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ORIGINAL ARTICLE

Effect of MDP agents on the bond strength of two self-adhesive resin cements to saliva-contaminated dentin substrates

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Abstract

This study investigated the influence of 10-methacryloyloxydecyl dihydrogen phosphate (MDP) agents and repeated saliva contamination on the microtensile bond strength (μTBS) of two self-adhesive resin cements (SARCs) bonded to dentin. Forty-eight sound human teeth were randomly divided into eight experimental groups, based on three factors: the type of SARC, the presence or absence of an MDP agent, and the presence or absence of multiple saliva exposures. The control group for each SARC received neither MDP agent nor multiple saliva contaminations. For each group, five teeth were sectioned to obtain 30–44 beams, followed by μTBS testing, fracture mode analysis, and scanning electron microscopy. Data were analyzed using one-way and two-way analysis of variance, Scheffé's test, and Weibull statistics. Under uncontaminated conditions, the application of MDP agents was found to significantly enhance the bond strength of both SARCs. Saliva contamination significantly reduced μTBS, regardless of the cement used. However, when MDP agents were applied prior to bonding, μTBS values remained comparable to those of uncontaminated controls, even after multiple saliva exposures. These results support the use of MDP agents to maintain adhesive integrity in the presence of repeated salivary contaminations, offering greater reliability in adverse clinical environments.

KEYWORDS

adhesion, dental tissue-conditioning, resin cement, saliva contamination

INTRODUCTION

Resin cements are increasingly being used to bond all-ceramic, metal, or composite indirect restorations due to their superior mechanical properties, and improved esthetics compared to conventional luting cements such as zinc phosphate and glass ionomers [1]. Resin cements include adhesive/multistep cements, self-adhesive (one-step) cements, and universal cements (which can be used as a self-adhesive or with an etch and rinse protocol) [2].

Self-adhesive resin cements (SARCs) were introduced in the early 21st century to simplify luting procedures while maintaining the performance of adhesive resin cements. The composition of SARC is based on carboxylic and/or

phosphoric acid methacrylate monomers. Upon mixing of the paste, the phosphoric acid groups react with the mineralized tissues and fillers of the luting material to form a bond, known as the cement reaction [3, 4]. At the same time, radical polymerization of the methacrylate monomers is initiated. As the material cures, the acid groups neutralize, changing the behavior of the material from hydrophilic to hydrophobic [3].

Several studies indicate that the bonding of SARCs relies predominantly on chemical interaction between the acidic functional monomers (e.g., phosphate or carboxylic acid groups) and calcium ions in the residual hydroxyapatite of dentin [4]. This is especially relevant given that SARCs exhibit limited ability to dissolve or penetrate the smear layer, and often do not form a distinct hybrid layer [5]. While micromechanical retention through hybrid layer formation and resin tag penetration is generally minimal, some reports have observed superficial hybridization or short resin tags under specific conditions [6]. Nonetheless, the literature consistently supports that chemical bonding rather than micromechanical interlocking plays the dominant role in the adhesion of SARCs to dentin [7].

Although SARCs have lower bond strength values than conventional multistep resin cements [8], a recent systematic review and meta-analysis study by Alavarenga et al. [9] reported that SARC can be recommended for cementing indirect single-tooth restorations, as they have a comparable risk of failure to conventional multistep cements.

However, performance may be compromised by contamination of the dental substrate, such as by saliva exposure [10]. Saliva contamination has long been a challenge in adhesive dentistry. Saliva is primarily composed of water, together with aggregates of macromolecules such as proteins, glycoproteins, enzymes, and inorganic particles such as calcium, sodium, and chloride, as well as organic particles such as urea, amino acids, fatty acids, and free glucose [11]. While surgical field isolation techniques (e.g., rubber dam, cotton rolls, saliva ejector) can prevent saliva exposure under favorable clinical conditions, certain situations in daily clinical dental practice make render their use impractical. These include patients with a gag reflex, noncompliant patients (e.g., those with mental or physical disorders), reluctant patients, or cases involving surgical sites near or even at the gingival margin and deep proximal restorations. The effect of saliva on bonding procedures has been extensively studied for direct restorations, but less so for indirect restorations and resin cements. Saliva can be detrimental for a number of reasons. The main problem is that water from saliva creates a wet surface, which hinders bonding. In addition, saliva contains hydrolytic enzymes (collagenase, amylase, peptidase, and esterase) that can degrade the collagen fibers and dentin proteins [12, 13]. In addition, saliva proteins form a thin organic film that reduces surface wettability and hinders the infiltration of resin monomers as well as the decalcification ability of the resin composite luting system [14–18]. Together, these factors can lead to restoration failure over time.

Methods for decontaminating saliva-contaminated substrates have been investigated [19]. However, bond strength results are dependent on factors such as the type of resin cement, the contaminated substrate, the timing of saliva contamination during the bonding procedure, and the cleaning methods used. In addition, the methodology of articles investigating the effect of saliva contamination on bonding procedures often does not accurately reflect real clinical scenarios [10, 20, 21]. While most articles on this topic introduce saliva contamination at a single step of the procedure, in real-life scenarios, moisture and saliva contamination may occur at every step of the bonding process when rubber dam application is ineffective or cannot be used.

