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Mechanical and Morphological Characterization of Halloysite-Nanotube (HNT)-Reinforced Urethane-Dimethacrylate (UDMA) Dental Composites

Amir Khan[1,*], Piyush Singhal[2]

Abstract

Dental resin composites are widely used in restorative dentistry because of their excellent aesthetics, conservative cavity preparation, and satisfactory mechanical properties. However, their long-term clinical performance is limited by polymerization shrinkage, wear, microcrack formation, water absorption, and degradation in the oral environment. This study investigates the mechanical and morphological characteristics of Halloysite Nanotube (HNT)-reinforced Urethane Dimethacrylate (UDMA) dental composites to enhance the structural performance and durability of restorative materials. UDMA-based composites containing different concentrations of HNTs were fabricated using controlled mixing and light-curing techniques. Their flexural strength, flexural modulus, compressive strength, Vickers microhardness, and water sorption behavior were evaluated. Morphological characterizations were performed using Scanning Electron Microscopy (SEM). The experimental results demonstrated that low to moderate HNT loadings significantly improved the mechanical performance of the composites by enhancing stress transfer, crack deflection, and filler matrix interactions. Increased microhardness indicated superior surface durability and wear resistance. SEM observations revealed uniform nanotube dispersion at lower filler concentrations, whereas higher HNT loadings resulted in particle agglomeration, increased viscosity, and localized void formation, adversely affecting mechanical performance. The findings confirm that HNTs are effective nano reinforcements for UDMA dental composites when incorporated at optimized concentrations and uniformly dispersed within the resin matrix. The developed nanocomposites exhibit enhanced stiffness, hardness, and fracture resistance, demonstrating considerable potential for advanced restorative dentistry. Future investigations should focus on long-term aging, simulated oral wear, antibacterial performance, cytocompatibility, and clinical validation to establish their suitability for next-generation dental restorative applications.

Keywords: Halloysite nanotubes (HNTs), dental composites, restorative dentistry, mechanical characterization, scanning electron microscopy (SEM)

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INTRODUCTION

Dental resin composites are among the most used restorative materials in modern dentistry due to their excellent esthetics, minimally invasive application procedures and improved adhesive bonding capabilities [1, 2]. The last few decades have seen the progressive development of polymer chemistry, filler technology, silane coupling chemistry and photopolymerization systems that have contributed greatly to the improved clinical performance of the resin-based restorative materials [1, 3]. These composites are commonly used for anterior and posterior restorations, core

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build-ups, veneers and indirect restorations because of their superior color matching, acceptable biocompatibility and conservative tooth preparation as compared to traditional metallic restorations such as dental amalgam [1, 2]. However, conventional dental resin composites still have several drawbacks such as polymerization shrinkage, poor wear resistance, water absorption, marginal degradation, microcrack formation, and poor long-term mechanical durability under cyclic masticatory loading. These disadvantages have led to the development of reinforced resin composites with nanostructured fillers, such as Halloysite Nanotubes (HNTs), by researchers to improve the mechanical performance and long-term durability of dental restorative materials [2, 4].

The performance of dental resin composites is largely governed by the synergistic interaction of the organic resin matrix, inorganic reinforcing fillers, and the silane coupling agent, which together determine the mechanical strength, wear resistance, polymerization behavior and long-term clinical durability of the restorative material [5, 6]. The resin matrix usually consists of dimethacrylate monomers like Bisphenol-A Glycidyl Methacrylate (Bis-GMA), Urethane Dimethacrylate (UDMA), Triethylene Glycol Dimethacrylate (TEGDMA), and Ethoxylated Bisphenol-A Dimethacrylate (Bis-EMA) [6, 7]. Among these monomers, UDMA has gained much attention because of its low viscosity, flexible molecular architecture, high degree of conversion and better mechanical performance as compared to conventional Bis-GMA-based systems [7, 8]. UDMA has better handling characteristics, greater polymer chain mobility during photopolymerization and improved cross-linking efficiency compared to Bis-GMA which has a rigid aromatic backbone and high viscosity [7, 9]. These characteristics are useful to make fracture toughness better, to reduce the brittleness, to improve the flexural properties and to reduce the polymerization shrinkage stress, which make UDMA one of the most promising resin matrices for development of advanced dental restorative composites [8, 9]. Accordingly, UDMA-based resin systems have become increasingly attractive for the fabrication of high-performance dental composites reinforced with nanostructured fillers to further improve their mechanical and clinical performance [9, 10].

UDMA-based dental composites have shown excellent mechanical performance and improved polymerization properties, but there is still a need to improve their strength, stiffness, wear resistance, fracture toughness, and resistance to hydrolytic degradation during long-term oral use [11, 12]. Recently, nanotechnology has emerged as one of the most promising strategies to improve the performance of polymer-based biomaterials by incorporation of nanoscale reinforcing fillers [12, 13]. The unique morphology, very high surface area-to-volume ratio and excellent interfacial interactions of nanofillers with the polymer matrix can significantly enhance the mechanical, thermal, tribological and biological properties of dental composites [13, 14]. Nanomaterials – such as silica nanoparticles, zirconia nanoparticles, carbon nanotubes, graphene oxide, nano-clays, and hydroxyapatite and aluminosilicate nanotubes – have been widely studied as reinforcing agents in resin-based dental restorative materials [14, 15]. Among them, Halloysite Nanotubes (HNTs) have attracted much attention as naturally occurring tubular aluminosilicates with high aspect ratio, excellent biocompatibility, low toxicity, chemical stability, cost-effectiveness and superior reinforcing capability [16, 17]. Moreover, the unique hollow tubular architecture of HNTs can contribute to efficient stress transfer, crack deflection, and good interfacial bonding with polymer matrices, which makes them a suitable candidate as multifunctional nanofillers for the development of high-performance UDMA-based dental nanocomposites [16, 18].

Halloysite nanotubes (HNTs) are natural aluminosilicate clay minerals belonging to the kaolinite family. Due to their unique structural and physicochemical properties, they have attracted considerable attention as multifunctional nanofillers for polymer composites [19]. HNTs are hollow tubes with external diameters in the range 30–70 nm, inner lumen diameters of 10–30 nm and lengths ranging from several hundred nanometers to a few micrometers. The exterior of HNTs primarily consists of siloxane (Si–O–Si) groups, while the interior lumen has a high concentration of aluminol (Al–OH) groups, suggesting that the surface chemistries of HNTs are different and allow for selective functionalization,

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better filler dispersion and good interfacial interactions with polymer matrices [19, 20]. The high aspect ratio and nanotubular structure of HNTs improve the mechanical properties of polymer composites through different reinforcement mechanisms such as effective stress transfer, crack deflection, crack bridging, reduced mobility of polymer chains, and enhanced interfacial adhesion between the filler and the matrix [20, 21]. In addition, HNTs demonstrate good thermal stability, chemical inertness, low cytotoxicity and excellent biocompatibility, which makes them highly attractive for biomedical and dental applications such as resin-based restorative composites, drug delivery systems, tissue engineering scaffolds, and antimicrobial biomaterials [20, 21]. These features make HNTs promising nano reinforcements for the development of next generation UDMA-based dental nanocomposites with enhanced mechanical durability and long-term clinical performance [21].

Several studies have investigated the addition of nanofillers into dental resin composites to improve the mechanical performance, physicochemical stability and long-term clinical durability [22, 23]. Previous studies have reported that nanofillers can significantly improve the flexural strength, compressive strength, surface hardness, fracture toughness, wear resistance and elastic modulus, while influencing the polymerization behavior, degree of conversion and water sorption properties of resin-based restorative materials [22, 24]. The enhancement in properties is mainly due to high surface area, nano-size and strong interfacial interactions between the nanofillers and polymer matrix. However, the reinforcement efficiency of nanomaterials depends on many factors, such as the dispersion of fillers, loading concentration, aspect ratio, particle morphology, surface functionalization, and compatibility with the resin matrix [23, 24]. Inadequate dispersion and/or overfilling of fillers often cause nanoparticle agglomeration, void formation, poor filler–matrix adhesion, and stress concentration sites, all of which can affect the mechanical properties of the composite [23]. Thus, achieving a homogenous distribution of nanofillers through adequate surface modification and optimized filler loading is still one of the major challenges in the development of high-performance dental nanocomposites with enhanced mechanical reliability and long-term clinical performance [22, 24].

