

The Effect of Food-Simulating Liquids on Surface Features of Single-Shade Universal Composites: An *In Vitro* Study

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ABSTRACT **Objectives:** This study aimed to investigate the microhardness, surface roughness, and field emission scanning electron microscopes (FE-SEM) images of three different single-shade composites (Essentia Universal, Omnicroma, and Vittra APS Unique) in food simulation liquids such as ethanol, citric acid, and distilled water. **Materials and Methods:** Three single-shade universal composites were selected for this study. For each composite resin group, 92 samples (5-mm diameter and 2-mm depth) were prepared in plexiglass molds ($N = 276$). Then, samples were separated into four groups randomly consisting of 23 samples each (10 for hardness, 10 for roughness, and 3 for FE-SEM analysis). Three groups were immersed in food-simulating liquids (FSL)—citric acid (0.02N), distilled water, and 75% ethanol stored in a glass at 37°C for the next 7 days to simulate a wet oral environment. Control samples were stored in an opaque-light-proof box at room temperature. After the conditioning period, roughness and microhardness were measured, and FE-SEM analysis was performed. For statistical analysis, the two-way analysis of variance and Tukey honestly significant difference tests were used to evaluate roughness and microhardness ($P < 0.05$). **Results:** There was a statistically significant difference between the composites in terms of roughness and hardness averages ($P = 0.001$; $P < 0.05$). Omnicroma showed the most surface changes in ethanol storage, whereas Vittra Unique showed the most surface changes in citric acid storage such as Essentia. **Conclusion:** FSL that mimic various oral environments affect single-shade universal resin composite restorations.

KEYWORDS: Food-simulating liquids, microhardness, roughness

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INTRODUCTION

Composite resins used frequently in restorative dentistry have some advantages as well as disadvantages. Numerous composites are produced to overcome the disadvantages of composites, such as polymerization shrinkage, bond strength to different materials and tooth tissue, and difficulties in color match. Sometimes, the use of a multilayer composite can be time-consuming, and it prolongs the chair time of the treatment.^[1] Also, color selection is a relative criterion depending on the clinician and the environmental factors.^[2] In restorative dentistry, achieving a good

color match is very complex because many factors affect the color selection. A trend to make shade selection easier has led to the development of materials called universal composites. Single-shade composites have been introduced to eliminate the complex shade selection problem and enhance this blending effect's efficacy. These resin-based composites reduce the step

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of shade selection, as single modification is supposedly able to match almost any color by blending in with the surrounding tooth structure. One of the most noticeable but not the only representative of “single-shade” is Omnichroma (Tokuyama, Japan), which reflects light in the yellow-to-red wavelength spectrum mostly due to a finetuned filler system.^[3]

Single or group shades universal composites carry fewer hues than previous composite resins due to features known as color adjustment potential. Composite resins reduce color differences by interacting with enamel and dentin.^[4] This reduction in the shade of colors facilitates achieving nearly undetectable restorations with single shades.^[5] Among the universal composite resins, Essentia Universal and Vittra APS Unique are available in single shade and Omnichroma has only one regular shade with an opaquer. Universal composites can obtain to finish a restoration easily, polishability of these composites is quite good. However, the color stability of a few available universal composite resins has been reported to be not ideal.^[6] Much research has been done on the optical properties of universal composites; however, more research is needed on their mechanical properties and surface features after exposure to different media and over the time.^[7-13]

Matrices of resin-based composites are suspected to be affected by various food and drink ingredients and organic acids.^[14] Clinically, composite restorations are exposed continuously or temporarily to chemicals in different food and drinks.^[15] McKinney *et al.*^[16] has shown that food contents can significantly affect the surface hardness and surface roughness of composites. Therefore, it can be stated that the interactions between components of food, saliva, drinks, and these factors in the oral environment can deteriorate and age composite restorations. In literature, the effect of gastric acid on the universal composites was investigated.^[17] The effects of food-simulating liquids (FSL) on different dental materials have been investigated in some studies, and no published study has evaluated the effects of FSL on the single-shade composite resins.^[18-20] The aim of this study was to investigate the microhardness, surface roughness, and field emission scanning electron microscopes (FE-SEM) images of three different single-shade composite resins (Essentia Universal, Omnichroma, Vittra APS Unique) in food simulation liquids (ethanol, citric acid, and distilled water). The first null hypothesis of this study is that the FSL do not affect the surface hardness of single-shade universal composites. The second null hypothesis of this study

is that FSL do not affect the surface roughness of single-shade universal composites.

