Effect of polymerization technique and glass fiber addition on the surface roughness and hardness of PMMA denture base material

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The current study evaluated the effects of autoclave polymerization both with and without glass fiber (GF) reinforcement on the surface roughness and hardness of acrylic denture base material. Ninety disc specimens (30×2.5 mm) were prepared from Vertex resin and divided according to polymerization techniques into a water bath, short and long autoclave polymerization groups. Tested groups were divided into three subgroups according to the GF concentration (0, 2.5, and 5 wt%). Profilometer and Vickers hardness tests were performed to measure surface roughness and hardness. ANOVA and Tukey-Kramer multiple comparison tests analyzed the results, and p≤0.05 was considered statistically significant. Autoclave polymerization significantly decreased the surface roughness and increased the hardness of acrylic resin without GF reinforcement (p<0.05). However, 5 wt% GF addition significantly increased surface roughness and decreased hardness of the autoclave polymerized denture base resin (p<0.05). Surface properties of Polymethyl methacrylate (PMMA) denture base material improved with autoclave polymerization and negatively affected with GFs addition.

Keywords: Autoclave polymerization, Glass fiber, Hardness, Surface roughness, PMMA denture base

INTRODUCTION

Polymethyl methacrylate (PMMA) acrylic resin has been the most popular and widely used denture base material for more than 60 years; however, it does not perform ideally⁴. In the past few years, PMMA polymerization techniques have been modified not only to improve PMMA's physical and mechanical properties but also to improve its working properties that facilitate laboratory processing techniques⁴,⁵. Various researchers have investigated modified procedures, using microwaves, visible light, and autoclave polymerization, all of which foster quicker PMMA polymerization while maintaining good material properties⁴,⁵.

To be competitive with the current PMMA manufacturing techniques, new polymerization techniques must provide results at least equivalent to the conventionally-approved water bath technique while also offering improved material properties. Charles Chamberland originally designed the autoclave technique in 1879 that is commonly used today in microbiology, medicine, and dentistry⁴,⁵. The autoclave is a device used for equipment sterilization by subjecting the contents to high-pressure saturated steam at 121°C for 15–20 min⁴,⁵. Using the same technique, it is possible to polymerize acrylic resin in a very short time⁵. Autoclave polymerization enhances the physical and mechanical properties of PMMA by increasing the degree of conversion and reducing the residual monomer content⁴,⁶ due to diffusion of the unreacted molecules into the polymer matrix by the pressure that is applied during autoclave polymerization. The applied pressure plays an important role in speeding up the initial polymerization by raising the temperature of the steam and elevating the boiling temperature of the monomer, which then may cause a reduction in the residual monomer content⁷. In addition, the increased temperature and pressure may enhance the degree of conversion resulting in lower level of residual monomer and subsequently lead to an improvement in the properties PMMA⁸-¹⁰. In previous studies²,⁶,¹¹-¹³, autoclave polymerization of PMMA has been used with different temperature and pressure levels; 55°C/2.5 bar¹¹, 120°C/6 bar¹², 121°C/2.1 bar¹², and 130°C/3 bar¹¹.

Alternative techniques that improve the mechanical and surface properties of the acrylic resins include the addition of glass fiber (GF) to the acrylic resin material¹⁴-¹⁶. Advantages of high mechanical properties, biocompatibility, wettability, favorable aesthetic qualities, and stability in the oral environment make GF a suitable material for acrylic resins’ reinforcement¹⁴,¹⁵. The efficiency of GF reinforcement is influenced by various factors, such as quantity, length and the orientation of GF²². Furthermore, GF treatment with a silane coupling agent increases the bond between fiber and resin matrix¹⁹,²⁰. Keyf et al. reported that GF addition without silane treatment might deteriorate the acrylic resin, as the GFs would not be bonded to the resin and may act simply as another material inside the resin²⁰.
A variety of physical properties can be used to assess the strength of denture materials, and it is important to determine the surface roughness and hardness of PMMA materials, as these properties influence the clinical performance and longevity of this material\(^{20}\). Rougher denture base surfaces are annoying to patients, leading to discoloration, increasing plaque accumulation, and bacterial and fungal adhesion\(^{24-26}\), affecting both the aesthetic appeal and longevity of the denture\(^{27}\). Therefore, the surface roughness properties of a denture base material should be evaluated prior to clinical usage. Moreover, when dentures are manufactured with harder acrylic resins, they are more resistant to all forms of damage\(^{10}\). Because the denture base material is subjected to scratching and abrasion during denture wear or mechanical cleansing, the hardness property indicates the proper material selection. Furthermore, resin materials with low surface hardness are liable to damage through brushing, which eventually results in increased surface roughness\(^{27}\). The appropriate method to evaluate the surface hardness is the Vickers hardness (micro-hardness) test, which determines the ability of the material to resist the penetration of a specific load\(^{19,24,28,29}\).

