ORIGINAL ARTICLE

Surface roughness of supra-nano spherical filled and nanohybrid composite resin after *Coffea arabica* immersion: an experimental study

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ABSTRACT

Introduction: Supra-nano spherical filled composite resin (Omnichroma, Tokuyama, Japan) with sol-gel method has 0.26 µm filler size, while nanohybrid composite resin (ENA HRi, Micerium, Italy) has 0.2-1 µm filler size. Nanofillers enhance resistance from chlorogenic acid contained in *Coffea arabica*. This study analyzed the surface roughness difference between supra-nano spherical-filled and nanohybrid composite resins after *Coffea arabica* immersion for 7 and 14 days, with 15 minutes of immersion each day. **Methods:** This study was an in vitro laboratory experiment with a pretest-posttest control group design. Twenty composite resin samples were immersed in artificial saliva and divided into four groups; supra-nano coffee, nanohybrid coffee, supra-nano saliva (control), and nanohybrid saliva (control). After 24 hours, immersion in *Coffea arabica* was started for supra-nano coffee group and nanohybrid coffee group for 15 minutes daily until days 7 and 14. Control groups were immersed in saliva. **Results:** One-way ANOVA showed significant differences in the surface roughness between composite resin groups. Post-hoc Tukey showed a significant difference between supra-nano coffee (0.91± 0.29 µm) and nanohybrid saliva (1.43±0.42 µm). The GLM Repeated Measure test showed significant surface roughness changes in supra-nano after 14 days of immersion in *Coffea arabica* (1.47±0.13 µm), while nanohybrid experienced surface roughness changes in 7 days of immersion in *Coffea arabica* (1.94±0.55 µm) and increased in 14 days (2.25±0.47 µm). In saliva immersion (control group), nanohybrid experienced an increase in surface roughness after 14 days, while supra-nano did not experience a significant increase in surface roughness. **Conclusion:** Supra-nano spherical filled composite resin exhibits greater resistance to changes in surface roughness following immersion in *Coffea arabica.*

KEYWORDS

Coffea arabica, nanohybrid, composite resin, supra-nano spherical filled, surface roughness

INTRODUCTION

Composite resin, frequently utilized in restorative dentistry, is a material that has undergone continuous technological advancement.[1] Composite resin serves as an effective substitute for tooth structure, offering a serial selection of colors and facilitating the replication of anatomic tooth contours to enhance aesthetic outcomes.[2,3] Composite resin restoration necessitates the creation of a smooth restoration surface. A good restoration surface is essential for composite resin restorations. Notably, the surface roughness of composite resin plays a pivotal role in extrinsic discoloration, with rough surfaces exceeding 200 nm promoting heightened biofilm accumulation and potentially compromising the material's mechanical strength.[4]

The ongoing development of composite resins encompasses the creation of supra-nano spherical filled composites, which are synthesized utilizing the sol-gel method. This innovative composite resin is engineered through a sol-gel technique that enables precise control over filler diameter and refractive index adjustment, thereby yielding favorable aesthetic properties.[5] Supra-nano spherical-filled composite resin tends to have few color changes over time due to reduced photochemical degradation.[6]

During treatment, this composite resin demonstrated the ability to seamlessly integrate with the surrounding tooth surface upon curing.[7] Notably, the supra-nano spherical filled composite resin represents a single-shade variant, facilitating simplified restoration procedures for clinicians by effectively matching the natural tooth color.[5] The use of optimized nano-sized fillers can improve the color-matching ability of composite resins.[8] The degree of conversion of composite resin is a critical factor in determining its mechanical strength. Supra-nano spherical-filled composite resin exhibited a lower degree of conversion compared to several nanohybrid composite resins in the present study.[9]

Nanohybrid composite resin is the most common type of composite resin used in modern dental practice. Distinguished by its reduced filler particle size in comparison to microhybrid composite resin, nanohybrid composite resin exhibits a smoother surface and promotes enhanced filler material dispersion within the matrix by amalgamating nanoparticles with submicron particles.[6,10] Nanohybrid composite resin is easier to polish, thereby offering superior aesthetics compared to microhybrid composite resin.[11] Notably, due to the substantial nano filler content, comprising 75-80% by weight of the composite resin, nanohybrid composite resin exhibits minimal susceptibility to shrinkage following polymerization.[12] A decrease in filler size leads to an increase in filler surface area, typically resulting in higher viscosity and necessitating an overall decrease in filler content.[13]

Coffea arabica is one of the most popular and widely cultivated coffee varieties in Indonesia. It is noteworthy that composite resin restorations are susceptible to degradation upon exposure to acidic liquids or the low pH characteristic of *Coffea arabica*. With a low pH level, *Coffea arabica* has a higher acidity level compared to *Coffea canephora*.[14] *Coffea arabica* is known for its potent aroma and tendency to have a sour taste.[15] Coffee consumption has become an integral aspect of individuals' daily routines.

