

The effects of food simulating liquids on surface roughness, hardness, and solubility of bulk fill composites

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Research article

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Abstract

Abstract Background: The aim of this study was to compare the effects of food simulating liquids (FSLs) on the surface roughness, surface hardness and solubility of bulk fill composites. **Methods:** Two-hundred samples were prepared from four high viscosity bulk fill composites (SonicFill TM , Tetric® EvoCeram Bulk Fill, Beautifil-Bulk Restorative, Filtek TM Bulk Fill Posterior Restorative) and a microhybrid composite (Filtek TM Z250). After the initial weights of the samples were recorded (m_1), surface roughness measurements were evaluated with a profilometer. The samples were stored in 4 different FSLs for 7 days, then the second surface roughness values were recorded. Then the samples were stored in a desiccator to reach the constant mass and the values were recorded as m_2 . The solubility levels were calculated according to the ISO 4049: 2009 specification. The surface hardness values of the samples were determined by using the Vickers microhardness measuring device. Twenty specimens from each group were evaluated in terms of surface morphology with scanning electron microscope (SEM). **Results:** Beautifil Bulk Restorative was the most affected group in terms of surface roughness after storage in FSLs and citric acid caused the highest values in this group ($p < 0,005$). Beautifil Bulk Restorative and Filtek Z250 groups showed the highest surface hardness values, while the Tetric EvoCeram group had the lowest. The highest solubility values were detected in Beautifil Bulk Restorative group, and citric acid and ethanol caused the highest solubility values on all the tested composites. **Conclusions:** Beautifil Bulk Restorative group showed the highest surface roughness and solubility values among the tested groups, while the same group showed the highest surface hardness. **Key Words:** Bulk fill composites, food simulating liquids, solubility, surface hardness, surface roughness.

Background

The advancement in the mechanical properties of resin-based materials in recent years created a wide area of use by addressing the increasing esthetical demands [1]. This progress in the properties of composite resins made their use in wide and deep cavities a routine [2,3]. In such cases, the incremental technique based on the polymerization of composite layers with 2 mm thickness is accepted as gold standard [4]. However, in order to overcome the problems due to placement and polymerization of each layer separately, such as failure of bonding between the layers, risk of gap formation, contamination risk, and prolonged treatment procedure [5], the bulk fill composites that can be polymerized up to 4 or 5mm were introduced to the market.

Various chemicals in foods and beverages consumed daily may cause destruction on the surface of composite restorations and it results in unaesthetic appearance, increased surface roughness, and abrasions [6]. According to the guideline prepared by the American Food and Drug Administration (FDA), the food simulating liquids (FSLs) were specified as citric acid, ethanol, and heptane [7]. The citric acid is present in beverages, fruits, and candy-like foods, whereas the ethanol is present in alcoholic beverages and mouthwashes and heptane simulates butter, fatty meats and vegetable oils. The reasons such as chemical destruction and mechanical abrasion due to exposure to various solutions within the mouth may damage the surfaces of resin composite restorations, and cause discoloration by making the

surface roughened, increase in the plaque accumulation, soft tissue inflammation, and recurrent caries [8,9].

One of the most important properties of restorative materials for the dentistry is the hardness of the material being used. The hardness is defined as the resistance of surface against penetration or deformation [10]. The high hardness values of resin composites enable them to successfully resist the effects that they are subject to within the mouth.

The solubility, which is another important parameter, is defined as the dissolution of resins in presence of oral liquids [11]. Having knowledge about the solubility of restorative materials is important in order to estimate their behavior in the oral environment [12]. The FSLs negatively affect their adaptation to the biological structures by increasing the chemical solubility of restorations. In conclusion, the solubility is an important parameter for the integrity of restoration, mechanical properties, surface characteristics, and esthetical appearance [13].

Although there are current studies carried out on bulk fill composites, the number of studies reporting the effects of FSLs on the bulk fill composites is limited in the literature. In this study, it is aimed to compare surface roughness, surface hardness and solubility of the bulk fill composites, which are increasingly used in the dentistry routine, with the traditional resin composite after exposed to FSLs.

The null hypothesis of the present study is that FSLs would not have any effect on the surface roughness, surface hardness, and solubility of bulk fill composites.

Methods

In the present study, 4 different high-viscosity bulk fill composites [Tetric® EvoCeram Bulk Fill (Ivoclar Vivadent), SonicFill (Kerr), Filtek Bulk Fill Posterior Restorative (3M ESPE), Beautifil-Bulk Restorative (Shofu Inc.)], a conventional nanohybrid composite resin [Filtek Z250 (3M ESPE)] and 4 different FSLs [ethanol (Teksoll 96%, Tekkim Chemical Ind. Trade. Co. Ltd., Turkey), heptane (n-Heptane, Tekkim Chemical Ind. Trade. Co. Ltd., Turkey), citric acid (citric acid 10%, Norateks, Turkey), and artificial saliva (control group)] were used.

The content, characteristics, and manufacturer information of the composite resins used in this study are presented in Table 1, whereas the FSLs are showed in Table 2.

Disc-shaped specimens (8 mm x 4 mm) were prepared using stainless-steel molds. Samples were prepared by applying bulk-fills in one step, whereas the conventional composite was applied as two 2-mm-thick layers. The specimens were polymerized for 20 seconds with a polymerization unit [Elipar S10 (3M ESPE, St. Paul, MN, USA)]. Light intensity was periodically checked by a radiometer (LED Radiometer, SDI, Australia) after the processing of every five specimens and it was verified to be higher than 1000 mW/cm². To ensure surface standardization, the coarse, medium, fine and super-fine grains of Sof-Lex aluminium oxide discs (Sof-Lex XT, 3M ESPE, St Paul, MN, USA) were respectively applied in

the same direction for 15 seconds in accordance with the manufacturer's recommendations. The specimens were inserted into a desiccator and maintained at $37\pm 1^\circ\text{C}$ for 22 h and at $23\pm 1^\circ\text{C}$ for 2 additional hours, and then weighed in a digital balance (Precisa XB 220 A, Zurich, Switzerland). This cycle was repeated until a constant mass was obtained and the values were recorded in microgram (μg) as M_1 . The diameters and thickness of specimens were measured using caliper (Mitutoyo–Absolute Digimatic NTD12-15C., Kanagawa, Japan) and the average volume of samples (V) was calculated in mm^3 .

To measure the surface roughness of the specimens, a surface profilometer (Perthometer M2, Mahr, Germany) was used, with a 0.25-mm cutoff value and 2-mm tracing length. Three measurements were performed, and the average surface roughness (Ra_1) was determined and recorded in μm for each specimen.

