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Original Research

Effect of Nanotube Fillers on Impact Strength and Fracture Toughness Forces of PMMA Denture Base Material

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ABSTRACT:

The main goal of this research was to create nanocomposites of poly methyl methacrylate (PMMA) with improved mechanical strength. The study aimed to explore how different amounts

of hybrid nano-fillers (4.25 wt% HNTs and 0.75 wt% MWCNTs) ranging from 1% to 6% affected the impact strength (IS) and fracture toughness (K_{IC}) properties of these materials. Additionally, the research involved examining the surface characteristics of the PMMA nanocomposites using Field Emission Scanning Electron Microscopy, after treating the hybrid nanofillers with a silane coupling agent. The results showed a significant increase in both impact strength and fracture toughness as the loading of hybrid nanofillers increased. Particularly noteworthy was the substantial improvement in IS and K_{IC}values when the nanofillers reached a loading of 5 wt%, measuring 10.25 KJ/m^2 and 2.58 MPa·$m^{1/2}$, respectively. In comparison, the pure PMMA matrix had significantly lower values of 5.27 KJ/m^2 for IS and 1.60 MPa·$m^{1/2}$ for K_{IC}. Consequently, the addition of hybrid nanofillers (HNTs/MWCNTs) into the PMMA matrix greatly enhanced the PMMA denture base material, indicating its potential for improving the longevity of dental composites.

KEYWORDS: PMMA, Nanotubes Fillers, Impact Strength, Fracture Toughness.

INTRODUCTION

In recent years, there has been significant advancement in the field of dental biomaterials research, result in insubstantial improvements in the properties and performance of dental materials and restorative techniques. A key focus of this research has been the development of innovative materials with enhanced biological and mechanical characteristics (Aati et al, 2022 and Alhotan et al, 2021) . Poly methyl methacrylate, commonly known as PMMA, is widely utilized in dentistry due to its numerous advantageous qualities. These advantages encompass its ease of handling, lightweight nature, cost-effectiveness, ability to adhere to teeth, aesthetic appeal, and stability within the oral environment. PMMA an acrylic material, is ease of manipulation and shape, making it an excellent choice for crafting dentures (Alageel, 2022 and Ali Sabri et al, 2021). Moreover, PMMA-based materials are preferred in various biomedical applications owing to their biocompatibility and other beneficial properties (Wang & Zhitomirsky, 2022 and Gautam et al, 2022). In dentistry, PMMA-based materials play a vital role in the fabrication of removable dentures, a common solution for missing teeth. The

biocompatibility, stability, and aesthetic attributes of PMMA make it highly favored in these applications (Ali Sabri et al, 2021). Nonetheless, PMMA denture resin has certain short comings in its mechanical attributes, making it less suitable for specific dental purposes (Raszewski et al, 2021). For instance, its limited flexural strength can lead to material breakage or fissures over time, and its reduced impact resistance makes it more susceptible to damage from accidental drops or impacts (Bacali et al, 2020). Additionally, PMMA denture resin can be prone to surface roughness and wear, potentially affecting the long-term fit and comfort of the denture (Baba et al, 2012).

Moreover, while maxillary dentures offer functional and cosmetic solutions for individuals with missing teeth, they can be susceptible to fracture or damage specific situations such as the potential for midline fractures (Jacob & Prathap, 2021 and Zidan, 2020). To minimize the risk of fractures, denture materials must excel in terms of their mechanical properties, requiring the ability to withstand the forces associated with normal oral function without breaking or developing cracks (Chander & Venkatraman, 2022). The dental materials field has witnessed technological advancements, resulting in the development of new denture materials with enhanced mechanical properties (Chladek et al, 2022 and Zidan et al, 2021). Recently, advances in nanotechnology have opened up new possibilities for improving dental materials, including the development of polymeric nanocomposites (Koo, 2019). These composites are created by incorporating nanoscale fillers into a polymer structure, which can lead to enhanced mechanical characteristics and other benefits (Omanović-Mikličanin, 2020 and Ghazanlou et al, 2021). In specific instances, these materials have shown promising results, including improved wear resistance, and bonding strength Ramburrun, 2021). Nanoscale reinforcing agents like nano-fillers have demonstrated the ability to introduce new physical, mechanical, and biomedical properties when incorporated into polymeric nanocomposites (Aga et al, 2021).