According to the Agricultural Data and Information System Center of the Ministry of Agriculture, national coffee consumption is projected to reach 370 thousand tons in 2021, with production estimated at 795 thousand tons. Notably, the demographic segment of coffee consumers falls primarily within the age range of 20 to 34 years.[16] The presence of chlorogenic acid in *Coffea arabica* can have a significant impact on composite resin's mechanical properties. Chlorogenic acid, in particular, is involved in the formation of pores within the filler, leading to matrix degradation. Chlorogenic acid has H+ ions, which can cause hydrolytic breakdown and break the methacrylate group ion in the bond between the filler and coupling agent. This can cause an increase in the roughness of composite resin.[17]

Previous study revealed that supra-nano spherical filled and nanohybrid composite resins immersed in a mixture of robusta and arabica coffee, exhibited an increase in surface roughness after 7 days.[18] Meanwhile another study stated that there was no increased roughness in single-shade composite resins and nanohybrids after immersion in arabica coffee for 7 and 30 days.[19] Further research is needed due to these differing findings.[19] In this study, a supra-nano spherical filled composite resin has a filler loading of 79 wt%, slightly lower than 82 wt% on previous research. It doesn't contain Bis-GMA monomer which is hydrophilic and tends to degrade more easily. Moreover, nanohybrid composite resin has silanized zirconium oxide nanoparticles.[7,18,19] The objective of this research was to analyze the surface roughness difference between supra-nano spherical-filled and nanohybrid composite resins after *Coffea arabica* immersion for 7 and 14 days.

METHODS

The research was conducted at the Dental Material and Testing Center of Research, Faculty of Dentistry, Universitas Trisakti, from September 2023 to October 2023. This genuine experimental study utilized a pretest-posttest control group design. The study involved supra-nano spherical-filled composite resin (Omnichroma, Tokuyama Dental, Japan, LOT 053E42) and nanohybrid composite resin (Micerium ENA HRi UE2, Italy, LOT 2023000058) with a diameter of 6 mm and a thickness of 3 mm according to ISO 4049. The composition of these materials can be seen in Table 1. The sample size calculation was determined by G*Power, and the minimum number of samples per group was 3. This study consisted of 5 samples per group, and a total of 20 samples were distributed into 4 groups.

The supra-nano spherical filled and nanohybrid composite resin samples were made using a stainless-steel mold. The process involved inserting the composite resin sample into the mold, flattening it with a celluloid strip, and covering it with a 1 mm-thick glass slide. Subsequently, the samples were light cured using an LED light-curing unit (LY→LY-B200 Dental LED Light Curing Unit, China) for 20 s, after which the initial surface roughness was assessed using a surface roughness tester (Taylor Hobson Surtronic, AMETEK, USA). All samples were transferred into a container and then subjected to a 24-h incubation (Labtech, Daihan, China) period with artificial saliva (Universitas Indonesia) at a temperature of 37°C.

The 6.25% *Coffea arabica* (Kopi Arabika Gunung Puntang Black Honey, Kiesta Roastery, Indonesia) was made fresh every day by mixing 15 g of *Coffea arabica* powder with 225 mL of water at a temperature of 100°C. The degree of acidity of *Coffea arabica* solution was measured every day, and a pH of 5 was obtained.

Groups I and II consisted of the supra-nano spherical-filled and nanohybrid composite resin groups. These groups were first immersed in artificial saliva for 24 hours, and then continued to be immersed in *Coffea arabica* for 15 minutes each day until days 7 and 14. Subsequently, the temperature of the *Coffea arabica* solution upon being placed into the container was 80°C, aligning with the temperature of the oral cavity, and then the solution was incubated at a temperature of 37°C. While not immersed in Coffea *arabica*, the samples were stored in artificial saliva with compositions of NaCl, KCl, Na_2HPO_4, $NaHCO_3$, and urea (Universitas Indonesia).