MATERIALS AND METHODS

Three single-shade universal composites were selected for this study [Table 1]. In the present study, only composite resins were used, no extracted teeth or any biological tissue; therefore, any application to ethics committee was not made. Sample size of the study was determined by the G*Power ver. 3.1.9.4 (Prof. Dr. Franz Faulstich, Uni Kiel, Germany) software with 95% power. Then, 276 composite samples (120 for microhardness, 120 for surface roughness, and 36 for FE-SEM analysis) (R = 5 mm; h = 2 mm) were prepared in plexiglass molds. Microsoft Excel Listbox method was used for the randomization of the composite samples. For each resin composite group, 92 samples were prepared, which were then divided into four groups consisting of 23 samples each. For example, for Omnichroma, 40 samples were prepared (10 samples for distilled water, 10 samples for ethanol, 10 samples for citric acid, and 10 for control group) for surface hardness, 40 samples for surface roughness, and 12 samples for SEM evaluation. For surface standardization, polyester strip is used and pressed together with the glass slab to minimize air bubbles and oxygen inhibition layer. The samples were polymerized for 40s with D- Light Pro (GC, Tokyo, Japan) according to the manufacturer’s instructions

Table 1: Chemical composition of the different composite restoratives

Material/Lot number	Type	Composition
Omnichroma (Tokuyama Dental, Tokyo, Japan) LOT: 010E69	Nano filled	Matrix: UDMA, TEGDMA. Fillers: 79% by weight uniform supranano spherical filler (SiO ₂ -ZrO ₂ 260 nm).
Essentia Universal (GC Corporation, Tokyo, Japan) LOT: 210526A	Micro hybrid	Matrix: UDMA, Bis-EMA, Bis-GMA, TEGDMA. Fillers: 65% by volume prepolymerized fillers, barium glass, silica
Vittra APS Unique (FGM, Joinville, Brazil) LOT: 021020	Nano filled	Matrix: Mixture of methacrylate monomers, UDMA, TEGDMA, photoinitiator compound (APS). Fillers: 72%–80% by weight, 52%–60% by volume boron-aluminum-silicate glass.

APS = advanced polymerization system, Bis-EMA = bisphenol A ethoxylated dimethacrylate, Bis-GMA = bisphenol A-glycidyl methacrylate, TEGDMA = triethylene glycol dimethacrylate, UDMA = urethane dimethacrylate

for each sample's bottom and top. Then, samples were randomly separated into four groups (control and three test groups). Three groups were immersed in FSL—citric acid (0.02N), distilled water, and 75% ethanol stored in a glass at 37°C for the next 7 days for aging the composite samples. Previous studies in the literature reported that the greatest change in the hardness of resin composites in aging procedure in FSL occurred within 7 days after the exposure.^[21] Therefore, the samples in this study were stored in FSL for 7 days before roughness and hardness tests. Control samples were stored in an opaque-light-proof box at room temperature [Figure 1].

SURFACE ROUGHNESS MEASUREMENT

After the storage period, the samples were washed with distilled water and air-dried, and to measure the surface roughness value (Ra), a 0.25-mm line scan was made across the surface of the sample using a profilometer (Surface SJ-301, Mitutoyo, Japan). The profilometer was calibrated with a cutoff of 0.25 mm, a reading length of 1.25 mm, and a velocity of 0.5 mm/s. Three measurements were obtained from three different locations from each sample, and the average was calculated.

VICKERS MICROHARDNESS MEASUREMENT

The Vickers microhardness tester (HMV-700 Microhardness Tester, Shimadzu, Japan) was used to

determine three different locations on each sample, and thus, the average Vickers hardness number (kg/mm²) was determined from these three measurements. A load of 500 gf was applied with a dwell time of 15 s.

