Knoop Microhardness of Conventional and Microwaved Denture Base Acrylic Resins

Abstract

Background: Microwave polymerization reduces the time of acrylic resin processing, but it is important to select a proper cycle to avoid overheating, porosity, and impairment to the materials’ properties. Aims: To evaluate the microhardness of conventional (Vipi Cril-VC) and microwaved (Vipi Wave-VW) acrylic resins after microwave polymerization cycles and the cycles recommended by the manufacturer. It was also evaluated the effect of thermocycling on the microhardness of these materials. Methods and Materials: Specimens (n = 10) were made: 1. WB (water-bath recommended for VC polymerization); 2. M630/25 (10 min at 270 W; 5 min at 0 W; 10 min at 360 W; recommended for VW polymerization); 3. M550/3 (3 min at 550 W); and 4. M650/5 (5 min at 650 W). After 48 h in distilled water at 37°C, specimens were subjected to Knoop test under 25 g during 5 s. The same specimens were submitted to thermocycling (5,000 cycles; 5°C and 55°C; 60 s) and the microhardness was again measured. The results were analyzed by repeated measures one-way analysis of variance/Bonferroni for each material (α = 0.05). Results: For both materials, no difference was obtained for groups polymerized using the cycles recommended by the manufacturer (VC = 19.8 ± 0.9 KHN; VW = 21.0 ± 1.6 KHN) and M650/5 (VC = 19.9 ± 1.5 KHN; VW = 20.9 ± 0.8 KHN). After thermocycling, microhardness decreased in experimental cycles for VC (M550/3 = 17.0 ± 1.9 to 14.6 ± 1.2 KHN; M650/5 = 19.9 ± 1.5 to 17.8 ± 0.8 KHN) and in all microwave cycles for VW (M630/25 = 21.0 ± 1.6 to 18.3 ± 0.9 KHN; M550/3 = 17.5 ± 3.0 to 15.7 ± 2.0 KHN; M650/5 = 20.9 ± 0.8 to 18.2 ± 2.0 KHN) (P = 0.000). The lowest hardness was observed for both resins processed by M550/3 (P = 0.001). Conclusions: Both materials could be polymerized in microwave during 5 min showing hardness similar to the cycles recommended by the manufacturer. Thermocycling did not decrease the hardness of VC polymerized with both cycles recommended by the manufacturer and VW polymerized with the water-bath conventional cycle.

Keywords: Denture bases, hardness, microwaves, polymerization, polymethyl methacrylate

Introduction
The polymethyl methacrylate acrylic resin has been the material of choice for manufacturing removable denture bases and artificial teeth since the early 20th century. The polymerization of these materials occurs by a series of chemical reactions, in which the polymers are formed from a large number of smaller molecules called monomers. The process of polymer formation and chain growth continues at a considerable rate. Theoretically, these reactions should continue with the increase in heat until all monomer was converted into polymer.[1] However, as the polymerization temperature is reduced, the formation of polymers is also reduced, and consequently some residual monomer remains in the polymerized resin.[1] Studies have demonstrated that, due to its plasticizing action, the presence of this monomer inside the polymeric bulk may negatively influence the physical and mechanical properties[2] of the materials. Other studies have also demonstrated that the quantity of residual monomer may be influenced by several factors as time, temperature, and the polymerization method employed, being greater for self-cured resins compared to heat-cured resins, and reduced in resins polymerized in microwave.[3]

Different from the conventional method, microwaves act only on the monomer, which decreases in the same proportion as the polymerization degree increases.[4] During polymerization in microwave, the movement of irradiated monomer molecules is guided by a high frequency electromagnetic field (2450

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MHz). In this movement, the molecules change their directions millions of times per second. Consequently, several intermolecular collisions occur, causing fast internal heating in which the energy is immediately absorbed by the resin, regardless of the thermal conductivity of materials involved in the processing of dentures, and occurs in a uniform manner. So, a form of self-regulation of the curing program is established, thus resulting in the complete polymerization of the resin. As a consequence, the time required to transfer the heat from water-bath to the resin inside the flask is eliminated. Therefore, in the conventional water-bath method, the resin is slowly heated in a passive manner, because the heat is externally generated and the monomer molecules collide due to this heating.

Kimura et al. used several time/power combinations inside the microwave oven and observed that it was possible to polymerize the material in reduced periods (500 W for 3 min), achieving accuracy and adaptation of the denture base, especially at the posterior palatal marginal area. Shlosberg et al. evaluated the flexural strength and hardness and did not observe differences between heat-cured resins polymerized conventionally and in microwave. Smith et al. analyzed the hardness, flexural strength, modulus of elasticity, and impact strength of acrylic resins polymerized conventionally, in microwave, and by visible light. These authors observed that polymerization in microwave increased the modulus of elasticity of two resins, reduced the impact strength of one resin and did not adversely affect the properties of the other two resins.