Meanwhile, groups III and IV, consisting of supra-nano spherical-filled and nanohybrid composite resin, were immersed in artificial saliva for a duration of 7 days and this immersion continued for an additional 14 days. Notably, both the *Coffea arabica* solution and artificial saliva were replaced with fresh solutions daily. Surface roughness measurements were conducted on the 7th and 14th days using a surface roughness tester (Taylor Hobson Surtronic, AMETEK, USA), in the center area of the sample, with 3x readings per sample measurements.

The normality and homogeneity of data were analyzed using the Shapiro-Wilk and Levene test, while the differences of surface roughness values among the 4 groups were tested with one-way ANOVA and post-hoc Tukey ($p<0.05$). The differences of surface roughness values in the same composite resin group at days 0, 7, and 14 were tested with the GLM repeated measures test. All data were analyzed using SPSS statistic 25 software (SPSS Inc, Chicago, USA).

Table 1. Composition of supra-nano spherical filled and nanohybrid composite resin[7,13,20]

	Omnichroma (Tokuyama,Japan)	Micerium ENA HRi UE2 (Avegno, Italia)
Type of fillers	Supra-nano spherical filled	Nanohybrid
Type of matrix	Urethane dimethacrylate (UDMA)/ Triethylene glycol dimethacrylate (TEGDMA) monomers	Diurethane Dimethacrylate, Iso-propyliden-bis (2(3)- hydroxy-3(2)-4(phenoxy)propyl)-bis(methacrylate)(Bis-GMA); 1,4 - Butanediol Dimethacrylate.
Filler volume	79%	80%
Filler size	0.26 µm	1 µm

RESULTS

Based on the normality test using Shapiro-Wilk, the data was found to be normally distributed (p>0.05). The data were presented in Table 2. Data analysis was performed using a one-way ANOVA and the GLM repeated measures test. The results of these tests are presented in Table 3. The one-way ANOVA test showed significant differences in surface roughness values between the supra-nano spherical filled and nanohybrid composite resins (p<0.05). Based on these findings, post-hoc Tukey testing was conducted (Table 4) to compare surface roughness between each composite resin group. The results of the post-hoc Tukey test revealed a substantial variance in surface roughness between the supra-nano coffee and nano saliva groups on days 0, 7, and 14, with p-values of 0.003, 0.005, and 0.007 (p<0.05), respectively. However, no significant differences were observed between the other groups on various immersion days.

According to the results of the GLM repeated measures test presented in Table 2, significant differences in surface roughness were observed in each group by comparing the same composite resin group at days 0, 7, and 14. Subsequently, the GLM post-hoc test in Table 5 indicated that immersing the supra-nano spherical filled composite resin group in *Coffea arabica* for 14 days led to a notable increase in surface roughness (p<0.05). Additionally, the GLM post-hoc test demonstrated that immersing the nanohybrid composite resin group in *Coffea arabica* for 7 and 14 days resulted in a significant increase in surface roughness (p<0.05). Furthermore, the nanohybrid composite resin group was immersed in artificial saliva for 14 days, resulting in a significant increase in surface roughness. Conversely, in the supra-nano spherical filled group, saliva immersion did not yield a significant change in roughness.

Table 2. Test of normality and homogeneity of variances in surface roughness values

Group	Tests of Normality p-value			Test of Homogeneity of Variances p-value		
	d-0 (n=5)	d-7 (n=5)	d-14 (n=5)	d-0 (n=5)	d-7 (n=5)	d-14 (n=5)
Supra-nano coffee	0.86	0.88	0.92	0.73	0.34	0.17
Nanohybrid coffee	0.92	0.43	0.43			
Supra-nano saliva	0.33	0.15	0.09			
Nanohybrid saliva	0.18	0.07	0.34			

Table 3. Differences in surface roughness values between composite resin groups on immersion days 0, 7, and 14

Group	Average ± SD (µm)			p-value
	d-0 (before immersion)	d-7	d-14	
Supra-nano coffee	0.91 ± 0.29	1.23 ± 0.23	1.47 ± 0.13	0.005†
Nanohybrid coffee	1.43 ± 0.42	1.94 ± 0.55	2.25 ± 0.47	0.001†
Supra-nano saliva	1.41 ± 0.56	1.81 ± 0.38	1.78 ± 0.39	0.044†
Nanohybrid saliva	2.05 ± 0.38	2.37 ± 0.53	2.54 ± 0.62	0.01†
p-value	0.006*	0.009*	0.007*	

