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Research Article

Effect of Hybrid Nanofillers on the Mechanical Characteristics of Polymethyl Methacrylate Denture Base Composite

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Abstract

The research study focused on enhancing the mechanical characteristics of polymethyl methacrylate (PMMA) denture bases. PMMA is commonly used in dentistry due to its easy fabrication, cost-effectiveness, and favourable physical properties. However, its limitations include low wear resistance, hardness, and mechanical strength, making it less suitable for long-term dental applications. To address these limitations, the study employed a combination of hybrid nano-fillers, specifically HNTs (halloysite nanotubes) and MWCNTs (multi-walled carbon nanotubes), at varying loading levels to improve the mechanical characteristics of the PMMA composite. These nano-fillers underwent treatment by using a coupling agent to enhance their compatibility with PMMA. Key findings of the research include that introducing HNTs/MWCNTs into the PMMA matrix led to a substantial increase in flexural strength, with a significant improvement of 109.1 MPa compared to unfilled PMMA. This indicates that the composite material became more resistant to bending or deformation. There was a substantial rise in flexural modulus values, suggesting improved stiffness in the nanocomposite compared to unfilled PMMA. In addition, the tensile strength of the PMMA composite increased by 64.4 MPa with the addition of the hybrid nano-fillers, indicating enhanced resistance to stretching or pulling forces. The study found that the improvement in flexural and tensile strength was dependent on the concentration of MWCNTs. Increasing the MWCNT concentration up to 0.75 wt.% led to improved mechanical properties, but further increases resulted in a reduction in PMMA properties. Although there was a modest improvement in Vickers hardness (approximately 18.93 kg/mm^2), the addition of HNTs/MWCNTs as hybrid nano-fillers effectively enhanced the properties of the PMMA nanocomposite. The study concludes that incorporating hybrid nano-fillers into PMMA could contribute to the longevity and durability of dental composites, addressing some of the material's inherent limitations. The specific combination and concentration of nano-fillers played a crucial role in determining the mechanical properties of the resulting nanocomposite.

Keywords

Material for Denture Bases, Halloysite Nanotubes, Multi-Walled Carbon Nanotubes, Flexural Characteristics, Tensile Properties, Vickers Hardness

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1. Introduction

In recent years, polymers have played a crucial role in satisfying the increasing demands across various fields. These adaptable materials consist of large molecules composed of repeating monomer subunits, demonstrating unique properties that render them indispensable in engineering, chemistry, physics, and biomaterials science [1]. Among the versatile polymers utilized in numerous industries for diverse applications is polymethyl methacrylate (PMMA), recognized by trade names like Plexiglas, Acrylite, and Perspex. PMMA boasts distinctive characteristics that qualify it as an excellent choice for biomaterial applications, including bone cement, lenses, dentures, and denture bases [2]. The polymerization of methyl methacrylate (MMA) monomers results in the formation of PMMA, with the size and characteristics of PMMA micro-particles in laboratory or industrial production influenced by stabilizers such as alginate and gelatin [2]. Despite its favourable attributes, PMMA has limitations in tribological (friction and wear), mechanical, and hardness properties, which may limit its suitability for certain applications, including dental materials. To overcome these limitations, researchers have explored the incorporation of nanoparticles into polymer resin, creating composites. This innovative approach aims to improve the material's overall performance, making it more suitable for specific applications such as dental prosthetics [2-4].

The augmentation of mechanical properties in a polymer matrix like PMMA can be achieved through the incorporation of nanoparticles, a process commonly referred to as nanocomposite reinforcement. This involves integrating various types of nanoparticles, including organic nanofillers, silica (SiO_2), alumina, carbon nanotubes (CNTs), carbon nanofibers (CNFs), graphene, nano-metals, or titanium dioxide (TiO_2), into the polymer matrix. These nanoparticles act as reinforcement agents, leading to multiple enhancements in the mechanical characteristics of the material [2, 5-7]. In a specific experiment, multi-walled carbon nanotubes (MWCNTs) were successfully introduced into nylon-6 fibres as reinforcing fillers for PMMA composites. Nylon-6 fibers were dissolved in hexafluoro propanol at a concentration of 10% wt., with MWCNTs incorporated at two different concentrations (0.5% wt. and 1.5% wt.). The study focused on the flexural strength of dental resin PMMA composites containing nylon-6 fibers. The results revealed that adding MWCNTs at a concentration of 0.5% wt. positively impacted the flexural strength of the dental resin PMMA composites. This suggests that the incorporation of MWCNTs, particularly at the lower concentration of 0.5% wt., has the potential to enhance the mechanical properties of dental materials [2, 8]. Furthermore, the improvement of physical and mechanical properties was observed when hydroxyapatite (HA) nanoparticles were incorporated into PMMA denture resin. The researchers tested various filler loadings of HA nanoparticles in the PMMA resin, ranging from 0% to 0.8% by weight. The study demonstrated that as the filler loading of HA nanoparticles increased, the mechanical properties of the PMMA/HA nanocomposites improved. This indicates that the addition of HA nanoparticles enhances the strength, stiffness, or other mechanical properties of denture resin. Additionally, the friction coefficient gradually decreased with increasing filler loading of HA nanoparticles, suggesting increased lubricity or reduced friction, which can be advantageous in dental applications. Similarly, wear loss decreased with higher filler loading, implying that denture resin with HA nanoparticles experiences less wear and tear, potentially enhancing its durability and lifespan [2, 9].

