Effect of repair resin type and surface treatment on the repair strength of polyamide denture base resin

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The purpose of the present study was to evaluate the effects of different repair resins and surface treatments on the repair strength of a polyamide denture base material. Polyamide resin specimens were prepared and divided into nine groups according to the surface treatments and repair materials. The flexural strengths were measured with a 3-point bending test. Data were analyzed with a 2-way analysis of variance, and the post-hoc Tukey test ($\alpha=0.05$). The effects of the surface treatments on the surface of the polyamide resin were examined using scanning electron microscopy. The repair resins and surface treatments significantly affected the repair strength of the polyamide denture base material ($p<0.05$); however, no significant differences were observed interaction between the factors ($p>0.05$). The flexural strength of the specimens repaired with the polyamide resin was significantly higher than that of those repaired with the heat-polymerized and autopolymerizing acrylic resins.

**Keywords**: Polyamide resin, Surface treatment, Repair strength

INTRODUCTION

The aim of prosthetic rehabilitation is to improve masticatory function, esthetics, and speech. Although different materials have been used for denture base materials, polymethyl methacrylate (PMMA) has been extensively used since 1937\(^1\),\(^2\). PMMA has the advantages of ease of manipulation and repair, accurate fit, ease of polishing, esthetic appearance, and stability in the oral environment\(^1\),\(^3\),\(^4\). However, PMMA also has several disadvantages, including allergic reactions to the residual monomer, insufficient surface hardness, poor wear resistance, and polymerization shrinkage\(^1\),\(^5\),\(^6\). Additionally, PMMA does not have the optimum mechanical properties of fracture resistance and flexural strength\(^7\),\(^8\). Denture base materials are subjected to compressive, tensile, and shear stresses during function, and fractures in the denture base may develop through repeated masticatory forces or high impact forces that may occur as a result of dropping the prosthesis\(^9\),\(^10\).

There have been different methods used to improve the mechanical properties of PMMA, such as modifying the PMMA chemical structure and reinforcing the PMMA with various fibers, metal wires, and frameworks\(^10\),\(^11\),\(^12\). However, cast metal frameworks have the disadvantages such as corrosion of the metal, allergic reactions, and the unappealing appearance of metal clasps\(^10\). Another method for improving the physical properties of the prosthesis is injection molding\(^13\) and the SR-Ivocap system (Ivoclar Vivadent AG, Schaan, Liechtenstein), which is an injectable PMMA\(^14\). In addition, alternative materials such as polyamide should be used for improving the physical properties of the prosthesis\(^10\).

Polyamide resins are thermoplastic polymers synthesized by the condensation reaction between a diamine and a dibasic acid. The use of nylon as a denture base material has been described since 1950. The early form of polyamides exhibited problems including high water sorption, surface roughness, and difficulty in polishing. Recently, the stiffness of the material was increased and flexibility was improved with glass fiber reinforcement\(^10\). Additional advantages of polyamide resins include superior esthetics, reduced allergic reactions, and decreased stress on abutment teeth\(^17\),\(^18\). However, the repair of a polyamide prosthesis is difficult and expensive, and often, the creation of a new prosthesis is more desirable than repairing a fractured polyamide prosthesis\(^10\).

There are a limited number of studies in the literature evaluating the repair of polyamide denture base materials. Therefore, the purpose of the present study was to investigate the effects of polyamide resin, heat-polymerized acrylic resin, and autopolymerizing resin used as repair materials and surface treatments with a methyl methacrylate monomer and ethyl acetate on the repair strength of a polyamide denture base material. For this research, firstly it was hypothesized that the repair resin type would not increase the repair strength of the polyamide denture base resin and secondly it was hypothesized that the surface treatments would not increase the repair strength of the polyamide denture base resin.

