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Synthesis and Characterization of PMMA/Nanoclay Composite as an Excellent Dental Material

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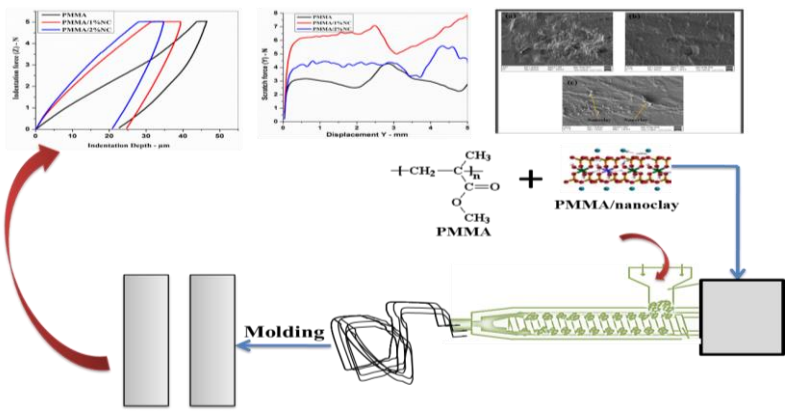
The present study reports the synthesis of poly (methyl methacrylate) (PMMA)/nanoclay-based nanocomposites via melt compounding using a twin extruder process. PMMA was chosen as a matrix material and various weight fractions of nanoclay (NC) nanoparticles (1 wt.% and 2 wt.%) were used as reinforcement. The processed nanocomposites were comprehensively characterized through simple indentation analysis, scratch analysis, and field emission scanning electron microscopy (FESEM) analysis. The variation in the mechanical properties with the addition of various wt. % of nanoclay in the composites was examined. The estimation of the mechanical aspects of the nanocomposites showed that the utilization of Nanoclay as a reinforcing factor increased specific strength of polymer. The morphological analysis depicted the formation of composites with uniform dispersion of nanoclay within the polymer matrix. The successful compatibilization in Nanoclay and the polymer pattern developed the engineering attributes. Finally, this event may combine the design aimed at the formation of a different compatibilized Nanoclay founded blend concerning dental treatments.

Graphical abstract



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1. Introduction

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In the current situation, polymer nanocomposites are believed to be a primary development of ancient materials known in the polymer domain. They have received significant attention from researchers across the globe. Potential reinforcements involve the use of calcium carbonate, silica, alumina, barium sulfate, talc, TiO_2, magnesium hydroxide, etc. to enhance to structural features and characteristics of the polymer matrix. Still, the significant developments in this field are unexplored and their consequences are unacceptable, especially when linked with lightweight polymers. To overcome the limitations, a novel approach of introducing nanomaterials in the polymer matrix to form nanocomposites is employed. The nano minerals ranging in the size domain of few to 100 nm, reinforce the Thermoplastics, thereby enhancing its characteristics [1, 2]. Amongst various nanoparticles, clay crystals, carbon nanotubes, and silica nanoparticles stand superfluous and are commonly utilized in improving the physical, mechanical, and thermal characteristics of polymers. PMMA has been regularly employed as an acrylic resin for denture base design [3]. This resin possesses sufficient mechanical, physical, and aesthetic characteristics [4]. Additionally, it has advantages such as low cost, easy invention, low power, complete aesthetics, good visible features, biocompatibility, color matching ability, and content of finishing and polishing [5]. However, PMMA has amazing applications but suffers from limitations such as low outer hardness and lower mechanical hardness upon failing, collision, and converting [6]. These deficiencies must be treated to increase the appearance of PMMA materials as orthodontic devices and detachable inadequate dentures [7-9]. PMMA is an optically clear amorphous thermoplastic exhibiting an extensive brightness in the visible region. It has largely been used as a replacement for inorganic glass owing to its high impression energy and flexibility. Still, the applications are limited due to the reduced thermal stability. The incorporation of nanoparticles in the PPMA matrix results in the formation of nanocomposites with improved characteristics and high stability [10]. Several reports are available on the preparation and design of PMMA nanocomposites [11-15]. Laachachi et al. [16] have illustrated that the PMMA nanocomposites comprising variable stuffing of boehmite ((AlO(OH)) and alumina (Al_2O_3) fillers prepared by the melt blending process at around 17–35 °C PMMA/alumina nanocomposites exhibit high thermal resistance as compared to pure PMMA. Singh et al. [17] reported the preparation of poly (methyl methacrylate) nanocomposites with superior impact strength by incorporation of alumina nanoparticles in the polymer matrix. The presence of alumina nanoparticles enhanced the effective strength of the nanocomposite by 84% as compared to the natural polymer. Kiersnowski et al. [18] synthesized PMMA nanocomposites using MMT improved with (3-acrylamidepropyl) trimethylammonium chloride via mixture and in situ polymerization system. In the present study, the preparation of PMMA-nanoclay, as a dental material was explored. The use of nanoclay in variable concentration act as reinforcement to the composites improving their strength and performance. The nanocomposites PMMA/1wt%nanoclay and PMMA/ 2wt%nanoclay were prepared using the melt blending processing method. Both nanocomposites have been investigated for physical and mechanical properties and then compared with the pristine PMMA specimen. The PMMA/2wt%nanoclay model revealed the best mechanical and physical characteristics, which can be a suitable material for dental schemes.

