MALAYSIAN JOURNAL OF MICROSCOPY

Journal homepage: https://malaysianjournalofmicroscopy.org

RESEARCH ARTICLE

MICROSTRUCTURE, SURFACE ROUGHNESS AND VICKERS HARDNESS ANALYSES OF MISWAK REINFORCED DENTAL COMPOSITE

Raja Azman Raja Awang, Muhammad Aqil Haikal Ab Halim Fauzi, Gao Guanlin, Noor Huda Ismail*

School of Dental Sciences, Health Campus, Universiti Sains Malaysia, Kubang Kerian, 16150 Kota Bharu, Kelantan, Malaysia.

Abstract. Despite the widespread use of dental composites, their mechanical properties and surface characteristics can sometimes fall short of clinical expectations, potentially affecting long-term durability and overall performance. Miswak, a natural product with known oral health benefits, has been proposed as a novel reinforcement agent to improve composite properties. This study investigated the effects of miswak on the microstructure, surface roughness, and Vickers hardness (VHN) of dental composites. Specimens were prepared in the following groups: negative control (no miswak), 1 wt% miswak, 3 wt% miswak, and positive control (Filtek™ Z350XT, 3M ESPE). Scanning electron microscopy (SEM) was used to examine the microstructure, while surface roughness and VHN were assessed using a profilometer and hardness tester, respectively, under standardised laboratory conditions. Data were analysed with one-way ANOVA and post-hoc Tukey's test. SEM analysis showed a homogeneous miswak distribution in all groups. The 3 wt% miswak group exhibited significantly lower surface roughness than the other groups ($p < 0.05$). The 1 wt% and 3 wt% miswak groups showed increased VHN compared to the negative control, but no significant difference was found between the 1 wt% and 3 wt% groups ($p > 0.05$). These results suggest that miswak reinforcement can improve the surface roughness and VHN of dental composites without compromising the resin matrix's homogeneity.

Keywords: Miswak, microstructure, surface roughness, Vickers hardness.

Article Info

Received 4 February 2025
Accepted 2 November 2025
Published 4 December 2025

***Corresponding author: noorhuda@usm.my**

ISSN: 1823-7010, eISSN: 2600-7444

1. INTRODUCTION

Dental composites, commercially introduced in the 1960s, are tooth-coloured restorations widely used in restorative dentistry due to their aesthetic appeal, lower toxicity compared to some traditional materials, and suitability for conservative treatment [1]. These composites typically consist of a resin matrix reinforced with fillers, such as silica, hydroxyapatite, zirconia, and alumina, to provide the mechanical and physical properties needed to withstand occlusal forces. Key properties include high strength, wear resistance, and low polymerisation shrinkage, all of which contribute to the clinical success of restorations [2,3]. However, traditional composites face challenges, particularly fatigue and crack propagation under masticatory forces, limiting their durability [3]. To address these issues, various reinforcement strategies have been developed, including the incorporation of inorganic fillers and/or natural minerals with different shapes and sizes into the resin matrix [4]. This study proposes miswak, a natural fiber with known oral health benefits, as a reinforcement agent to improve the longevity and biocompatibility of composite materials.

Miswak (*Salvadora persica*), traditionally used as an oral hygiene tool, has attracted attention for biomedical applications due to its safety, biocompatibility, and availability, particularly in the Middle East, Asia, and Africa. Its unique composition could enhance the structural integrity and impart bioactivity to composites [5]. Miswak contains carbohydrates, flavonoids, terpenes, sterols, alkaloids, glycosides, and essential trace elements such as fluoride, calcium, phosphorus, silica, and ascorbic acid. These bioactive compounds contribute antimicrobial, antiviral, antifungal, antibiofilm, and antioxidant properties [5,6]. Integrating miswak into dental composites could reinforce the material mechanically while also introducing bioactive advantages to dental restorations [7].

Surface roughness and hardness are critical parameters in assessing the performance and durability of dental composites. Surface roughness influences wear resistance, colour stability, optical properties, and hardness [8]. Rougher surfaces promote plaque accumulation, increasing bacterial growth and risking discolouration and degradation of restorations [9]. Hardness, defined as resistance to permanent deformation, is closely linked to wear resistance. Research has shown that lower hardness values are associated with reduced wear resistance, potentially limiting the composite's ability to withstand occlusal forces and resist long-term damage [8]. Conversely, higher hardness values enhance the material’s resistance to surface wear, a crucial property for maintaining the structural integrity of restorations, especially in high-stress areas of the oral cavity.

