ACTA SCIENTIFIC DENTAL SCIENCES (ISSN: 2581-4893)

Volume 3 Issue 3 March 2019

Research Article

Evaluation of Surface Roughness of Composite Resins with Three Different Polishing Systems and the Erosive Potential with Apple Cider Vinegar Using Atomic Force Microscopy-an *In Vitro* Study

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Received: January 30, 2019; **Published:** February 14, 2019

Abstract

The aim of this study was to evaluate the effect of polishing procedures on the Surface roughness of a Nano filled and Universal submicron hybrid composite and to study the effects of acidic drinks on these materials - An invitro study. 120 composite samples were divided into 2 groups based on the composite used. These were further divided into 4 subgroups based on the finishing and polishing system used. The surface roughness of samples was measured with an optical profilometer and atomic force microscope.

20 samples from each group was immersed in apple cider vinegar. The evaluation of surface topography and microhardness was done using AFM and Microhardness tester.

In the present study, Universal submicron hybrid composite presented lower Ra values when compared to Nanofilled composites in unpolished controls. Soflex diamond polishing system showed better polishing ability in both the groups than Politip and Optidisc. Highest mean microhardness values after immersion in acidic drink was observed for the Nanofilled composites compared to Universal Submicron hybrid composites. The immersion of restorative materials in acidic drinks can cause surface roughness and reduced microhardness which increases with time. Surface roughness associated with improper finishing and polishing can result in increased wear rates and plaque accumulation which can compromise the clinical performance of the restoration.

Keywords: Universal Submicron Hybrid Composite; Nanofilled Composite; Atomic Force Microscopy; Apple Cider Vinegar; Surface Roughness, Erosive Potential

Abbreviations

AFM: Atomic Force Microscope; VHN: Vickers Hardness Number; LED: Light Emitting Diode; NFC: Nanofilled Composite; OP: Optical Profilometer; RBC: Resin Based Composites; USHC: Universal Submicron Hybrid Composite.

Introduction

The search for an ideal esthetic material for restoring teeth has resulted in significant improvements in esthetic materials and techniques for using them. Composite resin proved to be the excellent esthetic material and was modified with respect to the resin matrix and filler [1].

Composite resins are heterogeneous materials composed of a resin matrix typically a dimethacrylate, reinforcing filler typically made of radiopaque glass, a silane coupling agent for binding the filler to the matrix and chemicals that promote or modulate the polymerization reaction. Within each type of composite resin, their characteristics of reinforcing fillers and particle size helps in distinguishing the macrofilled, microfilled and hybrid. Reduced particle size of 0.04 - 1 µm helps in the handling and polishing. Nanocomposites consist of nanomers (5nm to 75nm particles) and nanocluster agglomerate fillers (0.6 µm to 1.4 µm). Nanocomposites and universal submicron hybrid composites demonstrate mechanical and physical properties similar to those of hybrid composites along with excellent polishability and gloss retention producing restorations with better finish and esthetics [2].

Characterization of the surface texture of composites is important to enhance esthetics, color stability and longevity of restored teeth. The type of composite material and the finishing and polishing systems play an important role in bringing about adequate smoother surface which will avoid the onset of subclinical or even clinical inflammation [3]. The role of finishing helps in gross reduction of excess material, removes overhangs as well as restore the occlusion and morphology to achieve optimal function. Different types of finishing and polishing abrasive systems include aluminium oxide, carbide compounds, diamond abrasives, silicon dioxide, zirconium oxide and zirconium silicate and polishing instruments like coated abrasive discs and strips, stones, aluminium oxide or diamond pastes, soft or hard rubber cups or points, and wheels or brushes impregnated with abrasives [4].

