

INFLUENCE OF HIGH-PRESSURE POLYMERIZATION ON MECHANICAL PROPERTIES OF DENTURE BASE RESINS

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ABSTRACT

Purpose: The purpose of this study was to study the influence of high-pressure (HP) polymerization on the mechanical properties of denture base PMMA resins compared with conventional thermopolymerization and PMMA discs for digital dentures.

Materials and Methods: Three groups of blocks were prepared: Probase Hot (Ivoclar Vivadent, Lichtenstein) conventionally heat polymerized at 100°C, Probase Hot heat polymerized at 100°C under HP (200 MPa) and Ivobase CAD (Ivoclar Vivadent, Lichtenstein). Samples for mechanical/physical ($n = 30$) and samples for viscoelastic ($n = 10$) characterizations were cut from the blocks. Flexural strength (σ_f), elastic modulus (E_f), hardness, density (ρ), flexural deformation at maximal flexural stress, flexural load energy (U_f) and viscoelastic properties (E' , E'' , $\tan\delta$, T_g) were analyzed using one-way ANOVA ($\alpha = 0.05$), Scheffé multiple means comparisons ($\alpha = 0.05$) and Weibull statistics (for σ_f). SEM images of the fractured surfaces were obtained.

Results: E_f , E' , E'' and density of HP polymerized Probase hot were significantly higher than conventional heat polymerized Probase Hot, whereas T_g was significantly lower and σ_f , $\tan\delta$, hardness, flexural deformation at maximal flexural stress, U_f were not significantly different. The highest values for σ_f , flexural deformation at maximal flexural stress, U_f and Weibull modulus were obtained with Ivobase CAD.

Conclusion: HP polymerization does not significantly increase the mechanical properties of denture base resins.

KEYWORDS: Prosthetic dentistry/prosthodontics; PMMA denture base; high pressure polymerization; materials science(s); mechanical properties; viscoelastic properties.

Fracture is a frequent cause of failure of acrylic resin removable complete dentures, affecting around 30% of complete dentures,¹ as well as 9% to 15% of implant overdentures.^{2,3} In order to strengthen the prosthesis, cast metal reinforcement was shown to be the most effective.⁴ Indeed, compared to other prosthetic materials, the mechanical properties of poly (methylmethacrylate) (PMMA) resins are limited and their fracture toughness is significantly lower than metal alloys or ceramic materials such as zirconia.^{5,6} However, PMMA resins present other important advantages such as the ease of fabrication and repair, relining possibility, affordable cost and good appearance.⁷

Many approaches have been undertaken to improve the mechanical properties of materials used for removable complete dentures. The incorporation of fillers, such as carbon,⁸ glass or polyamide fibers,^{4,9-13} into the PMMA matrix was described. However, these materials are difficult to polish and are not adapted to milling processes. Other methods were explored such as incorporation of inorganic particles^{4,13,14} or changes of monomers formulation with crosslinking agents.¹⁵ On the other hand, advances in computer-aided design and manufacturing (CAD/CAM) processes and materials have offered a new landscape for dentures. These new developments may help offer cost-effective and straightforward treatments by improving overall accuracy and thus patient's satisfaction.¹⁶⁻²⁰ Compared to traditional pack and press, pour and injection techniques, CAD/CAM processes increase reproducibility, reduce time of fabrication and improve denture fit and retention.¹⁶ In fact, traditional processes induce distortion due to volume contraction during polymerization of the acrylic resins (0.45% to 0.9% linear distortion),²¹ while digital complete dentures are milled from prepolymerized block of acrylic resins, avoiding the polymerization shrinkage issue. Other benefits during laboratory steps have been observed, such as less artificial teeth movements during manufacturing compared to conventional process (pack and press)²² and digital archiving allows for faster and easier production of duplicate protheses.¹⁶ Finally, one of the major advantages of CAD/CAM processes is access to high performance materials. A recent study has shown that CAD/CAM PMMA discs (Ivoclar Base, Ivoclar) had 13% to 50% higher fracture toughness than classical heat polymerized PMMA (Probase Hot and Ivoclar, Ivoclar).⁵ Indeed, manufacturing of CAD-CAM blocks reduces the presence of voids and flaws,²³ and allows for the use of high-performance polymerization processes, which can improve material resistance. In particular, previous studies have shown that the use of high-temperature (180°C) combined with high-pressure (250 MPa) (HP) polymerization allows for a significant improvement in the mechanical properties of dimethacrylate-based CAD/CAM composites (Gradia GC, Grandio Voco, EsthetX Denstply, VitaVM LC Vita),²⁴ notably increasing the material flexural strength by 53.54% to 89.16%. Thus, a decrease in free monomer release and a better degree of conversion and crosslinking of the HP polymerized urethane dimethacrylate (UDMA) polymer were observed.^{25,26} These results could be explained by HP effects, such as reduced intermolecular distances, flaws, free volume, volumetric contraction, and internal stress.²⁷⁻²⁹ However, the influence of HP on mechanical and viscoelastic properties of PMMA has not yet been studied.

