Impact of Nanofiller Fractions on Selected Properties of Microfilled Composite Resin

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Objective: *To assess the impact of incorporating various weight fractions of nanometre-sized particulate fillers on specific properties of microfilled composite resin.*

Methods: *Microfilled composite resin was prepared by mixing 29 wt.% of resin matrix (Bis-GMA/TEGDMA) with the 71 wt.% of silane treated particulate fillers (Ø 0.4 µm). Then, various fractions of nanometre-sized (180 nm) fillers (0, 5, 10, 15, 20, 25, 30 and 35 wt.%) were added gradually using a high-speed mixing machine. For each composite resin, flexural properties (n = 8) were evaluated using a three-point bending test on a universal testing machine (ISO standard 4049). Fourier transform infrared (FTIR)-spectrometry was used to calculate the degree of monomer conversion (DC%). Surface microhardess (Vickers) was also determined. Surface gloss was measured before and after polishing (4,000-grit paper). A two-body wear test was performed in a ball-on-flat configuration using a chewing simulator with 15,000 cycles. A non-contact 3D optical profilometer was utilised to measure wear depth. An analysis of variance (ANOVA) was applied to interpret the results statistically, then a post hoc Tukey analysis was performed.*

Results: *ANOVA revealed that the fraction of nanofillers had a significant effect ($P < 0.05$) on flexural modulus, DC%, microhardness, gloss and wear depth. The group without nanofillers showed the highest DC% (56.6%), gloss after polishing (76.2 GU) and wear resistance (24.2 µm) values, whereas the group with 35 wt.% of nanofillers had the highest flexural modulus (9 GPa) and microhardness (70 VH).*

Conclusion: *It is beneficial to add nanofillers to microfilled composite resin; however, it is essential to assess the proportion ratio carefully. Optimising all the properties of composite resin at once with just one formulation is challenging.*

Keywords: *microfilled composite resin, nanofiller fractions, properties*
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Composites resins have become essential in restorative dentistry due to their versatility and the continuous improvement of their material properties. They typically consist of a polymeric matrix reinforced with fillers, where the filler–matrix interface, often mediated by silane coupling agents, plays a crucial role in the overall performance of the material.[1] By manipulating the proportion, size and shape of these fillers, significant enhancements in mechanical, physical and aesthetic properties can be achieved, allowing composite resins to meet the demands of a wide array of dental applications.[2]

Despite their widespread use, composite resins have not yet reached their full potential, and research efforts continue to focus on improving their clinical performance.[2,3] Innovations in the resin matrix predominantly involve the development of new monomer systems,[4-6] whereas advancements in fillers focus on optimising loading capacity, particle size and surface treatment, and exploring novel particulate or fibre technologies.[7-10] These areas of study are particularly important, as the filler content and particle size significantly influence many properties of dental composites.[7,8]

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One of the most promising advancements in this field is the integration of nanotechnology. Nanotechnology involves manipulating materials on a nanoscale (5 to 200 nm) to enhance specific properties through various physical and chemical approaches.[11] The incorporation of nanometre-sized fillers into microfilled composite resins has garnered significant attention, as these nanofillers occupy voids between larger microfillers, increasing the overall filler content and improving material performance.[12] Given that the resin matrix typically exhibits lower mechanical strength compared to the fillers, reducing the inter-filler voids is essential to improve the strength, hardness and wear resistance of the composite.[7,13] Furthermore, nanofillers offer additional benefits in dental composites, particularly by enhancing optical properties, which are critical for achieving highly aesthetic restorations.[14,15]

Despite these advancements, significant challenges remain with regard to optimising the mechanical and surface properties of composite resins, especially in high-stress areas. The relationship between filler size, shape, distribution and composite performance remains a critical focus of ongoing research.[2] Gaining a deeper understanding of how these variables influence material behaviour is essential for developing next-generation composite resins that combine superior aesthetics with long-lasting durability. Thus, this study aims to explore the optimal weight fraction of nanometre-sized particulate fillers that can significantly enhance the physical, mechanical and aesthetic properties of microfilled composite resins.

