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RESEARCH NOTE

The Effect of TiO_2 Nanoparticles on Mechanical Properties of Poly Methyl Methacrylate Nanocomposites

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A B S T R A C T

Various applications of nanocomposites were a good motivation to start a study on this wide spreading field of science. Current research is an investigation on incorporating different percentages of TiO_2 nanoparticle as reinforcement to a base material which here is poly methyl methacrylate (PMMA). In this study various percentages of TiO_2 (0.5, 1 and 2 wt%) were added to pure PMMA and effect of this combination on the mechanical properties of produced composite by performing several tests was investigated and compared to the base material. For producing samples, materials were compounded by melting compounding method using a twin screw extruder followed by injecting molding process. SEM images showed that almost all percentage of TiO_2 nanoparticles have been mixed suitably through base matrix. Rockwell hardness R, impact and tensile tests were carried out on all specimens. Almost all of the results illustrated that combination of TiO_2 nanoparticle with PMMA, improves mechanical properties of composite. The results also indicated amazing effect of TiO_2 nanoparticles on improvements of impact and flexural strengths. Highest recorded impact strength showed 229% increase in samples containing 2 wt% nanoparticles compared to the base material.

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1. INTRODUCTION

Queueing Recently, nanotechnology has been extensively studied in academic and industrial communities. Nanostructured materials have recently attracted lot of attention to researchers because of their potential uses in the development of new nano-devices. Nanoparticles are materials with at least one dimension between 1 and 100 nm. In such small range, materials have unique mechanical, chemical and physical properties. Nanocomposites are the materials with a base matrix and one or more nanoparticles as reinforcements. Basic matrix of the nanocomposites may be polymeric, metallic or even ceramic. Reinforcements are usually nano-size powdered metal oxides [1-4], single-walled and multi-walled carbon nanotubes [5, 6] and/or layered materials such as several types of clays [7].

Among nanocomposites, most attention is attracted to polymer-matrix composites due to their outstanding and considerable mechanical [8, 9], thermal [8, 10], physical [11], electrical [12] and optical [13] properties. Many researches have conducted research on the preparation and development of these materials. Different polymers have been used as matrix to produce nanocomposites and their mechanical, physical, morphological and thermal properties have been investigated.

Poly methyl methacrylate (PMMA) is a synthetic resin produced from the polymerization of methyl methacrylate and also one of the most frequently used commercial polymers with good dimensional stability, high mechanical strength, thermal stability, outdoor wearing properties and minimal inflammatory response. It is an important thermoplastic material with extensive applications in many technological fields due to its unique optical [14, 15], mechanical [16], thermal [15] and electrical [16] properties. Owing to these

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characteristics, PMMA is among the most extensively studied polymers for nano/micro composites [17, 18].

Recently, lot of research works on the mechanical and physical properties of nano composites is going on all over the world. Benjamin et al. [19] worked on mechanical properties of PMMA/Al_2O_3 Nanocomposites. PMMA/Alumina nanocomposites were produced by incorporating alumina nanoparticles, synthesized using the forced gas condensation method, into methyl methacrylate. At an optimum weight percent, the resulting nanocomposites showed, on average, a 600% increase in the strain-to-failure and the appearance of a well-defined yield point when tested in uniaxial tension. Al-Kawaz et al. [20] investigated tribological and mechanical properties of acrylic-based nanocomposite coatings reinforced with PMMA-grafted-MWCNT. They studied the tribological performances of 20mm coatings of these nanocomposites deposited on neat PMMA. The coefficient of friction was found to relatively decrease with the increase of the weight fraction of polymer grafted to the surface of MWCNT. Moreover, the elastic modulus also increased with increasing the weight fraction of PMMA coated MWCNT. Liu et al. [21] investigated the synthesis and characterization of PMMA/Al_2O_3 composite particles by in situ emulsion polymerization. They found that only when the concentration of sodium dodecyl sulfate (SDS) was much higher than its critical micelle concentration, could PMMA/Al_2O_3 composite particles with high percentage of grafting (PG) be prepared. Compared to Al_2O_3, thermal stability and dispersibility of the composite particles showed marked improvement. Bezy et al. [22] studied the effect of TiO_2 nanoparticles on mechanical properties of epoxy-resin systems and showed development in maximum flexural modulus. In this study, titanium dioxide nanoparticle was prepared by sol gel method, using titanium tetra isopropoxide and acetic acid. Mechanical studies of developed polymer sheets revealed much variation by incorporation of TiO_2. This is probably due to different dispersion of nanoparticles in the epoxy resin. Navidfar et al. [23] studied the influence of processing condition and carbon nanotube on mechanical properties of injection molded multi-walled carbon nanotube (MWCNT)/ poly (methyl methacrylate) nanocomposites. The results revealed when MWCNT concentration are increased from 0 to 1.5 wt %, tensile strength and elongation at break reduced about 30 and 40%, respectively, but a slight increase in hardness was observed. In addition, highest impact strength appears in the nanocomposite with 1 wt % MWCNT.