MATERIALS AND METHODS

Table 1 lists the materials used in the present study. Using a power analysis, it was determined that 72 specimens were required to detect a significant difference among 3 repair resins and 3 surface treatments (a total of 9 groups) on the repair strength of a polyamide
denture base resin. Seventy-two rectangular polyamide resin specimens (65.0×10.0×2.5 mm³) were prepared according to the American Dental Association Specification No. 12. The PR specimens were fabricated using an injection molding machine (Deflex MAD, Deflex, Nuxen SRL, Buenos Aires, Argentina) with an injection pressure of 0.6 MPa at 280°C and a preheating time of 15 min. The specimens were sectioned into 2 halves to create a repair gap (3.0×10.0×2.5 mm³) and then retention chamfers were prepared the bonding surfaces of all specimens with a tungsten carbide bur (Fig. 1). The bonding surfaces of all specimens were finished with 600-grit abrasive paper under running water (Norton, Saint-Gobain Abrasivos, São Paulo, Brazil).

The PR specimens that were processed into 2 halves were embedded in the denture flask, with a metal mold (3.0×10.0×2.5 mm³) placed in the center of the repair gap for standardization. The denture flask was then opened, the metal mold that formed the repair gap was removed, and the PR specimens were divided into 3 equal groups (24 specimens per group) according to the surface treatments: a group without any surface treatment (Control); treated with methyl methacrylate monomer for 120 s (MMA); and treated with ethyl acetate for 120 s (EA).

After surface preparation, PR, HP, or AP were applied to the repair gaps of the surface-treated and control specimens. The PR specimens were processed as described earlier. The HP specimens were polymerized by keeping the denture flask in a thermal chamber (Termotron P-100, Termotron do Brazil, São Paulo, Brazil) for 9 h once it reached the boiling temperature (74°C) using the long boiling method. The AP specimens were polymerized by keeping them under pressure at 55°C for 15 min. This process was carried out to enhance the strength and decrease porosity. After the polymerization processes, the specimens were carefully removed from the denture flask, and the residual repair resin was removed with a tungsten carbide bur at low speed. The specimens were molded to the final shape with 600-grit abrasive paper under running water. Eight specimens were made for each group, and were stored in distilled water at 37°C for 1 week before testing.

A three-point bend test was performed immediately after removing the specimens from the distilled water, without drying the specimens. This test was carried out on a universal testing machine (Model 2519-106, Instron, Northwood, MA, USA). A custom-made stainless steel device with a 50 mm span distance between the 2 supports was used, and the crosshead speed was set at 5 mm/min. A load was applied to the center of the specimens (center of the repair area). The specimens were loaded until the specimens fractured, and the maximum fracture load was recorded.

The flexural strength values of each specimen were calculated using the following formula: $S = \frac{3WL}{2bd^2}$, where $S$ is the flexural strength (in MPa), $W$ is the maximum fracture load (in Newtons), $L$ is the distance between the supports (50 mm), $b$ is the specimen width (10 mm), and $d$ is the specimen thickness (2.5 mm).

To evaluate the effects of the surface treatments on the surface of the denture base resin, 3 specimens (1 specimen each from the Control, MMA, and EA groups) were selected before repair process. These selected specimens were gold-sputtered and examined under a field emission scanning electron microscope (SEM) (Zeiss EVO LS 10, Carl Zeiss, Oberkochen, Germany) at 10.0 kV. The SEM photomicrographs were created using 2,000× magnification for visual inspection. In addition, the nature of the failure was noted as adhesive (interface), cohesive (only at the repair material), or mixed (interface and repair material).

A two-way analysis of variance (ANOVA) was used to study the effects of the repair resin type, surface treatments, and their interaction on the flexural strength, followed by the Tukey test with a confidence
level of 0.05 to determine the mean differences. These analyses were performed with SPSS statistical software (SPSS v16.0, SPSS, Inc, Chicago, IL, USA).

RESULTS

The two-way ANOVA results are presented in Table 2. Significant differences were found for repair resin type ($p<0.001$) and surface treatments ($p<0.05$); however, no significant differences were observed interaction between the factors ($p>0.05$). The mean and standard deviation values of the flexural strength for each of the experimental groups are presented in Table 3.

