THE STUDY OF ELECTRICAL AND MECHANICAL PROPERTIES OF MONTMORILLONITE CLAY POLYESTER NANO-COMPOSITE

[1*]Borisade, S.G; [2]Oyelaran, O.A; [1]Sanusi, O.M
[1]Department of Materials and Metallurgical Engineering, Federal University Oye-Ekiti.
[2,3]Department of Mechanical Engineering, Federal University Oye-Ekiti, Nigeria

*Corresponding Author e-mail: sunday.borisade@fuoye.edu.ng

ABSTRACT

Montmorillonite clay polyester nano composite were successfully prepared by melt insertion method at 5wt%, 10wt%, 15wt%, 20wt% and 25wt% of Montmorillonite clay. The electrical and mechanical properties of the produced composites were studied. Five specimens for each test was analysed. Results obtained indicates that while a drastic decrease in the impact energy of >100 was observed, the maximum tensile strength and young's modulus values of 50.27 MPa and 8.7 GPa respectively were obtained at 10% filler concentration. The compressive strength increases by 48% and 100% at 15wt% and 20wt% filler concentration respectively. An increase in dielectric strength (>100%) and capacitance (32%) of the samples with each filler addition of up to 25% with 3 times increase in hardness at 25wt%. was observed.

Keywords: Montmorillonite Clay, Mechanical Properties, Chemical Composition, Polyester Nano Composite.

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Introduction

Composite materials were created due to rigorous research for materials that offers high reinforcement levels, with better mechanical properties and lighter in weight (Ataiwi *et al* ., 2012). Polymer composites may be grouped according to fillers size as macro-composites, micro-composites and nano-composites (Ajayan *et al*., 2003).Synthetic fiber reinforced polymer composites have been applied for automotive, civil infrastructure, aerospace, defense, marine, sporting goods, etc. for the past thirty years giving better strength and stiffness, thermal properties and dimensional stability (Chowdhury *et al* ., 2008) The finite nature of the synthetic fibers and polymers and as a result of the new obligations on environmental pollution the attention of the researchers was shifted to bio-composites with the utilization areas remaining the same (Ahmeda *et al.,* 2008). Additionally, the dawn and utilization of nanotechnology have caused renewed curiosity in composites which show hopeful potential as next generation material for structural applications (Kumar et al ., 2010)

Majorly Montmorillonite are utilized because of their special properties especially their exchange capacity and swelling capability. They have two fused silica tetrahedral sheets of aluminum or magnesium hydroxide (Kumar *et al.,* 2010) Ion exchange ability are also enhance by its structures using an environmentally friendly approach based on direct polymer melt intercalation which are achieved by replacing the ion residing in the interlayer via ion exchange reactions rendering the hydrophilic silica organophilic (Dobrzanski *et al* ., 2010). Montmorillonite (MMT) are now been used in different areas such as thixotropic agents in cosmetics and paints, adsorbents in treatment of contaminated waste streams as well as fillers in nan-composites due to their unique intercalation/exfoliation properties (Ajayan *et al* ., 2003). Montmorillonite clay is receiving great consideration as reinforcement materials for polymer as a result of its exceptional intercalation/exfoliation characteristics and potentially high aspect ratio. A small inclusion of Montmorillonite clay to polymer matrix shows unexpected properties including improved solvent resistance, better mechanical properties and thermal stability, decreasing gas permeability, and improved flame retardant properties (Inceoglu *et al., 2010).*

