The Effect of Glass Fiber Distribution on the Transverse Strength and Surface Smoothness of Two Denture Resins

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The aim of this study was to evaluate the effect of glass fiber distribution on the transverse strength and surface smoothness of conventional heat cured acrylic and autopolymerizing acrylic of an injection-molding system.

Forty rectangular (65×10×2.5 mm) acrylic test specimens were prepared from both acrylic types: 10 with 5% (w/w) 6 mm length fiber and 10 without fiber for both groups. Transverse strength test was applied to these specimens. Surface samples were taken from the broken and polished surfaces of these specimens and evaluated using SEM.

The addition of fiber was found to cause a statistically significant increase in the transverse strength of the injection system’s acrylic. In SEM observation it was revealed that there was good adhesion between glass fiber and both acrylic resins. The glass fibers distribution was more even in the injection system’s acrylic. It is suggested that injection system’s acrylic be fiber-reinforced to reduce denture fractures.

Key words: Polymethylmethacrylate, Glass fiber, SEM

INTRODUCTION

Polymethylmethacrylate (PMMA), which is aesthetically pleasing, economic and easily constructed and repaired has been widely used as a denture base material. Despite these advantages its impact and fatigue strengths are low. Denture fractures occur frequently. Ways of modification or adding reinforcement have been tried to increase the strength of PMMA.

Various fibers such as carbon, aramid, metal, polyethylene (PE) and glass have been used as reinforcement materials.

For the improvement of the physical properties of acrylic resins-fiber composite some parameters such as selection of matrix, fiber thickness, content of fiber as volume or weight, its distribution, dimension, preignation with resin, selection and usage of silane agents, technique and conditions of construction are effective.

Fibers are made in chopped, unidirectional continuous, tape and woven forms.

It has been reported that glass fiber used in order to reinforce PMMA increases the fatigue, impact and transverse strengths and modulus of elasticity. Glass fiber is aesthetically pleasing and noncytotoxic. E-glass (electrical glass) fiber is used in dentistry. E-glass consist of 52-56% SiO₂, 16-25% CaO, 12-16% Al₂O₃, 8-13% B₂O₃, 1% Na₂O and K₂O and 0-6% MgO.
fiber and polymer matrix silanation is recommended.\textsuperscript{3,7,9,22}

The aim of this study was to evaluate the effect of glass fiber distribution on the transverse strength and polished surface smoothness of conventional heat cured acrylic and autopolymerizing acrylic of an injection-molding system.

**MATERIALS AND METHODS**

**Preparation of test specimens**

In this study silane treated continuous unidirectional glass fiber (KCR2(M), Cam Elyaf San. AS, Kocaeli, Turkey), conventional heat cured acrylic (Meliodent, Bayer Dental, Bayer, Germany) and autopolymerizing acrylic of an injection-molding system (Palaxpress, Heraeus Kulzer GmbH, Wehrheim, Germany) were used. The glass fibers were cleaned in boiling water for 10 min. After air-drying they were silanated by being dipped into a silane solution, $\gamma$-methacryloxypropyltrimethoxysilane ($\gamma$ MPS) (A174, HÜLS-Veba GmbH, Germany). The fibers were air-dried for 40 min and then placed in an oven (Nüve, PN500, Ankara, Turkey) for 1 hr at 115°C before being added into the acrylic resin.

Unidirectional continuous glass fiber was supplied as threads with 50 filaments per end with each filament of $12 \mu m$ nominal diameter. Unidirectional continuous glass fiber was chopped on a template of 6 mm using a surgical blade. Chopped fibers were added to polymer as 5% of the weight of the powder would be fiber and mixed for approximately 30 sec. Weight measurements were made with 0.0001 g sensitivity (Sartorious AG, Gottingen, Germany). Including the control groups four groups were formed, one of which had 10 test specimens.

All of the test specimens were prepared according to the sizes indicated by ADA standard No.12\textsuperscript{27}. Wax specimens were prepared in metal moulds measuring $65 \times 10 \times 2.5 \text{ mm}$. Acrylic test specimens were obtained from these waxes.

