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Analysis of the Colour Change, Surface Roughness, and Hardness of Nanofiller Composite Resin after Exposure to *Syzygium Cumini* Extract Mouthwash

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ABSTRACT

Nanofiller composite resins exhibit fluid absorption, making them susceptible to chemical interactions with oral care agent such as mouthwash. Syzygium cumini (Jamblang) extract mouthwash, which contains acidic compounds and anthocyanins that may degrade the resin's polymer matrix. This degradation can potentially lead to discolouration, increased surface roughness, and reduced hardness. This study aimed to assess the impact of Jamblang extract mouthwash on the colour stability, surface roughness, and hardness of nanofiller composite resins. Thirty-two cylindrical specimens (5 mm × 2 mm) were randomly assigned to five groups: immersion in Jamblang extract mouthwash, 0.2% chlorhexidine, commercial herbal mouthwash, distilled water, and a control group with no immersion. Specimens underwent 12 immersion cycles per day at 37 °C for five days to simulate two months of twice-daily use. Colour changes were visually evaluated using the VITA Toothguide 3D-Master, with assessments performed by five trained observers under standardized conditions. This method, while practical, involves subjective evaluation. Surface roughness was analyzed using Atomic Force Microscopy (AFM), and hardness was measured with a Micro-Vickers hardness tester. Statistical analyses included the Kruskal-Wallis test, Dunn's post hoc test, and one-way ANOVA. The results showed no significant colour change or hardness reduction in the Jamblang group (p > 0.05), but a statistically significant higher increase in surface roughness compared to commercial herbal mouthwash (p = 0.011) and distilled water (p = 0.036). In conclusion, short-term exposure to Jamblang extract mouthwash is safe for maintaining the colour and hardness of nanofiller composite resin, although it may increase surface roughness.

Keywords: Colour change, Surface roughness, Hardness, Nanofiller composite resin, Syzygium cumini

Introduction

Composite resin is a restorative material commonly used in dentistry due to the favorable mechanical characteristics and natural appearance. Among different types, nanofiller composite resin is distinct due to the superior modulus, strength, and polishing ability. However, the material remains prone to discolouration, increased surface roughness, and a decline in hardness. These changes are largely influenced by the chemical integrity of the polymer matrix, as well as external factors including tobacco, food, beverages, and chlorhexidine-based mouthwash. However, the material remains prone to discolouration, increased surface roughness, and a decline in hardness.

Exposure to acidic or basic solutions can deteriorate the polymer matrix, causing filler particles to develop and form irregularities on the surface. ^{8,9} These surface defects facilitate plaque accumulation and stain absorption. Furthermore, hardness of the composite resin is crucial in ensuring the durability of restorations. ^{10,11} As reported by Fadelan et al. (2023), nanofiller composite resin had surface hardness of 112.73 VHN, showing the ability to withstand masticatory pressure. ¹²

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Mouthwash has been shown to affect the physical characteristics of composite resin. ^{13,14} The various active ingredients, such as alcohol, detergents, and acids, weaken the polymer structure, thereby altering surface roughness, colour stability, and hardness. ^{15,16} Baig et al. (2016) found that non-alcohol-based mouthwash caused greater discolouration compared to alcohol-based types. ¹⁴ Meanwhile, Permatasari and Islamiah (2024) showed that different mouthwash could influence the surface roughness of resin composite after prolonged exposure. ¹⁷ Other commercial products, such as Listerine, Oral-B, and chlorhexidine, have been associated with a reduction in surface hardness due to the low pH, fluoride content, or alcohol base. ¹⁸⁻²⁰ Lepri et al. (2014) further stated that immersion in chlorhexidine-containing mouthwash reduced hardness by 1.29 KHN compared to artificial saliva. ²¹

Syzygium cumini (Jamblang) has gained significant interest as a natural mouthwash ingredient due to the bioactive components, such as flavonoids and anthocyanins, which have antibacterial effects against Streptococcus mutans. ²²⁻²⁵ However, the pigment and acidic content may pose a risk to the surface integrity of composite resin. ²⁶ Therefore, this study aims to evaluate the effect of Syzygium cumini fruit juice mouthwash on colour stability, surface roughness, and hardness of nanofiller composite resin.

Materials and Methods

A laboratory experimental method was used with a pre-post test group design. The study was carried out in several places, namely at the Laboratory of the Faculty of Dentistry, the Pharmaceutical Research Laboratory of the Pharmacy Department, and the Materials Laboratory of the Physics Department (Faculty of Mathematics and Natural Sciences) of Syiah Kuala University. The specimens used were 32 nanofiller composite resins (Filtek Z350 XT, 3M ESPE) with colour A2 in cylindrical shape measuring 5 x 2 mm.