FIELD EMISSION SCANNING ELECTRON MICROSCOPY ANALYSIS

For each composite group, three samples were prepared for FE-SEM analysis (N = 36). Three groups were immersed in FSL (citric acid, distilled water, and ethanol) and stored in a glass for the next 7 days, and control samples were stored in an opaque-light-proof box at room temperature for FE-SEM analysis. All samples were dried in a desiccator for 12 h and spray coated with gold/palladium in a vacuum coating device (LEICA ACE 200). After immersion in FSL, the entire surface of the universal composites was investigated under an FE-SEM; (HITACHI SU5000). Photomicrographs of the surface areas were taken at ×2500 magnification.

STATISTICAL ANALYSIS

While evaluating the findings obtained in the study, IBM SPSS Statistics 22 (IBM Corp., Chicago, USA) program was used for statistical analysis. The suitability of the parameters to the normal distribution was evaluated with the Kolmogorov–Smirnov and Shapiro–Wilk tests, and it was determined that the parameters were suitable for the normal distribution. Vickers hardness and surface roughness were evaluated with the two-way analysis

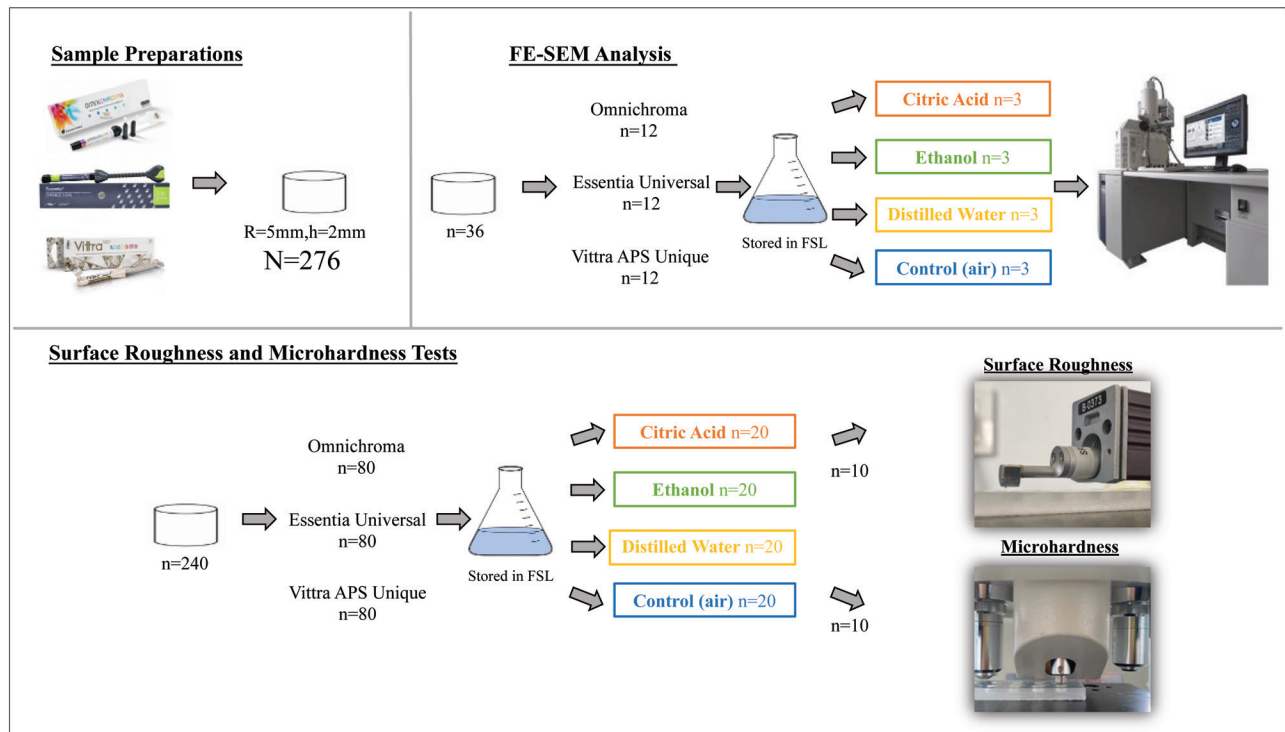


Figure 1: Flow chart of the experiments

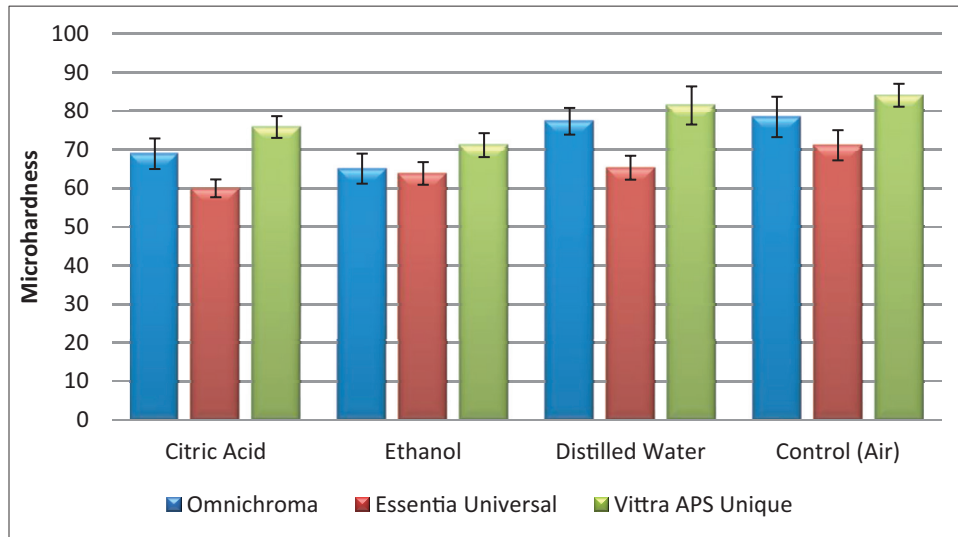


Figure 2: Vickers hardness numbers of universal composites in FSL. FSL = food-simulating liquids

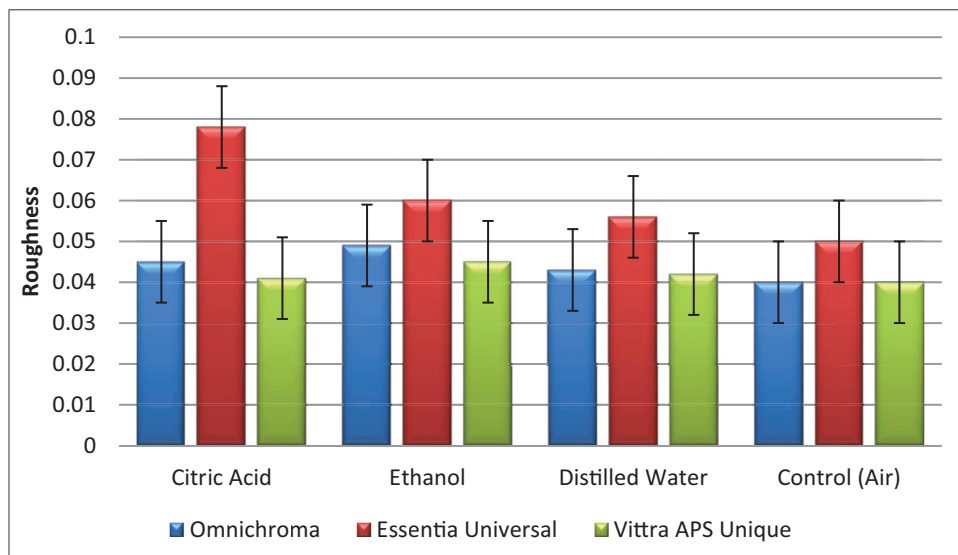


Figure 3: Surface roughness of universal composites in FSL. FSL = food-simulating liquids

of variance test, and the Tukey honestly significant difference test was used in post hoc analyses. Significance was evaluated at the $P < 0.05$ level.