Tooth wear and deterioration of restorative materials are a common problem which can be aggravated by using beverages like sports drinks, fruits and vegetable juices, soft drinks, coffee, green tea, probiotic milk, apple cider vinegar owing to their health benefits. Surface roughness of composite restorations is related to the type of composite, finishing and polishing systems used. So, the rougher surface produced by the consumption of these drinks allows the fast colonization by microorganisms and maturation of the biofilm, increasing the risk of the development of dental caries and periodontal disease as well as the susceptibility to staining of the restoration [5].

The significance of the present study was to assess the surface roughness of composite resins using different finishing and polishing methods and the erosive potential of the composite material using apple cider vinegar. Hence, the purpose of this *In vitro*-study was to evaluate a technique that will help achieve maximum esthetics and biological success when contouring, finishing, and polishing restorations.

Materials and Methods

For this study two composites and three finishing and polishing systems were used. The composites used were Universal Submicron Hybrid composite (Brilliant Everglow) and Nanofilled composite (Filtek Z350 XT). The finishing and polishing systems taken were Politip, Soflex TM Diamond polishing and Optidisc 4200.

The methodology used in this study explained under the following headings

Preparation of composite samples

- A total of 120 samples of composite resin were fabricated using plexiglass mold. The resin composites (Universal submicron hybrid composite and nanofilled) were compacted into the custom-made plexiglass mold of internal diameter (6 mm × 6 mm) using a teflon coated plastic filling instrument and the excess was carefully removed with an explorer.
- A mylar strip was sandwiched between the upper surface of composite resin and a glass slide of 1-2 mm thickness placed before curing with light activated source (LED Curing light) to flatten the surface.
- Following compaction of the resin, the samples were then cured in increments for 60s through mylar strip and glass slide. The curing unit was moved on both sides of the samples for an additional 20s after removing the strips and glass slides.
- The cured samples were then stored in distilled water at 37°C for 24 hours, prior to finishing procedures.

Grouping

- 120 samples were divided in to 2 groups based on the type of composite used into Group 1 - Universal submicron Hybrid Composite resin and Group II - Nanofill composite resin. Based on the finishing and polishing system used they were further subdivided into Subgroup A, B, C and D each consisting of 15 samples. Subgroup A - Control Group (No finishing treatment) Subgroup B: Politip, Subgroup C - SofLex Diamond Polishing system and Subgroup D - Optidisc 4200

Finishing and polishing of composite samples

- In Subgroup B polishing was carried out using Politip F (grey) for 30 seconds followed by Politip P (green) for additional 30 seconds. Surface of the samples in subgroup C were smoothened using 3M Sof-Lex pre-polishing spiral(beige). Sof-Lex Diamond polishing spiral was used for obtaining the final high gloss polishing. Spirals was used on wet surfaces at a medium to moderate pressure (30 - 60 grams force). All surfaces of the spiral (top, bottom, edge) was utilized for maximum access to the restorative surface. Polishing procedure in Subgroup D involved use of abrasive disk of all four grits in the Optidisc 4200 polishing kit in a dry condition. The samples were first contoured using extra-coarse grit (80 µm), finished with coarse/medium grit (40 µm), polished with fine grit (20 µm) and to obtain high gloss polish extra-fine grit (10 αm) was used sequentially.

- The Optishine, concave shaped polishing brush was used after the extra fine grit to obtain a smooth polished surface. All samples were polished using a planar motion using a slow speed handpiece running in a forward direction at a speed of 15000-20000 rpm in both the groups. After each step of polishing, all specimens were thoroughly rinsed with water and air - dried before the next step until final polishing. The polished resin composite samples with the molds was washed, allowed to dry and kept in distilled water at 37°C for 24 hrs before measuring the average surface roughness.

Measurement of surface roughness

- The mean surface roughness of samples was measured using an optical profilometer. The surface roughness was measured three times for each sample using this instrument. For each surface, Ra readings were taken three times, and it was scanned at higher resolution using an optical beam. The optical profilometer shows the three-dimensional topography of the samples.
- In oder to have a better clarity of the surface topography the samples were evaluated under Atomic Force Microscope.The images obtained were subjected to surface roughness analysis using software provided and the parameter Average roughness (Ra) was compared.

