Physical Properties of Different Composites

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The purpose of this study was to determine the flexural strength (FS), flexural modulus (FM), depth of cure (DC), polymerization shrinkage (PS), and microhardness (VH) of the following composites:

- Two packable composites — Filtek P-60 and Solitaire 2;
- One ion-releasing composite — Ariston AT; and
- Two hybrid composites — Charisma and Filtek Z-250.

Data of the different tests conducted were obtained as follows:

- FS and FM data were determined with a universal-testing machine;
- Polymerization shrinkage was determined using the apparatus of Watts and Cash;
- Depth of cure was measured with a micrometer (Mitotuyo, UK); and
- Microhardness was measured with a Shimadzu Microhardness tester (500 g, 15 seconds).

All data of the different tests were statistically analyzed by one-way variance analysis (ANOVA), which yielded the following results:

- Filtek Z-250 showed the statistically highest flexural strength and modulus values.
- Solitaire 2 exhibited the highest shrinkage, while the volumetric shrinkage results of Filtek Z-250 and Filtek P-60 ranked the lowest.
- For depth of cure, there were no statistically significant differences among all materials tested (p<0.05).
- Microhardness results revealed the following tendency: Filtek Z-250=Filtek P-60>Ariston AT=Solitaire 2=Charisma.

Key words: Composite, Physical property

INTRODUCTION

Light-cured composite resins were intended to be used in general for both anterior and posterior restorations⁶⁻⁷. However, in the current market, materials are clearly distinguished as either for the anterior or posterior region. Composite resins for posterior restoration — as so claimed by manufacturers — are easy to handle and capable of obtaining ideal contoured interproximal contacts⁸. Over the past few years, manufacturers have changed the composition of composite resins to improve their physical properties⁹⁻¹⁰. These changes include: increasing the volume of filler particles, varying the size and type of particles, altering the chemistry of resin matrix, and increasing the molecular weight. As a result of these changes, new categories of composite resin are introduced: hybrid, ion-releasing, and packable.

Manufacturers claim that these formulation changes result in improved composites, such as less shrinkage during polymerization as well as improvements in depth of cure and wear resistance¹¹⁺. These improvements thus suggest that composite resins might serve as a suitable replacement for amalgam.

Against this background, the purpose of this study was to determine the flexural strength (FS), flexural modulus (FM), depth of cure (DC), polymerization shrinkage (PS), and microhardness (MH) of two packable composites (which are especially recommended for posterior use), an ion-releasing composite, and two hybrid composites.

MATERIALS AND METHODS

Table 1 lists the inorganic particles and resin matrix properties of the materials used in this study. The materials were applied and polymerized according to manufacturers’ instructions. Finally, all data of the different tests were statistically analyzed with one-way variance analysis (ANOVA) and post-hoc Bonferroni test at p=0.05 level.

Flexural strength and modulus tests

Flexural strength and modulus of elasticity were evaluated according to ISO 4049 specification¹². Specimens of 2 mm × 2 mm × 25 mm were prepared using a stainless steel split mold. The composite resin was applied into the mold, pressed, cured at three points, and then removed. The cured specimens were stored in demineralized water at 37°C for 24 hours. The specimens were positioned in a three-point bending apparatus on parallel supports and
Table 1 Materials used in the present study