In this particular research, scientists investigated the improvement of the mechanical properties of PMMA base material by incorporating hybrid nano-fillers with different ratios. These nano-fillers include materials like halloysite nanotubes (HNTs) and multi-walled carbon nanotubes (MWCNTs). The primary goal was to assess how these nanofillers affected PMMA

mechanical characteristics, specifically its ability to withstand sudden impacts without cracking or breaking (impact strength) and its resistance to crack propagation (fracture toughness). By adding nanofillers to PMMA, the researchers aimed to enhance its mechanical properties and potentially extend its lifespan and durability.

MATERIALS AND METHODS

Materials

In the present investigation, the materials utilized encompassed PMMA with high molecular weight (i.e. 996.000, GPC –Aldrich U.S.A), plus 0.5% of benzoyl peroxide (BPO) (Merck chemical, Germany). While the liquid components consist of a mixture between 90% of methyl methacrylate (MMA) (Flucka,UK) and stabilized by 0.005% hydroquinone plus 10% ethylene glycol dimethacryate (EGDMA) (Aldrich U.S.A.). The primary focus of this study revolves around the utilization of hybrid nanofillers as strengthening materials within PMMA composites. These nanofillers comprise Halloysite nanotubes (HNTs) (Aldrich U.S.A.), and multi-walled carbon nanotubes (MWCNTs) (Aldrich U.S.A.). To enhance the interaction between PMMA and these hybrid nanofillers, acouplin gagent, namely Silane (3-trime thoxysilyl propyl methacrylate or γ-MPS), was utilized in their treatment and supplied by Sigma-Aldrich, Germany.

Salinization Process of Nano-Fillers Surface

The salinization process involvedtaking10 grams of filler powder, which could be either HNTs or MWCNTs, and mixing it with 200 milliliters of toluene.

Initially, the filler powder was dispersed in the toluene. In the subsequent step, a silane coupling agent was added to the filler powder at a concentration of 10 wt% while maintaining room temperature. The mixture was then continuously stirred at a speed of 150 rotations per minute (rpm) for a duration of 15 hours. Following this, the solution was filtered to separate and collect the modified filler powder from the liquid solution. To wash the collected modified filler powder, a Soxhlet apparatus was used, employing 300 Ml of fresh toluene over a 24 hour period. Subsequently, the modified filler powder was dried in a vacuum oven at 110°C for 3 hours to remove any remaining solvent or impurities from the modified filler material.

Preparation of the PMMA Denature Base Composite

The denture base materials were prepared in a dental laboratory following the ISO 1567:2001 standard method. The powder

components, consisting of PMMA and 0.5 wt.% BPO were used. The liquid medium was prepared by combining 90 % MMA monomer and 10 % EGMMA. To prevent rapid polymerization, a small amount of hydroquinone (0.025%) was added to the liquid medium. Additionally, hybrid nanofillers composed of HNTs/MWCNTs were included as reinforcement particles at concentration of 1 to 6 wt.% (G2 to G7 as detailed in Table , and Figure 1).

Table.1. Specimen grouping and coding.

Group/ Sub group Code	Powder			Liquid	
	PMMA	BPO	Mixture of 4.25wt.% HNTs and 0.75wt.%MWCNTsas Filler Loading	MMA	GDMA
G1	99.50	0.50	-	90%	10%
G2	98.50	0.50	1 wt.%	90%	10%
G3	97.50	0.50	2 wt.%	90%	10%
G4	96.50	0.50	3 wt.%	90%	10%
G5	95.50	0.50	4 wt.%	90%	10%
G6	94.50	0.50	5 wt.%	90%	10%
G7	93.50	0.50	6 wt.%	90%	10%