The objective of this work is to study the influence of HP polymerization on the mechanical properties of denture base PMMA resins in comparison with conventional heat polymerized PMMA and CAD/CAM PMMA discs for digital dentures. The null hypotheses tested were that the mechanical,

physical and viscoelastic properties of HP PMMA resins were not different from (1) those of conventional heat polymerized PMMA resins; and from (2) those of Ivobase CAD.

Materials and methods

A commercially available heat polymerized denture base PMMA resins, Probase Hot (Ivoclar Vivadent Inc., Lichtenstein), was selected for this study. Ivobase CAD (Ivoclar Vivadent Inc., Lichtenstein), a commercially available resins disc for CAD/CAM, was also included for comparison. Table 1 summarize details regarding the materials used.

Table 1. Details of materials used

Material	Manufacturer	Group	Composition	Polymerization parameters
Probase Hot	Ivoclar Vivadent Inc., Lichtenstein	Conventional heat polymerized Probase Hot	Liquid: methylmethacrylate (85-95%) Ethylene glycol dimethacrylate (5-10%) initiator	100°C, 45 min
			Powder: polymethylmethacrylate (>95%), plasticiser, pigments, Benzoyl Peroxide (1 - <2.5%)	
Probase Hot	Ivoclar Vivadent Inc., Lichtenstein	HP polymerized Probase Hot	Liquid: methylmethacrylate (85-95%) Ethylene glycol dimethacrylate (5-10%) initiator	100°C, 200
			Powder: polymethylmethacrylate (>95%), plasticiser, pigments, Benzoyl Peroxide (1 - <2.5%)	MPa, 1h
Ivobase CAD	Ivoclar Vivadent Inc., Lichtenstein	Ivobase CAD	polymethylmethacrylate (>90%)	unknown

The composition was obtained from manufacturer's data.

Conventional heat polymerized Probase Hot group blocks were manufactured with the conventional pack and press technique. A wax model of 80 mm wide × 80 mm length × 20 mm thick rectangle was invested in the denture flask. Two flasks were filled using type II gypsum (Gibraltar-Plaster, Henry Schein, USA). After removing the wax model with hot water, two layers of gypsum-resins insulator (Separating Fluid, Ivoclar Vivadent, Liechtenstein) were applied on the gypsum.

A mixture of heat polymerized acrylic resins was prepared according to the manufacturer's instructions with a proportion of 22.5 g of powder and 10 ml of liquid and at dough stage packed into the mold. The flask was closed and placed in the hydraulic press at an 8 MPa pressure. The flask was then placed in cold water, heated up to 100°C and boiled for 45 min according to the manufacturer's instructions.

A previously described HP polymerization process was applied to produce HP polymerized Probase Hot group blocks.²⁴⁻²⁶ 100 g of the mixture described above was placed inside a flexible silicone tube (25 mm internal diameter) and was then introduced into a custom-built autoclave with pressure and temperature control (LabVIEW version 8.6, National Instruments, USA). A thermocouple was placed close to the sample in order to monitor and control the temperature. At first the pressure was increased to 200 MPa at a rate of 1 MPa/s at ambient temperature. The temperature was then increased to 100°C at a rate of 2°C/min. The temperature and the pressure were then maintained at 200 MPa and 100°C for 60 min before cooling and releasing the pressure. The dimensions of HP polymerized Probase Hot out of the autoclave were 20 mm wide × 100 mm length × 20 mm thick rectangle.