<table>
<thead>
<tr>
<th>Material Category</th>
<th>Brand Name</th>
<th>Manufacturer</th>
<th>Resin System</th>
<th>Filler type</th>
<th>Average Filler Size (μm)</th>
<th>Filler Volume (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hybrid composite</td>
<td>Filtek Z-250</td>
<td>3M ESPE, Dental St Paul, MN, 55144</td>
<td>TEGDMA, UDMA, Bis-EMA (6)</td>
<td>Zirconia/Silica</td>
<td>0.6</td>
<td>60</td>
</tr>
<tr>
<td>Packable composite</td>
<td>Solitaire 2</td>
<td>Heraus Kulzer</td>
<td>UDMA, Bis-GA, TEGDMA</td>
<td>Ba-Al-B-F-Si Glas, Porous Siliciumdioxyde</td>
<td>0.7, 5, 8</td>
<td>66</td>
</tr>
<tr>
<td>Packable composite</td>
<td>Filtek P-60</td>
<td>3M ESPE, Dental St Paul, MN, 55144</td>
<td>TEGDMA, UDMA, Bis-EMA (6)</td>
<td>Zirconia/Silica</td>
<td>0.6</td>
<td>61</td>
</tr>
<tr>
<td>Ion-releasing composite</td>
<td>Ariston AT</td>
<td>Vivadent, Schaan Liechtenstein</td>
<td>Bis-GMA, UDMA, Dimetacrylate</td>
<td>Alkaline glass, Ba-Al Fluorosilicate, Ytterbiumtrifluoride, Silica</td>
<td>1.3</td>
<td>59</td>
</tr>
<tr>
<td>Hybrid composite</td>
<td>Charisma</td>
<td>Vivadent, Schaan Liechtenstein</td>
<td>Bis-GMA, UDMA, TEGDMA</td>
<td>Ba-Al F-Glas, Siliciumdioxyde</td>
<td>0.02-0.07</td>
<td>64</td>
</tr>
</tbody>
</table>

Polymerization shrinkage test
Five samples were prepared for each group, as described by Tolidis et al.10). Volumetric polymerization shrinkage was measured using the apparatus of Watts and Cash11) at 300 seconds. This apparatus consists of a linear vertical displacement transducer (LVDT), a light cure device, and a computerized recording system. Polymerization shrinkage was recorded by means of a data-logging software.

The testing procedure was as follows:
- A brass ring was placed on a glass microscope slide and filled with the restorative material.
- The brass ring, which was used to prepare the composite samples, had an internal diameter of 19 mm and a depth of 2 mm.
- A glass coverslip was put on the ring to flat the resin material.
- The LVDT, which was held in contact with testing resin composite sample, exerted approximately a 7-gram force during polymerization.
- The visible light irradiation unit (XL 3000, 3M, St Paul, MN, USA) was applied from the bottom of the testing samples and cured the material (as recommended by the manufacturer) during loading.
- Dimensional changes were monitored — during light polymerization and after the curing procedure (i.e., post-irradiation period) — for 300 seconds.

Depth of cure test
A stainless steel mold was used to prepare a cylindrical composite resin (8-mm height × 4-mm diameter). The mold was placed onto a strip of transparent film on a glass microscope slide. Wax was used to remove the specimen. The mold was filled with the test material (which was prepared according to manufacturer's instructions), and care was taken to avoid air bubbles. The mold was overfilled slightly, and a second transparent film strip was placed on the top followed by a second microscope slide. The mold and strips of film were pressed between the glass slides to extrude excess restorative material. Next, the mold was placed on a filter paper, the microscope slide covering the upper film strip was removed and the light cure device (Visiolux 105608, 3M, St Paul, MN, USA) gently placed through the orifice of the mold. The curing time applied was according to manufacturer's instructions. The specimens were removed from the mold, and the inadequately cured soft restorative material from the bottom of the mold with a plastic spatula. The height of the cylinder of cured material was measured with a micrometer to an accuracy of ±0.1 mm, and the obtained value was divided by two. This value was recorded as the depth of cure according to ISO 4049.
**Microhardness test**

A metal mold of 15-mm diameter × 10-mm height was prepared. The metal mold was filled with self-cured acrylic resin, and a 4-mm long × 4-mm wide × 4-mm deep hole was prepared. Test material was then applied into this 'cavity'. The composites were light-polymerized in 2-mm increments with a Visiolux (105608, 3M Dental Products Division, Germany) light curing unit according to manufacturer's curing instructions. A glass slide was placed over the composites, and pressure was applied to extrude excess material so as to avoid air bubbles and obtain a flat surface.