The utilization of hybrid nanofillers presents a promising strategy within the realm of nanocomposites, as it enables the amalgamation of beneficial properties from diverse filler types, including both nanoparticles and fibres. The core concept behind incorporating hybrid nanofillers is to harness the synergistic interactions among these constituents to elevate the overall physical and mechanical attributes of the nanocomposite material. When hybrid nanofillers were introduced into the PMMA polymer matrix, substantial enhancements in impact strength and other mechanical properties were observed [2, 10, 11]. An investigation was conducted to assess the impact of incorporating a 2wt.% hybrid filler consisting of zirconium dioxide (ZrO_2) and aluminium oxide (Al_2O_3) nanoparticles in a 1:1 weight ratio into a denture base made of heat-cured acrylic resin (PMMA). The study focused on measuring the impact strength, transverse strength, and thermal conductivity of the denture base with and without this filler. The addition of the 2wt.% filler of ZrO_2/Al_2O_3 nanoparticles in a 1:1 ratio resulted in a significant increase in both impact and transverse strength of the denture base. The study also noted a progressive influence on thermal conductivity with the 1:1 ratio of ZrO_2/Al_2O_3 nanoparticles [12]. Another investigation explored the impact of adding different amounts of glass fibres (GFs) and zirconium oxide nanoparticles to PMMA. The test samples featured varying mixtures of nano-ZrO_2 and glass fibres at a total concentration of 5% by weight, while a control sample had no additives. Compared to unreinforced PMMA samples, the most recent hybrid composites exhibited substantial improvements in impact and flexural strengths, with optimal values achieved at a 2.5% mixing ratio of both ZrO_2 NPs and GFs [13]. In a recent study, a hybrid filler composed of Al_2O_3/TiO_2 nanoparticles was employed to enhance the properties of PMMA denture bases. PMMA nanocomposite samples with various loading contents of this hybrid filler were fabricated and compared to an unfilled sample. The study demonstrated that incorporating the hybrid filler of Al_2O_3/TiO_2 nanoparticles into PMMA denture bases significantly enhanced their mechanical and tribological properties. The most favourable outcomes were achieved with a 0.8wt% filler content, resulting in a notable reduction in both the coefficient of friction (by up to 20%) and wear rate

(by up to 28%) compared to the unfilled sample [2]. Furthermore, an evaluation was conducted on adding polypropylene (PP) fibres and Al_2O_3 nanoparticles (NPs) as fillers to a PMMA resin for composite material creation. The filler content used was 0.5%wt and 1.0%wt for both polypropylene fibres and Al_2O_3 nanoparticles, leading to improvements in hardness, impact strength, and thermal conductivity of the composite, albeit with no enhancement in transverse strength [2, 14]. Finally, an intriguing approach involved the incorporation of nanographene (NG) and a hybrid SiO_2-TiO_2 filler into PMMA denture resin, showcasing potential benefits for denture materials. The addition of NG/SiO_2/TiO_2 fillers demonstrated a reduction in the friction coefficient and a lower wear rate, while also increasing the hardness of the PMMA denture resin. This suggests that nanographene can enhance the mechanical properties of composites, including hardness, strength, and stiffness [2, 15].

In previous studies, the incorporation of hybrid fillers into the PMMA matrix has been employed strategy in materials science to enhance the different characteristics of PMMA composite materials. This current investigation is centered on introducing a hybrid nanofiller composed of HNTs and MWCNTs nanoparticles into PMMA denture resin. The objective is to elevate the mechanical properties of the PMMA denture material. The primary aim of this study is to examine the influence of hybrid HNTs/MWCNTs nanoparticle addition on the mechanical properties, including flexural properties, tensile properties, and Viker hardness, of PMMA composite. Specifically, the research seeks to comprehend how varying nanofiller quantities impact the composite properties. Through the manipulation of filler amounts, the study aims to pinpoint the optimal composition that maximizes denture performance. Positive outcomes from this research have the potential to result in the development of improved denture materials with enhanced mechanical properties, potentially enhancing patient comfort and extending the lifespan of dentures.

2. Research Material and Methodology

2.1. Materials

In this ongoing investigation, the materials utilized consisted of PMMA, a polymer typically comprising two elements: powder of PMMA and liquid of methyl methacrylate (MMA). The initiation of polymerization reactions involved the use of benzoyl peroxide (BPO). Additionally, ethylene glycol dimethacrylate (EGDMA) served as a cross-linking agent in the composite. The primary focus of this research revolves around enhancing the strength of PMMA composites through the incorporation of hybrid nanofillers. These nanofillers include halloysite nanotubes (HNTs) with diameters ranging from 20 to 50 nanometers and lengths spanning 500 to 1500 nanometers, as well as multi-walled carbon nanotubes (MWCNTs) with diameters between 10 and 30 nanometers and lengths extending over several microns. To enhance the interaction between PMMA and these hybrid nanofillers, a coupling agent, specifically silane (3-trimethoxysilyl propyl methacrylate, or γ-MPS), was employed during their treatment.

2.2. Surface Treatment of Nanofillers

The treatment procedure commenced by taking 10 grams of filler powder, which could be either HNTs or MWCNTs and blending it with 200 millilitres of toluene. Initially, the filler powder was dispersed in the toluene. In the subsequent step, a silane coupling agent was introduced to the filler powder at a concentration of 10wt%, all while maintaining room temperature. The mixture was continuously stirred at a rate of 150 rotations per minute (rpm) for 15 hours. After this stage, the solution underwent filtration to segregate and gather the modified filler powder from the liquid solution. To purify the collected modified filler powder, a Soxhlet apparatus was utilized, employing 300 mL of fresh toluene over 24 hours. Subsequently, the modified filler powder underwent drying in a vacuum oven at 110 ℃ for 3 hours to eliminate any remaining solvents or impurities from the modified filler material.

2.3. Composite Material Preparation

The powder components, comprising PMMA and 0.5wt.% BPO were employed in this process. The liquid mixture was formulated by combining 90% MMA monomer and 10% EGMMA. To impede rapid polymerization, a small quantity of hydroquinone (0.025%) was introduced into the liquid medium. Additionally, hybrid nanofillers consisting of HNTs/MWCNTs were integrated as reinforcing particles at varying concentrations (as specified in Table 1, denoted as G2 to G7). The treated hybrid nanofillers were initially dispersed in the MMA monomer using an ultrasonic bath for 5 minutes. Subsequently, this mixture was blended with the PMMA powder until it reached a dough-like consistency, typically taking approximately 15 minutes. The resulting composite material is packed into moulds. The filled moulds are placed into a dental flask, and a pressure of 14MPa is applied using a hydraulic press. This pressurization process lasts for 35 minutes, and the pressurization is performed at room temperature. After pressurization, the moulds are subjected to polymerization. Polymerization is achieved by immersing the moulds in a water bath at a temperature of 79 ℃. Polymerization is maintained for 90 minutes. After polymerization, the moulds are removed from the water bath. The moulds are allowed to gradually cool to room temperature. The final step involves finishing and polishing the rigid samples. This is done using an X 35 electric handpiece or 240 emery paper.

Table 1. Proportions of the constituents in the powder blend, including treated HNTs and MWCNTs particles fixed at 5wt.%, serving as fillers in the PMMA denture base.

