Micromorphology, microstructure and topography characterization of resin materials

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Abstract

The morphologies, microstructures, of the adhesive restorative materials were systematically investigated. The objective of the present study was to make a comparative analysis of the physical properties by scanning electron microscopy and atomic force microscopy analysis of four different resins composite materials. For atomic force microscopy the materials were put between two matrix strips of celluloid and two glasses plates to obtain a flat surface. For scanning electron microscopy analysis the materials were put into standard dental human cavities performed with diamond burs The specimens were sectioned longitudinally, flattened, polished, thermo cycled from 50-500 C for 500 cycles, prepared for scanning electron microscopy observation (JEOLJSM 6390ª) and statistically analyzed using One-sample Test (p≤0.05). The surface roughness and topography were characterized by atomic force microscopy (Park SYSTEMS XE-100). There were differences in surface roughness microstructures and morphologies among the resin composite. The roughness surface of resin composite materials, in descending order, was achieved subsequent to the use of nanocomposite, ormocer, giomer, and microfiller resin composite material.

Key words: scanning electron microscopy, atomic force microscopy, ormocer, nanocomposite, giomer

Introduction

Evolution of dental plastics materials is in continuous expansion. Characteristics of an ideal restorative material are numerous. The finishing and polishing of dental materials are important stages of restorative technics for esthetics and clinical performance. [1, 2] Surface finish quality of tooth-colored restorations is a determinant factor in the esthetics and longevity of such restorations [3, 4] Surface roughness is the main factor determining bacterial adhesion and plaque formation on the dental surfaces in vivo and may cause gingival irritation and dental caries. [5]. Another important factor for the durability of restorations is adhesion of dental materials to dental substructures. This is dependent on a number of related factors: dentin substrate characteristics the type of approach to dentin substrate and the type of material used [6-8]. The objective of the present study was to make a comparative analysis of the physical properties of four different resins composite (RC) materials.

Materials and methods

Four resins composite were investigated as listed in Table 1.
Table 1. Lists of materials investigated

<table>
<thead>
<tr>
<th>Categorization</th>
<th>Brand Name</th>
<th>Code</th>
<th>Manufacturer</th>
<th>Setting Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin nanocomposite</td>
<td>Filtek Z 250</td>
<td>FiZ</td>
<td>3M ESPE, St Paul, MN, USA</td>
<td>L</td>
</tr>
<tr>
<td>Micro-filled resin composite</td>
<td>Gradia</td>
<td>Gr</td>
<td>GC Europe, Leuven</td>
<td>L</td>
</tr>
<tr>
<td>Ormocer</td>
<td>Admira</td>
<td>Am</td>
<td>Voco, Anton-Flettner-Str., Cuxhaven, Germany</td>
<td>L</td>
</tr>
<tr>
<td>Giomer</td>
<td>Beautifil II</td>
<td>Gi</td>
<td>Shofu Dental Corporation Osaka, Japan</td>
<td>L</td>
</tr>
</tbody>
</table>

L: Light-curing;

Surface roughness and topography characterization

The materials were prepared for one drop in conformity with manufacturer indication. After preparation the materials were put between two matrix strips of celluloid (Nr. 437 Alfred Becht GmbH-D 7600 Offenburg, Germany) and then between two glass to obtain a flat surface. The materials samples with photo initiator were light-cured for 40 seconds through the glass slide with a conventional quartz halogen lamp (QTH-3MESPE), flux density 570mW/cm². The microstructures, the composition and the dental penetration of the materials were characterized by scanning electron microscopy (SEM) (JEOLJSM 6390ª Japan). The roughness and the topography of the surface were characterized by atomic force microscopy (AFM) (Park SYSTEMS XE -100). AFM was used in non-contact mode using single crystal silicon tip (with nominal radius<10 nm), which was connected to a fixed substrate on a cantilever. The images were recorded with a scan rate of 0.5 Hz and a resolution of 256 × 256 pixels per image. For each specimen, two scans were carried out at each specimen surface quadrant at a scanning area of 10 µm×10 µm. The collected 3D topographical data was analyzed with data analysis software (XEI - Image Processing and Analysis). For each group, the surface roughness was calculated in nm.

