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Abstract

Aim: The purpose of this study was to evaluate the effect of Zirconium oxide (ZrO₂) nanofillers powder with different concentration (1.5%, 3%, 5% and 7%) on the transverse strength and impact strength of heat-polymerized acrylic resin. Materials and Methods: Zirconium oxide powders with different concentrations (1.5%, 3%, 5% and 7%) were incorporated into heat-cure acrylic resin (PMMA) and processed with optimal condition (2.5:1 Powder/monomer ratio, conventional packing method and water bath curing for 2 hours at 95˚C) to fabricate test specimens of PMMA of dimensions (65 x 10 x 2.5mm) for the transverse strength and (60 x 7 x 4mm) for impact strength. PMMA without additives was prepared as a test control. Two types of mechanical tests; transverse strength and impact strength were carried out on the samples. The recorded values transverse strength in (MPa) and impact strength in (J) were collected, tabulated and statistically analyzed. One way analysis of variance (ANOVA) and Tukey’s tests were used for testing the significance between the means of tested groups which are statistically significant when the P value ≤ 0.05. Results: Addition of Zirconium oxide nanofillers to PMMA significantly increased the transverse strength and impact strength Conclusion: Zirconium oxide nanofillers added to PMMA has a potential as a reliable denture base material with increased transverse strength and impact strength. According to the results of the present study, the best mechanical properties were achieved by adding 7%wt ZrO₂ concentration.

Keywords: Zirconium Oxide; polymethayl methacrylate; Impact Strength

1. Introduction

Acrylic resin polymethayl methacrylate (PMMA) has been the most popular material for the construction of dentures for many decades as it has many advantages such as good aesthetics, accurate fit, stability in the oral environment, easy laboratory and clinical manipulation, and inexpensive equipment’s [1]. Although it is the most widely used in dentistry for fabrication of denture bases, this material is still insufficient to fulfill the perfect mechanical requirements for dental applications. This issue was attributed mainly to its low fracture resistance and plaque accumulation [2] [3]. In a survey
to compare ten types of denture base resins it was found that nearly 70% of dentures had broken within the first 3 years of their delivery [2]. In a study evaluating the denture fracture, it was reported that 33% of the repairs were due to debonded/detached teeth, 29% of the repairs were because of midline fractures which were more commonly seen in the upper dentures and the rest were other types of fracture.

In another study the authors reported that the Mandibular partial denture was the most commonly needing repair [4]. So, the measuring of mechanical properties of the denture base materials is important to evaluate the effect of adding different strengthening materials [5].

Undoubtedly that, many trails were made to enhance mechanical properties of denture base materials either by adding chemical solutions such as a polyfunctional cross linking agent (polyethylene glycol dimethacrylate) [6] or by incorporating a rubber phase [7], metal fram [8], metal oxides [9], or fibers [10]. Despite these efforts to improve the fracture resistance of PMMA few have obtained promising results [11] [12]. The reinforcement of polymers used in dentistry with metal-composite systems has been a prime interest [12].

Zirconium oxide nano-particles powder has been selected to improve the properties of PMMA, as a bio-compatible material that possesses high fracture resistance, and to improve fracture toughness of ceramics by developing a new generation of ceramic-matrix composites [13] [14].

Since only limited amount of data regarding the effect of metal oxides on heat-cured PMMA are available in the literature, the purpose of this study was to investigate the influence of addition of metal oxides [zirconium oxide powder (ZrO2)] on some mechanical properties of heat cured PMMA.

2. Subjects and Methods

An in vitro study was conducted to evaluate the effect of Zirconium oxide nanofillers powder (ZrO2) (5 - 15 nm) with different concentration (1.5%, 3%, 5% and 7%) on transverse strength and impact strength of heat-polymerized acrylic resin.

One type of heat-cure acrylic resin (PMMA) was used as the control (Acrostone (A), Anglo-Egyptian Company. Hegaz, Cairo, Egypt, Batch No.505/04), Zirconium oxide nanofillers powder (ZrO2) (Sigma-Aldrich Germany, Trade 544,760) with different concentrations (1.5%, 3%, 5% and 7%) was added into heat-cure acrylic resin (PMMA) and processed with optimal condition (2.5:1 Powder/monomer ratio, conventional packing method and water bath curing for 2 hours at 95˚C) 100 bar shapes specimens were prepared to be used in this study. 50 specimens were used for each test [transverse strength (group A) and impact strength (group B)].