Samples code	Matrix of PMMA (wt%)		Hybrid nanofillers (wt%)	
	PMMA	BPO	HNTs	MWCNTs
G1	99.50	0.50	0	0
G2	94.50	0.50	4.75	0.25
G3	94.50	0.50	4.50	0.50
G4	94.50	0.50	4.25	0.75
G5	94.50	0.50	4.00	1.00
G6	94.50	0.50	3.50	1.50
G7	94.50	0.50	2.50	2.50

3. Preparation and Measurements of Specimens

3.1. Measurements of Flexural Properties

The evaluation of the flexural properties of composite materials commonly employs the ASTM D790-03 standards. In this analysis, a rectangular specimen undergoes bending until it fractures, with meticulous recording of both force and displacement. Mechanical property assessments were conducted using an Instron 3366-10kN machine. The specimens, tailored for this assessment, were rectangular, measuring 100mm in length, 13mm in width, and 3mm in thickness. The support span, denoting the distance between the two supports where the specimen rests during testing, was established at 50mm. The loading nose and support featured a 20mm diameter, representing the points of load application and specimen support. The test was executed at a cross-head speed of 2.00 mm/min, indicating the velocity of the machine's cross-head movement during the test. At least five samples were prepared for each formulation, aligning with the specific composition and processing conditions employed in material production. Flexural strength (FS) and flexural modulus (FM) data were recorded and calculated using Equations 1 and 2.

$$FS = \frac{3PL}{2bt^2} \quad (1)$$

$$FM = \frac{PL^3}{4bt^3d} \quad (2)$$

In the context of this equation, "P" denotes the maximum load in Newtons (N), "L" represents the span length (mm), "b" signifies the width of the sample (mm), "t" corresponds to the thickness of the specimen, and "d" refers to the slope of the tangent to the initial straight-line portion of the load-deflection curve (mm).

3.2. Measurements of Tensile Strength

The ASTM D-638 standard was utilized to measure the tensile properties of PMMA composites. The test was conducted using the INSTRON 3366 machine. The sample for this test was prepared with a length of 80mm, a width of 20mm, a thickness of 4mm, and a gauge length (distance between the grips) of 50mm. The crosshead speed for the tensile test was set at 5mm/min, indicating the rate at which the grips moved apart during the test. The calculation of the tensile strength and elongation of the PMMA composite involved the use of Equation 3.

$$TS = \frac{F}{A} \quad (3)$$

In this context, "TS" represents the tensile strength measured in Newtons per square millimetre (N/mm²), where "F" denotes the load at failure in Newtons (N), and "A" stands for the cross-sectional area at the point of failure measured in square millimetres (mm²). The calculation of" the elastic modulus (TM) was determined by utilizing Equation 4.

$$TM = \frac{\sigma}{\varepsilon} \; and \; \varepsilon = \frac{(I - I_0)}{I_0} \quad (4)$$

In this equation, "TM" represents the tensile modulus, "σ" stands for stress, "ε" denotes strain, "l" represents the pre-loading length of the specimens, and "l0" signifies the post-loading length of the specimens.

3.3. Vickers Hardness Measurements

The assessment of material hardness commonly involves the Vickers test, a method where a known load is applied to a diamond pyramid indenter with a square base, pressing it into the material's surface for a specified duration. The Vickers hardness (VH) is determined by measuring the lengths of the two diagonals of the resulting indentation. In this study, the VH test adhered to the ASTM 384:2008 standard. The sample dimensions for the VH test were as follows: length of 10mm, width of 10mm, and thickness of 3mm. The diamond pyramid indentation featured a square base at a relative angle of 136°, and 0.3kgf load was applied for 10seconds. To calculate the VH of the PMMA composite, the optical measurement of the mean diagonal length of indentation (μm) (d1 and d2) was performed in five different areas, and the average of these measurements was computed for each sample. The VH value was then determined using Equation 5.

$$VH = \frac{1.854\,L}{d^2} \quad (5)$$

In the equation, "VH" refer to Vickers hardness (kg/mm ^{2}), where "L" represents the load in kilograms and "d" signifies the diagonal length (mm).

4. Statistical Analysis

In this study, we utilized statistical methods, specifically ANOVA (One-way analysis of variance) and Tukey's post hoc analysis. ANOVA was applied to assess the data about flexural strength and modules, property of tensile, and surface hardness, aiming to identify any notable differences among the different groups and P value less than 0.05.

5. Results and Discussion

5.1. Confirmation of the Effect of Silane Coupling Agent Treatment of the Fillers

Figures 1 and 2 depict the outcomes of FESEM micrographs and EDX analysis for HNTs and MWCNTs nano-powder, both pre-silane and post-silane treatment. These visuals illustrate alterations in the surface structure and chemical composition of the ceramic fillers after undergoing the silane treatment procedure. The FESEM micrographs reveal shifts in surface morphology, while the EDX analysis highlights changes in the sample's chemical makeup. The EDX analysis indicates the utilization of a silane coupling agent to treat the nano-fillers in the HNTs/MWCNTs/PMMA composite, a common practice in composite production. Silane coupling agents serve to enhance the interaction between the filler and matrix materials, ultimately resulting in improved mechanical properties. The detection of Si elements at the filler-matrix interface provides concrete evidence of the silane coupling agent's presence. Additionally, assessing the weight and atom percentages of the filler elements and Si can offer further insights into the effectiveness of this treatment [16].

FESEM offers a means to obtain highly detailed images of a material's surface, allowing us to examine the structural characteristics of particles within the composite. Specifically, when assessing the 5wt% MWCNTs and HNTs in MWCNTs/PMMA and HNTs/PMMA composites, FESEM enables the observation of their shapes, dimensions, and distribution in the composite. In the case of the 5wt% MWCNTs and HNTs within the MWCNTs/HNTs/PMMA composite, EDX serves as a tool for confirming the presence of carbon and other elements within the MWCNTs and HNTs. Additionally, EDX can be utilized to quantify the relative concentrations of these elements in the composite, thereby providing valuable insights into the efficacy of the treatment process. The identification of the Si group in both the MWCNTs/PMMA and HNTs/PMMA composites indicates the successful treatment of ceramic fillers with a silane coupling agent, thus confirming the treatment process's effectiveness.

