EFFECT OF FOOD SIMULATING SOLUTIONS ON SURFACE ROUGHNESS OF FOUR RESTORATIVE MATERIALS

Asmaa M. Abdallah 1*, Rabab Mehesen 1

ABSTRACT

Objective: To evaluate the influence of FSSs on surface roughness of restorative materials. Materials and Methods: Eighty discs of Equia forte, Activa Bioactive composite, Cention-N, and Tetric-N Ceram Bulk Fill were prepared in a customized mold (2×10mm). The discs of each restorative material were divided into 2 subgroups as follows: one immersed in 50% aqueous ethanol (FSS1), and the other in methyl ethyl ketone (FSS2). The specimens were stored in the solutions for one week at 37°C. Surface roughness was measured before, and after immersion and the statistical analysis was performed by One Way (ANOVA) followed by post Hoc Tukey test for pairwise comparison. Percent of change was done using Z -test. Results: All the tested groups showed significance differences for ∆Ra in both FSSs. There were significance differences between FSS1 (∆Ra1) for all the tested groups, and FSS2 (∆Ra2) between Activa Bioactive and Cention-N. There was a statistical significance difference between ∆Ra 1 and ∆Ra 2 for Cention-N group. Conclusions: The effect of FSSs on alkasite based restorative material is comparable to that of the most commonly used tooth-colored direct restorative materials in clinical practice.

KEYWORDS: Alkasite based restorative material, FSSs, surface roughness

INTRODUCTION

The need for an ideal restorative material that can restore and replace natural enamel and dentin tissues with adequate mechanical, self-adhesive, and caries-preventive properties, as well as insensitive clinical application procedures, has sparked many revolutions in restorative materials technologies over decades of time (1). These restorative materials include resin composite, glass ionomer, and modified hybrid of both to exploit their perfect properties and improve their drawbacks. Resin composites have acceptable esthetic and mechanical properties, and glass ionomer has the ability to discharge fluoride ions into underlying dentin, and has self-adhesive monomers that attach chemically to hydroxyapatite in enamel and dentin (2).

Resin-modified glass ionomer is regarded as one of mostly used hybrid restorative material that was introduced to improve the mechanical and optical properties, despite of its higher incidence of degradation when compared to resin composite (3). Nanotechnology has been used to develop high viscosity glass ionomer to improve the physical characteristics and bioactivity of conventional glass ionomer (4). An alkasite material is introduced as one of the most recent modifications of hybrid restorative materials that is resin-based, and dual-curing in powder/liquid form. The organic monomers present in the liquid. The inorganic fillers comprise barium aluminium silicate glass filler, ytterbium trifluoride, iso filler, calcium barium aluminium fluorosilicate glass filler and a calcium fluoro silicate (alkaline) glass filler (5).

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It is radiopaque, contains alkaline fillers that release fluoride, calcium, and hydroxide ions, as well as an organic monomer with a high compressive strength that can release acid buffering ions. It has been created as a tooth-colored substitute to amalgam and can be utilized as a bulk restorative material.

Any restoration’s clinical effectiveness and durability are determined by how well it can withstand deterioration and erosion by extrinsic or intrinsic acids, which may cause changes in surface smoothness. Intraorally, the restorations are prone to chemical agents found in saliva, food, and various beverages on an intermittent or continuous basis. The chemical erosion by extrinsic factors may increase surface roughness of restorative materials leading to bacterial retention, carious lesions and periodontal diseases.

The erosion effect of organic acids and food simulating solutions on surface properties such as surface roughness had been previously explored. Ethanol is an organic solvent that is used as a food simulant by the US Food and Drug Administration (FDA). Methyl ethyl ketone (MEK) is naturally present in various types of fruits, meat, vegetables, and yogurt has been approved by the FDA as a food simulating liquid. Effectual in-vitro evaluation of restorative materials, including information on the effect of food-simulating solutions on the surface roughness of modified glass ionomers is currently scarce. Thus, the aim of this study was to investigate the effect of food simulating solutions on the surface roughness of different types of restorative materials; methyl methacrylate resin composite, resin modified glass ionomer, high viscous glass ionomer, and alkalsite based resin composite. This study designed to test the null hypothesis that there is no significant effect of food simulating solvents on surface roughness of these four restorative materials.