The flexural strength of the specimens repaired with PR was significantly higher than with HP and AP ($p<0.001$); however, no significant differences were found between the HP and AP groups ($p>0.05$). The flexural strength values of the PR group were 37.70 MPa to 46.25 MPa, the HP group were 6.80 MPa to 11.85 MPa, and the AP group were 5.60 MPa to 6.65 MPa.

The surface treated with the MMA in the PR, HP, and AP groups exhibited a significantly higher repair strength than those of the control groups ($p<0.05$). The specimens that were surface treated with EA showed a decrease in repair strength compared with the control groups, except in the PR group ($p>0.05$). However, no significant differences were found between the surfaces treated with EA and MMA, or EA and control specimens ($p>0.05$).

Representative SEM images of the control, MMA, and EA group specimens before repair process are presented in Fig. 2. It can be seen that the surface treatment resulted in irregularities, many small pits, and scratches on the surface of the denture base resin.

Table 2  Two-way analysis of variance to evaluate significant differences among groups

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of squares</th>
<th>df</th>
<th>Mean square</th>
<th>F</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Repair resin</td>
<td>19,320.582</td>
<td>2</td>
<td>9,660.291</td>
<td>400.347</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Surface treatment</td>
<td>244.878</td>
<td>2</td>
<td>122.439</td>
<td>5.074</td>
<td>0.009</td>
</tr>
<tr>
<td>Repair resin* surface treatment</td>
<td>163.339</td>
<td>4</td>
<td>40.835</td>
<td>1.692</td>
<td>0.163</td>
</tr>
<tr>
<td>Error</td>
<td>1,520.176</td>
<td>63</td>
<td>24.130</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Total</td>
<td>47,359.670</td>
<td>72</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
</tbody>
</table>

Table 3  Mean (SD) flexural strength (MPa) for repaired polyamide resin specimens subjected to different surface treatments and use of various repair resins

<table>
<thead>
<tr>
<th>Surface treatment</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PR</td>
</tr>
<tr>
<td>Control</td>
<td>37.70 (8.62)</td>
</tr>
<tr>
<td>Methyl methacrylate monomer</td>
<td>46.25 (8.96)</td>
</tr>
<tr>
<td>Ethyl acetate</td>
<td>42.51 (5.35)</td>
</tr>
</tbody>
</table>


Fig. 2  Scanning electron microscope image (×2,000 magnification) of surface-treated polyamide resins before repairing. (a) Control. (b) Treated with methyl methacrylate monomer. (c) Treated with ethyl acetate.
Different failure types were observed between the repair materials. In the PR group (n=24), 14 showed adhesive failure, 2 showed cohesive failure, and 8 showed mixed failure. In the HP (n=24) and AP groups (n=24), all specimens showed adhesive failure.

**DISCUSSION**

The present study evaluated the effects of different repair resins and surface treatments on the flexural strength of the polyamide denture base resin. For the repair resins, those repaired with PR showed a significant increase in flexural strength when compared to those with HP and AP (p<0.001), while no significant differences were found between those with HP and AP (p>0.05). Thus, the first null hypothesis was partially rejected. For the surface treatment groups, pretreatment with MMA showed a significant increase in flexural strength when compared to the control (p<0.05), while no significant differences were found between the control and EA groups or MMA and EA groups (p>0.05). Thus, the second null hypothesis was partially rejected.

Denture base resins should be made of materials which are strong, rigid, and biocompatible in order to serve successfully for a reasonable length of time. Fractures of denture bases and clasps are commonly repaired, because it is expensive and time consuming to remake the dental prosthesis. A satisfactory repair should possess sufficient strength and color, cost efficient and should be easy to apply, speedy, and dimensionally stable. Therefore, the purpose of the present study was to investigate the repair strength of polyamide denture base resin. The repair strength was tested by means of flexural strength, because it more closely simulated the stress distributions in dental prosthesis such as flexural fatigue failure caused by cyclic deformation of the denture base or clasp arms of removable partial dental prosthesis in the mouth.