Optimal surface roughness in dental composites prevents plaque accumulation [9,10]. While Vickers hardness is commonly used to assess the brittleness of dental composites. Noushad et al. have reported that silica from rice husk improves the hardness, compressive strength, and flexural strength of dental composites [2]. Modifying existing dental composites with miswak may further improve their mechanical properties. To our knowledge, no studies have been published on the surface roughness and Vickers hardness of dental composites incorporating silica rice husk reinforced with miswak.

In this study, silica extracted from rice husk and miswak was treated with a silane coupling agent and used to develop a hybrid dental composite. The objective was to characterise the morphological features of miswak powder to understand its interaction with the composite matrix and to investigate the effects of incorporating miswak powder at concentrations of 1 wt% and 3 wt% on the microstructure, surface roughness, and Vickers hardness of a newly developed experimental dental composite.

2. MATERIALS AND METHODS

2.1 Materials

The composite resins used in this study were bisphenol A-glycidyl methacrylate (Bis-GMA) (Esstech, Inc., Essington, PA, USA) and triethylene glycol dimethacrylate (TEGDMA) (Sigma-Aldrich,

USA). The fillers included miswak powder (Minature Siwak, Marudhar Impex, India) and silica from rice husk. For surface treatment, γ-methacryloxypropyltrimethoxysilane (γ-MPS) (Sigma-Aldrich, USA) was employed as a silane coupling agent. DL-camphorquinone (CQ) (Merck, Schuchardt OHG, Germany) served as the photoinitiator, while (2-dimethylaminoethyl) methacrylate (DMAEM) (Merck, Schuchardt OHG, Germany) was used as the amide co-initiator. Filtek™ Z350XT (3M ESPE, USA) was used as a reference material for comparison.

2.2 Fabrication of the Experimental Dental Composite

The silica and miswak powders were treated with γ-MPS, and their surface morphology was examined using Field Emission Scanning Electron Microscopy (FESEM) (Quanta 650 SEM, FEI, Germany). A 0.4 g sample of each filler was dried at 100 °C for 24 hours in a porcelain crucible. The dried particles were mounted on aluminium stubs, sputter-coated with gold using a cool sputter coater (EM SCD 005, Leica, Germany). Elemental analysis of silica was performed using FESEM at 15 kV. The dental composites were formulated using silica extracted from rice husk, following a method outlined in a prior study [2], with modifications made to the resin ratio for optimisation. Miswak powder was then incorporated into the composite to enhance its physical and mechanical properties. Disc-shaped composites were mounted, gold-coated, and analysed under the same SEM conditions. Surface images were captured at 150x magnification.

Table 1 presents the formulation for the experimental dental composite. The base materials Bis-GMA, TEGDMA, 0.5 wt% CQ, and 0.5 wt% DMAEM were initially premixed in a porcelain dish to ensure uniform distribution of the liquid components. The treated silica and miswak powders were then gradually incorporated into the mixture. To achieve thorough and homogenous mixing, a Kakuhunter mixer (Model DK-300S II, Japan), which utilises planetary centrifugal force, was employed under optimised speed and duration settings. This method ensured uniform dispersion of the fillers while minimising air entrapment. The resulting mixture was visually inspected for consistency and the absence of any powder agglomerates. Figure 1 depicts the preparation and fabrication procedure for the composite.