Then, the specimens were divided into 4 groups to be stored in FSLs. After this procedure, the specimens were placed in tubes containing different solutions and kept in a drying oven (EN025, Nüve, Turkey) at $37\pm 1^\circ\text{C}$ for 7 days. At the end of 7th day, the specimens were removed, and reconditioned in the desiccator until they reached a constant weight (M_2) using the same cycle described for M_1 . The water solubility values of composite specimens were calculated by using the formula below;

$$Ws \text{ (}\mu\text{g/mm}^3\text{)} = M_1 \text{ (}\mu\text{g)} - M_2 \text{ (}\mu\text{g)} / V \text{ (mm}^3\text{)}$$

Ws : Water solubility

After storage in FSLs, the second surface roughness measurements (Ra_2) of the specimens were performed.

The hardness value (P/d^2) of each specimen was determined using a microhardness tester (Buehler UK Ltd., England) with a diamond Vickers indenter. By applying 200g force for 10 seconds on the surface, a diagonal notch was made and then measured by using a microscope connected to the device. Vickers hardness value (VHN) was calculated by the device according to the formula below:

$$HV = 1.8544 (P/d^2)$$

P = Load, kgf

d = Mean length of the notch's two diagonals, mm

Three indentations per specimen were made and the mean values were obtained and recorded as VHN.

Five initial samples from each composite group and 20 samples from all the tested groups after storage in FSLs, were sputter coated with a 20 nm layer of Au/Pd to aid conductivity and examined using SEM (JEOL, JSM-7001FT-TLS LV, Oxford Nordlys EBSD) at an operating voltage of 10 kV at 1500 \times magnification.

Statistical Analysis

The IBM SPSS for Windows Version 22.0 software was used for the statistical analyses. The Shapiro-Wilk test was used to verify the normal distribution of the data. The two-way variance analysis (ANOVA) was used for the comparisons between the groups in terms of surface hardness and solubility values. Variance analysis was used for the repetitive measurements in intergroup and intragroup comparisons of surface roughness. The pairwise comparisons were performed using the Bonferroni test. The level of significance was set at $p < 0.05$.

Results

For five composite groups used in the present study, the mean and standard deviation of the initial and second surface roughness values (μm) are presented in Table 3 and Figure 1.

Examining the initial surface roughness values, it was determined that the highest value was in Group B (0.284 ± 0.072), whereas the lowest value was found in Group T (0.136 ± 0.037) and Group Z (0.150 ± 0.040) and there was no statistically significant difference between Group T and Z. The difference between the second surface roughness values of the composites tested after storage in FSLs was significant only for Group B (0.437 ± 0.194). The highest surface roughness value was obtained in Group B stored in citric acid (0.698 ± 0.120), and this value was statistically significant ($p < 0.01$). In Group B, there was no significant difference between the surface roughness values of ethanol, saliva, and heptane groups ($p > 0.05$).

The mean \pm standard deviation values of the surface hardness values of tested composites after exposure to each solution are presented in Table 4 and Figure 2.

Given the effects of FSLs on the surface hardness, it was determined that, among the specimens stored in ethanol, the highest surface hardness value was found in Group Z (108.87 ± 8.57) and the lowest value in Group T (67.99 ± 7.48). Among the specimens stored in saliva, the highest surface hardness without any statistically significant difference was determined in Group Z (111.57 ± 7.64) and Group B (105.93 ± 15.05), respectively, whereas the lowest value was found in Group T (64.78 ± 5.87). Among the specimens exposed to citric acid, the highest and lowest surface hardness values were found to be in Group B (164.84 ± 33.09) and Group T (61.66 ± 4.29), respectively. In heptane group, Group Z (101.44 ± 7.68) and Group B (90.37 ± 5.78) were found to have the highest surface hardness, whereas the lowest value was found in Group T (63.07 ± 2.82).

Evaluating each composite separately on the basis of 4 different solutions, a statistically significant result was obtained only in Group B ($p < 0.001$). In Group B, the highest surface hardness value was obtained in citric acid and the lowest value in ethanol and heptane groups.

The mean \pm standard deviation of the solubility values tested composites after the exposure to FSLs are shown in Table 5 and Figure 3.

Given the solubility values of the composites, it was determined that the highest solubility was obtained in Group B ($0.92 \pm 7.68 \mu\text{g}/\text{mm}^3$) and this value was statistically significantly higher than the other groups ($p < 0.05$), and the highest solubility value was found in citric acid.

SEM Images

SEM images (Figures 4-9) obtained after storing the tested composites in FSLs are presented below.

In Beautifil Bulk Restorative group, the statistically significant increase found in the surface roughness values in all solutions were supported by the surface images obtained from SEM. In these images, the gaps occurring because of the removal of inorganic fillers from the surface are pointed (Figure 4d).

Discussion

The null hypothesis that “the FSLs would not have any effect on the surface roughness, surface hardness, and solubility of bulk fill composites” was partially rejected since the solutions caused significant changes only in S-PRG fillers of Beautifil Bulk Restorative composite. The degradation of the fillers in this group caused significant increase on all tested parameters.

In the list of chemicals that were identified by the FDA and can be used as FSLs, there are solutions such as heptane, ethanol, citric acid, and distilled water [7]. Each solution causes the destruction of mainly one component of resin-based materials. As reported by Yap et al. [14], Bis-GMA matrix is sensitive to the softening effect of FSLs. Besides that, the inorganic fillers may be destroyed by weak acids such as citric acid and the water [15]. Thus, it can be said that the environment, to which the restorative materials are exposed, has an important effect on the destruction of materials. Kooi et al. [16] reported that various resin composite restorations were chemically destructed in FSLs (air, distilled water, citric acid, and ethanol) and there was a negative correlation between increased surface roughness values and decreased hardness values. Yap et al. [17] reported that the abrasion levels of composite materials may be affected by the chemical solvents, they might soften when contact with chemical materials, and fillers may come out.

In the previous studies, it can be said that various qualitative and quantitative methods were used in determining the surface roughness. Some of them are atomic force microscopy (AFM), two dimensional (mechanical) and three dimensional (optic) profilometer, and scanning electron microscope [18]. The mechanical profilometer is the most widely used measurement method in assessing the surface properties of resin composites, and it was also used in the present study. Also, the mechanical profilometers were found to be more useful in assessing applied treatments on the surfaces of materials [19]. Moreover, SEM was used in the present study and it was aimed to examine the surface topography of specimens in a more detailed manner.

Previous studies [16,20] stated that giomer based composites exhibited higher roughness values compared to traditional resin based materials because of large particle size of S-PRG fillers. Similarly, in the present study after ensuring surface standardization of the tested composites with Sof-Lex discs, initial Ra measurement exhibited that Beautiful Bulk Restorative group had the highest surface roughness values among the tested composites.