After light polymerization, the specimens were immersed in distilled water and stored at 37°C for 24 hours to achieve water sorption. Before hardness measurement, samples were polished to flat with 400-, 800-, and 1200-grit silicon carbide paper discs and alumina polishing paste (0.5 µ polishing). After 24-hour storage in distilled water at 37°C, the specimens were blotted dry and microhardness measurements proceeded. The specimens were subjected to a digital Vickers microhardness tester (Shimadzu MHT, HMV-2, Japan) and loaded with 500 g for 15 seconds. Three indentations were made and measured at different points on each specimen. Each specimen's average value was then determined. Five specimens were prepared for each composite resin group.

**RESULTS**

Table 2 lists the test results obtained in this study.

**Flexural strength test**

The FS test data showed that Filtek Z-250 had the statistically highest flexural strength value (172.6 ± 5.8 MPa) in comparison to other restorative materials.

Flexural strength data of Charisma restorative material showed the lowest (67.8 ± 13.2), and there were no significant differences in flexural strength between Charisma and Solitaire 2 (p > 0.05).

**Flexural modulus test**

Filtek Z-250 ranked the highest statistically (13 ± 0.5 GPa), while Solitaire 2 and Charisma ranked amongst the lowest at 5 ± 1 GPa and 6 ± 0.6 GPa respectively.

**Polymerization shrinkage test**

Volumetric shrinkage results showed that Solitaire 2 exhibited the highest shrinkage (2.7 ± 0.2%), while Filtek P-60 and Filtek Z-250 yielded the lowest shrinkage percentages at 1.7 ± 0.1% and 1.8 ± 0.1% respectively.

**Depth of cure test**

The depth of cure values ranged from 1.97 ± 0.12 to 3.07 ± 0.13 mm. There were no statistically significant differences among all groups (p > 0.05).

**Microhardness test**

Microhardness evaluation results were as follows:

<table>
<thead>
<tr>
<th>Material</th>
<th>FS (MPa)</th>
<th>FM (GPa)</th>
<th>PS (%)</th>
<th>DC (mm)</th>
<th>MH (VHN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtek Z-250</td>
<td>172.6 ± 5.8a</td>
<td>13 ± 0.5a</td>
<td>1.8 ± 0.1bc</td>
<td>3.07 ± 0.13</td>
<td>89.01 ± 6.87</td>
</tr>
<tr>
<td>Filtek P-60</td>
<td>130.3 ± 14.2b</td>
<td>11.8 ± 0.5b</td>
<td>1.7 ± 0.1a</td>
<td>2.82 ± 0.23</td>
<td>88.09 ± 2.71</td>
</tr>
<tr>
<td>Ariston AT</td>
<td>105.2 ± 15.6c</td>
<td>9.7 ± 0.6c</td>
<td>2.3 ± 0.1d</td>
<td>2.96 ± 0.15</td>
<td>63.4 ± 6.99</td>
</tr>
<tr>
<td>Solitaire 2</td>
<td>90.3 ± 2.2d</td>
<td>5 ± 1d</td>
<td>2.7 ± 0.2c</td>
<td>1.97 ± 0.12</td>
<td>59.86 ± 1.75</td>
</tr>
<tr>
<td>Charisma</td>
<td>67.8 ± 13.2e</td>
<td>6 ± 0.6e</td>
<td>2.1 ± 0.2ad</td>
<td>2.02 ± 0.57</td>
<td>60.35 ± 1.78</td>
</tr>
</tbody>
</table>

**DISCUSSION**

A restorative material should possess the properties of the lost hard tissue. Since dental restoratives are installed in a thermocycled, warm, and moist environment, their mechanical properties substantially influence a restorative material's quality and longevity. Therefore, it is necessary to investigate the factors that influence a restorative material's mechanical properties, which can be classified into two categories: nature of the restorative material and application procedure.

A resin material's nature is determined by filler type, load, size, and resin formulation. Hoffman et al. reported that filler particles incorporated into a matrix provided much better mechanical properties than the matrix itself. Therefore, up to a certain limit, a higher filler load may be expected to improve mechanical properties. In the present study, all restorative materials had a filler load near to 60%. Solitaire 2 had the highest filler load, but not the best physical properties, which leads to the postulation that resin formulations are important as well.