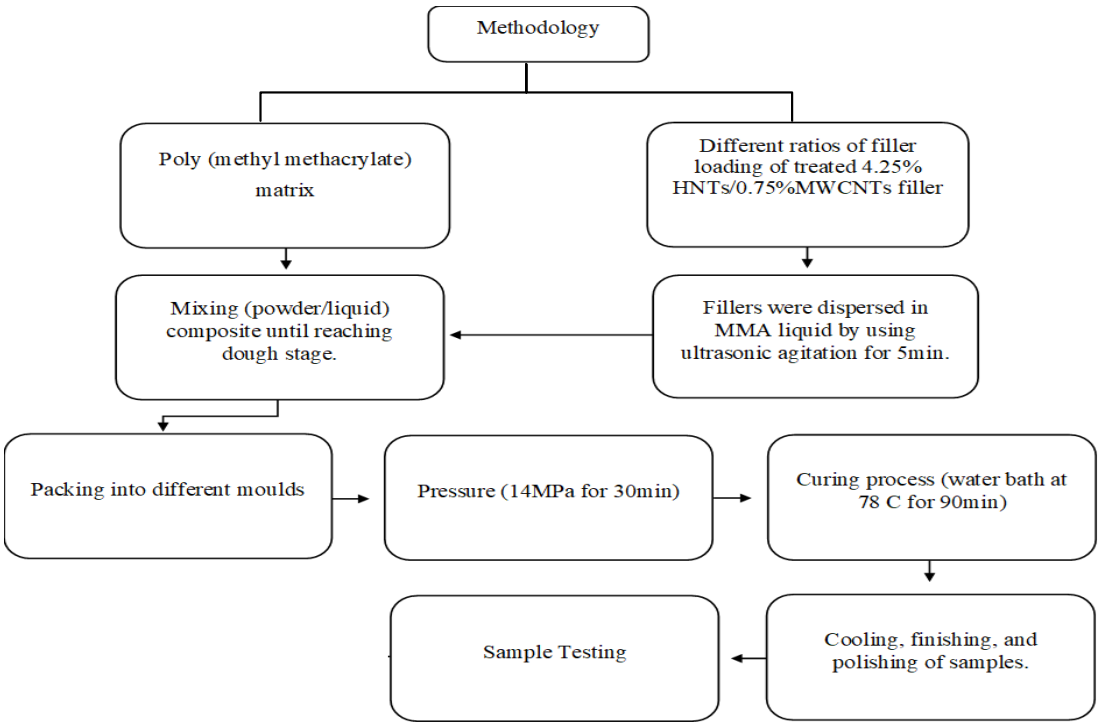

Figure.1. Flowchart of preparation of PMMA composite reinforced with ceramic fillers as filler loading.

Methods

The impact strength test is used to determine a material's ability to withstand impacts without breaking or developing cracks ISO 179-1:2023 is an international standard that outlines a procedure for evaluating the IS of plastic materials using an Charpy V-notch test (CVN). In this test the sample dimensions of length of 80 mm, width (b) of 10mm, width beneath the notch (bn) (V-notch of 0.25mm radius and a notch angle (rn) of 45°) of 9.75 mm, and thickness (d) of 4 mm. The test is conducted using a notched bar IS and span support of 62 mm. The mean impact strength of the samples is calculated using Equation 1:

$$\text{IS} = \frac{E}{bn\ d} 10^3 \qquad (1)$$

The evaluation of the material's K_{IC} was conducted by performing the Single Edge Span Notch Bending test (SEN-B) according to the ISO 13586:2018 standard. In this test the specimen's dimensions of 80 mm length, a 4 mm notch length, a 64 mm span length, a 4 mm thickness, and a20 mm width. To create a notch in the sample, a razor blade was used, and the notch featured a loading nose, a support span of 50 mm, and two supports with diameters of 20 mm and 10 mm. The testing was conducted using an Instron 3366 machine, with across head speed of 1.00 mm/min and applying a 10 KN load to the samples. The fracture toughness values were determined using Equations 2:

$$KIC = \frac{P\frac{S}{2}\,a^{\frac{1}{2}}}{t\frac{W^2}{3}}\ 1.93 - 3.07\left(\frac{a}{w}\right) + 14.53\left(\frac{a}{w}\right)^2 - 25.11\left(\frac{a}{w}\right)^3 + 25.8\left(\frac{a}{w}\right)^4 \qquad (2)$$

P= load at peak (N), S = span length (mm), a = notch length (mm), t = sample thickness (mm), w = sample width (mm)

Morphology of Filler Particles

The Zeiss Supra Model 35VP, a scanning electron microscope (SEM), was employed to inspect the fractured specimen surfaces. Morphology analysis involves the examination of particle shapes, dimensions, and structures, while microanalysis entails the investigation of the chemical composition of a material. To prepare samples for Field Emission Scanning Electron Microscopy (FESEM), a Bio-Red E5000 Sputter Coater from the USA was utilized to apply a thin metal layer. This was done to enhance image clarity and prevent electrostatic charging. By adjusting the electron beam voltage within the typical range of 5 kV to 50 kV during micrograph acquisition, various aspects of particle surfaces could be

explored. Lower voltages were employed for surface feature examination, whereas higher voltages facilitated the study of subsurface characteristics.