Regarding cleaning methods, several approaches have been proposed, including mechanical cleaning (e.g., sonic scaling [22], rotary instruments [22], rotary brushes [22], and blasting [23, 24]), chemical cleaning (e.g., ethylenediaminetetraacetic acid EDTA or polyacrylic acid [23, 25] or sodium hypochlorite [26]), or the use of intraoral cleaners [27, 28]. Intraoral cleaners have been developed to restore the original bond strength of luting cements. Intraoral cleaners and primers based on 10-methacryloyloxydecyl dihydrogen phosphate (MDP) differ in composition and function. MDP primers contain the acidic monomer that chemically bonds to hydroxyapatite, forming stable MDP–Ca salts responsible for durable adhesion [29, 30]. Thus, previous studies have emphasized the importance of distinguishing between MDP-based primers and MDP-salt cleaners, as they serve distinct purposes. MDP primers promote chemical bonding with hydroxyapatite or metal oxides on zirconia surfaces via the phosphate group of MDP [31]. In contrast, MDP-salt (a reaction product of MDP with triethanolamine) acts as a surfactant-based cleaner. MDP-salt has a structure that contains both hydrophilic and hydrophobic groups, allowing it to effectively remove saliva through its surfactant action [32]. According to the literature, MDP-salt has been shown to be effective in removing contaminants from saliva-contaminated resin core foundations, zirconia [20, 33–37] and dentin tissue [38]. It also helps provide a surface more suitable for bonding with resin luting cements. While MDP-salt has shown potential to enhance bonding, it should not be conflated with the function of primers [39] and is discussed here solely as a cleaning agent. The aim of a cleaner or saliva decontaminant is to "clean" the surface from saliva contamination in order to optimize the bonding of the resin adhesive [36]. This distinction is essential when assessing their clinical roles.

However, to the best of our knowledge, the effect of MDP monomer and MDP-salt on the microtensile bond strength (μTBS) of SARC in the presence of multiple saliva contamination of the dentin substrate has not been investigated. Although recent reports [38, 40] have investigated the

influence of MDP-salt–based agents on the bond strength to dentin contaminated with saliva, both studies implemented a single contamination episode prior to bonding. In contrast, the present study examines the effect of multiple saliva contamination events on dentin surfaces prior to cementation with SARCs. This experimental design more closely reflects real-world clinical conditions, where repeated contamination during each step of cementation is common. By addressing this gap, the present study provides new insights into the behavior of SARCs and the protective role of MDP agents under more demanding conditions.

The aim of this study was to investigate and analyze the effect of two MDP containing components—a MDP primer (GCem One Adhesion Enhancing Primer, GC) for the SARCa (GCem One, GC) and a MDP-salt cleaner (Katana Cleaner, Kuraray) for the SARCb (Panavia SA, Kuraray)—on the μTBS of these SARCs applied to the dentin surface in both favorable (no saliva exposure) and unfavorable (multiple saliva exposures) clinical situations.

The four null hypotheses tested were that the saliva contamination and the MPD-monomer–based primer have no effect on the μTBS of the SARCa to dentin; and that saliva contamination and the MPD-salt cleaner have no effect on the μTBS of the SARCb to dentin.

MATERIAL AND METHODS

The materials used in this study are listed in Table 1 [20, 21, 36, 41–47].

Specimen preparation

Two commercially available SARCs, SARCa (GCem One, GC), and SARCb (Panavia SA, Kuraray) with their respective MDP primer (GCem One Adhesion Enhancing Primer, GC) and MDP-salt cleaner (Katana Cleaner, Kuraray) were selected for this study. Table 1 summarizes the details regarding the materials used, while samples preparation and characterization are described in the following sections.

A total of 48 intact, caries free, extracted human molars obtained from the dental service of the Hospital Pitié Salpêtrière, from patients aged between 18 and 60 yr, were used in this study. All experiments were conducted in accordance with the tenets of the Declaration of Helsinki. All teeth were collected following the informed oral consent of all patients, in accordance with the ethical guidelines laid down by French law and the specific authorization of the Faculty of Dentistry of Paris Cité University (n°DC-2009-927, Cellule Bioethique DGRI/A5). Teeth were cleaned with a curette to remove soft tissue and bone tissue and have been stored in 0.5% chloramine-T in a temperature-controlled environment of 4°C and were used within 3 months of extraction. Enamel and the upper part of the teeth were removed by wet grinding with a polisher (ESC300 GT, Escil) using silicon carbide paper (220 grit, followed by 600 grit) to expose a flat dentin surface.

The saliva contamination protocol was performed according to Bolme et al. [48]: saliva was fresh, whole, unstimulated human saliva collected from the first author (A.F.) at the same time each morning. The saliva was used within 30 min of collection on the same day. The saliva was applied to the dentin surface with a microbrush for 10 s. The surface was then air dried thoroughly for 5 s. This saliva contamination was repeated after each step of the bonding procedure (Figure 1).

The SARC was applied in 2 mm horizontal layers using a transparent matrix strip roll (Hawe Striproll, Kerr) to obtain a height of 6 mm. Light curing was performed on each layer using an LED light (Valo Cordless, Ultradent) with a power of 890 mW/cm^2. A universal adhesive system (Clearfil Universal Bond Quick, Kuraray) was used with the SARCb.

Forty teeth were used for microtensile testing. The teeth were randomly assigned to eight test groups and received treatments as follows (five teeth per group):

(i) SARCa only (n_{beams} = 43): air dry for 5 s, application of SARCa with an auto-mix tip (provided by manufacturer), photopolymerization for 10 s.
(ii) SARCa + MDP primer (n_{beams} = 43): air dry for 5 s, application of MDP primer for 10 s followed by air dry for 5 s, application of SARCa with an auto-mix tip (provided by manufacturer), photopolymerization for 10 s.
(iii) SARCa + saliva (n_{beams} = 40): application of saliva for 10 s with microbrush, air dry for 5 s, application of SARCa with an auto-mix tip (provided by manufacturer), photopolymerization for 10 s.
(iv) SARCa +saliva + MDP primer (n_{beams} = 40): application of saliva for 10 s with microbrush, air dry for 5 s, application of MDP primer for 10 s followed by air dry for 5 s, application of saliva for 10 s with microbrush, air dry for 5 s, application of SARCa with an auto-mix tip (provided by manufacturer), photopolymerization for 10 s.
(v) SARCb only (n_{beams} = 34): air dry for 5 s, application of universal adhesive system and rub it for 20 s and air dry for 5 s, application of SARCb with an auto-mix tip (provided by manufacturer), photopolymerization for 10 s.
(vi) SARCb + MDP cleaner (n_{beams} = 36): air dry for 5 s, application of MDP-salt cleaner for 10 s and water rinse until the color of the product is removed and followed by air dry for 5 s, application of universal adhesive system and rub it for 20 s and air dry for 5 s, application of SARCb with an auto-mix tip (provided by manufacturer), photopolymerization for 10 s.