Surface characterization by scanning electron microscopy (SEM) analysis

Twenty caries-free human molars and premolars were extracted from orthodontically and periodontologically reasons. The teeth were obtained from patients who required an extraction as a routine part of their treatment. The research was conducted with the agreement of the Ethics Commission of the "Gr.T. Popa” University of Medicine and Pharmacy Iasi. The teeth were mechanically brushed with a non-fluorurate abrasive paste, rinsed with dionised water and stored in 0.5% chloramine solution at 4° C. Twenty standard cavities were performed at each tooth: 2.5 mm depth and 4mm wide. Cavities were performed mechanically (M) at slow and high speed under water spray with round and cylindrical diamond burs no:1. The teeth were randomly assigned in four equal groups and then were prepared and restored according to the manufacturers instruction as follows: Group 1 filled with FiZ (3MESPE); Group 2 filled with Gr (GC); Group 3 - filled with Am (Voco); Group 4- filled with Gi (Shofu). The samples were cut longitudinaly (mesio-distal) with diamond disc under water cooling and then washed with ethanol. The samples are submitted to a mechanically polishing process using diamond (particles size – 3µm) and Syton (particles size – 20nm) under continuous irrigation. The role of this step is to bring the materials to the same length on the surface and to obtain very smooth surfaces. The samples were etched with H₃PO₄ 35% for four seconds and washed for two minutes with distilled water [8]. The teeth
were then stored in saline solution 48 hours. The modality by adhesion was characterized by SEM with JEOL JSM 6390ª Japan. The measure was performed at walls of the restura in at least three different points, making the average for each sample.

**Results and discussions**

*Surface roughness and topography characterization*

**Table 2.** Analysis of the surface roughness Ra [nm] for 10 µm obtained from AFM images.

<table>
<thead>
<tr>
<th>Material</th>
<th>Rpm(nm)</th>
<th>Ra [nm]</th>
<th>Rz(nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FiZ</td>
<td>44,761</td>
<td>8,405</td>
<td>37,344</td>
</tr>
<tr>
<td>Gr</td>
<td>12,52</td>
<td>2,327</td>
<td>10,381</td>
</tr>
<tr>
<td>Am</td>
<td>15,59</td>
<td>4,125</td>
<td>15,043</td>
</tr>
<tr>
<td>Gi</td>
<td>5,790</td>
<td>4,963</td>
<td>20,474</td>
</tr>
</tbody>
</table>

The FiZ (3MESPE) has a higher roughness of the surface. (Table 2) This material contains: organic matrix Bis-GMA, Bis-GMA, TEGMA, UDMA; filler: 60% in volume and 77.6%wt of non-agglomerated silica nanoparticles with mean size of 20 nm and nano agglomerations composed of zircon-silica particles with a size varying from 5 to 20 nm. Mean agglomeration size varied from 0.6 to 1.4 ym. Filtek Z250 was granted the highest award by the Dental Advisor [9] for very good mechanical properties, excellent aesthetics and satisfaction by dentist and patient.

The lowest surface roughness was measured for the RC Gr (GC). (Table 2) This material contain silica and pre-polymerized fillers 75% by weight and 64% by volume (avg. particle size 0.85 microns) in a urethane dimethacrylate co-monomer matrix. Gradia Direct, was rated highly for polish ability, aesthetics, and shade matching by clinical evaluators. [10]

The roughness of the Am (Voco) was 2 times higher than that of the Gr (GC) but lowest 2 times than that of the FiZ (3MESPE). (Table 2)

The dual character of the Am as inorganic-organic copolymers is the key for improving the properties of filling composites. The organic, reactive monomers Bis-GMA, TEGMA, UDMA are bound in the sol-gel process by the formation of an inorganic network with filler: 3% apatite-sulfate-phosphate and 77% inorganic glass particles.

The roughness of the giomer was 2 times higher than that of the Gr (GC). (Table 2)

The giomer hybrid restorative materials containing mainly resin and glass ionomer particles prereacted (PRG-Prereacted Glass-Ionomer - they contain ions of glass, fluorine-amino-silicate in combination with polyacrylic acid) into organic matrix formed by bisphenol A-glycidyl-dimethacrylate, TEGMA, inorganic glass filler, aluminoxide and silica.

Morphologically, by AFM, the particle of the FiZ (3MESPE) materials appeared to be exposed to a higher degree than the particle of the giomer and than the particle of those micro filled resin composite, Gr (GC) and ormocer composite, Am (Voco) (Fig. 1-4).