Although several studies have investigated the effects of the addition of GFs to PMMA denture bases on their strength and fracture resistance, the effects of GFs’ addition on PMMA’s surface properties have not been well evaluated. Moreover, there were only a few investigations that assessed the effects of autoclave polymerization on the surface properties of acrylic resins. Hence, the effect of autoclave polymerization on the properties of GF-reinforced PMMA denture bases has been previously neglected. Therefore, this study evaluated the effects of autoclave polymerization with and without GFs reinforcement on the surface roughness and hardness properties of PMMA denture base materials. The first null hypothesis is that the autoclave polymerization does not improve the surface roughness and hardness properties of PMMA denture base materials. The second null hypothesis is that the GFs addition do not affect the surface roughness and hardness.

## MATERIALS AND METHODS

A total of 90 disc-shaped (\(n=10\)) (30 mm diameter×2.5 mm thickness) specimens were prepared from heat-polymerized denture base material (Vertex Dental, Zeist, The Netherlands). According to polymerization techniques, specimens were divided into three groups. Each group was divided into 3 subgroups according to GFs concentrations with ten specimens included within each group (\(n=10\)) (Table 1). For all groups, 90 wax models were prepared for the specific size of specimens and then were contained within metal flasks (61B Two Flask Compress, Handler Manufacturing, Westfield, NJ, USA) using a dental stone (Fujirock EP, GC, Leuven, Belgium). Next, flasks were placed into boiling water for 5 min to melt away all waxes and create mold spaces for the acrylic resin. After that, a separating medium (Isolmajor, Major Prodotti Dentari, Moncalieri, Italy) was applied to the exposed stone surface and then allowed to dry.

For the reinforced groups, GFs (E-glass, length=3 mm, diameter=12 μm, Shanghai Richem International, Shanghai, China) were weighed to create 2.5 and 5 wt% of acrylic powder on the electronic balance (S-234, Denver Instrument, Bohemia, NY, USA). Pre-weighed GFs were treated with silane coupling agent (3-trimethoxysilyl propyl methacrylate, 97%) (gamma-MPTS) (Shanghai Richem International) for the acrylic resin. After that, a separating medium (Isolmajor, Major Prodotti Dentari, Moncalieri, Italy) was applied to the exposed stone surface and then allowed to dry.

Table 1 Specimens grouping, codes, and description of denture base material with different GFs concentrations and polymerization techniques

| Group | Subgroups and codes | Denture base material and GFs concentrations | Polymerization technique |
|-------|---------------------|---------------------------------------------|-------------------------|
| I     | WB                  | Unreinforced acrylic resin (Control group)  | *Conventional water bath polymerization processed at 74°C for 8 h and then increasing temperature to 100°C for 1 h. |
|       | 2WB                 | Acrylic resin reinforced with 2.5 wt% GFs  | **Autoclave polymerization (short cycle); Specimens processed by the programmed cycles (60°C/2.1 bar, for 30 min followed by 120°C for 10 min) |
|       | 5WB                 | Acrylic resin reinforced with 5 wt% GFs    | **Autoclave polymerization (long cycle); Specimens processed by the programmed cycles (60°C/2.1 bar, for 30 min followed by 120°C for 20 min) |
| II    | AS                  | Unreinforced acrylic resin                 | **Autoclave polymerization (short cycle); Specimens processed by the programmed cycles (60°C/2.1 bar, for 30 min followed by 120°C for 10 min) |
|       | 2AS                 | Acrylic resin reinforced with 2.5 wt% GFs  | **Autoclave polymerization (long cycle); Specimens processed by the programmed cycles (60°C/2.1 bar, for 30 min followed by 120°C for 20 min) |
|       | 5AS                 | Acrylic resin reinforced with 5 wt% GFs    | **Autoclave polymerization (long cycle); Specimens processed by the programmed cycles (60°C/2.1 bar, for 30 min followed by 120°C for 20 min) |
| III   | AL                  | Unreinforced acrylic resin                 | **Autoclave polymerization (long cycle); Specimens processed by the programmed cycles (60°C/2.1 bar, for 30 min followed by 120°C for 20 min) |
|       | 2AL                 | Acrylic resin reinforced with 2.5 wt% GFs  | **Autoclave polymerization (long cycle); Specimens processed by the programmed cycles (60°C/2.1 bar, for 30 min followed by 120°C for 20 min) |
|       | 5AL                 | Acrylic resin reinforced with 5 wt% GFs    | **Autoclave polymerization (long cycle); Specimens processed by the programmed cycles (60°C/2.1 bar, for 30 min followed by 120°C for 20 min) |

