

Light curability and optical properties of boron nitride nanosheet reinforced dental resin-based composite

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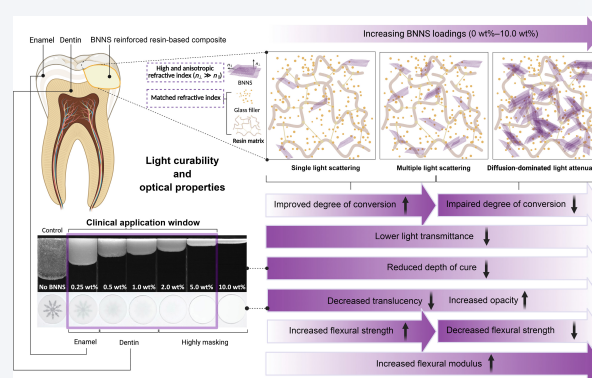
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ABSTRACT: Boron nitride nanosheets (BNNS), also known as "white graphene", combine a two-dimensional morphology with a high and anisotropic refractive index (RI), enabling strong and direction-dependent light-nanofiller interactions. While offering multifunctional reinforcement, BNNS also alter photon transport during photopolymerisation of dental resin-based composites (RBCs). Here, experimental RBCs containing increasing BNNS loadings (0 wt.%–10.0 wt.%) together with RI-matched glass fillers were formulated to elucidate how BNNS-induced optical anisotropy and RI mismatch govern light propagation, curing efficiency, and macroscopic optical properties. BNNS were characterised by scanning electron microscopy (SEM), atomic force microscopy (AFM) and dynamic light scattering (DLS).

The RI of fillers and resin were measured optically. The light curability and optical performance of BNNS-reinforced RBCs, including light transmittance, degree of conversion (DC), depth of cure (DoC), translucency, flexural strength (FS), flexural modulus (FM), and fracture microstructure were evaluated. BNNS exhibited a platelet-like morphology with a high aspect ratio and a substantially higher RI than the surrounding RBCs components, generating orientation-dependent scattering and refraction. Increasing BNNS loading with deteriorated dispersion and increased agglomeration altered the light-scattering regime, reduced photon flux at depth, resulting in decreased transmittance, DoC, and translucency while increasing FM. At low loadings, partial scattering enhanced local light distribution, producing peak DC and FS at 0.5 wt.%, whereas higher BNNS caused severe optical attenuation, drastically reduced DC and slightly decreased FS at 10.0 wt.%. These findings identify potential ranges for BNNS loadings, tailored to applications for repair of enamel, dentin replacement or masking. These established constraints for formulation of light-curable tooth-coloured nanocomposites.

KEYWORDS: boron nitride nanosheets, resin-based composite, light transmittance, degree of conversion (DC), depth of cure (DoC), translucency, mechanical properties



1 Introduction

Resin-based composites (RBCs) have developed considerably over the past six decades to become the most widely used direct restorative materials in modern dentistry [1, 2]. This rigid system

consists of an organic polymer matrix reinforced with filler particles and undergoes either chemical or photo-initiated polymerisation [3]. Extensive research has focused on improving filler technology to achieve superior clinical outcomes and simplified handling procedures compared with traditional amalgam restorations [4, 5]. Nevertheless, continuing advances in nanotechnology have created new opportunities to formulate novel RBCs with enhanced physicochemical and clinical performance.

Graphene family materials (GFMs) have been widely introduced into RBC systems as reinforcing filler [6], demonstrating notable enhancements in mechanical properties, tissue remineralisation,

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biocompatibility, and antibacterial efficacy [7–10]. However, owing to their intrinsic grey-to-black shade, their dental application especially as tooth-coloured restorative materials, is significantly restricted by aesthetics even at minimal filler loadings [6, 11].

Hexagonal boron nitride (h-BN) nanosheets (BNNS), also known as "white graphene", share a similar hexagonal layered lattice structure with graphene, but consist of alternating boron (B) and nitrogen (N) atoms instead of carbon [12]. These nanosheets are characterised by atomic thickness down to the nanometre scale and lateral dimensions that can vary from tens of nanometres to several micrometres [13]. From monolayered h-BN, various derivatives can be formed, including 0D fullerenes or nanospheres, 1D nanotubes (BNNT), nanoscrolls or nanoribbons, and 2D nanosheets (BNNS) [14, 15]. Since their discovery, these low-dimensional nanomaterials have attracted global attention as functional fillers in dentistry and beyond due to their distinctive atomic-level structure, outstanding physicochemical reinforcement, remineralisation potential, biocompatibility, antibacterial activity, and favourable aesthetics [10, 16, 17].

Pioneering studies investigating h-BN incorporation into resin-based dental composites have emerged since 2017. A wide range of resin systems have been examined, including bisphenol A-glycidyl methacrylate (Bis-GMA)/triethylene glycol dimethacrylate (TEGDMA) [18, 19], bisphenol A ethoxylate dimethacrylate (Bis-EMA)/urethane dimethacrylate (UDMA) resins [20], Bis-GMA/2-hydroxyethyl methacrylate (HEMA) adhesives [21–23], epoxy resin [24], polymethyl methacrylate (PMMA)-based denture materials [25–27], 3D-printed indirect restorations [28], and resin-based ceramics [29]. Despite these promising developments, the influence of BNNS on the light-curing behaviour and optical properties of RBCs remain insufficiently understood. Most previous studies typically explored relatively low h-BN loadings (≤ 1 wt.%) without optimising formulations or mixing strategies to balance mechanical reinforcement with optical aesthetics targeting real-world clinical needs. Furthermore, except for chemically polymerised systems such as PMMA-based resins [25–27], most of RBCs are light-cured systems, yet the impact of BNNS incorporation on photo-polymerisation mechanism and clinically relevant optical properties has received limited attention.

2D BNNS can be obtained either by exfoliating bulk h-BN crystals (3D) into mono- or multi-layered nanosheets via top-down methods [30] or synthesised through bottom-up approaches such as chemical vapour deposition (CVD) with boron and nitrogen precursors [31]. The top-down method [30] was firstly achieved shortly after the first report of graphene [32] in Manchester in 2005. Among h-BN derivatives, 2D BNNS exhibit unique optical features: (i) a white colour, (ii) high and anisotropic refractive index [33], and (iii) minimal absorption (optically transparent) in the blue and violet wavelength range commonly emitted by dental light curing units (LCUs), attributed to their ultrawide ~ 5.9 eV band gap [8]. Those optical features suggest that BNNS incorporation and dispersion within the polymer may critically influence filler-matrix interactions, especially the curing efficiency during photo-polymerisation, and consequently affect the final aesthetic appearance of the restorations.

In aesthetic restorative dentistry, close optical matching between filling materials and natural tooth structures (enamel and dentin) is crucial [34]. Human enamel, being the more translucent outer layer, contrasts with the opaquer dentin underneath [35]. The translucency of RBCs is influenced by several factors, including the

refractive index mismatch between filler and resin matrix [36–38], and filler morphology, size distribution and loading [39]. Commercial RBCs often incorporate minimal amount of metal oxides as opacifiers to modulate translucency and masking ability [40], particularly in cases requiring certain level of colour coverage and concealment of discolouration [3]. Translucency is commonly quantified through translucency parameter (TP_{ab} and TP_{00}) or contrast ratio (CR) [41], providing a basis for matching natural tissues such as human or bovine teeth with commercial restorative products.