Specimen Preparation

Stainless steel molds with a diameter of 5 mm and a height of 2 mm were washed clean and dried. The entire surface of the mold was smeared with vaseline using a plastic instrument. The composite resin paste was taken using a plastic instrument and inserted into the mold using the bulk technique. Subsequently, the top surface of the mold containing composite resin was covered using a matrix strip and a glass slide. A 1 kg load was placed on the glass slide to press the mold. The glass slide and load were removed after being left for 5 minutes, then each specimen was cured with a D-mate Curing Light LED for 20 seconds at a distance of 1 mm and perpendicular to the surface of the composite resin. The specimen was left for 1 hour and removed from the mold, then clamped using tweezers and washed using soap with running water. Finally, the specimens were immersed for 24 hours in distilled water before use.²⁷

Preparation of Jamblang Fruit Juice Mouthwash

Jamblang fruit (Syzygium cumini) samples were collected on 15 March 2024 from Lamreh Village, Masjid Raya Sub-district, Aceh Besar District, Aceh Province, Indonesia (5°36'10.8"N 95°32'43.3"E), with the criteria for fresh and intact fruit. The plant material was taxonomically identified by Dr. Saida Rasnovi, S.Ŝi, M.Si, Department of Biology, Faculty of Mathematics and Natural Sciences, Universitas A voucher specimen (Voucher Sviah Kuala. 1248/UN11.F8.4/TA.00.03/2024) was prepared and deposited at the Herbarium of Laboratorium Biosistematika, Department of Biology, Faculty of Mathematics and Natural Sciences, Universitas Syiah Kuala for future reference. The flesh was separated from the seeds until the weight was 10 g. Subsequently, 50 mL of distilled water was added to produce juice using a blender. To produce mouthwash, 4 mL of Jamblang fruit juice, 10 mL of glycerin, 10 mL of propylene glycol, 0.2 mL of sodium saccharin, and 100 mL of distilled water were added. All these ingredients were mixed in one sterile container and closed tightly. 18 The pH and viscosity tests were carried out using a Smart pH meter Milwaukee 100 MW, and an Ostwald viscometer.

Preparation of Immersion Group

Four groups of immersion were prepared, namely Jamblang fruit juice mouthwash, 0.2% chlorhexidine, commercial herbal mouthwash, and distilled water. Each specimen was immersed in a plastic vial containing the immersion group solution with a volume of 10 ml for 2 minutes then transferred to distilled water (volume 10 ml) for 10 minutes with 12 immersion cycles per day. After completing the last cycle of the day, the specimens were rinsed using running water and left to remain in distilled water until the next day. All immersion cycles were carried out at 37°C for 5 days to simulate the use of mouthwash twice a day for 1 minute during 2 months.

Measurement of Colour Change

Colour change measurements were carried out before and after immersion with Jamblang fruit juice mouthwash, 0.2% chlorhexidine, commercial herbal mouthwash, and distilled water. The measurements were conducted using the shade guide (VITA Toothguide 3D MASTER), which consists of 26 tooth colours (Figure 1). Colour of the specimen in each immersion group was compared to the shade guide. Colour change observation started after immersion by brushing the specimen gently using an extra-soft bristled toothbrush and a 16-way sweep pattern, followed by washing with running water. Meanwhile, observation before immersion did not require brushing. Each specimen to be measured was initially dried, 28,29 then colour changes were observed by 5 observers at once under the same conditions on a blue background. 30 All observers were thoroughly informed about the purpose and methodology of the study and signed an informed consent form before participation. Before the observation process started, all individuals were subjected to the Ishihara colour vision test to screen for colour blindness, in accordance with ISO/TR 28642:2016.31 There were no additional exclusion criteria applied in this study.

Measurements were conducted in three stages for each specimen. In the first stage, the value of the specimen was determined, ranging from group zero to five, and in the second stage, the chroma score was assessed by the observer, who opened and spread the shade tabs to

facilitate the evaluation process. In the third stage, the hue was determined by observing whether the specimen appeared more yellowish or reddish in tone. A one-level colour change was recorded when a shift of one level occurred on the shade guide positioned close to each specimen. Any observable colour changes by the observers were then recorded.

Measurement of Surface Roughness

The surface roughness of the specimens was measured twice, before and after immersion with Jamblang fruit juice mouthwash, 0.2% chlorhexidine, commercial herbal mouthwash, and distilled water. All specimens were measured using Atomic Force Microscopy (AFM, Nano Surft EasyScan 2). The specimens were placed on the tool, ensuring the tip was on the surface, then the device and computer screen were turned on. The tip or needle of the cantilever would move back and forth along the specimen surface during the scan. The measurement results would be shown on the computer screen.³²

Measurement of Hardness

The surface hardness measurement of nanofiller composite resin immersed in Jamblang fruit juice mouthwash, 0.2% chlorhexidine mouthwash, herbal mouthwash, distilled water, and without immersion treatment was carried out using a Micro-Vickers Hardness Tester (Shimadzu Microhardness Tester Type – M). This measurement was conducted at three points with a pressure of 100 grams within 15 seconds on the top surface of one side of the specimen. The results were obtained by calculating the marks formed after being pressed using an indenter.

