COMPARISON OF POLYMENTH METHACRYLATE CURING BY CONVENTIONAL HOT-WATER BATH AND MICROWAVE ENERGY
Gauri Madan, Sonal Madan, Gautam Madan

ABSTRACT
Background: Microwave irradiation is a cleaner, more time-efficient, more cost-effective, and less cumbersome method of polymerisation of denture base resins. Aims and objectives: To assess the transverse strength and residual monomer content of Polymethyl Methacrylate (PMMA) cured by conventional hot-water bath and microwave irradiation. Materials and Method: A preliminary experiment was conducted with twenty-six specimens of acrylic resins made by standard compression molding to find the optimum-curing program for microwave oven curing. Twenty specimens of 65mm x 10mm x 2.5mm were cured by both techniques to compare the transverse strength. Residual monomer content of PMMA cured by conventional hot-water bath and microwave irradiation was assessed by curing twenty specimens of 10mm x 10mm x 0.5mm measurements. SPSS V 16 was used to compare the Mean and significance between groups. Results: The microwave-cured specimens at the optimum curing cycle had statistically significant higher transverse strength and no difference in degree of polymerization than the conventional cured specimens. Conclusion: The transverse strength and residual monomer content of denture base material are optimum in microwave-cured acrylic.

Key words: Microwave Polymerisation; Polymethyl Methacrylate; Residual Monomer Content; Transverse Strength

Introduction
Acrylic polymers were introduced as a denture base material in 1937.1,2 Polymethylmethacrylate (PMMA) acrylic polymers are the most popular denture base material.3,4 The use of microwave energy was first reported in 1968 by Nishii5 as an alternative PMMA processing method and has become increasingly popular as to conventional water-bath processing.6,7 The advantages of this technique are a shortened dough forming time, more homogenous dough, a shorter curing time, minimal colour changes in the cured resin, cleaner and more time efficient method.8 New resins such as visible light-cured resins, rapid boil-out resins and resins specially designed for use in microwave polymerization are already in the market.9 The main aim of this study was to compare the transverse strength and residual monomer content of Polymethyl Methacrylate cured by conventional hot-water bath and microwave energy.

Materials and Methods
This in vitro study was conducted on 46 specimens made by pouring molten wax into metal mold that was of four types (Table 1). The conventional clear heat-cure denture base material with no crosslinking agent was used for specimen preparation. Compression molding was used for fabrication of all specimens in this study using standardized methods. Preliminary experiments were conducted on twenty-six specimens to find the optimum-curing program of the given microwave oven and the optimum program was used to cure the specimens for comparing the transverse strength and residual monomer content with conventional hot-water bath curing. Twenty type C specimens were used to compare transverse strength of cured resin by conventional hot-water bath and microwave energy. Conventional hot-water bath and microwave curing of twenty type D specimens were carried out to assess the residual monomer content of polymethyl methacrylate in the cured specimens.

<table>
<thead>
<tr>
<th>Type</th>
<th>N</th>
<th>Dimensions of the mold</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type A</td>
<td>13</td>
<td>55mm x 10mm x 2.5mm</td>
</tr>
<tr>
<td>Type B</td>
<td>13</td>
<td>30mm x 10mm x 10mm</td>
</tr>
<tr>
<td>Type C</td>
<td>20</td>
<td>65mm x 10mm x 2.5mm</td>
</tr>
<tr>
<td>Type D</td>
<td>20</td>
<td>10mm x 10mm x 0.5mm</td>
</tr>
</tbody>
</table>

Table 1. Measurement of Specimen for each study

Acrylic Curing: The acrylic was mixed strictly as instructed by the manufacturers. Special care was taken during the mixing and processing of the polymer and thereby eliminating their effect on porosity. Conventional packing procedures were carried out. Both types of flasks were allowed to stand for 30 minutes bench-curing.