The advantageous features of halloysite nanotubes (HNTs) over traditional nanofillers include the naturally occurring tubular morphology, high aspect ratio, good mechanical stability and ease of surface functionalization [25]. Surface modification methods, such as silanization with organosilane coupling agents (e.g., 3-aminopropyltriethoxysilane, APTES), dramatically enhance the compatibility of HNTs with methacrylate-based resin systems due to the formation of strong chemical bonds between the nanotube surface and the polymer network [25, 26]. Improved interfacial adhesion enables efficient stress transfer under mechanical loading, prevents crack initiation and propagation and increases the overall structural integrity, stiffness and fracture resistance of the composite material. Moreover, the peculiar hollow lumen of HNTs can be utilized as an efficient nanocontainer for the entrapment and controlled release of therapeutic agents, e.g., antimicrobials, drugs and bioactives, thus providing additional functionalities apart from mechanical reinforcement [26, 27]. These multifunctional properties make HNTs very attractive for the development of advanced restorative dental materials with improved clinical performance. However, the overall mechanical, morphological and hydrolytic behavior of HNT-reinforced UDMA dental composites has not been systematically investigated to a significant degree, especially with regard to the optimization of the filler loading and the understanding of the relationship between the nanotube dispersion, the interfacial bonding and the composite performance [27]. Further research is required to determine the optimum concentration of HNT for the maximum mechanical reliability and long-term durability of UDMA-based dental nanocomposites [25, 27].

Mechanical characterization is necessary to assess the applicability of dental composites in restorative applications. Properties – such as flexural strength, flexural modulus, compressive strength, fracture resistance, impact strength, and surface hardness – are of particular importance as restorative materials are constantly subjected to complex cyclic forces, thermal fluctuations and chemically aggressive conditions in the oral cavity [28]. Likewise, the morphological characterization by techniques – such as Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and Fourier Transform Infrared Spectroscopy (FTIR) – gives important information about filler

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distribution, interfacial adhesion, crack propagation behavior, elemental composition and polymerization characteristics of resin composites [28, 29]. The correlation between the morphological observations and the mechanical performance is critical to understand the reinforcement mechanisms of the Halloysite Nanotubes (HNTs) in the UDMA matrix and to optimize composite formulations for improved mechanical durability and long-term clinical performance [29].

Another important factor influencing the performance of dental composites is water sorption and hydrolytic degradation. Restorative materials are always in contact with saliva, changing pH, temperature variation and enzymatic activity in the oral environment. Water absorption may bring about matrix plasticization, debonding of filler, interfacial degradation and aging of the mechanical properties. The addition of HNTs could influence the hydrophilic behaviour of the composite because of the hydroxyl groups on the nanotube surfaces. Thus, evaluation of water sorption and long-term stability is important for the clinical applicability of HNT reinforced UDMA composites [29, 30].

The aim of the present study was to fabricate and characterize Halloysite Nanotube reinforced UDMA dental composites at different nanotube concentrations. The main aim is to investigate the influence of incorporation of HNT on the mechanical and morphological properties of UDMA-based restorative composites. Mechanical characterization comprises flexural strength, flexural modulus, compressive strength and microhardness. Morphological characterization includes SEM, EDS and FTIR analyses to assess nanotube dispersion and interfacial interactions. The study is also aimed to find the optimum HNT concentration that can improve composite performance without detrimental agglomeration effects. The results of this study should lead to the development of advanced nanostructured dental restorative materials with enhanced durability, mechanical reliability and long-term clinical performance [30].

Resin-based dental composites are one of the most used direct restorative materials due to their excellent aesthetic appearance, conservative tooth preparation, ability to bond adhesively and satisfactory mechanical performance. The composition of the resin matrix, the type and loading of the fillers, the interfacial adhesion between filler and matrix, the degree of polymerization, water sorption and resistance to occlusal wear and fatigue [31] strongly influence the long-term clinical success of these restorative materials. UDMA is one of the most used dimethacrylate monomers, and it has attracted much attention because of its relatively low viscosity, high degree of conversion, improved toughness and favorable mechanical properties compared to highly viscous Bis-GMA-based resin systems [31, 32].

Halloysite nanotubes (HNTs) are naturally occurring aluminosilicate nanomaterials with unique hollow tubular morphology, high aspect ratio, excellent biocompatibility and reactive surface hydroxyl groups. They are promising nanofillers for dental resin composites due to their white color, low cost, easy surface functionalization and reinforcing ability [33]. It has been shown in previous studies that the addition of HNT can enhance mechanical, morphological and biological properties of dental composites when it is uniformly dispersed in the polymer matrix [32, 33]. Based on these findings, the present study aims to investigate the mechanical and morphological characteristics of HNT-reinforced UDMA dental composites with different HNT loading concentrations to determine the optimum filler content for improved restorative performance.

Nanofillers can improve polymer composite properties by crack deflection, stress transfer and restriction of polymer chain mobility. However, the reinforcement effect is critically dependent on the quality of dispersion, interface bonding, filler loading, curing efficiency, and balance between strength and brittleness. In HNT-enhanced UDMA composites, agglomerates act as defects and can be formed by overloading. Thus, a systematic study is needed to determine the optimum HNT concentration and relate the mechanical behavior to the morphology observed by SEM. The primary purpose of the study is as follows:

- To fabricate UDMA dental composites containing different HNT loadings.
- To evaluate flexural strength, flexural modulus, compressive strength, microhardness, wear, and water sorption.

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- To analyze fracture morphology and nanotube distribution using SEM and EDS.
- To relate mechanical performance to filler dispersion and interfacial adhesion.
- To identify a practical HNT loading range for restorative dental composite development.

MATERIALS AND METHODS

Materials

The present research work aims to synthesize and characterize Halloysite Nanotube (HNT) reinforced Urethane Dimethacrylate (UDMA) dental composites with different HNT loading concentrations to study their mechanical and morphological properties for application in restorative dentistry. The experimental methodology consists of material selection, composite fabrication, specimen preparation, mechanical characterization, morphological analysis and data interpretation. The mechanical behavior of the developed composites will be systematically studied as a function of HNT loading to determine the optimum filler concentration that can improve the overall performance of UDMA-based dental restorative materials [34].

The UDMA resin will be used as the main polymer matrix initially because of its relatively low viscosity, high degree of conversion, excellent toughness and superior mechanical properties compared to conventional bis-GMA-rich resin systems [35]. Halloysite nanotubes (HNTs) will be incorporated as nanoscale reinforcing fillers owing to their naturally occurring tubular morphology, high aspect ratio, excellent biocompatibility and outstanding reinforcing capability. Triethylene Glycol Dimethacrylate (TEGDMA) will be used as a reactive diluent to adjust the resin viscosity to optimize the physicochemical properties of the composites as shown in Figure 1. The visible-light photoinitiating system will be camphorquinone (CQ) and a tertiary amine co-initiator. Additionally, silane coupling agents will be employed to enhance the filler–matrix interfacial adhesion so that efficient stress transfer can be achieved and the mechanical integrity and durability of the fabricated dental nanocomposites can be improved [35, 36].

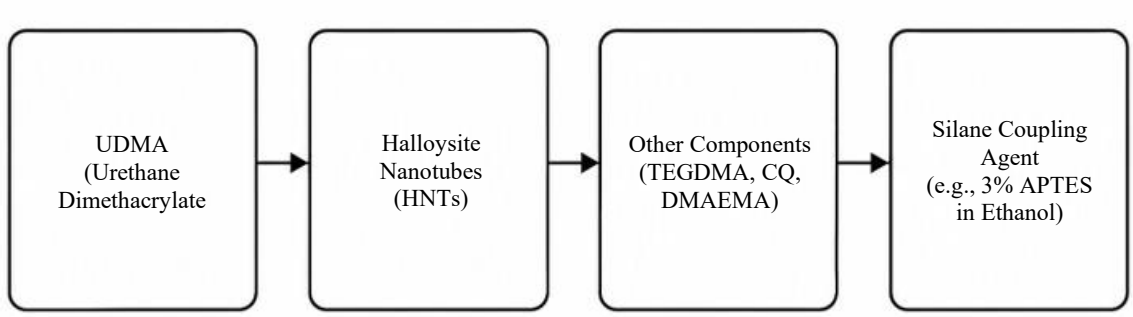


Figure 1. Schematic representation of the raw materials used for the fabrication of HNT-reinforced UDMA dental composites.

The Halloysite Nanotubes (HNTs) should be dried in a laboratory oven before the composite fabrication process, to remove absorbed moisture and decrease filler agglomeration which will result in better dispersion in the resin matrix. To further improve the compatibility of the inorganic nanotubes with the organic polymer matrix, the HNTs will be surface treated with a silane coupling agent – like 3-aminopropyltriethoxysilane (APTES) – to promote strong interfacial bonding and efficient stress transfer under mechanical loading. Then, composite formulations with different HNT loading concentrations (0 wt%, 0.5 wt%, 1 wt%, 2 wt% and 3 wt%) will be prepared to investigate the effect of filler content on the mechanical and morphological properties of the developed dental composites. The HNTs will be incorporated into the UDMA resin system step-by-step by magnetic stirring, ultrasonication and mechanical mixing to achieve uniform filler dispersion and to reduce the aggregation of nanoparticles as shown in Figure 2. The obtained composite paste is then packed into ISO 4049 standard ring molds and photopolymerized in a visible light curing unit under controlled irradiation conditions for complete polymerization and reproducible specimen preparation.

