Impact of surface treatments on the sorption and solubility of a heat-cured denture base material

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ABSTRACT

Introduction

Removable dentures fabricated from polymethylmethacrylate material are the most common prostheses used to treat edentulism worldwide.

Aims and Objectives

This research aimed to compare the sorption and solubility characteristics of a mechanically polished heat-cured acrylic denture material and a light-cured varnished material against non-treated material, all of which were soaked in distilled water.

Methods

A total of 45 specimens were prepared and tested according to the ISO Standard 20795-1: 2013 (E) to test for sorption and solubility. The data were analysed through one-way analysis of variance (ANOVA) and Tukey-Kramer multiple comparison test.

RESULTS AND CONCLUSIONS

The results indicated that both surface treatments were successful in reducing either the sorption or solubility level recorded by the specimens. The control group recorded mean sorption and solubility values which were both within the parameters set out by ISO 20795-1: 2013 (E) for a type-one polymer. Mechanical polishing was identified as the surface treatment that resulted in the largest reduction of solubility, with the lowest sorption values being recorded after the application of the light-cure varnish to the heat-cure denture base material. When mechanical polishing is compared to Optiglaze™ light-cured varnish, the results indicate that mechanical polishing may be a more effective surface treatment option.

Introduction

Removable dentures, whether complete or partial, are still the most prevalent dental prostheses worldwide. In recent times, there has been an increase in the demand for fixed prostheses due to their apparent advantages over removable prostheses, in terms of comfort, aesthetics and preserving underlying alveolar bone. This trend is however not as marked among those with lower socio-economic means or individuals in the older age groups due to the costs entailed. Sorption and solubility have been identified as two major drawbacks of denture base materials and their detrimental effects are well documented. Literature pertaining to the mixing ratios, polymerisation cycles and thickness of the denture bases are factors identified as external variables that may negatively influence the rate of sorption and solubility experienced by denture base materials. Measures to reduce the amount of sorption and solubility in denture base materials include various surface treatment procedures which may be applied in order to mitigate these effects.

Surface treatments are applied to improve the properties and characteristics of the material, for physical, mechanical, chemical or aesthetic purposes. However, as a result of continued use, masticatory erosion may result in the degradation of the polished surface over time. This degradation is associated with the increase in the surface roughness of the denture and may result in an increase in the sorption and solubility of the denture base material. As an alternative to mechanical polishing, a review of existing scientific literature has established that there are very few published studies investigating the effects that the application of a light-cured varnish to heat-cured PMMA...
denture base material have on the material’s sorption and solubility characteristics. Although there are studies that have reported the effect of mechanical polishing on the sorption and solubility of denture base materials \(^9,11–14\), establishing a standard has proved to be challenging due to inconsistencies across the board, as very few documented studies have precisely followed ISO regulations. This research therefore aimed to determine the sorption and solubility characteristics of mechanically polished heat-cured dental acrylic material and light-cure varnished material soaked in distilled water and to evaluate which surface treatment resulted in the least sorption and solubility when compared to untreated acrylic material.

Materials and Methods

A total of 45 specimens were prepared according to the ISO Standard 20795-1: 2013 (E) to test for sorption and solubility of a Type One, Class One denture base material. The specimens were required to have a thickness of 0.5 (± 0.05) mm and a diameter of 50 (± 0.1) mm. The total sample population consisted of three groups, comprising 15 specimens each: No surface treatment (Group A, Control), mechanically polished (Vertex, High Gloss polishing Paste, Vertex-Dental B.V, Zeist, NL) (Group B) and those that were treated with a light cure varnish (GC, Optiglaze, GC N.V. Europe, Belgium) (Group C). Vertex™ Rapid Simplified (Vertex-Dental B.V, Zeist, NL) heat-cured denture base material was the material of choice for this study. After fabrication, the specimens were all placed in individual air-tight bags, and stored in a portable cold storage unit (Snomaster, Classic Series CL60, Snomaster, Texas, USA) at a constant temperature of 7°C. A stainless-steel mould and cover were custom made to conform with ISO 20795-1: 2013 (E). The mould and cover were invested in a two-part denture flask with type two dental gypsum (Interdent, Alabaster, Interdent d.o.o., Celje, SI) mixed according to the manufacturer’s recommendation. One half of the flask contained the mould and the other half the cover. The laboratory environment was controlled at 23 (± 2) °C and at a relative humidity of 50 (± 10) °C. To produce the most accurate results possible and strictly to follow ISO protocols, the researcher used the same mould for the fabrication of all 45 specimens. A calibrated Denver Instruments S-403SN (Denver Instrument Inc., New York, USA) balance scale accurate to 1 mg and Biohit Proline Pipette (100-1000μl) (Biohit Oyj, Helsinki, FI) were used to measure the liquid-to-powder ratio of 1 ml: 2.3g recommended for Vertex™ Rapid Simplified (Vertex-Dental B.V, Zeist, NL) heat-cured denture base material. A polythene sheet from Metrodent™ (Metrodent Limited, West Yorkshire, UK) was placed over the mixed material to create a buffer between the material and the stainless-steel cover of the mould. The flasks were closed and placed in a pneumatic press (Dentalfarm SRL, Turin, IT). Pressure of two bars was applied and maintained until no loss in pressure was observed, after which the flask was removed and placed in its respective clamp. The flask was immersed in a curing bath (Eurocem, Water Polymeriser, Eurocem, Milan, IT) containing water heated to 100°C and cured for 20 minutes as recommended by the manufacturer. Following this, the flask was removed and allowed to bench cool until it reached the ambient temperature. Once all the specimens were fabricated, they were selected at random from the portable cold storage unit to receive their allocated surface treatment.