Table 1: Composition of experimental dental composites

Groups	Filler/Resin (50/50)		
	Filler loading (%)		Resin Matrix (%)
	Silica	Miswak	
CTR	100	-	BisGMA/TEGDMA
1 wt% miswak	99	1	(60/40)
3 wt% miswak	97	3	
Filtek™ Z350XT	N/A	N/A	BisGMA, BisEMA, UDMA, and TEGDMA

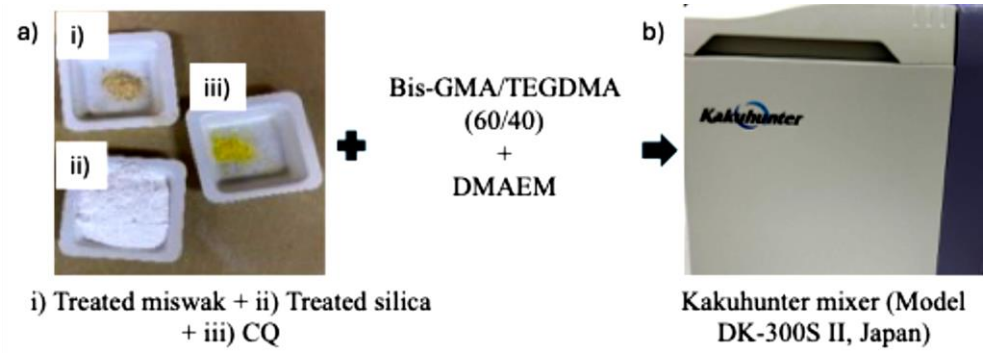

Figure 1: Preparation and fabrication of experimental dental composite

The experimental dental composite was divided into two groups based on the percentage of miswak filler reinforcement: 1 wt% miswak and 3 wt% miswak. A control group, consisting of a composite without miswak reinforcement (CTR), served as the negative control. In contrast, the commercial dental composite resin Filtek™ Z350XT (3M ESPE, USA) was used as the reference material. Filtek™ Z350XT was chosen for its clinical approval, durability, and aesthetic qualities. It resists wear, maintains colour stability, and mimics natural teeth. The experimental dental composites underwent evaluations of their physical and mechanical properties, including microstructural analysis, surface roughness (SR), and Vickers hardness (VHN). Descriptive statistics for SR and VHN were calculated, and a Tukey's post hoc multiple comparison test (one-way analysis of variance) with a significance level of $p < 0.05$ was performed.

2.3 Surface Roughness (SR)

A total of 40 specimens ($n = 10$ per group) were prepared for the SR test using a custom Perspex mold to create disc-shaped specimens (5 mm diameter, 2 mm thickness). A Mylar strip was placed over the free surface of each specimen and pressed with a glass slide to remove excess material before curing with an LED unit (Celalux II; Voco, Germany) for 40 seconds on both sides. The curing unit emitted light at a wavelength of 470 nm. To standardise the surface preparation, all experimental groups were polished with silicon carbide abrasive papers (Sof-Lex discs; 3M ESPE, St Paul, MN) in a sequential order of course, fine and extra-fine grits. The same operator performed the polishing manually using a slow-speed handpiece to minimize inter-operator variability. SR was measured using a profilometer (Surfcom Flex, Accretech, Japan). Three measurements were taken per specimen, and the average value was calculated.

2.4 Vickers Hardness (VHN)

For the VHN test, a total of 40 specimens ($n = 10$ per group) were prepared using the same procedure as for the SR test. Disc-shaped specimens (5 mm diameter, 2 mm thickness) were fabricated using a custom Perspex mold. A Mylar strip was pressed against the free surface of each specimen with a glass slide to remove excess material before curing with an LED unit (Celalux II; Voco, Germany) in standard mode for 40 seconds. The specimens were then polished using silicon carbide abrasive papers (Sof-Lex discs; 3M ESPE, St. Paul, MN) in the same sequential grit order as for the SR test. VHN was measured using an automatic hardness tester (Model FALCON 500G2, INNOVATEST, Netherlands) in accordance with the ASTM E384-22 standard [11]. Three indentations were made at different points on each specimen using a 1 kg load applied for 15 seconds. The average of the three measurements was calculated.

2.5 Statistical Analysis

Data analysis was performed using a one-way analysis of variance (ANOVA), complemented by post-hoc Tukey's honestly significant difference (HSD) multiple comparison test. The level of statistical significance was set at 0.05.

3. RESULTS AND DISCUSSION

3.1 Characterizations of Fillers

Figure 2 presents FESEM micrographs of mesoporous spherical silica and irregularly crystal-shaped miswak particles at magnifications of 40,000× and 2,000×, respectively. Different magnifications were selected to capture the morphological features of each filler appropriately, as the particle size of treated silica (63–346 nm) is significantly smaller than that of treated miswak (43–125.15 µm). These size ranges were calculated from the SEM images using ImageJ software (version 14), based on measurements of randomly selected particles.

