Evaluation of Surface Roughness and Hardness of Newer Nanoposterior Composite Resins after Immersion in Food-Simulating Liquids

Abstract

Introduction: Restorative resins during their prolonged use are exposed to variety foods and beverages are subjected to wear, degradation, and staining resulting in failure of restoration and require replacement. This study is aimed to evaluate surface roughness and hardness of five commercially available posterior resin composites following exposure to various food-simulating liquids (FSLs). **Materials and Methods:** Specimens were immersed in distilled water, ethanol, citric acid, and air and later examined using a profilometer, scanning electron microscope for the surface profiles. Hardness was measured by Vickers Hardness test. **Statistical Analysis:** Results were subjected to two-way ANOVA and Tukey's multiple *Post hoc* test. **Results:** There were significant differences in results among the composite resin tested. Inter comparison between materials after treating with FSLs, clear fill majesty (CFM) and Z350 showed better hardness values under the influence of ethanol, followed by Ever X, Tetric Evo Ceram and Sure fill SDR. None of the composites were unaffected by air compared to FSLs. Citric acid had reduced the hardness of CFM and had caused surface roughness on Sure fill SDR and Tetric Evo Ceram. Distilled water reduced hardness of CFM and SureFil SDR. EverX was not affected by any of the FSL either in hardness or surface roughness properties. All composites showed surface irregularities in all media. Ethanol and water had almost similar effect on all composites. **Conclusions:** Differences in hardness and surface roughness are due to different composition of resin matrix and different filler particles in all composite resin material tested.

Keywords: *Profilometric analysis, scanning electron microscope, surface roughness, Vickers hardness test*

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Introduction

Restorative resins are the most commonly used materials in clinical dentistry. These materials after months and years of use and exposure to a variety of different foods and beverages are subjected to wear, degradation, and staining resulting in failure of restoration and require replacement.[1] Yap *et al*. have shown that food substances and oral environment significantly affect the hardness and roughness of composites.[2]

The physical and mechanical properties of composite resins are composition dependent. The polymeric matrix is based on a mixture of dimethacrylate such as bisphenol-A glycol dimethacrylate (Bis-GMA), urethane dimethacrylate (UDMA), bisphenol-A dimethacrylate (Bis-EMA), and triethylene glycol dimethacrylate (TEGDMA).[3] The filler concentration of resin is generally 70%–80% by weight, comprising radiopaque silicon dioxide, boron silicate, lithium aluminum silicates,[4] and heavy metal particles such as barium, strontium, zinc, aluminum, or zirconium. Foods and beverages are extrinsic factors that can degrade and cause aging of composite resins in the oral cavity. Yap *et al*.[2] and Wu *et al*.[5] reported that food-simulating liquids (FSLs) such as citric acid, lactic acid, heptane, and ethanol softened the composite surface.

Aging of composite resins has been simulated by storage in water, citric acid immersion,[6] and ethanol in previous studies.[1,7] Water storage is considered to have detrimental effects on the composite resin surface according to Sideridou *et al*.[8] Citric acid immersion simulates effect of acid in foodstuffs such as vegetables, fruits, candy, syrup, and beverages according to Yap *et al*.[2] Aqueous ethanol solution has been the solvent of choice to stimulate and accelerate aging of restorations.[1]

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In the present study, distilled water, 50% ethanol, and 0.02N citric acid are used as food-simulating solutions. These liquids are recommended in guidelines from the US Food and Drug Administration to be used as food simulators.[9] Surface roughness is important for any restorative material as it affects the wear, color, optical, and hardness of restorative composites. Hardness of material is described as resistance of a material to permanent indentation.[10] Say and Kanchanavasita *et al*. in their study have shown link to low hardness values to lower wear resistance.[11,12] Many of these factors are associated with the morphological and physical characteristics of the inorganic particles, but there is lack of studies that systematically evaluate such properties before and after the degradation process. Surface roughness is principally determined by the presence of protruding filler particles above the resin matrix and intruding porosities.[12] In the present study, surface roughness was measured using profilometer, surface irregularities using scanning electron microscope (SEM), and hardness by Vickers hardness test (VHT) after immersion in various FSLs. The aim of the present study was to evaluate the effects of FSLs such as citric acid, 50% aqueous ethanol solution, and distilled water on the surface roughness and hardness of newer posterior composites.