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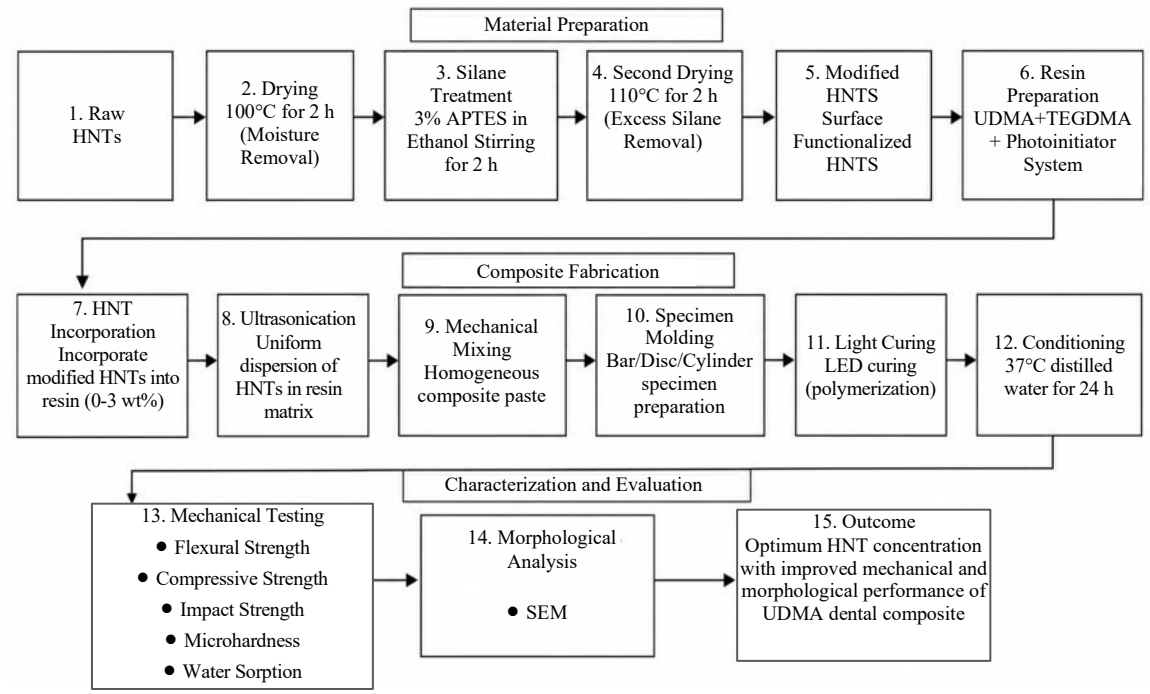


Figure 2. Experimental workflow for the fabrication and characterization of HNT-reinforced UDMA dental composites.

Experimental Groups/Sample

The experimental design was developed to evaluate the effect of halloysite nanotube (HNT) reinforcement on UDMA dental composites. UDMA was used as the primary resin matrix, while HNTs were incorporated at different loading levels as mentioned in Table 1. The HNTs and UDMA were mixed under controlled conditions to achieve uniform dispersion. The prepared composite formulations were then molded into specimens of predetermined dimensions.

Table 1. Composition and experimental design of halloysite nanotube (HNT)-reinforced UDMA dental composite specimens with different filler loading levels.

Group	UDMA resin system	HNT loading (wt%)	Suggested silane treatment	Specimen code	Purpose
G0	UDMA/TEGDMA	0	Not applicable	HNT-0	Control.
G1	UDMA/TEGDMA	0.5	Optional	HNT-0.5	Low filler.
G2	UDMA/TEGDMA	1.0	Recommended	HNT-1	Moderate filler.
G3	UDMA/TEGDMA	2.0	Recommended	HNT-2	High filler.
G4	UDMA/TEGDMA	3.0	Recommended	HNT-3	Agglomeration threshold study.

COMPOSITE FABRICATION

The fabrication of Halloysite Nanotube (HNT) reinforced Urethane Dimethacrylate (UDMA) dental composites was performed in a systematic process consisting of nanotube pre-treatment, surface modification, resin preparation, filler incorporation, homogenization, moulding and visible-light polymerization [37]. Firstly, the raw Halloysite Nanotubes (HNTs) were dried in a hot air oven at 100°C for 2 h to remove the absorbed moisture and minimize the particle agglomeration. The dried HNTs were then surface functionalized by a silane coupling agent to improve their compatibility with the methacrylate-based resin system and filler–matrix interfacial adhesion. For the silanization process, a 3 wt% solution of 3-aminopropyltriethoxysilane (APTES) in ethanol was added to the dried HNTs and magnetically stirred for 2 h at room temperature, to allow the silane molecules to react with the hydroxyl groups on the nanotube surface. The silanized HNTs are washed thoroughly to remove unreacted silane

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and then dried in a hot-air oven at 110°C for 2 h to remove residual solvent and ensure complete condensation of the silane layer after surface treatment. This surface modification greatly improves the dispersion of the nanotubes in the resin matrix, promotes a stronger interfacial bonding and enables an efficient stress transfer during the mechanical loading, thus improving the mechanical strength and long-term durability of the fabricated dental nanocomposites [38].

Urethane Dimethacrylate (UDMA) was chosen as the primary monomer and Triethylene Glycol Dimethacrylate (TEGDMA) was used as a reactive diluent to control the viscosity and improve handling properties. The photoinitiator system for polymerization by visible light was added by camphorquinone (CQ) and dimethylaminoethyl methacrylate (DMAEMA). Different composite formulations were prepared by gradually adding different concentrations of modified HNTs into the resin mixture, e.g., 0 wt%, 0.5 wt%, 1 wt%, 2 wt%, and 3 wt% as mentioned in Table 1 [39].

The HNT-resin mixture was initially stirred by magnetic stirring and then ultrasonicated for 15–30 minutes to assure uniform dispersion of nanotubes in the resin matrix, and to lower particle agglomeration. A homogeneous composite paste was obtained by ultrasonication and mixing mechanically. The prepared composite paste was carefully transferred into stainless steel mould of standard dimensions for mechanical testing. Polyester strips and glass slides were placed on the mould surfaces to get smooth specimen surfaces and to minimize oxygen inhibition during curing.

The specimens were polymerized by light curing unit (LED) with controlled curing conditions according to recommended curing intensity and exposure time of the manufacturer. The specimens were removed from the mold after curing and subsequently finished using abrasive polishing papers to eliminate surface irregularities. The fabricated samples were immersed in distilled water at 37°C for 24 hours before mechanical and morphological characterization as shown in Figures 3 and 4. The prepared specimens were then subjected to flexural strength testing, compressive strength evaluation, microhardness measurement, water sorption studies and morphological analyses like Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and Fourier Transform Infrared Spectroscopy (FTIR).

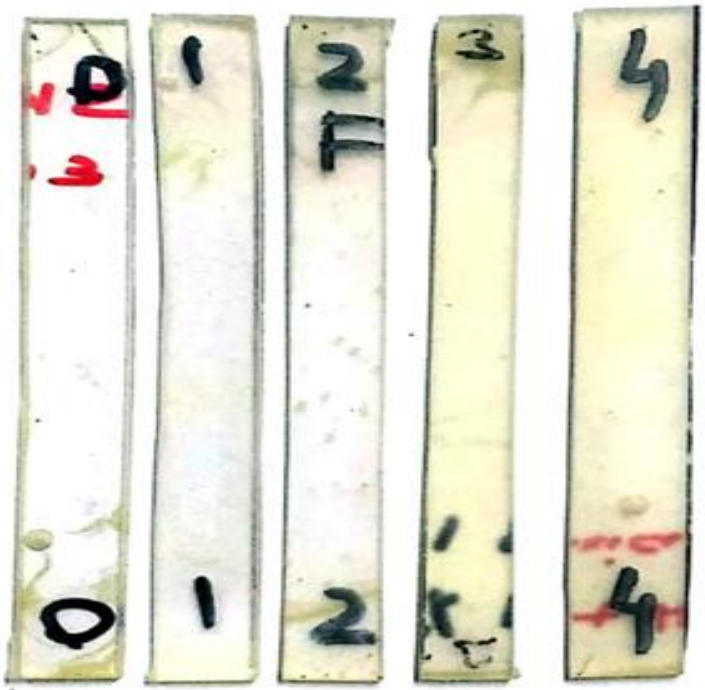


Figure 3. Fabricated HNT-reinforced UDMA composite specimens with different halloysite nanotube loadings (0–3 wt%).