2. Results and Discussion

2.1 Simple Indentation Analysis

The mechanical characteristics of pristine PMMA, PMMA/1 wt.% nanoclay and PMMA/2 wt.% nanoclay nanocomposites were analyzed under a micro-indentation analysis and micro-scratch analysis. Fig. 1 displays the characteristic loading/unloading curves for the indentation depth vs Indentation force applied. The analysis indicates that the specimen could sustain the maximum indentation load of 5 N. As usual, the status of the loading/unloading curvatures was comparable for all the samples. Yet, the extreme indentation depths under load and the following unloading were modified in the PMMA and its nanocomposites. The curvatures related to the nanocomposites were extremely more significant than those of PMMA, promoting their greater hardness and modulus benefits. While flexible gain during unloading was detected in various specimens. The highest dispersion penetration on an extreme indentation load and the identical modules are obtainable and shown in Table 1. The young hardness situations ignoring viscoelastic improvements are also presented in the table following Equation 1.

$$HM = \frac{P}{26.43h_{max}^2} \quad \text{---------- (1)}$$

where, HM is Martens hardness which depicts the unloading and loading of indentation curve, P stands for principal of maximum indentation force, and h stands for indentation depth, respectively. The depth of penetration indicated that the greatest approach was observed in PMMA, supported via PMMA/1wt%NC and PMMA/2wt%NC. Accordingly, the highest indentation modulus and Viker hardness were observed in PMMA/2wt%NC, supported via PMMA/1wt%NC and PMMA. The significant precision concerning particular hardness conditions of the PMMA and its nanocomposites were compared with the presence of a small number of NC nanoparticles as reinforcement. Saying that the decisions concerning particular elastic modulus and hardness of the absolute PMMA specimen reached in the present study stand show a standard deviation of ±10 % [19].

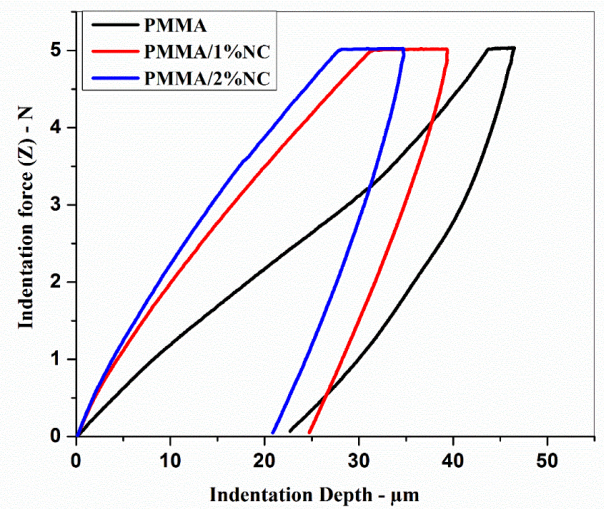

Fig. 1. Loading/unloading curve of micro-indentation of PMMA; PMMA/1%NC and PMMA/2%NC.