Polymerization of conventional acrylic: A flask including the wax specimens was immersed in boiling water and then flushed with detergent solution and boiling water. After the flask was cooled until it could be held comfortably in the bare hand, the sample cavities were painted with an undiluted alginate compound (Aislar, Heraeus Kulzer, Germany). The flask was left for 1 hr at a temperature of $23 \pm 2 \text{ ^\circ C}$. According to the suggestion of the manufacturer, the proportion of monomer/polymer was arranged so there would be $1 \text{ ml/2.34 g}$ in each control group. As for the group with fiber, additional monomer was added proportionally to the additional fiber added. In order to determine the amount of additional monomer of $3 \text{ ml}$ of monomer was put into a closed glass vessel. $1 \text{ g}$ chopped fiber was added. The volume of the remaining monomer was measured with a pipette after 10 min. It was determined that the amount of monomer saturating the $1 \text{ g}$ of fiber was $0.7 \text{ ml}$\textsuperscript{17,20}. After the acrylic dough reached a consistency where it did not stick to the edges of the mixing pot acrylic resin was packed. After pressing the flask for 10 min it was left in a water bath at $73 \pm 1^\circ \text{ C}$ for 90 min and boiling water for 30 min. After the heating process was completed the flask was cooled at $23 \pm 10^\circ \text{ C}$ room temperature.
Then it was cooled in water of $23\pm1{}^\circ\text{C}$ for 15 min.

Palaxpress test specimens were prepared in the following way according to the suggestions of the manufacturer. A special flask of the injection system including wax specimens was immersed in boiling water and then flushed with a detergent solution and boiling water. After the flask was cooled until it could be held comfortably in the bare hand, the sample cavities were painted with the undiluted alginate compound. The flask was left for 1 hr at a temperature of $23\pm2{}^\circ\text{C}$. The flask was closed and put into its place on the injection machine. The polymer/monomer proportion was set so there would be 2 g/1 ml in the control group. As for the group with fiber additional monomer was added proportionally to the amount of the fiber added (0.7 ml monomer per 1 g fiber). Acrylic dough was put into the pot of the injection system (Palajet, Heraeus Kulzer, Germany). The pot was located to flask’s entrance hole. Acrylic dough was injected into the flask under a 600 kPa pressure. After being under this pressure for 5 min acrylic was polymerized under 200 kPa for 30 min using a polymerization machine (Palamat, Heraeus Kulzer, Germany). The flask was cooled at room temperature for 15-20 min.

After curing and deflasking test specimens were leveled with a steel bur rotating at 15,000 rpm and their surface disorders were smoothly sandpapered with wet 600-grade silicon carbide paper. A 3-point loading test was applied (crosshead speed of 5 mm/min) to the test specimens Tensometre Machine H50KN, (Hounsfield, Croydon, England). The span of this 3-point loading test was 50 mm. This dimension represents the space between the maxillary molars in a complete denture. Transverse strengths were calculated with the following formula:\(^8\):

\[
S = \frac{3PL}{2WT^2}
\]

$P =$ Fracture load
$L =$ Distance between the supports (50 mm)
$W =$ Specimen width (10 mm)
$T =$ Specimen thickness (2.5 mm)

From the broken surfaces of the specimens with fiber cross sections of 1 mm were taken. After polishing the surfaces simulating the prosthesis polished surfaces of the same specimens, cross sections of 1 mm were taken from the polished surfaces. Fracture surfaces and polished surfaces were covered by gold under a vacuum (SC500 Sputter Coater, Polaron, England) and then examined in SEM (Scanning Electron Microscopy) (JSM 5600 SEM, Jeol, Tokyo, JAPAN). It was examined whether there was any relation between the transverse strengths of the specimens, the fiber resin connection and the fiber distribution. In addition, the locations of the randomly distributed, chopped fibers on polished surfaces were examined.

A statistical evaluation of the findings of the 4 groups was done using SPSS at a 95% confidence level. The diversity among the groups was evaluated by one-way ANOVA, but in two each comparisons Scheffe test was used.
**RESULTS**

The mean transverse strength values and standard deviations of both acrylic resin specimens are given in Table 1. One-way ANOVA indicated that there was a significant difference between the groups ($F=12.745$, $p=0$). According to the Scheffe test, the increase in transverse strength values caused by the addition of fiber in the acrylic group with injection was statistically significant ($p<0.05$), however, the increase in the transverse strength of the conventional acrylic was not statistically significant.