### MATERIALS AND METHODS

Four types of commercial restorative materials were used for this investigation and categorized as groups A, B, C, and D. The groups were respectively; Group A for high viscosity glass ionomer (Equia Forte, GC Corporation, Tokyo, Japan), Group B for resin-modified glass ionomer (ACTIVA BioACTIVE Restorative, Pulpdent Corporation Watertown, USA), Group C for alkalsite based composite resin (Cention N, Ivoclar Vivadent, Schaan, Liechtenstein), and Group D for bulk-fil resin composite (Tetric N Ceram Bulk, Ivoclar Vivadent, Schaan, Liechtenstein), Table 1. Twenty disc-shaped specimens from each material were prepared using a split Teflon mold (10 mm diameter x 2 mm height).

### TABLE (1) Materials used in the study.

<table>
<thead>
<tr>
<th>Product</th>
<th>Composition</th>
<th>Manufacturer</th>
<th>Lot number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Equia Forte</td>
<td>Powder: Fluoro-alumino-silicate glass, Polyacrylic acid powder, Pigment Liquid: Polyacrylic acid, Distilled water, Polybasic carboxylic acid</td>
<td>GC, Tokyo, Japan</td>
<td>150213B</td>
</tr>
<tr>
<td>Activa Bioactive</td>
<td>Blend of diurethane and methacrylates with modified polyacrylic acid (44.6%); reactive glass filler (21.8 wt.%); inorganic filler (56 wt.%), patented rubberized resin (Embrace), water.</td>
<td>Pulpdent, Watertown, USA</td>
<td>160314</td>
</tr>
<tr>
<td>Cention-N</td>
<td>Liquid: Dimethacrylates, initiators, stabilizers, additives and mint flavour. Powder: Calcium fluoro-silicate glass, barium glass, calcium-barium-aluminim fluoro-silicate glass, iso-fillers, ytterbium trifluoride, initiators and pigments. 78.4 wt%, or 57.6 vol% of inorganic fillers, particle size 0.1 and 7 μm.</td>
<td>Ivoclar vivadent, Liechtenstein</td>
<td>Z00547</td>
</tr>
<tr>
<td>Tetric-N Ceram</td>
<td>Monomer matrix (21 % weight): BIS-GMA, UDMA, BIS-EMA Inorganic fillers 75% (by weight): Bromium glass, ytterbium trifluoride, mixed oxides and coplyomers, and prepolymmerized filler (prepolymers)</td>
<td>Ivoclar vivadent, Liechtenstein</td>
<td>V24958</td>
</tr>
<tr>
<td>Bulk fill</td>
<td></td>
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</tbody>
</table>
**Group A;** was restored with EQUIA Forte capsules which were mixed in the capsule mixer for 10 s. With the help of the GC capsule applier the mixed cement was overfilled into the mold and was pressed between two glass plates with the Mylar strips placed between Teflon mold and the glass plate to prevent the adhesion of mixed material to glass plates. The glass plates were held firmly during setting to avoid the presence of air bubble and to obtain a smooth surface. **Group B;** was restored with Activa bioactive composite; Two-paste system dispensed directly from an automix syringe and the polymerization procedure was carried out through the polyester strip from top and bottom following the manufacturers’ recommended exposure time, using a light-curing device with a visible light intensity of 500 mw/cm² (DabiAtlante, RibeirãoPreto, SP, Brazil). The light intensity was controlled at 500 mw/cm² by measuring with curing radiometer.