### Table I: Sorption of samples in μg/mm³

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean</th>
<th>Median</th>
<th>Std Dev.</th>
<th>Std Error</th>
<th>Min.</th>
<th>Max.</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A</td>
<td>22.3690</td>
<td>22.1536</td>
<td>0.8619</td>
<td>0.2225</td>
<td>21.2659</td>
<td>24.5340</td>
<td>3.2681</td>
</tr>
<tr>
<td>Group B</td>
<td>21.8613</td>
<td>21.9569</td>
<td>0.2676</td>
<td>0.0691</td>
<td>21.4994</td>
<td>22.3403</td>
<td>0.8409</td>
</tr>
<tr>
<td>Group C</td>
<td>21.3713</td>
<td>21.3372</td>
<td>0.2873</td>
<td>0.0742</td>
<td>20.8905</td>
<td>21.9269</td>
<td>1.0364</td>
</tr>
</tbody>
</table>

### Table II: Solubility of samples in μg/mm³

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean</th>
<th>Median</th>
<th>Std Dev.</th>
<th>Std Error</th>
<th>Min.</th>
<th>Max.</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A</td>
<td>0.1843</td>
<td>0.1866</td>
<td>0.1367</td>
<td>0.0353</td>
<td>-0.189</td>
<td>0.4321</td>
<td>0.6212</td>
</tr>
<tr>
<td>Group B</td>
<td>0.1593</td>
<td>0.1600</td>
<td>0.0457</td>
<td>0.0118</td>
<td>0.0683</td>
<td>0.2315</td>
<td>0.1632</td>
</tr>
<tr>
<td>Group C</td>
<td>0.2406</td>
<td>0.2492</td>
<td>0.1080</td>
<td>0.0279</td>
<td>0.0223</td>
<td>0.4191</td>
<td>0.3968</td>
</tr>
</tbody>
</table>

### Table III: Wsl results for Tukey-Kramer multiple comparison test

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean</th>
<th>Different from Groups</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Surface Treatment, Distilled Water (A)</td>
<td>0.1843</td>
<td>None</td>
</tr>
<tr>
<td>Mechanical Polishing, Distilled Water (B)</td>
<td>0.1593</td>
<td>None</td>
</tr>
<tr>
<td>Light-Cured Varnish, Distilled Water (C)</td>
<td>0.2406</td>
<td>None</td>
</tr>
</tbody>
</table>

### Table IV: Wsp results for Tukey-Kramer multiple comparison test

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean</th>
<th>Different from Groups</th>
</tr>
</thead>
<tbody>
<tr>
<td>No Surface Treatment, Distilled Water (A)</td>
<td>22.3690</td>
<td>B and C</td>
</tr>
<tr>
<td>Mechanical Polishing, Distilled Water (B)</td>
<td>21.8613</td>
<td>A</td>
</tr>
<tr>
<td>Light-Cured Varnish, Distilled Water (C)</td>
<td>21.3713</td>
<td>A</td>
</tr>
</tbody>
</table>
Surface Treatments