Conventional hot water-bath curing: This method was carried out in a thermostatically controlled hot water-bath (+2°C). The curing tank had an AC power source of 230V, 1650W. It had three curing stations for low temperature, intermediate temperature and highest temperature. Each curing station had its own temperature and time control setting. The curing cycle used for all the specimens used by the hot water-bath curing method was the most recommended medium-curing cycle: 72°C for 7 hours followed by 100°C for 3 hours. The specimens thus obtained were subjected to transverse strength tests and residual monomer content estimation.
Microwave Curing: The curing by microwave curing method was carried out in a domestic microwave oven (Model 800G, BPL Electronics). This microwave oven had a power source of 220V, 50 MHz and a maximum power output of 800W with six wattage settings and separate time control. The optimum microwave-curing program was found by preliminary experiments to cure thin and thick specimens by using various time and wattage combinations. A series of experiments was carried out to determine the best curing conditions with the aim of avoiding two extremes like rapid heating that results gaseous porosity from the volatilization of monomer; or undercuring leading to excessive residual monomer in the resin.

Preliminary Experiment: Thirteen thick (type A) and thirteen thin (type B) acrylic resin specimen of the dimensions mentioned in Table 1 were prepared by curing the acrylic resin by curing programs using various time and wattage combinations as listed in Table 2. The acrylic specimens thus obtained were assessed by visual observation for porosity by a subjective scale of none, slight, moderate or high porosity. The specimens were also examined subjectively for hardness and smell of monomer to assess the degree of cure. The curing program that produced a specimen with no porosity in minimal time was chosen as the optimum microwave-curing program.

Transverse Strength Tests: Ten type C specimens were prepared by both curing methods. The specimens were stored in distilled water at 37°C for 48 hours before testing. A three-point bending test as suggested by the ADA specification No. 12 for denture base polymers was used. The specimens were loaded to fracture using a Lloyds Universal Testing Machine. The crosshead speed was 2.8m/min and the length of span between the two supports for the specimen was 50mm. The load at rupture was noted. The width and thickness of each specimen was measured by a digital micrometer and the transverse strength was calculated by Singer’s formula.

Estimation of Residual Monomer Content: Ten type D specimens were prepared by both curing methods and the acrylic chips were broken into a fine powder by a hammer. mg of the acrylic dust sample from each method was added to 25ml of each methanol in a conical flask. They were then heated at 65°C under reflux using a single surface condenser having 30cm cooling length and 1cm internal diameter for 75 minutes. The flask was raised 2mm above the hot plate to avoid bumping. The polymer residues were then filtered off. The supernatant methanolic extract obtained in this manner was set aside for analysis.

Gas chromatography was done by a Gas Chromatograph for Head Space Analysis at the Forensic Science Laboratory, Ahmedabad. 1µl methanolic extract was injected into the glass column 2M long, having internal diameter 3.5mm and coated with 15% Carbowax (polyethylene glycol 20,000). The oven temperature was 80°C, the injection temperature was 180°C, the thermostat temperature was 50°C, the thermostat time was 20min. and the flame ionization detector temperature was 150°C. The injection time was 0.05 min. Nitrogen was used as the carrier gas at a pressure of 35 PSI. The detector output was linked to a customized software. The monomer content was determined by comparing the area under the curve as obtained by the use of standard solutions of known amounts of monomer in methanol.

Results

Preliminary Experiments: The results of the preliminary experiments to find the optimum curing program are shown in Table 2. The curing programme, which produced a cured acrylic specimen with no porosity in minimal time, was: Programme K: 80W for 17 minutes, followed by 150W for 1 minute, followed by 450W for 2 minutes with total 20 minutes.

Transverse Strength: Table 3 shows the load at rupture and calculated transverse strength for specimens cured by con-
ventional hot-water bath and microwave energy curing method with the mean transverse strength and standard deviation of transverse strength.