TABLE 1 The commercial resin-based cement used and their primer/cleaner [9, 10, 16–23].

Abbreviated name	Commercial name (lot number)	Manufacturer	Composition
MPD Primer	GCem one adhesive enhancing primer (2308244)	GC	Ethyl alcohol (25-50%), 2-hydroxy-1,3 dimethacryloxypropane (10%–20%), 10-methacryloyloxydecyl dihydrogen phosphate (5%–10%), butylated hydroxytoluene (1%–2.5%), vanadyl acetylacetonate, 4-methacryloxyethyl trimellitate anhydride, water. pH: 1.5
SARCa	GCem one self-adhesive resin cement (2305171)	GC	Paste 1 2-hydroxy-1,3 dimethacryloxypropane, urethane dimethacrylate, 6-*tert*-butyl-2,4-xylenol diphenyl(2,4,6-trimethylbenzoyl), phosphine oxide, dimethacrylates, 4-methacryloxyethyl trimellitate anhydride, vanadyl acetylacetonate, phosphoric ester monomers, fluoroaluminosilicate, glass, TiO_2 (Filler: 65 wt %, average particle size of 1.7 μm), camphorquinone. Paste 2 urethane dimethacrylate, 2-hydroxy-1,3 dimethacryloxypropane, methacryloyloxydecyl dihydrogen phosphate, α,α-dimethylbenzyl hydroperoxide, 6-*tert*-butyl-2,4-xylenol.
SARCb	Panavia SA Cement Universal (1A0024)	Kuraray	Paste 1 10-methacryloyloxydecyl dihydrogen phosphate, bisphenol A diglycidylmethacrylate, triethyleneglycol dimethacrylate, hydrophobic aromatic dimethacrylate, 2-hydroxymethacrylate, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, peroxide, catalysts, pigments. Paste 2 hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated barium glass filler, surface treated sodium fluoride, accelerators, pigments. Total volume fillers 40% (particles size 0.2–20 μm)
Universal adhesive system	Clearfil Universal Bond Quick (5P0409)	Kuraray	Bisphenol A-glycidyl methacrylate (10%–25%), ethanol (10%–25%), 2-hydroxyethylmethacrylate (2.5%–10%), 10-methacryloyloxydecyl dihydrogen phosphate, hydrophylic amid monomers, sodium fluoride, dl-camphorquinone, colloidal silica.
MDP-salt cleaner	Katana Cleaner (7E0047)	Kuraray	Water, polyethyleneglycol, 10-methacryloyloxydecyl dihydrogen phosphate, triethanolamine, stabilizer, dyes, accelerator. pH:4.5

(vii) SARCb + saliva ($n_{beams} = 30$): application of saliva for 10 s with microbrush, air dry for 5 s, application of universal adhesive system and rub it for 20 s and air dry for 5 s, application of saliva for 10 s with microbrush, air dry for 5 s, application of SARCb with an auto-mix tip (provided by manufacturer), photopolymerization for 10 s.

(viii) SARCb + saliva + MDP cleaner ($n_{beams} = 44$): application of saliva for 10 s with microbrush, air dry for 5 s, application of MDP-salt cleaner for 10 sand water rinse until the color of the product is removed and followed by air dry for 5 s, application of saliva for 10 s with microbrush, air dry for 5 s, application of universal adhesive system and rub it for 20 s and air dry for 5 s, application of saliva for 10 s with microbrush, air dry for 5 s, application of SARCb with an auto-mix tip (provided by manufacturer), photopolymerization for 10 s.

The control groups consist of SARCa only and SARCb only. After SARC application procedure, the teeth were stored overnight at 100% air humidity before being cut into 30–44 rectangular beams (1 × 1 × 10 mm) per group using a saw (Isomet, Buehler) under water irrigation, the dimensions of which were measured using a digital caliper (Mitutoyo) before testing. The prepared beams were stored in water at 37˚C for 24 h to simulate ageing in the oral environment.

Microtensile bond strength

Bond strength was characterized in this study using the μTBS method (ISO/TS 11,405:2015). Each specimen was attached to a fixture with cyanoacrylate adhesive (Cyanboard). They were then subjected to a tensile stress in a computer-controlled (Bluehill, Instron) universal testing machine (Instron model 5943) at a crosshead speed of 0.5 mm/min, until fracture.