Materials and methods

Materials

The dimethacrylate (BisGMA 50% [bisphenol A-glycidyl dimethacrylate] and TEGDMA 50% [triethylenglycol dimethacrylate]) monomer resin system and radiopaque fillers of $BaAlSiO_2$ (0.4 µm in size) (UltraFine, Schott, Landshut, Germany) were used. Nanofillers (SiO_2, 180 nm in size) with various weight fractions (NanoFine, Schott) were incorporated into the resin system. Both types of fillers were received silanated from the manufacturer.

Preparation of the experimental composite resins

Experimental composite resins were prepared by mixing 29 wt.% of resin matrix with 71 wt.% of $BaAlSiO_2$ radiopaque fillers. Then, various weight fractions of nanofillers (0, 10, 15, 20, 25, 30 and 35 wt.%) were added gradually to the mixture. Mixing was carried out using a high-speed mixing machine for 4 minutes at 3,500 rpm (SpeedMixer DAC, Hauschild, Hamm, Germany).

All groups had the same resin matrix and a consistent fraction of microfillers but varied in their nanofiller fractions (resulting in different total filler fractions). Composite resins were transferred from mixing cups to syringes and mixed in a centrifuge (SPR centrifuge, Beckman Coulter, Brea, CA, USA) until all entrapped air was eliminated.

Flexural strength and modulus

Specimens of specific dimension ($2 \times 2 \times 25$ mm^3) were prepared for a three-point bending test from all evaluated composites. By using a half-split stainless-steel mould and transparent Mylar sheets, bar-shaped specimens were prepared. Light curing of dental composite was performed using a hand light-curing unit (Elipar DeepCure-L, 3M, St Paul, MN, USA) for a duration of 20 seconds in four different parts through metal moulds on both sides. The light tip of the curing device was 1 mm away from the composite. The light intensity was 1600 mW/cm^2, with a wavelength ranging from 400 to 480 nm (Marc Resin Calibrator; BlueLight Analytics, Halifax, NS, Canada). Before testing, the specimens of each group (n = 8) were kept dry for 2 days in an incubator (37°C). According to ISO 4049, a three-point bending test was performed (test span 20 mm, crosshead speed 1 mm/min, indenter 2 mm diameter, load cell 2500 N). Using a material-testing machine (model LRX, Lloyd Instruments, Fareham, UK), all specimens were loaded, and software was used to record the load-deflection curves (Nexygenf4.0, Lloyd Instruments).

Flexural strength (σf) as well as flexural modulus (Ef) were measured according to equations:

$\sigma f = 3FmI /(2bh^2)$

$Ef = SI^3 /(4bh^3)$

Where Fm is the load applied (N) at the maximum point of the load-extension curve, I is the length of span (20 mm), b is the width of the test specimens and h represents the thickness of the test specimens. S is the stiffness (N/m) S = F/d where d represents the deflection that corresponds to load F at a particular point along the straight-line portion of the trace.

Degree of monomer conversion

Fourier transform infrared (FTIR)-spectroscopy was employed using an attenuated total reflectance (ATR) accessory (Spectrum One, Perkin-Elmer, Beaconsfield, UK) for the quantification of carbon-carbon double bond conversion (DC%) both before and after photoinitiation of the polymerisation process. The assessment was conducted within a mould with a thickness of 1.5 mm and a diameter of 4.5 mm, encompassing the analysis of composite resins. The initial step involved the measurement of the spectrum of the non-polymerised specimen. Subsequently, the composite resin underwent polymerisation using a manual light-curing unit (Elipar DeepCure-L), with irradiation conducted through an upper glass slide for 40 seconds. Following the irradiation process, the FTIR spectrum of the specimen was subjected to scanning, then DC% was measured from the aliphaticfC = C peak at 1638 cm^{-1} and normalised against the aromaticfC = C peak at 1608 cm^{-1} following this formula:

$$DC\ \% = (1 - \frac{C_{aliphatic}\ /C_{aromatic}}{U_{aliphatic}\ /U_{aromatic}})$$

Where the C aliphatic is the absorption peak at 1638 cm^{-1} and C aromatic the reference peak of the polymerised specimen, whereas U aliphatic represents the absorption peak at 1638 cm^{-1} and U aromatic the reference peak of the unpolymerised specimen. Five trials were run for each experimental composite resin.