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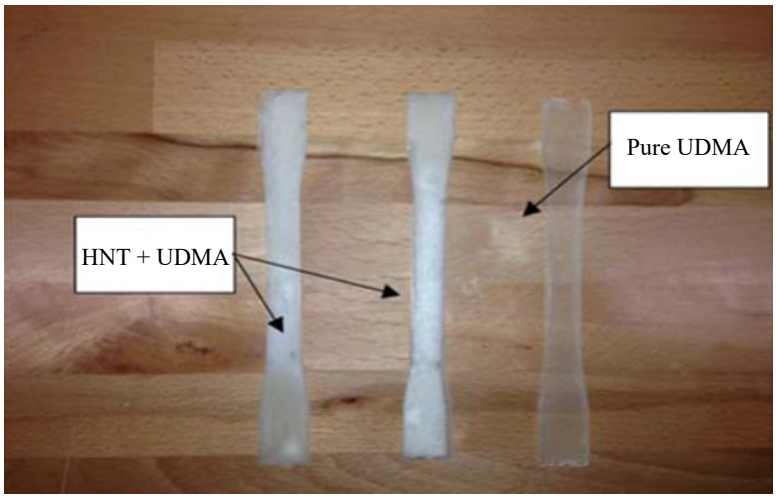


Figure 4. HNT distribution within the UDMA resin matrix.

CHARACTERIZATION METHODS

The prepared Halloysite Nanotube (HNT) reinforced Urethane Dimethacrylate (UDMA) dental composites were characterized using mechanical, physical and morphological evaluation techniques to investigate the effect of HNT incorporation on the overall performance of the developed nanocomposites. Standardized testing procedures were adopted to produce reliable and reproducible experimental results as mentioned in Table 2.

Table 2. Characterization techniques and evaluation parameters of HNT-reinforced UDMA dental composites.

Property	Suggested standard/instrument	Specimen size	Output	Interpretation
Flexural strength	Three-point bending; ISO 4049	25 x 2 x 2 mm	MPa	Resistance to bending fracture.
Flexural modulus	Calculated from load-deflection curve	Same as flexural test	GPa	Stiffness.
Compressive strength	Universal testing machine	Cylindrical specimens	MPa	Bulk load resistance.
Microhardness	Vickers hardness tester	Polished discs	HV	Surface resistance to indentation.
Water sorption	Gravimetric method; ISO 4049	Disc specimens	ug/mm3	Hydrolytic stability.
SEM	Scanning electron microscope	Fracture surface	Micrographs	Dispersion, voids, crack path.

Flexural Strength Analysis

Flexural strength is one of the most important parameters of dental restorative materials since restorations are constantly subjected to bending and tensile stresses during mastication [40]. Results showed that the flexural strength monotonically increased with the HNT loading up to an optimum loading of 1 wt%.

The flexural strength of the control specimen was 92 MPa. The addition of 0.5 wt% HNT resulted in an increase of strength to 104 MPa, that is an improvement of approximately 13%. The maximum strength of 116 MPa was attained at 1 wt% loading of HNT, showing an increase of nearly 26% with respect to the control. The enhancement can be ascribed to the effective stress transfer between the nanotubes and the resin matrix and the crack deflection mechanisms offered by the tubular structure of HNTs [41, 42].

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However, a small reduction in strength was noticed for the 2 wt% and 3 wt% HNT concentrations as shown in Figure 5. This reduction is primarily associated with nanotube agglomeration and the development of localized stress concentration regions that adversely influence the load transfer efficiency [41].

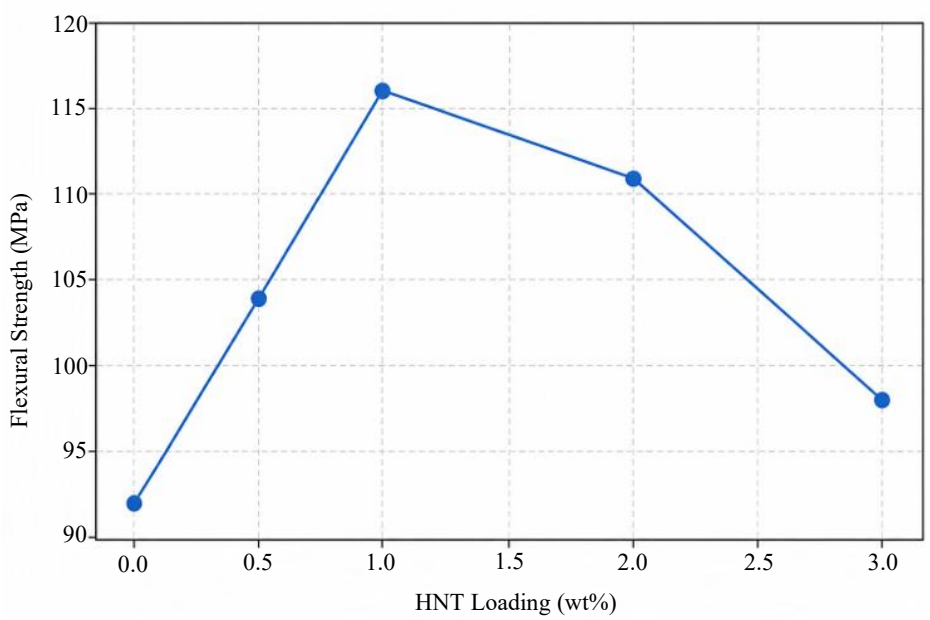


Figure 5. Effect of halloysite nanotube (HNT) loading on the flexural strength of UDMA dental composites.

Flexural Modulus Analysis

The flexural modulus was determined to assess the stiffness of the composite materials. With increasing nanotube loading, the flexural modulus increased monotonically up to 2 wt%. A maximum modulus value of 3.7 GPa was observed at 2 wt% HNT loading, showing a significant increase in stiffness as shown in Figure 6.

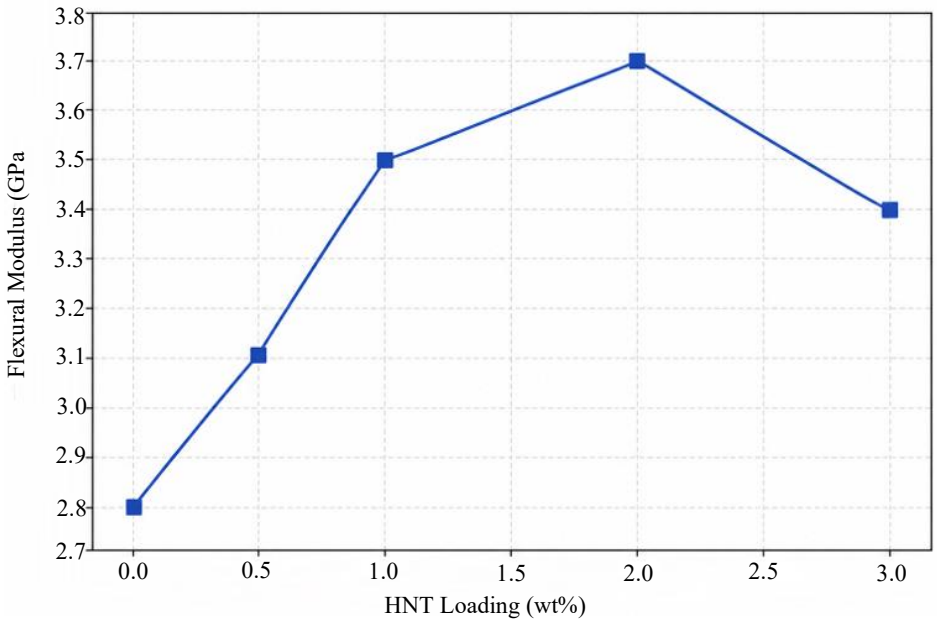


Figure 6. Variation in flexural modulus of UDMA dental composites with HNT loading.

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The enhancement of modulus is due to the rigid aluminosilicate structure of HNTs that restricts the mobility of polymer chains and improves the load-bearing ability. The modulus showed a slight decrease at 3 wt% loading due to filler clustering and reduced dispersion efficiency.

Compressive Strength Analysis

Compressive strength is an important property of dental restorations, since occlusal forces are predominantly compressive [43]. The control specimen had a compressive strength of 238 MPa. The addition of HNTs enhanced compression resistance significantly. The 1 wt% HNT composite exhibited the highest compression strength of 273 MPa as shown in Figure 7.