In the present study, the effect of addition of TiO_2 nanoparticles as reinforcement in various weight fractions on mechanical properties of poly methyl methacrylate (PMMA) was studied. For this purpose, materials were melt compounded using twin-screw extruder and then samples were produced by injection molding. Mechanical tests including flexural, impact, tensile and hardness were carried out according to appropriate standards. The results were presented and the effect of TiO_2 nanoparticles on mentioned properties was investigated.

2. EXPERIMENTAL DETAILS

2. 1. Materials and Equipment In this study, poly methyl methacrylate (PMMA) with melt flow index of 1.9ml/10min (230°C ×3.8kg) and with Trade name of ACRYREX CM-205 was used as base matrix and TiO_2 nanoparticles as reinforcement. PMMA is produced at Chi Mei Corporation and TiO_2 at US Nano Corporation with purity of 99% and nanoparticle size of 20 nm.

A ZSK-25 (Coperion Werner & Pfleiderer, Germany) twin-screw extruder with 10kg/h extruding capacity and 25mm extruder diameter was used for melt compounding of the materials. Mechanical test specimens were injected using an NBM HXF-128 injection molding machine [with L/D ratio of 21.1, screw diameter of 37 mm and maximum injection pressure of 196MPa].

A Universal STM-150 tensile testing machine (SANTAM) with maximum capacity of 50kN and extension resolution of 0.05mm and a SIT-200 impact test machine (SANTAM) used for carrying out tensile and Charpy impact tests, respectively. An Indentec universal hardness testing machine (Zwick/Roell, England) was employed in order to carry out the Rockwell hardness tests. Also, a GOTECH AI-7000-M machine was used for the bending test. For observing nano-structure of specimen a Hitachi S-4160 FE-SEM (field emission scanning electron microscopy) was used for SEM test.

2. 2. Preparation of Specimens Before compounding, PMMA was dried at 120°C for 2h using an industrial oven. Then, a 2 percent by weight master batch (2 wt% nanoparticle and 98 wt% PMMA) was extruded with twin-screw extruder machine at a screw speed of 250 rpm and melt temperature of 210°C. In the next step, 1 wt% and 0.5 wt% granules were produced by diluting and adding PMMA to 2 wt% granules till defined percentage by weight ratios be prepared. Subsequently, the prepared nanocomposite pellets were dried again in the hopper of injection molding machine at 80°C for 2 h. Then, experimental specimens were produced by injecting the melted pellets into the mold (see Figure 1). The processing parameters of injection molding are indicated in Table 1.

2. 3. Mechanical Properties Tensile test is one of the most important fundamental tests of a material’s mechanical response to investigate the mechanical properties of materials. Tensile strength is the maximum tensile stress that a material can endure before it undergoes cracking, fracture or plastic deformation. Tensile tests were carried out based on ASTM D638 standard at room temperature. At least three samples were examined for each trial and average tensile strength of the results is reported as tensile strength of a specimen. A universal Indentec hardness testing machine was used to perform hardness tests as a suitable surficial mechanical property of specimens. At least five points of a sample were examined and the average of recorded data was reported as the Rockwell-R hardness of the specimens based on ASTM-D785 standard at room temperature. Specimens were notched with angle of 60° and depth of 3 mm for impact tests (see Figure 2).

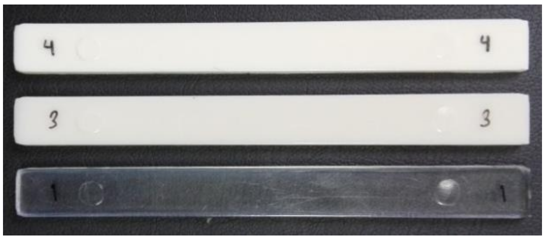

Figure 1. Produced nanocomposite samples

TABLE 1. The parameters of the injection molding

Parameter	Value
Injection temperature (°C)	260
Injection pressure (MPa)	80
Hoding pressure (MPa)	60
Holding pressure time (s)	2
Cooling time (s)	25
Mold temperature (°C)	60
Shot size (cm^3)	20

