Effect of Storage in Water and Thermocycling on Hardness and Roughness of Resin Materials for Temporary Restorations

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Purpose: This study evaluated the effect of storage in water and thermocycling on hardness and roughness of resin materials for temporary restorations. **Material and Methods:** Three acrylic resins (Dencor-De, Duralay-Du, and Vipi Cor-VC) were selected and one composite resin (Opallis-Op) was used as a parameter for comparison. The materials were prepared according to the manufacturers' instructions and were placed in stainless steel moulds (20 mm in diameter and 5 mm thick). Thirty samples of each resin were made and divided into three groups (n = 10) according to the moment of Vickers hardness (VHN) and roughness (Ra) analyses: C (control group): immediately after specimen preparation; Sw: after storage in distilled water at 37 °C for 24 hours; Tc: after thermocycling (3000 cycles; 5-55 °C, 30 seconds dwell time). Data were submitted to 2-way ANOVA followed by Tukey's test ($\alpha = 0.05$). **Results:** Op resin had higher surface hardness values (p < 0.0001; 25.4 ± 3.4) than the other ones (De = 4.5 ± 0.6; Du = 5.5 ± 0.4; VC = 6.1 ± 0.9). There was no statistical difference (p > 0.05) in roughness among materials (De = 0.31 ± 0.07; Du = 0.51 ± 0.20; VC = 0.41 ± 0.15; Op = 0.42 ± 0.18). Storage in water did not change hardness and roughness of the tested materials (p > 0.05). There was a significant increase in roughness after thermocycling (p < 0.05), except for material Du, which showed no significant change in roughness in any evaluated period (p = 0.99). **Conclusion:** Thermocycling increased the roughness in most tested materials without affecting hardness, while storage in water had no significant effect in the evaluated properties.

Keywords: acrylic resin, composite resin, Vickers hardness, surface roughness, thermocycling

1. Introduction

During prosthetic planning, several aspects, such as the mechanical, biological and esthetic principles of tooth preparation, must be respected to obtain successful oral rehabilitation. Temporary restorations should be inserted immediately after tooth preparation to reestablish esthetics and function, as well as to protect the adjacent periodontium and pulp tissue against exogenous damage. Furthermore, temporary restorations are used to assist diagnosis and planning oral rehabilitation, correcting changes in the occlusal plane, reestablishing occlusal vertical dimension, providing harmonious gingival contour and evaluating color, shape and size of the definitive restorations¹.

Temporary restorations may be made with polymethyl methacrylate, polyethyl methacrylate-based resins and bis-acrylic resin composites, which have an organic matrix/filler content with a chemical composition similar to that of resin composites²⁻⁴. It has been shown that bis-acrylic resin composites provide greater resistance to dissolution, fracture resistance, flexural strength, better anatomic contours and marginal fit when compared with acrylic resins^{2,5}. However, some studies have shown that acrylic resins exhibit a smoother surface than bis-acrylic resin composites after both polymerization^{2,3} and surface polishing^{2,3,6}.

According to Diaz-Arnold et al.⁷, surface hardness could be related to degradation of the restoration, since this property is directly related to the quality of polymerization and cross-link density^{8,9} of the material, and specifically for resin composites, to its filler content¹⁰.

The presence of a rough surface on the temporary restoration favors bacterial adhesion and dental biofilm formation, resulting in gingival inflammation and periodontal bone resorption¹¹. Furthermore, a rougher surface affects light reflection and brightness of the restorative material, as well as favors discoloration and staining¹². Ayuso-Montero et al.² also reported that the surface of the temporary restoration should be smooth enough to be comfortable, esthetic, and able to avoid staining and biofilm accumulation.

It has been demonstrated a decrease in the mechanical properties of the materials used for temporary restorations following prolonged exposure to humidity¹³⁻¹⁷. In addition, the temperature of the mouth varies considerably by the intake of hot and cold food and drinks, resulting in stress at the interface tooth-restoration. As a consequence, the stability of the cement may be compromised leading to microleakage and dissolution of the cement. Furthermore, constant temperature range between 4 and 60 °C might lead to fatigue or stress, which impair some material properties such as cohesive, compressive, and shear strength, as well as hardness and roughness.

Other factors may affect the surface hardness and roughness of temporary restorations, such as processing and polishing techniques, power to liquid ratio, and time of use¹⁸. Additionally, Silva Filho et al.¹⁹ verified that roughness of some chemically activated acrylic resins increased after thermocycling. Therefore, temporary restorations should not remain in the oral cavity for long time.

