

Comparative Evaluation of Surface Roughness and Wettability of an Alkaside with Nano Bulk-Fill and Nanofilled Resin Composite Restorative Materials: *In vitro* Study

Abstract

Context: Surface characteristics of resin-based composites (RBCs) can change with polishing and over time. **Aim:** The aim of the study was to compare the surface roughness and wettability of three different posterior RBCs after polishing and the change in these surface characteristics over time, after aqueous aging. **Settings and Design:** Experimental *in vitro* study. **Materials and Methods:** Eleven disc-shaped RBC specimens were fabricated. The RBCs used were, alkaside composite, bulk-fill nanocomposite, conventional nanofilled composite. All the specimens underwent polishing with Soflex Diamond Polishing System and then analyzed for roughness and wettability at baseline and after aqueous aging for 3 months with the help of an atomic force microscope and a contact angle goniometer respectively. **Statistical Analysis:** One-way ANOVA and Tamhane test were used for the multiple comparisons. **Results:** Alkaside composite showed significantly higher surface roughness ($P = 0.028$ and $P < 0.001$, respectively) and lower wettability ($P = 0.023$ and $P = 0.020$, respectively) than conventional nanofilled composite at baseline and 3 months. Surface roughness of alkaside composite was also significantly higher than bulk-fill nanocomposite ($P = 0.009$ and $P < 0.001$, respectively) at both the time points. **Conclusions:** Alkaside has higher surface roughness in comparison to conventional nanocomposite and bulk-fill nanocomposite and lower wettability than conventional nanocomposite after polishing and aqueous aging over a 3-month period. In terms of surface characteristics, alkaside composite may be advantageous in preventing initial plaque adhesion to the material surface, but the lower surface roughness of nano-filled composites may be more advantageous in terms of plaque retention prevention.

Keywords: Aging, atomic force microscopy, composite resin, polishing, wettability

Introduction

Resin based composites (RBCs) have an established application in restoration of primary and permanent teeth. Their ability to mimic tooth structure has given them a clear edge for use and acceptance by both patients and dental professionals.^[1] The type of filler affects their physical properties such as the strength, polymerization shrinkage, surface characteristics, and polishability.^[2] Formulation of filler particles has evolved from macro, micro, down to nanoparticles.^[3] Nanocomposites with filler particle size <100 nm, have low shrinkage, better polishability, and greater wear-resistance.^[4]

Bulk-fill resin composites that are currently extensively used allow the number of increments required to fill a cavity to

be reduced. Unlike the standard 2-mm increments recommended for traditional RBCs, increments of 4 mm–5 mm are used for the bulk-fill RBCs. This simplifies the restorative procedure, reduces clinical time, and improves patient compliance in cases of deep, wide cavities.^[5] However, the advantage of the faster restorative procedure with light-cured bulk-fill RBCs is offset by the uncertainty of sufficient in-depth curing. This has led to the development of dual-curing RBCs that are also suitable for bulk-filling procedure.^[6]

Recently, a dual-cured resin-based bulk-fill composite with alkaline fillers, referred to as alkasides was introduced as a tooth-colored, basic filling material for bulk placement in retentive preparations. They have bioactive properties and release acid-neutralizing ions to prevent tooth demineralization.^[6,7]

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The success of RBC restorations is limited by their vulnerability to secondary caries. Susceptibility of RBCs to plaque biofilm colonization, causative to secondary caries is dependent on the surface characteristics of the RBC such as surface roughness and wettability.^[8] Surface roughness has a significant effect on discoloration of restorations and gingival irritation.^[2,9] Finishing and polishing techniques are employed to decrease the surface roughness of RBC restorations. The type of inorganic filler, the size of the particles, and the magnitude of the filler loading influence the polishability of RBCs.^[10]

Surface wettability of the material is determined by contact angle measurements (angle formed by the solid sample surface and the tangent at the surface of the liquid drop) and gives an insight to the hydrophobicity of the material. As the angle increases, the wettability decreases and is an indicator of free surface energy, which plays an important role in bacterial adhesion.^[11,12] Earlier studies have shown that the wettability of the RBC varies with surface roughness and the composition of the RBCs.^[8,13]

In addition, surface characteristics of RBCs can change over time. Aqueous aging studies have shown a change in surface characteristics after immersion in water.^[3,14] No extensive studies have been done comparing the surface characteristics of commonly used RBCs such as nanocomposites and bulk-fill composites with the relatively newer alkasite material.