Currently, studies investigating BNNS mechanical reinforcement of methacrylate-based light-curable dental composites have reported inconsistent findings. No significant improvements in FS and FM were observed at BNNS loadings up to 0.5 wt.%–0.8 wt.% when hand-mixing methods were incorporated [18, 20]. In contrast, improvements in compressive strength (CS) and Vickers hardness (HV) have been reported at similar 0.8 wt.% loading [20]. More pronounced enhancement has been observed in hybrid BNNS nanofiller systems, such as BNNS/TiO₂ nanocomposites and BNNS/dimethylamino hexadecyl methacrylate (DMAHDM) [19, 20]. These contrasting results suggest that the reinforcing mechanism of BNNS is highly dependent on nanomorphology, loading level, dispersion quality, and mixing methods. Consequently, further investigation is required to explore the balance among BNNS incorporation, optical performance, and mechanical behaviour in clinically relevant RBC formulations.

Therefore, the objective of this study was to systematically investigate the effects of nano-layered BNNS incorporation on the light curability, optical properties, and mechanical performance of experimental flowable RBCs. Unlike previous studies that primarily examined low BNNS loadings, this work investigated BNNS loadings up to 10.0 wt.% to determine the feasibility of BNNS addition as supplementary reinforcing fillers for potential clinical applications. Specifically, the effects of BNNS incorporation on light transmittance, degree of conversion, depth of cure, translucency, flexural behaviour, and fractural morphology were assessed. The null hypotheses were as follows: (1) BNNS incorporation does not impact the light curability including light transmittance, degree of conversion and depth of cure; (2) BNNS addition does not alter the translucency of the light-curable RBCs; and (3) BNNS loading does not enhance the flexural properties.

2 Experimental

The materials used for experimental resin composites formulation are listed in Table S1 in the Electronic Supplementary Material (ESM). A boron nitride nanosheet powder (Hexotene-1, Versarien, Longhope, UK) was incorporated in this study. It was produced by similar mechano-chemical exfoliation of synthetic bulk h-BN crystals as in previous work from the National Graphene Institute, the University of Manchester [13].

Experimental resin composites were formulated by pre-dispersing varying BNNS loadings (0.25 wt.%, 0.5 wt.%, 1.0 wt.%, 2.0 wt.%, 5.0 wt.% and 10.0 wt.%) into a 50 wt.%:50 wt.% Bis-GMA:TEGDMA monomer mixture via 30 min ultrasonication. Each formulation was then further SpeedMixed with silanised barium silicate glass fillers (40 wt.%) and the photo initiation system (0.25 mol% camphorquinone (CQ), 0.5 mol% diphenyliodonium hexafluorophosphate (DPI) and 0.5 mol% ethyl 4-dimethylaminobenzoate (EDMAB)). To minimise air bubble

entrapment, the mixtures underwent vacuum degassing. The control group was formulated without BNNS incorporation. Specimens of all groups were freshly loaded and light-cured after resin formulation.

2.1 Filler particle characterisation

BNNS and glass powder were each separately dispersed into isopropanol (IPA) at 0.05 mg/mL via 30 min ultrasonication. Specimens ($n = 1$) of each powder were air-dried and sputter coated with a 10 nm gold-palladium layer (Au:Pd = 80:20). SEM images were obtained using a Tescan Mira3 SC field-emission SEM (TESCAN-UK, Huntingdon, UK) at an acceleration voltage of 5 kV and magnifications at 2000 $\times$ and 20,000 $\times$ to investigate the powder morphology on a silicon wafer. Atomic force microscopy (AFM) was used to determine the mean thickness of BNNS via a JPK Nanowizard 3 AFM (Bruker Nano, Berlin, Germany) operated in alternating current (AC) mode with a Tap300Al-G-10 probe. Thickness measurements were taken from 100 randomly selected particles. Particle size distribution was analysed using the BNNS-IPA dispersion ($n = 3$) via a Zetasizer Nano DLS (Malvern Instruments, Malvern, UK).

2.2 Refractive index measurement

The RI of the 50 wt.%.50 wt.% Bis-GMA:TEGDMA monomer mixture was measured using an Abbe refractometer (Bellingham & Stanley, Nottingham, UK) with sodium D line, 589 nm at 25 °C. The RI of the light-cured resin matrix and the barium silicate glass filler (manufacturer's specification: 1.53) were subsequently measured using the Becke-line method with refractive index liquid sets (Cargille Laboratories, Cedar Grove, NJ, USA) under a microscope (Revolve, ECO, San Diego, CA, USA).

2.3 Light curing unit (LCU) characterisation

A poly-wave Bluephase G4 LCU (Ivoclar Vivadent, Schaan, Liechtenstein) was used in high power (H)-mode to provide a 20 s exposure condition. The mean irradiance (mW/cm²), radiant exposure (J/cm²), and absolute spectral irradiance (mW/(cm²·nm)) were characterised at zero distance to the surface detector of a spectrophotometer (MARC™ Light Collector, BlueLight Analytics, Halifax, Canada).

The 2D and 3D beam profiles of the LCU tip were analysed using a wide beam imager (SP90612, Ophir-Spiricon, North Logan, USA) at 0 distance to the diffusive plate. Stray ambient light was removed digitally using the Ultracal baseline calibration in BeamGauge software (version 6.16, Ophir-Spiricon, North Logan, USA) prior to the LCU irradiance measurement. The "Top Hat" factor (THF), which represents the extent of uniformity across the beam profile, was also measured.

2.4 Transmittance of LCU wavelength during irradiation of RBC

The light transmittance through disc specimens ($n = 3$) of the experimental RBC paste were measured during the light irradiation and photopolymerisation process. This was achieved by placing the uncured RBC into polytetrafluoroethylene (PTFE) moulds (4 mm inner diameter $\times$ 1 mm thickness) with Mylar strips (DuPont Teijin Films, Chester, VA, USA) on both top and bottom surfaces. During the irradiation from the Bluephase G4 in high power (H) mode (irradiance 1200 mW/cm² $\pm$ 10%, according to the manufacturer's specifications) for 20 s on the top surface of specimens at zero

distance, the transmitted irradiance through the lower disc surface was recorded at the bottom surface detector of the same MARC™ Light Collector. The sensor trigger threshold was set at 5 mW.

The transmittance (T) was calculated as a function of time (from 0 to 20 s) using Eq. (1):

$$T(\%) = \frac{I_t}{I_0} \times 100\% \quad (1)$$

where I_0 is the irradiance of the incident light, measured as readings from light passing through the empty mould and two layers of Mylar strips, and I_t is the transmitted light irradiance collected with the RBC disc present. As a result, the transmittance values were determined as a function of time over the measurement period. Additionally, irradiance was measured as a spectrum, i.e., as a function of wavelength across the spectrum (360–540 nm) at each time point.

To compare effect of BNNS loading of RBCs on the violet (360–420 nm), blue (420–520 nm), and violet+blue (360–520 nm) spectral irradiance, respectively, the irradiance spectra from the LCU and delivered to the bottom of specimens were analysed using the area under curve (AUC) by GraphPad Prism (Version 10.3.1 for Mac, GraphPad Software, Boston, MA, USA).

2.5 Degree of conversion

Formulated resin composites ($n = 3$) were loaded into cylindrical PTFE moulds (5 mm diameter $\times$ 1 mm thickness) in contact with the attenuated total reflectance (ATR) crystal of the Fourier-transform infrared (FTIR) spectrometer (ALPHA II, Bruker Optik, Billerica, MA, USA). Each specimen was then cured with Bluephase G4 at H-mode for 20 s. Real-time 24 h FTIR spectra between 400 and 4000 cm⁻¹ were recorded using 32 scans at a resolution of 4 cm⁻¹. DC at 24 h post-curing (DC_{24h}) was calculated using Eq. (2):

$$DC(\%) = \left[1 - \frac{\left(\frac{C=C_{\text{aliphatic}}}{C=C_{\text{aromatic}}} \right)_{\text{cured}}}{\left(\frac{C=C_{\text{aliphatic}}}{C=C_{\text{aromatic}}} \right)_{\text{uncured}}} \right] \times 100 \quad (2)$$

where the absorption peak height of the aliphatic C=C at 1638 cm⁻¹ is used as the analytical frequency and the absorption peak height of the aromatic C=C from Bis-GMA at 1608 cm⁻¹ as an internal standard [42].