Considering that some rehabilitation treatments require the use of temporary restorations for a longer period of time, the aim of this study was to assess the effect of storage in water and thermocycling on the Vickers hardness and roughness of three autopolymerizing acrylic resins and one microhybrid composite resin. It was hypothesized that the evaluated properties would (1) vary among different materials and (2) change with storage in water and thermocycling.

2. Material and Methods

2.1. Materials used

Three autopolymerizing acrylic resins used for temporary restorations (Dencor, Duralay, and Vipi Cor) and one composite resin (Opallis), as a parameter for comparison, were selected (Table 1).

2.2. Specimen fabrication

The acrylic resins were mixed in accordance with the manufacturers' instructions (Table 1) and were inserted into hollow, disk-shaped stainless steel moulds with the dimensions of 20 mm in diameter and 5 mm thick. The composite resin test specimens were obtained by inserting and photopolymerizing them in the same matrix. During the polymerization, the test specimens were manually pressed between two Mylar strips and glass plates. After the polymerization time (Table 1), the specimens were removed with a plunger. The test specimens were then submitted to finishing using 600-grit silicon carbide paper to remove superficial irregularities. Polishing procedure was performed in a polishing lathe (Nevoni, São Paulo, Brazil) using pumice stone (Labordent, São Paulo, Brazil) along with white, black brushes, and felt tip for 1 minute each, respectively. Final polishing was performed with a flannel wheel and Spanish white paste (Labordent, São Paulo, Brazil) during 1 minute.

2.3. Experimental groups

The test specimens (n = 10) were submitted to one of the experimental conditions:

- Control (C): specimens were tested immediately after preparation;
- Storage in water (Sw): storage in distilled water at 37 °C for 24 hours²⁰.
- Thermocycling (Tc): thermal cycles were made in a thermocycling machine (model MSCT-3, Marcelo Nucci – ME, São Carlos, SP, Brazil) and consisted of 3000 cycles at 5 and 55 °C with a 30-second dwell time²¹.

2.4. Vickers hardness and roughness

Hardness measurements (VHN) were made with a microhardness indenter machine (Micromet 2100; Buehler, Lake Bluff, Illinois, USA) at a 25 gf load for 30 seconds. Twelve indentations were made on each specimen, and then the mean value was calculated.

The superficial roughness was evaluated using a surface roughness tester (Surftest SJ-401, Mitutoyo Sul Americana Ltda, Santo Amaro, SP, Brazil) with a standard cut-off value of 0.8 mm and a speed of 0.5 mm/s. Mean surface roughness (Ra) was measured in 5 areas on each specimen. The results were submitted to two-way analysis of variance, followed by Tukey's test (α =.05).

3. Results

A significant materials-experimental groups interaction indicated that experimental condition influenced both hardness and roughness of the tested materials (Table 2).

For hardness (Table 3), Dencor control specimens showed the lowest mean value (p < 0.05). After storage in water, Dencor and Duralay acrylic resins demonstrated the lowest mean values (p < 0.05). After thermocycling, there was no significant difference among the autopolymerizing acrylic resins (p > 0.05). For all the experimental groups, the composite resin exhibited the highest Vickers hardness values (p < 0.0001).

For roughness (Table 4), results demonstrated that there was no significant difference (p > 0.05) among the materials in the control group. Furthermore, the surface roughness of the materials evaluated was not changed after storage in water (p > 0.05). After thermocycling, there was an increase in the surface roughness of the acrylic resins Dencor (p < 0.0001) and Vipi Cor (p = 0.0003) and of the composite resin Opallis (p = 0.0017). The surface roughness of Duralay acrylic resin was not changed by thermocycling (p = 0.9926).

4. Discussion

This in vitro study simulated the use of temporary restorations for a period of three years, since each 1000 thermocycling represent the use of a fixed prosthesis for one year²². Storage in water during 24 hours was tested because in the indirect fabrication technique, temporary restorations should remain stored in water until cementation onto the tooth preparations.

Table 1. Materials evaluated in this study.

Product	Manufacturer	Type	Polymerization time	Powder / Liquid ratio	Composition
Dencor	Clássico LTDA, São Paulo, SP, Brazil	Autopolymerizing acrylic resin	8 minutes	20 g / 10 mL	Powder: PMMA, pigments, benzoyl peroxide; Liquid: MMA, DMT, EGDMA
Duralay	Reliance Dental Mfg Co., Worth, USA	Autopolymerizing acrylic resin	8 minutes	20 g / 10 mL	Powder: MMA copolymer, dialkyl phthalate, pigments, benzoyl peroxide; Liquid: MMA, DMT
Vipi Cor	Dental Vipi, Pirassununga, SP, Brazil	Autopolymerizing acrylic resin	5 minutes	15 g / 7.5 Ml	Powder: PMMA, pigments, benzoyl peroxide; Liquid: MMA, DMT, EGDMA
Opallis	FGM Produtos Odontológicos, Joinville, SC, Brazil	Micro-hybrid composite resin	40 seconds for each 2 mm increment	-	BisGMA, BisEMA, TEGDMA (resin matrix = 21-22.5%) and barium glass, aluminum silicate and silica dioxide (filler = 77.5-79% weight)

Table 2. Two-way ANOVA with materials and experimental conditions as independent variables for both hardness and roughness.