Table 1. Assessment of indentation modulus and Viker hardness (HV). Creep indentation time for each sample was 30 s.

Test samples	Indentation Analysis (*Pmax* = 5 N)		
Test samples	***Maximum penetration depth (μm) (×10³)***	***Indentation Module (GPa)***	***Viker Hardness (kg/mm²)***
PMMA	46.49	2.1	13.0
PMMA/1wt%NC	39.37	3.1	21.8
PMMA/2wt%NC	34.76	3.9	32.3

2.2 Micro-scratch Analysis

To determine the micro scratch hardness of the fabricated samples, a 9.8 N indentation weight were performed on clean specimens to prepare a first indent (regarding 50.4 μm depth). Figure 2 displays a significant quality of scratch forces with the scratch distance (in mm). The specimen was scratched vigorously for 6 mm. It can be realized that approximately developed scratch force is shown via the PMMA/1wt%NC specimen. The quick peek of scratch force in the PMMA/1wt%NC specimen displays the existence of compact nanoclay and his force to restrain the progress of the indenter during scraping of the specimen. On another hand, PMMA and PMMA/2wt%NC specimens revealed approximately similar scratch force, and at rates slightly below the scratch forces displayed by PMMA/2wt%NC specimens. These features may be connected to the strength of 2wt%NC to fracture down the predicted difficulties in the specimen so lessening the scratch force inside the instance of the PMMA/2wt%NC specimen. The exterior of the structure of scratched has been shown in Fig. 3 (a) and (b). Fig. 3 (a) and (b) shows the surface topology of the scratched surfaces. As can be seen PMMA/1wt%NC and PMMA/2wt%NC samples exhibited irregular surfaces which Figure 3. FESEM image of surface topography of scratch surfaces (a) PMMA/1wt%NC, and (b) PMMA/2wt%NC samples.

2.3 FESEM Analysis

The crack morphologies of the PMMA and its nanocomposites are shown in Fig. 4. The FESEM image shows the broken surface and exposed microvoids in whole the specimens. The non-uniform and indented exterior in the PMMA specimen was observed in the FESEM images (Fig. 4(a)). Meantime, a smooth-layered morphology amongst a terrace-like break exterior was observed for the PMMA/ 1wt%nanoclay nanocomposite (Fig. 4(b)). This characteristic break indicates the shear bending of the features. The high-magnification FESEM image of the cracked exterior of the PMMA/ 2wt%nanoclay nanocomposite (1.50kX) displayed a good amalgamation and sufficient adhesion of nanoclay to the PMMA pattern (Fig. 4(c)).

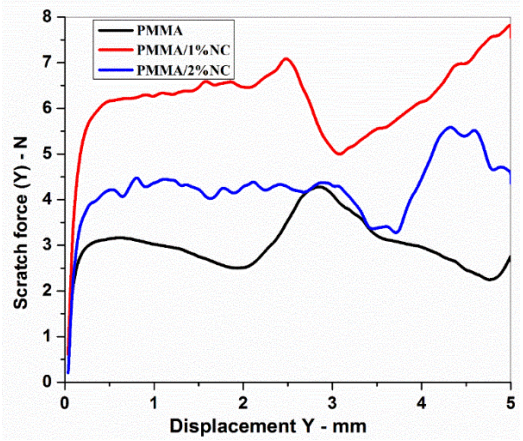

Fig. 2. Difference of scratch force along the scratch direction.