Heat-polymerized acrylic and autopolymerizing resins are used in the repair processes of denture base materials. Faot et al. reported that denture base acrylic resins repaired with autopolymerizing resins have a lower flexural strength than those of heat-polymerized acrylic resins, while Rached et al. reported that they have similar flexural strengths. In the present study, the highest repair strength was found in the PR group, and the lowest repair strength was in the AP group. It may be concluded that the repair strength is higher when the denture base and repair materials have similar chemical properties.

Several mechanical and chemical surface treatments have been used to improve the adhesion between the denture base and repair materials. Wetting the repair surfaces with MMA softens the PMMA, enhances the spread of superficial fissures, and forms pits in the bond surface. Alternatively, organic solvents, such as chloroform, acetone, and methylene chloride (dichloromethane), have been used to improve the adhesion. Methylene chloride has carcinogenic potential; therefore, ethyl acetate is used in chemical surface treatments as a safer alternative. Shimizu et al. reported that a 120 s application of ethyl acetate was as effective as a 5 s application of methylene chloride to repair the surfaces of heat-polymerized denture base resins. However, little information has been provided on the surface treatment methods that are effective for bonding polyamide denture base resins. Therefore, the repair surfaces were treated with MMA and EA in the current study.

Katsumata et al. reported that tribochemical silica coating followed by silane coupling was effective in improving the bond strength of nylon denture base polymers to autopolymerizing repair resin. Hamanaka et al. reported that the bond strength improved using a tribochemical silica coating system followed by the application of 4-META/MMA-TBB resin, and that an autopolymerizing repair resin bond to the polyamide denture base resin was difficult, which was supported by the observation that the failure mode was mostly adhesive. In the present study, the most effective surface treatment was the use of MMA and the failure mode was mostly adhesive. This may be due to mechanical interlocking between the repair resins and polyamide denture base resin. It can be seen that surface treatment with MMA resulted in irregularities, small pits and increased bonding area. However, EA was ineffective surface treatment, because the polyamide resin has high degree of crystallinity. This is supported with SEM image of EA that is similar to control. In addition, the repair strength was affected by mechanical fitting of the repair resin to the retention chamfer. Pretreatment with MMA showed a significant increase in repair strength when compared to the control (p<0.05). This can be explained that improved repair strength may be achieved by increased bonding area and mechanical interlocking. However, no significant differences were found between the control and EA groups (p>0.05), so the repair strength may be achieved only mechanical fitting of the repair resin to the retention chamfer.

When a fracture of denture base material is repaired, its flexural strength decreased to 22–58%, or to 36–65%, of the original strength. In addition, the maximum determined repair strength were 75–85% of the original strength. In the present study, repaired strength of the specimens were 8.62–71.15% of the required strength (65 MPa) according to International Standards Organization specification number 20795-1-10. Therefore, the flexural strength of the specimens repaired with the polyamide resin is acceptable for the clinical use. In addition, heat-polymerized acrylic resin and autopolymerizing acrylic resin is not suitable for the repair of polyamide denture base material.

There are a number of limitations to the present study. For example, only one polyamide denture base material and three repair resins were tested. Additionally, two different surface treatments were used, and rectangular specimens were used instead of more complex denture shapes. Finally, the length and in vitro nature of this investigation may not account for
changes inherent in the materials after long periods of use under oral fluid conditions. Further studies may be able to investigate the bond between the polyamide denture material and the prefabricated PMMA teeth. However, clinical studies are necessary to investigate the bond strength of the repair resins.

CONCLUSION

The present study has demonstrated that polyamide resin is the best material to repair a fracture in polyamide denture base resin. In addition, pretreatment with methyl methacrylate monomer increased the flexural strength of the polyamide denture base resin.

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REFERENCES