Measurement of water sorption of five different composite resin materials

Waleed M. Khalil, B.D.S., M.Sc. (1)

ABSTRACT

Background: Water sorption may affect composite resin materials by reducing their mechanical properties and wear resistance. The aim of this study is to measure the water sorption of five different composite resin materials, for different time intervals.

Materials and methods: A stainless steel mold of 4 mm width, 8 mm length and 4 mm depth was constructed to form composite blocks. Seven blocks were made of each test resin composite material. All specimens were placed in a silica-gel desiccator for 48 hours. The samples were then weighed thrice using calibrated electronic microbalance, the average reading was recorded to the nearest 0.0001g. The specimens were kept in individual containers in deionized distilled water at 37°C. All specimens were periodically weighed. The weight measurements were taken at 2, 8, 14, 28, 42, 56, 70, 100, 150 and 240 days intervals.

Results: The statistical analysis of the results showed that all composite resin specimens continued to gain weight for a period of 28 days. After that period, the weight gain continued, although at a reduced rate. The changes after the 28 days period were small for the Herculite XRV, Tetric ceram and Tetric composite resins, and greater for the Filtek P60 and Composan LCM composite resins.

Conclusion: In this study water absorption increased steadily for all materials. The percentage weight change showed a tendency to increase with the time of water storage. This study concludes that over a period of 8 months, the differences in water sorption capacity of the five composite resin materials are related to the differences in the amount of filler loading in the composite resin matrix. With one exception, the composite resin materials with the largest quantity of fillers had the least water sorption capacity after eight months of storage in water.

Keywords: Composite resin, water sorption, hygroscopic expansion. (J Bagh Coll Dentistry 2005; 17(3):37-41)

INTRODUCTION

Since the introduction of composite resin materials, one of their major disadvantages was that they undergo a sequence of dimensional changes during and following placement (1). These changes considered to be one of the main reasons for failures in the bond (2). The initial rapid polymerization shrinkage may be sufficient to disrupt the seal between the composite material and the structures to which it is bonded (1). The clinical effects of shrinkage can be minimized by using incremental placement technique, multiple-site light activation and slow low intensity light activation (3-5).

Once composite resins have polymerized, they are far from stable and will constantly be interacting with their surrounding environment. The principal interaction occurs with water since the restorative materials are continually bathed in saliva, and water absorption for some materials is inevitable (6). Water absorption by a material is the amount of water adsorbed through the exposed surface and into the body of the material (5).

Water diffuses into the matrix causing two opposing phenomena to take place. In some composites, water will leach out free unreacted monomers and ions (8). This outward movement of ions contributes to a further shrinkage and loss in weight of the material. Conversely, hygroscopic absorption of water leads to a swelling of the material and increase in weight (9-11). This phenomenon may allow for some degree of relaxation of the stresses which are set up within the matrix during polymerization shrinkage (2,12). Neither the original contraction stress nor the hygroscopic expansion will be uniform throughout the restoration. In contrast to rather rapid polymerization contraction stress development, the hygroscopic relief will proceed slowly and might even take days (2).

Water sorption may affect composite resin materials by reducing their mechanical properties and wear resistance. The water sorption by the polymer matrix could cause filler-matrix debonding or even hydrolytic degradation of the fillers (13,14). The water sorption by composite resin materials is a diffusion-controlled process, and the water uptake occurs largely in the resin matrix (3).

In the present study, the water sorption of different composite resin materials was determined as a function of time.

Table 1: Technical Profiles of the Composite Resins Evaluated
Physical and mechanical properties of glass ceramic crowns

Table 1. Physical and mechanical properties of glass ceramic crowns

<table>
<thead>
<tr>
<th>Product name</th>
<th>Composition</th>
<th>Filler volume</th>
<th>Filler size</th>
<th>Quantity of each Filler (wt%)</th>
<th>Index of Refraction</th>
<th>Hardness (Knoop)</th>
<th>Young’s Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X23</td>
<td>50</td>
<td>20</td>
<td>85</td>
<td>B2-SiO2</td>
<td>1.49</td>
<td>550</td>
<td>5.05</td>
</tr>
<tr>
<td>X24</td>
<td>50</td>
<td>20</td>
<td>85</td>
<td>B2-SiO2</td>
<td>1.49</td>
<td>550</td>
<td>5.05</td>
</tr>
</tbody>
</table>

MATERIALS AND METHODS

Table 1, lists the five types of composite resin materials investigated in this study.