**Group C;** was restored with Cention N in which 2 scoops of powder and 2 drops of liquid resin were dispensed and hand mixed to a smooth consistency on a mixing pad. The liquid was first mixed with half of the powder until well wetted, and then the remaining powder was added in small increments. Mixing time did not exceed 60 seconds. Then, the paste was applied with a spatula into the mold, covered with a Mylar matrix, and squeezed to a flat surface. The material was left untouched for 10 minutes from the beginning of mixing. **Group D;** was restored with Tetric N Ceram Bulk fill; the resin composite was inserted with Optra Sculp (Ivoclar Vivadent AG, Schaan, Liechtenstein) modeling instrument, adapted in the mold as in the other groups, and light cured through the polyester strip from top and bottom sides. After setting, the pellets were removed from the mold, and the excess was trimmed using a Bard-Parker blade. All the specimens were polished using disc-shaped polisher (Pogo, DENTSPLY, Caulk, USA), lower surface of each specimen was marked with a number to identify each side, and stored in artificial saliva for 24 H at 37°C in an incubator (BTC, Biotech, Egypt).

All groups were divided according to food simulating solution into two subgroups. Subgroup1; the specimens were immersed in 50% aqueous ethanol (FSS1), whereas subgroup 2; the specimens were immersed in methyl ethyl ketone (FSS2). Each specimen’s top surface was measured for surface roughness as a baseline record before the immersion in FSS1, and 2. The average surface roughness (Ra) was examined by a profilometer (Surttest SJ210, Mitutoyo Corp., Kawasaki, Japan) in which the probe was placed in the middle of the specimen surface. Each specimen was scanned 5 times and the mean roughness parameter (Ra) was recorded in µm. The tracing length was 0.8 mm, at scanning speed 0.5 mm/s, and the resolution of the recorded data was 0.01 µm.

According to Food and Drug Administration guidelines for chemistry and technology (FDA 1976, 1988), the chemical agents; were used in this study; are considered as food simulators. 50% aqueous ethanol (Ethyl Alcohol Mr 23 gm/mol, PIOCHEM, 6th October City, Egypt) is a simulator to mouth rinses, certain beverages, vegetables, fruits, candy, and syrup. Methyl ethyl ketone (MEK; 2-Butanone, PIOCHEM, 6th October City, Egypt) is a simulator to fruits, yogurt, butter, fat meals and vegetable oils. The specimens were kept in individual vials with 20 mL of each solution for 7 days at 37°C. After the immersion period; the specimens were rinsed with deionized water, air-dried, and surface roughness measured again by the profilometer as mentioned before.

The data collected, tabulated, and statistical analysis was carried out using IBM SPSS Corp. (2013, Armonk, NY) using software Statistical Package for the Social Sciences for Windows (SPSS version 22.0) at significance level P value ≤ 0.05. Quantitative data were described using mean, standard deviation for parametric data after testing normality using Shapiro–Wilks test. One Way ANOVA test was used to compare between subgroups with post Hoc Tukey test for pairwise comparison. Percent of change was calculated (control mean- ethyl alcohol
or ethyl ketone mean) for all the four tested restorative materials, and then comparison was done using Z -test.

RESULTS

In the present study, surface roughness values (Ra, \( \mu \text{m} \)) of different restorative materials after immersion in FSSs were determined. All tested restorative materials showed significant increase in the surface roughness after storage in FSSs (\( P<0.001 \)). Tukey test showed significant differences for \( \Delta \text{Ra} \) (surface roughness change) between pre- and after ethanol aging (p1), pre- and after MEK aging (p2), and ethanol and MEK aging (p3) as presented in Table 2.

