, Amann Girrbach AG, Koblach, Austria) (Fig. 1f). A CAD-CAM mock-up was performed in wax and tried to validate the restoration design and aesthetic result (Fig. 1g). Restorations corresponding to the estimated tissue loss (palatal veneer, posterior occlusal tabletops and veneerlays) were milled from PICN blocks (Vita Enamic HT, Vita Zahnfabrik, Germany; Ceramill Motion M, Saint-Paul, USA) and a silane (Silane Primer, Kerr, Orange, California, United States) was applied. Then a two-step self-etch adhesive (Optibond XTR, Kerr, Orange, California, United States) was applied following manufacturer recommendations and the adhesive layer was polymerized before restoration bonding. The restorations were bonded with a composite resin cement with the Nexus XTR system (NX3, Kerr, Orange, California, United States), polymerization was performed after excess removal, and final photopolymerization was performed under a film of glycerin to avoid the persistence of a polymerization inhibition layer. Major occlusal adjustments were realized immediately after bonding of the lower restorations with an Arkansas stone burr, followed by polishing with silicon gums and fine adjustments performed within the subsequent weeks and after a visit to a maxillofacial physiotherapist. A bleaching procedure (home bleaching with a night guard using Illumine 10 % tooth gel Kit, Dentsply Sirona, New York, USA) was also performed (which was not possible when the dentin was still exposed). To mask the junction between the palatal veneer and the buccal face of the upper anterior teeth, direct composite (Inspiro, Edelweiss, Zug, Switzerland) was added on a slight chamfer performed across the junction and where needed to optimize tooth shape (Figs. 1j–m, 2c, d). Finally, an acrylic nightguard (for the upper maxilla) (Orthocryl, Dentaaurum, Ispringen, Germany) was provided to all of the patients.

Prosthetic parameter analysis

For each restoration, the nature of the antagonist (enamel, dentin, direct composites, PICN restoration) was registered. The lowest thickness of each restoration was measured, and the VDO increase at the incisal guide pin was registered.

Clinical evaluation of restorations

Two independent and calibrated evaluators assessed restorations following the criteria of the World Dental Federation (FDI) after one month, six months, 1 year and 2 years [35].

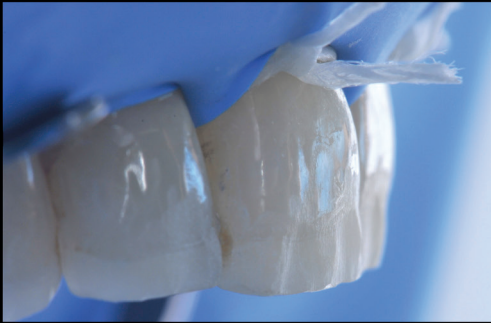


Fig. 1j



Fig. 1k

- Fig. 1 j) Direct composite was added on a slight chamfer performed across the junction between the tooth and the palatal veneer to mask the joint between the palatal veneer and the buccal face of the upper anterior teeth. In this case, the chamfer comprised two buccal grooves to fill with incisal direct composite to promote the aesthetic result. Direct composite was also added where needed to optimize tooth shape. This option is a minimally invasive alternative to ceramic buccal veneer, which can be performed a second time if the patient is not satisfied with the result or if the direct restoration is aging too quickly.
- k) Buccal views of upper anterior teeth before treatment, after palatal veneer bonding and after buccal joint masking with direct composite, respectively. Palatal veneers should be performed a bit longer than needed regarding the position of the incisal edge to be able to perfectly adapt this edge to the horizontal plane after bonding. Direct composite should not be present on the incisal edge for mechanical resistance issues.
- l) Smile pictures before and after treatment.
- m) Occlusal views after treatment.



Fig. 1l



Fig. 1m



Three dimensions, representing 18 items, were described: aesthetic, functional and biological. The functional and aesthetic dimension includes patient-reported satisfaction. Each item is assessed on a 5-point Likert scale (1 corresponding to an excellent restoration and 5 corresponding to a restoration that must be replaced). In case of discrepancies, agreement was found between evaluators to determine the final score.

Patient reported outcome measures (PROMs)

The Oral-Health-Impact-Profile-49 [36] questionnaire (OHIP-49, OHRQoL for Oral-Health-Related-Quality of Life) was filled out before treatment and at each evaluation time. The OHIP questionnaire includes 49 statements divided into seven areas, namely functional limitations, physical pain, psychological discomfort, physical disability, psychological disability, social disability and disability. For each question, a score between 1 and 5 is given, from never to very often. A high global score identifies poor quality of life related to oral health. In the present study, it was decided to present the global OHIP score [37] and the overall score for each of the seven areas, respectively.

Statistical analysis

Data analysis was carried out using GraphPad Prism (GraphPad Software, San Diego, CA). The data collected were expressed in terms of the prevalence or the mean \pm standard deviation (Mean \pm SD). The prevalence was defined as the proportion (%) of people with a given condition relative to the total population studied. The annual failure rate (AFR) of restorations was calculated on the basis of survival and success, respectively [45]. An analysis of the normal distribution of data was systematically performed with the Shapiro-Wilk test. In order to compare the mean scores of OHIP-49 variables, not normally distributed, before and after the intervention (1 month and 2 years), the Mann-Whitney *U* test and the Wilcoxon signed-rank test were used. In all tests, variations were considered statistically significant when p -value < 0.05 .

6 | 3. Results

Clinical data about the patients

Seven patients (n=192 PICN restorations) were included in this study. Among them, 6 were men. The mean age was 37.7 ± 12.8 years old. Six patients were in class I, and one patient was in class II.2. Of the patients 57.2 % (n=2) showed a group function, 14.3 % (n=1) had a canine function, and 28.5 % (n=2) had both types of function. Regarding the Basic Erosive Wear Examination (BEWE), 6 patients had a high risk level (BEWE score >14), and one patient had a medium risk level (BEWE score=13). Regarding the etiology of wear, all of the patients showed both chemical (erosion) and mechanical wear (bruxism) signs.

Most of the patients (71.4 %, n=5) reported drinking 1–2 liters of soda every day, and 28.6 % (n=2) had gastro-esophageal reflux. All of the patient reported grinding or clenching habits during the night or day, with a minimum score of 4 out of 10. Finally, 85.7 % (n=6) of patients described being stressed and anxious in their daily lives.

Clinical data about PICN restorations

In total, 192 PICN restorations were evaluated at 2 years. Six patients received PICN restorations on all of their teeth, and one patient received direct restorations for the

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Fig. 2 (Photo courtesy of A. Mainjot)
Case 2, severe tooth wear in a patient suffering from anorexia and bruxism.
a) Occlusal views before treatment.
b) Frontal view before treatment
c) Frontal view after treatment. The patient was sent to a specialist to consider soft tissue grafts for gingival recessions.
d) Occlusal views after treatment.

Table 1 2-year FDI rating of restorations. In bold, failures considered for the success rate calculation.

	Clinically excellent % (n)	Clinically good % (n)	Clinically sufficient % (n)	Clinically unsatisfactory % (n)	Acceptable %	Unacceptable %
A. Esthetic properties					100 (192)	
Surface luster	100 (192)					
Staining						
a. surface	100 (192)					
b. margin	90.6 (174)	9.4 (18)				
Color match and translucency	16.7 (32)	67.7 (130)	15.6 (30)			
Esthetic anatomical form	97.4 (187)	1.6 (3)	1.0 (2)			
B. Functional properties					100 (192)	
Fracture of material/retention	94.3 (181)	3.6 (7)	2.1 (4)			
Marginal adaptation		100 (192)				
Occlusal contour & wear						
a. Qualitatively	100 (192)					
b. Quantitatively	100 (192)					
Approximal anatomical form						
a. contact point	93.2 (179)	1 (2)	5.8 (11)			
b. contour	99.5 (191)	0.5 (1)				
Radiographic examination	100 (192)					
Patient's view	100 (192)					
C. Biological properties					100 (192)	
Postoperative sensitivity / tooth vitality	96.9 (186)	3.1 (6)				
Recurrence of caries, erosion, abfraction	100 (192)					
Tooth integrity	100 (192)					
Periodontal response	94.3 (181)	5.7 (11)				
Adjacent mucosa	100 (192)					
Oral and general health	100 (192)					

lower incisors and canines (Inspiro, Edelweiss). Consequently, 96.36 % (n=185) of restorations were in contact with PICN material and 3.64 % (n=7) with direct composite. The mean thickness registered at the thinnest point of restorations was 1.2±0.4 mm for upper incisors (n=28), 1.1±0.5 mm for lower incisors (n=24), 1.0±0.5 mm for canines (n=26), 0.7±0.3 mm for premolars (n=56) and 0.5±0.2 mm for molars (n=58). The lowest thickness measured was 0,11 mm on a molar. The mean VDO increase as measured at the incisal pin was 5.09±0.85 mm, and the greatest VDO increase registered was 6.00 mm (n=2).