SD: Standard deviation; *One way ANOVA (p<0,05)
†GLM repeated measures (p<0,05)

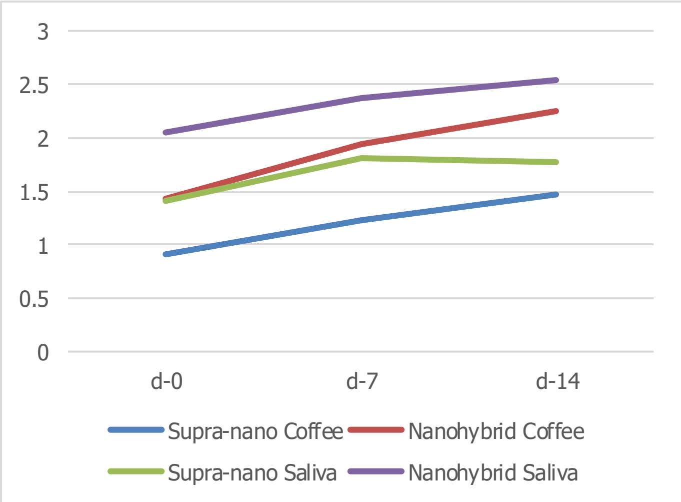

Figure 1. Differences in surface roughness values (µm) between composite resin groups on immersion days 0, 7, and 14

Table 4. Level of significance between composite resin groups on surface roughness value

Group		d-0 p-value	d-7 p-value	d-14 p-value
Supra-nano coffee	Nanohybrid coffee	0.26	0.09	0.057
Supra-nano coffee	Supra-nano saliva	0.28	0.20	0.68
Supra-nano coffee	Nanohybrid saliva	0.003*	0.005*	0.007*
Nanohybrid coffee	Supra-nano saliva	1.000	0.97	0.36
Nanohybrid coffee	Nanohybrid saliva	0.13	0.45	0.73
Supra-nano saliva	Nanohybrid saliva	0.12	0.24	0.06

*Post-hoc Tukey (p<0,05)

Table 5. Significance level of surface roughness on days 0, 7, and 14 in each composite resin group

Group	d-0 p-value	d-7 p-value	d-14 p-value
Supra-nano coffee	0.14	0.02*	0.07
Nanohybrid coffee	0.008*	0.01*	0.12
Supra-nano saliva	0.21	0.08	1.00
Nanohybrid saliva	0.07	0.04*	0.14

*Post-hoc GLM (p<0,05)

DISCUSSION

In the experimental group, specimens were immersed in *Coffea arabica*, known to contain chlorogenic acid. The chlorogenic acid present in *Coffea arabica* makes up 3 – 5.6 % of the coffee's dry weight.[21] Chlorogenic acid comprises ions capable of dissolving materials. When composite resin is immersed in H^+ ions, it exhibits high solubility, resulting in erosion of the composite resin surface and an increase in surface roughness. The composite resin absorption of H^+ ions initiates hydrolytic breakdown, causing the methacrylate group ions to disintegrate from the bond between the filler and coupling agent.[22] Following the disintegration of the group ions, the composite resin material is degraded.[23] The components in composite resin that can minimize water absorption include the matrix and filler. The nanohybrid composite resin has a hydrophobic UDMA matrix due to the absence of hydroxyl groups. Water absorption in composite resins can induce hydrolysis.[24] Both supra-nano spherical-filled composite resins and nanohybrid composite resins contain nano-sized glass particle fillers. Nano-sized fillers can be optimally distributed within the resin matrix, thereby enhancing the strength and resistance to degradation of a composite resin.[25,26]

The *Coffea arabica* solution and artificial saliva were replaced daily to maintain consistent pH levels and prevent any potential impact on the results. The 15-minute daily immersion period simulates the duration an individual consumes coffee each day over the course of one year. It is noted that 3 days of daily 15-minute immersion are equivalent to 1 year of coffee consumption, while 7 days of daily 15-minute immersion equate to 2 years of coffee consumption.[20] All treatment groups were immersed in artificial saliva for 24 hours prior to immersion in *Coffea arabica*. This is because the highest rate of composite resin monomer release occurs in the first 24 hours post-polymerization. In the control group, the samples were immersed in artificial saliva. In addition, salivary media was chosen because of ability to mimic the clinical conditions of the oral cavity.[27]