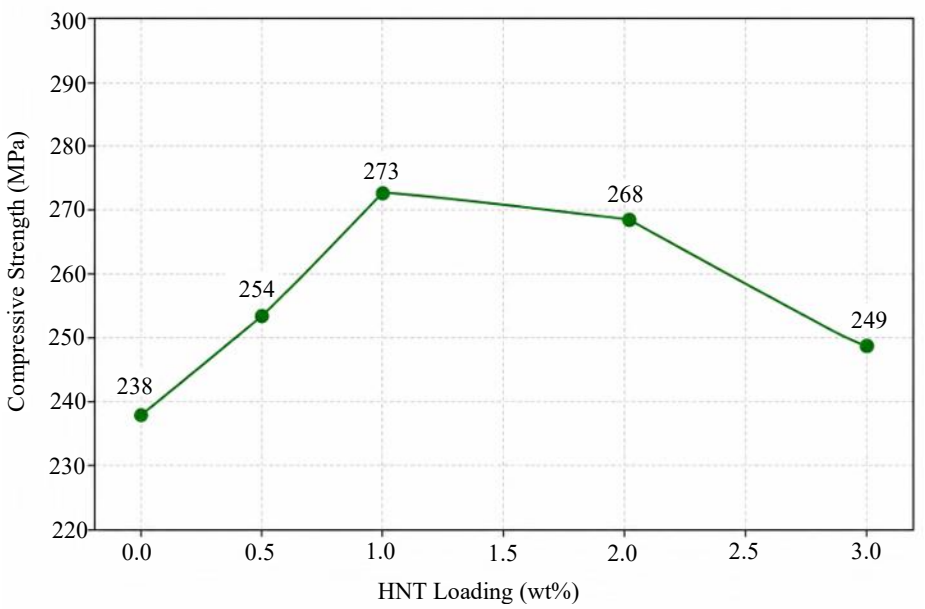


Figure 7. Influence of HNT content on the compressive strength of UDMA dental composites.

This improvement is due to the enhanced stress distribution and reinforcement from the uniformly dispersed nanotubes. Further increase in the HNT concentration led to a small decrease in the compressive strength due to agglomeration of nanotubes and increased defect formation in the matrix [44].

Vickers Microhardness Test

The surface hardness of fabricated composites was measured using Vickers micro hardness tester. Polished composite specimens were indented under a fixed load for a certain dwell time with a diamond indenter. The optical microscope was used to measure the diagonal lengths of the indentation marks after the indentation, and the Vickers Hardness Number (VHN) was calculated [45]. Resistance of the composites surface to the localized plastic deformation and wear was determined by microhardness test [46].

The results showed that the inclusion of HNTs significantly improved the surface hardness of the composites. The maximum hardness value of 66 HV was found for 1 wt% HNT composite as shown in Figure 8. The increase in the hardness value is due to the high rigidity and reinforcement effect of halloysite nanotubes. Improved hardness provides better resistance to abrasion and wear in clinical service.

Impact Strength

Impact strength results showed that the incorporation of Halloysite Nanotubes (HNTs) greatly improved the toughness and energy absorption capacity of UDMA dental composites.

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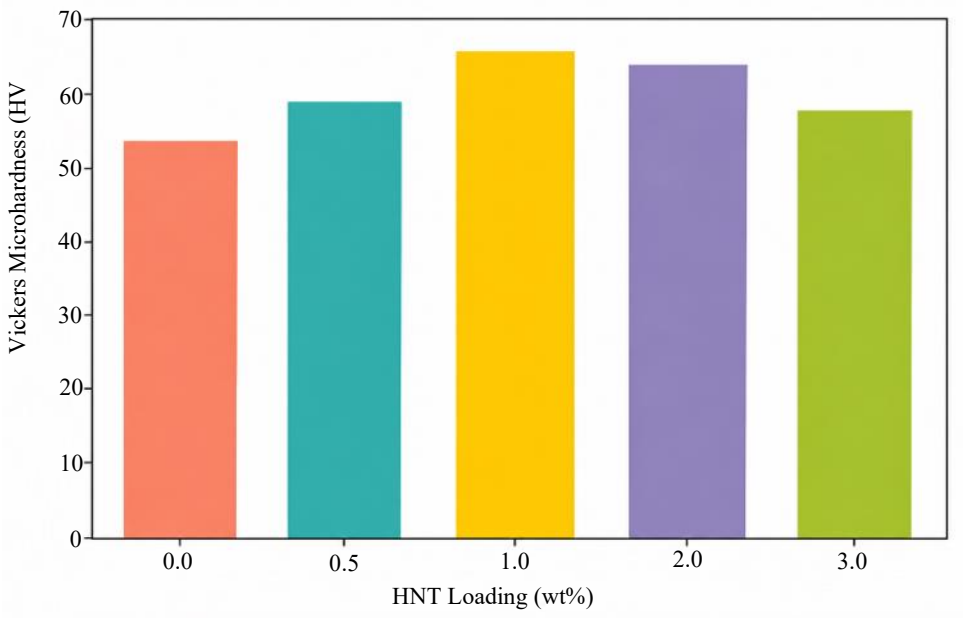


Figure 8. Effect of halloysite nanotube (HNT) loading on the Vickers microhardness of UDMA dental composites.

The impact strength was increased from 12.1 kJ/m² for the unfilled composite to 13.8 kJ/m², 16.6 kJ/m² and 17.9 kJ/m² for composites filled with 0.5 wt%, 1 wt% and 2 wt% of HNTs, respectively. The maximum impact strength was obtained at 2 wt% HNT loading which was approximately 48% higher than that of the control specimen. Such improvement is ascribed to the efficient crack-bridging, crack-deflection and energy dissipation mechanisms provided by the uniformly dispersed nanotubes in the UDMA matrix [47]. However, at 3 wt% HNT loading, impact strength reduced to 15.2 kJ/m^2 which could be attributed to nanotube agglomeration and formation of stress concentration sites as shown in Figure 9 [47, 48]. Overall, the results show that the optimal impact strength is obtained when 2 wt% HNTs are loaded, thus, the addition of 2 wt% HNTs is very suitable for the improvement of the durability and fracture resistance of UDMA-based dental restorative composites [48].

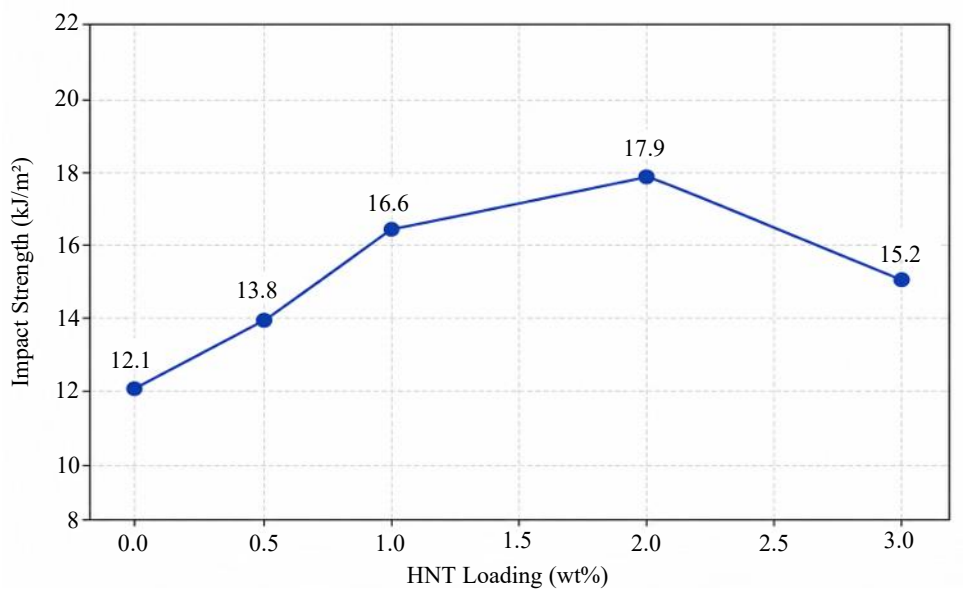


Figure 9. Impact strength of UDMA dental composites at different HNT loading levels.

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Water Sorption and Solubility Analysis

Water sorption and solubility behavior of the composites were evaluated according to the recommendations of ISO 4049 [49]. The specimens in the form of discs were dried initially in a desiccator to a constant weight and immersed in distilled water at 37°C for the specified duration. The specimens were weighed at regular intervals to study their water absorption behavior. The samples were reconditioned and reweighed after immersion to evaluate the material solubility. The analysis was carried out to study hydrolytic stability and moisture sensitivity of the developed composites in simulated oral conditions [50].