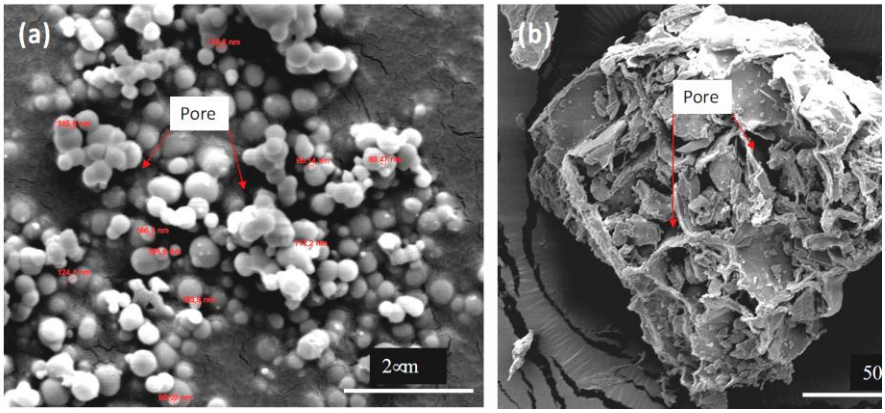

Figure 2: FESEM micrographs at magnification of (a) 40,000x of treated silica and (b) 2,000x of treated miswak

The results showed that the treated silica exhibited a narrower apparent pore size and more uniform surface morphology compared to the treated miswak. The FESEM analysis confirmed that the treated miswak, with its distinct crystal-like structure and larger particle size, displayed wider surface voids and rougher texture than the treated silica. Although these observations are qualitative, they provide useful insight into the relative differences in surface structure, consistent with previous studies [12,13].

3.2 Characterisation of the Experimental Dental Composite

The CTR group displayed a relatively smooth surface with slight microscale irregularities, indicating uniform surface contours, as shown in Figure 3. In contrast, composites containing additional fillers, such as 1 wt% miswak, 3 wt% miswak, and Z350XT, exhibited irregularities and granular structures.

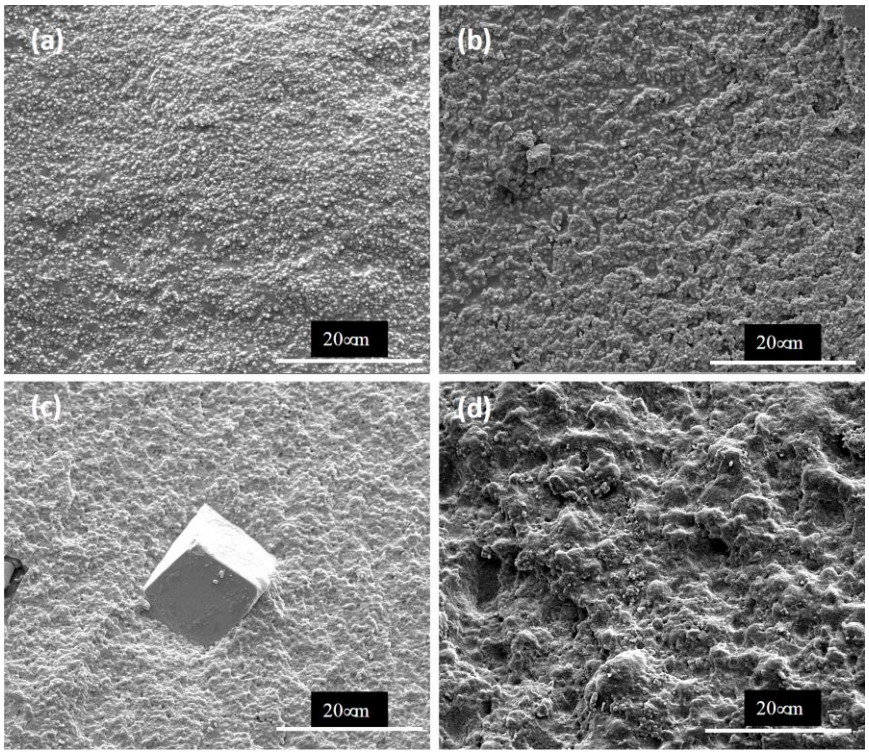

Figure 3: FESEM micrographs at 150x magnification of the specimen (a) CTR, (b) 1 wt% miswak, (c) 3 wt% miswak, and (d) Z350XT dental composites