Clinical evaluation of restorations

After periods of 1 and 2 years, the survival rates of PICN restorations (n=192) were 100 % and 100 %, respectively, and the success rates were 100 % and 93.75 %. The annual failure rate (AFR) on the basis of survival and success was 0 % and 3.1 %, respectively. FDI rating of restorations at 2 years is presented in *Table 1*. One hundred percent of restorations were scored as clinically acceptable for all properties at 2 years. The results showed 11 cases of minor chipping and one case of debonding (the weak link was the interface between the resin cement and the sclerotic dentin) (*Fig. 3*). Restorations presenting minor chipping were polished or repaired following this protocol: rubber dam placement, sandblasting with Cojet, silane application (Silane primer, Kerr), adhesive (Optibond XTR, Kerr) and direct composite placement (Els composite extra low shrinkage, Saremco Dental, Rebstein, Switzerland). The debonded restoration was rebonded following this procedure: elimination of the sclerotic dentin layer and of the surface layer of the composite resin cement in the restoration intaglio with a diamond burr, sandblasting of the resin cement and silane application, application of a three-step etch-and-rinse adhesive on tooth tissues following manufacturer recommendations (Optidbonf FL, Kerr) and bonding of the restoration with Inspiro direct composite (adaptation was not perfect anymore and direct composite was used to increase the mechanical resistance and aesthetics of the marginal joint). Those failures affected the success but not the survival rate of the restorations.

Patient reported outcome measures (PROMs)

The OHIP-49 results before treatment, at 1 month and 2 years, are presented in *Table 2*.



Fig. 3a

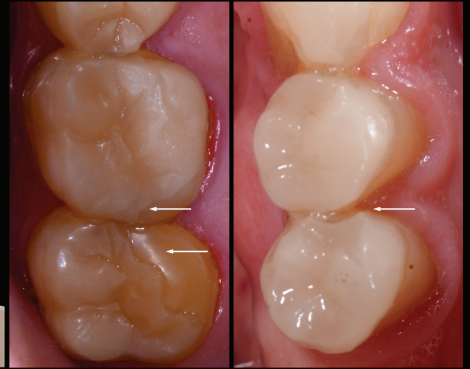


Fig. 3b

Fig. 3 (Photo courtesy of J. Oudkerk)

Observed technical complications.

a) In case 2, loss of the restoration on tooth #22, which was severely damaged, after a 19-mth follow-up.

The weak link was the interface between the resin cement and the sclerotic dentin. The restoration was successfully rebonded after eliminating the sclerotic layer with a diamond burr.

b) Typical minor chipping of thin restoration borders in occlusal contact.

Table 2 Comparison of global OHIP score and sub-scores before treatment and one month after treatment, and before treatment and two years after treatment, respectively (1 being the best score and 5 the worst).

	Before Treatment (Mean±SD)	1month (Mean±SD)	p value (Before treatment- 1 month)	2 years (Mean±SD)	p value (Before treatment- 2 years)
Global Score	1.88±1.26	1.54±0.92	0.0010***	1.36±0.92	<0.0001***
Functional limitation	2.02±1.34	1.79±1.03	0.5235 ns	1.56±1.09	0.02*
Physical pain	2.56±1.41	1.84±1.06	0.0035**	1.70±1.21	0.0003***
Psychological discomfort	2.49±1.36	1.71±1.15	0.0006***	1.49±1.7	≤0.0001***
Physical disability	1.66±1.15	1.52±0.89	0.7753 ns	1.17±0.52	0.0055**
Psychological disability	1.79±1.12	1.38±0.73	0.0462*	1.33±0.93	≤0.0002***
Social disability	1.09±0.37	1.09±0.28	1 ns	1.09±0.37	1 ns
Handicap	1.34±0.94	1.14±0.47	0.4345 ns	1.02±0.15	0.0431*

SD: Standard deviation, * Significant ($p \leq 0.05$), ** Significant ($p \leq 0.01$), *** Significant ($p \leq 0.001$), ns Insignificant

6 | 4. Discussion

Tooth wear is a multifactorial phenomenon, and in the present study, all of the patients showed both mechanical and chemical risk factors related to bruxism and soft drink consumption, highlighting the importance of early diagnosis and information in the prevention of this pathology [9]. The BEWE index was used for wear quantitative evaluation because of its common utilization and ease of use [30]. The high scores obtained (>13) and the presence of patients' aesthetic and functional complaints, particularly tooth pain, constitute indications for treatment, as recommended in recent European guidelines [9].

Despite its high risk regarding the one-step significant VDO increase (mean of 5.08 ± 0.84 mm at the incisal pin), i.e., without using provisional restorations or nightguards to test the new VDO as often recommended [22], the results of the One-step No-prep approach confirmed results obtained with the three pilot cases [23]. The global OHIP score was, as shown in a previous study related to severe tooth wear treatment with direct composites [38], significantly improved already one month after treatment and still after two years, while all sub-scores, except the one for social disability, were significantly improved after two years (Table 2). Detailed analysis of the sub-scores showed that, from a function and pain point of view, the patient's quality of life improves with time. In fact, patients quickly adapted to their new occlusal relationships, and only pronunciation troubles were present at one month but not later. The VDO determination is always an inaccurate and empirical process, and with the One-step No-prep approach, the principle is simply to recreate missing tissues on the basis of the remaining tooth anatomy. Although reported to be risky and not recommended for indirect restoration techniques, the one-step approach of VDO increase is already successfully used with direct techniques [16,12], and it was previously reported that a 5 mm increase at the incisal pin did not engender any undesirable effects on the temporomandibular joints and associated muscles [39,40]. In this limited clinical study, none of the patients reported any problems with the one-step VDO increase.

PICNs were confirmed to be well-adapted to the technique in terms of the ability to be milled in very low thickness (up to 0.11 mm on molars) and ease of in-mouth adjustment, particularly regarding occlusal contact points. Indeed, ceramic restorations cannot be easily adjusted and repolished after placement, with PICNs and other composite materials being more adapted to a one-step approach. PICNs also exhibit an elasticity modulus, which is comprised between the enamel and dentin, while ceramics are too stiff, and other composites are too flexible, and their damping behavior is particularly interesting in cases of bruxism and high occlusal stress [41]. Other advantages of PICNs are the

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bonding properties: in the present study, despite the presence of bruxism and the non-retentive prosthesis design, only one debonding was registered among 192 restorations. It was related to a severely damaged upper incisor, with sclerotic dentin and a small amount of enamel, and the fracture occurred at the interface with tooth tissues and not in the material (Fig. 2). Regarding mechanical strength, 5.73 % of minor chipping was observed after two years, most often on thin restoration edges, which were in occlusal contacts. Indeed, restorations were very thin and submitted to extreme conditions, particularly due to high occlusal stress. From this point of view, the new generation of PICNs should perform better due to their high flexural strength and flexural load energy [41]. Despite this fact, the survival rate and success rate of restorations were 100 % and 93.75 %, at two years, which were higher than recently reported survival and success rates at three years for partial coverage posterior restorations in PICN (95.6 % and 82.4 %, respectively, for 44 restorations) [27] and in lithium disilicate glass-ceramic (98.3 % and 85 %, respectively, for 60 restorations) [42]. However, to the authors' knowledge, there have been no clinical studies reporting the performance of partial indirect restorations for full-mouth rehabilitation of severe worn dentition. Several studies were published about treatment of those cases with direct composites showing various results [14,16,43,12]. Loomans et al. reported, for 1256 restorations, an overall success rate of 94.8 % at 3.5 years, and a survival rate of 99.3 % [12], which is similar to the present study, while Sundaram & Bartlett concluded that the use of direct (as artisanal indirect) composite resin for restoring worn posterior teeth is contraindicated [14]. Those contradictory results could be explained by the operator-dependent effect of direct techniques, or the difference between the applied composite resins (microfilled versus highly filled hybrid composite resin) [16]. The inconvenience of direct composites is, from a material point of view and compared to PICNs and other CAD-CAM composites, the degree of conversion of monomers, which is lower due to the polymerization mode (light curing compared to high temperature and high pressure for PICNs) [44]. In fact, this parameter influences all material properties, such as mechanical properties, biological properties (monomer release), chemical stability and aging [24,25]. Moreover, CAD-CAM blocks are more homogeneous with fewer flaws, also promoting mechanical behavior. Finally, with respect to the rapid evolution of digital dentistry and chairside systems, occlusion and tooth anatomy will be easier to design with computer help, promoting the use of CAD-CAM restorations, particularly in composite materials, which are well-adapted to those manufacturing processes.