In SEM pictures it was observed that fibers were lying obliquely in both acrylic groups, almost parallel to the surface (Fig. 1a, b and Fig. 3a, b). However, protruding ends of glass fibers were observed in polished surfaces here and there (Fig. 1b and Fig. 3b). Moreover, the fibers were found in bunches in the conventional acrylic group (Fig. 4b), but in the acrylic with injection group they were distributed evenly (Fig. 2a, b). When the fracture surfaces were examined there was no void space between fibers and both acrylic resins. Particles of acrylate that adhered to the surfaces of the fiber showed that there was good adhesion between the glass fiber and both acrylic resins (Fig. 2a and Fig. 4a).

<table>
<thead>
<tr>
<th>Groups</th>
<th>Mean (MPa)</th>
<th>Standard error (SE)</th>
<th>$F$-value</th>
<th>$p$-value</th>
</tr>
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<tr>
<td>Palaxpress</td>
<td>91.20</td>
<td>3.20</td>
<td></td>
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<td>Palaxpress+fiber</td>
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<td>2.15</td>
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<td>5.12</td>
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<tr>
<td>Meliodent+fiber</td>
<td>109.30</td>
<td>2.80</td>
<td>$F=12.745$</td>
<td>$p=0$</td>
</tr>
</tbody>
</table>

*Fig. 1 SEM photographs of polished surface of reinforced Palaxpress test specimens.*
Fig. 2 SEM photographs of fracture surface of reinforced Palaxpress test specimens.

Fig. 3 SEM photographs of polished surface of reinforced Meliodent test specimens.

Fig. 4 SEM photographs of fracture surface of reinforced Meliodent test specimens.
DISCUSSION

Glass fiber improves transverse, fatigue, and impact strengths, modulus of elasticity. It is aesthetic and it distributes in acrylic more evenly than other fibers. It is because of this and its intensity in the packing stage that has caused it to be successful in clinical use and noticed researchers\textsuperscript{20,22}. Vallittu and Ekstrand\textsuperscript{24} have shown using agar diffusion test that E-glass fiber with and without silane is not cytotoxic. We preferred glass fiber in this study because of the advantages mentioned above.

Though it has been reported that glass fibers in continuous unidirectional form increase the fracture strength of denture considerably\textsuperscript{3} and woven fiber is very applicable in fixed prostheses\textsuperscript{28}, some problems such as the difficulty of putting these fibers into their places and having an insufficient mixture of fibers with resin have been reported\textsuperscript{3}. It has also been reported that fiber bundles which impregnate insufficiently with polymer matrix cause a decrease in the transverse strength of denture base polymers with glass fiber, and that void spaces form in test specimens\textsuperscript{15,17}. Fibers in chopped form are easier to add fibers in than other forms\textsuperscript{21}. Furthermore, continuous unidirectional or woven form glass fiber can not be used in injection molding systems. Because of the reasons listed above glass fibers in chopped form were used.

Some researchers\textsuperscript{3,4,14} have reported that some good results were taken from fibers randomly put into acrylic without wasting time. Because of these reasons and the necessities caused by injection systems fiber was randomly added to PMMA powder.

The studies done on fiber reinforcement of injection acrylic are very limited\textsuperscript{29}. The distribution of chopped form glass fiber in conventional and autopolymerizing acrylic in injection molding systems has been a matter of interest for us. It was necessary for us to do this study to evaluate fiber distribution differences in both acrylics and the effects of the differences on the transverse strength and surface smoothness of these acrylics.

The methods of adding the fiber into the resin vary. Researchers have tried in different ways to increase the connection of fiber with polymer matrix, some\textsuperscript{3,30} adding fiber after impregnating it with monomer, some\textsuperscript{7,31–33} adding it after impregnating with a polymer-monomer mixture, and some\textsuperscript{3,4,15,22,25} adding it into acrylic resin without impregnating it first. In the studies above, a relation between the fiber incorporating technique and the homogeneity of fiber-resine composite has not been mentioned. However, we think that fiber preimpregnation, as fiber is wetted, will spoil the homogeneity of the fiber-resine composite. Therefore, we added fibers to acrylic resin without doing any preimpregnation process.