Hence, the purpose of this *in vitro* study was to estimate and compare the surface roughness and wettability of nanofilled, alkasite and bulk-fill RBCs used for posterior teeth restorations, after polishing. In addition, the changes in surface roughness and wettability over time, after aqueous aging were to be measured.

The null hypothesis was that there is no significant difference in the surface roughness and wettability between the resin composites after polishing and aqueous aging.

Materials and Methods

Sample preparation

The sample size was calculated by using a resource equation where E should be between 10 and 20 ($E = (n \times G) - G/G$), where n = total number of specimens; G = total number of groups.^[15] Assuming $n = 11$ and $G = 3$ and a 10% loss of sample, final sample size will be 11. Hence, the sample size was approximated to 11, since it is the closest number between 10 and 20.

The study was approved by the Institutional Ethics Committee, prior to the commencement of the study. Eleven -disc-shaped specimens of each RBC were fabricated:

Group 1

Alkasite composite (Cention N, Ivoclar Vivadent Inc, NY, USA).

Group 2

Bulk-fill nanofilled composite (3M Filtek™ One Bulk-fill Restorative, 3M ESPE, St Paul, MN, USA).

Group 3

Conventional nanofilled composite (Filtek Z350 XT, 3M ESPE, St Paul, MN, USA).

Details regarding the composition of the materials studied are given in Table 1. Each type of resin composite material was condensed in a single increment into a customized metal mold of 8 mm × 2 mm dimension, using a smooth surface round-ended condenser. Excess material was removed with a plastic filling instrument. To obtain a flat surface without any defects and entrapped air, specimens was covered with a Mylar strip (SS White Co, Philadelphia, Pennsylvania, USA) and a uniform pressure was applied with a microscope glass slide.

All specimens were polymerized through the glass slide using a light-emitting diode curing light (BLUEDENT, BG LIGHT LTD, Plovdiv, Bulgaria) at an intensity of 1200 mW/cm², as recorded by radiometer (Demetron 100, Demetron Research Corp., Danbury, CT, USA) for 20 s in the continuous mode. After retrieving the sample from the mold, the surfaces were examined for voids and checked for the dimensions with the help of a calliper. The samples with voids were discarded. All the specimens underwent finishing using a tungsten carbide 12-fluted bur (SS White, Lakewood, New Jersey, USA) in a single-direction to remove flash. Polishing was done with Soflex Diamond Polishing System (3M ESPE, St Paul, MN, USA). The specimens were first treated with prepolishing spiral followed by the diamond polishing spiral for 30 s, each with moderate pressure in a single-direction, using a micromotor handpiece (NSK Inc, Tokyo, Japan) at a speed of 10,000–20,000 rpm. The specimens were then rinsed with water for 10 s and air-dried for 5 s. The polishing spirals were replaced after each use and tungsten carbide bur were replaced after every three specimens. The specimens were then stored in distilled water for 24 h at 37°C in a plastic container with a sealed lid in an incubator (SANYO Incubator MIR-253, SANYO Electric Co., Ltd., Sakata, Oizumi-Machi, Ora-Gun, Gunma, Japan). The same operator performed all the procedures, to standardize the finishing and polishing procedures.