**Residual Monomer Content Estimation:** The calibration curves for both conventional and microwave-cured specimen extracts in methanol were similar and did showed no peak for carbon-carbon double bond indicating presence of methyl methacrylate. No residual monomer content was found in the acrylic dust samples of both types.

**Discussion**

In finding the optimum microwave–curing program, the goal was to completely polymerize the acrylic resin without porosity in the polymerized specimen. Porosity in a polymerized resin can be caused by an excessive rise in the exothermic temperature during polymerization, by under packing the resin or by insufficient pressure during packing procedures. Since the packing procedures were standardized, the porosity was assumed to be of the gaseous type, which arises as a result of overheating or boiling of the monomer at a temperature higher than its boiling point 100.8°C. This occurs when the temperature rises rapidly when a substantial quantity of unreacted monomer is still present in the mass. In this study, porosity was observed in thicker sections and when high wattage for short time period was used. When the wattage was lowered and the curing time lengthened, porosity was reduced. This is in agreement with the previous authors. By lowering the wattage and increasing the time, microwave–curing can be accomplished without porosity. The curing program advocated by Kimura et al was 90W for 13minutes followed by 500W for 2minutes. This can be considered similar to the curing program A of the preliminary experiments i.e. 80W for 13minutes followed by 450W for 22minutes. This program found no porosity in the thin specimen but the thick specimen contained moderate amount of porosity. Though denture bases are rarely more than 3mm thick, no porosity in cured thick specimens indicates complete polymerization with suitable physical properties. Lengthening the time for which the 80W power is used and then increasing the power to 150W ensures that the temperature rise is gradual and the polymerization occurs without overheating or boiling the monomer. The final 2minute period of curing at 450W ensures complete polymerization with no residual monomer content and can be correlated with the terminal boil used in the hot water-bath curing methods.

Gas chromatography is an established method of determining the residual monomer content. The extraction in methanol using reflux has the advantage of faster and complete extraction. Tissue sensitivity to denture base polymers depends on the quantity of residual monomer. It is also known that a high level of residual monomer content decreases the strength of denture base polymers. No residual monomer was found in both the types of specimens. This proves that both the methods produced complete polymerization. This is in agreement with the findings of Shlosberg et al.

The transverse strength of the microwave-cured specimens was found to be significantly higher than that of conventionally cured specimens. This correlated with the findings of Smith et al. Yet, the transverse strength of both types of specimens was higher than found in previous studies. In this study, only one brand of acrylic resin was used. The optimum curing microwave curing program for another brand may be different. If a microwave oven, which has lower wattage setting, such as 60W, is used, then another optimum microwave curing program may be found with perhaps even less time required for polymerization. Further research should be done to fabricate, indigenously, flasks, which can be used in microwave ovens as suggested in a method by McKinstry. The minimum time required for dewaxing by conventional method is 10minutes, while dewaxing by microwave method takes only 3-4minutes. The total time required for the suggested microwave curing method including the time required for bench curing and cooling is 80minutes. The minimum time required for the conventional hot water- bath-curing method excluding the time required for bench curing and cooling is 2hours up to a maximum of 14hours. The fiber-reinforced flasks are lighter than the heavy metal flasks and clamp.

**Conclusion**

In conclusion, the two most important properties, transverse strength and residual monomer content of denture base material are optimum in microwave cured acrylic. Thus microwave curing is a time efficient, cost effective and cleaner method that requires less cumbersome equipment.

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### Table 3. Comparison of transverse strength following two curing methods for each specimen (* kgf ** kgf/cm²)

<table>
<thead>
<tr>
<th>Microwave Curing</th>
<th>Load at Rupture*</th>
<th>Transverse strength**</th>
<th>Load at Rupture*</th>
<th>Transverse strength**</th>
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<tr>
<td>10.42</td>
<td>1094.577</td>
<td>11.44</td>
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<td>169.983</td>
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<td>61.419</td>
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References


How cite this article


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Source of Support: Nil
Conflict of Interest: None Declared