Statistical Analysis

In this research, we employed the statistical techniques of One-way Analysis of Variance (ANOVA) and Tukey's post hoc analysis. ANOVA was utilized to examine the data related to impact strength and fracture toughness in order to detect any significant differences among the various groups ($P < 0.05$).

RESULTS AND DISCUSSION

Figure 2 illustrates the impact strength (IS) outcomes of PMMA composite material that has been strengthened with a combination of hybrid nanofillers (HNTs/MWCNTs) at varying concentrations, ranging from 1 to 6 wt% as detailed in Table 1. These results are contrasted with the IS of the PMMA matrix. There indicate that the IS of the PMMA composite, which has been reinforced with hybrid nanofillers, surpasses that of the PMMA matrix. The highest recorded IS was achieved with the PMMA composite containing 5 wt% of the hybrid nano-fillers, registering at 89.18% (9.97 kJ/m^2), which is a substantial increase compared to the PMMA matrix, which only reached 5.27 kJ/m^2. Additionally, it's worth noting that these IS values were determined to be statistically significant ($p < 0.05$). The rise in IS can be ascribed to several factors. One contributing factor is the establishment of cross-links, which can enhance the interfacial shear strength between the fillers and the PMMA matrix (He et al, 2022). Additionally, another reason for the increased IS value is the elevated concentration of a cross-linking agent, such as EGDMA, which can lead to higher viscosity and decreased mixture flow ability (Fang et al, 2023). Alhotan et.al (2021), investigated the impact of E-glass fibers, ZrO_2, and TiO_2 nanoparticles on the IS of PMMA denture bases. Their findings revealed that incorporating E-glass fibers at concentrations of 3, 5, and 7 wt.% in PMMA composites resulted in significantly improved IS compared to the control group (Alhotan et al, 2021).The increase in IS may be attributed to factors such as the uniformity of concerning the compound, the effective infiltration of fillers in to the monomer, strong filler-resin interaction, an optimal filler to resin ratio, or a combination of organic resin and inorganic filler (Alhotan et al, 2021). However, introducing a high filler loading (HNTs/MWCNTs) of up to 6 wt% into the PMMA matrix led to a decrease in IS values compared to PMMA

composites with lower filler loading, up to 5wt%. This decrease in the IS of PMMA can be attributed to the limited compatibility between the increasing filler particles and the PMMA matrix. As the filler content rises, the composite's ability to distribute applied impact energy diminishes (Alhotan et al, 2021). As the ratio of fillers increases within a composite material, it can resultin a reduction in the interfacial adhesion between the reinforcement fillers and the PMMA matrix. Interfacial adhesion is a critical factor in determining the overall performance of composites. In addition to the energy-absorbing capability of fillers, establishing a strong interfacial bond is crucial for enhancing the IS of composites.

A robust bond between the fillers and the matrix generates micro-spaces or micro-cracks at the interface. These micro-spaces serve as barriers to crack propagation, making it more challenging for cracks to spread throughout the material. This result agrees with a study by Alhotanet.al (2021) which investigated the impact of ZrO2 and TiO2 nanoparticles on the IS of PMMA denture bases. The incorporation of ZrO_2 or TiO_2 at concentrations of 7 wt.% in PMMA composites did not show a statistically significant difference in IS when compared to PMMA matrix (Alhotan et al, 2021). Similarly, in a study conducted by Zhuet.al (2022), discovered that the IS increased when using 3 and 5 wt% nano-ZrO_2 fillers but exhibited a slight decrease with 7 wt% nano-ZrO_2 fillers. This decrease was attributed to the agglomeration of filler particles within the PMMA matrix (Zhu et al, 2022).

Figure 3 provides information about the values and variations of fracture toughness (K_{IC}) of unfilled and filled PMMA with 1 - 6 wt% hybrid nanofillers loading (HNTs/MWCNTs). According to the results of the statistic analysis, it was evident that there was significant difference (p<0.05) in K_{IC} between the groups that incorporated hybrid nanofillers and the unfilled PMMA, which had a K_{IC} value of 1.60 MPa.$m^{1/2}$. The K_{IC} of the specimens filled with 5 wt.% hybridnano-particle experienced a substantial increase (reaching 2.65 MPa.$m^{1/2}$), representing are makeable 65.63% improvement compared to the K_{IC} of the unfilled PMMA. Consequently, it attained the highest average K_{IC} among all the groups with reinforcements. The mean K_{IC} was statistically higher (p < 0.05) in PMMA samples filled with 3 and 4wt.% hybrid nanofillers when compared to the unfilled PMMA group. The mean K_{IC} in the remaining groups with reinforcement (1 and 2 wt% hybrid nanofillers) exhibited a slightly higher level in comparison to the