2.6 Depth of cure

The uncured RBC pastes from each group ($n = 3$) were slightly overloaded to a custom-made split stainless-steel mould with a rectangular cuboid slot (16 $\times$ 8 $\times$ 2 mm). Top and bottom were covered with Mylar strips and glass slides, and pressed to remove excess materials. The incident irradiance was above the top surface Mylar strip from a Bluephase G4 LCU on H-mode for 20 s. All post-irradiance specimens were incubated for 24 h at 37 °C.

HV mapping on the specimen was carried out with an auto hardness tester (DuraScan 80, ZwickRoell, Ulm, Germany). The specimen was aligned under the overview camera and then mapped with three rows (row gap = 2.5 mm) of HV indentation with an interspace of 0.5 mm under HV 0.3 mode (300 gf load, 15 s dwell time). The indentation was then evaluated under a 20 $\times$ objective lens (Fig. 1) and calculated automatically using the equations below (Eqs. (3) and (4)):

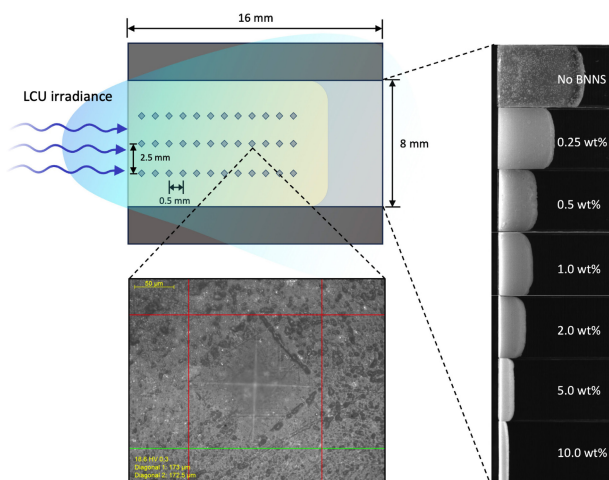


Figure 1 DoC measurement of 0 wt.%–10 wt.% BNNS-reinforced RBCs using HV mapping at successive 0.5 mm depths (left). Representative overview camera images of specimens from all testing groups (right).

$$HV = 0.1891 \times \frac{F}{d^2} \quad (3)$$

$$d = \frac{d_1 + d_2}{2} \quad (4)$$

where F is the test load (kgf), d is the mean diagonal length (d_1 and d_2) of an indentation (mm).

The HV mapping data were plotted as depth vs. hardness curves. The maximum HV ($HV_{\max}$) was identified for each group, and from this, the corresponding 80% $HV_{\max}$ was calculated. DoC, i.e. the depth at which the HV dropped to 80% of $HV_{\max}$, was defined based on that a reduction to 80% of $HV_{\max}$ into the depth of the RBC is considered clinically acceptable [43–45].

2.7 Translucency to a white light spectrum

Disc-shaped specimens (10 mm diameter, 1 mm thickness) of each group ($n = 5$) were prepared and incubated for 24 h at 37 °C prior to testing. Translucency parameters (TP_{ab} and TP_{00}) and CR were measured using a sphere reflectance spectrophotometer (Ci7630, X-Rite, Grand Rapids, MI, USA) with an aperture size of 6 mm. Measurements were based in the Commission Internationale de l'Éclairage (CIE) colour scale under a standard D65 illuminant, using a standard white background (CIE $L^* = 98.12$, $a^* = -0.29$, $b^* = 3.22$) and a black trap (CIE $L^* = 0.03$, $a^* = 0.00$, $b^* = -0.02$).

TP_{ab} was calculated using the CIELAB equation below (Eq. (5))

$$TP_{ab} = \sqrt{(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2} \quad (5)$$

where the subscript "W" refers to colour coordinates measured over the standard white background, and "B" refers to those over the black trap.

TP_{00} was the colour difference calculated using CIEΔE2000 formula by combining the differences of specimens over back trap and white background in lightness ($\Delta L'$), chroma ($\Delta C'$), and hue ($\Delta H'$) along with their weighting functions as below (Eq. (6))

$$TP_{00} = \sqrt{\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{K_C S_C}\right) \left(\frac{\Delta H'}{K_H S_H}\right)} \quad (6)$$

where these functions adjust the chroma and hue differences based on the colours being compared, accounting for the non-uniformity of human colour perception.

The CR, ranging from 0 to 1, was defined as the ratio of the spectral reflectance of the specimens over a black trap (Y_B) and a standard white background (Y_W), as in Eq. (7). A CR value closer to 0 indicates higher translucency.

$$CR = \frac{Y_B}{Y_W} \quad (7)$$

All the above data were obtained from Color iQC software (version 10.8.1; X-rite, Grand Rapids, MI, USA).

2.8 Flexural strength and flexural modulus

Flexural strength (FS) and flexural modulus (FM) were measured via a universal testing machine (Z020, Zwick/Roel, Leominster, UK) in accordance with International Organization for Standardization (ISO) Standard 4049.

A rectangular PTFE mould (internal size 25 × 2 × 2 mm) was used for specimen preparation ($n = 5$). All the specimens were stored in deionised water for 24 h at 37 °C before testing. The distance between the supports (L) was 20 mm and the centre of the specimens was placed to align with the tip of the crosshead. The specimens were loaded with a 0.5 kN load cell at a crosshead speed of 0.75 mm/min until fracture.

FS was calculated using Eq. (8)

$$\sigma_f = \frac{3FL}{2bh_{st}^2} \quad (8)$$

where F is the load at fracture (N), L is the distance between the supports (mm), b is the specimen width (mm), and h_{st} is the sample thickness (mm).

To determine FM, the load/displacement curve generated by FS measurement was used. FM was calculated by Eq. (9)

$$E_f = \frac{L^3 F}{4bh^2 d_{def}} \quad (9)$$

where F is the load at fracture (N), L is the distance between the supports (mm), b is the specimen width (mm), h is the height of the sample, and d_{def} is the deflection (mm).

2.9 Cross-sectional microstructure

BNNS distribution on the cross-sectional fractured surfaces of RBCs were characterised using a Tescan Mira3 SC field-emission SEM (TESCAN-UK, Huntingdon, UK) with a secondary electron (SE) detector at an acceleration voltage of 8 kV and magnification at 5000×. All specimens were sputter coated with a 10 nm gold-palladium layer (Au:Pd = 80:20).

2.10 Statistical analysis

Sample size calculation was conducted by G*POWER software (version 3.1.9.6; Heinrich-Heine University, Duesseldorf, Germany), with $\alpha = 0.05$, power ($1 - \beta$) = 80%, based on data from a pilot study. All data were assessed for normality using the Shapiro–Wilk test or Kolmogorov–Smirnov tests. One-way ANOVA followed by Tukey's post hoc multiple comparisons test was performed to compare the differences among the test groups using GraphPad Prism (version 10.3.1; GraphPad Software, Boston, MA, USA).

3 Results and discussion

The present study investigated the light curability and optical properties of experimental flowable RBCs that were formulated with an increasing BNNS loading (0 wt.%–10.0 wt.%). It was demonstrated that BNNS incorporation up to 10.0 wt.% adversely impacted the light transmittance, translucency and DoC. However, the DC was enhanced at low BNNS loadings (up to 1.0 wt.%) and remained practically acceptable up to 5.0 wt.%. Therefore, the three null hypotheses were all rejected since (1) BNNS incorporation significantly affected the light curability including light transmittance, degree of conversion and depth of cure; (2) BNNS addition remarkably reduced the translucency of the light-curable RBCs; and (3) BNNS loading significantly altered the flexural strength and flexural modulus.