The mechanical, physical and viscoelastic properties were evaluated following protocols described in previous studies.^{5, 24-26}

Specimens for the three-point bending test were cut from each material group with a low-speed saw (Isomet; Buehler, Lake Bluff, IL) under water irrigation to obtain 30 rectangular bars (10 mm × 3.3 mm × 65 mm) as described in ISO 20795-1.³⁰ Samples from Ivobase CAD group were cut directly from commercial discs of 98.5 mm diameter × 30 mm thick. Bars were then wet polished on 4000 grit silicon carbide (SiC) paper discs. Their dimensions were measured with a digital caliper (Mitutoyo Co., Kawasaki, Japan) before testing in a three-point bending device (50 mm span) at a crosshead speed of 5 mm/min using a computer controlled (INSTRON, USA) universal testing machine equipped with 1KN-cell force (Instron model 5565 with an extensometer). Flexural strength, σ_f , was calculated using the following formula:

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

where F is the load at fracture, L is the span (50 mm in this study), h is the specimen width, and c is the specimen thickness. The 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_f , was calculated according to the following formula:

$$E_f = \frac{FL^3}{4dhc^3}$$

where d is the deflection corresponding to load F at a point in the straight-line portion of the trace. The maximal flexural deformation was determined at the maximal flexural stress.

Thirty samples per group were 3 points fractured. Among them, 6 samples (half-bars) per group were surface coated with a 10 nm gold layer in a sputter-coater (SC500, Bio-Rad, UK). Microhardness was determined with a Vickers indenter (Struers Duramin, Denmark), under a 9.84 N loading and a 20 s dwell time for 5 samples a group, 6 values per sample have been recorded. Fractured surfaces were examined (one specimen per group) under a scanning electron microscope (SEM) (Hitachi S-3000N, Japan) (Fig 1) at X60 magnification for surface morphology characterization.

The assessment of the density was based on Archimedes's principle using a XS205 balance (Mettler Toledo, Switzerland), following the method explained a previous study.²⁴ The samples were weighed in air and in deionized water, and the density was then calculated using the following formula:

$$\rho = \frac{A(\rho_w - \rho_a)}{A - B} + \rho_a$$

where ρ is the density of the sample, A is the mass of the sample in air, and B is the mass of the sample in deionized water, ρ_w is the density of deionized water determined from the measurement of water temperature, and ρ_a is the density of air (0.0012 g/cm³).

The dynamic mechanical analysis (DMA) was carried out using a DMA 7/DX apparatus (PerkinElmer, Waltham, MA), following previous studies method that meant in 3 points bending mode with 15 mm distance, at a 1 Hz frequency.²⁶ Specimens suitable for DMA characterization were cut with a low-speed saw (Isomet; Buehler, Lake Bluff, IL) under water irrigation into rectangular specimens (4 mm x 1 mm x 20 mm) (n = 10 per each group),³¹ which were then wet polished on an 800-grit SiC paper and stored at room temperature under dry conditions. A static load of 0.2 N, a dynamic load of 0.18 N and a heating from 30°C to 180°C with 2°C/min ramp were applied. These measurements allowed the determination of storage modulus (E' at 32°C), loss modulus (E'' at 32°C), glass transition temperature (T_g) and damping factor (tanδ, at T_g) described in previous studies.^{26, 32} The dimensions of each sample were measured with a digital caliper (Mitutoyo Co, Kawasaki, Japan) before testing.

Data for mechanical, physical and viscoelastic properties were analyzed with 1-way ANOVA followed, by Scheffé multiple mean comparisons ($\alpha = 0.05$), using SPSS Statistics 21 (IBM, USA). The sample size was determined following the formula:³³

$$n = \frac{2s^2 (Z_{\alpha/2} - Z_{1-\beta})^2}{\Delta}$$

where n is the calculate sample size, s is the standard deviation, Δ is difference between the groups. Calculation was based on similar studies.⁵ A difference of 15% was chosen to have clinical significance. With $\alpha = 0.05$ and power (1- β) at 80%, $Z_\alpha = 1.96$ and $Z_{1-\beta} = 0.84$, the calculation gave n = 26.47, consequently samples size of 30 was set so that distribution could be considered as normal. The normality of the results distribution was assessed with the central limit theorem for groups with 30 samples and with the Shapiro-Wilks test for groups with 10 samples. The calculi of power (1- β) resulted in 99% to 100% for all tests except for Tanδ (30%).