RESULTS

There was a statistically significant difference in the average surface hardness of Omnichroma composite between different media groups ($P = 0.001$; $P < 0.05$) [Table 2]. According to the result of post hoc analyses, the mean hardness of the distilled water group was significantly higher than that of the citric acid and ethanol groups ($P < 0.05$) [Figure 2]. The mean hardness of the control group was found to be significantly higher than that of the citric acid and ethanol groups ($P < 0.05$). There was no significant difference between

the citric acid and ethanol groups in terms of hardness ($P > 0.05$). There was no significant difference between distilled water and control groups in terms of hardness ($P > 0.05$) [Table 2]. It was observed that the maximum decrease in surface hardness was caused by citric acid and ethanol. Thus, the foods containing citric acid can affect the surface hardness of Omnichroma composites.

There was a statistically significant difference in the average surface hardness of Essentia Universal composite between different media groups ($P = 0.001$; $P < 0.05$) [Table 2]. According to the result of post hoc analyses, the mean hardness of the citric acid group was significantly lower than the ethanol, distilled water, and control groups ($P < 0.05$). The mean hardness of the control group was significantly higher than that

Table 2: Vickers hardness numbers of composites in FSL

Food Simulating Liquids	Omnichroma	Essentia Universal	Vittra APS Unique	<i>P</i>
	Mean ± SD	Mean ± SD	Mean ± SD	
Citric acid	68.91 ± 3.96 ^{aa}	59.95 ± 2.31 ^{ba}	75.83 ± 2.82 ^{ca}	0.001*
Ethanol	67.05 ± 3.89 ^{aa}	63.81 ± 2.93 ^{ab}	72.14 ± 3.1 ^{bb}	0.001*
Distilled water	76.32 ± 3.46 ^{ab}	66.29 ± 3.1 ^{bb}	81.41 ± 4.94 ^{ac}	0.001*
Control (air)	77.44 ± 5.23 ^{ab}	75.1 ± 3.91 ^{bc}	82.06 ± 2.97 ^{cc}	0.001*
<i>P</i>	0.001*	0.001*	0.001*	

ANOVA = analysis of variance, APS = advanced polymerization system, FSL = food-simulating liquids, SD = standard deviation

Different lowercase letters in the lines indicate the difference between composite groups

Different capital letters in the columns indicate differences between media groups

**P* < 0.05; two-way ANOVA test

Table 3: Surface roughness of composites in FSL

Food Simulating Liquids	Omnichroma	Essentia Universal	Vittra APS Unique	<i>P</i>
	Mean ± SD	Mean ± SD	Mean ± SD	
Citric acid	0.045 ± 0.01 ^{aa}	0.078 ± 0.01 ^{ba}	0.041 ± 0.01 ^{aa}	0.001*
Ethanol	0.049 ± 0.01 ^{aa}	0.060 ± 0.01 ^{bb}	0.045 ± 0.01 ^{aa}	0.027*
Distilled water	0.043 ± 0.01 ^{aa}	0.056 ± 0.01 ^{bb}	0.042 ± 0.01 ^{aa}	0.047*
Control (air)	0.040 ± 0.01 ^{aa}	0.050 ± 0.01 ^{ab}	0.040 ± 0.01 ^{aa}	0.113
<i>P</i>	0.484	0.001*	0.813	

ANOVA = analysis of variance, APS = advanced polymerization system, FSL = food-simulating liquids, SD = standard deviation

Different lowercase letters in the lines indicate the difference between composite groups

Different capital letters in the columns indicate differences between media groups

**P* < 0.05; two-way ANOVA test

of the citric acid, ethanol, and distilled water groups (*P* < 0.05). There was no significant difference between distilled water and ethanol groups in terms of hardness (*P* > 0.05) [Table 2].

There was a statistically significant difference in the average surface hardness of Vittra APS Unique composite between different media groups (*P* = 0.001; *P* < 0.05) [Table 2]. As a result of post hoc analyses, the mean hardness of the ethanol group was significantly lower than that of the citric acid, distilled water, and control groups (*P* < 0.05). The mean hardness of the citric acid group was significantly lower than that of the distilled water and control groups (*P* < 0.05). There was no significant difference between distilled water and control groups in terms of hardness (*P* > 0.05) [Table 2]. The maximum decrease in surface hardness was caused by ethanol in this group.