Materials and Methods

Five commercially available posterior composite tested in this study were divided into five groups – A, B, C, D, and E. The details of the material are given in Table 1. A total of 70 specimens, 10 of each material was prepared using a customized circular mold (dimensions 5 mm [diameter] and 2 mm [height]). Excess flash, air bubbles, smooth surface, and minimization of oxygen inhibited layer of specimen material were achieved by covering with a transparent polyester strip on both sides over glass slab and pressed between glass slides and later cured by smart light light-emitting diode curing unit (Dentsply) using 40 s exposure to each specimen's top and bottom surface with standardized distance between light source according to manufacturer's instructions. The light intensity of curing light was checked regularly with the radiometer during specimen preparation. The specimens divided into four groups were immersed in FSL – air, distilled water, 50% ethanol, and 0.02N citric acid and conditioned in individual glass beaker at 37°C for 7 days (dwell time) which simulate wet oral environment as recommended in previous studies.[2,6]

After conditioning, the specimens were air-dried and subjected to surface roughness test with contact stylus surface profilometer (Surfcom 130A) with a 2 μm probe diameter to evaluate surface roughness value (Ra). The specimens were then subjected to load of 500 gf with dwell time of 15 s to the central top surface of each specimen through an indenter to check hardness using digital microhardness tester Instron, Wilson instrument to attain Vickers hardness value. Statistical analysis was performed using software (SPSS version 20.0 Inc., Chicago Illinois, USA). Intercomparison between groups was done using two-way analysis of variance and comparison of hardness by Tukey's multiple *post hoc* at statistical significance value $P \leq 0.05$.

Table 1: Composition of composite restorative resin materials

Group/trade name	Composition	Characteristic	Shade	Lot no
A-Surefil SDR (Dentsply)	UDMA, di methacrylate resin, di functional diluents, barium and strontium alumina –fluoro-silicate glasses, 68%wt and 45% vol, photo initiators and colouring agents	Flowable composite (bulkfill)	Universal shade	11221
B-Clearfil majesty posterior (KurarayEurope)	BisGMA, TEGDMA, hydrophobicaromatic, dimethacrylate, camphorquinone, accelerators, pigment, others Fillers: silanated glass ceramic fillers1.5μm, surface treated alumina micro fillers 20nm.Filler load 92%wt,(82%vol)	Nanohybrid	A2	00122C
C-EverX, GC Corporation, Japan	Bis-GMA10-20%, TEGDMA-5-10%, Silicondioxide5-10%, barium glass -60-70%, glass fiber 5-15%, polymethylmethacrylate, photoinitiators	Fiber reinforced (bulkfill)	Universal shade	1307022
D-TetricEvo Ceram, Ivoclar, vivadent	Bis-MA, UDMA 19.7%wtBarium glass, ytterbium tri fluoride62.5%wt, mixed oxide and prepolymers19.7%wt.	Nano hybrid (bulkfill)	A2	S08629
E-Filtek Z350 3M (ESPE)	Bis -GMA, UDMA, TEGDMA, Bis EMA, discrete nonagglomerated and nonaggregated silica and zirconia fillers of 20 nm and 4-11 nm in size.	Nano composite	A2	N562394

Bis EMA-ethoxy bisphenol A diglycidyl dimethaacrylate; Bis-GMA- bisphenol ether dimethacrylate, TEGDMA-triethylene glycol dimethaacrylate; UDMA- urethane dimethacrylate.

Table 2: Mean , SD of Hardness and Surface Roughness score in five groups and four FSL solutions

Group	Surface Hardness								Surface Roughness (Ra)							
	Distilled water		Ethanol		Citric acid		Air		Distilled water		Ethanol		Citric acid		Air	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
A- SDR	28.35	1.1	29.67	1.37	29.1	1.36	25.89	1.37	0.0584	0.0064	0.0778	0.0071	0.0641	0.007	0.0452	0.0036
B- CFM	96.76	3.75	107.4	3.75	101.84	3.03	104.29	3.45	0.0359	0.0045	0.0306	0.0039	0.0375	0.0049	0.0373	0.0025
C -EX	58.83	4.93	58.39	2.82	56.68	4.08	61.17	3.64	0.0492	0.0049	0.0454	0.004	0.0455	0.0036	0.0357	0.0025
D-TEC	53.71	7.23	37.49	1.48	41.43	1.25	44.04	1.25	0.0402	0.0025	0.0547	0.0045	0.0511	0.0072	0.0378	0.0052
Z -350	71.46	0.66	74.18	2.7	74.18	2.7	72.92	1.41	0.0385	0.0078	0.0377	0.004	0.0463	0.0039	0.0314	0.0042

Scanning electron microscopy

Changes in surface topography after immersion in FSL were observed by SEM (EVO 40 Oxford instrument UK) with an integrated Carl Zeiss camera (Carl Zeiss SMT Ltd., Cambridge, UK) at a ×1000.