Statistical Analysis

Data analysis was carried out using the Statistical Package for the Social Sciences (SPSS) version 26.0. The measurement of colour change was analyzed using descriptive statistics. Surface roughness data were analyzed using the Kruskal–Wallis test to identify significant differences among treatment groups, followed by Dunn's post hoc test for pairwise comparisons. Meanwhile, hardness measurements were analyzed using one-way analysis of variance (ANOVA).

Results and Discussion

Qualitative Analysis of Colour Changes and Surface Roughness Description of Nanofiller Composite Resin

Table I shows qualitative data on colour change of nanofiller composite resin specimens before and after immersion in various mouthwash solutions. Specimens immersed in Jamblang fruit juice mouthwash, 0.2% chlorhexidine, and distilled water showed no visible colour change, as indicated by consistent shade guide values (2M2) before and after immersion. In contrast, specimens exposed to commercial herbal mouthwash had perceptible colour changes, particularly 7 and 9, with a shift from 2M2 to 2R1.5. This shows a change in the hue group, suggesting that these specimens developed a more reddish-yellow appearance compared to others.

The results suggest that, among the tested groups, only the commercial herbal mouthwash caused significant discolouration in the composite resin, potentially due to the pigment content or chemical composition. The consistent colour stability observed in the other groups implied that Jamblang fruit juice, chlorhexidine, and distilled water did not significantly alter the aesthetic appearance of the composite resin under the test conditions.

Changes in surface roughness on the specimen were observed qualitatively through a three-dimensional topographic image (Figure 2). Dark areas on the topographic image marked with yellow circles show deep, uneven surface on the specimen. The presence of additional dark areas on specimens 1 (Figure 2 (A)), 6 (Figure 2 (B)), and 11 (Figure 2 (D)) after immersion in the group solution shows a deep and uneven surface, indicating an increase in roughness value.



Figure 1: VITA Toothguide 3D-MASTER shade guide used for colour assessment of composite resin specimens. The numerical scale (1–5) designates lightness values, ranging from the lightest (1) to the darkest (5), while the alphabetical notation specifies hue orientation: L (yellowish), M (neutral), and R (reddish).

However, specimen 9 (Figure 2 (C)) shows a reduction in the dark area, which suggests a decrease in the surface roughness value. The specimen showed a more homogeneous surface after immersion in commercial herbal mouthwash.

Based on the results, only the commercial herbal mouthwash induced a significant colour alteration in the nanofiller composite resin. This effect was observed through visible colour changes in resin specimens immersed in the solution, with two out of three showing a hue shift toward a more reddish tone, specifically to 2R1.5 (Table 1). Although the pH of this mouthwash was 4, similar to the pH 3.9 of the Jamblang fruit juice, the discolouration was more evident. This is presumably attributed to the presence of FD&C Red 40, a synthetic dye that imparts a vivid orange colour to mouthwash solution.

Table 1: Qualitative data on colour change scores in specimens

Immonsion Cuoung	No specimen	Colour Change		
Immersion Groups	No. specimen	Before	After	
Jamblang fruit juice	1	2M2	2M2	
mouthwash	2	2M2	2M2	
	3	2M2	2M2	
Chlorhexidine 0.2%	4	2M2	2M2	
	5	2M2	2M2	
	6	2M2	2M2	
Commercial herbal	7	2M2	2R1.5	
mouthwash	8	2M2	2M2	
	9	2M2	2R1.5	
Distilled water	10	2M2	2M2	
	11	2M2	2M2	
	12	2M2	2M2	

Explanation: 2M2, 2R1.5, and similar codes represent tooth shade designations according to the VITA Classical Shade Guide. The numerical scale (1–5) indicates lightness, ranging from the lightest (1) to the darkest (5), while the alphabetical notation specifies hue orientation: L = yellowish, M = neutral, and R = reddish.

The dominant colouring agent can enter the interface and cause more visible colour changes in the composite resin. The results are in line

with Assaf et al., (2020), who reported that coffee with a higher pH could provide greater colour changes than tomato sauce with a lower pH. It can be concluded that acidic foods or drinks have the potential to affect the integrity of composite resin matrix, but the pigments contained have a more significant influence on colour changes than the acidity level.²⁹

Regarding intrinsic factors, the organic matrix in nanofiller composite resin consists of BisEMA, UDMA, and TEGDMA monomers, which have hydrophilic properties originating from ether linkage (O-O), namely BisEMA and TEGDMA, as well as hydrogen bonds (N-H), namely UDMA.³⁴ Susceptibility of composite resin to colour change is attributed to the water absorption and hydrophilicity of the composite resin matrix. By absorbing water, the composite resin also captures other liquids, which cause colour changes. In general, FD & C Red 40 or Allura Red is a pigment often used in the food, beverage, and pharmaceutical industries.³⁵ This pigment can cause colour changes in composite resin due to the susceptibility to water. It is absorbed by ether linkage groups and hydrogen bonds, causing colour changes in the composite resin.³⁶

Figure 2 (C) shows the presence of darker areas on the specimen before immersion in the commercial herbal mouthwash. This shows that the surface of the specimen had a higher roughness value before immersion compared to those from the Jamblang fruit juice, chlorhexidine 0.2% and distilled water mouthwash groups. The rough surface before immersion will facilitate easy attachment of the pigment from mouthwash to the nanofiller composite resin.