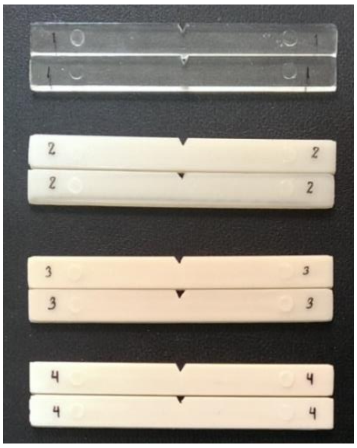

Figure 2. Notched specimens for Charpy impact test

The tests were carried out via a SIT-200 machine according to ASTM D6110 standard. At least three specimen of each sample were tested and reported results are the average of these three specimen.

3. RESULTS AND DISCUSSION

3. 1. SEM Results

Figure 3 shows SEM cross-section images of specimens. By comparing the base material with TiO_2 contained ones, it is clear that combination of nanoparticles is suitable and monotonous which means no agglomeration was observed in the structures of nanocomposites. This monotonousness in the structure of material promises increasing in the mechanical properties of nanocomposite specimens compared to pure PMMA specimens which will be studied in coming sections.

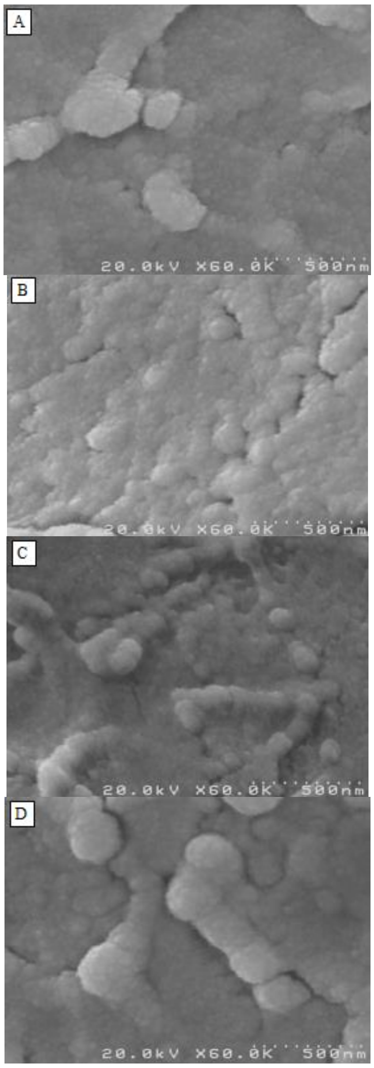

Figure 3. SEM cross-section view of a) Pure PMMA, b) PMMA/0.5 wt% TiO_2, c) PMMA/1 wt% TiO_2 and d) PMMA/2 wt% TiO_2

3. 1. Flexural Strength The first test carried out on the specimens was bending test. The loading rate was set on 5mm/s and the temperature of testing environment was 25°C based on standard test temperature. According to the results (see Table 2), flexural strength of specimen with 0.5 wt% was increased, but specimens containing 1 wt% and 2 wt% TiO_2 nanoparticles almost stayed unchanged compared to pure PMMA. This increase is because of the unique properties of nanoparticles. Figure 4 shows force-displacement curves for PMMA-0.5wt% TiO_2 and pure PMMA.

3. 2. Impact Strength Impact test was done on the specimens using Charpy method. Cross section of each specimen was measured. According to the test results (see Table 3), all specimens showed increasing behavior in the impact strength as shown by Ghasemi et al. [24] and Srivastava et al. [25] for other polymers as PP and Epoxy, respectively.

TABLE 2. Flexural strength of nanocomposite samples

Material	Flexural strength (MPa)
Pure PMMA	4.020
PMMA containing 0.5 wt% TiO_2	4.171
PMMA containing 1 wt% TiO_2	3.979
PMMA containing 2 wt% TiO_2	3.989

TABLE 3. Impact strength of nanocomposite samples

Material	Impact strength (kJ/m^2)
Pure PMMA	8.11
PMMA containing 0.5 wt% TiO_2	9.88
PMMA containing 1 wt% TiO_2	15.72
PMMA containing 2 wt% TiO_2	26.68

According to Figure 5, 21.8%, 93.8% and 228.9% increase in the impact strength of samples containing 0.5, 1 and 2 wt% TiO_2 nanoparticles were observed, respectively. For specimens with 2 wt% TiO_2, amazing number of 229% verifies incredible effect of added nanoparticles on the impact strength of pure polymer.