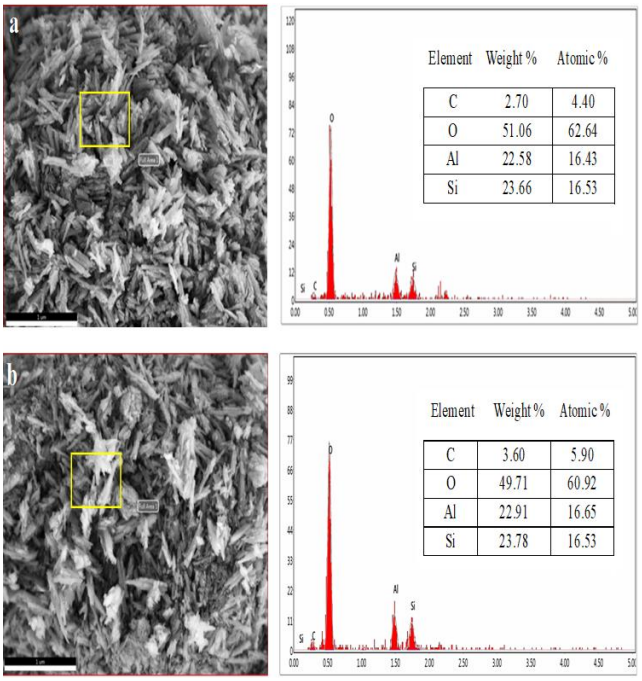

Figure 1. *Confirmation of silane treatment by FESEM and EDX of HNTs powder particles; (a) before silane treatment, (b) after silane treatment.*

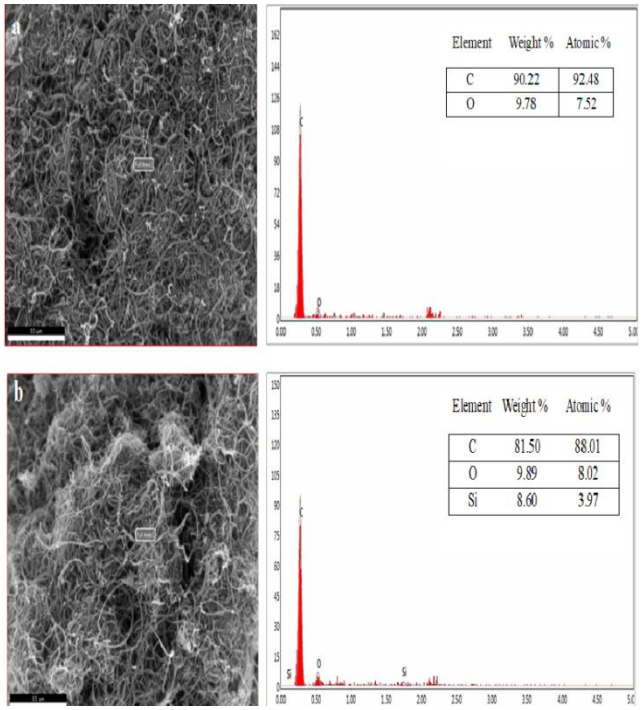

Figure *错误!文档中没有指定样式的文字。. Confirmation of silane treatment by FESEM and EDX of MWCNTs powder particles; (a) before silane treatment, (b) after silane treatment.*

5.2. Flexural Properties

The findings of the flexural properties of PMMA nanocomposites filled with hybrid nanofillers are presented in Figure 3 and Table 2. An increase in the amount of MWCNTs

in the composite resulted in a statistically significant rise in the flexural modulus of the HNTs/MWCNTs/PMMA nanocomposite ($p < 0.05$). Specifically, the flexural modulus increased from 2.50 GPa to 3.62 GPa. A higher flexural modulus denotes increased stiffness and resistance to bending or deformation under an applied load. The flexural modulus (FM) values of the hybrid nanofiller-filled PMMA composite exceeded those of the pure PMMA matrix and fell within an acceptable range for denture base applications. This adherence to standards, such as ISO 1567:2005, specifying a minimum mean FM value of at least 2.00 GPa for PMMA composites, demonstrates the suitability of the hybrid nanofiller-filled PMMA for such applications. The enhancement of the flexural modulus in the hybrid nanofillers HNTs/MWCNTs/PMMA is insightful.

The improvement in mechanical properties is primarily attributed to the hybrid nanofillers' ability to withstand stress and restrict the movement of molecular chains within the PMMA system when subjected to a load. The reinforcing effect of the hybrid nanofillers, which included HNTs and MWCNTs, is evident in their contribution to stress resistance and, consequently, the increase in flexural modulus. They possess the capacity to resist and distribute the applied stress on the composite material, preventing excessive deformation. The effectiveness of stress transfer between the fillers and the PMMA matrix relies on the quality of their interface, often enhanced through the use of coupling agents. These agents facilitate adhesion between the filler and matrix, ensuring improved stress transmission and reinforcing the composite material [16]. The filler's ability to withstand stress without excessive deformation, attributed to its extremely high modulus, is a crucial factor contributing to the enhanced mechanical properties of the nanocomposite material [17].

Furthermore, ensuring good compatibility between the constituents of the nanocomposite material is essential. The interaction between nanoparticles (NPs) and the PMMA matrix plays a pivotal role in boosting the flexural modulus of the filled-PMMA composite by preventing crack propagation. Additionally, proper distribution of NPs throughout the composite prevents nanofiller agglomeration. Well-distributed NPs reduce stress concentration near agglomerated particles, enabling the material to better withstand applied loads. The strength of the interfacial bonding between the NPs and the matrix is critical. Weak bonding can lead to stress concentration and material failure. Achieving good compatibility and interaction between the NPs and the matrix ensures that the interfacial bonding remains robust, even under minimal stresses [18, 19].

The observed increase in the modulus (stiffness) of the hybrid nano-composites aligns with prior research, which attributed the modulus rise to effective stress transfer occurring across the nano-Al_2O_3/PMMA interface. The incorporation of Al_2O_3 NPs into the matrix restricts the movement of the PMMA matrix near each particle, contributing to an overall modulus increase, as reported with a 3.56 GPa increase [20]. Lee et al.'s study [21] also supports the notion that the fillers' ability to resist deformation is directly linked to the filler material's modulus. Essentially, a higher modulus in the filler material enhances its effectiveness in resisting deformation, consistent with other research on PMMA nanocomposites using MWCNTs as fillers [22]. Flexural strength is a crucial parameter for assessing material fracture resistance, indicating how well a material can endure forces mimicking those in clinical situations. In the context of denture base materials, which must withstand various stresses during chewing and biting, flexural strength serves as a key measure. It also provides insight into the material's rigidity or stiffness. Analysis of Figure 3 and Table 2 reveals significant differences in flexural strength among the tested samples in the six groups ($p < 0.05$).