Specimens immersed in Jamblang fruit juice mouthwash, 0.2% chlorhexidine, and distilled water did not change colour. The Jamblang fruit juice mouthwash used contains a pigment in the form of anthocyanin, which presumably provides colour to the specimen.³⁷ The absence of colour changes is probably related to the anthocyanin levels. The Jamblang fruit juice mouthwash only consisted of 4 mL of fruit juice per 100 mL, producing a clear appearance with a pink colour. This level of colour density is significantly different compared to commercial herbal mouthwash, which causes colour changes in the specimen. Meanwhile, changes in roughness values were observed through topographic images in three dimensions (3D) and from above (top view) in this group of specimens.

Figure 2(A) shows the surface topography of the nanofiller composite resin with specimen number 1 before and after immersion in Jamblang fruit juice mouthwash. The large number of dark areas visible after immersion shows a deeper and rougher surface. The rougher surface facilitates easy attachment for the pigment, thereby causing colour change with prolonged exposure to Jamblang fruit juice mouthwash, even at a low concentration.

In the group immersed in 0.2% chlorhexidine, all specimens showed no perceptible colour changes, as also reported in previous studies. ElEmbaby (2014) reported that immersion of composite resins in chlorhexidine solutions for 24 hours, simulating two years of clinical use, did not cause significant colour alterations. The study concluded that potential discolouration of composite resins due to chlorhexidine exposure is primarily associated with extrinsic factors, such as dietary chromogens, rather than the intrinsic properties of mouthwash.³⁸ A systematic review by Morais Sampaio et al., (2020) reported 5 studies with clinically unobservable colour changes after immersion in mouthwash. This was related to various factors of the composite resin in the human oral cavity. Different factors can influence colour stability of restorative materials in clinical settings, including the presence of saliva, salivary films, as well as the effects of different foods and beverages difficult to simulate in an in vitro environment.^{2,16} Based on the topographic visualization in Figure 2(B), an increase in surface roughness is shown by the presence of darker areas and a more irregular surface morphology compared to the pre-immersion specimen. These changes may be associated with the acidic nature of mouthwash, which can enhance the degradation of the composite resin matrix and contribute to a rougher surface profile.

In the distilled water immersion group, the specimens did not show any colour change in the nanofiller composite resin.



Figure 2: Three-dimensional atomic force microscopy (AFM) images illustrating the surface morphology of nanofiller composite resin specimens before and after immersion in different mouthwash solutions: (A) *Syzygium cumini* (Jamblang) fruit juice mouthwash, (B) 0.2% chlorhexidine mouthwash, (C) commercial herbal mouthwash, and (D) distilled water. The left column (a) displays the surface topography prior to immersion, while the right column (b) presents the post-immersion condition. Yellow circles denote regions with increased or decreased surface roughness. Scale bars are located on the right side of each image. Surface irregularities are visually represented by darker areas, which correspond to deeper or rougher surface features.

Distilled water has a normal pH of 7 and does not contain pigment susceptible to colour changes. However, from the topography (Figure 2D), the specimen has additional dark areas showing a rough surface. A rough surface will affect the specimen ability to absorb and reflect light. Therefore, prolonged exposure will affect colour appearance of the specimen.

The duration of immersion influences the occurrence of colour changes in the nanofiller composite resin. In this study, immersion was carried out for 5 days to simulate the use of mouthwash for 2 months with use 2 times a day for 1 minute each time. The relatively short time has the potential to be one of the causes of no visible colour change.

Quantitative Analysis of Changes in Surface Roughness of Nanofiller Composite Resins

Table 2 shows that the average surface roughness value of the nanofiller composite resin changed after immersion in Jamblang fruit juice, 0.2% chlorhexidine, commercial herbal mouthwash, and distilled water for 5 days with 60 immersion cycles. The Kruskal-Wallis test showed a statistically significant difference in baseline surface roughness among the groups (p < 0.05). Following immersion, the group exposed to Jamblang fruit juice mouthwash showed a significant increase in surface roughness, from $0.0125 \pm 0.008~\mu m$ to $0.393 \pm 0.195~\mu m$.