Surface gloss

Disk-shaped specimens (15 mm diameter and 2 mm thickness) were fabricated from each material using a metal mould (n = 5). One side of the specimen surface facing the mould was polished with 4,000-grit paper, and this side was named the polished side. The other side of the specimen facing the glass slide and mylar strip remained unpolished and was named the unpolished side. After polishing, specimens were rinsed with water and stored in dry conditions at room temperature before testing. The surface gloss was measured at an incidence angle of 60 degrees, using a calibrated infrared Glossmeter (Zehntner Testing Instruments, Germany) with a square measurement area of 6 x 40 mm. The mean of four measurements was recorded per surface.

Surface microhardness

The surface microhardness of each group was measured using the same polished disk-shaped specimens (n = 5) and a Duramin hardness microscope (Struers, Copenhagen, Denmark) equipped with a 40x objective lens. A load of 1.96 N was applied for 15 seconds to each specimen, with five indentations made on the surface of each one. The diagonal lengths of the impressions were measured, and the Vickers hardness values were automatically converted into microhardness values by the machine. Microhardness was calculated using the following equation:

$$H = \frac{1854.4 \times P}{d^2}$$

where H is Vickers hardness in kg/mm^2, P is the load in grams and d is the length of the diagonals in μm.

Two-body wear

The two-body wear test was applied according to previous studies[16,17] to demonstrate the wear resistance of each composite. Two polished specimens (20 mm length × 10 mm width × 3 mm depth) of each composite were prepared in acrylic resin block. All specimens were immersed in distilled water at a temperature of 37°C for 24 hours prior to testing. Wear assessments were conducted using a chewing simulator (CS-4.2, SD-Mechatronik, Feldkirchen-Westerham, Germany), consisting of two distinct chambers designed to replicate vertical and horizontal masticatory movements sequentially, while operating within an aqueous environment. Each chamber composed of a lower sample holder for specimen insertion and an upper loading tip serving as the counteracting element to the specimens under examination. An upper antagonist, in the form of a steatite ball with a diameter of 6 mm, was employed. For each specimen, a total of 15,000 simulated chewing cycles were executed at a frequency of 1.5 Hz, employing a vertical load of 2 kg to emulate a chewing force of 20 N. Subsequent to the simulations, wear patterns were assessed via a 3D optical profilometer (ContourGT-I, Bruker Nano, Tucson, AZ, USA). The mean wear depth (in μm) was calculated based on the examination of the deepest points among six wear profiles from each group, representing a comprehensive measure of wear resistance (Fig 1).

Microstructure analysis

To evaluate the microstructure of the investigated composites, a scanning electron microscopy (SEM) (LEO, Oberkochen, Germany) was used. Polished specimens (n = 2) from the experimental composites (0 and 35 wt.%) were kept dry in a desiccator for 24 hours. After that, specimens were gold coated in a vacuum evaporator utilising a sputter coater (SCD 050 Sputter Coater, BAL-TEC, Balzers, Liechtenstein) prior to SEM inspection.