*Curing unit: KaVo Elektrotechnisches Werk D-88299, Germany.
**Autoclave: Ritter M11 UltraClave, Midmark International, Spain.

(WB): Water bath; (A): Autoclave; (S): Short cycle; (L): Long cycle; (2): 2.5 wt% GFs; (5): 5 wt% GFs; kPa: kilopascal.
mixer for 1 min. Then they were mixed with PMMA powder and stirred with a blender at a rotating speed of 400 rpm for 30 min to achieve an equal distribution of fiber within acrylic powder.

According to the manufacturer’s instructions, acrylic resin polymers (with and without GFs reinforcement) were dispersed in a methyl methacrylate monomer with a monomer/polymer mass ratio of 0.95 g liquid/2.3 g powder mixed and packed at the doughy stage into the molds. Then, flasks were closed and pressed under 20.68 MPa using the hydraulic pressure machine (WW-33 Hydraulic press, Wassermann Dental-Machinen, Hamburg, Germany) and kept at room temperature for 1 h. The flasks with acrylic resins were subjected to one of the polymerization techniques: the water bath method, a short autoclave polymerization cycle, or a long autoclave polymerization cycle (Table 1). After the specimens’ polymerization was completed, the specimens were finished and polished according to the protocol traditionally prescribed by the ISO 20795 standard No.12. Excess resins were removed with a tungsten carbide burr (HM251FX-040-HP, Meisinger, Centennial, CO, USA) then using a 600-grit abrasive paper under running water. Next, a soft bristle brush with fine pumice dust (Steribim super, Bego, Bremen, Germany), mixed to an equal volume of water, was used for polishing and stored in distilled water at 37°C for 72 h.

Testing procedures
A noncontact optical interferometric Profilometer (Contour Gt-K1 optical profiler, Bruker Nano, Tucson, AZ, USA) was used to measure the surface roughness (Ra) of the acrylic specimens at a 0.01 mm resolution. Each specimen was placed horizontally under a standard camera 20×, and scanned an area approximately 0.43×0.58 mm. Each specimen was scanned on its surface at 5 sites radially (across each specimen), and the average of the indentation was measured by microscope, and the hardness values were assessed. A total of five indentations were made at different points (the same selected areas for each specimen), on each specimen, and the means of individual specimens were calculated.

Statistical analysis
The statistical analyses of the obtained data were performed using a one-way analysis of variance test (ANOVA) (IBM SPSS Statistics). A Tukey-Kramer multiple-comparison test was used for results’ analysis, which showed statistical significance at p≤0.05.

RESULTS
Table 2 presents the mean values and standard deviation (SD) of surface roughness (Ra) for all tested groups. Generally, autoclave polymerization showed significantly lower surface roughness values (Ra) when compared to the water bath method (p<0.05). Statistical