Table 2: Result of changes in surface roughness

Immersion Groups Mo		Surface Roughness (Ra) in micrometer (µm)							
	Before (x)			After (y)			y-x	x.y	
	Mean	SD	pValue ^a	Mean	SD	pValue ^a	(ΔRa)	Pvalue ^b	pValue ^c
Jamblang fruit juice mouthwash	0.0125	0.008	0.031*	0.393	0.195		0.381	0.109	0.499
Chlorhexidine 0.2%	0.020	0.002		0.250	0.131	0.489	0.230	0.109	
Commercial herbal mouthwash	0.293	0.158		0.257	0.071		-0.0367	0.593	
Distilled water	0.147	0.023		0.185	0.173		0.0383	0.593	

^{*}significant difference (p < 0.05)

Although the Wilcoxon Signed Ranks test did not show a statistically significant difference post-treatment (p > 0.05), the magnitude of the change ($\Delta Ra=0.381~\mu m)$ was the highest among all groups.

In comparison, the 0.2% chlorhexidine group showed an increase in surface roughness from $0.020\pm0.002~\mu m$ to $0.250\pm0.131~\mu m$ ($\Delta Ra=0.230~\mu m$). The group immersed in the commercial herbal mouthwash had a slight decrease in roughness ($\Delta Ra=-0.0367~\mu m$), while the distilled water group showed a minimal increase ($\Delta Ra=0.0383~\mu m$). However, one-way ANOVA analysis showed no statistically significant difference in surface roughness changes among the four groups (p > 0.05).

The Dunn test results (Table 3) showed that exposure to Jamblang fruit juice mouthwash caused a significant change in the surface roughness of nanofiller composite resin compared to the commercial herbal mouthwash (p = 0.011) and distilled water (p = 0.036). However, there was no significant difference between Jamblang mouthwash and 0.2% chlorhexidine (p>0.05), showing that both have a comparable effect. Chlorhexidine also showed a significant difference when compared to the commercial herbal mouthwash (p<0.05), but not with distilled water (p>0.05). Meanwhile, no significant difference was found between the commercial herbal mouthwash and distilled water (p>0.05), suggesting a minimal effect on surface roughness.

The results suggest that the active compounds in Jamblang fruit juice may increase the surface roughness of resin, similar to the effects observed with chlorhexidine. This is a critical consideration, as increased roughness may affect the esthetics, plaque retention, and long-term durability of composite resin restorations. Although Jamblang fruit juice has recognized antimicrobial benefits, the potential impact on restorative materials should be carefully considered when used as mouthwash.

Exposure to Jamblang fruit juice mouthwash, 0.2% chlorhexidine, commercial herbal mouthwash, and distilled water led to variations in the surface roughness (Ra values) of the nanofiller composite resin. Although these changes were not statistically significant, the observed alterations show that immersion in different solutions can affect the surface characteristics of the material.

AFM test results showed that the specimens immersed in Jamblang fruit juice mouthwash experienced the highest increase in roughness values compared to the other groups, although statistically the change was not significant (Table 2). The Ra value of the specimen after immersion showed an average value of 0.393. This value has exceeded the roughness limit in the oral cavity, namely 0.2 μ m, potentially facilitating the attachment of plaque and pigment from food or drink in the oral cavity. However, in vivo, the existence of a buffer system in the

oral cavity caused by salivary factors can neutralize the pH of mouthwash to minimize the increase in surface roughness.^{2,39}

Continuous exposure to Jamblang fruit juice mouthwash containing flavonoids, tannins, several acid constituents such as ascorbic acid and malic acid, propylene glycol, and other acidic substances led to changes in the rougher surface. Acids generally function as an oxidizing agent and catalyst in the hydrolysis process. Damage to the hydrolysis reaction between resin matrix bond and the filler will cause water to enter the surface of the filler, leading to a reduction in cohesion of the material. Proken bonds cause material degradation through shortening of the polymer bond chain, which turns into oligomers and then monomers. Resin matrix and filler particles released from the surface have the potential to form holes and filler protrusions, causing the composite resin surface to become uneven, thereby increasing the surface roughness.

AFM test results showed a change in the roughness value of specimen immersed using 0.2% chlorhexidine, which was indicated by an increase in the average Ra value. This is presumably because mouthwash also has a low pH, resulting in a rougher specimen surface. Composite resin exposed to acidic solutions will cause roughness on the surface due to degradation of the filler components caused by acid particles, resulting in decreased physical properties and strength.³

In specimens immersed in commercial herbal mouthwash, the Ra value before immersion was relatively high on average compared to those in other immersion groups. Commercial herbal mouthwash has a fairly low pH of 4 with several acidic contents such as citric acid and other substances. After immersion, there was a decrease in the average Ra value in the specimens. This shows different results from other groups, which experienced an increase in the average Ra value. Similarly, Evgeny et al. (2016) showed that initially smooth solid surface may undergo surface abrasion when exposed to acidic solutions, due to chemical interactions between surface compounds and hydrogen ions (H⁺). On rougher surface, such exposure can lead to a reduction in surface irregularities. Immersion in 4 M hydrochloric acid (HCl) led to a decrease in surface roughness (Ra) values, suggesting that elevated peaks and cavities were gradually dissolved, resulting in a smoother surface profile. 42