Tanathanuch et al. [21] reported an increase in surface roughness and a decrease in microhardness values, for the bulk fill composites stored in food simulating solutions and beverages for 28 days.

The previous study researched the effect of FSLs (distilled water, 50% ethanol, 0.02 N citric acid, air-control) on the surface roughness and hardness values of giomer (Beautiful II, Beautiful Flow Plus F00), direct (Filtek Z250) and indirect (Ceramage) hybrid composites, it was reported that gomers were destroyed by these solutions, surface hardness was significantly affected by citric acid and ethanol, and also citric acid caused a significant increase in surface roughness [16].

In the present study after storage in FSLs, the specimens in Beautiful Bulk Restorative group were affected by the citric acid at most (Figure 5). The mean initial Ra value of specimens in this group was found to be $0.284 \pm 0.072 \mu\text{m}$, whereas the mean Ra value increased to $0.698 \pm 0.120 \mu\text{m}$ after storing 7 days in citric acid. Similar to the present study, Kooi et al. [16] stated that specimens in giomer-based Beautiful II group were significantly destructed by citric acid, and they reported that this might be because fluorosilicate glass fillers are more sensitive to the decomposition of weak acids [22].

Given the SEM images of the samples tested in the present study, it was observed that other composites were affected by the FSLs less than Beautiful Bulk Restorative group. Because they have smaller particle structure, the gaps occurring after removal of inorganic fillers did not cause a significant difference in the surface roughness. The size of gaps occurring because of the removal of S-PRG fillers as a result of the exposure of Beautiful Bulk Restorative group to solutions can be clearly distinguished by SEM images, and this is thought to be an indicator of a significant increase in the surface roughness.

It was determined that surface roughness of composite restorations is affected by the duration of contact with aqueous environment, in which they are [14,23]. In a previous study, it was reported that the properties of restorative material, the type of storage solution, and assessment period play effective role in the microhardness of composites, whereas the prolonged duration of exposure to the solutions may have more effect on the resin properties [24].

In a previous study, the surface hardness values of restorative materials were compared, after aging in distilled water for 30 days and applying 5000 thermal cycles, and it was reported that giomer (Beautiful) showed higher surface hardness (78.97 kgf/mm^2) than conventional composites (Esthet-x: 64.42 kgf/mm^2 , Admira: 49.11 kgf/mm^2) and compomer (Dyract: 56.01 kgf/mm^2) [25].

In their study on comparing the microhardness of various bulk fill composites (SDR, SonicFill, Tetric N-Ceram Bulk Fill, Filtek Bulk Fill) exposed to food simulating solutions and beverages, Tanathanuch et al.

[21] reported that the most significant decrease in microhardness values was observed with passionfruit juice having a high concentration of citric acid, whereas the bulk fill composite affected at most was found to be SDR.

In the present study, it is believed that white opaque layer, which can be macroscopically observed on the surfaces of specimens in giomer-based Beautifil Bulk Restorative group exposed to citric acid, might be due to the ions precipitating to the surface. Beautifil Bulk Restorative group is thought to exhibit higher surface hardness values due to this layer.

By storing the restorative materials (Beautifil II, Gradia Direct X, Tetric EvoCeram, and Fuji IX Extra) in deionized water (pH 6.5) and lactic acid (pH 4.0) in a previous study, it was comparatively examined how the fluor release, recharge properties, and mechanical properties (surface hardness and elasticity module) were affected [26]. Similar to the present study, it was reported that the surface hardness value of Beautifil II group stored in deionized water and lactic acid (0.7-0.8 GPa) was approximately twice as much, obtained in Tetric EvoCeram group (0.4-0.5 GPa).

In a study evaluating the specimens prepared by using giomer (Beautifil II), compomer (Dyract Extra), glass ionomer (Ketac), and resin composite (Tetric EvoCeram) in terms of surface hardness, Condò et al. [27] reported that the surface hardness value of Beautifil II group was approximately double of surface hardness values of Tetric EvoCeram and Dyract Extra groups.

In a previous study which comparing giomer- (Beautifil Bulk Restorative, Beautifil Bulk Flow) and resin-based composites' (Tetric EvoCeram Bulk Fill, SDR) characteristics such as surface hardness and polymerization depth, it was reported that micromechanical properties of giomer materials were higher than resin composites, as stated in the present study [28].

ISO 4049, which is used to determine water sorption and solubility values, is one of the reliable standards [29]. According to ISO 4049 (2009a), water sorption and solubility values must be lower than $40 \mu\text{g}/\text{mm}^3$ and $7.5 \mu\text{g}/\text{mm}^3$, respectively [30]. In this study, the solubility values obtained by using the formula given in the standard were found lower than the values specified in the ISO criteria in all groups except Beautifil Bulk Restorative group stored in citric acid. The solubility value of Beautifil Bulk Restorative group stored in citric acid was found to be $13.03 \pm 4.56 \mu\text{g}/\text{mm}^3$ at the end of 7th day. In the present study, it was determined that the solubility values of all composite groups, except Beautifil Bulk Restorative group, were negative. Considering that the materials might have not completed dehydration process, negative solubility values can be interpreted in the way that solubility of the materials occurred at low levels, rather than not occurring at all. In their study, Lopes et al. [31] reported that negative solubility values of tested composites might be because the resin composite is more sensitive to water sorption, which causes an increase in mass and which may mask the real solubility. In a previous study, it was stated that negative solubility values might be because water absorbed partially become bound water within the resin matrix and they cannot be removed irreversibly [32].

According to the solubility values in the present study, it can be concluded that as the fluorosilicate glass fillers are very sensitive to weak acids [16], Beautifil Bulk Restorative dissolved in citric acid at the highest level.

Conclusions

Beautifil Bulk Restorative group showed the highest surface roughness and solubility values after storage in FSLs due to the degradation of S-PRG fillers. Moreover, the result that the highest surface hardness values in this group might be related with the residual layer due to the ion release from these particles, and further studies are needed on this subject. There was no significant difference between the other composites and control group in terms of the tested parameters.

Within the limitations of this in vitro study, composites were exposed to only FSLs, but in the oral cavity the continuous buffering effect of saliva would affect the materials so the findings should be supported with in vivo studies.

Abbreviations

AFM: atomic force microscopy, ANOVA: analysis of variance, FDA: Food and Drug Administration, FSL: food simulating liquids, SEM: scanning electron microscopy, VHN: Vickers hardness value

Declarations

Ethics approval and consent to participate

Not applicable

Consent for publication

Not applicable

Availability of data and material

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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instrumental testing.

Authors' contributions

ÖGC: acquisition of data, analysis and interpretation of data, drafting the article, final approval of the version to be published. NG: conception and design of the study, revising it critically for important intellectual content, final approval of the version to be published. All authors read and approved the final manuscript.