The water sorption was decreased with the loading of HNT up to 1 wt%. This behavior shows better packing of matrices and less free volume in the composite structure as shown in Figure 10. However, at higher concentrations the water uptake increased, probably because of the presence of agglomerates and hydroxyl-rich nanotube surfaces that can promote moisture uptake.

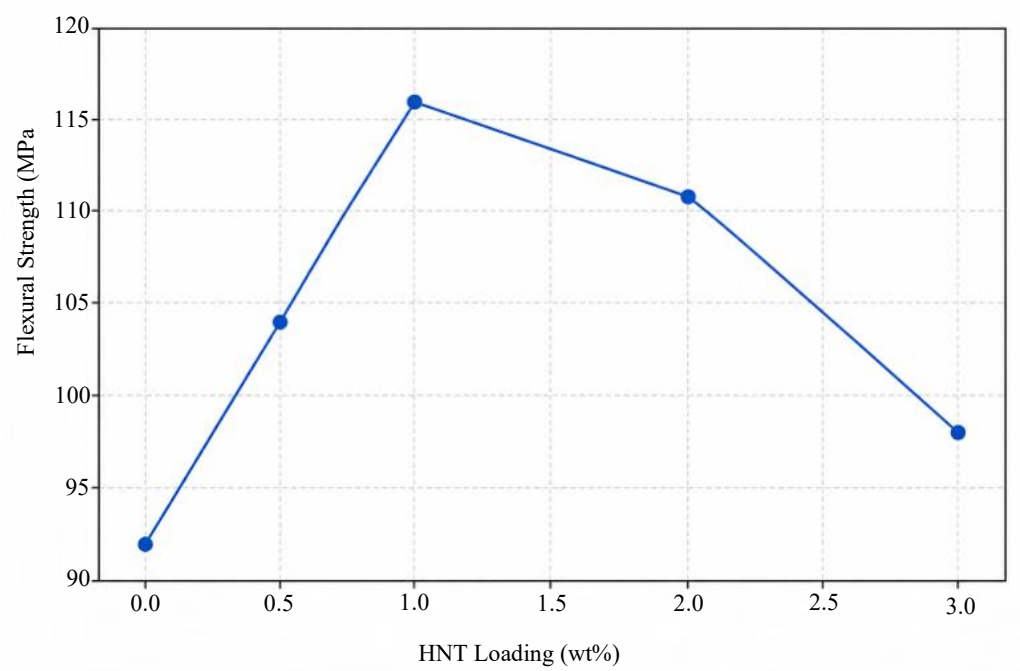


Figure 10. Influence of halloysite nanotube content on the water sorption behavior of UDMA-based dental composites.

Scanning Electron Microscopy (SEM)

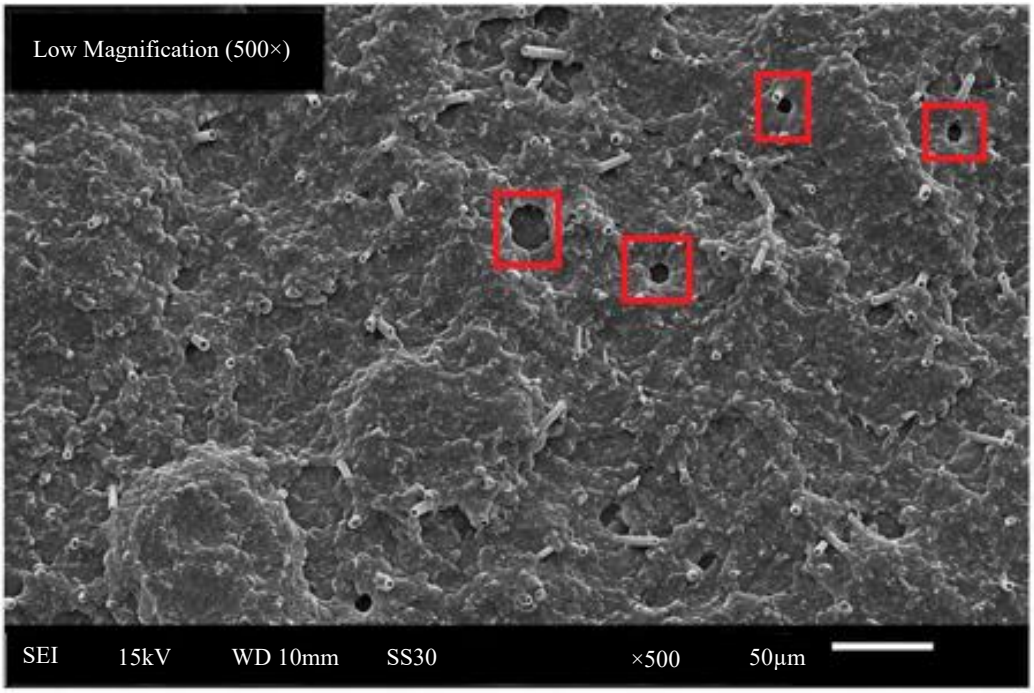
The surface morphology and fracture characteristics of the fabricated composites were investigated by Scanning Electron Microscopy (SEM). The fractured specimens after mechanical testing were cleaned, dried and coated with a thin conductive metallic layer by sputtering for examination [51]. SEM analysis was performed to observe the dispersion of HNTs in the resin matrix, filler pull out, crack propagation, void formation, agglomeration behavior and interfacial adhesion between the nanotubes and the polymer matrix. Morphological observations obtained through SEM were correlated with the mechanical performance of the composites [52].

The 0.5 wt% HNT composite exhibited a uniform dispersion of nanotubes with minimum void formation. The 1 wt% HNT well dispersed nanotubes served as crack arresters and facilitated effective stress transfer throughout the matrix. By contrast, the 3 wt% HNT composite showed substantial nanotube agglomeration and filler clustering as shown in Figure 11. These agglomerates acted as defect sites and lowered the reinforcing efficiency of the nanotubes.

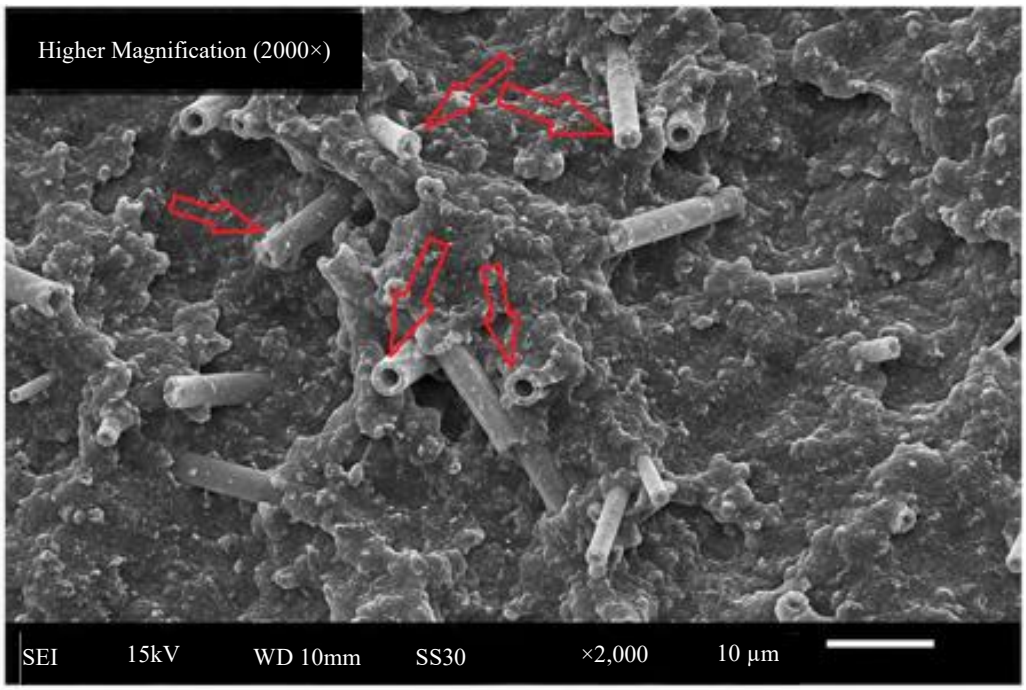
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RESULTS

The mechanical and morphological characterization of Halloysite Nanotube (HNT) reinforced Urethane Dimethacrylate (UDMA) dental composites were performed to evaluate the influence of nanotube incorporation on the overall performance of the developed restorative material. Composite specimens with 0 wt%, 0.5 wt%, 1 wt%, 2 wt% and 3 wt% HNTs were prepared and characterized by different testing techniques. The results obtained showed that the addition of HNTs significantly affected the mechanical, physical and morphological behavior of the UDMA matrix.