group without fillers, these differences were observed. Nevertheless, did not attain statistical significance ($p > 0.05$). Regarding the specimens with hybrid reinforced nanoparticles the K_{IC} of PMMA filled with 6 wt.% hybrid nano- fillers showed improvement but without any statistically significant difference.In summary, the K_{IC} demonstrated a significant increase at nanofiller loadings of 4 and 5 wt.% (reaching 2.47 and 2.65 $MPa.m^{1/2}$, respectively), followed by a gradual decrease when the nanofiller loading concentration reached 6 wt.% (where it was measured at 1.98 $MPa.m^{1/2}$).The elevation in K_{IC} values of PMMA was attributed to various factors, which encompassed the effective dispersion of nanoparticles within the PMMA composite, strong adhesion between the fillers and the PMMA matrix, interactions between the matrix and fillers, as well as the utilization of high-strength fillers [24]. These findings were corroborated by earlier research conducted by Alhotan, et al (2021). In their study, the rise in K_{IC}within the PMMA composite might be ascribed to several elements, such as the even distribution of the compound, the successful infiltration of fillers into the monomer, a robust connection between the filler and the resin, an optimal quantity of filler within the resin, or the synergy between the organic resin and the inorganic filler (Alhotan, et al, 2021). Also, the study of Alhotan, A.,etal (2021), confirmed the observation that lower filler loading can lead to an enhancement in K_{IC} within PMMA composites. According to their research, the incorporation of ZrO_2 or TiO_2 at concentrations of 3 and 5 wt.% into PMMA composites resulted in notably elevated K_{IC} values. Specifically, the K_{IC} increased by 23.24% when 5 wt% ZrO_2 was added and by 19.72% when 3wt% TiO_2 was added, in comparison to the control group. This increase in K_{IC} may be attributed to the uniform distribution of the filler within the composite material (Alhotan, et al, 2021). Similar finding was reported by Wang, et al (2020), which emphasized that a strong bond between filler particles and the resin matrix leads to superior mechanical properties in composites. On the other hand, the decline in K_{IC} value when 6 wt% of hybrid nanofillers were added, especially at higher filler loading concentrations, can be attributed to several factors (Wang, et al, 2020). These factors include the potential risk of filler particles clustering together, the uneven dispersion of the additive within the PMMA matrix, and the formation of anointer facial layer between the PMMA matrix and the additives (Abushowmi et al, 2020). The inadequate dispersion of filler particles within the composite and the

occurrence of particle clustering can result the creation of voids, which in turn can establish areas of stress concentration within the polymer matrix. This disrupts the even distribution of stress and diminishes the strength of the bond between the matrix and the anoparticles. These observations are in line with study by Zidan et al (2019). Furthermore, the reduction in PMMA K_{IC}occurs because the PMMA composite eventually reaches a saturation point, which is the point at which it cannot accommodate additional filler particles (Zidan et al, 2019). This saturation leads to a disruption in the continuity of the PMMA matrix. Additionally, as the content of filler loading increases, the interaction between the filler particles and the PMMA matrix becomes less robust (Nabhan et al, 2023).