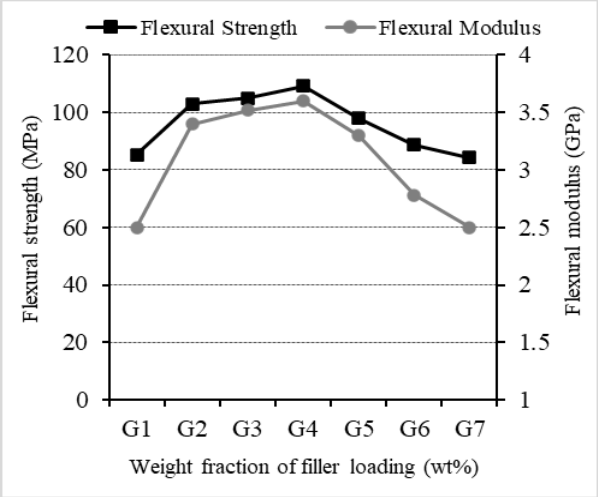

Figure 3. *The impact of various proportions of hybrid nano-fillers comprising HNTs and MWCNTs on the flexural characteristics of PMMA composite.*

Typically, composites without fillers exhibit lower mechanical properties, including flexural strength, when compared to their filled counterparts. The addition of hybrid nano-fillers generally leads to an initial increase in flexural strength, reaching a peak at an optimal filler loading point. However, beyond this point, further increases in filler loading may not significantly enhance the material's strength and could result in a slight decrease in flexural strength due to factors related to the internal structure and properties of the composite. Despite this, the flexural strength values of the nanocomposites surpassed those of the pure PMMA matrix. Specifically, the flexural strength showed an increase from 85.20 MPa to 109.1 MPa, indicating that the nanocomposites were more robust and resistant to bending forces than pure PMMA. This enhancement in PMMA flexural strength is attributed to the effective dispersion of hybrid nanofillers within the PMMA matrix. The even distribution of nanofillers enhances the material's mechanical properties, facilitated by

good interaction and molecular-level bonding, leading to a homogeneous composite structure. This compatibility contributes to the overall improvement in mechanical properties. The incorporation of hybrid nanofillers (HNTs/MWCNTs) into PMMA nanocomposites, along with efforts to enhance their dispersion and compatibility within the matrix, has been found to enhance flexural strength. The application of a silane coupling agent for surface treatment further strengthens the chemical bonding between the nanofillers and the matrix, resulting in a more robust material with improved flexural strength.

Prior research findings corroborate the observation that composites containing nanoparticles tend to demonstrate notably elevated flexural strength. This suggests that introducing nanoparticles has the potential to augment the mechanical properties of composite materials. To attain this enhancement in strength, it is imperative to ensure the uniform and effective dispersion of nanoparticles within the resin matrix. When nanoparticles are well-dispersed, they establish a substantial interfacial area between the filler (e.g., nano-barium titanate, NBT) and the polymer matrix (e.g., PMMA). This expanded interfacial area facilitates the absorption and efficient dissipation of energy by the nanoparticles, resulting in the heightened strength of the modified composites [16]. The strength of composite materials hinges on two primary factors: firstly, the robustness of the bond or adhesion between the filler and the polymer matrix. A sturdy interfacial adhesion is essential for the transfer of loads and the preservation of the composite's integrity. Secondly, smaller filler particles possess a greater surface area relative to their volume. This increased surface area translates to higher surface energy at the interface between the filler and the polymer matrix, thereby enhancing strength properties. Furthermore, the addition of fine-sized nano-fillers to a polymer matrix can bolster the resin's rigidity. These fine particles occupy inter-chain spaces in the polymer matrix, constraining the segmental movements of polymer chains and, consequently, diminishing stress concentration at the filler/matrix interface. The flexural strength of a material encompasses its compressive, tensile, and shear strengths, reflecting its ability to withstand bending or flexing forces, which involve a combination of these stress types [16].

When both tensile and compressive strengths experience augmentation, there is a corresponding increase in the force necessary to fracture the material. This fundamental principle in materials science posits that heightened strength generally results in enhanced resistance to breaking or fracturing under diverse loads. Notably, at elevated filler loadings of multi-walled carbon nanotubes (MWCNTs), a decline in the flexural strength of polymethyl methacrylate (PMMA) nanocomposites was observed. This reduction in flexural strength can be attributed to factors such as the overcrowding of filler particles or alterations in the material's microstructure. Nevertheless, even with this reduction, the flexural strength of the nanocomposites persisted at levels surpassing those of pure PMMA. The international standard ISO 1567 is a point of reference, specifying requirements for denture base materials.

Table 2. *Provides a statistical summary of mean values, standard deviations, as well as minimum and maximum values for flexural properties.*

Samples code	Mean value of flexural strength	Mean value of flexural modulus	*P*- value
G1	85.20±1.82	2.50±0.35	0.355
G2	103.0±2.50	3.40±0.55	0.001
G3	105.0±2.80	3.52±0.58	0.001
G4	109.1±2.89	3.62±0.61	0.001
G5	98.00±2.40	3.3±0.53	0.001
G6	88.70±2.11	2.78±0.58	0.122
G7	84.30±2.15	2.50±0.35	0.152