ADA standard No.12 recommends that the preparation of test samples in acrylic without fiber is to be done by cutting from the block. However, in our study acrylic reinforced with fiber was used. For this reason specimens were prepared one by one so that the length and proportions of the fibers in each specimen would be the same.

It is known that the direction of fiber is important for the reinforcement of
Optimal results have been obtained by placing the fibers parallel to the surface of the denture perpendicular to the direction of mastication force. It is possible to do this with fibers in woven or continuous form, however, it has been determined in our study using SEM pictures that as the length of chopped fibers exceeds the thickness of the denture base, fibers lie obliquely almost parallel to the surface of the denture.

It has been observed that the fracture strength of acrylic resin with shortly cut and randomly mixed glass fiber 1% by weight has increased. According to Vallittu et al., when combined with acrylic resin material the fibers increase the fracture strength of PMMA test specimens. It has also been reported that it increases the fracture strength of fiber further at high concentrations (21.9%) by weight; however, Ladizesky et al. do not suggest the use of fiber more than 4% by weight. Stipho has reported that glass fiber more than 5% by weight does not give a meaningful mechanical advantage. Chen et al. claimed that they had successful results from the acrylic resin reinforcement they did with 6 mm PE and glass fiber. In Marei's study randomly distributed short glass fibers with a mean length of 88 mm have been used and it has been indicated that these fibers of different lengths increase the transverse strength of acrylic resin. It has been indicated that this is similar to the reinforcement done with fibers in unidirectional form. In accordance with the explanations above and as the addition of fiber more than 5% by weight in the injection system make the injection process difficult, fiber 5% by weight was used in this study.

It is necessary to provide good adhesion between the fiber and the polymer matrix for the reinforcement of resins with fiber. Fibers without silane added to the resin act as a foreign body. Instead of reinforcing the resin it weakens it. Fibers without silane may spoil the homogenous structure of the matrix. Silane agents chemically combining glass fibers to resin matrix may alter the mixture to a more homogenous state and may reinforce the PMMA. Vallittu has shown using SEM pictures that the application of silane improves adhesion between, fibers and polymer matrices. Solnit has reported that the strength values gathered by the use of fiber with silane have exceeded the normal strength values of PMMA. In addition, it has also been reported that the more PMMA and fiber are mixed homogenously, the more the strength of the resin increases. In our study the areas where acrylate particles on glass fiber were determined in the SEM pictures were taken from the fracture surfaces in both acrylic groups. Besides it was observed that there is not much void space between the fiber and matrix, and that they are fairly harmonious. However, it has been determined that in some areas fiber slipped off the resin as the lengths of fibers are short. It is considered that the good fiber-matrix connection designated by SEM contributes to the increase in transverse strength.

It was determined that the addition of glass-fiber increases the transverse strength of Palaxpress test specimens significantly; however, there was not a statistically significant increase in the Meliodent specimens' strengths. As the connections of both acrylics with fiber in SEM pictures are good, the reason for the difference
between these acrylic groups was thought to be the presence of fibers as bunches in the Meliodent group and their homogenous distribution in Palaxpress. We think that fibers distribute in Palaxpress more homogenously since palaxpress settles into the flask, flowing by means of injection strength. We think that the considerable increase in the transverse strength of Palaxpress resulted from the homogeneity of fiber distribution.

One serious disadvantage of using short fibers was the presence of protruding ends in the finished specimens\(^3\). Some researchers\(^{20,21}\) have reported that as a result of the use of fiber in chopped form for the reinforcement of PMMA fiber ends slipped off the surface, an inconvenience for mouth mucosa. It has been observed also in our study that the ends of the fiber come out in the areas where fibers are close to the surface of the acrylic when the cross-sections taken from the polished surfaces of the specimens are studied.

**CONCLUSIONS**

1. The addition of glass fiber increased the transverse strength of both acrylic types. However, only the increase in the Palaxpress group was found to be statistically significant.
2. It was understood as a result of the SEM investigation that there was good adhesion between fibers and both acrylic resins and glass fibers lied down obliquely almost parallel to the surface of acrylic.
3. It was seen by means of SEM pictures that fibers were distributed as bunches in Meliodent and homogeneously in Palaxpress.
4. It was observed by SEM that the edges of fiber closer to the surface in polished surfaces come out in some places.

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