**Figure 1.** AFM view of the surface filled with FiZ (3MESPE)
AFM images show a relatively smooth natural surface at Gr (GC) material (Figure 2). Decreased surface irregularities are observed in the AFM images of ormocer Am (Fig. 3) and giomer Gi (Fig. 4).

AFM has several advantages for surface analysis, including higher resolution and the ability to provide 3D topographic images of the surface and suitability for qualitative and quantitative comparison of the surface texture and roughness [11].

The surface roughness of the giomer specimens treated with all prophylaxis methods was greater than 0.2 µm, which is a threshold value for bacterial adherence [12]. The differences in the substrate of materials can be considered another reason for different results. It has been reported that the hardness, the initial surface roughness, filler size, filler content and water absorption of the substrate affect wear resistance [3, 4, 13 and 14].

**Surface characterization by SEM analysis**

Surface characterization by SEM showed the microstructures and morphologies of the RC materials is different (Figure 5).
Micromorphology, microstructure and topography characterization of resin materials

Figure 5. Top-view SEM photomicrographs of a template filled with FiZ (3MESPE) (a), Gr (GC) (b), Am (Voco)(c) Gi (Shofu)(d)

Morphologically by SEM micrograph shows no difference between the diameter and the shape of the micro particles of the RC and giomer. Filler particle size range from 0.60 µm to 2.00 µm for FiZ (3MESPE) and from 0.80 µm to 1.40 µm for the other. Also these materials are homogeneous. Top-view SEM micrographs of a sample filled with Fi (3MESPE) is present in Figure 2a, for Gr (GC) materials in Figure 2b, for ormocer Am (Voco) in Figure 2c, and for Gi (Shofu) in Figure 2d. RC have an omogeneous structure with a little variety filler particle size.

The results for a study indicated that the ormocer, like Amira (Voco), which was developed by Ormocer technology, demonstrate a higher micro hardness and wear resistance when it is compared with a hybrid composite [13]

The quality of the resin-tooth structure interface determines the integrity and durability of the adhesive restorations [15-17]. Our study put in evidence a continuously hybrid layer for all the samples, but with differences between thickness. (Fig. 6 a-d)

Figure 6. Top-view SEM photomicrographs of the template filled with FiZ (3MESPE) (a), Gr (GC) (b), Am (Voco)(c) Gi (Shofu)(d)

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SEM micrograph analysis of hybrid layer (Fig.6 a-d) show that the best results were obtained for the samples, filled with Gi=4.24 (±1.33)µm, followed by Am=4.16 (±2.28)µm, Gr.=2.7(±0.19)µm and FiZ=1.50 (±0.38)µm. The differences were statistically significant p≤0.05 (Table 3).

Table 3. One-Sample Test

<table>
<thead>
<tr>
<th></th>
<th>Test Value = 0</th>
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<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>t</td>
<td>df</td>
<td>Sig.(2-tailed)</td>
<td>Mean Difference</td>
<td>95% Confidence Interval of the Difference</td>
<td>Lower</td>
</tr>
<tr>
<td>GR. FILTEK Z 250</td>
<td>8.79</td>
<td>4</td>
<td>0.001</td>
<td>1.50</td>
<td>1.03</td>
<td>1.98</td>
</tr>
<tr>
<td>GR. GRADIO</td>
<td>31.83</td>
<td>4</td>
<td>0.000</td>
<td>2.70</td>
<td>2.47</td>
<td>2.93</td>
</tr>
<tr>
<td>GR. ADMIRA</td>
<td>4.06</td>
<td>4</td>
<td>0.015</td>
<td>4.16</td>
<td>1.32</td>
<td>7.00</td>
</tr>
<tr>
<td>GR. BEAUTIFIL</td>
<td>7.10</td>
<td>4</td>
<td>0.002</td>
<td>4.24</td>
<td>2.58</td>
<td>5.90</td>
</tr>
</tbody>
</table>

Conclusion

Given the results of the current study, further investigations on the micro morphology, microstructure and topography of resin composite restoratives materials are warranted. Within the limitations of this study it was concluded that all the materials have a degree of roughness. The roughest surface of resin composite materials, in descending order, was achieved subsequent to the use of nanocomposite, ormocer, giomer, and micro-filler resin composite material. Morphologically by SEM, micrograph shows the fact that the diameter and the shape of micro particles of RC are different. The resin restoration material influences the dimensional level of intrication of the material into the teeth.

Acknowledgement

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References