3.1 Filler powders characterisation

Characterisation was achieved as follows: (1) the morphology by SEM, (2) the BNNS thickness profile by AFM, and (3) the particle size distribution by DLS.

In Fig. 2(a), the barium silicate glass exhibits an irregular granular shape, distinguishing it from the platelet-like, two-dimensional (2D) structures of BNNS in Figs. 2(b)–2(d). This difference allows for easy identification of both particles in the RBC. Without ultrasonication facilitated dispersion in IPA, Fig. 2(b) at 20,000 $\times$ reveals significantly agglomerated BNNS. Despite ultrasonication, stackings can still be identified at a reduced level.

Figure 2(e) suggests considerable variation in BNNS thickness, with the three measured spots (from left to right) corresponding to approximately 11, 20, and 9 BN monolayers, respectively. The measured thickness of 100 particles ranges from 1 to 10 nm, with a

mean thickness of 5.4 nm (~ 16 layers, with an interlayer spacing of ~ 0.33 nm [46, 47]). The interlayer spacing in bulk h-BN crystals is widely accepted to be 0.33 nm [46], while high-resolution transmission electron microscopy (HRTEM) measurements of few-layer BNNS reported a very close value of 0.325 ± 0.01 nm [47]. Therefore, 0.33 nm was adopted for all calculations on layer numbers.

Figure 2(f) shows the particle size distribution by intensity for both BNNS and barium-silicate glass powders. The Z-average of BNNS is 1585.0 nm (~ 1.6 μm), while that of barium-silicate glass is 768.5 nm (~ 0.77 μm), which aligns with the manufacturer's specifications. Therefore, the aspect ratio (length/thickness) of this BNNS was estimated to be ~ 294 . The true particle size of BNNS may be smaller than that assigned by DLS, as DLS assumes that the particles are spherical and undergo isotropic Brownian motion. Instead, it can be represented by their radius as a single parameter [48]. This does not apply to BNNS with thin, platelet-like 2D morphology and high aspect ratio (ca. 294). Anisotropic diffusion may be slower than with spheres of the same lateral size. Hence, DLS results are less accurate than SEM and AFM data but can be adopted to extract the mean nanosheets length [49].

3.2 Refractive index

The measured RI of the experiment RBC components are summarised in Table 1. In addition, the refractive index of BNNS from published studies is also listed below [50, 51]. The refractive indices of the barium silicate glass (1.53) as primary filler and the monomer matrix (1.52–1.54) are similar, i.e., closely matched. These constituted the reference RBC without BNNS addition (Table 1). This RBC was highly translucent and allowed for adequate light penetration (Fig. 1, No BNNS group). However, in

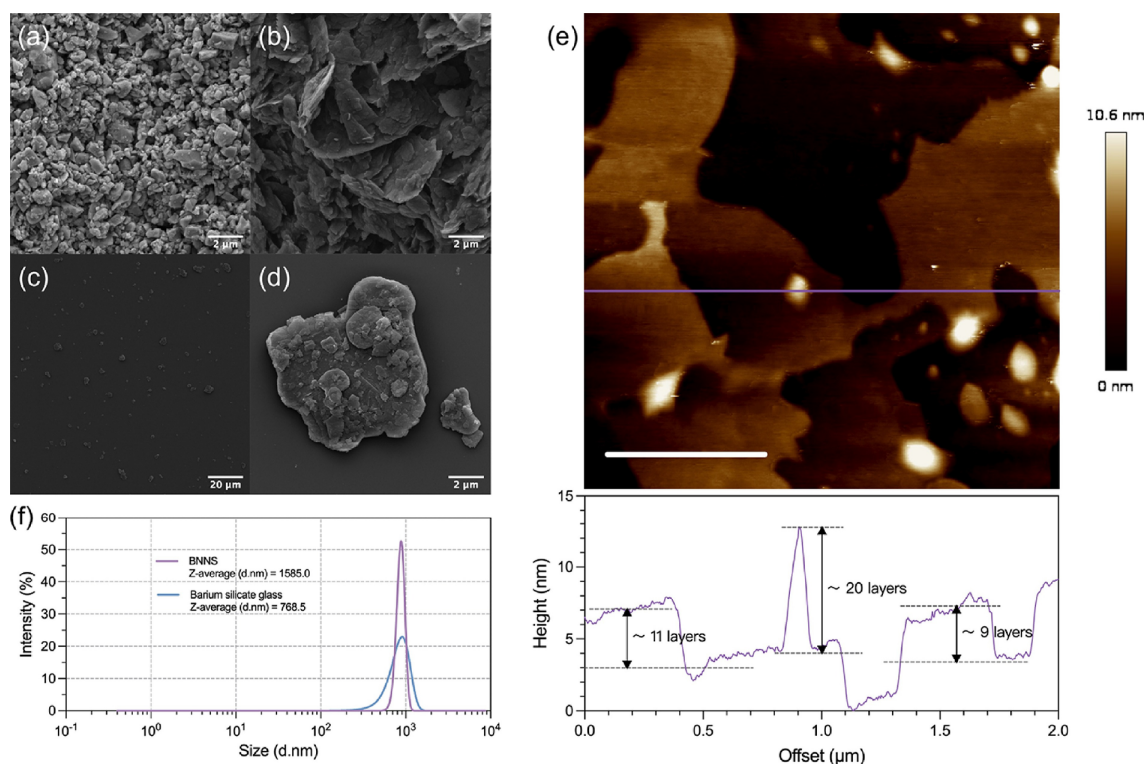


Figure 2 SEM images of (a) barium silicate glass powder at 20,000 $\times$; (b) agglomerated BNNS without ultrasonication at 20,000 $\times$; ultrasonically dispersed BNNS at (c) 2000 $\times$ and (d) at 20,000 $\times$. (e) AFM image of BNNS and height profile along the purple scanning line. (f) Particle size distribution by intensity of BNNS and barium silicate glass using Zetasizer Nano DLS.

Table 1 Refractive index of resin matrix and filler particles

Components	Refractive index
50 wt. %:50 wt. % Bis-GMA/TEGDMA monomer (uncured)	1.517 ± 0.005
50 wt. %:50 wt. % Bis-GMA/TEGDMA polymer (cured)	1.544 ± 0.004
Barium silicate glass	1.532 ± 0.012
BNNS	1.7–2.8 [50, 51]

real clinical settings, metal oxides including titanium oxide (TiO_2), aluminium oxide (Al_2O_3), and zirconium oxide (ZrO_2) with a RI ranging from 1.7 to 2.7, are commonly added to RBCs as opacifiers for shade-matching [40].

Exfoliated BNNS also exhibit a high RI, ranging from 2.1 to 2.7 [33, 51]. Owing to the high aspect ratio (ca. 294), BNNS are expected to be highly anisotropic in refractive index, and thus poorly matched to the resin. Hence, they are strongly light scattering [52]. This has been demonstrated by previous studies for blue light wavelength of 450 nm [50]. The in-plane component ($n_{\parallel}$) was 2.35 for light propagating parallel to the hexagonal layers. This is substantially higher than the out-of-plane value ($n_{\perp} \sim 1.65$), for light propagating perpendicular to the hexagonal layers. Similarly, researchers reported a measurement of $n_{\perp} \sim 2.7$ and $n_{\parallel} \sim 1.5$ at the UV wavelength of 280 nm [33]. In this case, its RI-related scattering behaviour is critical in understanding the optical behaviour of the formulated RBCs. BNNS is also often regarded as "optically transparent" due to their negligible absorption in the visible range, attributed to its ultrawide band gap (~ 5.9 eV) [53]. Therefore, their light scattering features, rather than light absorption ability, are the focus of the present study.