When the surface roughness of Omnichroma composite was evaluated, there was no statistically significant difference between different media groups in terms of roughness averages (*P* = 0.484; *P* > 0.05) [Table 3]. When the surface roughness of Essentia Universal composite was evaluated, there was a statistically significant difference in roughness averages between different media groups (*P* = 0.001; *P* < 0.05) [Table 3]. As

a result of post hoc analyses, the mean roughness of the citric acid group was significantly higher than the ethanol, distilled water, and control groups (*P* < 0.05) [Figure 3]. There was no significant difference between the other groups in terms of roughness (*P* > 0.05) [Table 3].

When the surface roughness of Vittra APS Unique composite was evaluated, there was no statistically significant difference between the different media groups in terms of roughness averages (*P* = 0.813; *P* > 0.05) [Table 3].

DISCUSSION

In the present study, the null hypothesis was rejected. The FSL affected the surface hardness and roughness of these single-shade universal composites. The maximum effect of FSL on surface hardness was obtained in the citric acid and ethanol groups.

Despite advances in composite resins, the longevity of composite resin restorations is still a concern for dental clinicians because these materials are subject to wear, degradation, and staining after exposure to various foods and beverages in the oral cavity, which causes failure of restoration and requires replacement.^[18] As in the previous study, water, ethanol, and citric acid were used as FSL in this study. These liquids were selected

in accordance with the guidelines of the US Food and Drug Administration.^[22] In this study, water immersion simulates the wet oral environment provided by saliva; citric acid immersion simulates vegetables, fruits, and sugar; and ethanol is the solvent of choice to stimulate and age the dental restorations.^[18,23] In a previous study, it was reported that significant changes occurred in the hardness of composite resins in the first week of exposure to FSL.^[21] Therefore, in this study, specimens were stored in FSL for 7 days before evaluation.

FSL, which have softening and hydrolyzing effects on resin composites, may cause a decrease in the physicomechanical features of composites due to the deterioration of the polymer matrix.^[23,24] Kumari *et al.*^[18] found changes in surface roughness and surface hardness of composite resins after immersion in FSL similar to the present study. They reported that these changes are due to different filler particles and different compositions of resin matrix in all composite resin materials tested. In recent years, the effect of FSL also used on computer aided design/computer aided manufacturing (CAD/CAM) materials (Tetric computer aided design and Lava Ultimate CAD/CAM blocks).^[25] After storage for 15 days in FSL, the surface roughness and hardness were not significantly decreased but when they were compared with initial values, Lava Ultimate showed higher surface roughness and hardness values. In another study, the effect of FSL has also been used on CAD/CAM polymer composites. According to the results, FSL affected the mechanical and surface properties of carbon fiber-reinforced composite, glass fiber-reinforced composite (TRINIA; TR), and a reinforced polyether ether ketone.^[26] Regarding microhardness in this study, after 7 days of storage in water, aqueous ethanol solution, and citric acid, Essentia composite suffered more changes in the percentage of microhardness in all tested environments and lost its hardness properties in all solutions. This might be owing to the presence of highly soluble monomers in the resin composition, such as bisphenol A ethoxylated dimethacrylate (Bis-EMA), triethylene glycol dimethacrylate, urethane dimethacrylate, and bisphenol A-glycidyl methacrylate (Bis-GMA).^[27] Another reason for the low value might be increased solution absorption by Bis-GMA and Bis-EMA molecules, which are absent from the organic matrices of Omnichroma and Vittra but present in Essentia.^[18] In FE-SEM evaluation, the most surface changes of Essentia composite occurred in citric acid solution storage [Figure 4I]. The changes such as pores with different sizes occurred. These FE-SEM results suggest the surface hardness results of Essentia.

Also, Omnichroma showed the most surface changes in ethanol storage, whereas Vittra Unique showed the most surface changes in citric acid storage such as Essentia. Among the tested single-shade universal composites, Omnichroma showed the least surface change in all media. These dimensional changes in the organic matrix induce stress at the matrix-silane-filler particle interfaces, leading to the degradation of this bond. As a result, inorganic particles detach from the surface, leading to increased roughness.^[18] The shape and size of resin composite fillers determine the surface properties of restorations.^[28] This is because the filler particles leaving the surface can leave small or large defects, depending on the size.^[29,30] Tested single-shade composite materials in this study had different filler sizes; Omnichroma contains uniformly spaced and arranged 260-nm spherical particles [Figure 4A], Vittra Unique is a nanoparticulate composite with a charge composed of nanospheres with an average particle size of 200 nm, whereas Essentia is micro hybrid composed of filler particles ranging from 0.85 to 17 μm in diameter.