<table>
<thead>
<tr>
<th>Equia forte</th>
<th>Activa Bioactive</th>
<th>Cention-N</th>
<th>Tetric N Ceram Bulk fill</th>
</tr>
</thead>
<tbody>
<tr>
<td>Baseline</td>
<td>0.555±0.123</td>
<td>0.166±0.022</td>
<td>1.069±0.269</td>
</tr>
<tr>
<td>FSS1</td>
<td>0.882±0.129</td>
<td>0.486±0.033</td>
<td>1.375±0.269</td>
</tr>
<tr>
<td>FSS2</td>
<td>1.445±0.15</td>
<td>0.971±0.076</td>
<td>1.961±0.27</td>
</tr>
<tr>
<td>P value</td>
<td>F=111.68</td>
<td>F=662.01</td>
<td>F=28.07</td>
</tr>
<tr>
<td></td>
<td>( P&lt;0.001^* )</td>
<td>( P&lt;0.001^* )</td>
<td>( P&lt;0.001^* )</td>
</tr>
<tr>
<td>Post hoc Tukey test</td>
<td>P1&lt;0.001*</td>
<td>P1&lt;0.001*</td>
<td>P1=0.018*</td>
</tr>
<tr>
<td></td>
<td>P2&lt;0.001*</td>
<td>P2&lt;0.001*</td>
<td>P2&lt;0.001*</td>
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<tr>
<td></td>
<td>P3&lt;0.001*</td>
<td>P3&lt;0.001*</td>
<td>P3&lt;0.001*</td>
</tr>
</tbody>
</table>

By comparing the percent of \( \Delta \text{Ra} 1 \) caused by ethanol for all groups using Z test, there were statistical significance differences between Activa Bioactive and Cention-N (p1), Activa Bioactive and Equia forte (p2), and Activa Bioactive and Tetric N Ceram Bulk fill (p3). While, there were no statistical significance differences between Cention-N and Equia forte (p4), Cention and Tetric N- Ceram Bulk fill (p5), and Equia forte and Tetric- N Ceram Bulk fill (p6). Also, the comparison of the percent of \( \Delta \text{Ra} 2 \) caused by MEK for all groups showed there was a statistical significance difference between Activa Bioactive and Cention-N only as presented in Table 3, Figure 1.

TABLE (2) Mean and SD values of surface roughness of tested restorative materials pre- and post-aging.

<table>
<thead>
<tr>
<th></th>
<th>Equia forte</th>
<th>Activa Bioactive</th>
<th>Cention-N</th>
<th>Tetric N Ceram Bulk fill</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percent of ( \Delta \text{Ra} 1 )</td>
<td>65.8%</td>
<td>22.2%</td>
<td>37.1%</td>
<td>39.7%</td>
</tr>
<tr>
<td>Percent of ( \Delta \text{Ra} 2 )</td>
<td>82.9%</td>
<td>45.5%</td>
<td>61.6%</td>
<td>61.7%</td>
</tr>
</tbody>
</table>

TABLE (3) The comparison of the percent of surface roughness changes in all groups.
DISCUSSION

Food and liquids in the oral cavity expose tooth-colored restorative materials to temperature variations and acidic-base environments roughness \(^1\). Some chemicals in foods and beverages can cause their damage, which leads to rapid wear of dental materials. The choice of ethanol and MEK is based on the basis that they are food-simulating solvents reflecting extreme dietary exposure scenarios. Second, they demonstrate increasing powers of solubilization on the resin phase of the composites, according to fundamental polymer science \(^14\). The clinical quality and efficacy of any restorative material are determined by surface \(^15\). Surface roughness and imperfections render restorations more susceptible to dental plaque deposition, gingival irritation, poor esthetics and bad prognosis \(^2\). Based on the results of this study, all the tested restorative materials were affected by FSSs and surface roughness increased. Therefore, the null hypothesis of the study was rejected.