5.3. Tensile Properties

The impact of hybrid nano-fillers on the tensile characteristics of a PMMA composite material incorporating HNTs/MWCNTs is detailed in Figure 4 and Table 3. The tensile modulus values of the PMMA composite, filled with hybrid nano-fillers (HNTs/MWCNTs), exhibit a notable augmentation compared to the pristine PMMA matrix. This augmentation is statistically significant ($p < 0.05$). Notably, the elastic modulus continues to rise with the increased incorporation of hybrid nano-fillers, particularly in groups G2 to G4. This escalation is attributed to robust bonding at the interface between the hybrid nano-fillers (HNTs/MWCNTs) and the PMMA matrix, restricting the mobility of PMMA molecular chains and resulting in heightened stiffness. The key observation from this investigation is that the introduction of HNTs/MWCNTs nanofillers into the PMMA matrix enhances the stiffness of the resulting nanocomposites. Consequently, the composite material exhibits increased strength and resistance to deformation under tensile forces. The enhancement in tensile modulus primarily stems from the favorable interaction between the hybrid nano-fillers (HNTs/MWCNTs) and the PMMA matrix. This interaction is further improved by the addition of γ-MPS, enhancing the dispersion and interaction of the composite filler within the PMMA matrix. Improved filler dispersion within the PMMA matrix enhances the composite material's ability to uniformly distribute stress, leading to heightened resistance to deformation and an elevated tensile modulus. Essentially, well-dispersed fillers effectively reinforce the polymer matrix [24]. However, when the MWCNT filler content reaches a specific threshold (up to 1 wt.%), the tensile modulus remains unaffected by further filler incorporation, as observed in

groups G5 to G7 (refer to Table 3). This suggests that there might be an optimal filler content beyond which additional fillers do not significantly enhance the tensile modulus. The modulus measurements were conducted before plastic deformation occurred, contributing to the observed behaviour. This implies that the tensile modulus was measured in the elastic region of the material's stress-strain curve, where deformation is reversible. In contrast, plastic deformation results in permanent changes in shape or size [16]. Previous work by Shirkavand and Moslehifard found that the highest increase in the elastic modulus occurred when 1wt% nano-TiO_2 was added to the polymer matrix. However, with higher filler amounts, the increase in modulus became less pronounced, underscoring the variability in filler-polymer matrix interactions based on the type and quantity of fillers used [25].

Tensile strength is a measure of how well a restorative material can withstand lateral forces during function without breaking, essentially assessing its resistance to stretching forces [16]. The study's findings revealed statistically significant variations in tensile strength among the examined samples, with a p-value less than 0.05. The PMMA composites containing hybrid nano-fillers demonstrated superior tensile strength compared to the pure PMMA matrix, indicating an improved ability to resist stretching forces. Among the tested composites, the PMMA nanocomposite filled with HNTs/MWCNTs hybrid nano-fillers exhibited the most favorable tensile properties. It achieved the highest tensile strength at 64.40 MPa, representing a 16.31% increase compared to pure PMMA's tensile strength of 55.37 MPa. Moreover, the nanocomposite displayed a tensile modulus of 1.65 GPa, surpassing the pure PMMA's modulus of 1.58 GPa. This suggests a significant enhancement in both tensile strength and modulus with the addition of HNTs/MWCNTs, as detailed in Table 3.

The enhanced mechanical characteristics, such as increased tensile strength, are attributed to the even dispersion of filler particles within the polymer matrix. When fillers are uniformly distributed, they form a network that can strengthen crack propagation paths and facilitate plastic deformation, thereby absorbing energy in the process. This ultimately culminates in improved mechanical performance. Notably, the robust chemical bonds formed between the fillers and a silane coupling agent play a pivotal role in this enhancement. This chemical linkage heightens the interaction between the fillers and the PMMA matrix, resulting in a more consistent and stable composite material. The heightened interaction between fillers and the matrix contributes to an overall improvement in mechanical properties [16, 23, 26]. The composite's surface energy increases proportionally with the hybrid nanofiller content. This heightened surface energy correlates with improved composite strength. The elevated surface energy fosters better adhesion between the filler particles and the polymer matrix, positively impacting the composite's mechanical properties [16]. Additionally, the shape of HNTs and MWCNTs nanoparticles is acknowledged to influence the mechanical properties of the composite. Distinct particle shapes can alter their interaction with the polymer matrix, influencing the material's overall behaviour. The specific mechanisms involved depend on the shapes of these nanoparticles [27]. Smaller and more uniformly sized particles provide an increased surface area for bonding with the polymer matrix, resulting in stronger interactions and contributing to improved mechanical properties. Conversely, larger particles may not bond as effectively with the matrix, leading to weaker reinforcement [28].

According to Elshereksi et al., composites containing spherical particles may demonstrate increased mechanical strength [16]. Nevertheless, when the concentration of MWCNTs (up to 1wt.% or more) nanoparticles exceeded a certain threshold, their dispersion within the PMMA matrix became inadequate. This suboptimal dispersion had an adverse effect on the strength of the nanocomposite, as evident in G6 and G7 (refer to Figure 4 and Table 3). The accumulation of filler particles resulted in ineffective stress distribution, causing a reduction in PMMA tensile strength due to the weakened strength of these agglomerates. The agglomeration of filler particles intensified with higher loadings of MWCNTs nano-fillers. An increase in filler loading, such as MWCNTs, can weaken the bond between the filler particles and the resin matrix, further promoting filler aggregation and adversely affecting the composite's mechanical properties. Consistent with prior studies, the aggregation of filler particles induces non-uniform stress distribution within the composite, leading to localized stress concentrations and potential crack formation, especially in areas where filler-matrix and filler-filler interactions are compromised or stressed [16, 25, 29]. Although filled samples exhibited higher tensile strain and energy at break values compared to unfilled samples, this improvement is attributed to improved dispersion and favourable filler-matrix interaction. Enhanced compatibility between the composite phases facilitates effective stress transfer to the hybrid nano-fillers from the PMMA matrix, resulting in increased energy absorption and improved mechanical properties [16].

The inclusion of higher quantities of MWCNTs nanofiller (beyond 1 wt.%) in the composite results in diminished tensile strain and energy at break values. This outcome is ascribed to filler agglomeration, wherein the nanofillers aggregate together, causing inadequate uniformity in local stress distribution within the composite. Consequently, deformations occur at specific points, disrupting the even distribution of stress and negatively impacting the material's capacity to absorb energy before breaking. The introduction of ceramic fillers to the composite restricts the mobility of the amorphous phase in the polymer matrix. Consequently, the damping of the composite material is diminished. Damping, in this context, refers to the material's ability to absorb and dissipate energy, particularly in the form of vibrations or mechanical shocks. In this scenario, the augmentation of filler content reduces the proportion of the polymer matrix in the composite, subsequently diminishing the

overall damping capacity of the composite [16, 17].