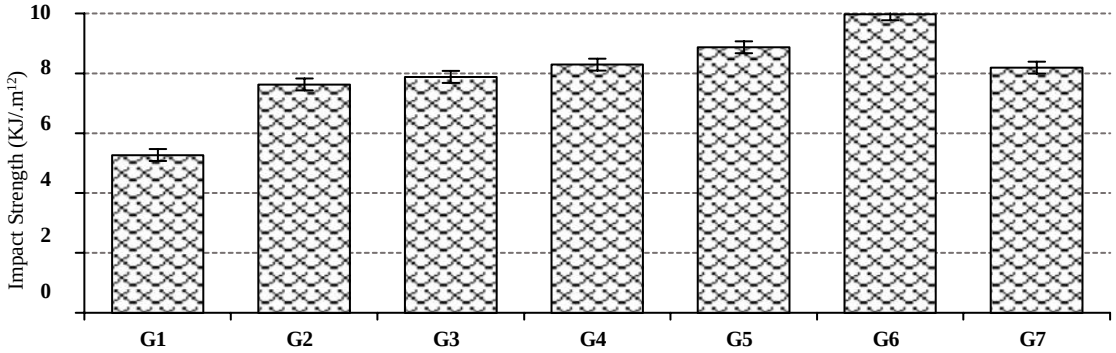

Figure. 2 Weigh Fraction of Filler

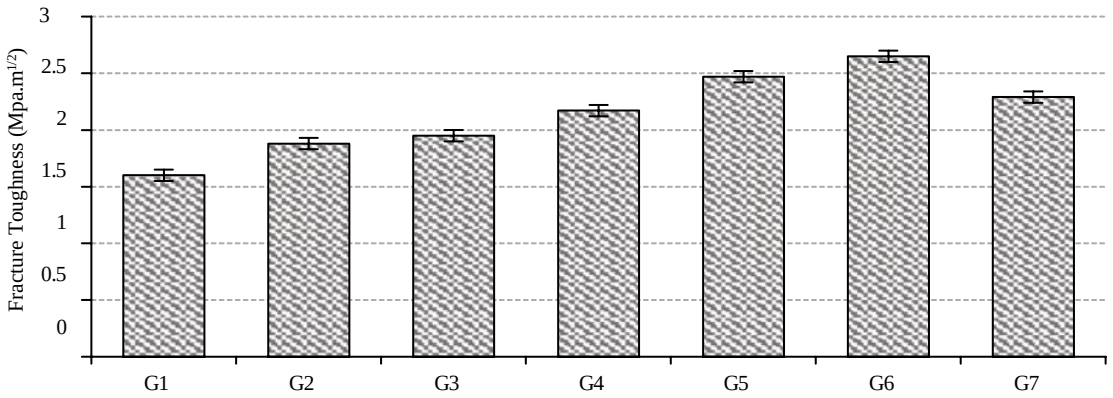

Figure. 3 Weigh Fraction of Filler Loading

Morphology of PMMA Specimens Fracture

The microstructure morphology of both unfilled and filled PMMA, containing hybrid nan-fillers (HNTs/MWCNTs) at concentrations ranging from 1 to 6wt.%), was examined using field emission scanning electron microscopy (FESEM) (see Figures 4 to 7. The SEM micrograph of unfilled PMMA sample revealed a surface that was notably smooth with small pores. This surface

morphology suggested a brittle mode of fracture (see Fig. 4a). This observation agrees with the finding by Sadati et al (2022), who reported that the fracture morphology unfilled PMMA matrix is characterized by a smooth surface, indicative of uncontrolled crack propagation throughout the PMMA matrix (Sadati et al, 2022). This brittle behavior is defined by smooth areas and a lack of resistance to crack propagation (de Menezes et al, 2022).

The fracture surface of PMMA composite containing 1and 2 wt% of nano- fillers displayed a rough texture and exhibited small cracks. These characteristics were a con sequence of the nanofiller particles resisting the fracture energy, (see Figure 4b and Figure 5a). The distribution of the filler particles was nearly uniform within the PMMA composite, and the interaction between these filler particles and the PMMA matrix was relatively robust. A rough fracture surface typically indicates a longer crack propagation path. This phenomenon is attributed to various factors, including the material properties of the fractured object, the applied load or stress conditions, and the specific circum stances under which the fracture occurred (Sharafi et al, 2021). With an increment in the loading of hybrid nanofillers up to 3, 4, and 5 wt% in PMMA composites, there is a notice able enhancement in the bonding between the filler particles and the PMMA matrix. The PMMA specimens filled with 3, 4, and 5wt.% hybrid nano-fillers, a strong bond between the This observation suggests that the nanofillers (HNTs/MWCNTs) were more efficiently embedded within the PMMA matrix which lead to increase in the resistance of the fracture surface. Moreover, the inclusion of γ-MPS in the filler particles contributed to a more robust bonding and even distribution of these particles within the PMMA (poly