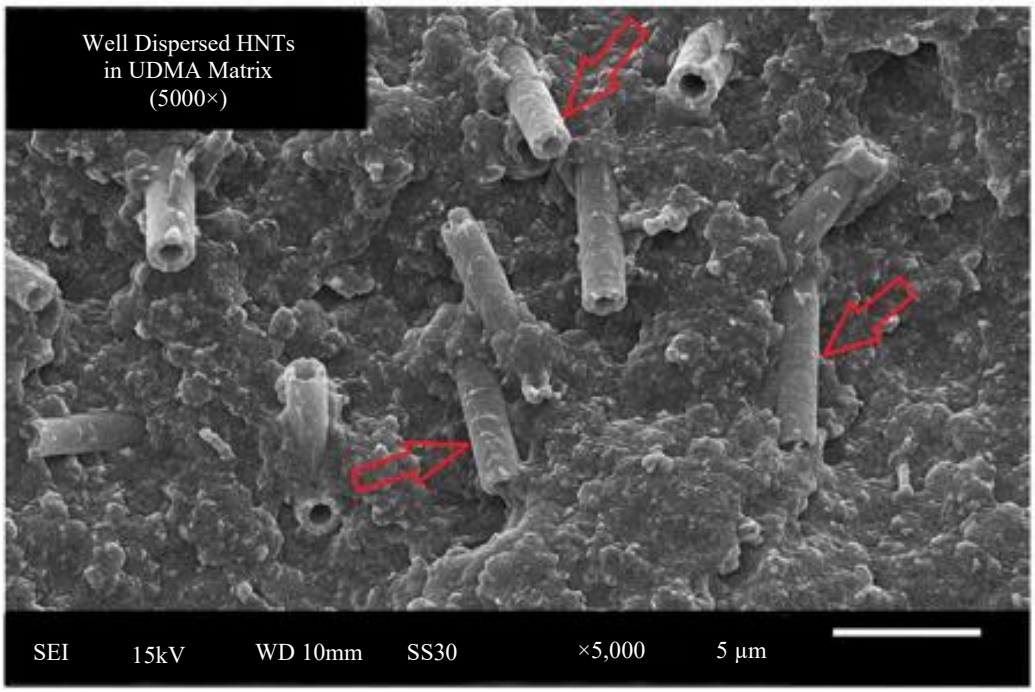


(a)

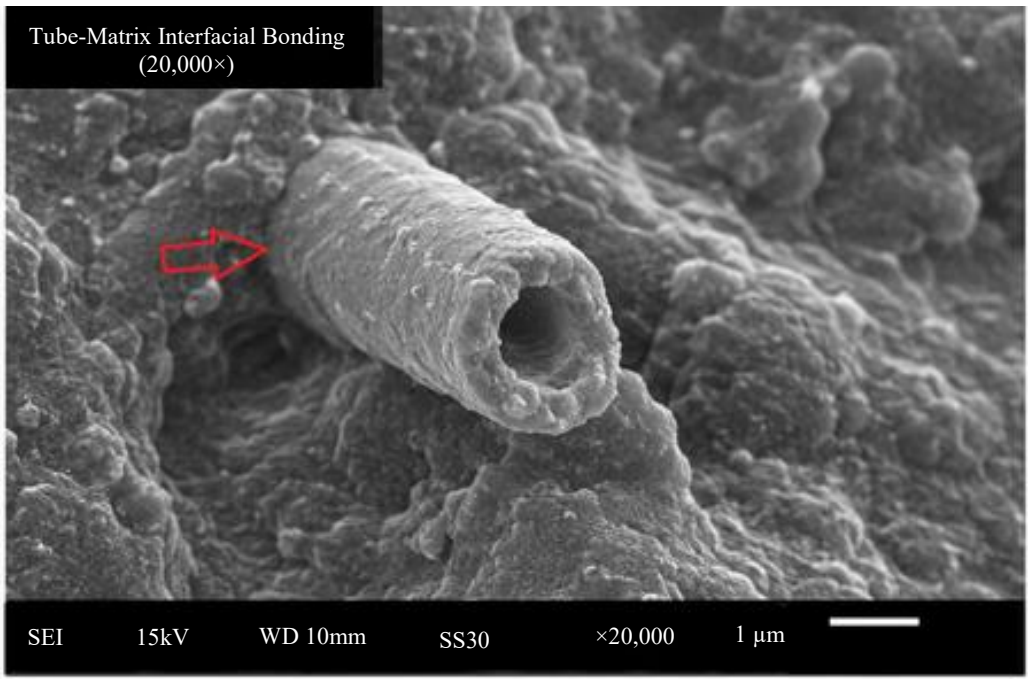


(b)

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(c)



(d)

Figure 11. Schematic SEM interpretation: (a) 0.5 wt.% minimum voids formation, (b) 3 wt.%, agglomerated regions at excessive loading, (c)1 wt.% well dispersed HNT nanotubes, (d) Tube–matrix interfacial bonding between HNT and UDMA.

Representative Mechanical Results

The mechanical properties and water sorption behavior of HNT-reinforced UDMA dental composites were evaluated to determine the effect of HNT incorporation on composite performance. The addition of HNTs influenced the flexural strength, flexural modulus, compressive strength, impact strength, and surface hardness of the UDMA matrix. At appropriate HNT loading levels, the nanotubes can improve stress transfer between the filler and resin matrix, resulting in enhanced mechanical performance.

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At low levels of HNT, the improvement in flexural strength and hardness is due to the high aspect ratio of halloysite nanotubes, mechanical interlocking, crack deflection, and stress transfer at the resin-filler interface. Further improvement of the interfacial compatibility can be achieved by silanization of the surface with methacrylate groups that can participate in the polymer network.

The reduction of strength at high HNT concentrations agrees with the usual behavior of nano filled polymer composites as mentioned in Table 3. Agglomerates of nanotubes can reduce effective interfacial area, increase local viscosity during fabrication, trap voids, decrease polish ability and create stress concentration sites. Therefore, optimum loading should be selected based on combined assessment of mechanical, morphological and handling properties, not simply strength.

Table 3. Mechanical properties and water sorption behavior of HNT-reinforced UDMA dental composites.

Group	HNT (wt%)	Flexural strength (MPa)	Flexural modulus (GPa)	Compressive strength (MPa)	Microhardness (HV)	Water sorption (ug/mm^3)
G0	0	92 +/– 6	2.8 +/– 0.2	238 +/– 13	54 +/– 3	21.5 +/– 1.2
G1	0.5	104 +/– 5	3.1 +/– 0.2	254 +/– 11	59 +/– 2	20.3 +/– 1.0
G2	1.0	116 +/– 7	3.5 +/– 0.3	273 +/– 14	66 +/– 3	19.1 +/– 0.8
G3	2.0	111 +/– 6	3.7 +/– 0.3	268 +/– 12	64 +/– 2	19.8 +/– 0.9
G4	3.0	98 +/– 8	3.4 +/– 0.4	249 +/– 16	58 +/– 4	22.4 +/– 1.4

Water sorption and degree of conversion are also important for dental applications. The surface hydroxyl groups in HNTs could affect hydrophilicity, and a high filler content could hinder the light transmission and the depth of polymerization. Therefore, the mechanical testing should be accompanied by FTIR-based degree of conversion analysis and depth-of-cure testing.

Morphological Changes After Mechanical Failure and Their Relation to Fracture Behavior

When HNT/UDMA composites fail (in flexure, compression, or fracture toughness testing), the fracture surface morphology examined by SEM reveals several characteristic features that map directly onto the failure mechanisms and the macroscopic mechanical response:

- *Nanotube Pull-Out:* Empty cylindrical sockets/holes on the fracture surface, sometimes with protruding tube segments, indicate the HNT debonded from the matrix and was pulled out rather than fractured in place. This is generally a *toughening* signature pull-out consumes fracture energy through interfacial friction and is associated with improved fracture toughness/work-of-fracture, provided pull-out length is moderate (not indicative of near-zero adhesion).
- *Nanotube Fracture:* Clean, flush-broken tube ends embedded in the matrix suggest strong interfacial bonding (often after silanization) load was transferred efficiently enough that the filler itself fractured rather than debonding. This usually correlates with higher strength/modulus but can reduce toughness if it removes crack-deflection pathways.
- *Crack Deflection and Bridging:* Tortuous, non-planar crack paths that deviate around nanotube clusters, versus flat/mirror-like fracture in unfilled resin, indicate deflection toughening. “Bridging” ligaments (HNTs spanning the crack faces before failure) are sometimes visible near the crack tip region.
- *Debonding-Induced Voids/Microcracking:* At higher loadings or with poor silanization, interfacial gaps/micro voids around agglomerates are visible even before macroscopic failure (sometimes evident as porosity in the bulk cross-section, not just the fracture plane).These act as stress concentrators and crack initiation sites – this explains the classic “increase-then-decrease” mechanical trend: at low wt%, HNTs toughen via pull-out/deflection; beyond an optimum (~1–5 wt% depending on the study), agglomerate-associated voids dominate and properties fall.