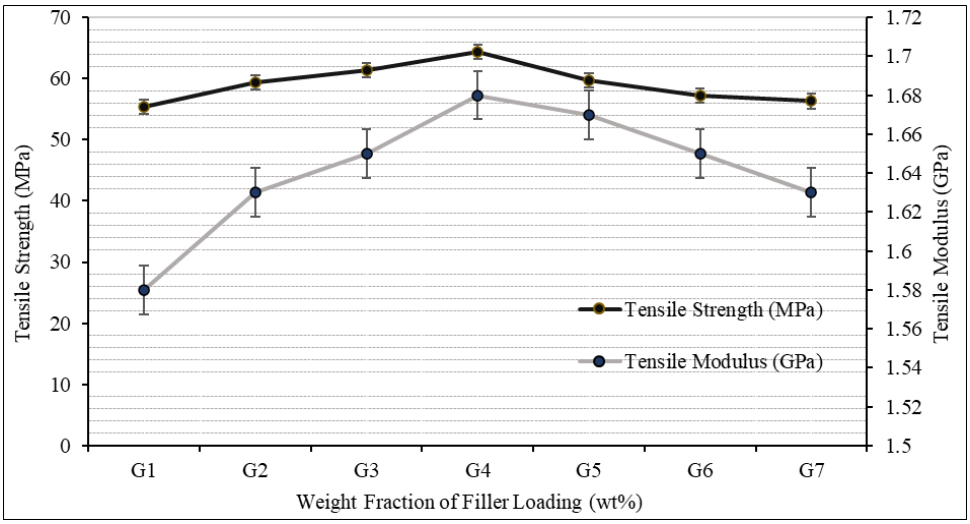

Figure 4. *The impact of various ratios of hybrid nano-fillers (comprising HNTs/MWCNTs) on the tensile properties of a PMMA composite in comparison to the pure PMMA matrix.*

Table 3. *Provides a statistical summary of the mean value, standard deviation, minimum, and maximum values of tensile strength.*

Samples code	Tensile Strength (MPa)	Tensile Modulus (GPa)	Tensile Strain (%)	Energy at Break (N/m^2)
G1	55.37 ±1.72	1.58 ±0.80	4.90 ±0.41	1.15
G2	59.38 ±1.06	1.63 ±0.06	5.41 ±0.27	1.11
G3	61.31 ±1.80	1.65 ±0.50	5.50 ±0.32	1.08
G4	64.40 ±1.20	1.68 ±0.50	5.55 ±0.35	1.06
G5	59.71 ±1.30	1.67 ±0.40	5.51 ±0.40	1.03
G6	57.24 ±1.90	1.65 ±0.30	5.46 ±0.38	0.96
G7	56.30 ±1.80	1.63 ±0.30	5.30 ±0.37	0.83

5.4. Vickers Hardness Surface

Figure 5 and Table 4 present the surface hardness results of PMMA nanocomposites filled with hybrid nano-fillers (HNTs/MWCNTs) in comparison to pure PMMA samples. There were notable statistical disparities ($p < 0.05$) observed in the hardness values of PMMA nanocomposites as opposed to those of pure PMMA. The assessment of composite hardness is pivotal for characterizing and ranking dental restorative materials, as it can serve as an indicator of the material's ability to withstand wear and abrasion, which is particularly crucial in dental applications. The research revealed significant enhancements in surface hardness when incorporating hybrid nano-fillers (HNTs/MWCNTs) in PMMA nanocomposites compared to pure PMMA.

The elevated Vickers Hardness values observed in the PMMA composites imply "that the incorporation of HNTs and MWCNTs effectively boosts the hardness characteristics of the composites. This validates that the inclusion of these specific nano-fillers plays a significant role in enhancing material hardness. Utilizing multiple types of fillers with distinct properties offers several advantages in composite materials, such as synergistic effects, improved dispersion, and heightened mechanical strength. Consequently, the combination of HNTs and MWCNTs resulted in composite material achieving a balance between stiffness and toughness, thereby leading to the observed improvement in Vickers hardness values. The PMMA nanocomposite filled with hybrid nano-fillers exhibited a Vickers hardness value of 18.93 kg/mm², whereas the pure PMMA matrix displayed a lower Vickers hardness value of 16.26 kg/mm². This outcome signifies a robust connection between surface hardness and the filler volume fraction. Put simply, as the concentration of

hybrid nano-fillers (HNTs/MWCNTs) in the PMMA nanocomposite increases, the surface hardness also increases. One reason for this association is the inherently high hardness of the dispersed phase, specifically the HNTs and MWCNTs. As these nano-fillers, characterized by their intrinsic hardness, are well-dispersed within the PMMA matrix, they contribute significantly to the overall hardness of the composite. The resilient bond formed between HNTs/MWCNTs, and the resin matrix indicates the effective integration of these nano-fillers into the composite. This robust bond imparts greater rigidity to the polymerized acrylic resin, reducing its susceptibility to deformation and further enhancing its hardness.

The elevation in Vickers hardness values in PMMA composites can be attributed to two primary factors: the effective "dispersion of hybrid nano-fillers within the PMMA matrix and the subsequent constraint on matrix deformation. When these nano-fillers are evenly distributed throughout the matrix, they act as hindrances to the movement of polymer chains, enhancing the stiffness and brittleness of the PMMA matrix. This, in turn, leads to an increase in the Vickers hardness value. Various factors, including the type, size, and quantity of nano-fillers, as well as the processing parameters employed during material synthesis, can impact the stiffness and brittleness of the composite. This observation aligns with findings supported by Nabhan et al. [30]. The results present a deviation from previous studies suggesting that dental composites often exhibit high hardness values due to their substantial filler contents, emphasizing the established principle in dental material science of using fillers to enhance hardness [16, 17]. As per Elshereksi et al. [16], the Tricyclic antidepressants agent (TCA) serves the purpose of safeguarding the surface of the NBT nanofiller. This protective measure prevents the hindrance of polymerization, ensuring that the nanofiller does not interfere with the polymerization process. Consequently, augmenting the content of treated nanofillers in the polymer matrix has been found to enhance the hardness of the composite [16].