Grouping of the specimens:

Each group was further divided into five subgroups (1, 2, 3, 4 and 5) of 10 specimens each as shown in Table 1.

Transverse strength testing:

The transverse strength test (TS) was conducted according to International Standards Organization specification 1567 for denture base polymers [15]. Rectangular-shaped specimens (65 x 10 x 2.5) were prepared. Testing was conducted under 3-point loading, with a crosshead speed of 5mm/min. using a Lloyd universal testing machine (model LRX plus II, Fareham, England).

Impact strength testing:

Rectangular-shaped specimens (60 x 7 x 4 mm) were prepared for impact strength testing (IS). Strength test method and specimens dimensions were similar to those used by Uzun et al [16]. Using a notch cutter (Hounsfield notching machine, Tensometer Ltd., Croydon, U.K.), a 3.5 mm notch was prepared in each specimen. A Charpy-type impact tester (Hounsfield plastic impact machine, Tensometer Ltd.) was used to apply force to the specimens from the un-notched side. During testing those specimens that did not fracture at the first trial were excluded from the study.

The recorded values of transverse strength and impact strength were collected, tabulated and statistically analyzed. One way analysis of variance (ANOVA) and Tukey’s tests were used for testing the significance between the means of tested groups which are statistically significant when the P value ≤ 0.05.

3. Results

Transverse strength

Both Table 2 and Figure 1 show a comparison between mean transverse strength in (MPa) of the tested groups of PMMA. ANOVA test showed statistically significant difference between all groups. PMMA specimen with 7% zirconium oxide nanofillers (ZrO2) (group A5) showed significantly highest mean transverse strength followed by
### Table 1: Classification and grouping of the specimens.

<table>
<thead>
<tr>
<th>Groups</th>
<th>Subgroups</th>
<th>Description</th>
<th>No. of Specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A</td>
<td>Group A1</td>
<td>Heat-cure acrylic resin (PMMA) without additives as control.</td>
<td>10 specimens</td>
</tr>
<tr>
<td></td>
<td>Group A2</td>
<td>PMMA with 1.5% zirconium oxide nanofillers powder (ZrO₂).</td>
<td>10 specimens</td>
</tr>
<tr>
<td></td>
<td>Group A3</td>
<td>PMMA with 3% ZrO₂.</td>
<td>10 specimens</td>
</tr>
<tr>
<td></td>
<td>Group A4</td>
<td>PMMA with 5% ZrO₂.</td>
<td>10 specimens</td>
</tr>
<tr>
<td></td>
<td>Group A5</td>
<td>PMMA with 7% ZrO₂.</td>
<td>10 specimens</td>
</tr>
<tr>
<td>Group B</td>
<td>Group B1</td>
<td>Heat-cure acrylic resin (PMMA) without additives as control.</td>
<td>10 specimens</td>
</tr>
<tr>
<td></td>
<td>Group B2</td>
<td>PMMA with 1.5% zirconium oxide nanofillers powder (ZrO₂).</td>
<td>10 specimens</td>
</tr>
<tr>
<td></td>
<td>Group B3</td>
<td>PMMA with 3% ZrO₂.</td>
<td>10 specimens</td>
</tr>
<tr>
<td></td>
<td>Group B4</td>
<td>PMMA with 5% ZrO₂.</td>
<td>10 specimens</td>
</tr>
<tr>
<td></td>
<td>Group B5</td>
<td>PMMA with 7% ZrO₂.</td>
<td>10 specimens</td>
</tr>
</tbody>
</table>

| Total | 100 specimens |

### Table 2: Comparison between mean transverse strength (MPa) of the tested groups of PMMA.

<table>
<thead>
<tr>
<th>Group</th>
<th>Control group</th>
<th>Group A2 (1.5% ZrO₂)</th>
<th>Group A3 (3% ZrO₂)</th>
<th>Group A4 (5% ZrO₂)</th>
<th>Group A5 (7% ZrO₂)</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>79.1e</td>
<td>86.58d</td>
<td>95.03c</td>
<td>101.8b</td>
<td>128.6a</td>
<td>0.000*</td>
</tr>
<tr>
<td>SD</td>
<td>1.148</td>
<td>3.42</td>
<td>3.67</td>
<td>3.076</td>
<td>1.96</td>
<td></td>
</tr>
</tbody>
</table>

*Significant at P ≤ 0.05, Means with different letters are significantly different according to Tukey’s test.