This alternative method of polymerization in microwave presents advantages as easy cleaning during processing of acrylic resin, homogeneously distributed heating, fast temperature increase and consequent reduction in fabrication time, minimum discoloration of the denture base, and lower risk of fracture of artificial teeth during removal from the flask. Despite the great number of studies analyzing the effect of polymerization in microwave on the properties of acrylic resins, this method still has limited clinical acceptance.

The importance of adequate selection of the polymerization cycle in microwave has been reported to avoid overheating of the monomer, which might cause degradation porosity in the material and consequent damage to the physical and mechanical properties of the denture. According to Bafile et al., resins fabricated for utilization in microwave oven may contain triethylene- or tetraethylene glycol dimethacrylate in their composition. These dimethacrylates present low steam pressure, allowing polymerization at high temperatures (between 100 and 150°C) without risk of porosities, which does not occur with the polymethyl methacrylate that presents high steam pressure.

Hardness is considered a relevant surface property and is defined as the material resistance to indentation or permanent penetration. Since microhardness is a simple and effective method to evaluate the degree of polymer conversion, measurements of this property have been used as an indirect method to determine the degree of conversion of conventional and self-cured denture base acrylic resins. Also, hardness represents the viscoelastic properties of the material and its resistance to wear; being that materials with low hardness values may present increased roughness, predisposing to microbial adhesion and staining, and esthetic problems.

Thermocycling is a usual laboratory method for testing of dental materials, serving as a guide for adequacy and utilization in vivo. Thermocycling represents a mean of exposure of polymeric materials to fatigue tests, aiming to achieve an indicator of the material performance in the humid oral environment. Based on this information, this study aimed to evaluate the microhardness of denture base heat-cured acrylic resins (Vipi Cril and Vipi Wave) after accomplishment of experimental polymerization cycles in microwave and in cycles recommended by the manufacturer. The study also aimed to evaluate the effect of thermocycling on the microhardness of these materials. The null hypotheses tested in this study were: 1) there would be no difference in hardness between groups polymerized using recommended and experimental cycles for each material; and 2) thermocycling would not decrease the microhardness of materials polymerized in the different cycles.

Materials and Methods

Material

The heat-cured denture base acrylic resins selected for this study are presented in Table 1.

Specimen preparation

Specimens (n = 10) were fabricated using moulds with dimensions $64 \times 10 \times 3.3$ mm. For this, impressions were taken from metallic templates with these dimensions using laboratory silicone (Zetalabor; Zhermack, Labordental, SP, Brazil) between two glass plates. The impression/template assembly was placed in metallic or plastic flask using type III stone plaster. A glass plate was placed over the impression to provide a smooth specimen surface. The flask was filled with stone plaster, closed, and kept under a load of 0.5 t in a hydraulic press during plaster setting. After this period, the flask was opened and the metallic template was removed.

The materials were manipulated following the manufacturers’ instructions [Table 1] and inserted in the silicone impression. The flask was closed and kept under a load of 1.25 t in a hydraulic press during 30 min. After this period, the specimens were submitted to conventional or microwave polymerization cycles, as presented in Table 2. The flask were cooled in tap water for 30 min and on the bench for 15 min. The specimens were then removed from the silicone impressions and submitted to metallographic
polishing (Aropol E; Arotec, Cotia, SP, Brazil) using silicon carbide sandpaper grits #240, #400 and #600 (3M, Campinas, SP, Brazil), under constant cooling, for removal of irregularities. After polishing, the specimens were stored in distilled water in an oven (Nova Instruments, Piracicaba, SP, Brazil) at 37°C during 48 h.\[16\]

**Knoop microhardness**

The Knoop microhardness test was performed (Shimadzu HMV-2000; Shimadzu Corporation, Kyoto, Japan) with a load of 25 gf for 5 seconds. Five indents were made in each specimen and the mean value represented the specimen hardness. A single examiner assessed the greatest diagonal immediately after each indent, allowing a minimum interval (around 10 seconds) between indent and reading. The smooth and flat surface of specimens enhanced observation of the diagonal during the analyses. After the initial measurements, the specimens were submitted to thermocycling (5,000 cycles, 5 and 55°C, immersion interval 60 seconds) and microhardness was once again measured.

**Statistics**

Data were submitted to repeated measures one-way analysis of variance followed by Bonferroni test for each material at a confidence level of 95% ($\alpha = 0.05$), using the software IBM SPSS 19 (SPSS Inc., IBM Company, Armonk, NY, USA). Power sample analyses were also performed using the software IBM SPSS 19.

**Results**

For the number of specimens used for measurement of microhardness ($n = 10$), this study presented adequate power for the factor “polymerization cycle” for both materials (VC = 98.2% and VW = 96.1%; $\alpha = 0.05$).