A stainless steel mold of 4 mm width, 8 mm length and 4 mm depth was constructed to form composite resin blocks. The composite material was placed into the mold using a plastic instrument. Over the composite, a transparent celluloid strip and a microscope slide were placed and pressed to extrude the excess material, remove voids and produce a flat surface. The material was then photo polymerized using light curing unit (Kulzer Tranlux CS, Germany) by applying the tip of the light probe directly against the glass slide. Light activation was carried out for 1 minute, then the specimen was removed from the mold by applying slight finger pressure. Seven blocks were made of each test material. All specimens were placed in a silica-gel desiccator for 48 hours to ensure that the material would contract to a maximum capacity following light activation. Then the samples were weighed thrice using calibrated electronic microbalance (Sartorius-Germany), the average reading was recorded to the nearest 0.0001g.

The specimens of each group were kept in individual containers in deionized distilled water at 37 °C. All specimens were periodically weighed. Prior to weighing, the specimens were taken out of water, gently dried with blotting paper and left undisturbed for 4 minutes in order to allow stabilization of each specimen. The weight measurements were taken at 2, 8, 14, 28, 42, 56, 70, 100, 150 and 240 days intervals. The percentage weight changes were calculated using the following formula:

\[
\text{Weight change} = \frac{W_A - W_B}{W_B} \times 100\%
\]

Where \( W_A \) is the weight of the sample after immersion and \( W_B \) is the original weight of the sample before immersion.

One way Analysis of Variance test (ANOVA) was used to test whether there is any significant difference between the five composite resins at each storage interval. In case of a significant difference at (P < 0.05), then Least Significant Difference test (LSD) was used.

RESULTS

All of the composite resin materials increased in weight during immersion in water. The mean percentages weight changes after a period of 240 days were: Herculite XRV = 0.8444 % (S.D. = 0.1201), Composan LCM = 1.2934 % (S.D. = 0.3049), Filtek P60 = 1.1880 % (S.D. = 0.0889), Tetric ceram = 0.8014 % (S.D. = 0.1093), Tetric = 0.7973 % (S.D. = 0.1744). The rate of change in weight over the selected time intervals is presented in figure 1.

The graph shows how the specimens continued to gain weight for a period of 28 days.

Restorative Dentistry
After that period, these increases continued, although at a reduced rate. The changes after this time were small for the Herculite XRV, Tetric ceram and Tetric composite resins, and greater for the Filtek P60 and Composan LCM composite resins.

![Figure 1: Mean percentage changes in weight over 240 days](image)

**DISCUSSION**

Weight change in water was evaluated because saliva is a dilute fluid consisting of 99% water. The concentrations of dissolved solids (organic and inorganic) are characterized by wide variations, both between individuals and within a single individual therefore, deionized distilled water was used for a test standard (6).

The consequences of polymerization shrinkage in restorative dentistry have considerable effects like flexion and crack formation of the natural tooth structure or a break in the bond which will lead to the formation of gaps, micro leakage (15) and the potential for caries lesions formation (16,17). The polymerization shrinkage stresses can be relieved by the hygroscopic expansion of the material (2). The expansion resulting from water sorption can be clinically desirable if it fully counteracts the effects of shrinkage. If the hygroscopic expansion exceeds the polymerization shrinkage, further stresses may be introduced into the tooth (1).

The results of this study showed wide range of water sorption by the different composite resin materials. The resin matrix of these composite materials contains hydrophilic resins like Bis-GMA, TEGDMA and UDMA. These hydrophilic resins absorb more water and consequently expand to a greater degree than the hydrophobic resins (1,2,5,12). The volume of water that can be absorbed by a material is determined by the content of hydrophilic monomers that are present (1).

The rate of water sorption depends also on the type of filler, filler loading and filler-matrix adhesion. The volume of the filler occupying the total volume of the composite resin material will determine the amount of the resin matrix and the resultant capacity of the composite material to absorb water (18). Therefore, the material with lower filler content and higher resin matrix content had higher water sorption (6,8). The results of this study coincide with the above findings. The Tetric ceram and Tetric composite resins contain slightly higher filler volume than the Composan LCM and Filtek P60. An exception was the Herculite XRV, which showed low water sorption capacity in spite of its low filler volume. Two studies had shown that the Herculite XRV composite resin material contains higher filler loading (up to 87.1% by weight), than what is mentioned in it's manufacturer's instructions (up to 79% by weight) (19,20).