Surface roughness measurement

After specimen preparation, a baseline measurement of the surface roughness was done after 24-h by observing under an atomic force microscope (AFM) (Innova™, Bruker Corporation, Coventry, UK) with a probe with a length of 125 μm, width of 40 μm and tip thickness of 3.4 μm, under tapping mode. Specimens were air-dried prior to observation under AFM and were placed on a sample-mounting disk made of stainless steel (15 mm diameter). Specimen scanning was done

Table 1: Details of the tested materials: Alkasite composite, bulk-fill nanocomposite, conventional nanocomposite

| Material group | Type | Matrix | Filler type and filler size | Filler loading (volume percentage/weight percentage) | Manufacturer, shade and lot number |
|----------------|--------------------------------|---|---|--|--|
| Group 1 | Alkasite composite | UDMA, DCP, an aromatic aliphatic-UDMA and PEG-400 DMA | Barium aluminum silicate glass filler, ytterbium trifluoride, isofiller (tetric N-ceram technology), calcium barium aluminum fluorosilicate glass filler, calcium fluorosilicate (alkaline) glass filler 0.1-35 µm | 57.6/78.4 | Ivoclar Vivadent Inc, NY, USA A2 Lot number Y25391 |
| Group 2 | Bulk-fill nanofilled composite | AUDMA, AFM | Nonagglomerated silica filler ~20 nm Nonagglomerated zirconia filler ~4-11 nm Agglomerated ytterbium trifluoride ~100 nm | 58.4/76.5 | 3M Filtek™ one bulk-fill restorative, St Paul, MN, USA A2 Lot number N719050 |
| Group 3 | Nanofilled composite | Bis-GMA, UDMA, TEGDMA, and bis-EMA resins | Nonagglomerated silica filler (20 nm) Nonagglomerated/zirconia filler (4-11 nm) Aggregated zirconia/silica Cluster filler (comprised of 20 nm silica and 4-11 nm zirconia particles) | 63.3/78.5 | 3M™ ESPE, Filtek™ Z350 XT, St Paul, MN, USA A2 Lot number NA21364 |

DCP: Dimethanol dimethacrylate; PEG-400: Polyethylene glycol 400; UDMA: Urethane dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; BisGMA: Diglycidildimethacrylate; AUDMA: Aromatic urethane dimethacrylate; AFM: Addition-fragmentation monomer; BisEMA: Ethoxylated version

at three points (one point each at the center of the specimen, at specimen's perimeter and at half distance between specimen's center and perimeter), and the mean average value was obtained.^[2] Three images (751 × 751 pixels) were collected for each specimen with a scan size of 10 µm × 10 µm and a scan rate of 1.00 Hz. Measured topography data were analyzed using Nanoscope Analysis Version 1.5 software. (Bruker Corporation, Coventry, West Midlands, UK)

Surface roughness was expressed as average Ra value (arithmetical average value of all absolute distances of the roughness profile) in nanometers. These measurements were repeated after 90 days with the same specimens. During this time interval, all the specimens were stored in distilled water, away from the direct sunlight, in a plastic container with a sealed lid and placed in an incubator at a temperature of 37°C, for aqueous aging. The distilled water in the plastic container was changed every day. All AFM scans were performed by the same, trained operator, who was blinded to the type of the composite resin analyzed.

Contact angle measurement

To assess wettability, contact angle measurements were obtained using a contact angle goniometer (Holmarc opto-mechatronics Pvt. Ltd, Kochi, Kerala, India) with an attached camera to capture the image at a resolution of 2592 × 1944 pixels. The measurement was done by sessile-drop-method in which deionised water was dropped perpendicular to the substrate surface at the room temperature to the center of the sample, placed in a perfectly horizontal position. The volume for each drop of water was standardized to 2 µL using 50 µL syringe dispenser. The time interval between each recording was

standardized to 5-s and two readings were taken (left and right) each time at four regions (upper-right, upper-left, lower-right, lower-left) of the specimen. The image obtained at each time point was analyzed with the inbuilt image analyser software of the goniometer, to obtain the contact angle (the angle formed between the liquid – Solid interfaces). Ten readings at 5-s interval were taken in each of the four regions per sample and the average mean angle was calculated.^[13] These measurements were repeated after 90 days with the same specimens that were stored in the incubator at 37°C in distilled water for aqueous aging and thus a total of 1760 measurements per group were taken in the span of 3 months. The procedure for measuring the contact angle was the same for all groups and was performed by the same, trained operator who was blinded to the type of composite used.