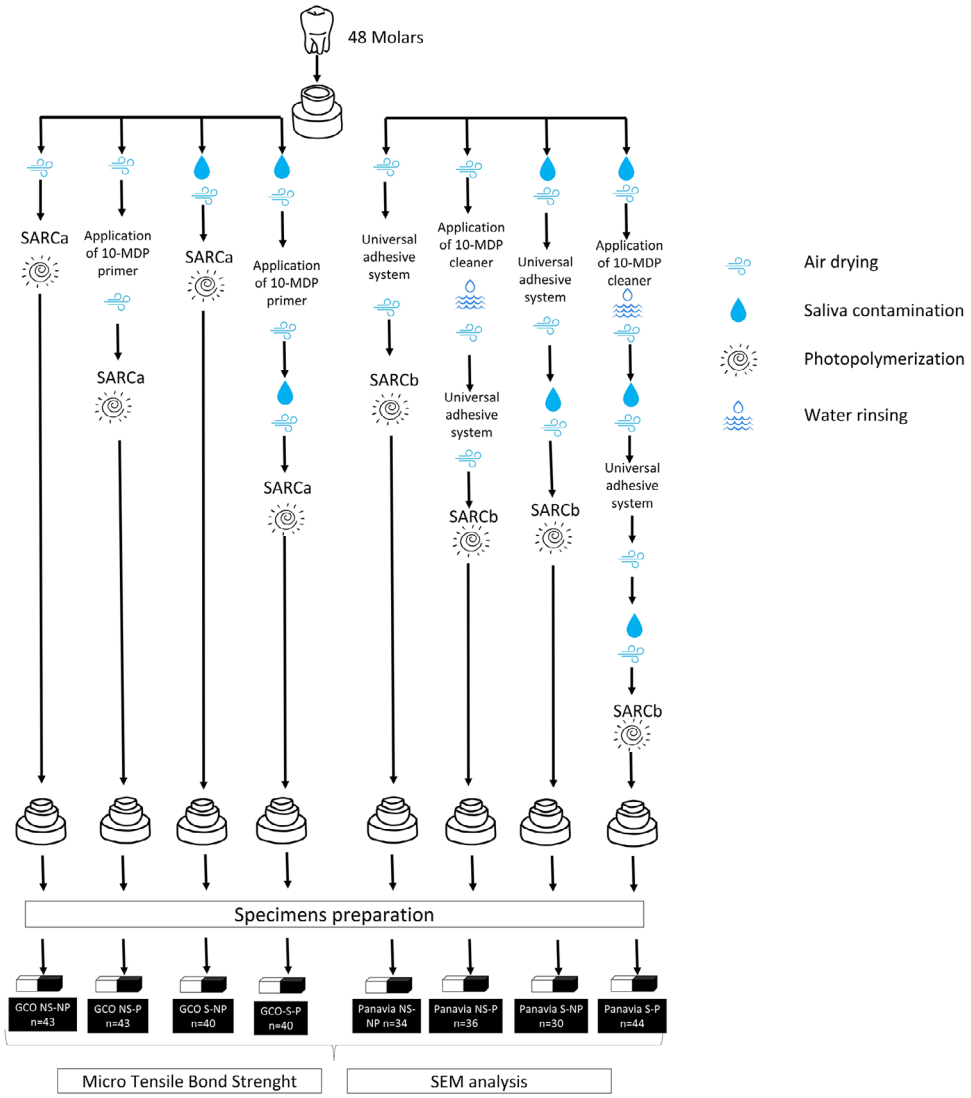

FIGURE 1 Experimental design and bonding protocols. Saliva contamination at various stages of the bonding process. Groups SARCa only and group SARCb only serve as controls.

The failure load (N) was measured and the μTBS (MPa) was calculated.

The interface failure mode was analyzed by observing the specimens under a stereomicroscope (Leica S9D, Weltzar) at ×50 magnification, and classified into one of the following three categories: cohesive fracture (failure occurred in the dentin or in the bonding material), adhesive fracture (failure occurred in the interface) and mixed fracture (failure occurred partially in the bonding material and partially in the interface) [49].

Scanning electron microscopy analysis

One tooth from each group was retained for scanning electron microscopy (SEM) analysis. They were re-embedded after application of the bonding material. A new longitudinal cut with a saw (Isomet, Buehler) under water irrigation was made in the center of the tooth to obtain a cross-section of the interface, resulting in two square-shaped samples, each approximately 1 × 1 cm in size. The surface was polished to 4000 grit. The samples were then dehydrated in a pure alcohol bath for 1 h. They were then metallized for observation using a SEM (JSM-6400, JEOL) at ×2000 magnification.

Statistical analysis

The results of μTBS for each material were analyzed using both one-way and two-way analysis of variance (ANOVA) ($\alpha = 0.05$), followed by Scheffé's post hoc test for multiple comparisons ($\alpha = 0.05$), performed with SPSS 21 software (IBM). Although multiple beams were obtained from the

same tooth, each beam was treated as an independent unit of analysis, as commonly reported in microtensile testing protocols. A total of five teeth per group were used to ensure biological relevance and variability, in line with recommendations by Armstrong et al. [50]. The potential effect of intra-tooth interdependence is acknowledged as a limitation of the study.

One-way ANOVA was used to compare the different bonding protocols within each SARC group, treating each protocol as a distinct treatment level. Two-way ANOVA was used to analyze the individual and interaction effects of two factors (saliva contamination and use of MDP primer or cleaner) on the μTBS results for each SARC. For pretest failure specimens, a bond strength value of 0 MPa was used in the statistical analysis.

As the Weibull distribution cannot handle zero values, pretest failures were replaced with a random value between zero and the lowest measured value in the respective group, and the resulting Weibull parameters were obtained [51].

Weibull statistical parameters were calculated for the bond strength data. The description of the Weibull distribution is given by:

$$P_f = 1 - e^{-\left(\frac{t}{\eta}\right)}$$

where P_f is the fracture probability, defined by the relation estimated using Bernard's estimation:

$$Pf = \frac{K - 0.3}{N + 0.4},$$

where K is the rank of strength from least to greatest, N is the total number of specimens in the sample, β is the shape parameter (Weibull modulus), t is bond strength, and η is the scale parameter or characteristic bond strength (63.2% probability of failure) [52, 53]. Weibull modulus, characteristic strength and strength at 10% probability of failure were obtained using the Weibull statistics option in Excel (Microsoft).

RESULTS

The results of the μTBS measurements are summarized in Table 2, together with the results of the statistical analysis. For SARCa, μTBS values ranged from 0 to 50.06 ± 16.25 MPa depending on the presence of MDP primer conditioning and saliva contamination. MDP primer conditioning significantly increased the bond strength of SARCa with a mean μTBS value of 50.06 ± 16.25 (95% CI = 45.06, 55.06) MPa for SARCa + MDP primer and 49.76 ± 22.85 (95% CI = 42.55, 56.97) for SARCa + saliva+ MDP primer compared to 4.39 ± 9.26 (95% CI = 1.54, 7.24) MPa of SARCa only (control group) and 0 MPa for SARCa + saliva ($p < 0.05$).