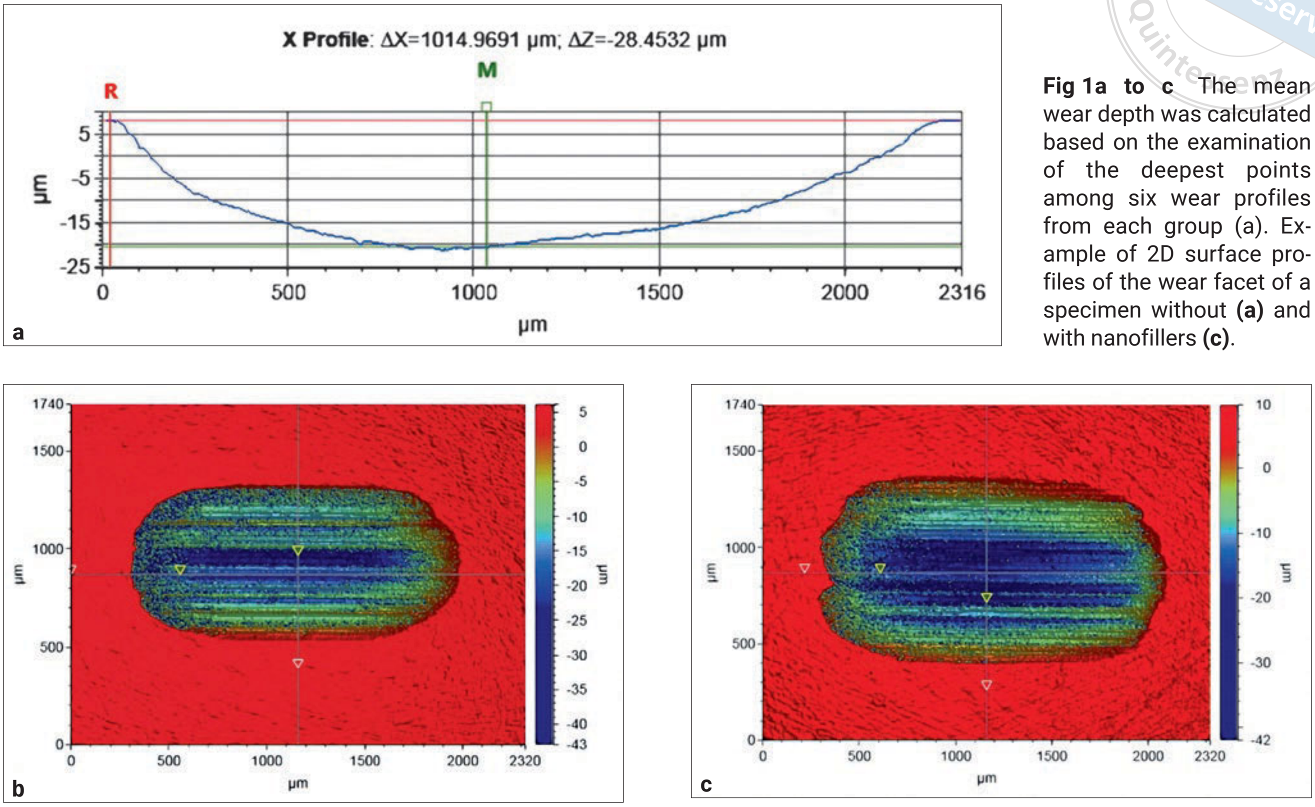

Fig 1a to c The mean wear depth was calculated based on the examination of the deepest points among six wear profiles from each group (a). Example of 2D surface profiles of the wear facet of a specimen without **(a)** and with nanofillers **(c)**.

Statistical analysis

Analysis of variance (ANOVA) with the level of significance set at 0.05 was employed to statistically analyse the data with SPSS version 23 (IBM, Armonk, NY, USA). The results were primarily assessed using a Levene test to evaluate equality of variances. Tukey HSD post hoc analysis was used to ascertain the variations between tested groups. The Pearson correlation coefficient was calculated to examine the relationship between the investigated material properties and the nanofiller weight percentage.

Results

The results presented in Table 1 indicate that the fraction of nanofillers had a significant effect ($P < 0.05$) on flexural modulus (positive correlation, $R^2 = 0.8$), degree of conversion (DC%) (negative correlation, $R^2 = 0.9$), microhardness (positive correlation, $R^2 = 0.9$), gloss (negative correlation, $R^2 = 0.8$) and wear depth (negative correlation, $R^2 = 0.9$). However, the addition of nanofillers did not correlate with flexural strength values ($R^2 = 0.2$).

The group without nanofillers exhibited the highest DC% (56.6%), gloss after polishing (76.2 GU) and wear resistance (24.2 µm). In contrast, the group with 35 wt.% nanofillers showed the highest flexural modulus (9 GPa) and microhardness (70 VH).

The polishing protocol used in this study (4,000 grit) reduced the surface gloss of the composite surfaces compared to the unpolished surfaces polymerised against a mylar matrix strip ($P < 0.05$). Nanofillers had no effect on the gloss values of the unpolished specimens.

Figure 1 illustrates a typical wear facet profile of specimens with and without nanofillers. The addition of nanofillers increased both the wear depth values and the size of the wear facet area. Moreover, SEM images (Fig 2) provide further insight into the structural differences, showing that nanofillers effectively filled the spaces between micro-sized fillers, resulting in a more uniform material. Specimens without nanofillers, on the other hand, exhibited greater porosity, suggesting that these unfilled gaps could negatively impact certain material properties.