Statistical analysis

Data were analyzed using IBM SPSS Statistics for Windows, Version 20 (IBM Corp., Armonk, NY, USA). Mean surface roughness (Ra) and contact angle values for each group were calculated. Intergroup and intragroup comparison at two-time interval was done using the one-way ANOVA and paired *t*-test, respectively. Multiple comparisons were done using Tamhane test. To analyze the effect of time and type of composite on the Ra and contact angle values, a multivariate regression analysis was performed using the general linear model. *P* < 0.05 was considered statistically significant.

Results

Quantitative analysis of the Ra values using one-way ANOVA showed a significant difference between the

groups [Table 2] with the highest value for Group 1 and lowest for Group 2. For further intergroup comparison, *post hoc* test (Tamhane) was applied and it was found that Group 1 values were significantly higher than Group 2 at both baseline and 3 months ($P = 0.009$ and $P < 0.001$, respectively). Group 1 also showed a significantly higher value than Group 3 at both baseline and 3 months ($P = 0.028$ and $P < 0.001$, respectively). There was no significant difference between Group 2 and Group 3 at both time points ($P = 0.867$ and $P = 0.705$, respectively). Thus, the alkasite composite had higher surface roughness than both conventional nanofilled and bulk-fill nanocomposite.

The surfaces showed slight increase in Ra over a 3-month period of aqueous aging for both Groups 1 and 2 but a slight decrease for Group 3 [Table 2]. However, the differences were not significant ($P = 0.431$, 0.949 , and 0.938 , respectively, for Groups 1, 2, and 3), as shown by paired *t*-test.

Qualitative analysis of the AFM images showed that the polishing procedure generated lines and scratches on the material surfaces. The polishing system formed a surface with furrowed and micro ploughed topography, with valleys and peaks formed due to dislodgment of resin matrix as well as filler particles, exposing the remaining filler particles. While Group 1 (alkasite) showed multiple depression areas indicating loss of matrix as well as filler particles, Group 2 (bulk-fill nanocomposite) showed a more uniform surface with deep scratch lines and occasional blunted peaks and ridges, whereas Group 3 showed a regular pattern of peaks and valley distribution. Comparing the images at baseline and 3 months, Group 1 showed increase in irregularities with shortening and blunting of the peaks while Group 2 showed an increase in irregularity with more prominent peaks and appearance of new areas of depression, signifying loss of fillers and matrix. For Group 3, compared to the 1st month, peaks were wider and more rounded, with deeper valleys and grooves seen, compatible with marginal increase in irregularity [Figures 1 and 2].

The comparison of the mean contact angles of the three groups also revealed a significant difference between the groups at baseline and 3 months [Table 3] with highest value in Group 1 and lowest value in Group 3. Intergroup comparisons with Tamhane test revealed a significant difference in the contact angle values between Group 1 and Group 3 ($P = 0.023$ and 0.020 at baseline and 3 months, respectively). There was no significant difference between Groups 1 and 2 ($P = 0.309$ at both baseline and 3 months) and Groups 2 and 3 ($P = 0.451$ and 0.301 at baseline and 3 months, respectively). Thus, the alkasite showed significantly lower surface wettability than nanofilled composite, while it was comparable with bulk-fill nanocomposite.

Contact angle values [Table 3] showed a slight increase over the period of aqueous aging for both Groups 1 and 2, but a negligible decrease for Group 3, the differences not shown to be significant by the paired *t*-test analysis ($P = 0.126$, 0.211 , and 0.519 for Groups 1, 2, and 3, respectively).

Since the values obtained varied by two categorical variables (type of composite and time), the mean values of surface roughness and wettability were entered into general linear models with *post hoc* tests. There was a significant difference in Ra values between the composites irrespective of aqueous aging for 3 months ($F = 16.94$; $P < 0.001$) with *post hoc* test revealing a significant difference between Groups 1 and 2 ($P < 0.001$) as well as Group 3 ($P < 0.001$). Significant differences were also seen in the surface wettability using general linear model ($F = 5.19$; $P = 0.012$) with a significant difference between Group 1 and 3 ($P = 0.021$), regardless of aqueous aging.