Regarding the restorations' color matching and translucency, the FDI criteria yielded high scores for evaluators and patients after two years. Furthermore, OHIP results showed significant better psychological comfort after treatment (Table 2).

Future perspectives include the study of new PICN materials, particularly the recently introduced biomimetic PICN, which exhibits a gradient in mechanical and optical properties to mimic tooth tissues. Its hardness is similar to enamel on the surface and dentin in the depths, and its high flexural strength and flexural load energy are promising in terms of wear behavior, chipping resistance and damping effect.

6 | 5. Conclusion

The One-step No-prep technique is a minimally invasive and straightforward approach for the treatment of severe and generalized tooth wear. The treatment protocol was shown to yield successful clinical results from the functional and aesthetic points of view, the one-step approach of VDO increase was well tolerated and the global OHIP score, as 6 sub-scores on 7, were significantly improved. The present study provided significant data about the clinical performance of PICN partial restorations with respect to the existing literature, constituting the largest sample of restorations examined until now. The observed survival (100 %) and success (93.75 %) rates of restorations at two years were high, despite the extreme conditions to which the material was submitted. Minor chipping of very thin borders submitted to occlusal stress was the most frequent complication encountered. Finally, with respect to the rapid evolution of digital dentistry and the development of chairside systems, occlusion and tooth anatomy will be easier to design with computer help, which promotes the use of indirect CAD-CAM composite restorations for the treatment of severe worn dentition.

Future perspectives could include the development of clinical research to confirm the present results and the performance of future generations of PICN materials, particularly biomimetic PICN.

6 | 6. CRediT authorship contribution statement

J Oudkerk: Methodology, Validation, Investigation, Data curation, Writing - original draft, Writing - review & editing, Visualization, Project administration. **M Eldafrawy:** Investigation, Data curation, Writing - review & editing. **S Bekaert:** Validation, Formal analysis, Data curation, Writing - review & editing, Project administration. **C Grenade:** Investigation, Writing - review & editing. **A Vanheusden:** Investigation, Writing - review & editing. **A Mainjot:** Conceptualization, Methodology, Validation, Investigation, Writing - original draft, Writing - review & editing, Visualization, Supervision.

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6 | 7. Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

6 | 8. Acknowledgements

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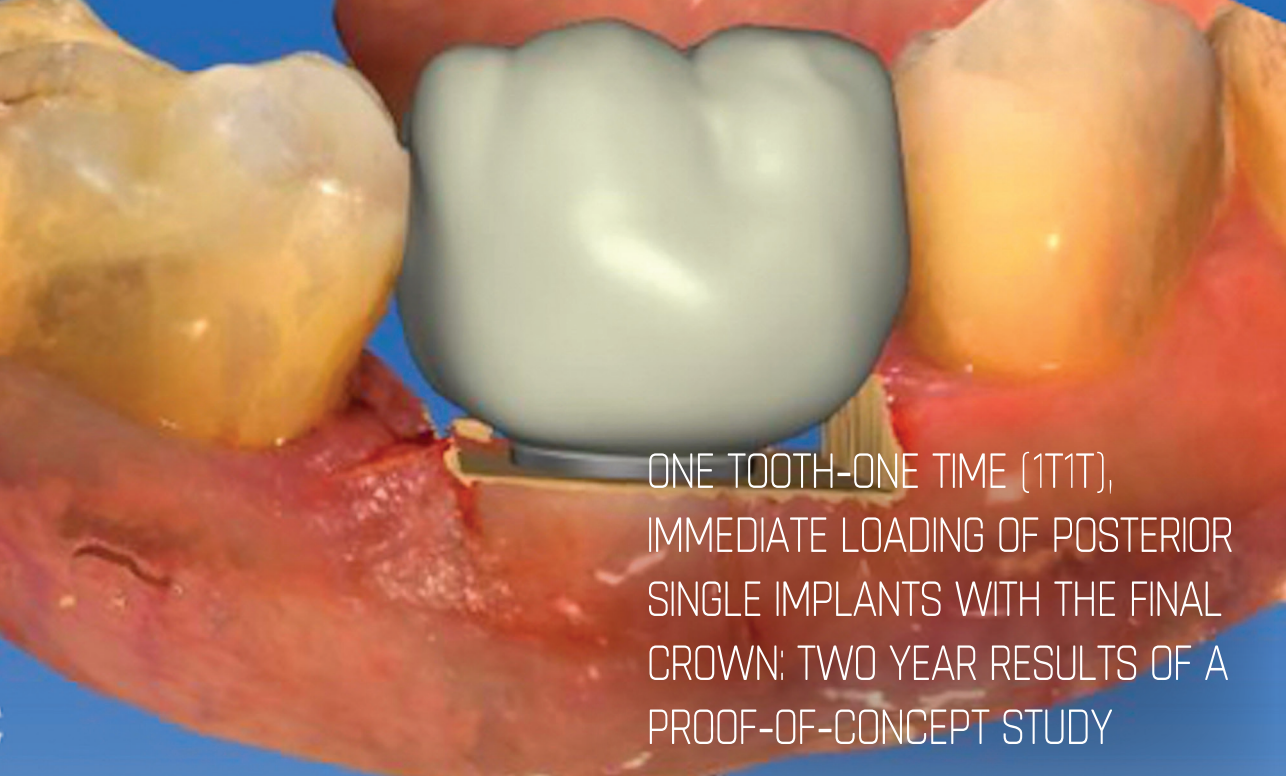
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ONE TOOTH-ONE TIME (1T1T),
IMMEDIATE LOADING OF POSTERIOR
SINGLE IMPLANTS WITH THE FINAL
CROWN: TWO YEAR RESULTS OF A
PROOF-OF-CONCEPT STUDY

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Chapter **7**

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7 | Abstract

Aims: The aim of this study was to evaluate the two-year outcomes of the One Tooth-One Time (1T1T) completely digital workflow, allowing the immediate loading of a single implant in the posterior region with a final CAD-CAM crown made of a polymer-infiltrated ceramic network (PICN).

Materials and methods: A series of 10 implants were placed, and an intra-oral scan was taken after the surgery. A final screw-retained PICN crown was manufactured chair-side and placed the same day in full occlusion. Peri-implant bone remodeling and soft-tissue health were evaluated, as restoration performance using World Dental Federation criteria and Pink and White Esthetic Scores. Patient-related outcome measures (PROMs) and time consumption data were collected.

Results: After two years, the implant survival rate was 100%. The debonding of one crown from its base led to a 90% prosthodontic survival rate and the remaining crowns were all considered successful. The mean peri-implant bone remodelling yielded 0.87 (SD: 0.96) and 0.55 (SD: 0.53) mm after one and two years, respectively. Mild or no inflammation of peri-implant soft tissue was observed in most implants. The overall treatment time reached 175 min and PROMs displayed high patient satisfaction.

Conclusions: This study constitutes the first report considering immediate loading of a single implant in the posterior region with a final crown in occlusion. In this case-series, the two-year outcomes of the 1T1T protocol seem rather promising and fulfil patient expectations. However, those preliminary results need to be confirmed by randomized control trials and patient selection is probably a key factor in this procedure success.

Conflict of interest: The authors declare that they have no conflict of interest.

Keywords: Immediate loading, digital workflow, intra-oral scanner (IOS), dental implant, polymer-infiltrated ceramic network (PICN), CAD/CAM

7 | 1. Introduction

In today's world, things are going increasingly fast, mainly due to the impact of digital technologies. The trend is similar in the dental field: digital workflows are increasingly used, especially for single-unit restorations, and a recent systematic review concluded that patients tend to prefer digital over conventional techniques [1]. In particular, advances in computer-aided design and manufacturing (CAD-CAM) materials and chair-side CAD-CAM systems offer a new landscape for implant dentistry and a complete virtual environment without any physical model situations [2]. These new developments can contribute to improvement in patients' expectations and satisfaction in offering straightforward and cost-efficient protocols, but those breakthroughs should not reduce treatment quality and prognosis.