3. 3. Rockwell Hardness R The hardness tests were performed according to Rockwell-R method. The results are given in Table 4. As seen in this table, hardness of base material is 76.60 HRR, and all specimens containing various weight fractions of nanoparticles showed increase in hardness. This could be because of microstructural bonds between base matrix and added nanoparticles. These bonds endure the applied force instead of base matrix and because of this effect, more energy is needed to break these bonds.

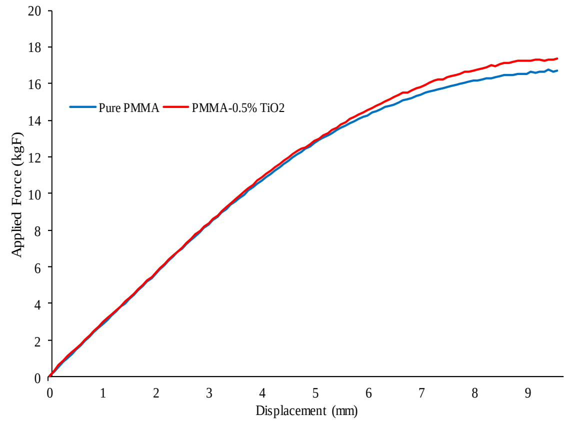

Figure 4. Comparison between flexural test results of pure PMMA and PMMA containing 0.5 wt% TiO_2

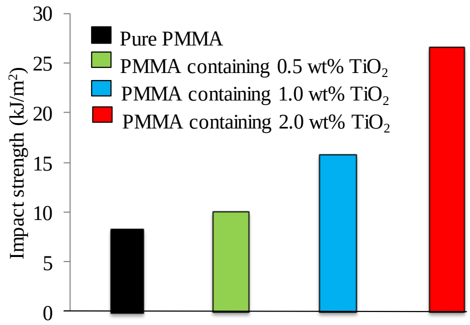

Figure 5. Comparison between impact strengths of nanocomposite samples

TABLE 4. R-Rockwell hardness of samples

Material	Rockwell hardness R
Pure PMMA	76.60
PMMA containing 0.5 wt% TiO_2	76.95
PMMA containing 1 wt% TiO_2	77.03
PMMA containing 2 wt% TiO_2	80.37

3. 4. Tensile Test For the tensile test, rate of loading is set to 10 mm/s. This study will focus on 2 important properties (Young's modulus and ultimate tensile strength) obtained from tensile test. The results are based on ASTM D638 standard.

3. 4. 1. Young's Modulus First outcome of tensile test is Young's Modulus. Based on Young's modulus results (see Table 5), adding nanoparticles at various percentages of 0.5, 1 and 2 wt% shows increase in the Young's modulus; The same influence on Epoxy has been shown by Srivastava and Tiwari [25]. The increase in Young's modulus is uniform (see Figure 6) with increasing the percentage of TiO_2 nanoparticles. This proceeding confirms TiO_2 nanoparticles reinforcement effect on the base material.

TABLE 5. Young's modulus of nanocomposite samples

Material	Young's modulus (MPa)
Pure PMMA	2914
PMMA containing 0.5 wt% TiO_2	2949
PMMA containing 1 wt% TiO_2	3023
PMMA containing 2 wt% TiO_2	3112

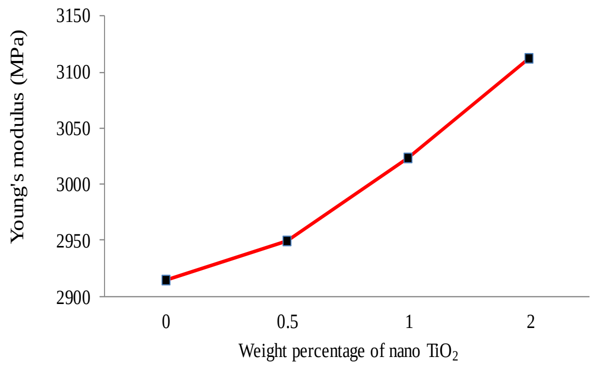

Figure 6. Linearly increasing behavior of Young's modulus

3. 4. 2. Tensile Strength According to results of tensile test (see Table 6), it is clear that all specimens containing nanoparticles showed reduction in tensile strength as shown in literature [25] for Epoxy. Another noticeable result obtained from mechanical behavior of the nanocomposites is a considerable reduction in the elongation at break of the samples containing nanoparticles in tensile test. Figure 7 demonstrates stress-strain curves and elongations at break for pure and nanoparticle filled specimens under tensile test.