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Tables

Due to technical limitations the Tables are available as a download in the Supplementary Files.

Figures

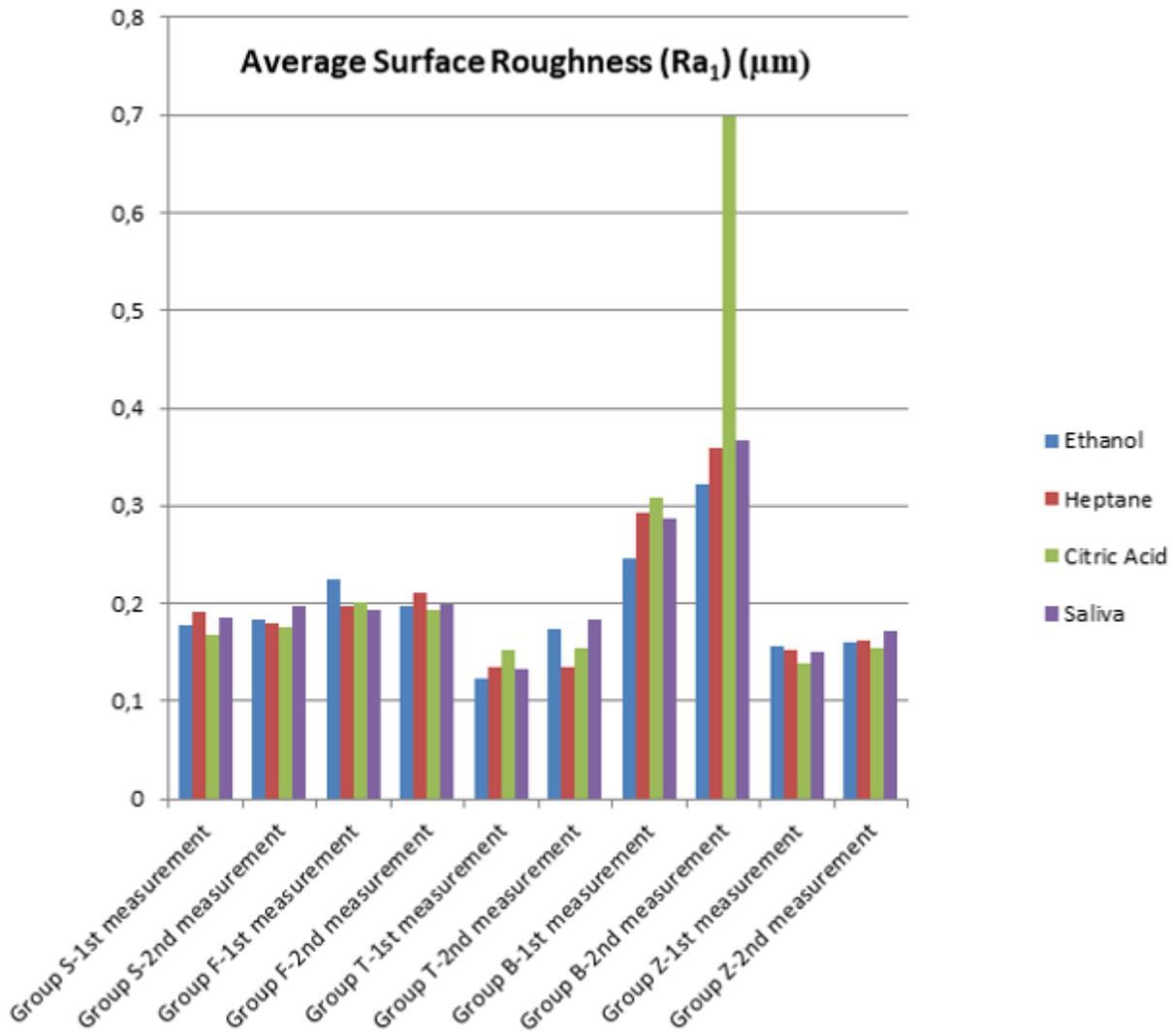


Figure 1

The graphical appearance of the average surface roughness values of the composites ($Ra-\mu\text{m}$).

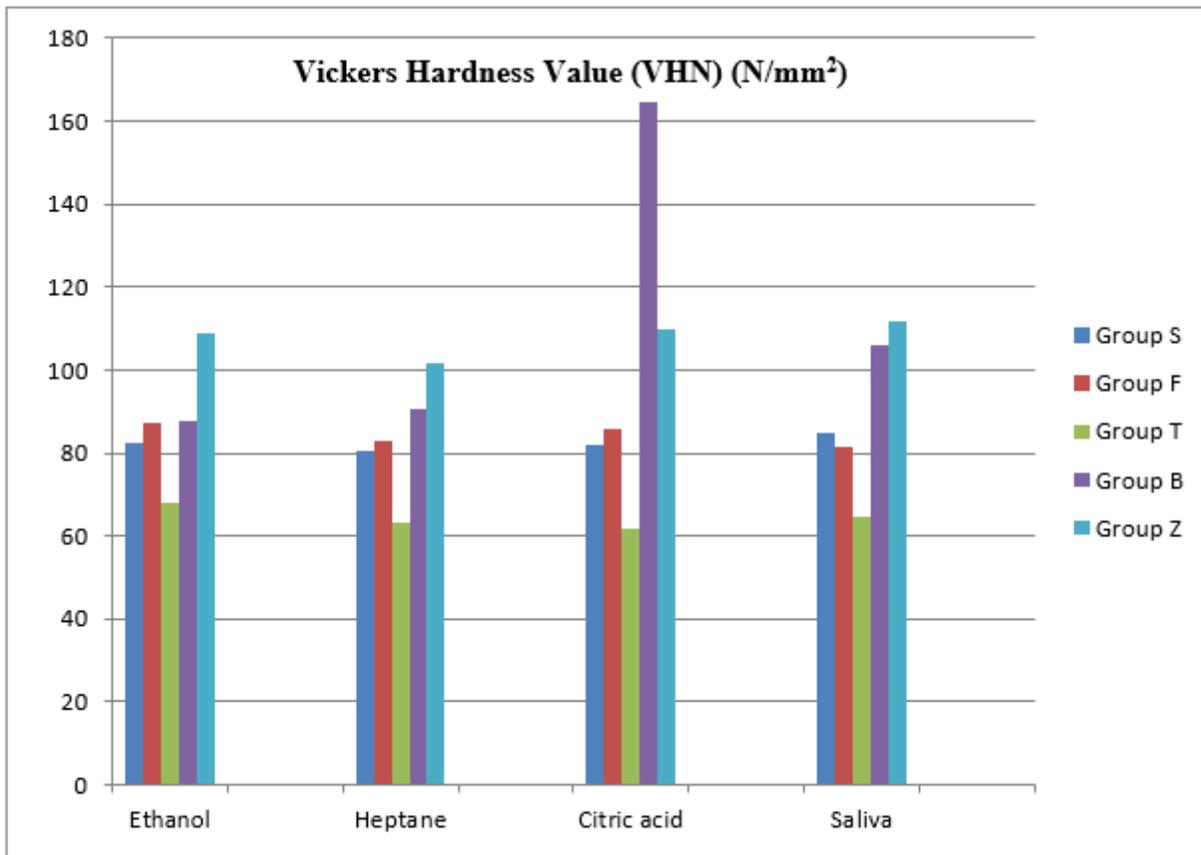


Figure 2

The graphical appearance of the Vickers hardness values (VHN) of the composites.