All the specimens were ground with wet pumice (Interdent, Pumice, Interdent d.o.o., Celje, SI) and a wet muslin wheel for one minute per surface at a circumferential speed of 650 ± 350 meters per minute as specified by ISO 20795-1:2013(E). The mechanically polished (Group B) specimens were polished for two minutes per surface using an unstitched muslin wheel and Vertex™ High Gloss Polishing Paste (Vertex-Dental B.V, Zeist, NL). After polishing, the specimens were visually inspected to ensure they complied with ISO requirements, presenting a smooth surface with a high gloss. The application surface of the specimens in Group C was wiped with Vertex™ Rapid Simplified monomer (Vertex-Dental B.V, Zeist, NL), to remove any smear layer from the surface. A thin layer of Optiglaze™ (GC N.V. Europe, Belgium) protective coating agent was applied, after which it was cured in a light-curing unit (Max Stir, MS Multiact, Max Stir srl, Cavriago, IT) for 40 seconds on each side. Once the respective surface treatment procedures were complete, the specimens were marked using a black waterproof marker.

Sorption and solubility

The sorption and solubility of the samples were based upon measurement of the uptake and release of a solute under controlled conditions. The guidelines established by the ISO 20795-1 (2013) document to test for sorption and solubility were followed precisely. The specimens were placed in a desiccator containing freshly dried silica gel, which was stored in an incubator for 23 (± 1) hours at 37 (± 1) °C. Once the time had elapsed, the specimens were removed from the desiccator and placed in a second desiccator containing freshly dried silica gel for 60 (± 10) minutes at 23 (± 2) °C. Once the specimens reached a conditioned mass, the volume of each specimen was calculated. The racks containing the specimens were then submerged in a glass bowl filled with grade two distilled water. The bowl was sealed with plastic wrap and placed in an incubator kept at a constant of 37 (± 1) °C for seven days (± 2 hours). After the time had elapsed, the specimens were individually removed, dried and weighed. The specimens were reconditioned for a final time until a constant mass was reached. Using the recorded variables, and formulae provided by ISO 20795-1:2013(E), the sorption and solubility of the specimens were calculated according to the following formulae:

Water sorption (Wsp) was calculated in μg/mm³ using the formula recommended by ISO 20795-1:2013(E):

$$Wsp = \frac{m_1 - m_3}{V}$$

Water solubility (Wsl) was calculated in μg/mm³ using the formula recommended by ISO 20795-1:2013(E):

$$Wsl = \frac{m_1 - m_2}{V}$$

A Mettler AE 240 (Mettler Toledo, Ohio, USA) analytical balance scale mounted on a granite top was used to provide readings accurate to 0,1mg and indicating up to five decimal places. The weighing plate of the scale was situated in a glass enclosure with sliding doors to prevent any external variables such as moisture in the air from affecting the weight reading of the specimen. To calculate the volume of the specimens, the thickness readings were measured by means of a Toolquip & Allied Digital Outside Micrometer 0-25mm (Toolquip and Allied Ltd, Johannesburg, ZA) accurate to three decimal places, and the diameter readings with a Mitutoyo CD-15 DCX Digital Caliper (Mitutoyo Corporation, Kanagawa, JP) indicating up to 2 decimal places. All the instruments were calibrated by the internationally accredited “Lasec Group” prior to the commencement of the study.

Data analysis

Determination of any statistically significant differences between the means of the sorption and solubility characteristics of the surface-treated specimens was conducted by one-way analysis of variance (ANOVA). A p-value of 0.05 was considered to be statistically significant and values less than 0.05 were used to
conclude that a significant difference existed between the variables. The Tukey-Kramer multiple comparison test was further used to establish significant differences among the means of the different sample groups.

RESULTS
All analyses were performed using the NCSS (2019) Statistical Software package. The first analysis was a one-way analysis of variance on the Wsl and Wsp variables to test whether the within sample variances were equal and if the data showed a normal distribution. For Wsl a p-value of 0.102597 was recorded, indicating that no significant difference existed between the different treatment groups (p > 0.05). For Wsp a p-value of 0.000056 was recorded, indicating that a significant difference was found between the treatment groups (p < 0.05).