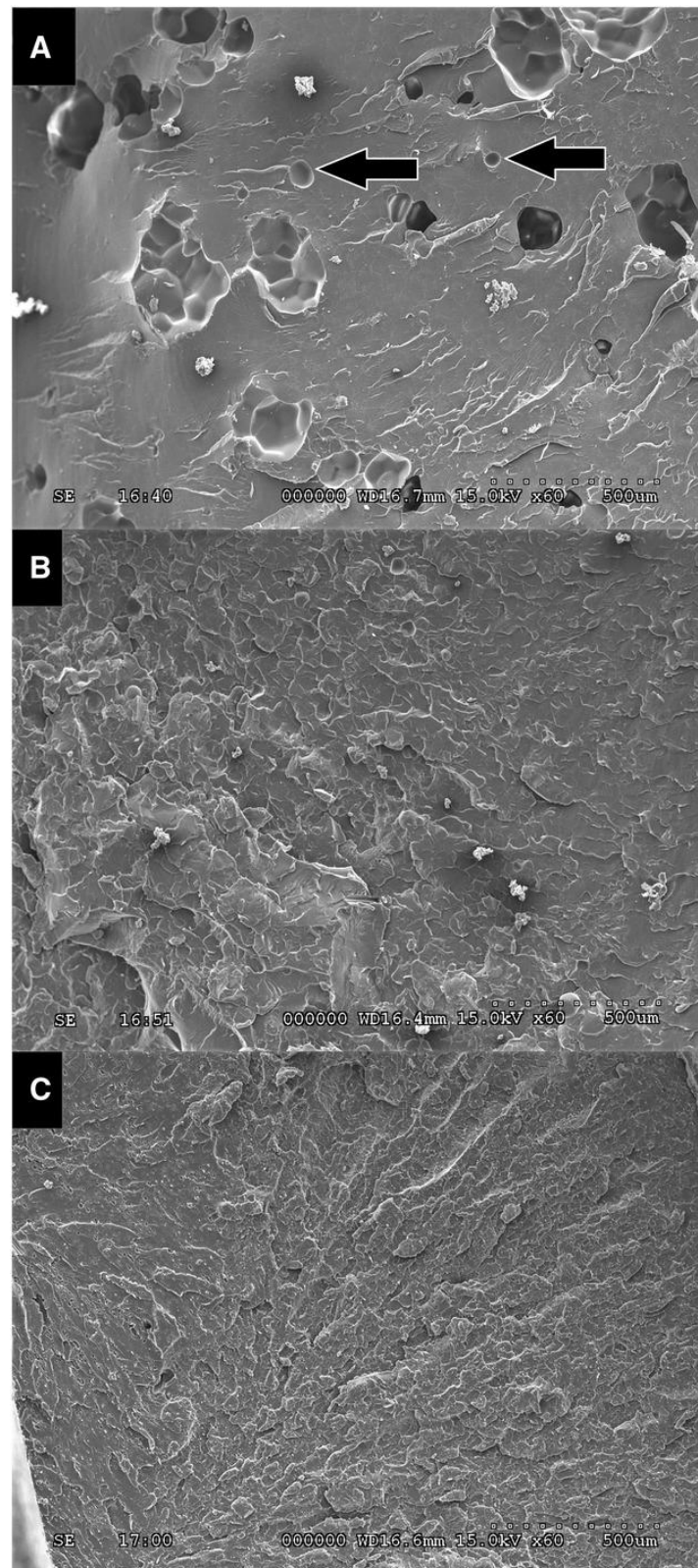


Figure 1. SEM micrographs of the fractured surfaces (60×): (A) Probases Hot conventional heat polymerized Probases Hot (black arrows: voids). (B) High pressure polymerized Probases Hot (at 100°C, 200 MPa). (C) Ivobase CAD. Conventional heat polymerized Probases Hot shows significantly more voids than HP polymerized Probases Hot and Ivobase CAD, which are more homogeneous.

Weibull statistics (Fig 2) parameters were calculated for the σ_f data. The description of the Weibull distribution is given by

$$P_f = 1 - e^{-\left(\frac{\sigma}{\sigma_0}\right)^m}$$

where m , the shape parameter, is the Weibull modulus, σ_0 is the scale parameter or characteristic strength $\sigma_{63.21\%}$.^{34,35} P_f , the fracture probability, is defined by the relation

$$P_f = \frac{\kappa}{N + 1}$$

where K is the rank in strength from least to greatest and N the total number of specimens.