| Group | Codes | Surface roughness | | | Hardness | |
|-------|-------|-------------------|---|---|-------------|---|
| | | Mean±SD | SDS | | Mean±SD | SDS |
| I | WB | 0.115±0.006 | AL, AS, 2AL, 2AS, 5AL, 5AS, 2WB, 5WB | 19.3±0.36 | 5AS, 5AL, 2AS, 2AL, AS, AL |
| | 2WB | 0.124±0.003 | AL, AS, 2AL, 2AS, 5AL, 5AS, WB, 5WB | 18.8±0.31 | 5AS, 5AL, 2AS, 2AL, AS, AL |
| | 5WB | 0.129±0.005 | AL, AS, 2AL, 2AS, 5AL, 5AS, WB, 2WB | 19.5±0.38 | 5AL, 2AS, 2AL, AS, AL |
| II | AS | 0.072±0.002 | AL, 2AS, 5AL, 5AS, WB, 2WB, 5WB | 24.4±0.31 | 2WB, WB, 5WB, 5AS, 5AL |
| | 2AS | 0.078±0.006 | AL, AS, 5AS, WB, 2WB, 5WB | 23.16±0.5 | 2WB, WB, 5WB, 5AS, AL |
| | 5AS | 0.087±0.004 | AL, AS, 2AL, 2AS, WB, 2WB, 5WB | 21.66±0.3 | 2WB, WB, 2AS, 2AL, AS, AL |
| III | AL | 0.065±0.003 | AS, 2AL, 2AS, 5AL, 5AS, WB, 2WB, 5WB | 25.1±0.25 | 2WB, WB, 5WB, 5AS, 5AL, 2AS |
| | 2AL | 0.073±0.004 | AL, 2AS, 5AL, 5AS, WB, 2WB, 5WB | 23.5±0.21 | 2WB, WB, 5WB, 5AS |
| | 5AL | 0.083±0.002 | AL, AS, 2AL, WB, 2WB, 5WB | 22.7±0.17 | 2WB, WB, 5WB, AS, AL |

Multiple comparison tests for all pairwise differences between the means. SDS: Statically significant difference from group p≤0.050.
analysis showed that the 5WB had significantly the highest surface roughness value (0.129±0.005), while the AL represented the lowest surface roughness mean values (0.065±0.003 μm) among the tested groups (Table 2). Within group I, statistical analysis showed significant differences between WB (0.115±0.006) and 2WB (0.124±0.003) & 5WB (0.129±0.005) subgroups. For group II, AS significantly showed the lowest surface roughness compared to 2AS and 5AS while 5AS significantly showed the highest surface roughness. For group III, AL significantly showed the lowest surface roughness compared to 2AL and 5AL while 5AL significantly showed the highest surface roughness. Within unreinforced subgroups, WB showed higher Ra value while AL showed the lowest values significantly. Within the reinforced subgroups, 5WB showed the highest Ra while the 2AL had significantly the lowest surface roughness as compared to the other reinforced tested subgroups. Figures 1, 2, and 3 show the average surface roughness values by depicting this parameters in two colors: red and blue. Red exhibited the peaks’ height while the blue represented the valleys’ depth from a mean line. The graduated colors between red and blue, and readings on the top and bottom of the parameter, represented the whole surface roughness. Figure 4 showed SEM images for surface characteristics before GFs addition (Figs. 4A, E) and after GFs addition (Figs. 4B, C, D, F). Some fibers present as boundless on the surface in horizontal direction (Figs. 4B and 5B) while others emerge longitudinal with the level of the specimen’s surface or come out resulted in the change of surfaces (Fig. 5A).

The mean and SD values of the Vickers hardness (VH) for each tested group are represented in Table 2. The statistical analysis showed that the autoclave polymerization groups had significantly the highest VH mean values in comparison to water bath group (p<0.05) except 5WB compared to 5AS (p>0.05). It was noted that
DISCUSSION

The current study evaluated the effect of autoclave polymerization and GFs addition on surface roughness and hardness of PMMA denture base material in comparison to water bath polymerization. The study results showed that the autoclave polymerization cycles significantly improved the Ra and hardness ($p<0.05$) and GFs addition significantly affected the Ra and hardness ($p<0.05$), thus both null hypotheses were rejected.

Based on the results of the current study, autoclave polymerization led to a statistically significant reduction in the materials' surface roughness. The improvement in Ra values could be attributed to the levels of heat and pressure during autoclave polymerization, where the increase in the degree of conversion reduced the residual monomer and also improved surface roughness\(^{31}\). Autoclave polymerization significantly showed a lower Ra value in comparison to the control group, which was in agreement with previous research\(^{32}\). However, this study's findings were in disagreement with Nazhat \(et\ al.\)\(^{33}\) who found that there were no significant differences between the autoclave polymerization and the water bath methods. These variations may be due to using a different autoclave polymerization cycles and/or another resin type.