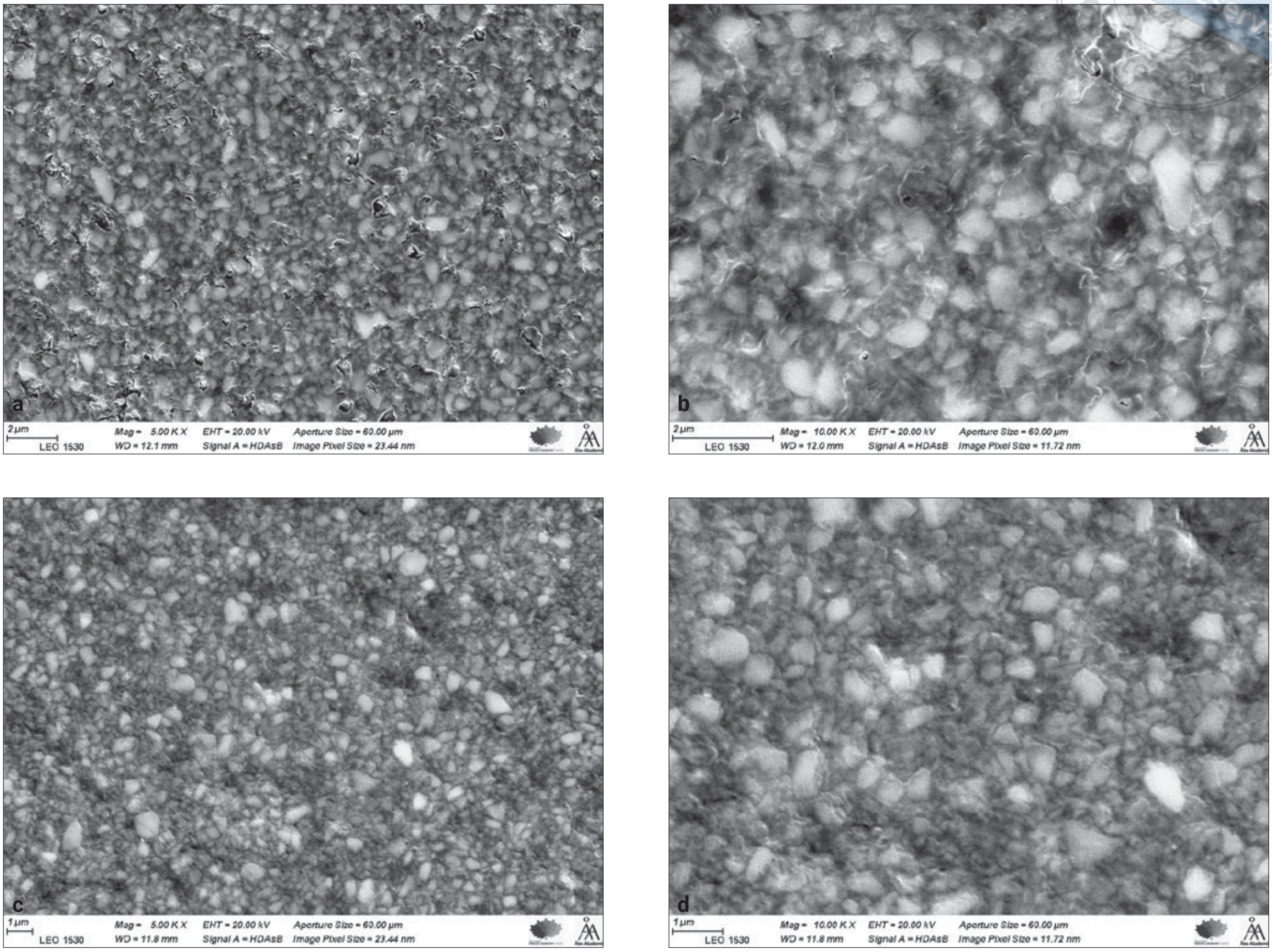

Fig 2a to d SEM images at different magnifications of the group without nanofillers **(a and b)**, showing some porosity, and the group with 30 wt.% nanofillers **(c and d)**, where the nanofillers are filling the spaces between the micro-sized fillers.