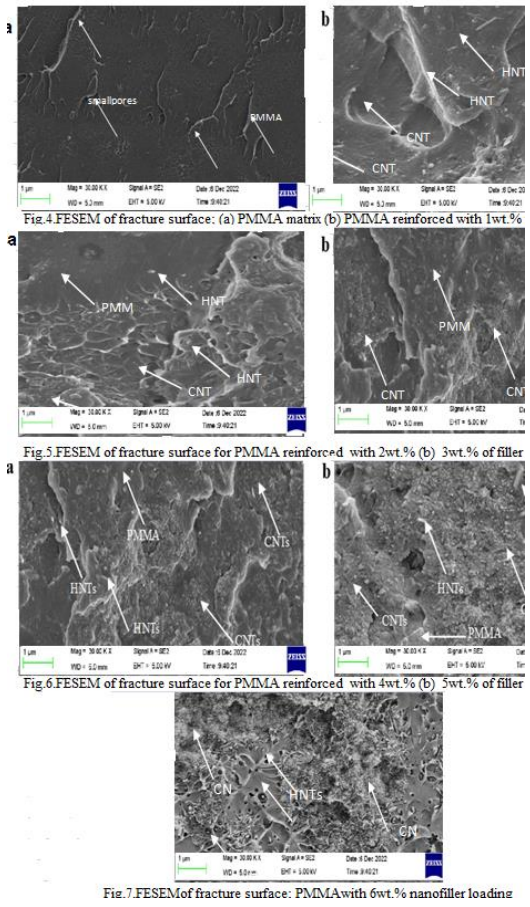

Figure. 4 Methyl Methacrylate Matrix. [31]

Nevertheless, the fracture surface of the specimen in the PMMA filled with 6 wt% hybrid nanofillers (see Figure 7) displayed small gaps and slight clumping, suggesting a sub optimal interaction between the nanoparticles and the PMMA matrix.

Furthermore, these flaws can contribute to early crack formation and expansion, ultimately diminishing the material's fracture resistance. This finding agrees with Lim et.al (2021), who observed that incorporating a substantial quantity of talc filler into a PMMA polymer composite can result in the formation of sizable clusters of filler particles, adversely affecting the composite material's flexibility and strength (Lim et al, 2021).

CONCLUSION

The incorporation of Hybrid nanofillers mixture into PMMA composite material has proven to be effective in boosting its mechanical properties. This improvement includes increased IS and K_{IC}. The synergy between the two nanotube types, their well-dispersed particles, and their improved bonding with the PMMA matrix are critical factors contributing to the enhanced properties of the composite. Therefore, combining treated HNTs/MWCNTs nanotubes as reinforcing agents shows great promise for enhancing the properties of PMMA composites and developing innovative materials with superior performance.

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الملخص

الهدف الرئيسي لهذا البحث هو إنشاء مركبات نانوية من بولي ميثيل ميثا اكريلات (PMMA) بقوة ميكانيكية محسنة. تهدف الدراسة إلى استكشاف كيفية تأثير كميات مختلفة من الحشوات النانوية الهجينة (4.25 wt% HNTs و0.75wt% MWCNTs) التي تتراوح من 1٪ إلى 6٪ على قوة التأثير (IS) وصلابة الكسر (K_{IC}) لهذه المواد. بالإضافة إلى ذلك، تضمنت الدراسة فحص الخصائص السطحية للمركبات النانوية من بولي ميثيل ميثاكريلات (PMMA) باستخدام مجهر الإلكترون الماسح، بعد معالجة الحشوات النانوية الهجينة بواسطة عامل سيلاني المعالج. أظهرت النتائج زيادة كبيرة في كل من قوة التأثير وصلابة الكسر مع زيادة تحميل الحشوات النانوية الهجينة. كان التحسن الكبير في قيم IS و K_{IC} عندما وصلت الحشوات النانوية إلى إضافة 5wt%، حيث بلغت KJ/m^2 10.25 و$2.58\ MPa{\cdot}m^{1/2}$ ، على التوالي. في المقارنة، كانت بولي ميثيل ميثاكريلات النقية قيم أقل بكثير، حيث بلغت $5.27\ KJ/m^2$ لقوة التأثير(IS) و $1.60MPa{\cdot}m^{1/2}$ لصلابة الكسر(K_{IC}). وبالتالي، فإن إضافة الحشوات النانوية الهجينة (HNTs/MWCNTs) إلى مصفوفة PMMAعززت بشكل كبير مادة قاعدة الطقم الصناعية (PMMA)، مما يشير إلى إمكانية تحسين طول عمر خليط التركيبات السنية.

الكلمات المفتاحية: بولي ميثيل ميثا اكريلات، الحشوات النانوية الهجينة، قوة التأثير، صلابة الكسر.