3.3 LCU characterisation and light transmittance

When incident light hits the uncured RBC, a portion of light may be surface reflected. The transmitted light attenuates primarily through two mechanisms: (1) absorption and (2) scattering, which can be characterised together by the Beer–Lambert law (Eq. (10)):

$$I(d_m) = I_{\text{top}}(1 - r)e^{-\mu d_m} \quad (10)$$

where $I(d_m)$ is the irradiance at depth d_m , I_{top} is the irradiance at the top surface, r is the fraction of light undergoing specular and/or

diffuse reflection from the surface, μ is the attenuation coefficient (cm^{-1}) the sum of the absorption coefficient μ_a and the scattering coefficient μ_s , and d_m is the depth of the material (cm). Within the current optical characterisation framework, it is practically impossible to separately characterise the individual contributions of the absorption coefficient and the scattering coefficient for this polymer composite system.

Transmitted light is essential to activate photo-polymerisation. This study employed a ternary system consisting of CQ with DPI and EDMAB, based on the enhanced photo-polymerisation reaction with reduced amount of CQ [54]. This can help minimise residual yellowish colouration from excess of CQ post photo-polymerisation [55], making the material more aesthetically pleasing for white-shades restorations, particularly following teeth whitening treatments [3].

In this study, the Bluephase G4 LCU in H mode had a uniformly distributed beam profile across the tip (THF = 0.615), as demonstrated by the 2D and 3D beam profiles in Fig. 3(a). Its emission spectrum in Fig. 3(b) showed dual peak emissions at 409.5 nm (violet) and 451.8 nm (blue), which aligned with a previous report [56]. The LCU tip data including mean radiant exitance (mW/cm^2) and radiant exposure (J/cm^2) measured during 20 s standard curing time using the top surface detector of a MARCTM LC spectrophotometer can be found in Table S2 in the ESM. This ensured the quality of LCU incident light and this wavelength range closely matches the absorption spectrum of CQ, which peaks at 468 nm with a range from 400 to 500 nm [57].

Unlike CQ, the co-initiators are not directly activated by radiant exposure. Instead, DPI with a single-peak absorption peak at 326 nm [58] serves primarily as an electron acceptor. EDMAB with an absorption peak at 310 nm [59] functions as a hydrogen donor. Both co-initiators have only minimal to weak absorption in the LCU wavelength and together facilitate radical generation and polymerisation following the photoexcitation of CQ, suggesting that their role is secondary and dependent on CQ activation.

Table S3 in the ESM shows that the radiant exitance, irradiance at the LCU tip characterised by the 4 mm diameter bottom surface detector of MARCTM LC, was 4294.5 ± 34.8 mW/cm^2 . However, due to reflection losses at the top and bottom Mylar strip-air interfaces and the empty 1 mm thick mould, the incident light irradiance (I_0) was measured at 3789.5 ± 31.3 mW/cm^2 , with 526.7 mW/cm^2 (13.9%) in the "violet" (360–420 nm) and 3262.8 mW/cm^2 (86.1%)

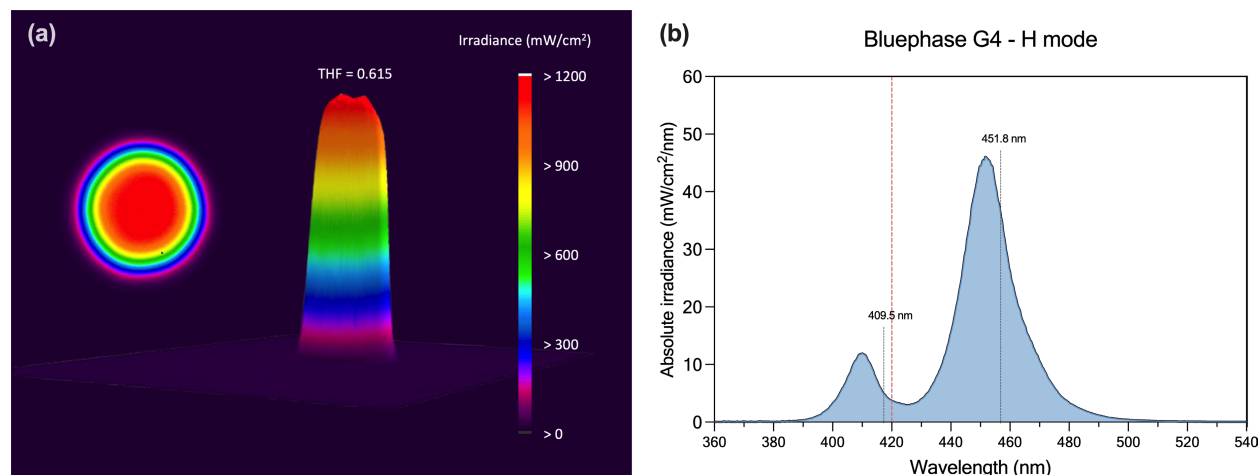


Figure 3 (a) Beam profiles of the poly-wave Bluephase G4 LCU H mode in 2D and 3D using a beam profiler, with a THF of 0.615. (b) The emission spectrum peaked at 409.5 nm in violet light region and 451.8 nm in blue light region, and the irradiance was measured at 1238.6 ± 1.6 mW/cm^2 illustrated by the AUC in blue colour.

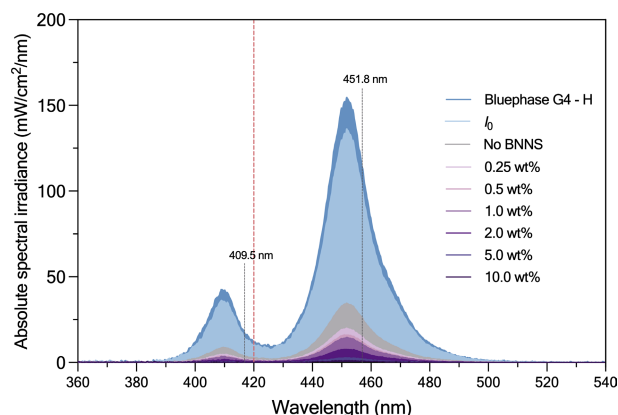


Figure 4 Spectral irradiance of Bluephase G4-H mode, the incident irradiance (I_0) under the experimental setting, and through 1 mm thick 0 wt.%–10.0 wt.% BNNS reinforced RBCs. The emission spectrum of the LCU peaked at 409.5 and 451.8 nm (marked by the grey dash lines), respectively. The peak AUC represents the mean spectral irradiance during a 20 s exposure. The peak area on the left side (360–420 nm) is the violet light region and the peak area on the right side (420–520 nm) is the blue light region ($\lambda = 420$ nm is marked as the red dash).

in the "blue" (420–520 nm) spectral ranges. The delivered spectral irradiance peaked at 409.5 and 451.8 nm, respectively (Fig. 4). Thus, a small portion (13.8%) of the spectral radiant power was in the violet wavelength region (10.6 J/cm²), whereas the majority (86.2%) was in the blue region (66.3 J/cm²).

Table S4 in the ESM presents the spectral irradiance values in the "violet", "blue", and "violet+blue" wavelength ranges through 1 mm specimen, along with their calculated transmittance T (%). As plotted in Fig. 5, a two-phase exponential decline ($R^2 > 0.99$) in light transmittance ($T\%$) was observed with increasing filler loading across all three wavelength regions. Notably, when BNNS loadings exceeded 5.0 wt.%, transmittance drops below 5% (2.0%), and at 10 wt.% BNNS, it approaches zero (0.61%).