Assessment of erosive potential of acidic drinks on composite resin after immersion

In this study, apple cider vinegar was used to assess the erosive potential on composite resin. pH of apple cider vinegar was determined using a pH meter. Twenty samples from each group were immersed in 10 ml of 1:1 dilution of apple cider vinegar.The assay was performed simulating the time contact of the restorative material in the oral cavity. After the immersion period in the test solutions, the samples were washed with deionized water and were maintained at 37°C during the rest of the day.

Evaluation of surface hardness and topography

After the immersion sequence is completed, the samples were rinsed with distilled water, blotted dry and subjected to Vicker's microhardness testing before immersion and at the end of 30 days. To assess the erosive potential of apple cider vinegar on both the groups surfaces were evaluated using atomic force microscope.

Statistical analysis

Data were statistically analyzed using Kruskal Wallis test, Mann Whitney and Wilcoxon Signed Rank test.

Statistical Package for Social Sciences [SPSS] for Windows Version 22.0 Released 2013. Armonk, NY: IBM Corp., was used to perform statistical analyses.

Polishing system	Group	N	Mean OP values (µm)	Mean Diff	Mean AFM values (nm)	Mean Diff
Control	USHC	15	0.0141	-0.0764	25.28	-8.54
	NFC	15	0.0905		33.82	
Politip	USHC	15	1.7239	0.4208	53.09	-3.10
	NFC	15	1.3031		56.19	
Soflex	USHC	15	0.1776	0.0641	29.87	-12.06
	NFC	15	0.1135		41.93	
Optidisc	USHC	15	1.0902	-0.7039	50.45	-7.30
	NFC	15	1.7941		57.75	

Table 1: Comparison of Surface Roughness [Ra] between Group I and II using Optical Profilometry (OP) and Atomic force microscope (AFM) (Mann Whitney U Test).

Group	Immersion	N	Mean	SD	Mean Diff	Z	P-Value
USHC	Before	10	58.90	3.90	16.30	-2.807	0.005*
	After	10	42.60	5.08			
NFC	Before	10	79.20	8.70	17.10	-2.701	0.007*
	After	10	62.10	5.04			

Table 2: Comparison of mean Microhardness [VHN] between Before and After Immersion of Group I and II in Apple Cider Vinegar using Wilcoxon Signed Rank Test.

Solution	Composite	N	Mean	SD	Mean Diff	Z	P-Value
AC Vinegar	USHC	10	40.10	4.91	-15.40	-3.705	<0.001*
	NFC	10	55.50	4.22			

Table 3: Comparison of mean AFM values for Erosive Potential between USHC and NFC samples after immersion in Apple cider Vinegar using Mann Whitney Test.