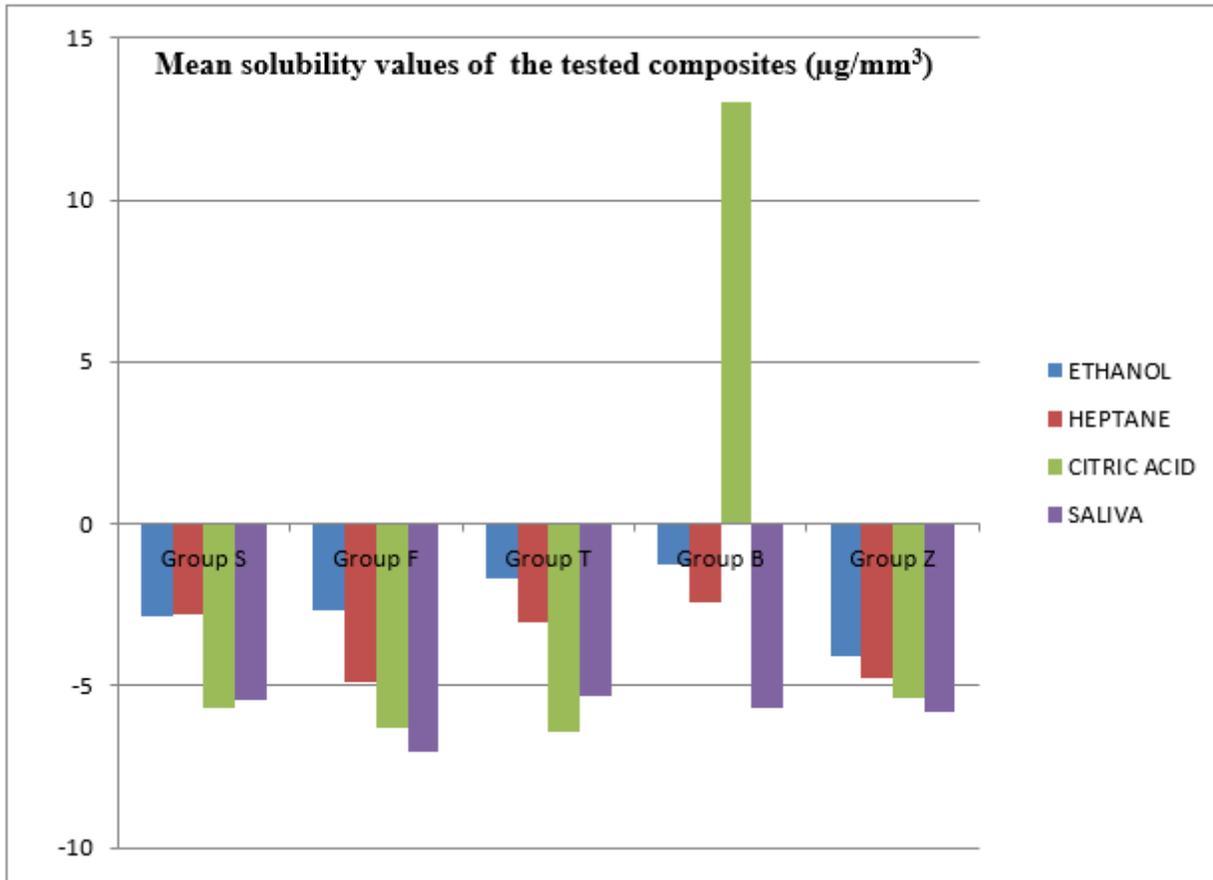


Figure 3

The graphical appearance of the mean solubility values of the composites ($\mu\text{g}/\text{mm}^3$).