The reliability of immediate loading on single implants in the posterior mandible, using provisional crowns, has been demonstrated in several reports; the evidence is weaker for the posterior maxilla [3, 4]. In the reports describing immediate restoration in the posterior regions, non-occlusal immediate loading using resin-based provisional is often described, and the final crowns would be realised several weeks after implant placement once the osseointegration was achieved [5]. Although such protocols may improve patient satisfaction, several appointments and a significant contribution by the dentist and the lab technician are required, which affects the overall treatment cost [6].

On the other hand CAD-CAM composites, particularly of polymer-infiltrated ceramic network (PICN), also called hybrid ceramics (Vita Enamic, Vita Zahnfabrik, BadSackingen, Germany), now constitute an alternative to ceramics for definitive, single-unit restorations. They exhibit some advantages over ceramics, such as their better machinability (faster and with lower-thickness milling, with less edge chipping) [7, 8], the absence of post-milling firing and the ease of in-mouth adjustments. Most of all, the material's stiffness (elastic modulus value between enamel and dentin, whereas ceramics are stiffer than enamel) and damping effect ability make them potential candidates for implant restorations [9, 10], particularly for immediate loading.

Intraoral scanning (IOS) of single-unit implants directly after surgery and the chair-side manufacture of a PICN crown enables the delivery of a final restoration within the same visit. Such a protocol was previously introduced, describing two pilot cases, as the One-tooth One-time (1T1T) approach [11]. However, clinical research remains to be developed to validate this approach.

The aim of the present study was to evaluate the clinical and radiographic outcomes prospectively after a two-year follow-up of 10 single implants placed according to the 1T1T protocol. In addition, the time necessary to perform the procedures and the patients' reported outcome measures (PROMs) were assessed.

7 | 2. Materials and methods

Regulatory approvals, screening and consent

The study was approved by the Ethical Committee of Liège University Hospital and was conducted in accordance with the Declaration of Helsinki Helsinki of 1975, as revised in 2000 and all applicable local regulations and standards (file: B707201629113, approved on 18/08/2016). Participants complying with the inclusion criteria were provided with written information about the study, advising them of the study requirements and possible risks. Enrolled patients signed the informed consent form according to local regulations.

Study Design

The present study was designed as a single-centre prospective case series of nine consecutive patients presenting one or several maxillary or mandibular single-unit missing teeth in the posterior region. Patients were treated in the Dental Department of the University Hospital of Liège and recruited between June 2016 and September 2016. Both surgical and prosthodontic procedures were performed by a single periodontist and a single prosthodontist, respectively. The patients were followed for a period of two years. At each follow-up visit, the implant and prosthodontic data were collected. In addition, possible patient dropouts and withdrawals, as well as adverse events, were carefully monitored during the entire investigation period.

Patient selection

Inclusion criteria

- Voluntarily signed the informed consent form before any study-related action
- Age: at least 18, with one or two missing teeth in the posterior area, seeking for implant therapy
- In good systemic health (ASA I/II)
- Healed alveolar crest (at least 12 weeks post-extraction)
- Bone volume allowing the placement of an implant of at least 10 mm in length and 4.1 mm in diameter

Exclusion criteria

- Medical conditions contraindicating implant placement
- Smokers
- Full-mouth plaque score (FMPI) lower than or equal to 25%
- Sites treated with socket preservation or bone reconstruction techniques
- Presence of intraoral infection (periodontitis, caries and so on)
- Absence of antagonistic fixed teeth
- Less than 2 mm of keratinised gingival tissue in the implant site

Clinical procedure (Figure 1)

The patients were treated according to the previously described 1T1T protocol [11]. They were subjected to a cone-beam computed tomography (CT) scan to evaluate the

Fig. 1 (a) Implant placement (TE implant, Straumann Group, Switzerland), using a minimally invasive surgical approach.
(b) Digital image of IOS with the scan body placed on the Variobase.
(c) Digital setup of the crown.
(d) Crown just after the chair-side manufacturing cemented on a titanium base (Variobase, Straumann Group, Switzerland). Note the emergence profile.
(e) Final CAD/CAM crown made of PICN placed a couple of hours after the surgical procedure.
(f) Occlusal view two months after the placement of the crown. Note the occlusal contact points.
(g) Clinical outcome after one year.
(h) Intra-oral X-ray after one-year follow-up.



bone dimensions in the area of interest and to confirm that they comply with the inclusion criteria. All subjects received preoperative antibiotic (amoxicillin 2 g or, if allergic, clindamycin, 600 mg) one hour before the surgery. After local anaesthesia, minimally invasive incisions were made. The drilling sequence was carried out according to the protocol of the manufacturer for the placement of a tapered-effect (TE) implant, (Straumann Group, Switzerland) of at least 10 mm in length and 4.1 mm in diameter. The implant stability was measured using the wrench key and recorded in N/cm. The loading procedure was considered only if the insertion torque was higher than or equal to 35 N/cm. The implants were covered with healing abutments before suturing with a resorbable suture (Vikryl, 5/0).

Directly after the surgery, titanium Variobase abutments (Straumann Group, Basel, Switzerland) and a CEREC plastic scan body were placed on the implant in order to take an IOS (Omnica camera, CEREC System, Sirona, Salzburg, Austria) of the upper and lower jaws as well as of bite registration, according to manufacturer recommendations. Healing abutments were placed during the chair-side crown manufacturing process. Screw-retained implant restorations were designed with CEREC 4 software and manufactured with the CEREC MCXL (CEREC System), using the dedicated PICN blocks, with a pre-shaped hole fitting the abutment (Vita Enamic IS-16L, Vita Zahnfabrik, Bad Säckingen, Germany). Special attention was given to the restoration's emergence profile. The PICN crown intaglio was etched with hydrofluoric acid for 60 seconds, subsequently cleaned in a 90% ethanol ultrasonic bath for five minutes and covered by a primer layer (Monobond Plus, Ivoclar Vivadent, Schaan, Lichtenstein), which was applied using a microbrush and left for 60 seconds and then air dried for 10 seconds. The Variobase abutment was screwed onto an implant replica to facilitate handling. After neck protection with a silicone impression material, the abutment was sandblasted (50- μ m alumina particles, two bars), cleaned and covered by a primer layer in the same way as the crown intaglio. After screw head protection with Teflon, the crown was cemented on the abutment with a resin composite cement (Multilink Hybrid Abutment, Ivoclar Vivadent), according to the manufacturer's recommendations. Finally, if needed for aesthetic purposes, the restoration was stained with a light-cured nano-filled composite coating agent (GC Optiglaze colour, GC Corporation, Tokyo, Japan), according to the manufacturer's recommendation and after crown etching and silanisation, as performed for bonding. The final crown was subsequently polished with dedicated instruments (Vita Enamic Polishing set). Before placement, the crowns were cleaned for 2 min in three consecutive ultrasonic baths (cleaning agent, sterile water and then 90° ethanol). Once manufactured and cleaned, the PICN definitive crowns were directly placed on the freshly placed implants. If necessary, the crowns were subjected to occlusal or contact point adjustments and were screwed with a torque of 15 N/cm. The access channels were filled with Teflon and temporary filling material

(Cavit, 3M ESPE, St. Paul, MN, USA), and X-rays were taken for baseline radiologic data. Chlorhexidine spray (0.12%) was prescribed twice daily on the surgical sites for seven days; ibuprofen, 600 mg TID, was prescribed for 4–5 days according to the patient's need. Patients were advised to avoid tooth brushing at the implant site for seven days.

Follow-up and data collection

Patients were followed up at seven days, two months, six months, one year and two years. In case of any complications, the patients were asked to contact one of the study dentists directly. After two months, the osseointegration was verified with a radiograph and a clinical assessment. The restorations were torqued at 35 N/cm and the access channel filled with a light-cured composite (Els Extra Low Shrinkage® Saremco, Rebstein, Switzerland). At six months, one year and two years, the patients were recalled to evaluate both implant and prosthodontic outcomes, and patient-reported outcome measures were collected.

Occlusal risk factors

Occlusal relationships were classified based on the clinical examination of two independent evaluators. Class I, class II.1, class II.2 or class III as well as malocclusion, anterior or posterior crossbite, edge to edge or open bite were identified. The presence of bruxism was recorded if the patient fulfilled at least two criteria: A) reporting of tooth grinding during the night or day; or B) the presence of at least one clinical sign among the following: abnormal attrition wear facets on the teeth; transitory pain or fatigue on waking felt in the jaw muscles; temporal headaches on waking; and jaw locking on waking related to teeth grinding during sleep [12]. The use of an occlusal nightguard was noted.