Discussion

A mean surface roughness of $0.3 \mu\text{m}$ can be easily detected by the tongue tip, causing discomfort to patients. When the value is above $0.2 \mu\text{m}$, there is high risk of plaque accumulation, increasing the risk of secondary caries.^[12,16,17] The range of mean surface roughness values obtained in this study ($0.058 \mu\text{m}$ – $0.098 \mu\text{m}$) is thus clinically acceptable.

The results of our study showed that the alkasite had the highest roughness when compared to conventional nanofilled and bulk-fill nanocomposite. Previous studies

Table 2: Surface roughness measurements (Ra in nm) at baseline and 3 months

| Groups | Mean±SD | |
|----------|----------------------------|----------------------------|
| | Baseline | 3 months |
| Group 1 | 64.13±0.08 ^{a,ab} | 64.37±0.71 ^{a,ab} |
| Group 2 | 61.94±4.21 ^{b,ab} | 62.37±3.81 ^{b,ab} |
| Group 3 | 59.25±4.87 ^b | 59.21±5.02 ^b |
| <i>F</i> | 4.75 | 5.56 |
| <i>P</i> | 0.016* | 0.009* |

* $P < 0.05$: Significant; Same alphabets in superscript denote no statistical difference. SD: Standard deviation

Table 3: Contact angle measurements (°) at baseline and 3 months

| Groups | Mean±SD | |
|----------|--------------------------|---------------------------|
| | Baseline | 3 months |
| Group 1 | 98.80±29.58 ^a | 105.61±13.04 ^a |
| Group 2 | 58.06±27.26 ^b | 58.36±20.41 ^b |
| Group 3 | 65.74±23.76 ^b | 65.24±10.28 ^b |
| <i>F</i> | 7.08 | 31.06 |
| <i>P</i> | 0.003* | <0.001* |

* $P < 0.05$: Significant; Same alphabets in superscript denote no statistical difference. SD: Standard deviation