Discussion

The use of inorganic particles as a reinforcement method for polymer-based materials is well-documented, with various studies showing that incorporating nanofillers into polymer matrices enhances multiple properties of the resulting composites.[18] In the present study, the effect of adding nanofillers on the properties of microfilled composite resin was explored by testing eight experimental composite groups, each containing varying amounts of nanofillers. The parameters were assessed in accordance with the dental standard ISO 4094 or other reliable testing methods.[19-21] Despite the experimental resin-based materials not being fully optimised, the results indicated that the selected formulations fell within the property range of standard commercial products.[21-23]

The findings demonstrate the significant influence of nanofiller content on the mechanical and surface properties of the microfilled composite resins tested. Specifically, the positive correlation between the fraction of nanofillers and the flexural modulus ($R^2 = 0.8$) and microhardness ($R^2 = 0.9$) suggests that increasing the nanofiller content enhances the stiffness and surface hardness of the microfilled composite resin. Consistent with previous studies, the combination of micro- and nano-sized fillers allows for denser packing, which in turn increases the filler volume fraction of the composite resins.[7,24,25]

The flexural strength results ($R^2 = 0.2$) indicate that there is no significant correlation between the addition of nanofillers and the flexural strength of the composite. Although an initial increase in strength was observed at low nanofiller loadings (5 wt.%), a significant decrease in strength occurred at higher filler loadings (Table 1). This suggests that while small amounts of nanofiller may enhance flexural strength, further increasing the filler content beyond a certain threshold

Table 1 Results, mean ± standard deviation (SD).

Group	Flexural strength (MPa)	Flexural modulus (GPa)	DC%	Surface gloss (GU)		Hardness (VH)	Wear (µm)
				Unpolished	Polished		
0 wt.%	135 ± 19^{a}	7.7 ± 0.6^{a}	56.6 ± 0.6^{a}	90.8 ± 0.5^{a}	76.2 ± 2.3^{a}	56.1 ± 5.0^{a}	24.2 ± 1.4^{a}
5 wt.%	158 ± 20^{b}	8.2 ± 0.3^{a}	56.4 ± 0.4^{a}	90.9 ± 2.0^{a}	74.9 ± 2.6^{a}	61.0 ± 3.0^{a}	25.9 ± 1.8^{b}
10 wt.%	139 ± 18^{a}	8.2 ± 0.1^{a}	55.0 ± 0.5^{b}	90.7 ± 1.2^{a}	75.8 ± 3.5^{a}	61.2 ± 3.0^{b}	26.4 ± 1.9^{c}
15 wt.%	144 ± 19^{c}	8.6 ± 0.2^{b}	54.1 ± 0.6^{b}	90.0 ± 1.5^{a}	75.6 ± 3.4^{a}	61.2 ± 2.0^{a}	27.4 ± 1.8^{d}
20 wt.%	149 ± 25^{d}	9.0 ± 0.5^{c}	54.4 ± 0.2^{b}	89.1 ± 0.6^{a}	68.5 ± 1.3^{b}	65.2 ± 2.6^{c}	27.5 ± 1.3^{d}
25 wt.%	146 ± 22^{e}	8.6 ± 0.5^{c}	53.8 ± 0.3^{c}	90.6 ± 1.4^{a}	68.2 ± 1.2^{b}	67.0 ± 2.4^{c}	30.0 ± 2.1^{e}
30 wt.%	130 ± 11^{f}	8.9 ± 0.5^{d}	52.2 ± 0.8^{d}	91.0 ± 0.7^{a}	68.9 ± 2.2^{b}	67.9 ± 4.4^{d}	28.9 ± 1.6^{f}
35 wt.%	121 ± 12^{g}	9.0 ± 0.4^{d}	51.4 ± 0.5^{d}	89.1 ± 0.9^{a}	65.5 ± 0.1^{c}	70.4 ± 3.5^{d}	31.0 ± 1.7^{g}

The same superscript letter after the values indicates groups that were statistically similar ($P > 0.05$).