Peri-implant bone remodeling and soft-tissue health

Peri-implant bone remodeling of the mesial and distal bone levels was assessed based on intra-oral X-rays, using the parallel technique performed immediately after surgery and 1 and 2 years after the procedures. Marginal bone remodeling was measured using the Image J64 (National Institutes of Health, Bethesda, MD, USA) image processing software. The peri-implant soft-tissue health was also assessed by scoring peri-implant bleeding on probing (BOP) using the Silness and Loe index on each implant [13]. Gingival index scores below or equal to one were considered healthy peri-implant soft tissues. Moreover, at each follow-up visit, a full-mouth plaque score was collected using the O'Leary plaque index [14].

Clinical evaluation of PICN screw-retained restorations

At each follow-up visit, clinical pictures of the restorations were taken, and two independent evaluators clinically evaluated the restorations, following the criteria of the World Dental Federation (FDI) [15, 16]. Three dimensions, which represented 18 items, were described: esthetic, functional, and biological. Each item was assessed on a five-point Likert scale, 1 corresponding to an excellent restoration and 5 corresponding to a restoration that needs to be replaced. In cases of discrepancy, an agreement was found between evaluators to determine the final score. Scores 4 and 5 were considered failure.

Aesthetic outcomes (PES-WES)

Although it was initially described to evaluate the esthetic outcomes in the anterior region, the pink aesthetic score/white aesthetic score (PES/WES) index was used in this case in the posterior region. A score of 2, 1, or 0 was assigned to each PES/WES parameter. Hence, the highest possible score was 14 for the PES according to Furhauser et al. [17] and 10 for the WES according to Belser et al. [18], which represented, respectively, a close match of the peri-implant soft-tissue conditions and the clinical single-tooth crown compared to the respective features present at the contralateral natural tooth site.

Time

The time necessary for each step of the clinical procedure and chair-side manufacturing (respectively, implant placement, IOS, chair-side manufacturing and restoration placement) were recorded in minutes. In addition, the overall time necessary to perform the 1T1T procedure was calculated by summing up the time necessary for each of these steps.

Patient reported outcomes measures

The patients received satisfaction questionnaires related to the procedure, the function and the aesthetics of their crown(s) following the intervention as well as at the one-week, one-year and two-year follow-ups.

Statistical analyses

Results were expressed as means \pm standard deviations (SDs) or medians for quantitative variables, whereas frequencies and proportions (%) were used for categorical variables. Statistics are only descriptive, because there was no failure. Calculations were always carried out with the maximum amount of data available. Missing data were not replaced. Data analysis was carried out using GraphPad Prism (GraphPad Software, San Diego, CA).

7 | 3. Results

Patients' and site characteristics

A total of 10 implants were placed in nine patients; four were female and five were male, with a mean age of 45.7 years (ranging from 30 to 62 years). No patients dropped out during the follow-up of the study. Patient-related parameters, including occlusal risk factors, are detailed in *Table I*. The presence of clinical signs of bruxism, and consequently high occlusal stress, was suspected in six patients out of nine, and only one patient wore a nightguard for this reason.

Table I <i>Patients and implant-related data</i>		
Patients (n tot=9)		% (n)
Tobacco use	Yes	0 (0)
	No	100 (9)
Occlusal relationships	Class I, II1	55.6 (5)
	Class II2	11.1 (1)
	Class III	0 (0)
	Crossbite	11.1 (1)
	Edge to edge	22.2 (2)
Guidance	Group	55.6 (5)
	Canine	44.4 (4)
Occlusal stress	Yes	66.7 (6)
	No	33.3 (3)
Nightguard	Yes	11.1 (1)
	No	88.9 (8)
Implants (n tot = 10)		% (n)
Location	Maxillary premolar	20 (2)
	Maxillary molar	20 (2)
	Mandibular premolar	0 (0)
	Mandibular molar	60 (6)

Implant survival rates

All implants reached a primary stability of at least 35 N/cm and all were immediately loaded with the final crown. No implants failed over the two-year follow-up period, leading to an implant survival rate of 100%.

Peri-implant bone remodeling and soft-tissue health

The mean peri-implant bone remodelling from baseline to one and two years post-loading was, respectively, 0.87 (SD: 0.96) mm and 0.55 (SD: 0.53) mm. Most of the implants (80%) showed no or mild inflammation on probing, whereas more severe BOP was found on two implants at both one and two years, respectively. Full-mouth plaque scores above 15% were observed in two and four patients, respectively, at the one- and two-year follow-ups. Details related to survival rates and clinical parameters are displayed in *Tables II and III*.

Table II <i>Survival rates and peri-implant bone remodelling</i>		
	1Year	2 Years
Implant survival rate (n tot = 10)	100%	100%
Peri-implant bone remodelling (mean±SD)	0.87 ± 0.96mm	0.55 ± 0.53mm
Prosthetic survival rate (n tot = 10)	100%	90%

Table III <i>Plaque and gingival indexes</i>										
	1 week		2 months		6 months		1 year		2 years	
Full-mouth plaque index	<15% (n)	>15% (n)	<15% (n)	>15% (n)	<15% (n)	>15% (n)	<15% (n)	>15% (n)	<15% (n)	>15% (n)
Patients (n tot = 9)	8	1	2	7	5	4	7	2	5	4

	1 week		2 months		6 months		1 year		2 years	
Gingival index	0-1 (n)	≥2 (n)	0-1 (n)	≥2 (n)	0-1 (n)	≥2 (n)	0-1 (n)	≥2	0-1 (n)	≥2 (n)
Implants (n tot = 10)	9	1	9	1	9	1	8	2	8	2

Clinical evaluation of PICN screw-retained restorations

All crowns could be successfully manufactured and placed. Only minor occlusal or interproximal adjustments were necessary. The most frequent complication was crown unscrewing, which occurred in half of the crowns within two months after the procedure. These restorations were then torqued at 35 Ncm and no further unscrewing occurred. In addition, at the two-year recall in a single case, the PICN crown appeared to be slightly moving. In fact, it was debonded from the titanium Variobase and the crown was immediately replaced with a new one within the same appointment, using the

original file in the CEREC system, that is, without taking a new optical impression. The replacement of this crown led to a prosthodontic survival rate of 90%. Aesthetic, functional and biological properties related to the remaining original restorations (nine out of 10) were sufficient to excellent, according to the FDI rating. Details are presented in *Table IV*. No complications were detected on antagonistic teeth.

Table IV FDI criteria

	Clinically excellent		Clinically good		Clinically sufficient		Clinically unsatisfactory		Clinically poor		Acceptable		Unacceptable	
	1 yr %	2 yrs %	1 yr %	2 yrs %	1 yr %	2 yrs %	1 yr %	2 yrs %	1 yr %	2 yrs %	1 yr %	2 yrs %	1 yr %	2 yrs %
A. Esthetic properties														
Surface luster	30	30	70	70							100	100		
Staining														
a. surface	70	70	30	30							100	100		
b. margin	80	80	20	20							100	100		
Color match and translucency	40	40	50	50	10	10					100	100		
Anatomical form	80	80	20	20							100	100		
B. Functional properties														
Fracture of material and retention	100	90								10	100	90		
Approximal anatomical form														
a. contact point	90	80			10	20					100	100		
b. contour	100	100									100	100		
Radiographic examination	100	100									100	100		
Patient's view	90	90	10	10							100	100		
C. Biological properties														
Periodontal response	100	100									100	100		
Adjacent mucosa	100	100									100	100		
Oral and general health	100	100									100	100		

PES/WES

The mean global PES score yielded 11.6 out of 14 after two years. The lowest values were found for the 'alveolar process' and the 'presence of the distal papilla'. The mean global WES reached 9.2 out of 10 after two years, and the lower value was found for the surface texture. Details for the PES/WES scores appear in *Table V*.

Table V Detailed description of PES/WES needed restorations (n tot =10). PES: pink aesthetic score, WES: white aesthetic score, SD: standard deviation

PES	Mesial papilla (mean±SD)	Distal papilla (mean±SD)	Level of soft-tissue margin (mean±SD)	Soft-tissue contour (mean±SD)	Alveolar process (mean±SD)	Soft-tissue color (mean±SD)	Soft-tissue texture (mean±SD)	Total PES Score (mean±SD)
1 year	1.7±0.67	1.3±0.67	2.0±0.0	1.4±0.52	1.3±0.48	1.8±0.42	1.8±0.42	11.30±1.49
2 years	1.7±0.67	1.20±0.79	1.80±0.63	1.90±0.31	1.60±0.52	1.70±0.48	1.70±0.48	11.60±1.50
WES	Tooth form (mean±SD)	Tooth volume/outline (mean±SD)	Color (hue/value) (mean±SD)	Surface texture (mean±SD)	Translucency (mean±SD)			Total WES Score (mean±SD)
1 year	1.80±0.42	1.90±0.32	1.40±0.52	1.30±0.48	2.0±0.0			8.4±1.07
2 years	2.0±0.0	2.0±0.0	1.80±0.42	1.50±0.53	1.90±0.32			9.2±0.79

Time

The mean overall treatment time, including clinical and chair-side procedures, reached 175.7 ± 60.7 minutes. The mean necessary time, respectively, for the surgery, the prosthodontic steps and the manufacturing process were 15.7, 54.9 and 105.1 minutes. Details appear in *Table VI*.