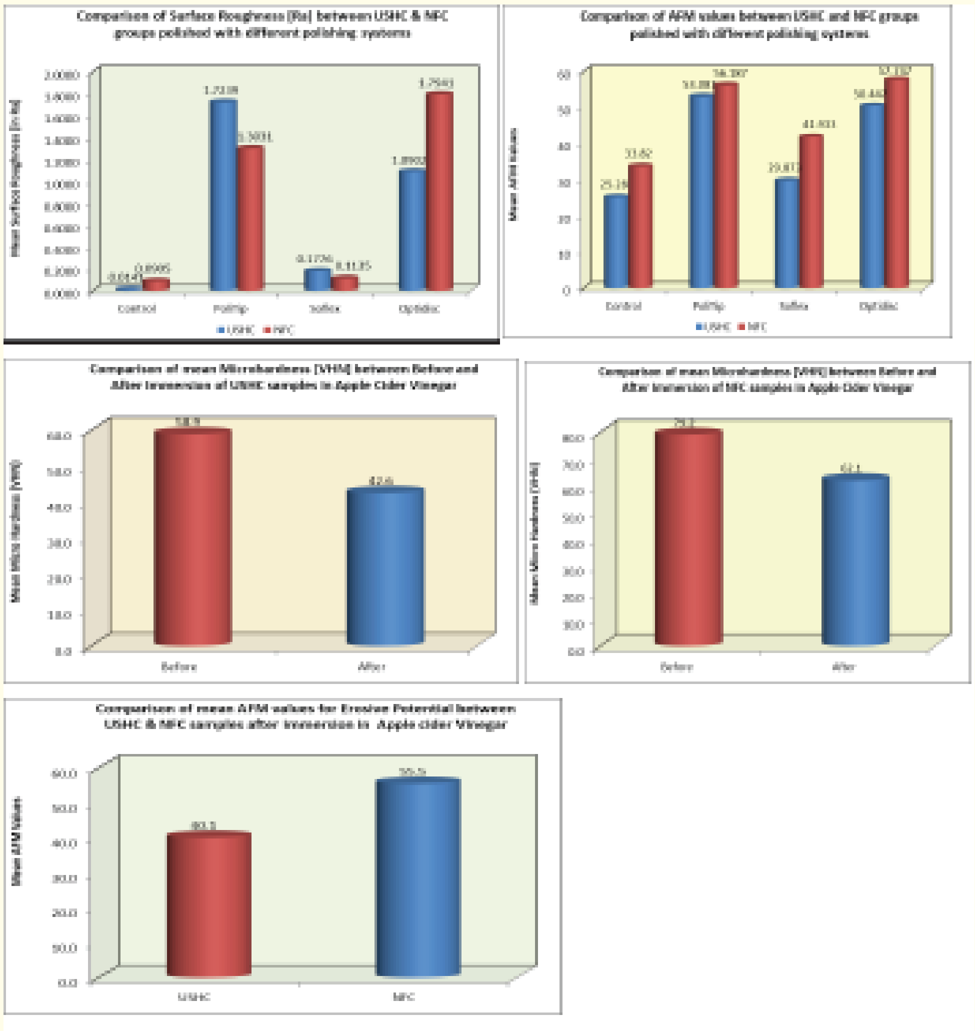

Figure a

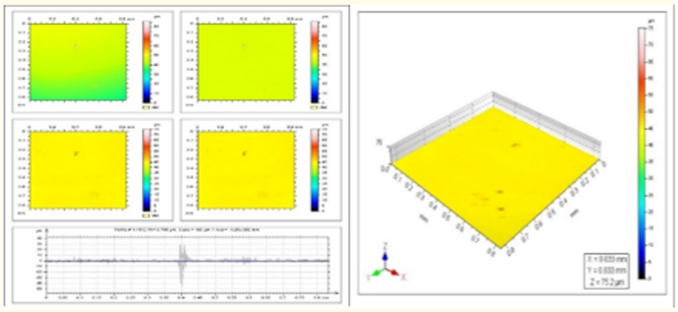

Figure 1: Optical profilometry image for group I, sub group C.

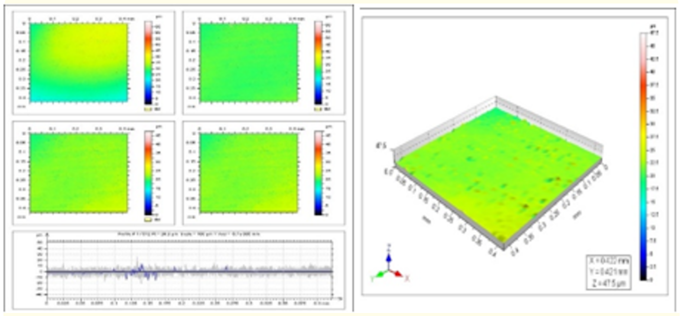

Figure 2: Optical profilometry image for group II, sub group C.