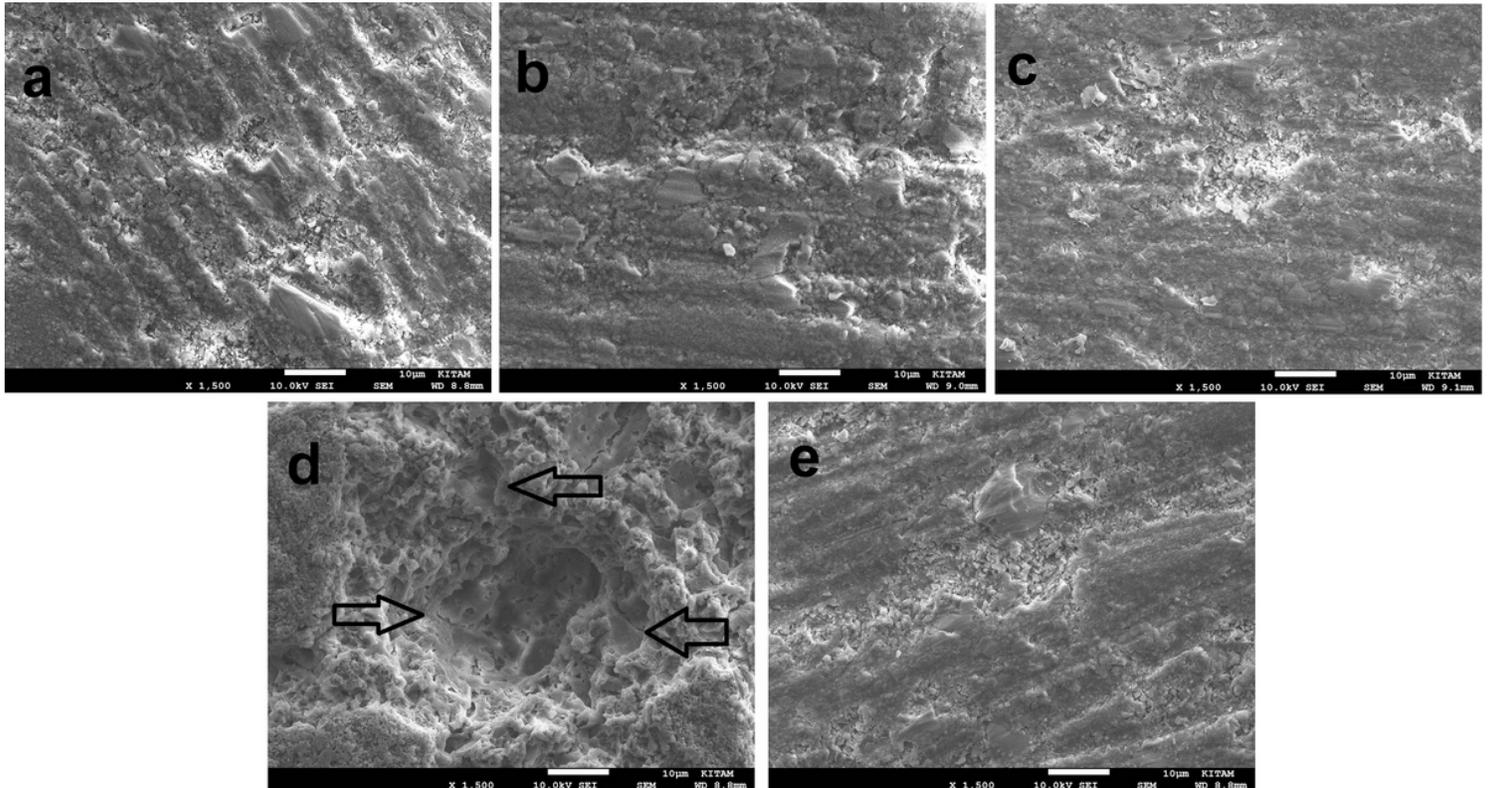


Figure 4

Beautiful Bulk Restorative

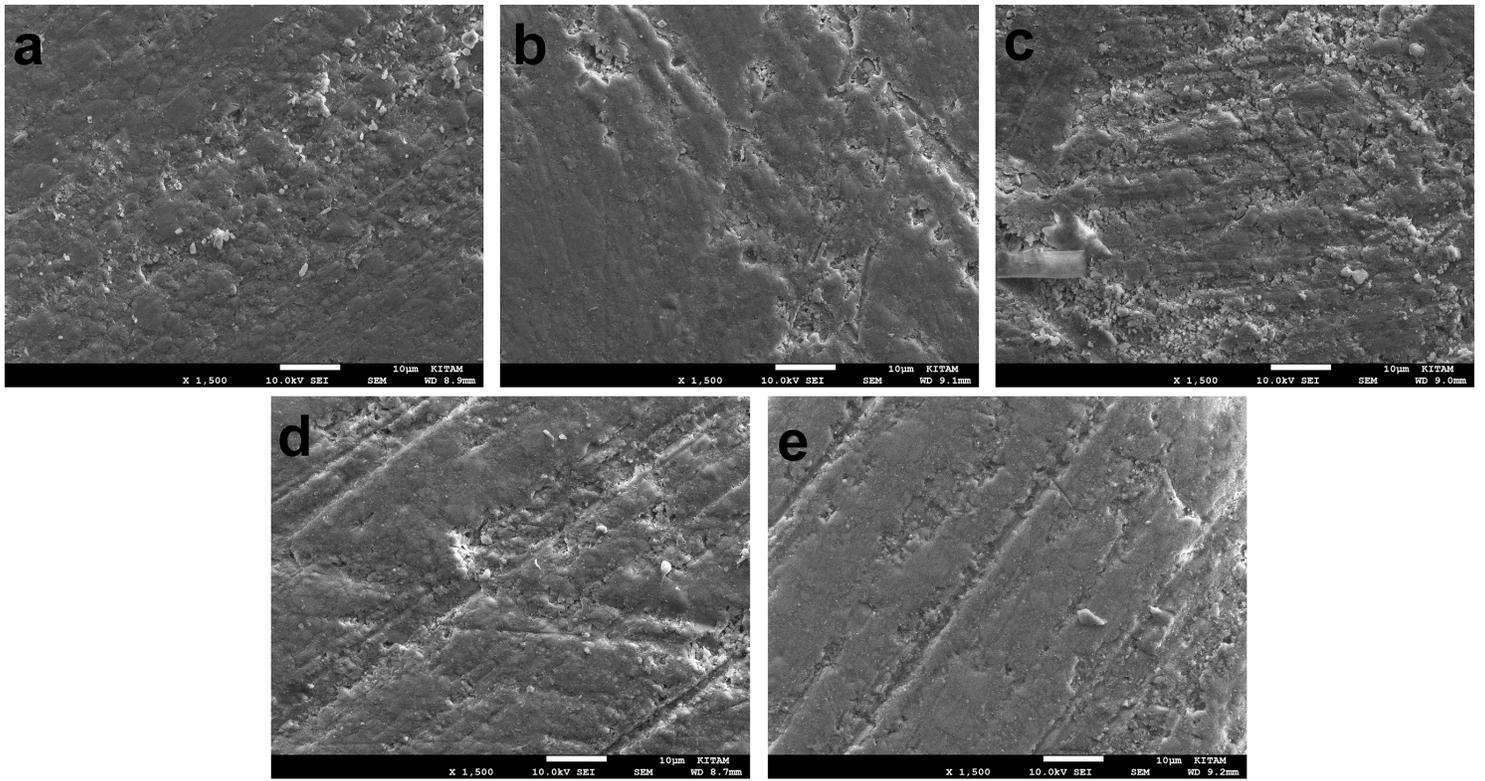


Figure 5

Bulk Fill Posterior Restorative