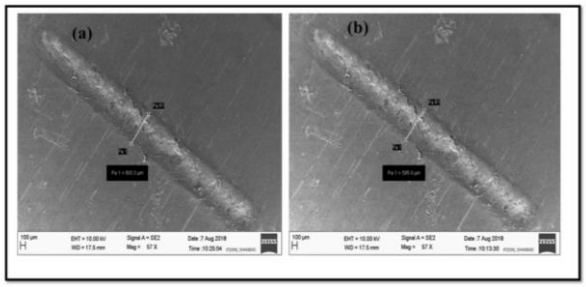

Fig. 3. FESEM image of surface topography of scratch surfaces (a) PMMA/1%NC and (b) PMMA/2%NC.

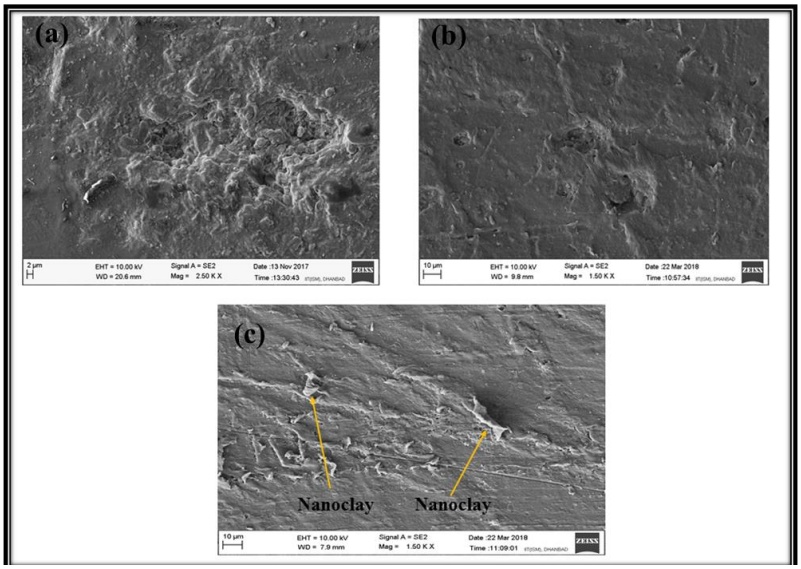

Fig. 4. FESEM images showing crack exteriors of (a) PMMA; (b) PMMA/1wt%NC; (c) PMMA/2wt%NC.

3. Material and Methods

3.1 Materials

PMMA/NC nanocomposites were prepared by the incorporation of nanoclay powders in a polymer (PMMA) matrix. PMMA (grade LG 2S) was employed while some model material and nanoclay acted as reinforcement. Materialistic nanoclay nanopowder with a purity of nearly 99.9 % was procured from Alfa Aesar, USA.

3.2 Manufacture of PMMA/nanoclay nanocomposites

A series of blend nanocomposites of PMMA with variable loading of nanoclay (1 wt. % and 2 wt. %) were prepared by melt blending compounding using a twin-screw extruder. The sample was purified by mechanical separation and was molded in a traditional design using compression molding at 260 °C and a strength of 12 MPa. The specimens were then cooled to room temperature. A related approach was used to combine a real PMMA specimen externally, starting with each reinforcement. The synthetic process involved the preparation of pure PMMA, PMMA/1wt.% nanoclay, and PMMA/2wt.% nanoclay, and the synthetic details such as formulation composition and preparation conditions are listed in Table 2. The schematic representation of preparation and molding of the nanocomposites is depicted in Fig. 5.