negatively impacts this property. This finding aligns with reports in the literature on particulate-filled composite resins, where increasing nanofiller loading can counteract the expected reinforcement due to a higher concentration of particles.[25,26] The observed decrease in strength is likely due to increased mechanical failure at the interface between the resin matrix and the inorganic fillers.[27] In other words, the wettability of the nanofillers is reduced. Conversely, the negative correlation between nanofiller content and DC% ($R^2 = 0.9$), gloss ($R^2 = 0.8$) and wear depth ($R^2 = 0.9$) indicates that higher concentrations of nanofiller may hinder the polymerisation process, reduce the surface gloss and increase the susceptibility to wear. The reduction in DC% observed in groups with higher nanofiller content may be attributed to the increased viscosity of the composite resin, which restricts monomer mobility and diminishes the efficiency of the curing process. The inclusion of nanofillers can increase the refractive index and reduce the extinction coefficient, which could hinder the polymerisation of monomers.[28] Additionally, the formation of a dense or highly filled structure (Fig 2) might impede light penetration or cause light scattering, particularly when the filler particle size is close to the wavelength of the light from the curing unit.[29,30] As a result, increased scattering reduces light intensity, further impacting the curing process.

Gloss refers to a surface's ability to reflect light, with high gloss typically indicating a smooth restoration surface.[31] In the present study, the gloss values of the composite surface polymerised against a Mylar strip (unpolished surface) were higher than those obtained after polishing. Similar findings have been reported in other studies.[32,33] Although light curing against a Mylar sheet produces a smoother surface, restorations usually require finishing to remove excess material and recontour the surface, which decreases smoothness and necessitates further polishing. The observed reduction in gloss, particularly after polishing, suggests that composites with higher nanofiller content may exhibit less reflectivity, likely due to increased surface roughness and the loss of filler particles during the polishing process.[33]

These results contrast with the findings of Valente et al,[34] who reported that nanofilled composites (mean particle size Ø 175 nm) retained higher surface gloss both before and after brushing, suggesting that smaller inorganic fillers can better maintain aesthetic properties under oral conditions. However, Kaizer et al[35] noted that there is no in vitro evidence to favour nanofilled composite resins over traditional microfilled composites in terms of superior surface smoothness or gloss.

The wear facet profiles shown in Fig 1 provide further insight into the effect of nanofillers on wear performance. The increased wear depth and facet area observed in the specimens containing nanofillers suggest that, despite enhancing certain mechanical properties, nanofillers can also make the composite more vulnerable to wear under specific conditions. This outcome is not unexpected, as previous studies have shown that flowable composite resins with lower filler content often exhibit better wear resistance than packable, highly filled composites.[36,37] This may be partly attributed to the elastic deformation of the low-viscosity composite matrix, which offers some shock-absorbing properties.[23,36] The literature presents mixed results: some studies report a positive[38,39] or negative correlation[23,37] between surface hardness and wear resistance, whereas others found no relationship.[21,40] Bayne et al[41] emphasise that filler quantity is less critical than how it is distributed, with inter-particle spacing being a key factor in protecting the composite surface. However, the wear of composite materials remains a complex process, and no definitive conclusions have been reached in the literature so far.

It is important to highlight that the nanofillers in the present study were well distributed throughout the matrix, as no agglomeration was observed in the SEM images (Fig 2). This uniform distribution is most likely attributable to the silane treatment.

Conclusion

In summary, the incorporation of nanofillers into composite materials presents a complex balance of benefits and drawbacks. While nanofillers enhance flexural modulus and microhardness, they may also reduce the DC%, gloss and wear resistance. These findings emphasise the importance of carefully optimising nanofiller content in composite formulations to achieve the desired combination of mechanical strength, surface quality and durability for specific applications. Further studies are needed to explore the mechanisms underlying these effects and to identify strategies for mitigating the negative impacts of nanofillers on composite performance.

Conflicts of interest

The authors declare no conflicts of interest related to this study.

Author contribution

Dr Enni PARPO contributed to the data collection/analysis and manuscript draft; Dr Lippo LASSILA contributed to the study design, data analysis/interpretation and manuscript revision; Dr contributed to the manuscript editing and revision; Dr Sufyan GAROUSHI contributed to the study design, manuscript writing and revision.

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