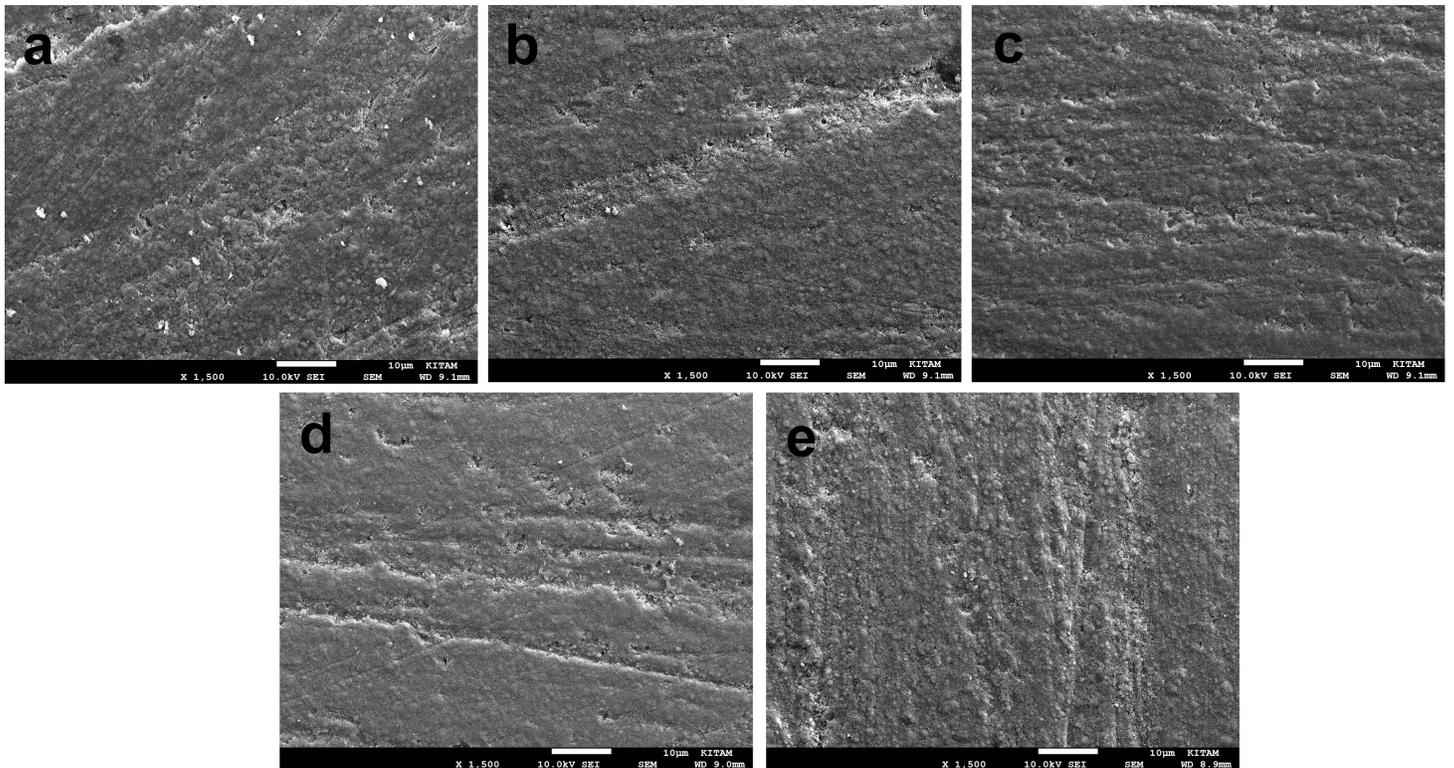


Figure 6

FiltekTV Z250

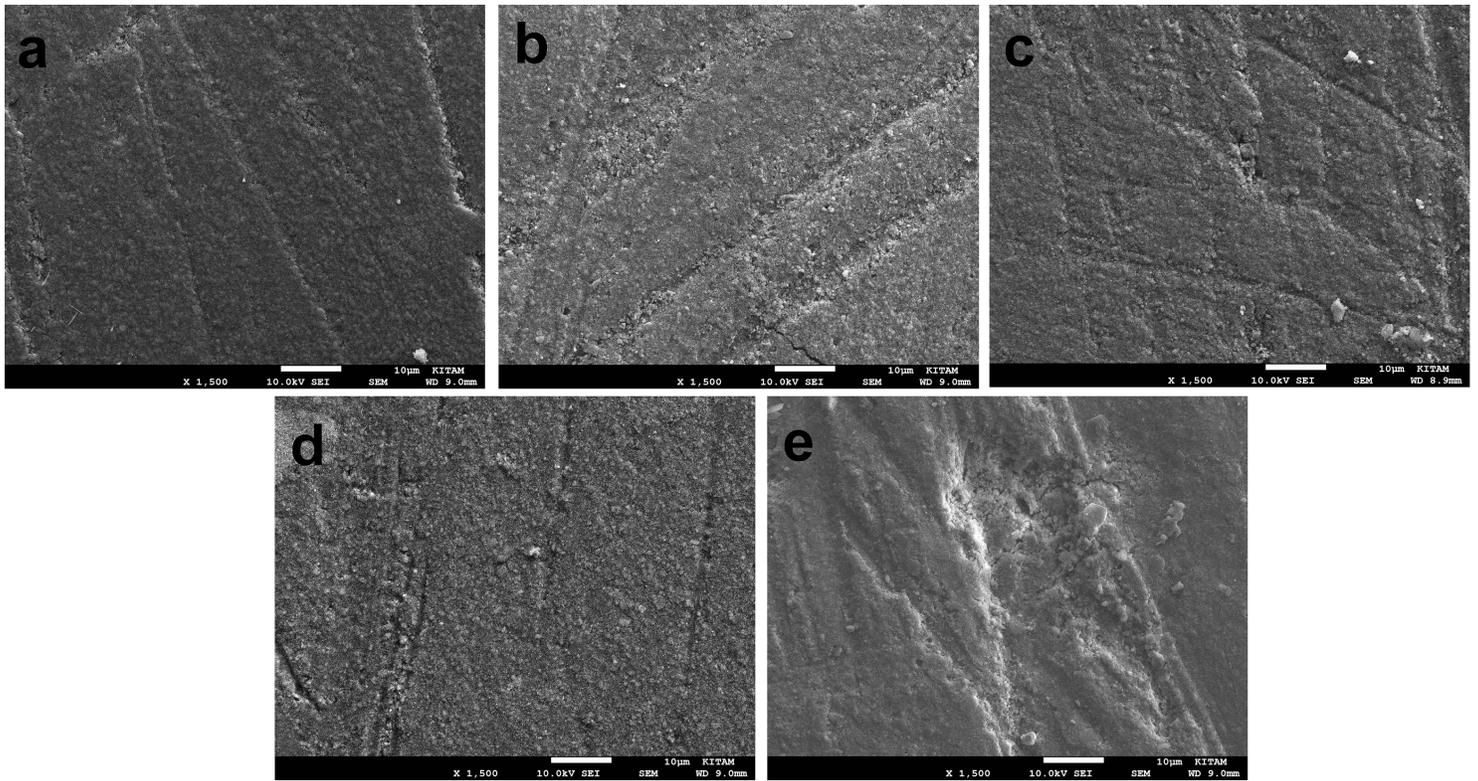


Figure 7

Tetric EvoCeram Bulk Fill

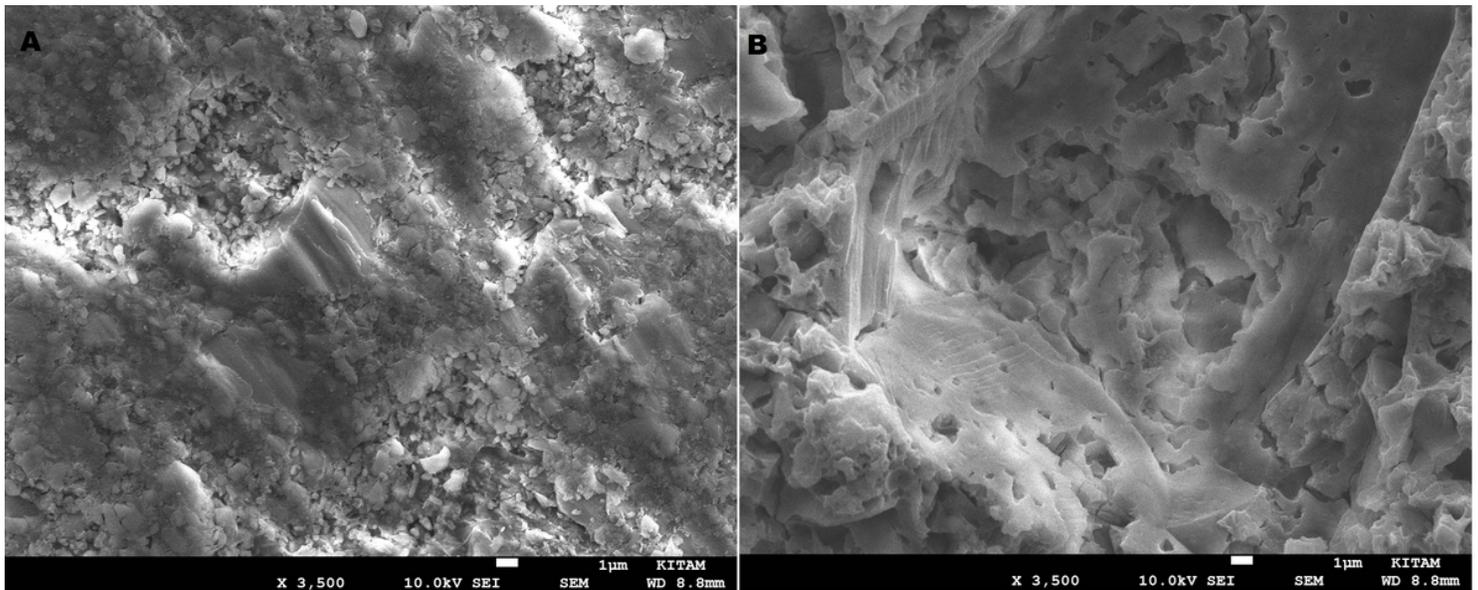