At low concentrations with an efficient dispersion method, BNNS are expected to be evenly dispersed and randomly oriented in the resin matrix, allowing for more effective light propagation via single photon scattering. According to classic scattering theory for spherical particles, the interaction between photons and particles

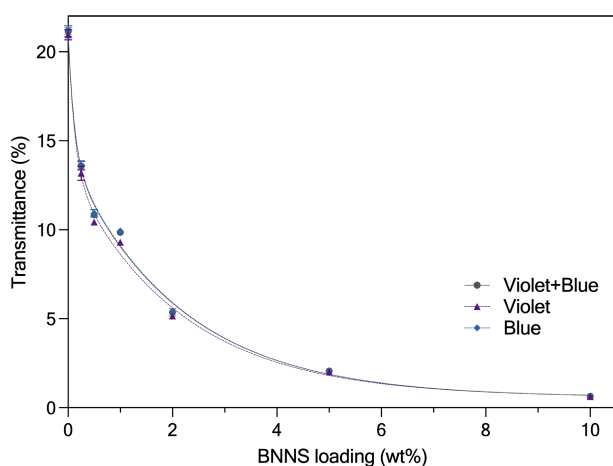


Figure 5 Transmittance of 1 mm thick experimental RBCs across the violet (360–420 nm), blue (420–520 nm) and violet+blue (360–520 nm) spectral regions vs. BNNS loading (0 wt.%–10.0 wt.%), cured with Bluephase G4-H for 20 s. The curves were fitted with a two-phase exponential decay model ($R^2 > 0.99$), revealing the decline in transmittance with an increasing BNNS loading (0 wt.%–10.0 wt.%).

depends on the ratio of spherical particle radius (r) to wavelength of incident light (e.g., blue light $\lambda \sim 450$ nm) [3]. However, for non-spherical particles, there is no generally acknowledged theory. Their scattering pattern depends on many factors including the shape, size, and orientation of the particles [60]. In our study, the 2D structure of BNNS (~ 1.6 μm lateral size, 5.4 nm thickness) enables a unique combination of Rayleigh-like ($r \leq \lambda$), Mie-like ($r \sim \lambda$), and geometric scattering-like ($r \geq \lambda$) modes when interacting with visible light (e.g., blue light $\lambda \sim 450$ nm) at minimal addition [61].

However, at higher concentrations, it is inevitable that BNNS become agglomerated, and multiple scattering effects dominate. These optical disruptions led to excessive backscattering and light attenuation [61], reducing light transmission and polymerisation efficiency. Advanced modelling, such as Monte Carlo simulations [62], can be employed to simulate and understand their scattering behaviour at high loadings. Researchers observed similar behaviour between BNNS and polystyrene (PS) nanospheres of similar size at higher wt.% for multiple scattering regime [60]. When this higher limit concentration is reached, and the RBC becomes opaque, the light propagation falls into a diffusive photo transport regime as observed with dental ceramics [63] and dentin tissue [64].

3.4 Degree of conversion

The $DC_{24\text{h}}$ at 1 mm depth of RBC at different BNNS loadings (0 wt.%–10 wt.%) is listed in Table 2 and presented in Fig. 6. The $DC_{24\text{h}}$ showed a slight increase with a peak at 0.5 wt.% BNNS and remained stable up to 1 wt.%. This scattering behaviour with RI mismatch probably contributed to the slightly enhanced DC [37]. Improved DC has been reported in several studies on h-BN reinforced dental adhesives including low concentrations of BNNT [21] and BNNS [23]. However, other studies reported no DC enhancing effect of h-BN addition in light-curing systems. This can be due to the poor filler dispersion by hand-mixing without ultrasonic pre-dispersion [18–20]. Ensuring the uniform dispersion is one of major challenges faced by nanomaterials. Surface functionalisation with chemical groups and modifying the surface charge may facilitate good dispersion. For example, methacrylate-functionalised BNNS demonstrated improved DCs [23] while melamine-functionalised BNNS also achieved good dispersion in epoxy resin up to 2 wt.% [24].

No formal regulatory standard defining a minimum 24 h post-irradiance DC of RBC has been established. However, DC of $> 55\%$

Table 2 DC of 0 wt.%–10 wt.% 1 mm thick BNNS reinforced RBC and DoC at 80% HV_{max} at 24 h post-irradiance, cured with Bluephase G4-H for 20 s ($n = 3$)

BNNS loading (wt.%)	$DC_{24\text{h}}$ (%)	DoC at 80% HV_{max} (mm)
0	77.3 ± 0.5^b	5.4 ± 0.5^a
0.25	80.6 ± 2.1^a	3.9 ± 0.4^b
0.5	80.3 ± 2.2^a	2.2 ± 0.2^c
1.0	78.7 ± 0.2^{ab}	1.6 ± 0.2^d
2.0	73.4 ± 0.6^c	1.4 ± 0.1^{de}
5.0	69.9 ± 2.3^d	1.0 ± 0.1^e
10.0	30.4 ± 0.5^e	$< 0.9^f$

^{a–e}Different letters (a, b, c, d, and e) within the same column indicate statistically significant differences between groups via one-way ANOVA ($P < 0.05$). ^fThe DoC of 10.0 wt.% BNNS group was unable to obtain due to the insufficient depth (< 0.9 mm) of cured materials for HV mapping at 0.5 mm interval, as shown in Fig. 1.

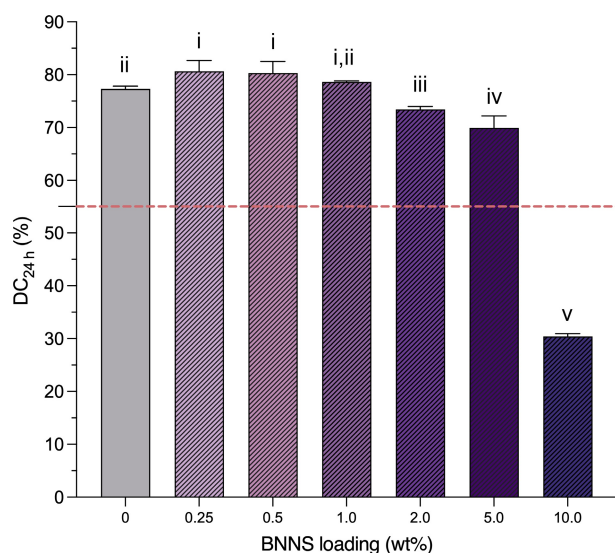


Figure 6 DC (%) of 1 mm thick 0 wt.%–10 wt.% BNNS RBC measured at 24 h post-irradiance, cured with Bluephase G4-H for 20 s ($n = 3$). The horizontal red dashed line indicates the practically acceptable threshold of DC (55%) reported by previous studies [65–67]. Different letters (i, ii, iii, iv, v) indicate statistically significant differences between groups via one-way ANOVA ($P < 0.05$).

is considered as a practical threshold for occlusal or bulk-filled restorations based on previous studies [65–67]. In the present study, DCs remained practically acceptable for BNNS loadings up to 5.0 wt.% but declined drastically at 10 wt.%, reaching only $30.41\% \pm 0.54\%$. DCs falling below this level are widely associated with increased wear and compromised mechanical performance [68].

Overall, while low BNNS concentrations (0.25 wt.%–1.0 wt.%) slightly enhanced polymerisation, excessive BNNS incorporation (≥ 2.0 wt.%) hindered the polymerisation process, leading to a significant reduction in DC_{24h}.

3.5 Depth of cure

Figure 1 shows representative images of specimens from each group used for HV mapping. As light transmittance decreased with higher BNNS loading, the DoC showed a similar decreasing trend (Fig. 1) consistent with previous studies using a simple scraping method in accordance with ISO 4049 [69]. These investigated: up to (i) 0.5 wt.% BNNS [18], (ii) 1.6 wt.% BNNS/TiO₂ addition [19], and (iii) 1.6 wt.% BNNS/DMAHDM composites [20]. However, the ISO 4049 method was reported to overestimate the DoC of bulk fill RBCs [43].