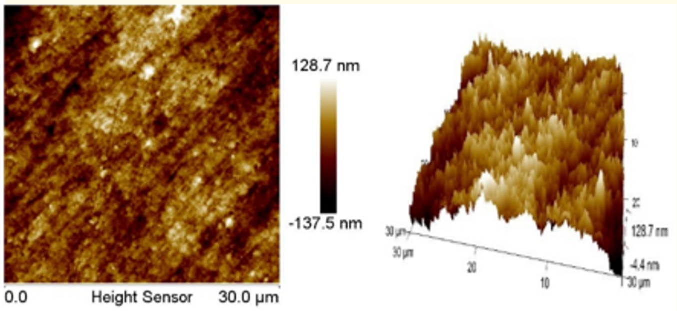

Figure 3: AFM images – group I, subgroup C.

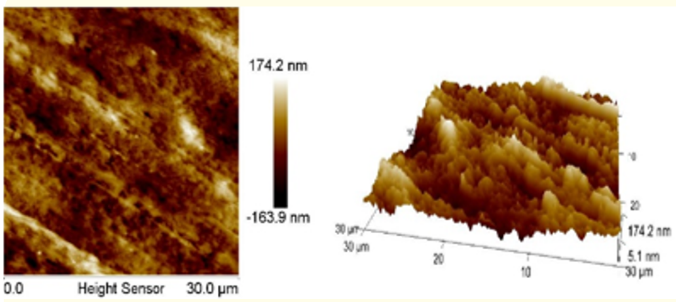

Figure 4: AFM images – group II, subgroup C.

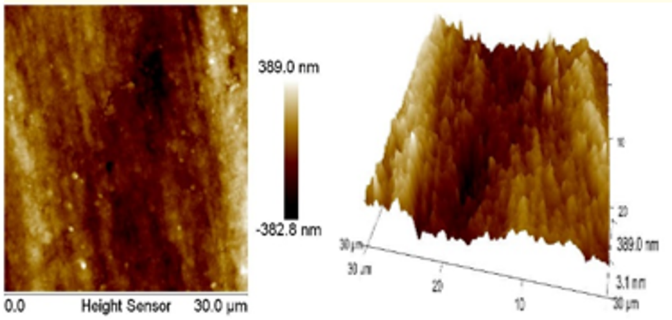

Figure 5: AFM images – post immersion of group I in apple cider vinegar.

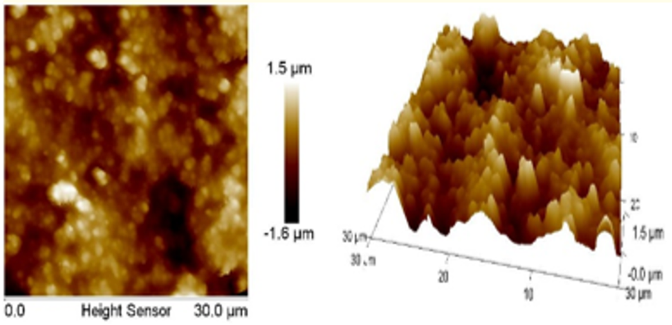

Figure 6: AFM images – post immersion of group II in apple cider vinegar.

Discussion

- In the field of esthetic dentistry there is a continuous development with respect to the adhesive restorative materials which is tooth colored. The critical steps to enhance the esthetics and longevity of the composite restorations depend on excellent finishing and polishing. A resin composite for a restoration requires the evaluation of its functional properties, including high strength, fracture toughness, surface hardness, optimum polishability and gloss [6].
- Restorations which are poorly polished are susceptible to discoloration, gingival irritation and recurrent caries. Hence, the factors like composition of the resin with respect to matrix and filler particle size, type of polishing system, degree of polymerization plays an important role.
- Exposure to acidic environment may affect the dental restorations since the pH level may vary causing erosion of the restorative materials by leaching out of the matrix forming substances. The influence of the chemical degradation reflects a change in the microhardness and surface roughness [7]. When selecting materials for

repairing erosive lesions, acid resistance is an important property to consider [8].