Figure 1 demonstrates that, for the conventional heat-cured VC resin, there was no significant difference for hardness values among WB, M630/25, and M650/5 groups. Regardless of thermocycling, the lowest hardness value was observed for the cycle M550/3 (17.0 ± 1.9 to 14.6 ± 1.2 KHN) ($P = 0.000$). After thermocycling, a reduction was observed in the microhardness values only for the experimental polymerization cycles in microwave (M550/3 = 17.0 ± 1.9 to 14.6 ± 1.2 KHN; M650/5 = 19.9 ± 1.5 to 17.8 ± 0.8 KHN) ($P = 0.000$).

![Figure 1](image_url)

**Figure 1**: Mean values and standard deviation of microhardness for VC and VW resins for the different polymerization cycles in water-bath and in microwave, before and after thermocycling. For each material, different upper case letters indicate statistically different values in the comparison among polymerization cycles ($P < 0.05$). For each material, different lower case letters indicate statistically different values before and after thermocycling ($P < 0.05$)

| Material (acronym) | Type | Composition* (batch) | Powder/liquid ratio | Manufacturer |
|-------------------|------|----------------------|---------------------|--------------|
| Vipi Cril® (VC)   | Conventional | PMMA, benzoyl peroxide, pigments (00773) | 2.15 g/1 mL | Vipi Ltda., Pirassunga, SP, Brazil |
| Vipi Wave® (VW)   | Microwaved-processed | PMMA, benzoyl peroxide, pigments (11467) | 2.15 g/1 mL | Vipi Ltda., Pirassunga, SP, Brazil |

*According to the MSDS provided by the manufacturer. PMMA: polymethyl methacrylate; MMA: methyl methacrylate; EGDMA: ethylene glycol dimethacrylate

| Group | Polymerization cycle | Reference |
|-------|----------------------|-----------|
| WB (water-bath - conventional polymerization) | • immerse the flask in cold water; he heat water up to 65°C during 30 min and maintain for 1 h; heat water until boiling point during 30 min and maintain for 1 h; turn off the water-bath and let cool down | Manufacturer’s recommended cycle to cure VC resin |
| M630/25 | 270 W for ten min 0 W for five min 360 W for ten min | Manufacturer’s recommended cycle to cure VW resin |
| M550/3 | 550 W for three min | Ilbay et al.\[33\] |
| M650/5 | 650 W for five min | Patil et al.\[34\] |
There was no difference between microhardness values before and after thermocycling for both cycles recommended by the manufacturer, even for that indicated for polymerization of VW resin (microwave) when used for polymerization of VC resin (WB = 19.8 ± 0.9 to 19.9 ± 0.9 KHN; M630/25 = 18.6 ± 2.8 to 18.8 ± 0.5 KHN).

For VW resin, similar findings were observed when the polymerization cycles were compared to each other before thermocycling and the lowest microhardness value was also obtained for the cycle M550/3 (P = 0.001). However, for this material, only when the conventional water-bath cycle was used, microhardness values (20.6 ± 1.3 KHN) were not decreased after thermocycling (20.7 ± 0.7 KHN).

In all other groups, there was a reduction in hardness of VW after thermocycling (M630/25 = 21.0 ± 1.6 to 18.3 ± 0.9 KHN; M550/3 = 17.5 ± 3.0 to 15.7 ± 2.0 KHN; M650/5 = 20.9 ± 0.8 to 18.2 ± 2.0 KHN) (P = 0.000).

**Discussion**

Both null hypotheses tested that “there would be no difference in hardness between groups polymerized using recommended and experimental cycles for each material” and “thermocycling would not decrease the microhardness of materials polymerized in the different cycles” were not accepted.

It has been reported that the polymerization temperature and time interfere with the rate of polymer conversion. Also, the higher the temperature, the lower the residual monomer content. Hardness has been sensitive to the residual monomer content in the polymerized sample and has been directly related to the degree of dental polymer conversion. Since this is a simple and fast test, measurements of microhardness have been used as an indirect method to evaluate the polymerization of conventional heat-cured and self-cured acrylic resins.

In this study, the lowest hardness values were observed for samples polymerized in microwave using the cycle at 550 W for 3 min for both materials. Spartalis et al. evaluated the polymerization temperature in microwave reached at completion of the cycle of Vipi Cril and Vipi Wave resins in the same polymerization conditions analyzed in the present study. The authors observed that the lowest temperature values were achieved in M550/3 cycle (102.8 ± 12.6°C and 107.6 ± 18.8°C) for Vipi Cril and Vipi Wave resins, respectively. Moreover, in a previous study by Figuerôa et al., the highest solubility values of the both acrylic resins (Vipi Cril and Vipi Wave) were detected by using the microwave polymerization at 550 W for 3 min. Therefore, it is possible to assume that the lower temperature associated with a shorter polymerization as well as the higher leaching of the remaining unreacted monomer and other soluble constituents that might exert a plasticizing effect were responsible for the lower hardness values in this cycle.