Figure 8

Beautiful Bulk Restorative

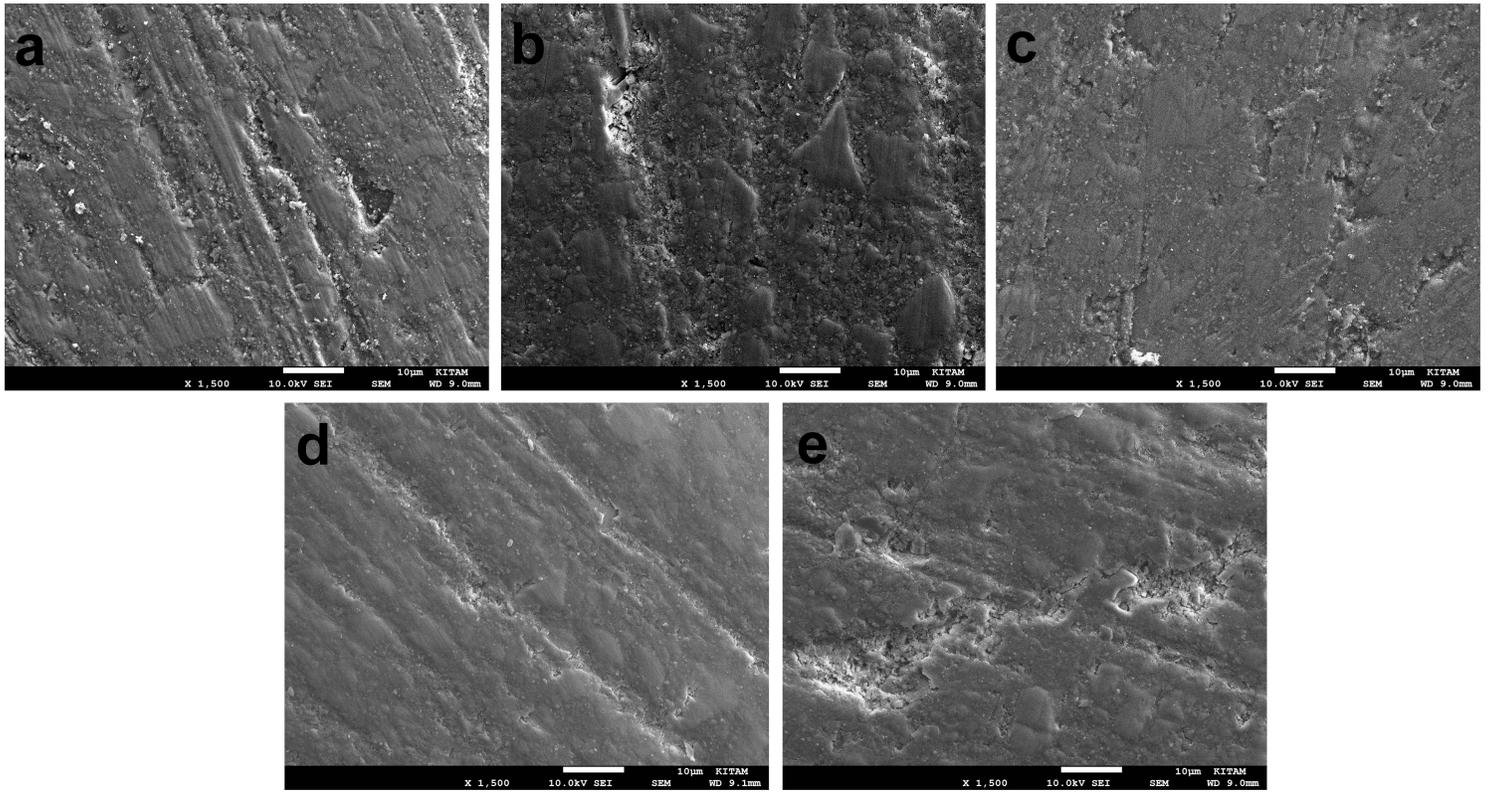


Figure 9

Sonic Fill

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [Tables.pdf](#)