This explains why the increase in the surface roughness of Essentia compared with the control group was statistically significant in all tested environments, whereas this difference was not significant in Omnichroma and Vittra.

Tabatabaei *et al.*^[31] reported that the highest amount of monomer is released from composite samples immersed in ethanol solution, citric acid, and lactic acid, and the lowest amount of monomer is released from the samples immersed in distilled water. In this study, the hardness values of other composite resins immersed in ethanol, except Essentia, were significantly lower than those immersed in citric acid. The organic matrix of composite resins can be damaged by organic solutions such as ethanol and heptane solution, whereas fillers can be damaged by citric acid and water.^[19] In this study, citric acid might have caused more surface degradation as GC Essentia was a microhybrid [Figure 4].

It is important to note that the experiments in this study were performed *in vitro*, and thus, some limitations still exist for simulating oral cavity environmental challenges. However, important results were found for Omnichroma and Vittra regarding resistance to food-simulated liquids. Vittra has an advanced polymerization system (APS) for better performance, which has contributed to good results after immersion in food imitation liquids. This recent technology has been developed to increase the important properties of composite resins: guaranteeing a high degree of

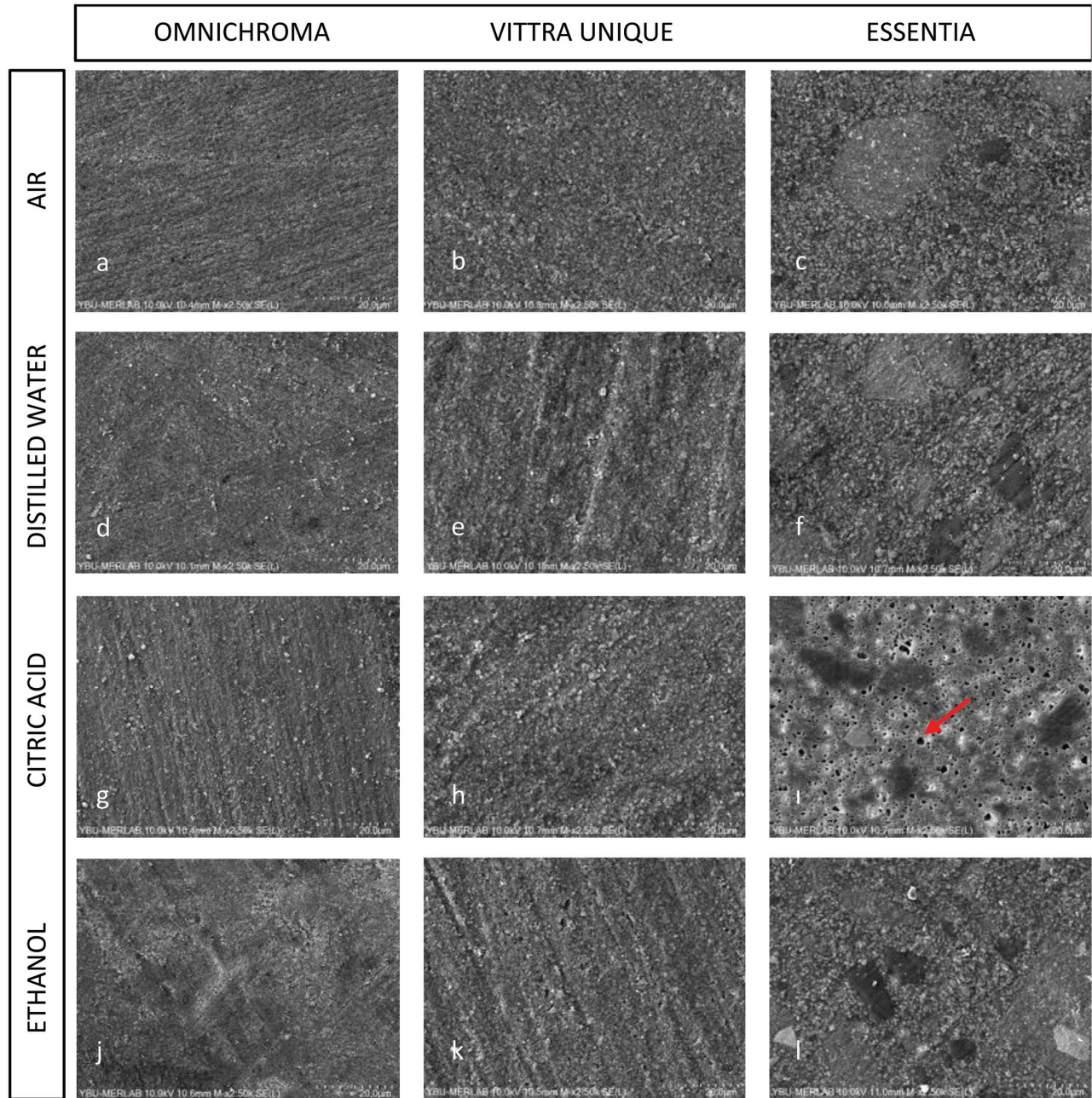


Figure 4: FE-SEM images of universal composites in FSL. (A–C) Composites' surface changes after stored in an opaque-light-proof box in air. (D–F) Composites' surface changes after immersion in distilled water. (G–I) Composites' surface changes after immersion in citric acid. (K–L) Composites surface changes after immersion in ethanol. FE-SEM = field emission scanning electron microscopes, FSL = food-simulating liquids

conversion, increasing bond strength, and providing a high-quality esthetic profile.^[32,33] Other points to consider are the absence of Bis-GMA in the organic matrix of Omnichroma and Vittra, and the nano-sized inorganic fillers of these composites. In addition, the high levels of chemical elements such as aluminum and silica in Vittra and zirconia in Omnichroma contributed to their better performance after immersion in FSL.