Despite the lack of sufficient scientific evidence on the effects of different polymerization methods with GF reinforcement for the purpose of comparison, the results of the present study showed that the autoclave polymerization of acrylic resins with GF reinforcements increased the Ra values. Any addition of GFs resulted in significant increase in Ra between unreinforced, 2.5 and 5 wt% GFs subgroups which declared that Ra values increased as GFs concentrations increased in all tested groups. This factor implies that the fibers' effects depend on their concentrations\(^{22}\). The increase of surface roughness can be explained by the random arrangement of the GFs at the surface and the possibility of protruding GFs from the surface (Figs. 4B and 5A). Moreover, this increase may be attributed to specimens' surface defects due to the lack of bonding between the acrylic resin and GFs (Fig. 5B). Although the high roughness values recorded with 5%GFs, the surface roughness of the tested groups were lower than the reported clinically acceptable value (0.2 μm)\(^{34}\).

The results of the surface hardness test revealed that the autoclave polymerized groups presented the higher VH values when compared to WB group. Heat and pressure inside the autoclave led to a high degree of conversion\(^{35}\) which consequently increased the materials' hardness property and decreased residual monomer content\(^{11,36}\). It was reported that hardness values are directly proportional to the amount of residual monomer content\(^{37,38}\). Additionally, the plasticizing effect of residual monomer, that weakens the surface hardness, was reduced due to high monomer conversion\(^{11,36}\). In agreement with results of the current study, previous studies by Ayaz \(et\ al.\)\(^{11}\) and Ali \(et\ al.\)\(^{39}\) found that the maximum strength could be attained with high temperature in the processing unit ensured complete polymerization due to high monomer conversion. In contrast, Ming \(et\ al.\)\(^{12}\) found that there was no statically significant difference between the autoclave and the
Fig. 4 Representative SEM micrographs images of tested specimens.
A: (WB); B: (5WB); C: (2AS); D: (5AS); E: (AL); F: (2AL).

Fig. 5 Representative SEM micrographs images with high magnifications of tested specimens.
A: (2AL); B: (5AL).
water bath polymerization methods. This difference may be due to using another resin type and/or different polymerization cycles. In addition, they used a pressure cooker filled with water, inflated with 6 bar air pressure, heated to 120°C, and maintained for 10 min.

The present study demonstrated a decrease in the VH values of acrylic resins when GF was added to the polymers matrix as compared to unreinforced groups. Among the GF reinforced groups, in autoclave groups, the addition of 5 wt% GF showed a significant decrease in VH values in comparison to unreinforced groups, while no significant difference between unreinforced and 2.5% GFs groups which confirmed that VH did not affect with low concentration while decreased with 5% GF concentrations. Alternately, this study found that during specimens’ fabrication there was a possibility of the GFs overlapping and clustering creating structural defects at the surface of the specimen, which might affect the integrity of the matrix, and this possibility became greater as the GF concentration increased. Chen et al. (20) found that the random orientation and alignment of the GFs produced the lowest indentation resistance, and a higher percentage of residual monomer content, which has a plasticizing effect (13), reduced the resistance of the material (41).

Finally, autoclave polymerization improved both surface roughness and hardness of the tested material, in addition to introducing easier laboratory work and time-saving procedures for removable prosthesis fabrication. Consequently, autoclave polymerization could be used as an alternative method instead of the conventional water bath polymerization technique. Moreover, silanized GFs addition in low concentrations to autoclave polymerized PMMA seems to be the most effective technique among the tested materials for denture base material, whereas many studies used GFs primarily to improve the mechanical properties (4, 16, 18, 40). So we would recommend of GFs addition in low concentrations to prevent the deterioration of the materials’ surface properties.

The limitations of this study included using of bar specimens alternative to multifaceted denture shapes as well as the absence of environment that mimic oral conditions and absence of aging procedures as thermal cycling effects. Further research should be conducted to investigate the effects of autoclave polymerization on GF-reinforced PMMA resin; particularly its effects on mechanical properties and biocompatibility, taking into consideration aging factors to obtain the most appropriate material for denture base construction. Despite the autoclave polymerization advantages, elevated temperature may limit its use (22). So, the results of this study must be interpreted with cautions, because a different performance can be obtained with different temperature and pressure levels which indicate further investigations.

CONCLUSION

Within the limitations of this study, autoclave polymerization significantly reduced the surface roughness and increased the hardness of the acrylic resin denture base material and could be used as an alternative to the water bath polymerization method. GF reinforcement negatively affected the surface roughness and the hardness properties of autoclave polymerized PMMA denture base resin, particularly at high GF concentrations.

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CONFLICT OF INTERESTS

Authors declare that they have no conflict of interests regarding the publication of this manuscript.

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