The air-filled voids incorporated in the composite resin during the handling of the material manually, may lead to inhibition zones with unpolymerized materials, which may result in higher water sorption (1,9). Also it can
be hypothesized that these voids may be filled with water, thus increasing the weight. The rate of water sorption also depends on the accessibility of water to the composite resin material (21). The rate and degree of water sorption and stress relief in composite restoration will be much lower than those found in the in vitro studies in which, composite resin blocks or disks were allowed to absorb water freely through all surfaces (2,15). Composite restorations with a large surface area of resin exposed to the mouth will absorb water more than smaller restorations in which, the resin is confined within two or three tooth surfaces (1).

Table 2: Descriptive statistics with ANOVA and LSD tests between the five composite resins for the same storage intervals

<table>
<thead>
<tr>
<th>Water storage intervals</th>
<th>Statistical analysis</th>
<th>Composite resin materials</th>
<th>Herculite XRV</th>
<th>Composan LCM</th>
<th>Filtek P60</th>
<th>Tetric ceram</th>
<th>Tetric</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 days</td>
<td>ANOVA Non significant difference</td>
<td>Mean %</td>
<td>0.3024</td>
<td>0.4101</td>
<td>0.3920</td>
<td>0.4383</td>
<td>0.3267</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1101</td>
<td>0.1713</td>
<td>0.1327</td>
<td>0.167</td>
<td>0.1168</td>
</tr>
<tr>
<td>8 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.4170</td>
<td>0.7087</td>
<td>0.5220</td>
<td>0.4983</td>
<td>0.3916</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1145</td>
<td>0.2822</td>
<td>0.0756</td>
<td>0.2136</td>
<td>0.1684</td>
</tr>
<tr>
<td>14 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.5729</td>
<td>0.8596</td>
<td>0.5597</td>
<td>0.6360</td>
<td>0.5077</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.0918</td>
<td>0.2386</td>
<td>0.1215</td>
<td>0.1615</td>
<td>0.1804</td>
</tr>
<tr>
<td>28 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.6581</td>
<td>1.0390</td>
<td>0.8170</td>
<td>0.7056</td>
<td>0.6846</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1497</td>
<td>0.3070</td>
<td>0.1068</td>
<td>0.1604</td>
<td>0.1828</td>
</tr>
<tr>
<td>42 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.7036</td>
<td>1.0931</td>
<td>0.8733</td>
<td>0.7281</td>
<td>0.7363</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1247</td>
<td>0.2840</td>
<td>0.0717</td>
<td>0.1569</td>
<td>0.2160</td>
</tr>
<tr>
<td>56 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.7436</td>
<td>1.0933</td>
<td>0.9231</td>
<td>0.7100</td>
<td>0.7413</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1194</td>
<td>0.2913</td>
<td>0.0451</td>
<td>0.1589</td>
<td>0.1780</td>
</tr>
<tr>
<td>70 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.7959</td>
<td>1.1464</td>
<td>0.9221</td>
<td>0.7203</td>
<td>0.7420</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1313</td>
<td>0.2695</td>
<td>0.0253</td>
<td>0.1110</td>
<td>0.1716</td>
</tr>
<tr>
<td>100 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.8417</td>
<td>1.2397</td>
<td>1.0110</td>
<td>0.7537</td>
<td>0.7797</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1508</td>
<td>0.2983</td>
<td>0.0611</td>
<td>0.1479</td>
<td>0.1856</td>
</tr>
<tr>
<td>150 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.8413</td>
<td>1.2714</td>
<td>1.0550</td>
<td>0.8000</td>
<td>0.7934</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1284</td>
<td>0.2808</td>
<td>0.0971</td>
<td>0.1555</td>
<td>0.1917</td>
</tr>
<tr>
<td>240 days</td>
<td>ANOVA Significant difference</td>
<td>Mean %</td>
<td>0.8444</td>
<td>1.2934</td>
<td>1.1880</td>
<td>0.8014</td>
<td>0.7973</td>
</tr>
<tr>
<td></td>
<td></td>
<td>SD</td>
<td>0.1201</td>
<td>0.3049</td>
<td>0.0889</td>
<td>0.1093</td>
<td>0.1744</td>
</tr>
</tbody>
</table>
REFERENCES