The second influencing factor is the application procedure which includes the layering technique and polymerization efficacy. Light source application direction, curing time, and apparatus intensity can in-
fluence polymerization quality, which consequently affects a restorative material’s mechanical properties and its clinical performance.

A material’s fracture-related properties — such as fracture resistance, elasticity, and marginal degradation under stress — are usually determined by these parameters: flexural strength, flexural modulus, and fracture toughness15). In this study, the flexural strength (FS), flexural modulus (FM), depth of cure (DC), polymerization shrinkage (PS), and microhardness (VH) of two packable composites (Filtek P-60, Solitaire 2), one ion-releasing composite (Ariston AT), and two hybrid composites (Charisma, Filtek Z-250) were evaluated.

**Flexural strength**

Flexural strength is a more clinically relevant test of material strength and is especially important if the material is used for Class I, II and IV cavities, which are usually subjected to high forces. Higher flexural strength materials are less prone to bulk fracture of the filling as well as fracture of the margins12,15).

Flexural strength tests are sensitive to surface imperfections such as cracks, voids, and related flaws which can influence the fracture strength of brittle materials. High flexural strength values reflect a limited tendency to crazing and high resistance to surface defects and erosion16).

A complete resin cure is necessary to achieve its maximum mechanical strength and therefore provide higher bond strengths17). In this study, the hybrid composite Filtek Z-250 showed higher flexural strength data than the packable restorative materials, Filtek P-60 and Solitaire 2. However, these tests need further clinical observation because occlusal forces may influence flexural strength.

Filtek Z-250 and Filtek P-60, due to their higher molecular weight and filler volume, resulted in higher flexural strength values. The strengthening effect of inorganic fillers depends on their chemical structure (e.g., pyrogenic silica or glass filler), as well as size and distribution of particles used. Larger and harder particles result in higher strengthening effects1).

**Flexural modulus**

Flexural modulus is a means of defining a material’s stiffness16). Flexural modulus is obtained from the load-deflection trace during flexural strength testing15). Charisma and Solitaire 2 showed the lowest flexural moduli in this study. A low modulus indicates a flexible material. Filtek Z-250 and Filtek P-60, which have higher molecular weight and filler volume, yielded higher flexural modulus values — which means they are less flexible materials. It can be concluded that high flexural strength materials (see Table 2) need to be coupled with stress absorbing material, filled bonding or more layers of bonding agent to fortify the restoration material against biting forces. Many researches have focused on this topic18,19). Civelek et al.20) reported that Filtek Z-250 showed less microleakage with a flowable composite, as opposed to applying a total etch adhesive system (i.e., the hybrid composite resin alone without any flowable resin).

**Polymerization shrinkage**

Polymerization shrinkage of a composite resin occurs when the composite monomer polymerizes21). The setting reaction is accompanied by volumetric shrinkage of varying magnitudes, depending on the resin formulation22). Manufacturers claim that if resin has higher molecular weight, it will lead to less shrinkage, reduced aging, and a slightly softer resin matrix. UDMA (urethane dimethacrylate) and Bis-EMA (Bisphenol A polyethylene glycol diether dimethacrylate) impart a greater hydrophobicity and are less sensitive to changes in atmospheric moisture. Therefore, higher molecular weight results in less shrinkage23).

Both Filtek Z-250 and Filtek P-60 have higher molecular weight (Bis-GMA (512 g/mole), TEGDMA (286 g/mole), and UDMA (629 g/mole)) and filler volume. The filler load contents of Filtek Z-250 and Filtek P-60 are 60 vol% and 61 vol% respectively, which accounted for the lowest polymerization shrinkage in the present study — at 1.8±0.1% and 1.7±0.1% respectively.

It was reported that inorganic filler’s amount, type, and size are also factors which influence polymerization shrinkage23). The higher the filler amount in a resin composite, the smaller would be the shrinkage24). Solitaire 2 was found to have the highest filler load, but not the lowest polymerization rate in this study. This is because Solitaire 2 and Charisma posses glass fillers (see Table 1), and glass inserts or pre-polymerized balls of resin composite minimize resin volume which shrinks during polymerization25).