Table VI Duration of each step of the 1T1T protocol and total time needed

Procedure step	Minutes (mean±SD)
Implant placement	15.70±4.7
Digital impression	14.5±4.3
Crown manufacturing	105.1±40.8
Placement and occlusal adjustments	40.4±18.1
TOTAL	175.7±60.66

PROMs

The general satisfaction related to the overall treatment and the aesthetic perception of the crown both reached a score of 9.5. The scores for comfort during the surgery and the prosthodontic procedures were, respectively, 9.3 and 9.5. All patients said that they would do the treatment again and that they would recommend it to a friend. PROMs-related data is available in *Table VII*.

Table VII PROMs as measured using a 10-point Likert scale. NA: Not Applicable

Score	Intervention (mean±SD)	1 week (mean±SD)	1 year (mean±SD)	2 years (mean±SD)
Comfort during surgery	9.3±0.87	NA	NA	NA
Comfort during prosthodontic procedure	9.5±0.73	NA	NA	NA
Aesthetic perception	9.5±0.53	NA	NA	NA
General satisfaction	NA	9.4±1.01	9.3±1.0	9.8±0.44
Would recommend to a friend	NA	100%	100%	100%

7 | 4. Discussion

According to the present preliminary study, the innovative 1T1T protocol displayed rather successful outcomes after a two-year follow-up. To our knowledge, this is the first report considering data of an immediately loaded single implant in the posterior region with a final crown in occlusion, using a fully digital workflow without the use of physical models. These preliminary implant survival and success rates are encouraging; however, it is important to emphasise that several key diagnostic factors, as described by Bahat et al., were taken into account and may have positively influenced the results [19]. First, the patients were carefully selected regarding bone volume and quality; all sites were healed, and the bone availability allowed placement of regular-diameter implants of 10 and 12 mm. Moreover, smoking patients were excluded from the study. Although tobacco use was not considered a risk for early implant failure in immediately loaded implants, it seems to influence long-term peri-implant bone remodelling and therefore implant success [20, 21]. Yet, it must be noted that patients exhibiting clinical signs of bruxism were not excluded. Consequently, the majority of implants (70%) were probably submitted to high occlusal stress, which could have negatively influenced the treatment prognosis.

The choice of implant may also have influenced the present results. Tissue-level, tapered implants with a highly hydrophilic surface were used to implement, respectively, the primary and secondary implant stability and to respect soft-tissue integration. Indeed, as demonstrated by some authors, it is preferable to insert single implants with a medium (>35 N/cm) to high (80 N/cm) insertion to minimise early implant failures when loading them immediately [22]. *In vitro* and clinical studies have emphasised the relevance of tapered implant design as employed in the present study to ensure primary stability [23 - 25]. Highly hydrophilic implant surfaces have been shown to improve the speed of osseointegration as well as the quality and quantity of osseointegration [26 - 28]. Reaching faster secondary implant stability is, in principle, an argument for an immediate loading protocol because the total stability of the implant would not drop in the first

weeks after implantation; therefore, such a hydrophilic implant surface was chosen for the 1T1T protocol. The choice of a PICN CAD/CAM restorative material characterised by a lower elasticity modulus than ceramics [29] and, most of all, an ability to absorb occlusal stress by reversible deformation [30] was also considered by the authors to be a key aspect of the procedure to have a restoration with a damping effect. Because of their specific microstructure, PICN materials resulting from the infiltration of a partially sintered glass–ceramic block secondarily infiltrated by monomers may positively influence the biomechanics of immediately loaded implants and therefore the osseointegration process. Although it was not demonstrated yet, this type of material may also have an impact on peri-implant bone remodelling and would therefore be an advantage in the long-term outcomes of implants.

After two years of loading, a 100% implant survival rate was yielded: on average, peri-implant bone loss was less than 1 mm. This peri-implant bone remodelling is comparable to what is described in the literature when using tissue-level implants in a classic loading procedure [31].

As suggested by recent European Federation of Periodontology (EFP) consensus statements, peri-implant soft-tissue health is an important criterion for implant success [32], and bleeding on probing may be the first indicator of peri-implant disease such as mucositis or peri-implantitis [28, 33 - 35]. In the present study, after two years of loading, 80% of the implants displayed no or mild peri-implant soft-tissue inflammation, whereas some implants exhibited signs of mucositis with moderate bleeding on probing (20%) [36]. Recently, experimental PICNs have been shown to exhibit biocompatibility properties comparable to lithium disilicate and no monomer release, due to the original polymerisation process involving high temperature and high pressure [37, 38]. Those properties can contribute to the acceptable peri-implant soft-tissue health found in the present study.

The aesthetic results of implant restorations are also a critical parameter for treatment success, especially because patient expectations tend to increase even in the posterior region in the maxilla when they show up to the first molar. Although the PES/WES scores were described for the anterior region, it is the only index proven to be very reliable and reproducible for implant-supported single crowns [39] and it was therefore used in the present study. The PES was rather good, and the variable with the lowest score was generally the 'alveolar process resorption', which is related to buccal bone resorption after extraction [40]. Indeed, the study criteria excluded previous ridge preservation techniques or extraction and immediate implants that may have limited this buccal bone remodelling [41 - 43]. Also, the score related to the papilla depends on the anatomy of the adjacent tooth bone level and anatomy [44, 45]. These parameters are not necessarily related to the materials or protocol used in the present study; therefore, this data should be interpreted cautiously. The PES scores may have

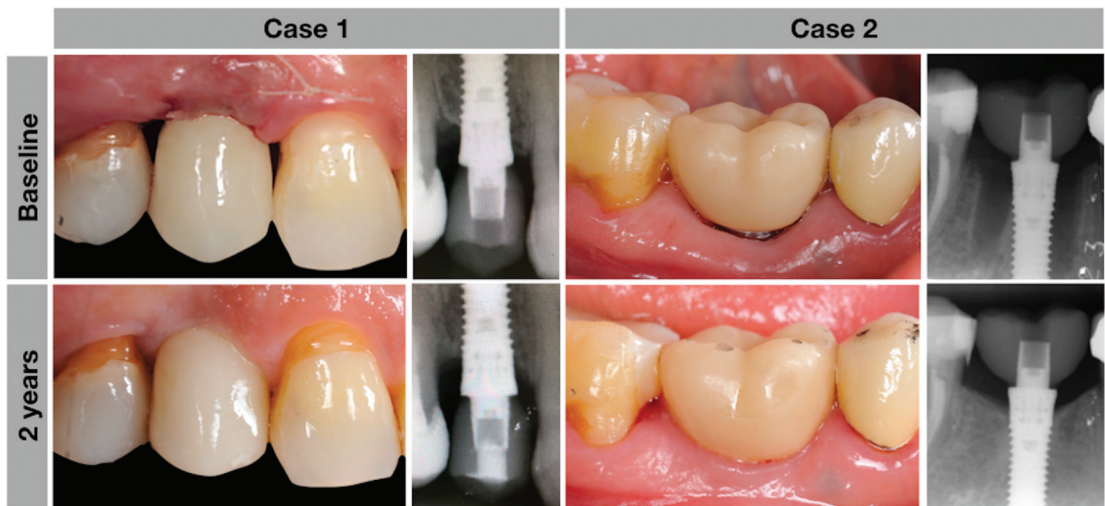


Fig. 2 In these two cases, PICN crowns placed with the 1T1T protocol at baseline and two-year follow-up. We note the improvement of the peri-implant soft-tissue contours over time as well as the excellent peri-implant bone-level stability from baseline to two years.

also been influenced by the immediate placement of the final crown with the ideal emergence profile allowing prosthesis-driven soft-tissue healing. This aspect is a potential additional benefit of the 1T1T protocol (Figure 2).