Sorption and solubility of the unpolished acrylic control group (Group A)
Group A, the control group was used to record baseline sorption and solubility values to assess the effectiveness of surface treatments on reducing the levels of sorption and solubility observed in Vertex™ Rapid Simplified denture base material (Fig.1a & Fig.1b). The specimens in sample group A obtained a mean sorption value of 22.3690 μg/mm³ (Table I) and a mean solubility value of 0.1843 μg/mm³ (Table II), which are both within the parameters set out by ISO 20795-1: 2013 (E) for a type-one polymer. The ISO 20795-1: 2013 (E) states that the sorption and solubility of type-one polymers should not exceed 32 μg/mm³ and 1.6 μg/mm³ respectively, in order to be deemed viable for clinical use. A single specimen in the group recorded a negative solubility value of -0.1891 μg/mm³ (Fig.1a) which indicated that it was not able to expel all the moisture it adsorbed during the saturation process. It was decided to keep this value in the recorded data as it is believed that the variance is not of such an extent as to affect the conclusions drawn from the study.

Sorption and solubility of mechanically polished specimens (Group B)
The specimens in sample group B (Fig.2a & Fig.2b) obtained a mean sorption value of 21.8613 μg/mm³ (Table I) and a mean solubility value of 0.1593 μg/mm³ (Table II), which are both within the parameters set out by ISO 20795-1: 2013 (E) for a type-one polymer. The Tukey-Kramer Multiple Comparison Test indicated that the lower sorption values were statistically significant in comparison to Group A. Even though mechanical polishing reduced the solubility values, it was not deemed statistically significant (Table III and Table IV).

Sorption and solubility of light-cured varnished acrylic soaked in distilled water (Group C)
The specimens in this group (Fig.3a & Fig.3b) obtained a mean sorption value of 21.3713 μg/mm³ (Table I) and a mean solubility value of 0.2406 μg/mm³ (Table II) which are both within the parameters set out by ISO 20795-1: 2013 (E) for a type-one polymer. The mean sorption value was lower and the mean solubility value was higher for this group than for Group A. The Tukey-Kramer Multiple Comparison Test indicated that the lower sorption values recorded by the specimens that were treated with the light-cured varnish soaked in distilled water were statistically significant. Even though the light-cured varnish increased the solubility values observed in Vertex™ Rapid Simplified denture base material, this was deemed not to be of statistical significance (Table III and Table IV).

Overall it was found that for sorption, sample Group C recorded the lowest mean value (21.3713 μg/mm³), followed by Group B (21.8613 μg/mm³) and Group A (22.3690 μg/mm³), whereas for solubility, sample Group B recorded the lowest mean value (0.1593 μg/mm³), followed by Group A (0.1843 μg/mm³) and Group C (0.2406 μg/mm³). Both groups B and C exhibited significantly lower Wsp values than the control group.

DISCUSSION
For this study, the objective of the control group was to record baseline sorption and solubility values to assess the effectiveness of surface treatments on reducing the levels of sorption and solubility observed in Vertex™ Rapid Simplified denture base material. A single specimen in the group recorded a negative solubility value of -0.1891 μg/mm³ which indicated that it was not able to expel all the moisture it adsorbed during the saturation process. Negative solubility values were also recorded by Tuna et al. [8], who suggested that the material or content within the material was responsible for bonding with the water.
molecules chemically. Due to the sensitivity of the scale, the possibility exists that this negative value was a result of human error. The sorption values recorded for this sample group were similar to those recorded by Engelbrecht (14), who also used an unpolished sample group soaked in distilled water fabricated from Vertex™ Rapid Simplified denture base material. Engelbrecht recorded a mean sorption value of 23 μg/mm³ for the sample group that received no surface treatment, soaked in distilled water. The solubility value recorded was however considerably higher, with a mean value of 1.1 μg/mm³. It is possible that the higher solubility value recorded is because of the thicker specimens and a different fabrication method from that used by this author. (14)

For sample group B, these specimens obtained lower mean sorption and solubility values than the control group, with only the reduction in sorption being deemed statistically significant. These findings are also in line with those of Engelbrecht and Al-Muthaffar (13), who found that surface treatment in the form of mechanical polishing was successful in reducing the mean sorption and solubility values experienced by heat-cured denture base material. Al-Muthaffar tested the effect of a conventional polishing procedure on the sorption of heat- and cold-cured denture base material and found that the conventional polishing procedure significantly reduced the amount of sorption experienced by the materials. Engelbrecht recorded similar results, with the conventional polishing procedure reducing both mean sorption and solubility values of the heat-cured denture base material, although only the reduction in sorption was deemed significant. It is noteworthy that the mechanical polishing of denture base material is associated with high levels of friction which may generate a considerable amount of heat within the material. As the heat generated is of a greater temperature than the flashpoint of methyl methacrylate monomer, it has been thought that the heat generated during the mechanical polishing procedure may serve to reduce the amount of residual monomer within the specimen, resulting in lower solubility levels.