Figure 3(c) illustrates the FESEM micrograph of the 3 wt% treated miswak composite. The surface appears relatively smooth and compact, indicating good filler dispersion and strong adhesion between the miswak particles and the resin matrix. Compared with the 1 wt% miswak composite, the 3 wt% sample exhibits a slightly smoother texture and the presence of a crystal-like morphology, suggesting improved filler–matrix interaction and reduced surface irregularities. The varying sizes and irregular shapes of miswak particles and the surface modification techniques influenced their incorporation into the resin matrix. These findings align with previous studies, which have shown that surface morphology is closely related to the size and distribution of filler particles [14,15]. Additionally, hybrid fillers often appear as discrete particles embedded within the resin matrix, as observed in the 1 wt% miswak and 3 wt% miswak groups, which is in line with previous studies [7,16,17]. However, when well-dispersed, fillers contribute to a smoother and more uniform surface, while poor dispersion can lead to filler agglomeration, resulting in rougher areas [15,18].

3.3 Analysis of Surface Roughness

Table 2 presents the mean (SD) values of surface roughness (SR) for each specimen group. The SR test results indicate a significant difference between the study groups. While the addition of 1 wt% miswak did not significantly affect SR, the addition of 3 wt% miswak resulted in a significant reduction in SR compared to the CTR group. Surface roughness improves with the optimum addition of hybrid reinforcement or miswak filler, as reflected by lower SR values indicating a smoother surface finish. The findings suggest that the addition of miswak improves SR compared to both the CTR and Z350XT groups. Additionally, the SR values were close to the ideal limit of 0.2 µm, consistent with previous studies and in accordance with the ISO 4049:2019 [19-21] standard, which is linked to a reduction in plaque accumulation.

Table 2: Surface roughness of the dental composite

Dental composite	Surface roughness (µm) Mean (SD)	F statistic[a] (df)	*p* value[a]
CTR	0.345 (0.063)[b]		
1 wt% miswak	0.265 (0.080)[c]	6.337	0.001
3 wt% miswak	0.244 (0.071)[bd]	(3, 36)	
Filtek™ Z350XT	0.376 (0.100)[cd]		

*Statistical analysis was carried out using One-way ANOVA[a], followed by post-hoc Tukey's test. Significance level set at $p = 0.05$. The same letter indicates a statistically significant difference ($p < 0.05$).

3.4 Analysis of Vickers Hardness

Table 3 presents the mean Vickers hardness (VHN) values for the study groups. In this study, miswak reinforcement increased in the VHN of the dental composite. The 1 wt% miswak group showed a significantly higher VHN value compared to the control (CTR) group. This improvement is attributed to the hybrid-treated silica and miswak, which strengthened the resin matrix's microstructure and reduced pore volume. These findings are consistent with those of Khan et al., who incorporated miswak into a dental composite [17].

Table 3: Vickers hardness of the dental composite

Groups	Vickers hardness (VHN) Mean (SD)	F statistic[a] (df)	*p* value[a]
CTR	37.629 (2.268)[b]		
1 wt% miswak	39.833 (1.371)[b]	3350.222	0.000
3 wt% miswak	38.783 (0.841)[c]	(3, 36)	
Filtek™ Z350XT	94.357 (1.229)[bc]		

*Statistical analysis was carried out using One-way ANOVA[a], followed by post-hoc Tukey's test. Significance level set at $p = 0.05$. The same letter indicates a statistically significant difference ($p < 0.05$).

There are very limited studies that have incorporated both miswak and silica derived from rice husk into dental composites, particularly in relation to Vickers hardness. Despite the slight increase in VHN with 1 wt% miswak, the difference was not statistically significant when compared to the 3 wt% miswak group in this study, which is consistent with findings from a previous study [17]. This could be due to the higher content of crystal shape and larger miswak particles, which may affect the mechanical properties. It remains unclear whether the improvement was solely due to miswak fillers or the hybrid reinforcement of silica and miswak. The increased hardness may be due to the presence of crystal-shaped structures and larger miswak particles, which resist surface deformation and act as rigid fillers.