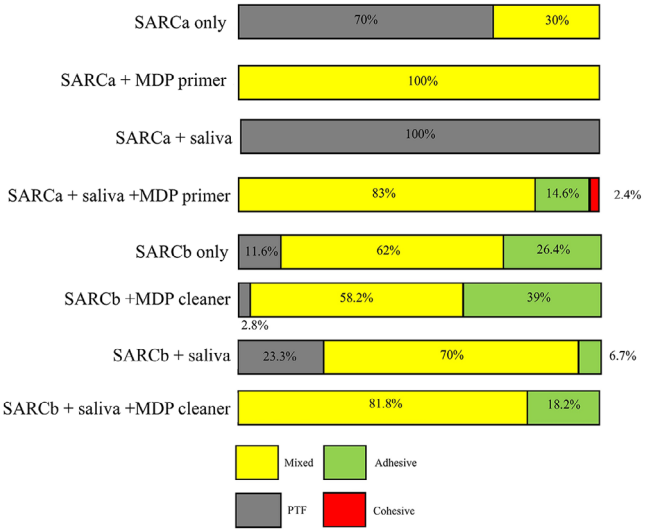

FIGURE 2 Fracture analysis of specimens subjected to tensile bond strength test. PTF, pretest failure.

For SARCb, the highest significant mean μTBS was observed in SARCb +MDP cleaner (21.57 ± 13.02 MPa; 95% CI = 17.18, 25.96) ($p < 0.05$), while SARCb + saliva had the lowest significant value (5.91 ± 4.67 MPa; 95% CI = 4.17, 7.65). Thus, despite saliva exposure, the application of MDP cleaner in SARCb + saliva + MDP cleaner (12.57 ± 6.83 MPa; 95% CI = 10.50, 14.65) maintained μTBS values comparable to those of SARCb only (control group) (12.44 ± 7.53 MPa; 95% CI = 9.82, 15.06).

For SARCa, two-way ANOVA showed that MDP primer (yes/no) was the only statistically significant factor ($p < 0.05$), with no apparent interaction between saliva contamination (yes/no) and MDP (yes/no) ($p > 0.05$). In contrast, for SARCb, two-way ANOVA indicated that both MDP (yes/no) and saliva (yes/no) were statistically significant factors for bond strength ($p < 0.05$), but no interaction between saliva (yes/no) and MDP (yes/no) was observed ($p > 0.05$).

Therefore, the second, third, and fourth null hypotheses were rejected, while the first null hypothesis was not rejected.

The failure modes are illustrated in Figure 2. No cohesive fracture was observed, except for SARCa + saliva + MDP primer. The failures modes were mainly mixed for the two SARCs (Figure 2). It should be noted that groups subject to pretest failures were essentially without MDP agent application, thus 100% of specimens of SARCa + saliva had pretest failures, while specimens of SARCa only and SARCb + saliva had, respectively, 70% and 23% pretest failures. All occurred during the cutting of the specimens.

The Weibull plot in Figure 3 shows the reliability of the μTBS of each material. The use of MDP primer resulted in an increased Weibull modulus for the SARCa, indicating a more

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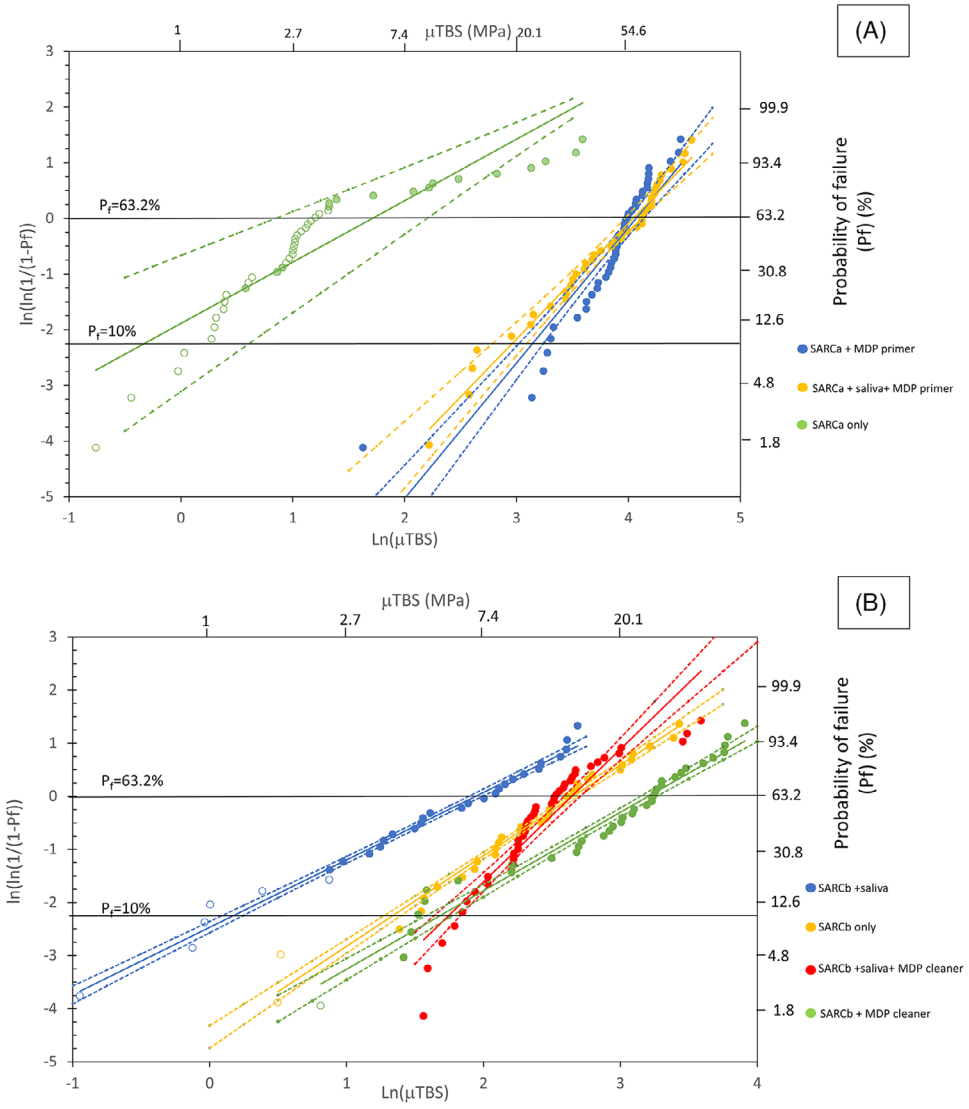

FIGURE 3 Weibull plot for SARCa (A) and SARCb (B). Dotted lines represent the 95% confidence limits for each group. True values are represented by full circles, while each value representing pre-test failures are represented by empty circles.

reliable bond. For the SARCb, the use of MDP cleaner in the presence of saliva contamination resulted in a more reliable bond.