Al-Muthaffar explains that the increase in temperature during the polishing procedure may also exceed the glass transition temperature of the material, resulting in the smearing of the material’s surface. The smeared surface is thought to decrease the surface polarity of the material, and effectively reduces the concentration of polar sites for water molecules to form hydrogen bonds with. As the resin’s polarity is one of the main factors governing the uptake of water into the structure of denture base acrylics, the reduction in the concentration of polar sites on denture base acrylics may reduce the rate of sorption observed in the material. (15) It is therefore possible that the generation of heat and the smearing of the specimens’ surface during the polishing procedure contribute to the positive effect that mechanical polishing has on the sorption and solubility of heat-cured denture base material. The extent of this is however unknown and more tests would need to be conducted to determine the exact effects that the heat generated during the polishing procedure has on the sorption and solubility of the material. There is also the possibility that the surface roughness of denture base materials may affect their sorption and solubility. Rough surfaces essentially have a larger surface area, which increases the contact interface between the water molecules and the surface of the denture base. The phenomenon is also explicable in terms of contact angle hysteresis. A study published by Rahal et al. investigated the influence of chemical and mechanical polishing on the water sorption and solubility of denture base acrylic resins. The authors noted that reducing the surface roughness of the material not only results in a smaller surface area but may also affect the hydrophilic nature of the material. (11) Monse ‘ne ‘go et al. suggest that water droplets form lower contact angles with rougher surfaces. Surfaces that produce lower contact angles are of a more hydrophilic nature, increasing the material’s affinity to water.

With respect to the third aspect of the study, the specimens in sample group C obtained a mean sorption value that was lower and a mean solubility value that was higher than the mean values recorded for the control group, with only the lower sorption values being statistically significant. These findings indicate that the application of Optiglaze™ light-cured varnish increased the observed levels of solubility. The increase in solubility may perhaps be attributed to the composition of this specific light-cured varnish. The application of Optiglaze™ had a positive result on the sorption of Vertex™ Rapid Simplified denture base material soaked in distilled water. A possible explanation for this occurrence may be that the application of Optiglaze™ alters the polarity of the specimen’s surface. As explained...
by Malacarne et al., indicates that Optiglaze™ may act as a surface sealer, sealing microscopic cracks, pores and irregularities on the surface of the specimens. It has also been suggested that water molecules are adsorbed to the surface of the material and are further absorbed into the body of the denture base through porosity and intermolecular spaces via diffusion. If the quantity of irregularities on the surface is reduced by the application of Optiglaze™, this may inhibit the uptake of water into the body of the material.

CONCLUSION

This study has established that the mechanical polishing of prostheses fabricated from Vertex™ Rapid Simplified heat-cured acrylic will reduce their sorption and solubility, but only the reduction in sorption can be expected to be statistically significant. The application of Optiglaze™ light-cured varnish to prostheses fabricated from Vertex™ Rapid Simplified heat-cured acrylic also significantly reduces the sorption of the material but will result in higher solubility values. When mechanical polishing is compared to Optiglaze™ light-cured varnish as a surface treatment, the sorption and solubility results indicate that mechanical polishing may be a more effective surface treatment option. However, Optiglaze™ light-cured varnish may be considered as an alternative surface treatment to mechanical polishing or used in conjunction with it as the sorption and solubility levels recorded were within the thresholds stipulated by ISO.

Limitations of the study

This study has identified some limitations: a test for statistical power was not conducted to determine the sample size, but rather the sample size was chosen in order to substantially exceed any previously published study of a similar nature. Secondly, while light-cured varnishes with different compositions may exhibit different sorption and solubility properties, this study, only investigated the effect of one light-cured varnish. In addition, the dimensions of the specimens recommended by ISO significantly differ to that of a removable prosthesis fabricated from heat-cured acrylic, thus the possibility exists that the results of this study may not accurately represent the sorption and solubility of complete dental prostheses fabricated from heat-cure acrylic. Concerning the solutes leaching from denture base materials during function, the exact substances leaching from the specimens are unknown so it is unknown whether the variables in this study would reduce the cytotoxicity of heat-cured acrylic material. Finally, the sorption and solubility of the study specimens were determined after being soaked for seven days, as recommended by ISO. It is therefore unclear what the long-term effects of surface treatments on the sorption and solubility of heat-cured acrylic materials would be.

REFERENCES