### Table 3: Comparison between mean impact strength (J) of the tested groups of PMMA.

<table>
<thead>
<tr>
<th>Group</th>
<th>Control group</th>
<th>Group B2 (1.5% ZrO₂)</th>
<th>Group B3 (3% ZrO₂)</th>
<th>Group B4 (5% ZrO₂)</th>
<th>Group B5 (7% ZrO₂)</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean</td>
<td>1.42e</td>
<td>1.95d</td>
<td>2.55c</td>
<td>2.93b</td>
<td>3.45a</td>
<td>0.000*</td>
</tr>
<tr>
<td>SD</td>
<td>0.96</td>
<td>0.63</td>
<td>1.07</td>
<td>0.56</td>
<td>1.05</td>
<td></td>
</tr>
</tbody>
</table>

*Significant at P ≤ 0.05, Means with different letters are significantly different according to Tukey’s test.
PMMA specimen with 5% (ZrO₂, group A4) followed by PMMA specimen with 3% (ZrO₂, group A3) then PMMA specimen with 1.5% (ZrO₂, group A2). There were significant differences (P < 0.05) between studied groups. PMMA specimen without any additives (control group) showed significantly lowest mean transverse strength.

Impact strength

The impact strength data showed there was significant improvement in the tested groups which were reinforced with zirconium oxide nanofillers (ZrO₂) (Table 3 and Figure 2).

There was significant increase in the impact strength for groups reinforced with (1.5%, 3%, 5% and 7%) ZrO₂ when compared with control group.

4. Discussion

We principally aimed to assess possible improvements in the mechanical properties of PMMA, in particular, the transverse strength, and impact strength, through incorporating of ZrO₂ Nano particles. There are three ways to improve the mechanical properties of PMMA: replacing PMMA with an alternative material; chemically modifying it; and reinforcing the PMMA with other materials [17] [18].

Addition of Zirconia Nano fillers to acrylic resin was found to improve mechanical properties. In addition to that ZrO₂ was used because it has excellent biocompatibility and white color which less likely to alter esthetic. The Nano-filler particles were used in this study as it yields a better
dispersion eliminate aggregation and improve its compatibility with organic polymer [19] [20]. Proper percentage range of zirconium oxide Nano-fillers (Percentages of 1.5% - 7% by weight) was selected because percentages above 7% was leads to massive changes occurred in the color of acrylic [21].

The transverse strength test, one of the mechanical strength tests, is especially useful in comparing denture base materials in which a stress of this type is applied to the denture during mastication [22]. The transverse (flexural) strength is a combination of compressive, tensile and shear strengths, all of which directly reflect the stiffness and resistance of a material to fracture [23].

The fracture of acrylic resin dentures as a result of being dropped is a common occurrence and research continues to dates to produce a denture base material with improved impact resistance. Impact strength is an important parameter as it can reflect the contact force required to cause fracture in a denture under situations such as accidental dropping [24].

The Results of the present study demonstrated a significant increase in transverse strength and impact strengths as the percentage of ZrO2 fillers increased. This improvement in mechanical properties could be attributed to the high interfacial shear strength between the nanofillers and resin matrix as a result of formation of cross-links or supra molecular bonding which cover or shield the nanofillers which in turn prevent propagation of crack, also complete wetting of the nanofillers by resin lead to increase in flexural strength, fracture toughness, and hardness as volume of filler increased [25].

The results of this study are in agreement with the findings reported by others [26-28] who concluded that reinforcement of ceramics, dental restorative resins as well as acrylic resin with zirconia could exhibit improvement in their mechanical properties.

5. Conclusion:

Within the limitation of this study, we can conclude that addition of zirconium oxide nanofillers to PMMA increased transverse strength and impact strength of heat polymerized acrylic resin. According to the results of the present study, the best result was got when using the concentration of 7%wt.

Further studies are needed to investigate its effect on other mechanical and physical properties with different concentrations.

6. References