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Effect of HNT Surface Treatment/Functionalization on Mechanical and Morphological Properties

Because raw HNTs have a hydrophilic, hydroxylated surface (Al–OH on the outer/inner surfaces, siloxane character on parts of the tube) that is poorly compatible with the hydrophobic UDMA network, functionalization is usually the single biggest lever on composite performance. Table 4 shows how HNT surface treatment affects mechanical and morphological properties.

Table 4. Effect of HNT surface treatment on mechanical and morphological properties.

Treatment	Mechanism	Typical effect on mechanical properties	typical effect on morphology
Silanization (e.g., γ-MPS, γ-methacryloxypropyltrimethoxysilane)	Silane's methoxy groups hydrolyze/condense with surface –OH on HNT; methacrylate end co-polymerizes with UDMA/TEGDMA during curing.	Generally, the most effective improves flexural strength, modulus, hardness, and fracture toughness vs. untreated HNT at equivalent loading; allows higher wt% loading before decline.	Fracture surfaces show tube fracture/embedded tubes rather than clean pull-out sockets; SEM/TEM shows more uniform dispersion, fewer visible agglomerates; FTIR confirms new Si–O–Si and C=O ester peaks.
Acid treatment/etching (HCl, H2SO4)	Removes surface impurities (Al_2O_3 layer), can partially widen lumen, increases surface –OH density and roughness.	Can improve mechanical properties modestly by increasing mechanical interlocking/surface area for subsequent silanization; used mainly as pretreatment before silanization.	Rougher, more porous tube surface in TEM/SEM; increased surface area confirmed by BET.
Calcination/thermal treatment	Removes physisorbed/interlayer water, alters surface hydroxyl density.	Can improve dispersion and thermal stability; effect on mechanical properties is more modest/inconsistent than silanization	TGA shows altered dehydroxylation behavior; XRD shows loss of interlayer water peak.
Polymer grafting (e.g., PMMA, PDA/polydopamine, PEG)	Grafted chains provide steric compatibility and sometimes covalent linkage points.	Can enhance toughness (energy dissipation via chain pull-out) more than strength; polydopamine coating also imparts some functional/antibacterial benefit	TEM shows visible coating layer (few-nm shell) around tubes; SEM fracture shows fibrous/stringy residue at pull-out sites (polymer bridging).
Lumen loading (drug/ion loading, not strictly "surface" treatment)	Vacuum-assisted loading of Ag+, chlorhexidine, Zn, or remineralizing ions into the lumen	Usually mechanically neutral to slightly negative (lumen filling does not much effect outer-surface bonding, but processing/vacuum cycling can introduce defects) main purpose is functional (antibacterial/remineralization), not mechanical	EDS/XPS confirms loaded species; SEM cross-sections occasionally show lumen content; no major fracture surface change unless loading damages tube walls.

Effect of HNT Reinforcement on Polymerization Shrinkage and Degree of Conversion (DC)

This is a somewhat separate but clinically important axis, since polymerization shrinkage stress is a major cause of marginal leakage/debonding in restorations, and DC governs both mechanical properties and biocompatibility (unreacted monomer leaching).

Polymerization Shrinkage

- *General Trend:* HNT addition tends to reduce volumetric polymerization shrinkage in a dose-dependent way, because the inorganic filler is a non-reactive diluent that reduces the *volume*

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fraction of reactive monomer per unit composite volume the same "filler dilution" effect seen with any rigid inorganic filler (silica, glass, etc.) in dental composites.

The magnitude of shrinkage reduction depends on:

- *Filler Loading:* Higher wt%/vol% generally more shrinkage reduction, up to the point where dispersion problems emerge)
- *Filler–Matrix Interfacial Bonding:* Well-bonded (silanized) HNTs constrain matrix contraction more effectively than poorly-bonded ones, which can locally debond and only partially resist shrinkage
- *Nanotube Geometry/Aspect Ratio:* The tubular/rod-like shape may impart some geometric constraint on shrinkage vectors different from spherical fillers, though this is less well quantified than the simple dilution effect
- Shrinkage is typically measured by bonded-disk/linometer methods, or occasionally by micro-CT/dilatometry.

Degree of Conversion (DC)

- *General Trend:* DC often shows a *slight decrease with increasing HNT content*, particularly at higher loadings (this is the more commonly and consistently reported effect across nanoclay/nanotube-filled dental resins generally, not unique to HNT).
- *Mechanisms:*
  - *Light Scattering/Attenuation:* HNTs (being an opaque/semi-opaque aluminosilicate) scatter and absorb curing light, reducing the effective light dose reaching deeper/more distant regions of the specimen this is the dominant explanation, especially for thicker specimens or greater cure depths.
  - *Increased Viscosity:* Higher filler loading increases paste viscosity, restricting monomer/radical mobility late in cure, which can trap some double bonds unreacted (vitrification-limited conversion).
  - *Potential Radical Scavenging/Inhibition at Filler Surface:* Particularly for untreated HNTs with unreacted surface hydroxyls, though this is a comparatively minor/inconsistent effect versus the optical/viscosity effects.

COMPARATIVE SUMMARY

The comparative analysis was performed to evaluate the effect of HNT reinforcement on the overall performance of UDMA dental composites as mentioned in Table 5. The mechanical properties of the HNT-reinforced specimens were compared with different loading rates of HNT and effect of surface treatment and water behavior are analyzed.

Table 5. Influence of halloysite nanotube (HNT) reinforcement on the performance of UDMA dental composites.

Factor	Positive effect of HNT	Possible negative effect	Recommended evaluation	Design implication
Low loading	Improved strength and hardness	Minor viscosity increase	Flexural and hardness tests	Useful reinforcement range.
High loading	Higher stiffness	Agglomeration and brittleness	SEM and fracture analysis	Avoid excessive filler.
Silanization	Better interface bonding	Extra processing step	FTIR, SEM, mechanical tests	Recommended stable reinforcement.
Morphology	Crack deflection	Void formation if poorly mixed	SEM/EDS mapping	Optimize dispersion.
Water behavior	Potential barrier effect	Hydroxyl-driven sorption	Water sorption/solubility	Balance hydrophilicity.

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LIMITATIONS

The present study demonstrates the potential of Halloysite Nanotube (HNT) reinforced UDMA dental composites; however, some limitations should be considered. The study is mainly based on in vitro testing conditions that may not completely reflect the complex oral environment including temperature variations, saliva, pH changes, microbial activity, and cyclic masticatory forces. The study is mainly focused on mechanical and morphological characterization, while other clinically relevant properties – such as wear resistance, fatigue behavior, color stability, biocompatibility, antibacterial activity, and long-term aging performance – were not thoroughly evaluated. Moreover, a uniform dispersion of HNTs within the resin matrix remains a challenge, especially at higher filler loadings where nanotube agglomeration can adversely affect the performance of the composites. The sample size and range of HNT concentrations tested may also limit the generalizability of the results. Thus, further long-term in vivo studies and clinical trials are required to confirm the effectiveness and durability of HNT-reinforced UDMA dental composites for routine restorative dental applications.

CONCLUSIONS

The present study indicates the potential of Halloysite Nanotubes (HNTs) as effective reinforcing nanofillers for Urethane Dimethacrylate (UDMA)-based dental composites. The addition of HNTs considerably enhanced the mechanical and morphological properties of the composites, improving flexural strength, compressive strength, stiffness and surface hardness. Morphological analyses indicated that the excellent dispersion of the nanotubes and the strong interfacial bonding between the HNTs and the resin matrix were important for improving the performance of the composites. Moderate HNT loading proved to be the best compromise between mechanical reinforcement and structural integrity among the studied formulations, while higher filler loadings gave agglomeration of the nanotubes and performance decreases. Moreover, the research emphasizes the significance of filler dispersion, surface modification, and optimized filler content for achieving high-performance dental restorative materials. In conclusion, UDMA composites reinforced with HNTs have shown great potential for advanced restorative dentistry applications and may be used for the development of durable, reliable and long-lasting dental restorations.

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Declaration of Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Ethical Approval

This study did not involve human participants, human tissues, animals, or clinical trials. Therefore, ethical approval and informed consent were not required.

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