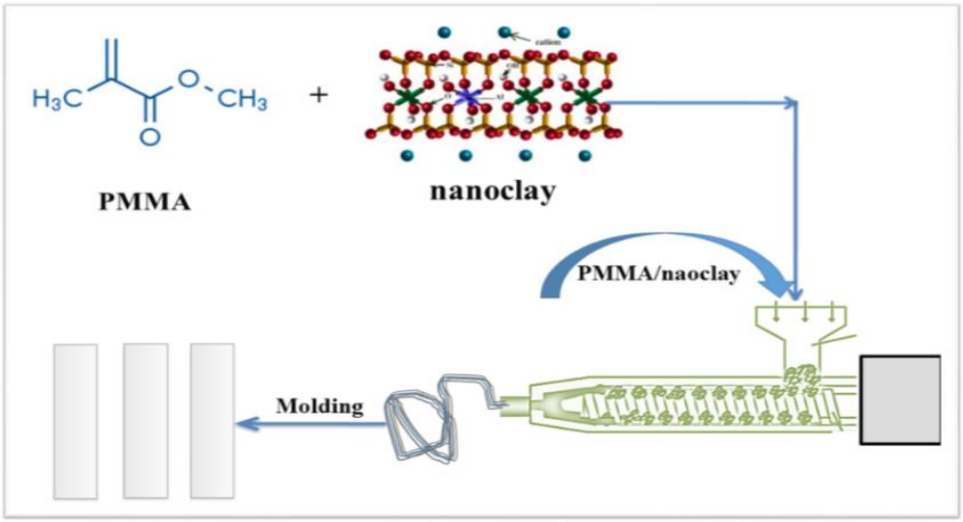

Fig. 5 Schematic demonstration of PMMA, PMMA/1%nanoclay, and PMMA/2%nanoclay

Table 2. Combining formulation and dispensation conditions

Model	Preparations and collaborating order	Handling conditions
PMMA	PMMA (60g) + nanoclay (0g)	260 °C, 12 MPa.
PMMA/1wt%NC	PMMA (60g) + nanoclay (0.6g)	260 °C, 12 MPa.
PMMA/2wt%NC	PMMA (60g) + nanoclay (1.2g)	260 °C, 12 MPa.

3.3 Micro Indentation Analysis

The Indentation module and Vickers hardness of the PMMA and PMMA nanocomposites were evaluated by micro-indentation analysis using a completely automatic micro-indentation machine (model MTR3/50–50/NI, MICRO TEST S.A, Spain). The device was regulated by Tribotester software installed in the indentation machine. The properties of the composites were compared with those of pure PMMA via a test procedure carried out under force control at the maximum indentation load of 5 N. The specimens were unloaded after holding 30 sec at 5 N load. The experiments were repeated in triplicates to ensure the reproducibility of results.

3.4 Micro-scratch Analysis

Micro-scratch measurement was conducted using an auditory radiation-based Microtest Scratch Validator (MTR3/50–50/NI) instrument. A diamond slope indenter of the radius 200 mm (Indenter category Rockwell C) was applied for scratching the exteriors of the models. A regular weight of 9.8 N was implemented aimed at opening infiltration prior to the scratching process. The specimens were scratched for 6 mm in one minute with a standardized scratching speed. The scratching strength and the quantity were calculated from Tribotester software.

3.5 FESEM Analysis

The crack exterior morphology and scratch exteriors of the bulk PMMA and its nanocomposite were analyzed by field emission scanning electron microscopy (FESEM) (Pattern: Supra 55 Carl Zeiss, Germany). The fracture exteriors were acquired by separating the specimens in liquid nitrogen. The cracked samples and the scratched samples were formerly glazed with platinum to produce certain the samples are beginning below an accelerating voltage of 5–15 kV.

4. Conclusions

Polymeric dental materials, i.e., PMMA and PMMA/NC nanocomposites, were completely formed by melt blending compounding using a twin-screw extruder machine. The Viker hardness, Indentation Module, and scratch hardness were showed significant results for the nanocomposites than for the pristine PMMA. The increased popularity of these resources is largely connected to the excellent protection of NC against polymer design. In general, this effort reveals the proper dental administration of PMMA/NC nanocomposites.

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Author Contributions

Md Alamgir: Conceptualization, data curation, formal analysis, methodology, investigation, software, validation, formal analysis, investigation, visualization, resorces, writing—original draft, and writing—review and editing. **Zenab Darban**: Formal analysis and writing-review and editing. **Pratyush Verma**: Formal analysis, and investigation. **Siti Nor Atika Baharin**: Formal analysis, investigation, and validation. **Syed Shahabuddin:** Conceptualization, methodology, software, validation, investigation, resources, writing—original draft, writing—review and editing, supervision, and funding acquisition.

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