Source	df	<i>p</i> -value		
		Vickers hardness	Roughness	
Materials (A)	3	< 0.0001	0.0033	
Experimental condition (B)	2	0.5293	< 0.0001	
Interaction $(A \times B)$	6	0.0015	< 0.0001	

Table 3. Vickers Hardness means $(VHN) \pm standard$ deviations for the experimental groups.

	С	Sw	Тс
Opallis	$25.6 \pm 3.4 \mathrm{Ac}$	$24.0 \pm 2.3 \text{ Ac}$	$16.3 \pm 1.4 \mathrm{Ab}$
Vipi Cor	$6.1 \pm 0.9 \mathrm{Aa}$	$6.4 \pm 0.8 \mathrm{Aa}$	$6.4 \pm 0.8 \mathrm{Aa}$
Duralay	$5.5 \pm 0.4 \mathrm{Aa}$	$4.8 \pm 0.7 \text{ Ab}$	$5.3 \pm 0.3 \text{ Aa}$
Dencor	$4.5 \pm 0.6 \mathrm{Ab}$	$5.0 \pm 0.8 \text{ Ab}$	$5.4 \pm 0.8 \mathrm{Aa}$

Horizontally, means with identical capital letters were not significantly different (p > 0.05). Vertically, means with identical small letters were not significantly different (p > 0.05).

Table 4. Roughness means (μm) ± standard deviations for the experimental groups.

	С	Sw	Тс
Opallis	$0.42 \pm 0.18 \mathrm{Aa}$	0.46 ± 0.22 Aab	$0.83 \pm 0.21 \text{ Ba}$
Vipi Cor	$0.41 \pm 0.15 \mathrm{Aa}$	$0.58 \pm 0.14 \mathrm{Aa}$	$0.95 \pm 0.35 \; \mathrm{Ba}$
Duralay	$0.51 \pm 0.20 \mathrm{Aa}$	$0.50 \pm 0.16 \mathrm{Aab}$	$0.43 \pm 0.18 \mathrm{Ab}$
Dencor	$0.31 \pm 0.07 \mathrm{Aa}$	0.31 ± 0.08 Aab	$0.81 \pm 0.29 \text{ Ba}$

Horizontally, means with identical capital letters were not significantly different (p > 0.05). Vertically, means with identical small letters were not significantly different (p > 0.05).

The tested hypotheses that hardness and roughness would vary among different brands and would change with storage in water and thernocycling were partially proved. There was no significant difference among the materials in the control and the storage in water groups for roughness. Storage in water did not change both hardness and roughness values of the materials and thermocycling did not affect the hardness of the materials and the roughness of Duralay acrylic resin.

Regarding the hardness, the composite resin showed higher values than the acrylic resins in all the experimental groups. These results may be explained by the difference in composition between the two types of material. The composition of Opallis resin composite presents as organic matrix BisGMA, BisEMA and TEGDMA monomers and as filler particles barium glass, aluminum silicate and silica dioxide (77.5 to 79%). On the other hand, the acrylic resins are basically composed of MMA monomers and EGDMA as a crosslinking agent, with the exception of Duralay resin that does not have a crosslinking agent in its composition and presents a plasticizer (dialkyl phthalate).

It has been reported that surface hardness of composite resins is influenced by both the organic matrix (monomers) and the inorganic filler^{10,23}. With regard to the organic matrix, hardness depends on the density and structure of the polymer formed²³ and the degree of conversion after the polymerization¹⁰. It is known that the presence of aromatic groups in the monomers BisGMA and BisEMA provides a polymeric structure with higher rigidity²⁴. Hardness has also been used as an indirect method to measure the degree of conversion of

resins: the higher the degree of conversion, the higher the hardness values²³. Moreover, the use of bifunctional monomers such as EGDMA and TEGDMA¹⁰ to form crosslinked chains improves the hardness values. Regarding the inorganic filler, the hardness values obtained are also dependent on the volume added to the material. When the quantity of particles increases, the surface hardness of the material also increases^{10,23,25}. Therefore, it may be suppose that the quality of polymerization of the composite resin with respect to the polymer density and the degree of conversion obtained was superior to that of the acrylic resins tested.