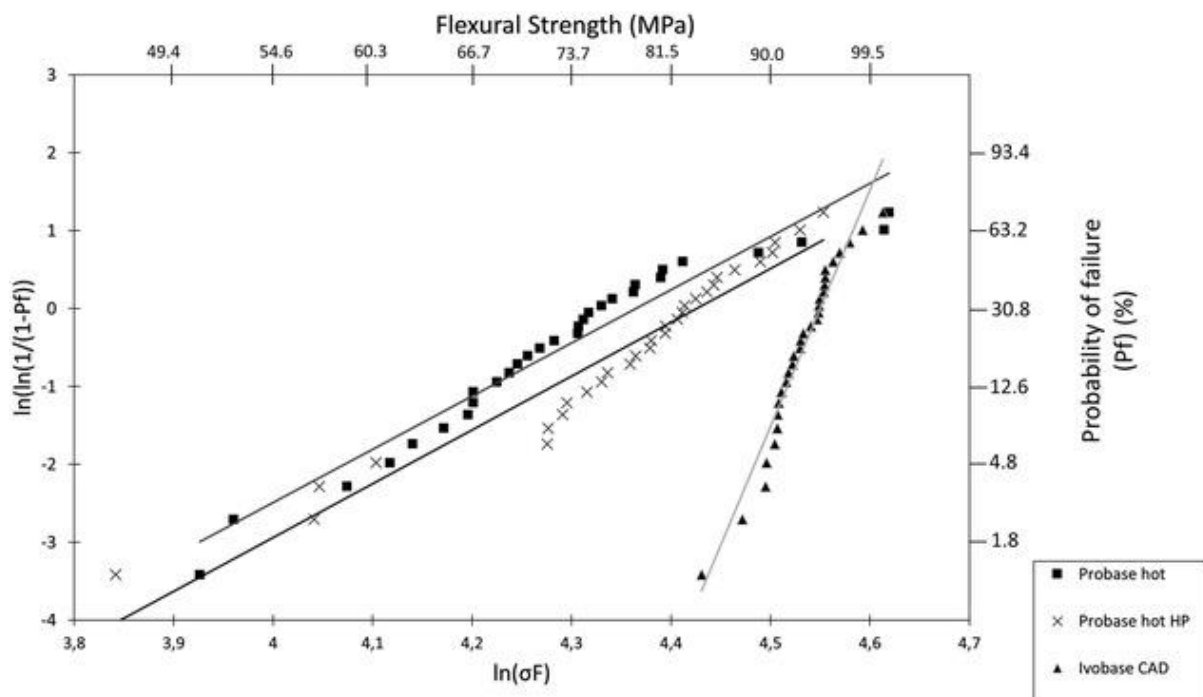


Figure 2. Weibull plots of the flexural strength (σ_f) results.

Results

The results of the mechanical, physical and viscoelastic properties are summarized in Table 2, along with the results of the statistical analysis. The results showed that the HP polymerization of Probases Hot significantly increased E_f ($p < 10^{-8}$), E' ($p < 2.6 \times 10^{-4}$), E'' ($p < 1.8 \times 10^{-4}$), and the density ($p < 10^{-8}$), while it significantly decreased T_g ($p < 5 \times 10^{-5}$). However, σ_f , hardness, flexural deformation at maximal stress, U_r , $\tan\delta$ were not significantly different compared to conventional thermopolymerization ($p = 0.184$ for σ_f ; $p = 0.289$ for hardness, $p = 0.713$ for flexural deformation at maximal stress; $p = 0.990$ for U_r ; $p = 0.227$ for $\tan\delta$). Results for Ivobase CAD were significantly higher than for HP polymerized Probases Hot regarding σ_f ($p < 10^{-6}$), U_r ($p < 10^{-8}$), and flexural deformation at maximal stress ($p < 10^{-8}$), while E_f , hardness, density, E' and E'' were significantly lower ($p < 10^{-8}$ for E_f , hardness and density; $p < 10^{-4}$ for E' ; $p < 0.036$ for E''). T_g and $\tan\delta$ of Ivobase CAD were not significantly

different from HP polymerized Probased Hot. The highest σ_f , U_r , flexural deformation at maximal stress and Weibull modulus were obtained with Ivobase CAD.