Figure 3 shows changes in surface roughness observed in specimens immersed in commercial herbal mouthwash, suggesting a reduction in Ra values. These results suggest that exposure to acidic or low pH solutions can compromise the integrity of resin surface, regardless of whether the initial surface is smooth or rough. The alteration in roughness, either an increase or a decrease, is primarily caused by the breakdown or release of certain polymer fractions at the surface, resulting from chemical reactions between the liquid and resin.

a. Kruskal Wallis test

b. wilcoxon Signed Ranks test

c. One way ANOVA test

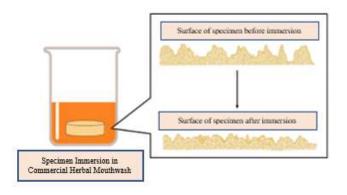


Figure 3: The surface of the specimen becomes smooth after immersion in commercial herbal mouthwash

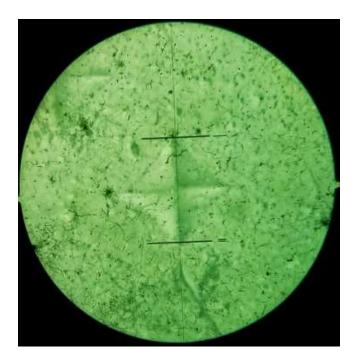


Figure 4: Surface scars results of specimen with the Indenter Micro-Vickers Hardness Tester

Although different immersion groups showed varying patterns, such as increased roughness in Jamblang fruit juice, 0.2% chlorhexidine, and distilled water, and decreased roughness in commercial herbal mouthwash, the underlying mechanism remains the same, comprising chemical interaction at the polymer surface.

In specimens immersed in distilled water, an increase in Ra values was also recorded. This may be attributed to prolonged water exposure, which can lead to the degradation of the silane coupling agent at the interface between resin matrix and filler particles, facilitating water absorption. 43 Due to the hydrophilic nature of composite resin, water absorption potentially reduces physical and mechanical properties.⁴⁴ Camilotti et al. (2021) reported that an increase in surface roughness was caused by hydrolytic degradation, where water penetrates the composite resin, accumulates at the matrix and filler interface, degrades filler particles, and weakens the bond between the matrix and fillers. 45 The results showed no significant difference in the surface roughness value of the nanofiller composite resin immersed in Jamblang fruit juice mouthwash, 0.2% chlorhexidine, commercial herbal mouthwash, and distilled water. This is presumably due to differences in initial surface roughness values before immersion. The initial roughness value of the Jamblang fruit juice mouthwash group specimens was lower compared to that of the commercial group. Therefore, in the commercial herbal

mouthwash group, the roughness value decreased because the initial value was higher. Jamblang fruit juice mouthwash experienced an increase in surface roughness value, above the normal limits accepted by the oral cavity ($\leq 0.2~\mu m$). The pattern of increasing roughness values between Jamblang fruit juice mouthwash and distilled water was similar. However, the presence of an acidic pH factor in Jamblang fruit juice increased the roughness value compared to distilled water, although within the acceptable range of the oral cavity.

Quantitative Analysis of Nanofiller Composite Resins Hardness

The surface hardness of the nanofiller composite resin was assessed by calculating the size of the scars from the indenter formed on the surface of the specimen, as shown in Figure 4. Based on the data in Table 4, the mean hardness values after immersion in various solutions showed no statistically significant differences (p>0.05). The group immersed in Jamblang fruit juice mouthwash had the highest mean hardness (112.42 \pm 28.47 VHN), followed by 0.2% chlorhexidine (109.58 \pm 47.52 VHN), commercial herbal mouthwash (107.17 \pm 20.98 VHN), and distilled water (105.37 \pm 30.46 VHN). However, these differences were not statistically significant according to ANOVA (p > 0.05), showing that immersion in the solutions did not cause marked changes in resin hardness.

The results suggest that despite minor variations, the internal structure of the composite resin remained relatively stable after exposure, including to acidic solutions such as Jamblang fruit juice. The stability implies that nanofiller composite resin possesses sufficient mechanical resistance to short-term degradation in hardness caused by exposure to different types of mouthwash, including those with acidic or bioactive components.

The clinical relevance of this study is grounded in the identification of how different types of mouthwash, particularly those with acidic and pigmented content, can influence the surface characteristics of nanofiller composite resin. Although Jamblang fruit juice mouthwash did not cause visible discolouration, it significantly increased surface roughness beyond the clinically acceptable threshold of 0.2 μm . This elevation in roughness could enhance plaque accumulation, pigment retention, and bacterial adhesion, which may compromise the long-term esthetics and hygiene of resin-based restorations in actual clinical settings. Therefore, careful selection and formulation of herbal mouthwash is essential when used by patients with composite restorations.

Surface hardness of resin remained relatively stable after exposure to various mouthwash types, including those with low pH and moderate viscosity. This suggests that short-term use of the products may not immediately affect the material mechanical resistance to chewing forces. However, due to the in vitro nature and limited immersion duration, these results should be interpreted with caution. For more definitive clinical recommendations, further in vivo studies that simulate the dynamic conditions of the oral cavity over longer periods are needed.