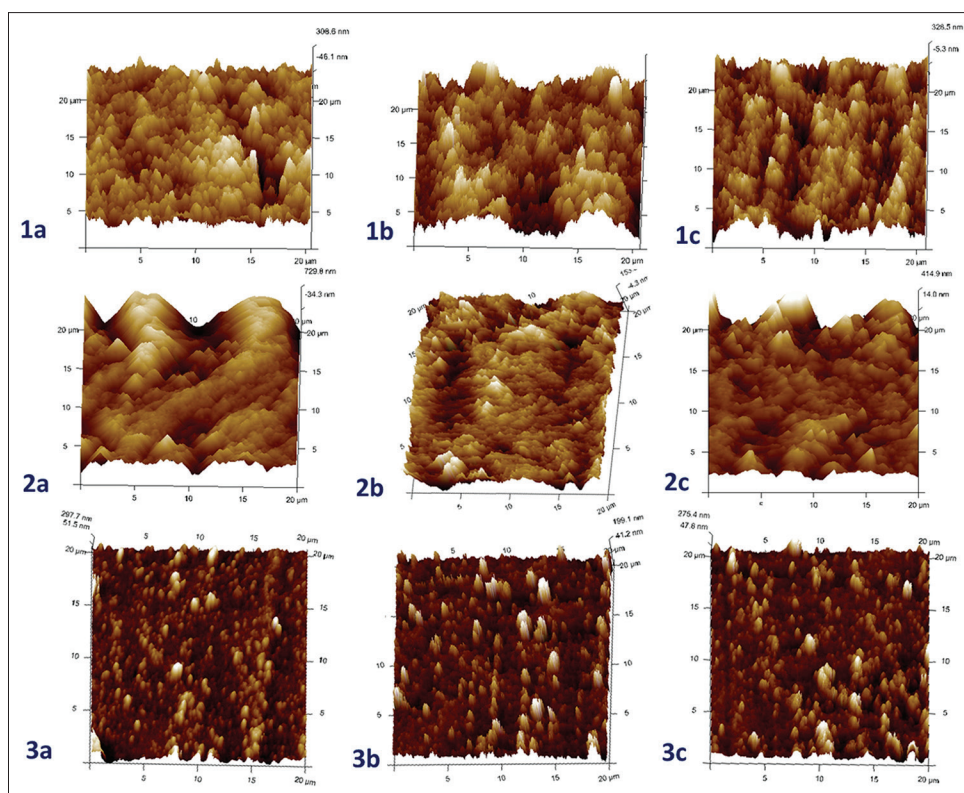


Figure 1: Atomic force microscope image of the polished surfaces of the resin-based composite specimens at baseline. 1a, 2a, 3a: center of the specimen; 1b, 2b, and 3b at half distance between specimen's center and perimeter and 1c, 2c, 3c at specimen's perimeter

comparing surface roughness of various composite materials have shown that the surface roughness of the RBCs is affected by their composition, especially the size, shape, hardness, and amount of filler particles.^[2,9,18] Alkasite resin composite has higher filler particle size and least filler load of all the composites and thus showed highest surface roughness values. Qualitative analysis of the AFM images also showed higher areas of loss of resin matrix and fillers in this group when compared to the more regular surface topography for nanocomposites. The results of our study are similar to a study done by Senawongse and Pongprueksa^[19] which stated that nanofilled resins show the least surface roughness due to the presence of nanoclusters and their higher filler content.

During finishing and polishing, some amount of resin matrix is lost, exposing the fillers.^[20] Higher filler loading also means more filler particles are in contact with the polishing instrument, which minimizes excessive abrasion of the resin matrix. Some amount of the filler particles may also be lost during polishing, and the loss is greater when the hardness level, which is determined by filler load, is lower. Loss of larger and irregular filler particles leave larger voids, increasing the surface roughness.^[21,22] Alkasite has larger dimensions of filler particle size when compared to nanofilled RBCs, with a heterogeneous range of 0.1–35 μm .

In vitro aging method uses exposure of the material to chemicals such as water or ethanol.^[23] They give us an

understanding of the degradation of properties of the material in the oral environment, as *in vivo* studies are more time-consuming and expensive to carry out.^[24] Exposure of RBCs to water results in rapid elution of the unreacted monomers in the first 1–4 weeks, resulting in the formation of voids/pores.^[25] Simultaneously, water absorbed by the resin occupies the voids/pores and the space between the polymer chains by a slow, diffusion-controlled process. This affects the mechanical properties of the resin as secondary chain interaction during polymerization is affected.^[24,25] It has been shown that urethane dimethacrylate (UDMA)-based resins show more degradation in aqueous environment than bisphenol A-glycidyl methacrylate (Bis-GMA) resins.^[24] This explains the increase in surface roughness values after 90 days of aqueous aging in this study, although not significant, for alkasite and bulk-fill nanocomposites, as both are UDMA based. Weakening of resin matrix due to aging results in dislodgments of the filler particles leading to roughening of the surface. RBCs with higher filler loading show decreased surface degradation during aging.^[24,26]

In this study, contact angle measurement was higher for alkasite resin composite, implying less wettability and hence less susceptible to bacterial adhesion than conventional nanofilled resin composite. Ability of the resin composite to release fluoride influences the surface wettability which may be one of the factors leading to lower wettability of the alkasite.^[13,27] For nanocomposites,