A resin composite demonstrating low polymerization shrinkage percentage in vitro is clinically preferred23), but must be tested under in vivo conditions too. In this study, Filtek Z-250 and Filtek P-60 showed the lowest polymerization shrinkage values.

The polymerization shrinkage apparatus evaluates a restorative material’s shrinkage without taking into account any cavity factor or influence from any bonding agent. Nonetheless, a previous research reported that no relationship was observed between in vitro polymerization shrinkage percentages and the dye penetration scores for restoratives Filtek Z-250, Admira, and Ariston AT26).

In the present study, no direct relationships could be found between polymerization shrinkage data and resin formulation or filler particle properties. Instead, the relationship differs amongst the
materials. The obtained results revealed that the interaction between inorganic filler particles and resin matrix could also be an influencing factor on polymerization shrinkage.

**Depth of cure**

Manufacturer of Filtek Z-250 and P-60 claim that with the new resin system of higher molecular weight and filler content materials, these composites yield fewer double bonds to cross-link so that the resin is cured more efficiently. The depth of cure values of the restoratives ranged from 1.97±0.12 to 3.07±0.13 mm. There were no statistically significant differences among all groups (p>0.05). The results showed that all resins could provide a more adequate depth of cure and more effective bonding capacity when the layering technique was utilized. Moreover it is not possible to apply the packable composite material (Filtek P-60) by the bulk technique in cavities over 2.5 mm — because Filtek P-60 has a 2.82±0.23 mm depth of cure. Cobb et al. researched the physical properties of packable and conventional posterior resin-based composites. However, their strength data represent incremental placement — not bulk cure — of composites tested; whereas depth of cure test data is based on bulk polymerization. In this study, the height of the cylinder of cured material was measured with a micrometer and the mean values evaluated. The height of the material was divided by two, as recommended by ISO 4049. The obtained value highlighted a change to what is recommended for layering technique.

Light intensity, cure time, and composite material (resin, filler, opacity, and shade) impact depth of cure. Partially polymerized and therefore weakened composite at the interface creates a weak area for bond failure to occur. Lovell et al. reported that highly cross-linked dimethacrylate systems, such as Bis-GMA/TEGDMA, exhibit similar network structure and properties as a function of double bond conversion, regardless of curing method or time. In this study, a light source (XL 3000, 3M, St Paul, MN, USA) with a halogen lamp of 650 mW/cm² light intensity was used for curing procedure and applied in the direction according to manufacturer's instructions for all composite resins.

**Microhardness**

In the present study, Filtek Z-250 and Filtek P-60 (which have higher molecular weight and filler volume) exhibited the highest microhardness values, while Ariston AT, Solitaire 2, and Charisma showed the lowest.

Composites with harder and larger particles have higher strengthening effect, but worse polishability. The strength and chemical stability of the interfacial bond between resin and filler also greatly influence the clinical behavior of the composite material.

Therefore microhardness-versus-wear and microhardness-versus-fracture toughness relationships are important topics for researchers. Filler level, particle size and distribution, and resin matrix properties do significantly influence microhardness. Materials for posterior use typically display good Vickers hardness values (as compared to those used for dentin) and relatively high compressive strength values. Hence these materials should be able to support occlusal stresses.

Physical property studies are accepted methods to evaluate resin materials. However, clinical follow-ups are necessary to determine their long-term performance under physiological conditions. The choice of restorative material rests upon the expected properties that each cavity or restoration area demands. Be it flexibility, wear or esthetic demand, it varies in every clinical situation: Class I, II, III, IV, V or direct veneering restoration.

**CONCLUSION**

This study showed that stringent prediction of physical properties for a particular category of resin materials is not feasible. This is because the results differed statistically even between materials of the same category, for example between two packable or two hybrid composites. It was concluded that higher molecular weight materials used in this study (Filtek P-60 and Filtek Z-250) are more suitable for posterior restorations.

Future studies will need to focus on correlations between physical property tests and clinical evaluations, such as marginal discoloration, staining, fracture resistance, durability in stress bearing situations, and wear in oral conditions.

**REFERENCES**