The result is clinically relevant, as surface hardness correlates with the wear resistance and durability of restorative materials. Although the low pH and moderate viscosity of Jamblang mouthwash may affect surface roughness, these factors do not appear to significantly alter the material hardness, suggesting a limited impact on the internal mechanical properties.

Before immersion, the pH of Jamblang fruit juice mouthwash, chlorhexidine 0.2% and herbal mouthwash was measured using a pH meter, and the results are shown in Table 5. The highest pH was obtained from 0.2% Chlorhexidine (4.7), and the pH was found in Jamblang fruit mouthwash (3.9).

There were significant differences in the pH and viscosity values among the immersion solution groups, as confirmed by ANOVA with highly significant p-values (p < 0.01). The Jamblang fruit juice mouthwash had the lowest pH value, at $3.9\pm0.15,$ which was significantly different from the other groups (p < 0.01). This shows that the solution is more acidic compared to 0.2% chlorhexidine (pH 4.7), the commercial herbal mouthwash (pH 4.0), and neutral distilled water (pH 7.0).

Table 3: Pairwise comparisons results of dunn's test for surface roughness change

Immersion Groups	Comparison Groups					
	Jamblang fruit juice	Chlorhexidine	Commercial herbal	Distilled water		
	mouthwash	0.2%	mouthwash			
Jamblang fruit juice		0.571	0.011*	0.036*		
mouthwash						
Chlorhexidine 0.2%	0.571		0.047*	0.126		
Commercial herbal	0.011*	0.047*		0.650		
mouthwash						
Distilled water	0.036*	0.126	0.650			

^{*}significant difference (p < 0.05), Anova

Table 4: Average hardness value of nanofiller composite resin

Immersion Groups	Hardness Valu	pValue	
	Mean	SD	
Jamblang fruit juice	112.42	28.47	
mouthwash			
Chlorhexidine 0.2%	109.58	47.52	0.994
Commercial herbal	107.17	20.98	0.994
mouthwash			
Distilled water	105.37	30.46	

^{*}significant difference (p < 0.05), Anova

The high acidity may contribute to surface degradation of the composite resin through erosion of resin matrix and leaching of filler particles, in line with previous studies, which recorded an increase in surface roughness following exposure to the Jamblang mouthwash.

The Jamblang fruit juice mouthwash had a viscosity of 1.395±0.013, significantly higher than chlorhexidine 0.2% (0.960±0.015), and distilled water (0.818±0.008), but lower than the commercial herbal mouthwash (1.526±0.012) (P<0.01). Viscosity influences the contact time of the solution on resin surface. Higher viscosity can prolong exposure, enhancing the interaction of active compounds such as organic acids with the material. This effect, combined with the acidic nature of the solution, contributes to alterations in the surface roughness and hardness of the nanofiller composite resin.

The results support the hypothesis that the physicochemical properties of immersion solutions, specifically pH and viscosity, play a crucial role in determining the dimensional stability and surface properties of nanofiller composite resin. The acidic nature and moderate viscosity of Jamblang fruit juice mouthwash raise important considerations for application as an active ingredient in formulations, particularly for patients with resin-based restorations.

Surface hardness measurements were carried out using a micro-Vickers hardness tester, which aims to determine the resistance of the nanofiller composite resin to chewing pressure on the surface. ⁴⁶ The surface hardness value of restoration materials, both enamel and dentin, ranges from 50-370 VHN. ⁴⁷ Fadelan et al. (2023) measured hardness value of the Z350 XT (3M ESPE), which is the gold standard, using a micro-Vickers hardness tester (Shimadzu, Japan) and obtained an average value of 112.73 VHN. ¹² The study by Puspita et al. (2020) reinforced previous results by reporting that the nanofiller composite resin Z350 XT (3M ESPE) showed hardness value of 115.2 VHN, followed by silk fiber-reinforced composite at 109.4 VHN, and the lowest value was observed in the unfilled composite at 32.2 VHN. ⁴⁸ Meanwhile, in this study, the mean hardness value of the Z350 XT (3M ESPE) nanofiller composite resin was 106.74 VHN.

The difference in hardness values can be influenced by several factors, such as light intensity, thickness of restoration material, light cure distance, and exposure time, which affect the polymerization of

composite resin.⁴⁹ This study used a light intensity of 1200 mW/cm², a thickness of 2 mm nanofiller composite resin restoration material, a light cure within a distance of 1 mm, and an exposure time of around 20 seconds, in line with the gold standard for optimal polymerization for composite resins. However, the lower surface hardness of the specimens is presumably due to the use of glass objects when curing with light cure. This causes light dispersion due to the presence of glass between the tip and the specimen. In addition, the condition of the specimen in a non-moist condition can affect the molecular mobility of the polymer chain of the unpolymerized composite resin. Unpolymerized monomers will form cross-links when moist, thereby increasing the mechanical properties of the nanofiller composite resin.⁵⁰ Nanofiller composite resin immersed in mouthwash and distilled water showed an increase in surface hardness values compared to specimens without immersion treatment. This is presumably because immersion at 37°C affects the molecular mobility of the polymer chains. Some monomers do not polymerize during curing but are mobilized to form more cross-links, thereby increasing the mechanical properties of the composite resin.