- In the present study we investigated the surface roughness of 2 composites (Universal submicron hybrid composite and Nanofill composite) using 3 polishing systems (Soflex Diamond polishing system, Politip and Optidisc 4200) with Optical profilometer and Atomic force microscope. We also found out the erosive potential of restorative materials after immersion in apple cider vinegar for a period of 30 days while monitoring changes in hardness.
- Quantitative analysis to evaluate surface roughness was done using Optical profilometry and qualitative evaluation by atomic force microscopy (AFM).
- In our study lowest surface roughness in both the groups was found to be less for samples which were in contact with the Mylar strip (subgroup A). The mean Ra values found using the matrix strips in this study (Group I = 0.0141 µm and Group II = 0.0905 µm) were lower than the threshold Ra value of 0.2 µm in accordance with the studies done by Bollen., *et al*. Gedik., *et al* and Uctasli., *et al*. The use of clear polyester strips over the last increment of material in composite resin restorations is a usual step to avoid the oxygen inhibition layer on the resin surface. However, the resulting surface is rich in organic matrix, leading to a relatively unstable surface. Resin rich layer on the surface of composite resin need to be removed by polishing to avoid accelerated clinical wear. In addition, the oral environment will be exposed to inorganic filler content if no polishing procedure is carried out. [11] However, this method is not commonly used in clinical practice, because the correct anatomical contour of the restoration is rarely achieved using only mylar strip**.** Moreover, the high content of submerging organic matrix promotes an insufficient polymerization, which results in reduced hardness and discoloration of the surface [9,10,11].
- An increase in surface roughness values was observed in all the 3 subgroups i.e. Politip group, Soflex diamond polishing group and Optidisc group than the control group. Among the polished groups, the smoother surfaces were seen in Soflex diamond group compared to the samples in Politip and Optidisc.
- The mean Ra value for Soflex spirals was found to be 0.1776 µm which showed that it had better polishing ability followed by Optidisc (1.0902 µm) and Politip (1.7239 µm) in Group I. The mean Ra value for Soflex spirals was 0.1135 µm which was less when compared to Politip (1.3031 µm) and Optidisc (1.7941) in Group II. Thus, Soflex diamond polishing produced less surface roughness in both the groups as seen by optical profilometry and AFM results. The above results were in accordance with studies done by Guhati., *et al*, Hegde., *et al*. and Turssi., *et al*.
- Soflex spiral wheel had least Ra value as compared to other subgroups and it may be attributed to the presence of diamond particles and aluminum oxide in Soflex spiral wheel, which promote homogenous abrasion of fillers and resin matrix. This is in accordance with a study done by Mohammed S Abzal., *et al*. and Mensudar Rathakrishnan., *et al*. The excellent polishing ability of Soflex spirals may be attributed to lower surface roughness and harder diamond particles (7000KHN) and aluminium oxide particles (2100 KHN) compared to aluminium oxide particles (2100KHN) alone in Optidisc and silicon carbide particles (2500KHN) in Politip. The hardness of aluminium oxide is significantly higher than silicon dioxide, generally, higher than most filler materials used in composite formulations. The trend of Sof-Lex discs is to provide a slightly smoother surface with the aluminium oxide abrasive on rigid matrix as this can flatten the filler particles and abrade the softer resin matrix at an equal rate [12-14].
- Politip and Optidisc subgroups presented higher Ra may be because they were not capable to reduce both the resin and filler particles at an equal rate. Filler particles should be situated together as close as possible in order to protect the resin matrix from abrasives. The discrepancy between the size of abrasive particles present in the abrasive disks and abraded material should be minimal to reduce the creation of scratches or roughness on the polished surface [15].
- The reason for the aluminum oxide (SofLex) discs giving better surface smoothness could be due to the non-displacement of the composite filler particles by SofLex as stated by Herrgott., *et al*. The aluminum oxide discs (Sof-Lex) performed better because the fillers in composite are so small that their stiffness is reduced and so their malleability promotes a homogeneous abrasion of the fillers and the resin matrix (Yap., *et al*. 1997). Study by Mitra., *et al*. (2003) also supported the concept of homogeneous abrasion.
- Limited use of Aluminium oxide discs is because of their shape, which makes them difficult to use efficiently, particularly in the posterior regions of the mouth. Soflex spiral wheels have a unique, flexible shape which easily adapts to irregular, convex and concave tooth surfaces and is effective from any angle since abrasive particles are embedded throughout the tool. The unique shape of the Soflex spiral wheel is an advantage over Soflex discs in adapting to all tooth surfaces. Hence, we have used Soflex spirals which has both aluminium oxide and diamond particles in this study.
- Fruits have reported that three types of motion may be critical to the development of optimal surface smoothness rotary motion (circular), planar motion, or reciprocating motion. It was found that for all the possible combinations of materials and abrasive grits, the planar motion achieved the lowest Ra values [16]. In the current study, a planar motion was used for all the polishing systems. The planar motion is a rotational