Results

The mean and standard deviation of VHT and Ra of five composites restorative materials are shown in Table 2. The hardness values were material dependent. Comparison of hardness values of materials showed that Clearfil Majesty (CFM) posterior and Z350 showed better results followed by Ever X and Tetric EvoCeram. Surefill SDR showed least hardness values [Table 2]. Comparison among FSL, none of the materials was affected by air. CFM showed reduction in hardness values when simulated with distilled water and was not affected by any other FSL. Tetric EvoCeram showed reduction in hardness values when immersed in ethanol and was not affected by any other FSL. Z350 and EverX were not affected by any of the FSL. Surface roughness and irregularities of material were again material dependent. CFM and Z350 showed least roughness followed by Ever X and Tetric EvoCeram. SureFil SDR showed maximum roughness values, and none of the materials were affected by air. Tetric Evo Ceram (TEC) and Z350 had influence on surface roughness after immersion in citric acid, and TEC and SDR were affected by ethanol solution. Significant difference was observed with respect to groups and FSL and surface hardness and surface irregularities.

Discussion

Clinical success of a composite restoration is mainly attributed to resistance to many factors changes in color, less roughness, good polishing, reduced wear, less shrinkage, and good optical properties. Despite improvements in materials, the longevity of composite restorations is the concern for all clinicians. The degradation of composite resins is a process that involves several factors such as wear, staining, absorption of liquids, inadequate finishing, and polishing. The failures of composites are mainly due to behavior of different resin matrix and type and percentage of filler in composite resins. The resin composites used in this study are based on Bis-GMA,UDMA, TEGDMA and Bis EMA and use ethoxylated bisphenol-A dimethacrylate (EBPADMA). The monomer is a hydrophobic analogue of Bis-GMA, with a relatively flexible structure, lower vinyl group concentration, and lower viscosity than Bis-GMA systems[13] [Table 1]. Peutzfeldt and Munksgaard *et al*. in their studies have shown various food and beverage constituents cause degradation, leaching of monomer components, filler dissolution, reduction of the hardness,[3] increased surface roughness,[14] and premature aging of composite restorations. Significant changes in hardness have also been reported during 1st week of exposure to FSL by Beyth *et al*.,[15] so a 7-day conditioning was selected in this study. In the present study, there was difference in surface microhardness and surface irregularities for the different composite resins after conditioning in the various FSLs. This finding could be attributed to the different chemical compositions of the tested composites [Table 1] along with the effect of the FSLs on the various chemical components.

Composite resin when placed in ethanol which depicts alcohol, released monomer in less time than if it were placed in water. Soderholm in their study discusses the mechanism of action of ethanol ion composites, the molecule diffuses into the composite resulting in microcracking which further promotes the infusion of ethanol which is retained in the monomeric matrix, causing increase in distance between the polymer chains, resulting in a soft matrix into the composite leading to greater damage.[16] In the present study, CFM showed greater resistance to surface degradation and higher hardness values, Surefil SDR showed least values [Figure 1Ea] after immersion in ethanol when compared to other groups. This could be because of hydrophobic monomer in CFM matrix. The reason for least value could be due to the higher amount of absorption of ethanol by the Bis-GMA molecule in SDR matrix causing swelling of the material. This dimensional change in the matrix causes stress at the matrix-silane-filler particle interfaces, resulting in degradation of this bond. In consequence, inorganic particles detach from the surface, causing an increase in roughness. Asmussen in their study concluded that the composites with matrix UDMA, Bis-EMA, and a little TEGDMA in its formulation showed decrease in hardness.[17] This is in agreement in our study, the inorganic phase composed of a zirconia filler could be the reason for greater hardness values and less surface