Polymer Nano-composites are new group particle-filled polymers composites of which at least one of the dispersed particles dimension is of nanometre range . Inceoglu and Yilmazer stated that the tensile strength, tensile modulus, flexural strength, and flexural modulus of neat UP were enhanced with the inclusion of up to 5wt% clay. However above 5wt% of clay, both tensile and flexural properties were decreased. The first mention of polymer / clay Nano-composite technology in the literature was in 1949 and credited to Bower that performed DNA absorption using montmorillonite clay (Inceoglu *et al ., 2010).* Other studies also revealed that monomers can be interposed amid clay mineral platelets (and clay surface can act as a polymerization motivator (Inceoglu *et al., 2010)*. in 1963, polyvinyl alcohol / montmorillonite Nano-composites was prepared in aqueous medium. In the late 1980s a great breakthrough in the polymer clay nano-composite was achieved by the preparation and characterization of polyamide 6 organophilic clay nano-composite used as timing belts in cars. This new material, that only had only 4.2 wt. %, had an increase of 68% in the Young modulus. 40% in the rupture tension, and 126% in the flexural modulus in addition to a raise in the heat distortion temperature from 65 to 152℃ when compared with pure polymer. Since then, begin the introduction of thermoplastic nano-composites, such as polyamide and polypropylene, in automotive application. The properties achieved by the nano-composite are as a result of phase interactions that occur between the nanoparticles and polymer matrix at the interface .This is because many vital chemical and physical interactions are always governed by surfaces (Pothan, 2007). The hybrid nano-composite systems are currently receiving increased attention as they are based on organic and inorganic layer silicate. As a result of their unexpected hybrid properties which can be utilized by synergistic combinatorial benefits of the constituents Polymer (Pothan, 2007). nano-composites materials consist of organic and inorganic fillers at nano-scale usually less than 100µ.

The abundance and ease of exploitation are excellent sources of nano particle inorganic materials that can be used in diverse applications. The utilization of polymer-clay nano composite (PCN) is now been considered because of their enhance properties and are one of the promising materials for energy storage applications. They are based on the amalgamation of inorganic materials of high permittivity with polymer of high breakdown strength may promote the energy storage capacity of the produced nano-composite (Inceoglu *et al.,* 2003). Researchers have successfully synthesized a number of polymer/clay nano-composites (Pothan, 2007). The concept have been extended to the production of biodegradable polymer nano composite (Inceoglu *et al.,* 2003). The focus of these studies is to extract montmorillonite clay deposits found around surulere area, Oyo state in Nigeria and formulate nano-composite with polyester as the matrix at high filler concentration for possible use in electrical and structural installations with the aim of enhancing their potential in the exploitation of local resources for industrial applications.

2.0 Experimental Procedures

2.1 Collection and preparation of clay sample

Clay samples were collected from a site in Aleru village in Surulere area, Lagos State, Nigeria. The clay collected were crushed with mortar and pestle with the aims of removing the impurities and to obtained smaller particle size of the clay. The crushed samples after removal of impurities were soaked in water overnight after which it was decanted and sun-dried. They were further crushed to fine sample and were sieved through sieve aperture of 53µm. In determining the chemical constituents of the clay, the Atomic Absorption Spectroscopy method was used. In this process, the instrument was calibrated while solutions of the specimens were prepared.

2.2 Polyester resin and other chemicals

Three litres of polyester resin were purchased in liquid form, cobalt-naphthalene (accelerator), methyl-ethyl-ketone (catalysing agent) and petroleum jelly (deboning agent) were all purchased from a chemical store in Ibadan, Nigeria.

2.3 Modification of montmorillonite clay

The melt intercalation method as used by (32) was adopted in carrying out the montmorillonite treatment. 200 gram of clay was added to 100ml 0.5M ferric sulphate solution and stirred for 4 hours by magnetic stirrer and kept overnight. It was then filtered and washed with distilled water to make them free from SO_4^{2-} ions and were dried at 150℃ in an oven to have Fe^{3+} exchanged montmorillonite. 10g of the exchange ion montmorillonite clay was added to 50ml water in a beaker, stirred for 3 hours and allow for a night making the montmorillonite clay to swell. The swelled montmorillonite clay suspension was added to 1M HCl aqueous solution and also stirred for an hour. To the suspension of clay was added aniline in the ratio of 50 ml aniline: 10 g of clay. The suspension was stirred for the intercalation of aniline into silicate layers and kept for at 0℃ overnight. The solution was then filtered and substrate washed with distilled water to remove excess oxidant, impurities and residual aniline after which it was allowed to dry in an oven for 2 hours at 105℃.