In contrast, a decrease in Vickers hardness was observed with 3, 4, and 5 wt% miswak incorporated into the dental composite [17], while another study reported a reduction in compressive strength with 10 wt% miswak in glass ionomer cement [12]. Additionally, nanohybrid composite resin exhibited smaller voids due to the smaller filler particles debonding from the resin while maintaining their micro-sized particles in situ [22]. This characteristic helps explain the force distribution in miswak-reinforced dental composites. Overall, achieving optimal filler loading and uniform dispersion is crucial for improving the physical and mechanical properties of the composite, as improper dispersion may lead to stress concentrations and weak interfacial bonding. Notably, the Z350XT group exhibited significantly higher VHN values than all other study groups, suggesting that further improvements or modifications may be required for the experimental composites to reach clinical suitability.

This study suggests that filler loading, despite variations in the size and shape of miswak particles, can enhance the physical and mechanical strength of hybrid dental composites when added to silica, consistent with previous research [23,24]. Furthermore, the improvements in surface roughness and Vickers hardness indicate that adding miswak may help the composite better withstand mechanical forces, contributing to its longevity.

4. CONCLUSIONS

The miswak filler improves surface quality by reducing surface roughness and increasing Vickers hardness of the experimental composite. The treated miswak filler also aids force distribution within the composite's hardness. However, achieving the optimal miswak filler content is crucial to minimise agglomeration, which could negatively impact the physical and mechanical properties.

Acknowledgements

The authors thank the technicians for their support in the laboratory. This research was funded by the External Research Grant Scheme (Grant no. R504-LR-GAL008-0000000496-D131) and the Universiti Sains Malaysia Bridging Research Grant Scheme (Grant no. R501-LR-RND003-0000002248-0000).

Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

References

[1] Rajan, G., Shouha, P., Ellakwa, A., Bhowmik, K., Xi, J. & Prusty, G. (2016). Evaluation of the physical properties of dental resin composites using optical fiber sensing technology. *Dental Materials,* 32(9), 1113–1123.

[2] Noushad, M., Rahman, I. A., Husein, A. & Mohamad, D. (2016). Nanohybrid dental composite using silica from biomass waste. *Powder Technology,* 299*,* 19–25.

[3] Yang, D., Sun, Q., Niu, H., Wang, R., Wang, D. & Wang, J. (2020). The properties of dental resin composites reinforced with silica colloidal nanoparticle clusters: Effects of heat treatment and filler composition. *Composites Part B: Engineering,* 186, 107791.

[4] Yan, S., Wang, K. & Wang, Z. (2023). A comparative study on the microscale and macroscale mechanical properties of dental resin composites. *Polymers*, 15(5), 1129.

[5] Adam, F. A., Mohd, N., Rani, H., Yusof, M. Y. P. M. & Baharin, B. (2023). A systematic review and meta-analysis on the comparative effectiveness of Salvadora persica - extract mouthwash with chlorhexidine gluconate in periodontal health. *Journal of Ethnopharmacology*, 302(Pt A), 115863.

[6] Aljarbou, F., Almobarak, A., Binrayes, A. & Alamri, H. M. (2022). Salvadora persica's biological properties and applications in different dental specialties: A narrative review. *Evidence-based Complementary and Alternative Medicine,* 2022. 8667687.

[7] Chaaben, R., Taktak, R., Mnif, B., Guermazi, N. & Elleuch, K. (2022). Innovative biocomposite development based on the incorporation of *Salvadora persica* in acrylic resin for dental material. *Journal of Thermoplastic Composite Materials,* 35(11), 1815-1831.

[8] Kumari, C., Bhat, K., Bansal, R., Singh, N., Anupama, A. & Lavanya, T. (2019). Evaluation of surface roughness and hardness of newer nanoposterior composite resins after immersion in food-simulating liquids. *Contemporary Clinical Dentistry*, 10(2), 289-293.