SEM analysis of the SARCa–dentin interface showed that the adhesive interface was continuous under uncontaminated conditions and without the application of an MDP primer, with no resin tag penetration into the dentinal tubules (Figure 4A). In contrast, when the MDP primer was applied, clear resin tag infiltration into the tubules was observed (Green arrow Figure 4B), indicating enhanced micromechanical retention. This side-by-side comparison highlighted the essential role of MDP primer application in enhancing SARCa–dentin adhesion. Moreover, SARCa + saliva showed a gap between dentin and resin cement (blue arrow Figure 4C), whereas for SARCa + saliva + MDP primer intratubular resin penetration can be visualized (green arrow Figure 4D). Saliva residues on the dentin surface were also observed with SARCa + saliva + MDP primer (red arrow Figure 4D).

For SARCb, intratubular penetration of the resin cement (green arrows) was observed in both the SARCb-only and SARCb + MDP cleaner groups (Figure 5A,B). Voids within the resin cement (white arrows) were also noted under both uncontaminated and saliva-contaminated conditions (Figure 5B–D). Additionally, degradation of the adhesive layer was evident in the SARCb + saliva group (blue arrow, Figure 5C).

DISCUSSION

This study investigated the effects of MDP agents on the bond strength between two SARCs in both favorable (no saliva exposure) and unfavorable (multiple saliva

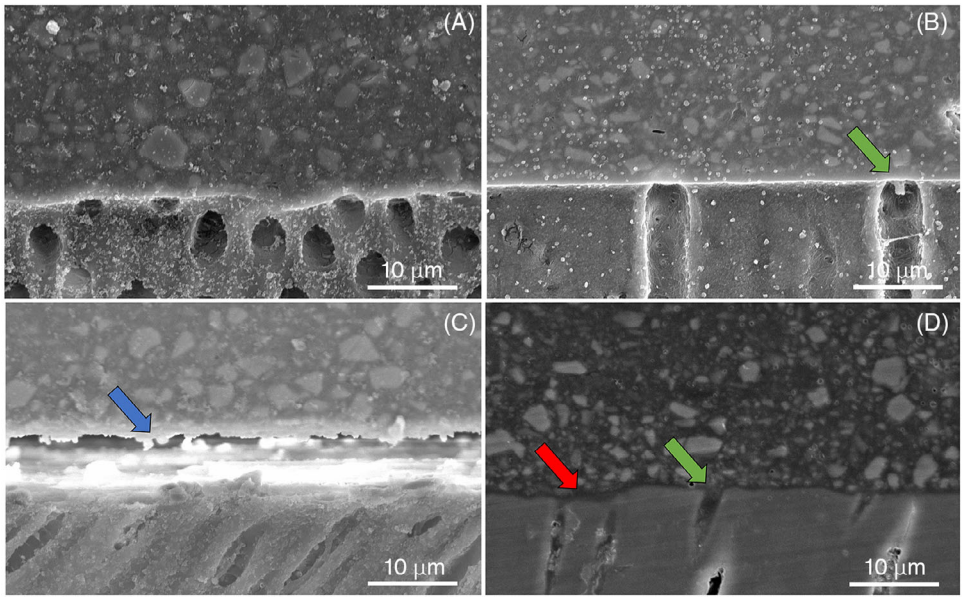

FIGURE 4 Scanning electron microscope images (×2000 magnification) of dentin substrates. (A) SARCa only, (B) SARCa + MDP primer, (C) SARCa + saliva, and (D) SARCa + saliva + MDP primer. Green arrow shows intertubular resin cement penetration. Red arrow shows saliva residue on the dentine surface. Blue arrow indicates gap between dentin and resin cement.

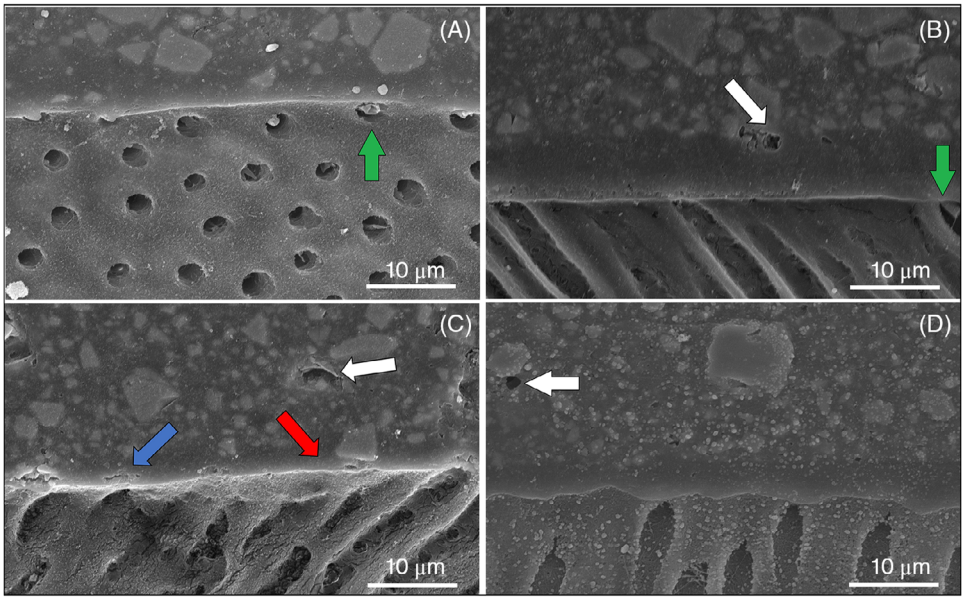

FIGURE 5 Scanning electron microscope images (×2000 magnification) of dentin substrates. (A) SARCb only, (B) SARCb + MDP primer, (C) SARCb + saliva, and (D) SARCb + saliva + MDP primer. Red arrow indicated adhesive layer. White arrow shows voids. Blue arrow shows degradation of the adhesive layer. Green arrow shows intertubular resin cement penetration.