movement with the axis of the rotation of the abrasive device perpendicular to the surface being smoothened. Sarita., *et al*. and Nitin., *et al*. reported that specimens polished with planar motion (Sof-Lex disks) gave lower surface roughness values than the specimens polished with rotary motion (Shofu) in microhybrid and nanofilled composites.

- The mean Ra value in subgroup A of Group I (0.0141 µm) was found to be less than Group II (0.0905 µm) suggesting better polishability and gloss retention for Group I (Universal submicron hybrid composite) when compared to Group II (Nanofilled composite).
- The microhybrid composite resins demonstrated significantly lesser roughness (0.0141 µm) than the nanofilled composite resin (0.0905 µm) under matrix strips. (Refer table 1). The filler particle size and the interparticle spacing in the composite resin attributes to the differences in surface topography. In this study Filtek Z350 XT (nanofilled resin composite), having an average filler particle size of 0.005-0.02 µm and universal submicron hybrid composite with a filler particle size of 0.01 - 0.2 µm were used. Submicron hybrids contain a broad distribution of particle sizes with the larger particle sizes (1 micron). This influences the optical properties and detracts from polish retention [17]. Yap., *et al*. and Tjan., *et al*. suggested that materials with fillers of larger sizes generally show more surface roughness than those with smaller sizes after polishing. Therefore, the lower filler loading and higher resin ratio of microhybrid composite compared with nano-filled composites may be responsible for the significantly reduced surface roughness within control group. This assumption is reinforced by Reis., *et al*. Nagem Filho., *et al*. Turkun., *et al*. They emphasize that the composite resins with a higher percentage of loading and better distributed particles in the resinous matrix have greater surface smoothness [18,19].
- In this study, apple cider vinegar was used to evaluate its effect on microhardness and surface roughness of composite resins. The material's microhardness is one of the most important properties, which correlates with resistance to intra-oral softening, compressive strength and degree of conversion (Voltarelli., *et al*. 2010). A low surface microhardness value is largely related to inadequate wear resistance and proclivity to scratching, which can compromise fatigue strength and lead to failure of the restoration (Erdemir., *et al*. 2013). The factors determining the hardness include composite characteristics such as type of the organic matrix, size, distribution of loading particulates including the material's exposure to low pH food, drinks and mouth rinse solutions and factors related to the abrasive system such as flexibility of the material in which the abrasive is impregnated, hardness of the abrasive, grit size and geometry, speed and form of application of the instruments used [20,21]. The correlation between filler particle size and erosion of Resin based Composites have been studied [22].
- The lowest mean Ra value after immersion was observed in Universal submicron hybrid composite (25.28nm) when compared to Nanofilled composite (33.82nm) (Refer Table 3) This indicates a smoother surface for Universal Submicron hybrid composite than nanofilled composite. (Refer figure 5). Higher microhardness after immersion was observed for Nanofilled composites (62.10VHN) when compared to Universal Submicron Hybrid composites. (42.60 VHN). In our study a correlation between microhardness and surface roughness was observed. The materials with higher surface hardness presented higher surface roughness which was in agreement with the studies done by Tjan and Chan and Marcos Aurelio., *et al*.
- In this study, the reduction in microhardness is due to the difference in their filler or monomer ratio and hydrolytic breakdown of the silane/filler particle bond which is in consensus with studies conducted by Soderholm., *et al*. Medeiros., *et al*. and Bagheri., *et al*. Filler particles dislodge from the outer surface of the material causing surface roughness and decreasing hardness as stated by Santos C., *et al*. and Clarke RL., *et al*. This situation causes plasticizer effect of the organic matrix and consequent degradation. The microhardness value depends on the Degree of conversion (DC) Therefore, lower microhardness of universal submicron hybrid composites compared with the nanofilled composites might be related to lower Degree of conversion and and the type of fillers [23]. Filtek Z350 XT composite resins have an organic matrix of Bis EMA monomer with high molecular weight and it is more resistant to degradation due to removal of terminals OH-groups, which are susceptible to absorption and solubility [24]. The zirconia and silica fillers in the Filtek Z350XT have greater hardness and less solubility when compared with fillers in Brilliant Ever glow. In addition to the Bis EMA monomer other monomers present in Filtek Z350 XT composites such as UDMA, TEGDMA, and Bis-GMA are highly susceptible to absorption and solubility in contact with low pH substances, causing softening and degradation of the organic matrix [25]. Therefore after immersion surface roughness increased considerably when compared to Universal Submicron hybrid composite.
- The acidic drinks can penetrate the resin matrix and accelerates the release of unreacted monomers via reducing the surface hardness. The increase of the interaction and reaction between solution and resin materials such as water absorption and erosion due to the acidic condition leads to the decrease in the surface hardness of resin composites [26].
- The surface nano-indentations of resin samples is accounted to the various components of the acid. In the present study Apple cider containing acetic acid, citric acid and malic acid which produced the surface indentation effect. Apple cider has the highest titratable acidity as seen in a study by Saijai Tanthanuch., *et al*.