In this study, it was demonstrated that *Coffea arabica* led to an increase in the surface roughness of supra-nano spherical filled composite resin and nanohybrid composite resin. The one-way ANOVA test (Table 3) confirmed a disparity in roughness between the supra-nano spherical-filled composite resin and the nanohybrid composite resin. Clinically, supra-nano spherical-filled composite resin yielded a smoother surface compared to nanohybrid composite resin. Although one statistical test indicated that a specific group of nanohybrid composite resins exhibited higher surface roughness than the supra-nano spherical-filled composite resins, this finding could not be conclusively proven, as the other samples of nanohybrid composite resins did not exhibit rougher surfaces than the supra-nano spherical-filled composite resins.

Nanohybrid composite resin primarily consists of a matrix composition based on Bis-GMA. Composite resins with a Bis-GMA matrix as the base material tend to be hydrophilic, making them more susceptible to liquid absorption and surface degradation, thus increasing surface roughness. Under acidic conditions, composite resins containing Bis-GMA as the main component demonstrate higher hydrophilicity compared to composite resins containing UDMA, such as supra-nano spherical filled composite resins.[20]

In this research, the spherical-filled supra nano immersed in coffee significantly had a lower surface roughness value compared to the nanohybrid immersed in saliva on days 0, 7, and 14. The notable distinction in roughness could be attributed to the significant distinctions between these groups from the outset, before immersion. Study by Aydin et al. indicated that the change in roughness of supra-nano spherical filled composite resin after immersion in coffee was the lowest compared to the nanohybrid composite resin type. The smaller filler particle size can improve the composite resin's resistance to degradation because it will make the composition denser and more resistant to acid.[18]

On the 14th day, supra-nano spherical-filled composite resin exhibited lower surface roughness results. However, there was no significant difference in surface roughness between the types of composite resin immersed in coffee. This suggests that both supra-nano spherical-filled and nanohybrid composite resins offer similar resistance to acid degradation. On the other hand, upon completion, nanohybrid composite resins tend to yield a rougher surface from the outset compared to supra-nano spherical-filled composite resins, which produce a finer roughness.

These research findings revealed that supra-nano spherical-filled composite resins did not experience significant roughness changes in saliva immersion. However, the nanohybrid composite resin experienced significant roughness changes after 14 days of immersion in saliva. Moreover, the supra-nano spherical-filled composite resin experienced significant roughness changes after 14 days of immersion in *Coffea arabica*. On the other hand, the nanohybrid composite resin experienced significant roughness changes after 7 days of immersion in *Coffea arabica* and continued to increase its roughness on the 14th day of immersion. The smaller filler size of spherical filled supra-nano composite resin results in a denser filler content, which contributes to the production of a smoother surface.

Various factors, such as matrix, filler size, and coupling agent, can influence the increased surface roughness of composite resin. The filler content is associated with the depth of polymerization, stability, and resistance to degradation. Damage to the matrix caused by water absorption and hydrolytic degradation can lead to changes in the composite's mechanical properties. In acidic conditions, water absorption properties can cause the filler and matrix to break down. These findings were align with the research conducted by Atmaja et al., as the H^+ ions present in *Coffea arabica* can destabilize the chemical double bonds of the composite resin polymer matrix. This instability arises from the cross-linking with H^+ ions and the breaking double bonds in the polymer matrix.[20] In this study, there were some limitations encountered, especially in making composite resin samples with the same surface roughness across the samples.

The hypothesis posited in this study can be validated based on the post hoc GLM test results, which demonstrate significant differences with $p<0.05$ in the supra-nano spherical-filled composite resin after being immersed in *Coffea arabica* for 14 days, and in the nanohybrid composite resin after immersion for 7 and 14 days. However, immersion of the supra-nano spherical filled composite resin in *Coffea arabica* for 7 days did not yield significant changes. The study's limitations include the small number of samples and the difference in initial surface roughness values between groups of similar composite resins before coffee immersion.

CONCLUSION

Coffea arabica can notably increase the surface roughness of supra-nano spherical filled composite resin after 14 days of immersion, and substantially increase the surface roughness of nanohybrid composite resin after 7 and 14 days of immersion. Supra-nano spherical filled composite resin has greater resistance to changes in surface roughness compared to nanohybrid composite resin when immersed in *Coffea arabica*. Implication of these findings underscore the importance of considering coffee exposure when selecting dental composite materials.

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