Immersion in various types of mouthwash produced nanofiller composite resin with variations in surface hardness, but there were no significant differences (p>0.05). Nanofiller composite resin immersed in Jamblang fruit juice mouthwash produced the highest surface hardness value. This is presumably due to the high viscosity of Jamblang fruit juice mouthwash (1.39) containing the glycerin component. Given that the viscosity value of glycerin is higher than water, 42 the diffusion rate for Jamblang fruit juice mouthwash by the nanofiller composite resin polymer matrix becomes slow and the quantity is minimal. This prevents a decrease in hardness of the nanofiller composite resin. However, commercial herbal mouthwash with the highest viscosity (1.53) produced lower hardness values than Jamblang fruit juice. This can be attributed to the presence of other chemical substances.

The commercial herbal mouthwash contains sorbitol, Poloxamer 407, peg-40 hydrogenated castor oil, Citric Acid, and other active ingredients, namely solvents, cleaners, and acids. 51 Mouthwash solvents cause degradation and erosion of composite resin materials, softening of polymers, and a decrease in mechanical properties.

Table 5: Average pH and viscosity values of immersion solution

Immersion Groups		pН			Viscosity	
	Mean	SD	pValue	Mean	SD	pValue
Jamblang fruit juice mouthwash	3.9	0.15	0.000**	1.395	0.013	0.000**
Chlorhexidine 0.2%	4.7	0.08		0.960	0.015	
Commercial herbal mouthwash	4.0	0.13		1.526	0.012	0.000
Distilled water	7.0	0.14		0.818	0.008	

^{*}significant difference (p < 0.01), Anova

Citric Acid is an organic acid that produces high erosion on teeth and causes degradation of composite resin. In the degradation process, the matrix weakens by forming rough holes in the filler. This causes the filler to come off when exposed to chemicals such as detergents and certain foods. Acid solutions containing hydrogen ions break the bonds, resulting in degradation of resin by shortening the polymer chain bonds, which turn into oligomers, and then to monomers.⁵²

The substances degrade the surface of composite resin by removing monomers from the polymer bond structure. Therefore, water and liquids easily enter the opened polymer structure and cause a decrease in hardness. The decrease can be attributed to the process of absorbing water in the matrix, dissolving and softening the matrix by alcohol and acid.⁵³ The combination of these different factors reduces hardness value.

Nanofiller composite resin immersed in 0.2% chlorhexidine mouthwash produced a lower hardness value than Jamblang fruit juice but higher than commercial herbal mouthwash. This was caused by the low viscosity of 0.2% chlorhexidine (0.9) and the higher pH value (4.7), suggesting a lower acidity compared to Jamblang fruit juice and commercial herbal mouthwash. A lower viscosity facilitates easy absorption of water into the composite resin polymer matrix, thereby increasing the diffusion coefficient value.⁵⁴ On the other hand, the higher the viscosity, the lower the diffusion rate.⁵⁵ The diffusion coefficient influences the rate at which water diffuses into the polymer network, thereby affecting the degradation of the composite resin. The higher the diffusion coefficient, the faster the degradation process. The lower acidity level of 0.2% chlorhexidine did not increase the degradation process. Therefore, hardness value was higher than immersion in commercial herbal mouthwash which had a more acidic pH.

The lowest viscosity of distilled water and continuous absorption during the immersion cycle imply that diffusion occurs more quickly during the absorption process by the polymer matrix. This ultimately causes degradation of resin matrix and weakens the bond of the filler with the matrix. Therefore, hardness value of the nanofiller composite resin was lower compared to immersion in mouthwash and specimens without immersion treatment.

This study was conducted under controlled laboratory (in vitro) conditions, which do not fully reflect the complex environment of the oral cavity. Factors such as salivary flow, enzymatic activity, mechanical forces (brushing and chewing), and temperature fluctuations were not simulated, which may influence the actual performance of composite resin in clinical use. Additionally, the immersion period applied was relatively short and may not represent the long-term exposure typically associated with routine mouthwash use. Further in vivo studies with extended exposure durations under more clinically relevant conditions are recommended to obtain more applicable results.

Conclusion

In conclusion, exposure to Jamblang fruit juice mouthwash on nanofiller composite resin restorative material did not cause colour change or reduce hardness, but increased surface roughness. Further *in vivo* studies are highly recommended to explore long-term effects and compare various types of mouthwash to better understand the clinical relevance.

Conflict of Interest

The authors declare no conflicts of interest.

Authors' Declaration

The authors hereby declare that the work presented in this article are original and that any liability for claims relating to the content of this article will be borne by them.

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