In this study, DoC was measured using a more accurate Vickers hardness mapping method with a cuboid slot mould [43–45]. The observed HV_{max} values of all groups ranged from 31.4 ± 1.5 , and 80% HV_{max} values were calculated accordingly. Figure 7 illustrates how the DoC at 80% HV_{max} was determined via depth vs. hardness curve fitting. As the RBCs were turning less translucent with increasing BNNS addition (Table 2 and Fig. 1), the light penetration and polymerisation were detrimentally affected, thereby resulting in a significant loading-dependent decrease in DoC ($P < 0.05$). According to ISO 4049 [69], the DoC should meet at least 1 mm for opaque shade restorative materials and 1.5 mm for other materials. All experimental formulations met the requirement except for the 10 wt.% BNNS group, where the cured material depth notably reduced to less than 0.9 mm (Table 2 and Fig. 1), making it insufficient to perform HV mapping at 0.5 mm intervals.

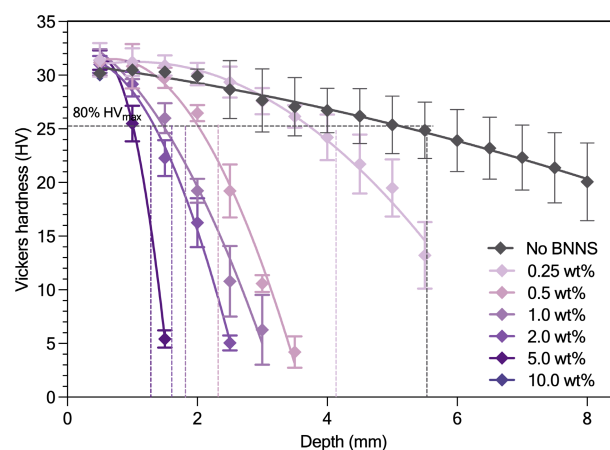


Figure 7 DoC measurement of all BNNS-reinforced RBCs using HV vs. depth curves. The vertical dashed lines are corresponding to the DoC at 80% HV_{max} for each group. The 10.0 wt.% group was not plotted due to the insufficient depth (< 1 mm) for HV mapping.

3.6 Translucency and clinical relevance

The evaluation of translucency in dental restorative materials is crucial to achieve optimal aesthetic outcomes. However, no specific translucency requirements are prescribed in current standards or guidelines, including ISO 4049:2019 [69], ISO/TR 28642:2016 [70], or those of the American Dental Association (ADA). This is largely due to the versatility and flexibility of RBCs as direct restorative material with various shades and applications.

Figure 8(a) shows the translucency of the experimental RBCs of various BNNS loading against black snowflake patterns on a white background. As shown in Table 3 and Fig. 8(b), TP_{ab} and TP₀₀ metrics exhibit strong correspondence as an exponential decline with increasing BNNS loadings, while TP₀₀ tends to yield slightly higher values and is considered more perceptually accurate ($P < 0.05$).

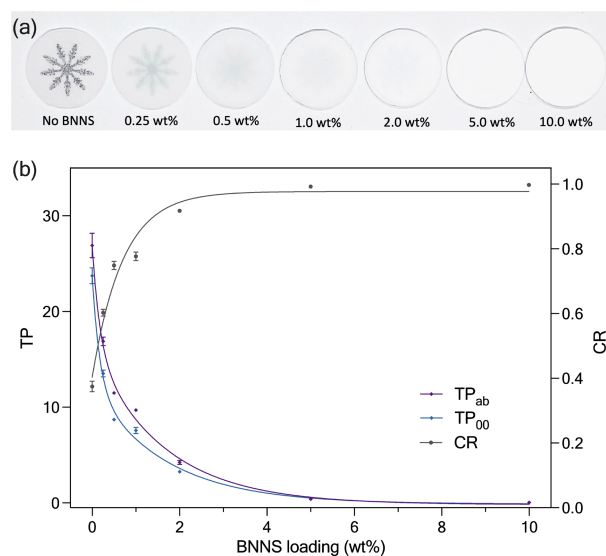


Figure 8 (a) Images of 1 mm thick BNNS-reinforced RBCs discs against black snowflake patterns on a white background: from left to right, the discs correspond BNNS loadings of 0 wt.%–10.0 wt.%. (b) TP and CR of 1 mm thick experimental RBCs as a function of BNNS loading (0 wt.%–10 wt.%). The TP_{ab} and TP₀₀ curves were fitted using a two-phase exponential decay model ($R^2 > 0.99$), while the CR curve was fitted using an exponential plateau model ($R^2 > 0.96$), revealing a significant decrease in translucency and a corresponding increase in CR with increasing BNNS content.

0.05). CR, which quantifies opacity, demonstrates an expected inverse relationship with TP values ($P < 0.05$).

The translucency of natural human teeth is both site-dependent [35] and strongly influenced by tissue thickness [71]. For example, human incisors exhibit a gradual decrease in TP from the incisal to the cervical region [72]. Reported TP values for 1 mm human enamel commonly fall within 15–20 [34, 35, 72], reflecting its highly translucent nature arising from its inorganic and prismatic microstructure. By contrast, dentin tissue typically shows lower and more variable translucency [64]. Published TP values for 1 mm human dentin span a wide range (approximately 4–16) [34, 35, 72]. This can be characterised with dentinal tubules, a higher organic fraction and significant structural heterogeneity [64].

Previous studies suggested both increasing filler loading [71] and greater RI mismatch [73], both reduce the translucency of RBCs. In this present study, BNNS possess a substantially higher RI than the base RBC (Table 1). Consequently, its incorporation produced a loading-dependent decrease in translucency (lower TP) and increase in opacity (higher CR). At low BNNS loading (< 0.25 wt.%), the RBC remained highly translucent and exhibited optical behaviour comparable to natural enamel [34, 35, 72]. With increasing BNNS content, opacity increased progressively. At BNNS addition ≥ 2.0 wt.%, the RBC became distinctly opaque (CR = 0.9–1.0) [74]. This suggested potential applications in dark discolouration cases requiring a high masking ability [75].

3.7 Flexural strength and flexural modulus

The results of FS and FM are presented in Fig. 9 and Table S5 in the ESM. FS initially increased with 0 wt.%–0.5 wt.% BNNS loadings and remained relatively stable for additions up to 2.0 wt.%. A more noticeable reduction was observed at higher BNNS concentrations (5.0 wt.% and 10.0 wt.%), as shown in Fig. 9(a). Nevertheless, the FS of all groups remained above the minimum requirement of 80 MPa specified by ISO 4049 for intraorally light-cured RBCs intended for occlusal load-bearing areas [69].

In contrast, FM increased significantly with BNNS incorporation, indicating enhanced composite stiffness. The highest FM was recorded at 10.0 wt.% BNNS (4663.8 ± 139.9 MPa), demonstrating a progressive stiffening effect with increasing BNNS content.

Previous studies reported no improvement in FS or FM up to 0.5 wt.%–0.8 wt.% BNNS loadings, which may be attributed to the relatively small lateral dimensions (0.1–0.4 μm) and low aspect ratio of the nanosheets, combined with the limited effectiveness of hand-mixing procedures [18, 20]. In contrast, flowable RBCs reinforced

with BNNS/TiO₂ showed approximately 82% and 55% enhancement in FS and FM, respectively, at a loading of 0.8 wt.% [19]. The present findings suggest that the reinforcing effect of BNNS is strongly affected by filler loading and dispersion state. While moderate BNNS addition did not substantially compromise strength, excessive loadings resulted in reduced FS, possibly owing to agglomeration-induced stress concentrations, which can be validated by SEM imaging discussed below.