- In our study, AFM images showed erosion of nanofilled composite surface. This finding indicated that the superficial layer has undergone corrosion, but less softening has occurred in the subsurface layer compared with submicron hybrid composites as seen in Figure 5 and 6. This is in accordance with Nuran Yanikoglu., *et al*.
- The limitations of the current study include incomplete replication of the complex oral environment. While future studies may examine the *In vivo* effects of acidic drinks, this study attempts to confirm the erosive potential of certain acidic drinks, a potentially damaging factor of which the public should be aware of. The erosive potential of an acidic drink is not exclusively dependent on its pH, but also strongly influenced by its titratable acid content in beverages. But the present study did not take into account the titratable acid content.
- Therefore, the results of the present in-vitro study provide an insight of the commonly consumed apple cider vinegar on the surface roughness and microhardness of the restoration.

Conclusion

From the present study, it was concluded that:

1. The type of resin-composite and the polishing technique used are important factors affecting the surface roughness and gloss of composites.
2. Universal submicron hybrid composites (Brilliant Ever glow) showed better polishing ability than nanofilled composites in the unpolished controls.
3. Soflex diamond polishing system showed better polishing ability for both Universal submicron hybrid and Nanofilled composites.
4. Nanofill composite (Filtek Z350) showed higher surface hardness than Universal submicron hybrid composites.
5. Apple cider showed reduction in surface microhardness of both the composite resins tested.
6. Nanofilled composites showed more resistance to fracture and wear, therefore having a better clinical performance for restorations especially in areas of more masticatory load. The use of Universal submicron hybrid composite can be taken into consideration for restoring anterior esthetic areas.

However, further studies are extensively required to evaluate the effect of finishing and polishing agents on the restorations and their co-relations.

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Volume 3 Issue 3 March 2019

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