contamination) clinical situations on dentin surfaces. The results showed that the first null hypothesis, which posited that saliva contamination does not affect the μTBS of the SARCa on dentin, could not be rejected. In contrast, the second, third, and fourth null hypotheses were rejected. These stated that the MDP primer does not influence the μTBS of the SARCa, that saliva contamination and the MPD-salt cleaner have no effect on the μTBS of the SARCb on dentin.

One of the major disadvantages of SARCs is their poor wettability due to their high viscosity, which limits infiltration into tooth hard tissue [54, 55]. Furthermore, it has been reported in the literature that no hybrid layer or resin tag for-

mation was observed when SARCs were used alone [56]. We also observed a lack of resin tag formation using SEM and 26% of adhesive failures in samples with SARCb without MDP cleaner, confirming that the bond is weak when SARCb is used alone. The presence of voids within the material was also observed (Figure 5).

Under favorable clinical conditions (without saliva contamination), ANOVA results showed for SARCa that the application of MDP primer resulted in a statistically significant increase in μTBS. This SARCa has been little studied in the literature. For example, Atalay et al. [55] reported highest shear bond strength to dentin in the presence of MDP primer. Dentin is a hybrid tissue that is hydrophilic due to its low mineral content and high organic compound ratio. The higher μTBS values observed with the use of MDP primer in favorable clinical conditions may be attributed to a "touch-cure" mechanism occurring at the dentin–cement interface. This reaction is likely facilitated by the presence of redox-active compounds in the primer formulation. Specifically, the MDP primer has been reported to contain vanadyl acetylacetonate, a compound capable of reacting with peroxide-based initiators. In the case of the SARCa, the initiator α,α-dimethylbenzyl hydroperoxide is found in Paste 2 (Table 1) and could undergo redox activation in the presence of vanadyl acetylacetonate, thereby initiating radical polymerization [57]. A previous study reported that "touch-cure" resulted in higher degree of conversion at the interface between the resin cement (Panavia V5, Kuraray) and dentin in the presence of the MDP primer (Panavia V5 Tooth Primer, Kuraray), which contains a vanadyl compound. The study also found higher bond strength [58], which is consistent with the results shown in Table 2. Significantly higher μTBS observed in groups with primer/cleaner may be due to MDP. Previous reports have shown that MDP monomer interacts with Ca ions in hydroxyapatite through carboxyl and phosphate groups and can copolymerize with methacrylate groups of resin monomers [59]. It can also bind ionically to the calcium of hydroxyapatite to form an insoluble MDP–Ca salt at the interface. MDP monomer can also form a thick acid–base-resistant zone, which is believed to protect the adhesive interface from the acidic oral environment [60]. This enhances the adhesive's ability to absorb water and penetrate the demineralized dentin matrix, promoting better resin infiltration and surface adaptation. The extremely stable chemical bonds between MDP and hydroxyapatite contribute to long-lasting adhesive restorations, reducing the risk of bond degradation, marginal discoloration, and postoperative sensitivity. For exposed collagen, this salt fills the water-rich, resin-poor regions within the hybrid layer, preventing invasion by exogenous proteases and endogenous matrix metalloproteinases (MMPs), thus providing mechanical protection for the underlying demineralized collagen. MDP–Ca salts protect the exposed collagen fiber network by providing mechanical protection and reducing collagenase activity [59]. In addition, they interact with MMP-9 to disrupt its recognition and binding to collagen, thereby inhibiting enzymatic activity. MDP-treated demineralized dentin collagen showed reduced hydroxyproline release compared to the negative control, indicating improved resistance to exogenous enzymes and a direct inhibitory effect against MMPs [59].

An increase in μTBS for SARCb was also observed in the present study, which is consistent with the findings reported by Çeliksöz et al. [38], where the use of an MDP-based cleaner enhanced the bond strength of SARCb to uncontaminated dentin. Unlike the MDP primer, the manufacturer has not disclosed the chemical composition of the "accelerator" contained in MDP-salt–based cleaners, and no published chemical analyses have confirmed the presence of vanadyl acetylacetonate or a similar compound. Consequently, while the possibility of a "touch-cure" effect involving MDP-salt cleaner and SARCb cannot be ruled out, this remains a hypothesis that should be investigated in future studies. Overall, the results of the present study, along with those of Çeliksöz et al. [38], suggest that MDP-salt–based cleaners may have a beneficial effect on bond strength to uncontaminated dentin substrates.

Under unfavorable conditions (with multiple saliva contaminations), the results for SARCb demonstrated that saliva contamination weakened its bond strength to dentin surfaces, confirming findings in the literature. On dentin, it has been well established that salivary contamination alters the surface pH, as demonstrated by Hiraishi et al. [61]. This alteration negatively affects bonding effectiveness, as confirmed by both bond strength measurements [10, 48, 62] and biochemical assessments such as protein adsorption assays [63]. Similarly, on zirconia surfaces, the adsorption of salivary proteins forms a biofilm that impairs surface energy and disrupts adhesive interactions [64, 65]. Further reports demonstrated that this protein layer reduces the effectiveness of bonding [66, 67]. Specifically, Pitta et al. [68] reported a significant reduction in the bond strength of SARCb to zirconia following saliva contamination. These findings underscore the critical importance of implementing effective surface cleaning strategies—such as the application of MDP-containing cleaners or MDP salts—to restore the chemical reactivity of contaminated substrates prior to cementation [37].