Regarding aesthetic properties of the PICN restorations, both WES and FDI evaluation at two years displayed very good results; excellent or good scores were found for all crowns regarding luster, staining and anatomical form; for colour match and translucency, only one crown was scored as sufficient, whereas the others were good or excellent (Table IV). Moreover, the FDI results did not change after two years, which is promising regarding material ageing. The FDI evaluation and PROMs also highlighted high patient satisfaction from the aesthetic point of view. In fact, if CAD/CAM composite restorations can be suspected to be less aesthetic and glazy than a veneered ceramic crown, patients were shown to be totally satisfied with the result in the posterior area, whereas future perspectives include the development of more aesthetic PICN materials [30].

Over the two-year follow-up, crown unscrewing occurred only within the two months after the procedures in 50% of the crowns. The restorations were initially screwed with a torque of 15 Ncm, which does not seem enough considering that the single crowns were in full occlusion. Because primary implant stability of 35 Ncm was required to apply the 1T1T protocol, we recommend torqueing the restoration at 35 Ncm from the first day. Indeed, in cases of unscrewing, the crowns were tight to 35 Ncm and no further complication of that type occurred.

It must be noted that no material fracture was observed, despite the presence of high occlusal stress in the majority of patients, which was previously shown to

promote chipping of zirconia-based restorations [46]. This can be attributed to the monolithic restoration design and the material's ability to absorb occlusal stress by reversible deformation. No antagonistic tooth complication was detected. The PICN material was easy to manufacture and to adjust on occlusal and interproximal contact points. Those occlusal contact points were shown to be able to evolve from one evaluation time to the other (*Figure 1*), which indicates that the material can adapt to occlusal relationships by a slight wear process, which could reduce the risk of occlusal contact point interference compared to harder materials. The only observed failure was related to a PICN crown debonding from the titanium Variobase at two years while proper surface pre-treatments were performed. The failure was located at the interface between the resin cement and the titanium base, and the resin cement was still present on the PICN surface. Indeed, the PICNs were shown to exhibit very good bonding properties, significantly higher than other CAD-CAM composites and similar to lithium disilicate glass-ceramics, which are considered the gold-standard [30]. Consequently, it seems that the design of the titanium base, which is round and short, should be improved to promote a better crown retention.

Finally, the 1T1T protocol is performed in a single day, compared to the conventional approach that would require three or more appointments. Overall, the procedure took about three hours. However, the time to welcome the patients, install and uninstall them in the operation room as well as the breaks in between were not taken into account. It is fair to say that it takes half a day and that the learning curve may improve the results found in this case series. All in all, it may take the same time as a conventional approach, with the main difference that the patient comes only once, which in certain cases is seen as a strong benefit from the patient's side, and the technical needs are reduced. At this stage, time efficiency and cost-effectiveness are difficult to extrapolate from the present study design, and further controlled randomised trials are necessary.

Patient-reported outcome measures are becoming important aspects in assessing the outcomes of implant therapies [47, 48]. Although excellent results related to the overall treatment satisfaction as well as comfort and aesthetic perception of patients were found, the data should be interpreted cautiously because, as already suggested by some authors, the perception of new technologies may have influenced the figures [49]. Moreover, in the absence of validated tools to assess PROMs for single-tooth replacement, the satisfaction questionnaires used in the present study might be discussable. However, according to the collected information, the 1T1T procedures seemed highly appreciated by the patients, particularly the single-visit characteristic, which is easier to manage with their own professional activity and can decrease global stress related to the procedure of tooth replacement.

7 | 5. Conclusion

Despite the limitation of the present study, this is the first report considering single implant immediate loading in the posterior region with a final crown in occlusion. These 2-year outcomes of the 1T1T protocol, using a fully digital workflow, seem rather promising. The straightforward technique provides immediate results, allows prosthesis-driven soft-tissue healing, is free of substantial complications and highly appreciated by the patients. However, patient selection is probably a key factor for the success of this procedure. Further clinical research is needed, including randomised controlled trials (RCTs) comparing this new protocol to conventional procedures.

7 | 6. Author contributions

France Lambert, contributed to conception and design, acquisition, analysis and interpretation, drafted manuscript; Maher Eldafrawy, contributed to acquisition, critically revised manuscript; Sandrine Bekaert, contributed to analysis and interpretation, critically revised manuscript; Amélie Mainjot, contributed to conception and design, acquisition, analysis and interpretation, drafted manuscript. All authors gave final approval and agree to be accountable for all aspects of the work.

7 | 7. References

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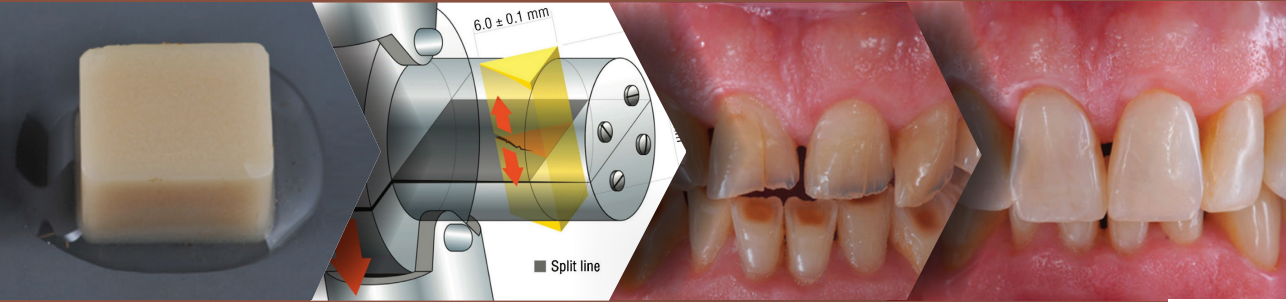
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CONCLUSIONS



Studying new materials from the bench to the clinic is a rich experience, which provides some insight of their potential and limitations in clinical practice. Moreover, translational research is not uni-directional and it provides a dynamic flow of information with the aim of improving the product. The *in vitro* investigations of PICN bonding performance, in particular the interfacial fracture toughness (IFT) with resin cement and the material microstructure influence, in addition to the introduction of novel functionally graded PICN materials constitute the bulk of this thesis. This was completed by the participation in clinical research on the commercial PICN (Vita Enamic) providing a highlight on its clinical performance in extreme conditions, which provides information about properties to be improved.

In this thesis, the bonding properties of PICN materials with a resin composite luting agent (RCLA) were evaluated by fracture mechanics using IFT measurement with the notchless triangular prism (NTP) specimen K_{IC} test, which constitutes an original approach compared to conventional bond strength test methods. IFT allows the evaluation of the interface properties rather than the mechanical strength of the whole assembly. It provides a good level of control and stable crack growth at a low cross-speed (0.05 mm/min), thus offering reliable measurements and the possibility of performing a fatigue process study, controlling crack propagation for a given K_I . It also avoids bias related to the creation and alignment of a notch, which could present some difficulties when the interface thickness is low. Moreover, the NTP specimen K_{IC} test requires no further preparation of samples after bonding, a procedure that may introduce flaws to the bonded specimens. Further modifications were implemented to the system, in which the IFT measurements were carried out in a water bath at mouth temperature to avoid any bias related to differences in temperature and to provide close approximation to mouth conditions. From the author's point of view, there are some limitations in the NTP specimen K_{IC} test. Those limitations include the time required to prepare the prisms, in which prisms are prepared one-by-one in a procedure that requires time and patience to reproduce the prisms with the correct dimensions. Bonding of the prisms is also time-consuming, in which each prism is bonded separately; however, this is considered as an advantage because all of the samples are submitted to light-curing in the same way. In addition to time, the initial cost of the materials for preparation and testing the prisms could be considered expensive. It is advisable to select the RCLA with a different shade than the restorative material for easier location of the adhesive interface while presenting the crack initiation point. A clinically relevant approach would be to implement the IFT tests into fatigue studies.

Results of the first and the second IFT studies highlighted the importance of material microstructure, and then class (Dispersed Filler versus PICN) on bonding interface performance. Particularly, surface roughness after pre-treatment appeared as a key factor,

which can be enhanced by silane application. The IFT of experimental homogenous PICN was shown to be superior than the gold standard glass-ceramic (IPS e.max CAD) when etched with 5% hydrofluoric acid and better than other dispersed filler (DF) CAD-CAM composites, namely Lava Ultimate and Cerasmart, when etched or gritblasted with 50 μm Al_2O_3 particles. PICN showed an original polymer-based honeycomb microstructure after etching, which leads to the selective dissolution of the glass-ceramic at the surface, exposing a polymer-based structure with micro- and nano-porosities that would, in turn, enhance the micromechanical retention. This specific microstructure was not shown in DF. This influencing effect of micromechanical retention on interface performance with resin cement, was supported by the strong correlation found between IFT and the developed interfacial area ratio (Sdr) of representative samples of the materials subjected to the same pretreatment protocols (either etching or gritblasting). It was proven from the IFT studies that PICN should be etched while DF should be gritblasted to achieve the highest interface performance with RCLA. Silane pre-treatment, as well, proved to be a critical factor in enhancing the IFT of PICNs with RCLA by increasing their wettability, thus promoting their spreading into the microroughness created by either etching or gritblasting.