CONCLUSIONS

Omnichroma showed the most surface changes in ethanol media, whereas Vittra Unique showed the most surface changes in citric acid media such as Essentia. Among the tested single-shade universal composites, Omnichroma showed the least surface change in all media. As a result, it can be said that FSL affecting surface properties is an open area of research today. This may affect the properties of

the composite such as color and plaque uptake, and further studies are needed. For instance, long-term *in vitro* studies and *in vivo* studies can be planned to determine these or other effects of FSL on these composites.

Within the limitations of this study, all single-shade resin composites showed changes in surface roughness and hardness in FSL. It may be concluded that these changes are due to the different composition of the resin matrix and different filler particles of the tested single-shade composite resins.

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Nil.

CONFLICTS OF INTEREST

There are no conflicts of interest.

AUTHOR CONTRIBUTIONS

C. K.A.: conceived the research idea, interpreted the statistical results and wrote the article. B.A.D.: elaborated the article and performed the methodology of the article and H.A.: helped writing and performed the critical review of the article. All authors approved the final version of the article.

ETHICAL POLICY AND INSTITUTIONAL REVIEW BOARD STATEMENT

In the present study, ethics approval was not required.

PATIENT CONSENTS STATEMENTS

No references from previously published articles were used in the current study.

DATA AVAILABILITY STATEMENT

Not applicable.

PERMISSION TO REPRODUCE MATERIALS FROM OTHER SOURCES

No published material (including figures/diagrams, or short extracts, or content taken from websites) from previously published articles was used in the current study.

CLINICAL TRIAL REGISTRATION

The present study is an *in vitro* study, there is no need for a clinical trial registration number.

LIST OF ABBREVIATIONS

APS: advanced polymerization system
Bis-EMA: bisphenol A ethoxylated dimethacrylate
BisGMA: bisphenol A-glycidyl methacrylate

FDA: US Food and Drug Administration
FE-SEM: field emission scanning electron microscopes
FSL: food-simulating liquids
TEGDMA: triethylene glycol dimethacrylate
UDMA: urethane dimethacrylate

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