3.8 Cross-sectional fracture surface microstructure

At lower concentrations (up to 1.0 wt.%), BNNS were homogeneously dispersed in the cured resin matrix with a predominantly isotropic orientation, as shown in Figs. 10(a)–10(c). However, as BNNS content increased, more voids and aggregated BNNS were observed on the cross-sectional fracture surfaces following three-point bending testing. Specifically, at higher loadings (above 2 wt.%), the dispersion quality and interaction between BNNS and the resin matrix deteriorated, with notably increased agglomeration and residual voids formation, as presented in Figs. 10(e)–10(f).

Direct observation of BNNS dispersion within RBCs is challenging particularly at rather low loadings with nanoscale particle dimensions [18]. Consistent with previous reports, homogeneous dispersion was achieved at lower BNNS addition, whereas higher loadings resulted in significant agglomerations [19]. Such agglomerates can act as structural defects and stress-concentration centers, deteriorating stress-transfer efficiency within the composites and consequently compromising overall mechanical performance [14, 15]. Similarly, light attenuation may be explained with dominated multiple scattering model applicable for aggregated BNNS. This observation is well aligned with the reduction in light transmittance, depth of cure, translucency, and FS observed at higher concentrations.

3.9 Future work

The dispersion state and spatial alignment of BNNS within the resin matrix are critical to mediate the trade-off between mechanical reinforcement and light-curability, both of which are essential in the development of aesthetic and functional restorative materials. Further investigations should aim at achieving controlled orientation of BNNS orientation and improved dispersion efficiency, as well as evaluate long-term including wear resistance, hydrolytic stability, biocompatibility, and antibacterial activities. Such studies will provide a more comprehensive understanding of structural reinforcing mechanisms and facilitate the optimisation of

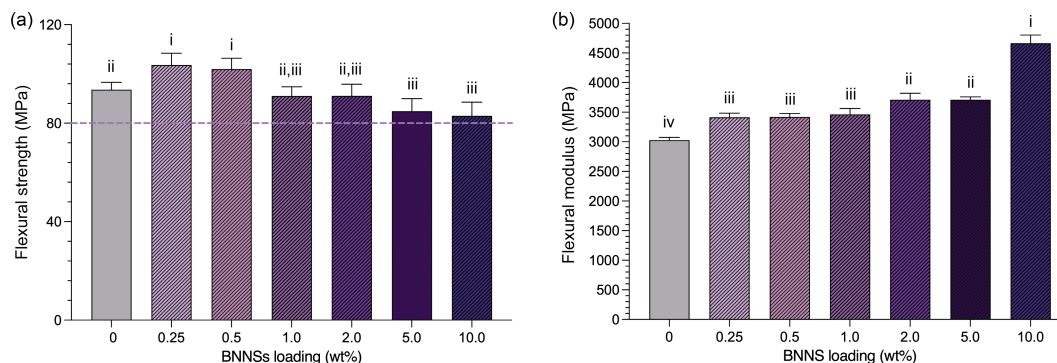


Figure 9 (a) Flexural strength and (b) flexural modulus of all BNNS-reinforced RBCs after 24 h in 37 °C deionised water storage. The horizontal purple dash in panel (a) indicates the clinical acceptable FS threshold of 80 MPa based on ISO 4049:2019 [69]. Different letters (i, ii, iii, iv) indicate statistically significant differences between groups via one-way ANOVA ($P < 0.05$).

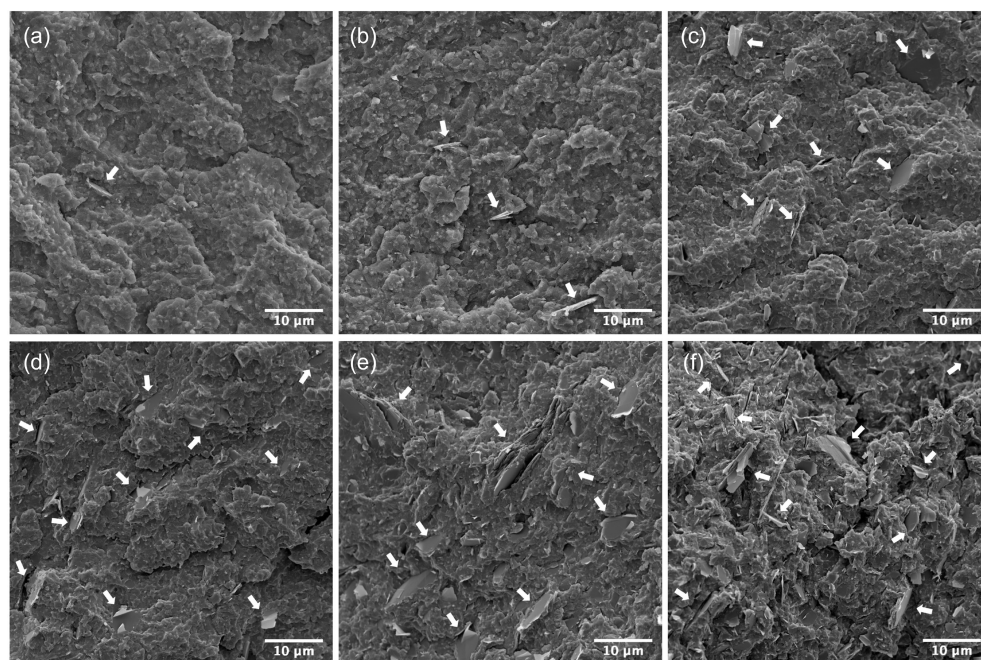


Figure 10 Cross-sectional SEM images of fractured surface morphology of BNNS (marked by white arrows) reinforced RBCs at 5000 $\times$ magnification after three-point bending test, with BNNS loading at (a) 0.25 wt.%, (b) 0.5 wt.%, (c) 1.0 wt.%, (d) 2.0 wt.%, (e) 5.0 wt.%, and (f) 10.0 wt.%, respectively.

BNNS-reinforced RBC formulations for clinical translations.

4 Conclusions

This study provides comprehensive insights into the light-curability, optical properties, and mechanical performance associated with incorporating 2D BNNS as a reinforcing filler into light-cured RBC systems. BNNS addition (up to 10 wt.%) adversely impacted the light transmittance, depth of cure, and translucency in a loading-dependent manner. However, the degree of conversion was slightly enhanced at low BNNS loadings (up to 1.0 wt.%) and remained practically acceptable up to 5.0 wt.%. The formulations all met the minimal flexural strength requirement for occlusal load-bearing restorations, with a progressively increased flexural modulus. These findings identify potential ranges for formulating BNNS nanofiller-reinforced RBC tailored to clinical application, ranging from highly translucent materials suitable for enamel restorations, to more opaque options for dentin fillings and highly opaque aesthetic masking applications. Moreover, this work highlights the importance of future optimisations on BNNS morphology, dispersion efficiency, and spatial orientation to maximise reinforcement while maintaining adequate light-curability and aesthetic performance.

Electronic Supplementary Material: Supplementary material (further details on the materials used, LCU output and incident light characterisation, light transmittance calculations, FS and FM results) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94908977>.

Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and/or the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

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Declaration of competing interest

All the contributing authors report no conflict of interests in this work.

Author contribution statement

J. Q. F.: Writing – original draft, writing – review & editing, data curation, investigation, conceptualisation, formal analysis, software, visualisation, funding acquisition. J. L. G.: Resources, project administration, funding acquisition. N. S.: Writing – review & editing, methodology, validation, funding acquisition. D. C. W.: Writing – review & editing, resources, methodology, validation, project administration, conceptualisation, supervision, funding acquisition. All the authors have approved the final manuscript.

Informed consent

Not applicable.

Ethics statement

Not applicable.

Use of AI statement

None.

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