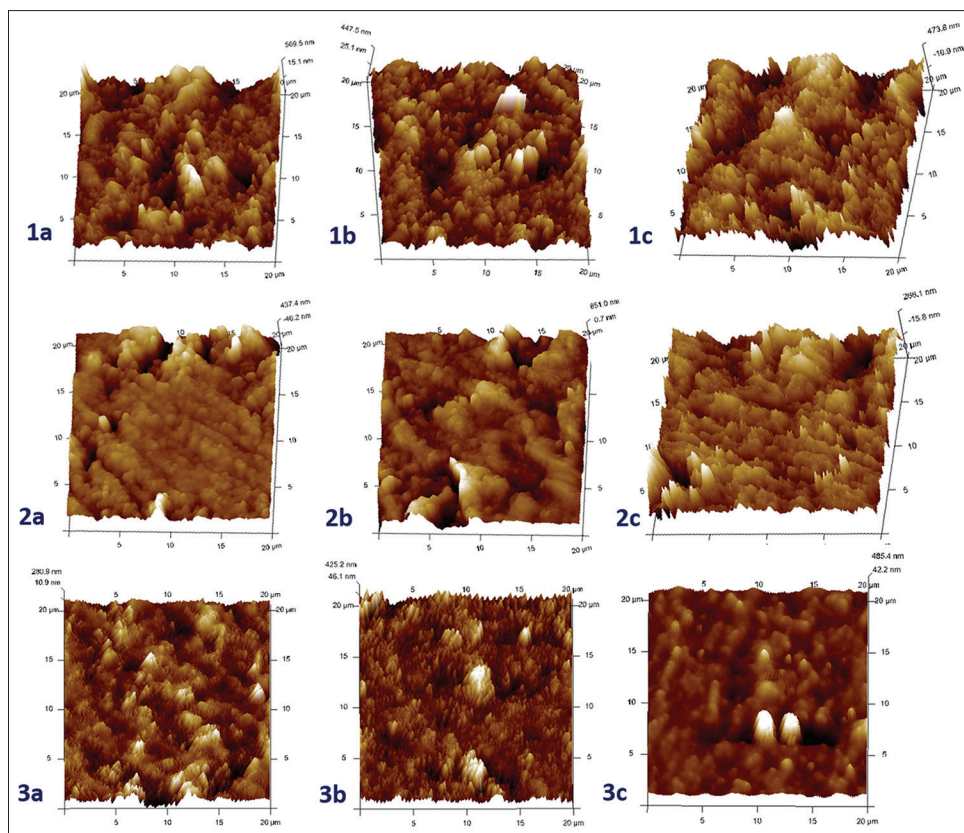


Figure 2: Atomic force microscope image of the polished surfaces of the resin-based composite specimens after 3 months. 1a, 2a, 3a: center of the specimen; 1b, 2b, 3b at half distance between specimen's center and perimeter and 1c, 2c, 3c at specimen's perimeter

lower contact angles were observed due to higher filler content.^[3] This difference in wettability plays an important role in initial plaque adhesion.^[3,18]

If the contact angle is 60° – 86° , surface roughness has no influence on surface wettability.^[28] This was the case with alkasite resin composite, which had the highest surface roughness but least wettability. However, surface roughness influences the bacterial adhesion to the material surface to a greater extent than the surface free energy and has an important role in plaque retention, as the rough surface protects bacteria against shear forces.^[12,27] Thus, it may be inferred that nanofilled composites are more advantageous in terms of surface characteristics than alkasite resin composite.

AFM method allows 3D imaging at a nanometric resolution, giving a better description about the surface topography with more precise readings.^[20] However, it gives only localized values of surface roughness, as compared to surface profilometry, which gives more global values.^[9] Another limitation is the use of only water as the aging medium. The effect of other mediums such as artificial saliva, acids, and ethanol needs to be evaluated. It should be noted that *in vivo* conditions, the change in the surface roughness due to aqueous aging and the wettability of the material is altered by the presence of salivary pellicle, which is not considered in an *in vitro* design.^[27,29]

Hence, further clinical studies are needed to evaluate the surface characteristics of these composites.

Conclusions

Within the limitations of the study, it can be concluded that alkasite has higher surface roughness in comparison to conventional and bulk-fill nanocomposite and lower wettability in comparison to conventional nanocomposite after polishing with a diamond polishing system and aqueous aging over 3 months. In terms of surface characteristics of the resin composite material, which influences plaque biofilm colonization and hence secondary caries, alkasite composite may be advantageous only in preventing initial plaque adhesion to the material surface, but the lower surface roughness of nanocomposites may be more advantageous in terms of plaque retention prevention.

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Conflicts of interest

There are no conflicts of interest.

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