Even though time and temperature interfere with the degree of conversion of materials, it was possible to polymerize the heat-cured acrylic resins in shorter cycles using temperatures higher than 100°C and achieve adequate conversion values, as observed in the evaluation of hardness in this study. Other studies evaluated the Knoop microhardness of conventionally polymerized denture base acrylic resins and obtained mean values in the range of 16.9–19.66 KHN. These values were similar to those observed in this study when the materials were polymerized using the recommended cycles (VC = 19.8 and VW = 21.0 KHN).

Both materials presented adequate hardness values, regardless of the polymerization cycle in which they were processed. Concerning the conventional water-bath cycle, utilization of the final temperature during at least 30 min of boiling leads to a reduced residual monomer content. This study evaluated the water-bath cycle recommended by the manufacturer that included 1 h of boiling for polymerization of both resins Vipi Cril and Vipi Wave. Reduction of the residual monomer is related to the increase in polymerization temperature, which results in greater mobility of molecular chains, enhancing the monomer conversion into polymer. Concerning the polymerization cycles in microwave, shorter polymerization time and lower residual monomer content are considered two advantages for this alternative processing. A possible explanation is related to the high frequency of mobility of monomer molecules inside the polymeric bulk during polymerization, causing fast internal heating and consequently leading to greater monomer conversion into polymer.

The similar performance of materials in this study is related to their similar composition. Truong and Thomasz also did not observe differences in composition between conventional or microwave heat-cured acrylic resins. Bonatti et al. related the similar outcomes of color stability to the fact that materials were made by the same manufacturer, consequently presenting similar composition.

It has been observed that thermocycling effectively reduces the mechanical properties of acrylic denture base resins, including hardness. This study evidenced a reduction in microhardness values of both materials after utilization of experimental cycles in microwave for Vipi Cril resin and after all microwave cycles for Vipi Wave resin, including the cycle recommended by the manufacturer for polymerization of this material. The denture bases are routinely subjected to thermal stresses in the oral cavity, especially during food and drink intake. These temperature variations may influence the water absorption of materials, which is a temperature-dependent process. The water absorption inside the resin is influenced by the polarity of polymethyl methacrylate molecules and by the diffusion of water molecules between interstitial spaces of polymeric chains. The thermal stress may influence the increase...
in water absorption by the polymer, increasing the distance of the polymeric chains. The increase in temperature (in that case, 55°C) may also allow faster diffusion of the water molecules inside the base resin. The absorbed water acts as a plasticizer, allowing the polymeric chains to move more easily over one another under load, and therefore the mechanical properties may be negatively affected. However, though statistically significant, the lower hardness values after thermocycling observed in this study were still close to those reported in the literature for nonaged specimens and thus they might not result in clinical significance.

Even after thermocycling, there was no reduction in the microhardness of Vipi Cril after accomplishment of the conventional cycle in water-bath and in microwave recommended by the manufacturer and of Vipi Wave after the conventional polymerization. The reduced hardness observed especially for cycles in microwave is due to the de-arrangement of polymeric chains under the action of microwave energy. In longer polymerization cycles, the polymers are formed more slowly, yielding longer polymeric chains with greater molecular weight compared to the microwave energy. Thus, it is possible that the polymeric structure formed in polymerization in microwave was more sensitive to the stresses induced by thermocycling. It was also observed that Vipi Wave was more affected by thermocycling than Vipi Cril. Another investigation conducted in the same conditions as the present study reported that, even though the resins presented the same chemical composition, it was likely that Vipi Cril presented greater concentration of ethylene glycol dimethacrylate as cross-linking agent than Vipi Wave, because the conventional resin presented higher flexural strength and modulus of elasticity values. Therefore, the polymeric structure achieved is denser and more resistant to the action of thermocycling.

It should be highlighted that this study analyzed only two brands of heat-cured resins. Additional studies on the physical–mechanical and biological properties and performance of materials after aging conditions are required to observe if faster polymerization cycles in microwave may be used for polymerization of conventional resins.

**Conclusion**

Within the limits of the study, the following conclusions were drawn:

- Both materials could be polymerized by the experimental cycle at 650 W during 5 min showing hardness similar to the cycles recommended by the manufacturer.
- Thermocycling did not decrease the hardness of Vipi Cril resin polymerized with water-bath and microwave cycles recommended by the manufacturer and Vipi Wave resin polymerized with the water-bath conventional cycle.

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**Conflicts of interest**

There are no conflicts of interest.

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