Previous investigations reported that the effect of FSSs on the surface properties occurs during the first 7 days of immersion. For this reason, the present study was designed for a 7 days storage period \(^16\). Restorative materials are susceptible to surface degradation by daily exposure to food and drinks components \(^17\). Roughness depends on filler particle size, percentage of surface area, filler particles volume, degree of conversion, and filler-matrix bond \(^18\). According to the results of this study, all materials tested became rougher after soaking in FSSs as they are solvents that promote dissolution of the matrix, and induce erosive wear in restorative materials surface \(^19\). As reported in a previous study, \(^8\) ethyl alcohol FSS has been the solvent of choice to simulate accelerated ageing of restorations as it has a solubility parameter, and ethyl ketone FSS is an organic solvent has the potential for polymeric network damage \(^14\). Percent of ∆Ra for all tested materials was significantly higher for MEK than ethanol which could be explained by the solubility parameter. The Hildebrand solubility parameter (δ) is a numerical estimate of the degree of interaction between materials which could be a good indicator of solubility, especially for nonpolar materials like polymers. Materials with similar values of δ are likely to be miscible. The solubility parameters of E and MEK (26.2 δ/MPa\(^{1/2}\) and 19.3 δ/MPa\(^{1/2}\), resp.) are close to that of poly methyl methacrylate resin (18.6 δ/MPa\(^{1/2}\)). But higher resemblance of the solubility parameters of MEK and the dimethacryлатemonomer systems of the tested composite resins than in case of Ethanol \(^20\).

In the presence of such environment, glass filler particles tend to slip out of the material, and the matrix component decomposes \(^21\). The greater surface roughness corresponded to the larger average particle sizes. Resin composite is vulnerable to the polymer softening that can be clarified by the solvent ability to diffuse in it to bond to the polymer chains and replace the polymer inter-chain bonds. As a result, matrix softening, surface erosion, and structural ion loss occur. Also, these solutions’ acid molecules could enter the resin matrix, speeding up the release of unreacted monomers and reducing the load resistance \(^22\). Due to the hydrolytic breakdown of the bond between silane and the filler particles, a chemical degradation occurs via hydrolysis. This progressive degradation altered the microstructure of the composite bulk through the formation of pores \(^23\).
The glass-ionomer based materials were reported with surface loss in acidic solutions \(^{(24)}\). Similar to this study results, it was reported that glass ionomer-based materials are able to absorb acidic fluids, resulting in material degradation \(^{(9)}\). Another study on hybrid resinous glass-ionomer showed notable increase in surface roughness after aging by FSSs \(^{(13)}\). Furthermore, surface deterioration of resin-based Alkasite could be caused by debonding of the filler matrix or chemical degradation via hydrolysis, as it contains calcium fluoro silicate glass fillers in powder and resin in liquid. This means that the effect of FSSs on surface degradation and roughness is material dependent.

According to this study results, the comparison of ∆Ra percent showed no differences among Tetric N Ceram Bulk, Cention -N, and Equia forte, while Activa had the maximum change in surface roughness. This may be due to Cention- N is modern restorative material claimed to be an esthetic alternative to amalgam materials for filling of posterior tooth because of its much higher compressive strength \(^{(25)}\). It contains alkaline fillers that can release acid-neutralizing ions comparable to Activa \(^{(26)}\). This could explain the difference between the two materials. Moreover, depending on composition; Cention -N has some structural qualities similar to resin composite. The liquid contains urethane dimethacrylate (UDMA), which produces stiff networks with no hydroxyl side groups and are hydrophobic in nature to decrease water absorption rate \(^{(27)}\). In addition to the effect of the monomers; the fillers provide an essential part in defining the resistance to the plasticizing effect of organic solvents. An increase in the filler loading is most probably associated with higher resistance of the tested composite resins to degradation \(^{(28)}\). Cention-N had better surface properties that could make it analogous to resin composite. The current study was conducted in vitro, and it did not accurately represent the complex environment of the oral cavity, which was one of the limitations. Other materials should also be examined. Finally, other variables could be taken into account when determining the mechanical behavior of various materials. As a result, more clinical research is needed to broaden the clinical applicability of this new restorative material.

**CONCLUSIONS**

Within the limitation of this study, it can be concluded that different direct tooth-colored restorative materials surface roughness increased by food simulating solutions. This increase is material dependent. Cention-N provided a surface resistance to food aging that was comparable to that of mostly used tooth-colored direct restorative materials.

**REFERENCES**