[9] Soliman, H. A. N., Elkholany, N. R., Hamama, H. H., El-Sharkawy, F. M., Mahmoud, S. H. & Comisi, J. C. (2020). Effect of different polishing systems on the surface roughness and gloss of novel nanohybrid resin composites. *European Journal of Dentistry,* 15(2), 259–265.

[10] Isabel, C. A. C., Dominguette, A. A. S., Santos, S. G. D., Ribeiro, J. C. R. & Moysés, M. R. (2016). Surface roughness of a resin composite. *Revista Gaúcha de Odontologia*, 64, 50-55.

[11] ASTM E384 (2022). *Standard Test Method for Microindentation Hardness of Materials.* ASTM International: West Conshohocken, PA, USA.

[12] Devi, K., Pualraj, J., Shanmugam, R. & Maiti, S. (2024). Miswak-infused glass ionomer cement: A comparative in vitro analysis of antibacterial efficacy and compressive strength. *Cureus*, 16(2), e53484.

[13] Ismail, N. H., Raja Awang, R. A., Kannan, T. P., Abdul Hamid, Z. A. & Jaafar, M. (2022). Film thickness, diametral tensile strength and fracture surface of alumina reinforced dental cement. *Malaysian Journal of Microscopy,* 18(1), 12-23.

[14] Randolph, L. D., Palin, W. M., Leloup, G. & Leprince, J. G. (2016). Filler characteristics of modern dental resin composites and their influence on physico-mechanical properties. *Dental Materials*, 32(12), 1586–1599.

[15] Kundie, F., Azhari, C. H., Muchtar, A. & Ahmad, Z. A. (2018). Effects of filler size on the mechanical properties of polymer-filled dental composites: A review of recent developments. *Journal of Physical Science,* 29(1), 141–165.

[16] Zeeshan, M., Amin, N., Muhammad, N., Siddiqui, U., Sultan, Z., Jabeen, H. & Liaqat, S. (2024). Synthesis and characterisation of novel miswak powder-based dental composites. *Cellulose Chemistry and Technology,* 58(7-8), 727-736.

[17] Khan, M. A., Muhammad, N., Liaqat, S., Ejaz, M., Fayyaz, S., Ali, H. & Khan, T. A. (2024). Mechanical and antibacterial properties of conventional pit and fissure sealants with addition of miswak fibers. *Biomaterial Investigations in Dentistry,* 10(1), 2271972.

[18] Xiong, J., Sun, X., Chen, J. & Tang, L. (2013). Influence of filler content on physicomechanical and bonding properties of an experimental dental resin cement. *Journal of Applied Polymer Science,* 127(4), 2427–2434.

[19] Ramírez-Vargas, G. G., Ladera-Castañeda, M. I., López-Gurreonero, C., Cornejo-Pinto, A., Cachay-Criado, H., Cervantes-Ganoza, L. A. & Cayo-Rojas, C. F. (2022). Surface roughness in nanoparticle resin composites subjected to two polishing systems: An in vitro comparative study. *Journal of International Society of Preventive and Community Dentistry,* 13(2), 114-123.

[20] Alkhudhayri, S. L., Alhassani, S. L., Alahmadi, R. J., Alharbi, M. R., Redwan, H. S. & Hawsawi, R. A. (2024). Water sorption and solubility of highly aesthetic single-shade nano-hybrid resin composites. *Cureus,* 16(7), e65548.

[21] International Organization for Standardization (2019). *ISO 4049-2019: Dentistry: Polymer-based restorative materials*. 5th Edition. (International Organization for Standardization).

[22] Păstrav, M., Păstrav, O., Chisnoiu A. M., Chisnoiu, R. M., Cuc, S., Ioan, P., Sarosi, C. & Feştila, D. (2024). Properties of nanohybrid dental composites-A comparative in vitro study. *Biomedicines*, 12(1), 243.

[23] Chaaben, R., Ketata, R. T., Elleuch, K., Ellouz, M. & Kordisch, T. (2020). Wear behaviour of new biomaterial composite for dental application. *Polymers and Polymer Composites,* 28(8-9), 654-662.

[24] Naguib, G., Maghrabi, A. A., Mira, A. I., Mously, H. A., Hajjaj, M. & Hamed, M. T. (2023). Influence of inorganic nanoparticles on dental material's mechanical properties. A narrative review. *BMC Oral Health,* 23(1), 897.