Table 2. Results of mechanical, physical and viscoelastic properties (Mean $\pm$ SD) and statistical analysis

Group				
Properties		Conventional heat polymerized Probased Hot	HP polymerized Probased Hot	Ivobase CAD
Mechanical Properties	σ_f (in MPa) (n = 30)	73.6 $\pm$ 11.9 ^b	78.2 $\pm$ 11.1 ^b	93.1 $\pm$ 3.4 ^a
	E_f (in GPa) (n = 30)	2.99 $\pm$ 0.13 ^b	3.32 $\pm$ 0.23 ^a	2.6 $\pm$ 0.11 ^c
	Flexural deformation at maximal flexural stress (in%) (n = 30)	2.80 $\pm$ 0.53 ^b	2.7 $\pm$ 0.49 ^b	6.64 $\pm$ 0.28 ^a
	U_r (in mJ) (n = 30)	200.6 $\pm$ 81.4 ^b	203.3 $\pm$ 66.7 ^b	754.6 $\pm$ 65.0 ^a
	Hardness (in HVN) (n = 30)	23.9 $\pm$ 2.1 ^a	23.1 $\pm$ 1.9 ^a	18.7 $\pm$ 1.7 ^b
	Weibull modulus	6.83	6.90	30.36
	$\sigma_{63.21\%}$ (in MPa)	78.7	83.6	94.7
Physical Properties	Density (in g/cm ³) (n = 30)	1.14847 $\pm$ 0.00039 ^c	1.19284 $\pm$ 0.00132 ^a	1.1698 $\pm$ 0.00022 ^b
Visco Elastic properties	E' (in GPa) (n = 10)	2.69 $\pm$ 0.32 ^b	3.16 $\pm$ 0.12 ^a	2.64 $\pm$ 0.16 ^b
	E'' (in GPa) (n = 10)	0.31 $\pm$ 0.04 ^b	0.39 $\pm$ 0.02 ^a	0.34 $\pm$ 0.03 ^b
	Tan δ (n = 10)	1.76 $\pm$ 0.44 ^a	1.52 $\pm$ 0.20 ^a	1.62 $\pm$ 0.19 ^a
	T_g (in°C) (n = 10)	119.8 $\pm$ 1.7 ^a	115.4 $\pm$ 1.5 ^b	115.6 $\pm$ 2.1 ^b

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 no significant differences horizontally per row.

HP is high pressure (at 100°C, 200 MPa).

σ_f is flexural strength.

E_f is elastic modulus.

U_r is the flexural load energy.

$\sigma_{63.21}\%$ is Weibull characteristic strength.

E' is storage modulus at 32°C.

E'' is loss modulus at 32°C.

$\tan\delta$ is damping factor at T_g .

T_g is glass temperature transition.

SEM observation of HP polymerized Probase Hot and Ivobase CAD fractured specimens showed less flaws than for conventional heat polymerized Probase Hot (Fig 1). Nevertheless, no difference between mechanical properties of conventional heat polymerized Probase Hot and HP polymerized Probase Hot was observed.

Discussion

The null hypotheses tested were that the mechanical, physical and viscoelastic properties of HP PMMA resins were not different from (1) those of conventional heat polymerized PMMA resins and from (2) those of Ivobase CAD. In view of the above findings, the first null hypothesis was rejected for T_g , E_f , E' , E'' , density, and was not rejected for σ_f , hardness, flexural deformation at maximal stress, U_r and $\tan\delta$. The second null hypothesis was rejected for σ_f , flexural deformation at maximal stress and U_r of HP polymerized Probase Hot, which were shown to be significantly inferior to Ivobase CAD. It was also rejected for U_r , E_f , hardness, density, E' and E'' , which were shown to be significantly superior to Ivobase CAD. However, the second null hypothesis was not rejected for T_g and $\tan\delta$.

The HP process chosen (100°C-200 MPa) was based on previous literature, which showed that polymerization under 250 MPa significantly increases the mechanical, physical properties of resins composites,²⁴ with 200 MPa being the optimal pressure for UDMA.²⁶ HP polymerization was conducted under T_g at 100°C to reduce thermal degradation, which can occur at higher temperatures.³⁶ Previous studies have described polymer main chain and side group scission as depolymerization, at approximately 150°C to 165°C, which engenders radical transfer to the unsaturated chain end and H-H bond scissions.^{37, 38} Indeed, altered molecular structure of degraded polymer affects mechanical properties.³⁹ Moreover, polymerizing at 100°C, as in the conventional heat polymerized Probase Hot group, allowed to discriminate the HP influence.