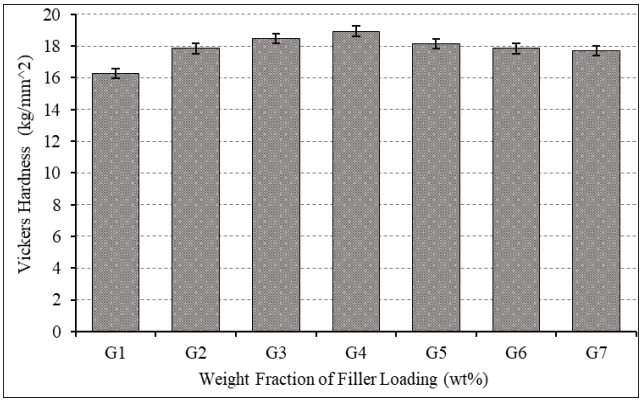

Figure 5. *The impact of varying ratios of hybrid nano-fillers composed of HNTs/MWCNTs on the Vickers surface hardness of a PMMA composite in comparison to the pure PMMA matrix.*

Table 4. *Provides a statistical summary for Vickers hardness (expressed in Kg/mm²), including mean values, standard deviation, minimum, and maximum values.*

Samples code	Mean value of Vickers hardness	*P*- value
G1	16.26±0.4	0.435
G2	17.85±0.2	0.410
G3	18.46±0.7	0.372
G4	18.93±0.3	0.001
G5	18.15±0.6	0.001
G6	17.84±0.1	0.001
G7	17.71±0.4	0.615

While the introduction of hybrid nano-fillers generally boosts hardness, a specific observation highlights a decline in Vickers hardness values for the PMMA nanocomposite when incorporating hybrid nano-fillers. This decline occurs when the MWCNTs nanofiller content surpasses 1 to 2.5wt% in the composite, as illustrated in Figure 5. Beyond this threshold, the inclusion of MWCNTs may have a diminishing impact on hardness. Various potential factors contribute to the observed decrease in Vickers hardness values when the MWCNTs nanofiller content exceeds a certain limit in the PMMA composite. The incorporation of fillers into a polymer matrix can lead to diverse interactions with the matrix. These interactions may involve weak bonding or the generation of stress concentrations at the interface between the filler and the matrix. Such interactions can adversely affect the composite's mechanical properties, particularly its hardness. Weaker bonding has the potential to diminish the material's overall strength and stiffness. The common occurrence of filler particle agglomeration, where particles cluster instead of uniformly dispersing throughout the matrix, presents a challenge in composite materials. Agglomerated fillers create localized stress concentrations within the material, rendering it more susceptible to cracking or failure. Furthermore, agglomeration can compromise the effectiveness of interfacial bonding between the matrix and the filler, further compromising the composite's strength. Additionally, the presence of MWCNTs beyond a saturation limit can weaken the PMMA composite and have adverse effects on its mechanical properties, as indicated in previous research [31].

6. Conclusions

Incorporating treated hybrid nanofillers (HNTs/MWCNTs) into a PMMA composite has demonstrated a notable enhancement in mechanical properties, including flexural characteristics, tensile strength, and hardness. This improvement positions PMMA composites as viable options for diverse industrial applications. The synergistic outcome

arising from the interaction between the two nanotube varieties, coupled with their uniformly dispersed particles and enhanced bonding with the PMMA matrix, plays a pivotal role in augmenting the composite's properties. The amalgamation of treated HNTs and MWCNTs as reinforcing agents presents a promising approach for the development of novel materials with superior performance.

7. Recommendations for Further Research

The dental materials field is in a constant state of evolution, and these suggestions can serve as a foundation for future advancements. Below is a condensed list of recommendations for future research aimed at enhancing PMMA denture base materials:

1. Explore the incorporation of nano-fillers like HNTs and MWCNTs into PMMA denture base composites to enhance properties such as strength, stiffness, and wear resistance.
2. Examine water absorption at varying storage durations, colour stability when exposed to different beverages, hardness under various loads, and other mechanical and physical attributes across different storage periods to assess PMMA resin material performance improvements.
3. Size specimens to mimic actual dental configurations, enabling testing in an environment that replicates real oral conditions.
4. Investigate the long-term stability and biocompatibility of PMMA denture base materials containing nano-fillers such as HNTs and MWCNTs.
5. Analyze the impact of ageing, UV radiation, and storage conditions on the properties of PMMA denture base composites.
6. Enhance grafting percentage in reinforced PMMA nanocomposites by using a vacuum or inert gas environment during the free radical functionalization process.
7. Investigate various processes (e.g., solution blending, melt blending, in situ polymerization) to improve the dispersion of reinforcement particles within the polymer matrix. Study the influence of different temperatures, mixing methods, and particle characteristics on final material properties.
8. Thoroughly assess the effects of nano-fillers on both denture base material properties and their potential impact on human health.
9. Determine the most effective balance of nano-fillers to PMMA to enhance material properties.
10. Explore the use of various types of reinforcement, such as fibres or particles, in combination with nano-fillers (e.g., HNTs or MWCNTs) to further enhance the properties of PMMA denture base material.

Abbreviations

Al_2O_3	Alumina Oxide
ANOVA	One-Way Analysis of Variance
ASTM	American Society for Testing and Materials
BPO	Benzoyl Peroxide
CNFs	Carbon Nanofibers
CNTs	Carbon Nanotubes
EGDMA	Ethylene Glycol Dimethacrylate
FM	Flexural Modulus
FS	Flexural Strength
GFs	Glass Fibers
HA	Hydroxyapatite
HNTs	Halloysite Nanotube
ISO	International Organization for Standardization
MMA	Methyl Methacrylate
MWCNTs	Multi-Walled Carbon Nanotubes
NBT	Nano Barium Titanate
NG	Nanographene
NPs	Nanoparticles
PMMA	Polymethyl Methacrylate
PP	Polypropylene Fibers
SD	Standard Deviation
SiO_2	Silicon Dioxide
TiO_2	Titanium Dioxide
TM	Tensile Modulus
TS	Tensile Strength
VH	Vickers Hardness
ZrO2	Zirconium Dioxide
γ-MPS	3-methacryloxypropyltrimethoxysilane
TCA	Tricyclic Antidepressant Agent

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Author Contributions

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Hazizan Md Akil: Funding acquisition, Project administration, Software, Supervision, Writing – review & editing

Zuratul Ain Abdul Hamid: Methodology, Software, Validation, Writing – review & editing

Ahmed Omran Alhareb: Methodology, Validation, Writing – review & editing

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Data Availability Statement

The data is available from the corresponding author upon reasonable request.

Conflicts of Interest

The authors declare no conflicts of interest.

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Zuratul Ain Abdul Hamid: Research field1-1 Biopolymers, research field1-2 Biodegradables, research field1-3 Biomaterial, research field1-4 Graft Engineering, research field1-5 Regenerative Technology, research field1-6 Polymers, research field1-7 Artificial Organs & Prosthetics, research field1-8 Materials Engineering, research field1-9, Nanoparticles.