Secondly, the novel functionally graded PICN (FG-PICN) can be considered as a breakthrough in the composite CAD-CAM industry for its original gradient in mechanical, as well as optical, properties it provides, which resemble enamel at the surface and dentin deeper in the block, particularly in terms of hardness and elastic modulus. This important step towards a biomimetic material could promote its wear and biomechanical behavior. In addition, the flexural strength of the dentin-like layer was found to be similar to lithium disilicate glass-ceramics, which can improve chipping resistance, whereas the flexural load energy was higher than glass-ceramics and translucent zirconia. This last property could promote occlusal stress absorbance in treatment of patients with bruxism (such as worn dentition cases) and on implant restorations, which can constitute a real improvement from a clinical point of view. The main limitation of the FG-PICN is that the gradient is in one-dimension, while it is in 3-dimensions in natural teeth, which should not cause a problem in manufacturing inlays, onlays, tabletops, and palatal and buccal veneers, but is not ideal for crowns.

Clinical applications of PICN materials were also explored in two studies; 1) the One-step No-prep approach of full-mouth worn dentition and 2) One-Tooth One-Time (1T1T) approach for replacing missing teeth. The first clinical study, One-step No-prep, is a minimally invasive and straightforward approach for restoring severely worn dentition. Despite the extreme conditions the restorations were subjected to, their survival rate was 100% after 2 years, with a success rate of 93.75%, which was mainly attributed to chipping at the edges. The higher flexural strength and flexural load energy of FG-

PICN could yield better success rates should they be used in similar clinical situations. The second clinical study, 1T1T, is related to a completely digital workflow of the immediate loading of a single implant in a posterior edentulous area with the final CAD-CAM PICN restoration. This original concept was based on material occlusal stress absorbance effect compared to a ceramic material, the hypothesis being that it would not encounter implant osseointegration by generating important peri-implant stresses when loading the restoration. In this case series, a 100% survival rate of the implants was observed after two years, which is promising. For the Vita Enamic crowns it was 90% due to the debonding of one crown from its titanium base.

The future perspectives of this thesis work include:

- Fatigue testing of the homogenous experimental PICN material compared to other CAD-CAM materials such as lithium disilicate glass-ceramic and other commercially available DF composites.
- Clinical studies involving the homogenous experimental PICN and the FG-PICN, particularly on the One-step No-prep protocol.
- Long-term evaluation of the One-Step No-prep and 1T1T protocols with Vita Enamic.
- Analysis of PICN and the opposing teeth wear in those clinical studies using the casted impressions and optical profilometry.

ASSOCIATED STUDENTS WORK

Bonding properties of innovative CAD-CAM composite biomaterials for dental prostheses.
Thesis presented by Marie Ebroin to obtain Master degree in Biomedical Sciences, 2016, University of Liège (ULiège).

The influence of silane on the bonding properties of CAD-CAM composite materials for dental prostheses.

Thesis presented by Laura Greimers to obtain Master degree in Biomedical Sciences, 2017, University of Liège (ULiège).

Propriétés des biomatériaux composites innovants pour CFAO des prothèses dentaires
"Properties of novel CAD-CAM composite materials for dental prosthesis"

Thesis presented by Francine Bopda to obtain Master degree in Biomedical Sciences, 2018, University of Liège (ULiège).

Bonding properties of new CAD/CAM restorative materials: The effect of different surface treatment methods on the bond strength.

Thesis presented by Emilie Gelin to obtain Master degree in Dental Sciences, 2016, University of Liège (ULiège).

Evaluation of the effectiveness of chemical bonding versus micromechanical retention with PICN and dispersed filler materials and evaluate silane aging sensitivity on the bond strength.

Thesis presented by Kelly Wechseler to obtain Master degree in Dental Sciences, 2017, University of Liège (ULiège).

A three-years prospective and retrospective study on single unit PICN CAD-CAM restorations « Etude prospective et rétrospective sur 3 ans de restaurations unitaires en PICN réalisées par technique CFAO directe »

Thesis presented by Tatiana Aissi and Marie Laurent to obtain Master degree in Dental Sciences, 2018, University of Liège (ULiège).

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- IADR Kulzer Travel Award, July 2018

POSTERS AND PRESENTATIONS

- 1 Bonding properties of third generation zirconia materials for monolithic restorations. Eldafrawy M, Bekaert S, Sadoun M, Mainjot A. European Dental Materials Conference (EDMC 2019), Brussels, August 2019.
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PROPERTIES AND APPLICATIONS OF POLYMER-INFILTRATED CERAMIC NETWORK MATERIALS

Computer-aided design and manufacturing (CAD-CAM) materials are gaining popularity in the field of restorative dentistry. Among recently developed materials are polymer-infiltrated ceramic network (PICN) materials, a sub-class of CAD-CAM composites, comprised of 75 vol% sintered glass-ceramic network that is secondarily infiltrated with monomers and polymerized under high-temperature and pressure; whereas the other sub-class of CAD-CAM composites, dispersed fillers (DF), consist of inorganic fillers classically incorporated by mixing in an organic matrix that is secondarily polymerized under high-temperature.

The first objective of this work was to use fracture mechanics, particularly the notchless triangular prism (NTP) specimen fracture toughness test to: 1) evaluate the interfacial fracture toughness (IFT) of a resin composite luting agent (RCLA) with PICNs, represented by an experimental and a commercial PICN (Vita Enamic), versus DF materials. Lithium disilicate glass-ceramic (IPS e.max CAD, EMX) was also tested as a gold standard for comparison. Samples were pretreated with hydrofluoric acid (HF) or gritblasting (GR), and the results were correlated with the developed interfacial area ratio (Sdr) of representative samples subjected to the same pretreatment procedures. 2) Evaluate the influence of silane on the IFT of RCLA with PICN and DF after HF and GR, and correlate the results with the Sdr and surface wettability of representative samples. The results showed that the IFT of PICNs was significantly superior to DF, and IFT of etched experimental PICN was significantly higher than EMX. In addition, there was a strong correlation between the IFT and the Sdr of the representative samples, PICNs demonstrating significantly higher surface roughness than DF when pretreated. These results highlighted the importance of material microstructure and then class (DF vs PICN) on the bonding interface performance. Etching of PICN led to the selective dissolution of the glass-ceramic at the surface, creating an original polymer-based honeycomb structure that promoted the micromechanical retention of RCLA. This micromechanical retention is enhanced by the application of silane, which allows RCLA penetration in surface roughness. On the other hand, IFT of DF was not influenced by silane, regardless of the surface pretreatment, which was less effective in creating surface roughness.

The second objective was to introduce of a functionally-graded (FG) PICN block as a biomimetic material for CAD-CAM prostheses. FG-PICN is characterized by a gradient of mechanical and optical properties, in which the surface properties resemble the hardness and modulus of enamel, while the deeper layers resemble those of dentin. The flexural strength values at the dentin-like layer was similar to glass-ceramic EMX and flexural load energy was significantly higher than EMX and monolithic zirconia. These properties could promote occlusal stress absorbance in treatment of patients with bruxism, such as worn dentition cases, and on implant restorations.

Finally, the third objective was to participate in two clinical studies performed following new treatment protocols developed with commercial PICN; a minimally invasive approach of worn dentition treatment with Vita Enamic bonded restorations; the "One-step No-prep" protocol and a novel approach for restoring a missing posterior tooth with immediate loading of an implant and a final crown made of PICN in a single visit; the "One-tooth One-time, 1T1T" protocol. The restorations success rate after 2 years was high (93.75% for the former and 90% for the latter), highlighting some edge chipping in the first study, and debonding from the ti-base in the second.

In conclusion, PICNs show a promising potential to be used in a variety of restorative applications, especially in minimally invasive dentistry, such as demonstrated in novel clinical applications of Vita Enamic. Indeed, *in vitro* studies highlighted the superiority of PICN, particularly the new PICN generation, to DF and lithium disilicate glass-ceramic in terms of IFT with RCLA. In addition, the original gradient of mechanical properties of the FG-PICN, opens the door for restorations with an improved bio-mechanical behavior compared to other CAD-CAM materials available on the market. Future perspectives include fatigue tests and clinical studies with experimental PICN materials.

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