The results showed that polymerizing PMMA under HP decreases flaws and voids and promotes the cohesion between PMMA particles and matrix (Fig 1B). However, the observed improvements did not significantly increase mechanical properties compared to conventional heat polymerized Probase Hot and Ivobase CAD (except for E_f), contrary to commercial composite resins or UDMA polymer.^{24, 25} In fact, HP induces two effect on molecules: HP brings the monomers closer, and the free volume reduction increases the probability of molecular matching as well as the reactivity,^{27, 28} while if the pressure is too high, HP can decrease the molecular mobility and slow down the

polymerization kinetics, as reported by Kaminski and al⁴⁰ with triethyleneglycol dimethacrylate (TEGDMA). Consequently, future research should focus on different pressure values to determine the most efficient approach.

On the other hand, radical polymerization of acrylic resins produces an important exothermic reaction.⁴¹ Indeed, compared to most dimethacrylate monomers used in dentistry, methylmethacrylate (MMA) has a low molecular weight and a high concentration of vinyl groups, which makes the reaction more exothermic and induces more volumetric contraction.⁴² This effect can engender thermal degradation and internal stress,^{37, 38, 43} as demonstrated by the decreased T_g of HP polymerized Probase Hot compared to conventional heat polymerized Probase Hot, which is evidence of an alteration of polymers and a lower polymerization.³⁹ As T_g and hardness allowed to evaluate the degree of crosslinking and the degree of conversion of a polymer,⁴⁴⁻⁴⁶ lower hardness and T_g of Ivobase CAD compared to conventional heat polymerized Probase Hot suggested that the degree of conversion of commercial Ivobase CAD disc was lower than conventional heat polymerized Probase Hot. As shown in SEM images (Fig 1) the industrial process of Ivobase CAD allows to obtain a more homogeneous material with less flaws than conventional heat polymerized Probase Hot, that could partly explain significantly higher σ_f , flexural deformation at maximal flexural stress and U_f and Weibull modulus. Thus, improved Weibull modulus of Ivobase CAD allows for higher reliability.

A previous study characterized Ivobase CAD disc and conventional heat polymerized Probase Hot, with different results for σ_f and microhardness.⁵ These differences can be considered as limitations between studies due to several factors: the operator-dependent conventional press pack polymerization process, and the use of a less accurate cell force in the previous study (5KN). Indeed, the 5KN-cell force can lead to a variation in 6 to 7 N while a 1KN-cell force leads to a variation of 1 to 2 N, which engenders a variation of 5% in the results for σ_f (force was approximately 100-130 N for the samples).

Finally, HP significantly increased E_f and E' , which are essential properties of denture base. Indeed, stiffness improves occlusal stress distribution on soft tissues.⁴⁷ This higher E_f could be explained by the observed increased density and a higher cohesive density energy.⁴⁸

Limitations of the present study are related to the exothermic reaction characteristic of the mixture of monomers and polymers in conventional heat polymerized Probase Hot and HP polymerized Probase Hot.⁴¹ This exothermic reaction can be increased by the high pressure used in HP polymerized Probase Hot and can engender internal stresses and volume variations, which can, despite better material homogeneity compared to conventional heat polymerized Probase Hot, explain the absence of most mechanical properties improvement for HP polymerized Probase Hot. For Ivobase CAD, the manufacturing process and exact composition are unknown, which limits data interpretation.

Conclusion

HP polymerization at 200 MPa did not significantly improve the mechanical properties of PMMA denture base resins compared to conventional heat polymerization, these properties being inferior to CAD/CAM PMMA (Ivobase, Ivoclar). However, viscoelastic properties and density were significantly increased. Studies are necessary to understand and potentially optimize the PMMA HP polymerization. Ivobase CAD/CAM material constitutes a significant improvement in terms of mechanical resistance compared to conventional heat polymerized materials and should be recommended in the context of new digital protocols. Nevertheless, manufacturers should provide better information in terms of composition and manufacturing process of those CAD/CAM materials.

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