TABLE 6. Tensile strength of nanocomposite samples

Material	Tensile strength (MPa)
Pure PMMA	79.16
PMMA containing 0.5 wt% TiO_2	75.64
PMMA containing 1 wt% TiO_2	38.17
PMMA containing 2 wt% TiO_2	48.98

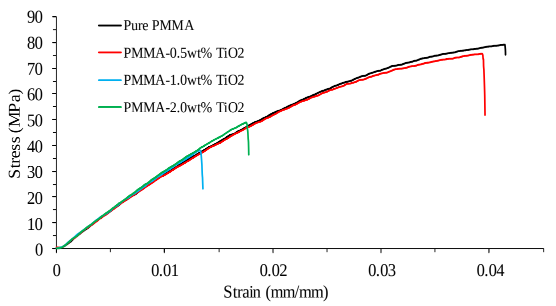

Figure 7. True Stress-Strain Curves of tensile test for nanocomposite samples

As shown in Figure 7, a simple calculation shows more than 50% reduction in elongation for specimen with 2 wt% of TiO_2 and almost 70% for 1 wt% of TiO_2. But, for 0.5 wt% of TiO_2 this reduction is about 5%.

4. CONCLUSION

In this research, the effect of incorporating different amounts of TiO_2 nanoparticles on PMMA was investigated. The results of SEM tests showed that nanoparticles were distributed in polymeric matrix appropriately in all contents. Four mechanical tests were carried out on samples, and following conclusions were obtained:

1. Flexural strength of all specimens almost stayed unchanged. Samples with 0.5% wt. TiO_2 showed 3.75% by mean improvement in the flexural strength compared to the pure PMMA.
2. Impact strength of all specimens increased as well, and this increasing was uniform. Specimens with 2% wt. TiO_2 showed the highest impact strength. Improvement was about 229% (compared to the base material).
3. Results of hardness test illustrated that Rockwell-R Hardness of all specimens improved. The highest was for samples with 2% wt. TiO_2.
4. Young's modulus for all samples was improved. Again, the highest was for samples with 2% wt. TiO_2.

True stress-strain curves of specimens indicated that ultimate tensile strength of all samples has reduced. This could be because of agglomerating effect. The agglomerated compounds can act as stress concentrating centers in the matrix and adversely affect the mechanical properties of the polymerized material. The additional TiO_2 nanoparticles act as impurities and the tensile strength decreases as a result of the extra additive.

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RESEARCH NOTE

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چکیده

کاربردهای وسیع نانوکامپوزیت‌ها، محرک مناسبی برای شروع یک مطالعه در این زمینه‌ی گسترده‌ی علمی می‌باشد. تحقیق حاضر، یک مطالعه روی پلی‌مر پلی‌متیل متاکریلات حاوی درصدهای مختلف نانوذرات اکسید تیتانیوم به عنوان تقویت-کننده می‌باشد. در این تحقیق درصدهای وزنی مختلف نانوذرات اکسید تیتانیوم شامل ۰/۵، ۱ و ۲ درصد به پلی‌متیل متاکریلات خالص اضافه شده و تاثیر این افزایش نانوذرات بر روی خواص مکانیکی کامپوزیت‌های تولید شده بررسی شد. برای تولید نمونه‌ها، مواد به روش اختلاط ذوبی توسط دستگاه اکسترودر دوماردونه ترکیب و سپس توسط دستگاه تزریق، قالب‌گیری شدند. نتایج میکروسکوپ الکترونی روبشی مشخص ساخت که نانوذرات اکسید تیتانیوم تقریباً در تمامی درصدهای وزنی به صورت مناسب داخل پلی‌مر پایه پخش شده است. آزمون‌های مکانیکی انجام شده شامل خمش، سختی راکول، ضربه و کشش می‌باشد. مطالعات اولیه نشان داد که تقریباً نتایج تمامی آزمون‌ها مبین این امر هستند که افزودن نانوذرات به پلی‌مر پایه سبب بهبود خواص مکانیکی آن می‌شود. مطالعات عمیق‌تر مشخص ساخت که افزودن نانوذرات اکسید تیتانیوم به صورت چشم‌گیری خواص ضربه‌ای و خمشی پلی‌متیل متاکریلات را تحت‌الشعاع قرار داده است. بهبود ۲۲۹٪ در استحکام ضربه‌ای نمونه‌های حاوی ۲ درصد وزنی نانوذرات اکسید تیتانیوم مشاهده گردید.

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