A decrease in surface hardness could be expected after storage in water and thermocycling due to water absorption. The action of the water molecules inside the polymeric structure has been described as plasticizing effect^{3,26} and the decrease in hardness would be associated with the reduction in the interchain interactions²⁶. Furthermore, considering that the temporary restoration will be submitted to temperature changes inside the mouth, the restorative material could undergo fatigue or stress, similar to the application of cyclic loads from the cumulative effect of shrinkage and expansion. One of the harmful consequences of this phenomenon would be the decrease in the hardness values²⁷. However, the hardness of the evaluated resins did not change neither after storage in water nor after thermocycling. It has been reported that polymeric chains with high crosslink density decrease water absorption of the material due to the reduction of the free space. Moreover, in materials with a denser polymer network, less water absorption is expected. The fact that the experimental conditions did not affect the hardness values may be explained by the presence of crosslinked chains in the polymeric structure of the acrylic resins Dencor and Vipi Cor and the composite resin Opallis. In spite of Duralay acrylic resin does not have crosslinking agents in its composition, one could suppose that this material has a denser polymeric structure. However, further studies are needed to confirm this assumption. In addition, residual monomer molecules acting as plasticizers²⁸ may have been released²⁹ during the storage period, thus compensating the plasticizer effect of water absorption³⁰, although this was not assessed in this study.

With regard to the roughness results, no significant differences were observed among the acrylic resins evaluated and the composite resin used as a parameter for comparison. In another study³¹, there were also no significant differences in the roughness of the acrylic resins Dencor, Duralay and Vipi Cor. The study of Şen et al.³ showed that the methacrylate-based resin specimens showed smoother surfaces than the bis-acrylic composite specimens. This fact was related to the homogeneous composition of the acrylic in contrast of the heterogeneous composition of the composite. Although there were differences in the chemical composition of the acrylic resins and the composite resin evaluated, the fact that the surface roughness of the materials did not differ may be explained by the standard polishing procedure performed in all specimens.

An increase in the surface roughness values of all the materials could have been expected after specimens had been submitted to the experimental conditions because water absorption and solubility result in a variety of chemical and physical processes, such as the release of substances that did not react²⁹ and the formation of byproducts by hydrolytic degradation, affecting the structure as well as the biocompatibility^{32,33} of the polymeric materials²⁶. Particularly, exposing composite materials to water has been shown to degrade dental composites due to the degradation of filler particles, the weakening of polymer matrix or the debonding of filler-matrix interfaces³⁴. However, an increase in surface roughness was only observed after thermocycling for Dencor, Vipi Cor and Opallis products. Only Duralay acrylic resin did not undergo the influence of thermocycling. This result is in agreement with Silva Filho et al.¹⁹,

who observed that Duralay acrylic resin showed low surface change after thermocycling, probably due to its high density by the presence of smaller and more regular pre-polymeric particles compared with the other acrylic resins.

Although the absorption of water is reduced in materials with high filler content, the increase in the roughness of the composite resin Opallis after thermocycling may be attributed to the hydrolysis of silane coupling agents as well as the stress at the filler-matrix interface^{4,35}. As a consequence, the filler particles located at the surface of the material would debond and the grooves created would promote the increase in the roughness, as observed in this study.

The increase in surface roughness suggests that in clinical conditions, considerable discomfort to the patient and risk for periodontal diseases by the accumulation of biofilm on the temporary restoration may also increase 36,37 . According to Bollen et al. 38 , surface roughness values higher than 0.2 µm facilitate microbial adhesion both in vitro and in vivo studies. Although the results exhibited roughness higher than 0.2 µm, the materials evaluated in this study may be considered as materials with low roughness according to Zissis et al. 39 , once the materials' roughness ranged from 0.7 to 3.4 µm. However, the prolonged use of temporary restorations made with composite resin or acrylic resin should be avoided in order to reduce microbial colonization caused by the presence of rougher material surfaces.

Although thermocycling simulates aging by submitting materials to extreme temperatures, temporary restorations in the clinical condition are exposed to the detrimental effects of food, beverages, saliva components, and to the mechanical action of chewing and brushing. Therefore, these aspects should be considered in future investigations.

5. Conclusion

Within the limitations of this in vitro study, it can be concluded that:

- 1. Storage in water had no significant effect in the hardness and the roughness of the tested materials.
- Thermocycling did not change the hardness of the tested materials.
- Thermocycling increased the roughness in most tested materials, with the exception of Duralay which was not affected by thermocycling.
- 4. Dencor acrylic resin and Opallis composite resin showed the lowest and the highest hardness values, respectively.
- 5. Surface roughness values did not differ for the tested materials in the control group.

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