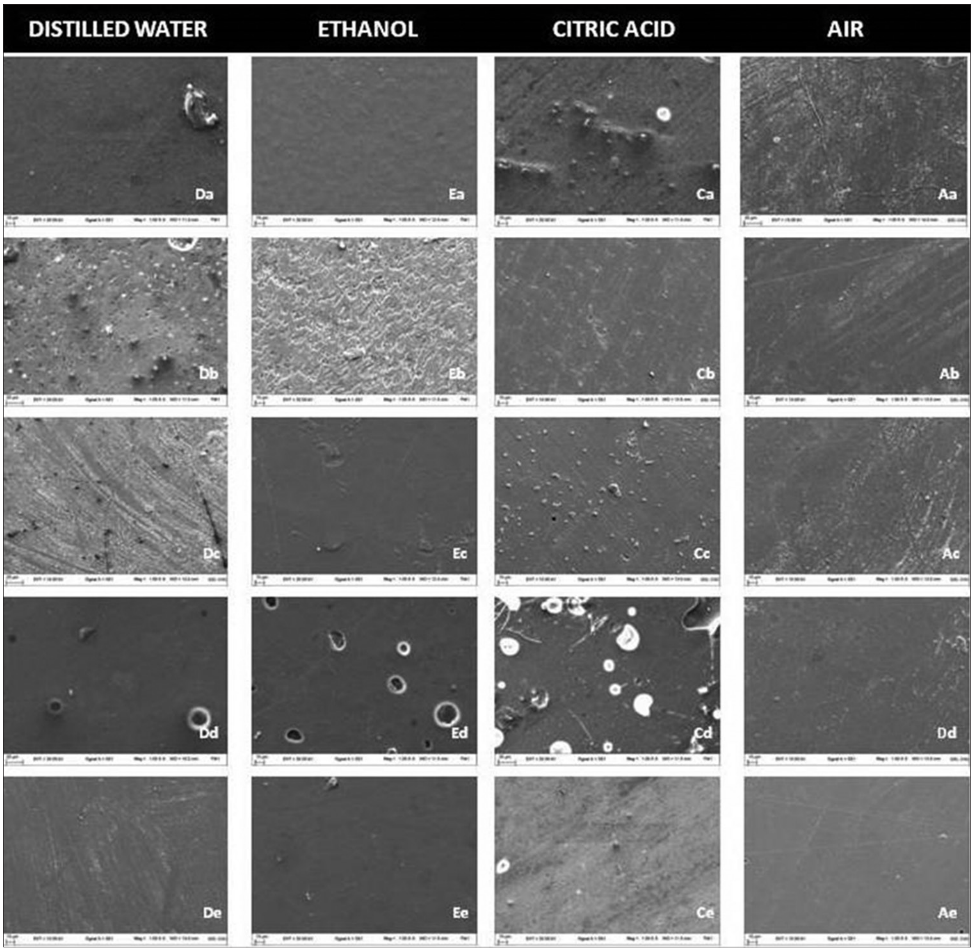

Figure 1: (Da-e, Ea-e, Ca-e and Aa-e) Scanning electronic microscopic images showing surface changes after immersion in different food simulating liquids

roughness of Z350 [Figure 1Ee] when compared to all the other materials tested.

Sideridou *et al*. have explained the degradation of composite surface by hydrolysis and release of filler particles because water uptake in the resin matrix. The absorbed water causes softening of the matrix, microcrack formation, resin degradation, and debonding of the filler-matrix interfaces.[8] The CFM according to manufacturer contains hydrophobic monomer EBPADMA, Bis EMA which reduces water sorption in hardened composites. Cattani-Lorente *et al*. in their study conclude that the TEGDMA content in resin matrix systems led to an increase in water uptake, as this monomer presents higher hydrophilicity when compared with Bis-GMA and UDMA.[18] This might be the reason for increased roughness and less hardness values with Z350 [Figure 1De]. Tetric EvoCeram [Figure 1Dd] and CFM [Figure 1b] both contain hydrophobic dimethacrylate resin which might be the reason for reduced surface roughness and greater hardness when compared to Z350, EverX [Figure 1Dc and De], and SDR [Figure 1Da]. This property of Tetric EvoCeram prevents composites to degrade and can be recommended for large cavities.

In the present study, citric acid was used to simulate acidic environment for composite resin materials. According to Ferracane and Marker[19] Prakki[20] *et al*., the process of chemical degradation has been associated with hydrolysis stimulated by oral conditions and reduced pH because of acids generated by microorganisms affecting the surface integrity by dissolution of the inorganic fillers of resin-based composites leading to erosive loss of material leading to surface roughness. In the present study, all composites in citric acid showed almost similar surface roughness changes. SEM evaluation showed pitted area filler debonding [Figure 1Ca-e]. This might be the reason for less hardness values with EverX when compared with other composites. In the present study, there is a significant difference in hardness between groups after immersion in citric acid solution.

Soderholm[16] and Yap *et al*.[21] in their study concluded that composites containing zinc, barium glass fillers, and zirconia glass fillers were shown to be more susceptible to aqueous attack than those containing quartz fillers, this is in agreement with our study as most composites used have barium glass in their filler composition. Therefore, differences in filler composition could be a possible reason for the decreased hardness values of Z350, TEC, and CFM in ethanol, water, and citric acid solution. Composites exposed to air and later measured for surface roughness and SEM [Figure 1Aa-e] did not show much significant difference; however, SDR and Z350 showed changes in hardness when compared to other groups.

Conclusions

Recent resin-based composite materials because of improvements in the composition have high rates of success and high patient acceptance. Composite restoration materials have been shown to behave differently when stored in different types of food-simulating solutions. In the present *in vitro* study, all materials showed changes in surface hardness and surface roughness after immersion in FSL. In this study, it can be concluded that the differences in hardness and surface roughness are mainly due to the different composition of resin matrix and different filler particles of all composite resin material tested.

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Conflicts of interest

There are no conflicts of interest.

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