Furthermore, our results indicated that the bond strength increased when primer/cleaner was used for both SARCs in the presence of saliva contamination. Although the use of MDP primer with SARCa is optional according to the manufacturer, the present results demonstrated that its application led to a substantial increase in bond strength and reliability (μTBS and Weibull modulus). These findings support the importance of using the primer to optimize SARCa performance. These results for the SARCa may be due to the pH level of 1.5 in MDP. This is consistent with previous data

TABLE 2 Microtensile bond strength values (mean ± SD, MPa) of SARCa and SARCb. Also given is the Weibull modulus, and the microtensile bond strength (μTBS) values at which 10% (PF10), respectively 63.2% (PF63.2) of the specimens fractured.

Experimental groups	μTBS (in MPa) mean ± SD [95% CI]	Weibull modulus (β shape) [95% CI]	PF10 [95% CI]	PF63.2 (η) [95% CI]
SARCa only	4.39 ± 9.26[b] [1.54, 7.24]	1.10 [0.93, 1.26]	0.72 [0.53, 0.91]	5.55 [4.53, 6.57]
SARCa + MDP primer	50.06 ± 16.25[a] [45.06, 55.06]	2.43 [2.11, 2.75]	23.14 [20.25, 25.89]	58.40 [52.04, 68.03]
SARCa + saliva	0	–	–	–
SARCa + saliva + MDP primer	49.76 ± 22.85[a] [42.55, 56.97]	2.08 [1.99, 2.18]	19.26 [18.49, 20.00]	56.83 [56.77, 56.88]
SARCb only	12.44 ± 7.53[b] [9.82, 15.06]	1.71 [1.62, 1.80]	3.81 [3.56, 4.04]	14.23 [13.59, 14.94]
SARCb + MDP cleaner	21.57 ± 13.02[a] [17.18, 25.96]	1.48 [1.38, 1.58]	5.37 [4.85, 5.88]	24.65 [23.17, 26.34]
SARCb + saliva	5.91 ± 4.67 [4.17, 7.65]	1.27 [1.21, 1.33]	1.19 [1.09, 1.27]	6.94 [6.67, 7.34]
SARCb + saliva + MDP cleaner	12.57 ± 6.83[b] [10.50, 14.65]	2.50 [2.21, 2.79]	5.73 [5.15, 6.24]	14.09 [13.34, 14.92]

Note: Identical superscript letters in a column indicate no significant differences between materials within a material category (one-way analysis of variance followed by Scheffe test, $\alpha = 0.05$).

obtained after the application of acids such as phosphoric acid, which is effective in removing saliva from ceramic surfaces [69]. Acids can penetrate the saliva film and etch the adherent surface, freeing it from the saliva. In the Hayashi et al. [20] study, more favorable shear bond strength results were observed when acidic solutions were used, suggesting that acidic etching materials are effective in removing saliva components [17, 70]. The results of this study also suggest the effectiveness of the MDP primer as a saliva decontaminant.

For the SARCb group, the application of the MDP cleaner statistically significantly improved bond strength compared to conditions under repeated saliva contamination without MDP cleaner use. Notably, it helped maintain μTBS values comparable to the uncontaminated SARCb control group.

These findings are consistent with those reported by Çeliksöz et al. [38], although their study involved only a single instance of saliva contamination. In contrast, the present study included three consecutive contamination steps (SARCb + saliva + cleaner group), highlighting the persistent cleaning efficacy of MDP-salt even after multiple saliva exposures.

This study is original because the contamination procedure was designed to closely mimic clinical scenarios where isolation is ineffective or absent. Unlike previous studies [14], where saliva exposure occurred only once, typically before primer application, we used a protocial where saliva was applied at each step. Furthermore, the results of this study highlight the persistent effect of MPD, as saliva was applied before and after the application of MPD.

The μTBS test used in this study has great discriminatory potential. Although the μTBS test procedure is labor intensive and technique sensitive, it remains a recognized adhesion test [70]. The relatively high standard deviations across all groups could be attributed to this sensitivity, supported by the observation of high pretest failure rates in the contaminated groups, where a significant proportion of samples did not survive the sectioning process. This is often a result of manual handling, which can stress the already fragile adhesive layer [60]. Although the μTBS test is known for its sensitivity, it can yield consistent and reproducible results when carefully performed under controlled conditions. Several studies have reported low coefficients of variation, indicating the reliability of the technique [21]. However, in the present study, higher variability was observed in some groups which can be attributed to both the inherent variability of SARC bonding [71–73] and the added complexity of simulating salivary contamination [54, 74]. In the SARCa only group, approximately 70% of the specimens exhibited adhesive failure at the interface, resulting in zero bond strength. This generated a highly skewed distribution with a low mean and large standard deviation. Such patterns are expected in μTBS testing, particularly under contaminated or suboptimal conditions. In fact, a coefficient of variation between 20% and 50% is commonly reported in the literature, even under favorable circumstances, due to the test's sensitivity to factors such as dentin heterogeneity, specimen preparation, and technique [28, 49, 60, 75]. This should be considered a methodological limitation when interpreting μTBS data under such conditions. Despite the observed variability, statistically significant differences were confirmed between groups, supporting the validity and clinical relevance of our findings. Another limitation of this study is its in vitro

design, which does not simulate the actual stresses found in the oral environment. Future research should focus on the longevity and predictability of the results described in this study.

Overall, the results of this study suggest that the use of MDP agents can provide moisture tolerance. In situations where multiple exposures to saliva are expected, MDP agents can be used to achieve sufficient adhesion to dentin compatible with good clinical results.

AUTHOR CONTRIBUTIONS

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

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