2.4 Compounding and casting of nano-composite

Measured quantities of treated montmorillonite clay as shown in Table 1, were first mixed thoroughly with polyester resin until homogeneity is achieved allowing for intercalation of clay with the resin after which catalyst agent and accelerator were added and mixed. The compound mixture which is molten was poured into two piece open mould which was allowed to cure at room temperature for 8 hours and was again cure in an oven for 2 hours at 80℃. the cured samples were removed from the mould, fettled and stored in a plastic for test specimen preparation. Five specimen were produced from each sample as shown in Table 1, below for each test conducted.

Table 1- Montmorillonite/Polyester nano-composite formulation composition

MMT clay (wt %)	Polyester resin (cm^3)	Methyl-ethyl-ketone (cm^3)	cobalt-naphthalene (cm^3)
5	245	2.5	2.5
10	245	2.5	2.5
15	245	2.5	2.5
20	245	2.5	2.5
25	245	2.5	2.5

2.5 Determination of mechanical properties

2.5.1 Tensile strength determination

The tensile strength test was carried out using Hounsfield tensometer in accordance with ASTM D638 using a gauge length of 25 mm. Load applications was carried out by turning the handle of the tensometer until the test piece failed. The maximum load and the extension at breakage were recorded. The test was repeated five times on each specimen.

2.5.2 Compression test determination

The compression test was carried out using Hounsfield tensometer in accordance with ASTM D695-02A using specimen of 5 ×5 ×10 mm dimension. The test specimens were placed in between the plates of the tensometer and compressive load were applied until failure occurs. The test was conducted at 32℃ and 28 ±2%. The test was repeated five times on each specimen.

2.5.3 Flexural strength determination

Flexural Strength of samples was also tested on the computerized universal testing machine. The three-point bend flexural test was conducted in accordance with ASTM D 790 method. The σbh flexural strength, namely the maximum stress at break, was calculated using the formula. The test was repeated five times on each specimen.

$$\sigma bh = \frac{3FL}{2bh^2} \quad (1)$$

Where σbh = Flexural strength;

F = Breaking force (Newton);

L = Support distance (mm);

b = Width of Specimen (mm);

h = Thickness of Specimen (mm).

2.5.4 Hardness test determination

Micro-hardness measurement was done using a Leitz Hardness (OS-2H) tester. This tester had a diamond indenter, in the form of a right pyramid with a square base and an angle 136˚ between opposite faces under a load of 3 N in accordance with ASTM E384. The test was repeated five times on each specimen.

2.5.5 Impact energy determination

Impact energy testing was carried out using ATS FAAR impact tested an Izod impact tester according to ASTM D256 standard at room temperature with a single swing of the hammer. The specimen dimension was 65 ×12.5 ×3 mm with 1.2 mm depth under notch. The test was repeated five times on each specimen.

2.6 Determination of electrical properties

2.6.1 Determination of Capacitance

A digital multimeter with precision of ±0.0001 with model of DT9205A (CE) was used for the measurement of capacitance. The results obtained was used to calculate the dielectric constant (ε) using Equation 2 .

$$\varepsilon = \frac{Ct}{\varepsilon_0 A} \qquad (2)$$

Where C is capacitance of the material (ρF), t is thickness of the sample (mm), ε_0 is permittivity of free space (8.85 $\times 10^{-12}$CNM^2) and A is area of sample under electrode (mm^2).

3 Results and Discussions

Chemical composition of the clay

The result of the chemical composition of the clay is shown below.

Table 2 - Chemical constituents of the clay

S/N	Constituent	Composition
1	SiO_2	56.96
2	Al_2O_3	18.19
3	TiO_2	1.06
4	Fe_2O_3	6.39
5	M_gO	0.74
6	ZnO_2	0.03

3.2 Results of mechanical properties of the composites

Effect of the MMT nano-clay addition on the hardness behaviour of MMT clay/polyester nano-composite is shown in Figure 1. From the figure it can be seen that the hardness of the composite material increases as the filler increases. This is in agreement with the findings of (34) wrote that increase in hardness is a function of the filler addition in clay/epoxy nano-composite. They opined that the clay dispersed within the epoxy prevents crystals on the surface of epoxy from being damaged by an external force and also prevents micro cracks existing inside the epoxy from growing up to the surface.

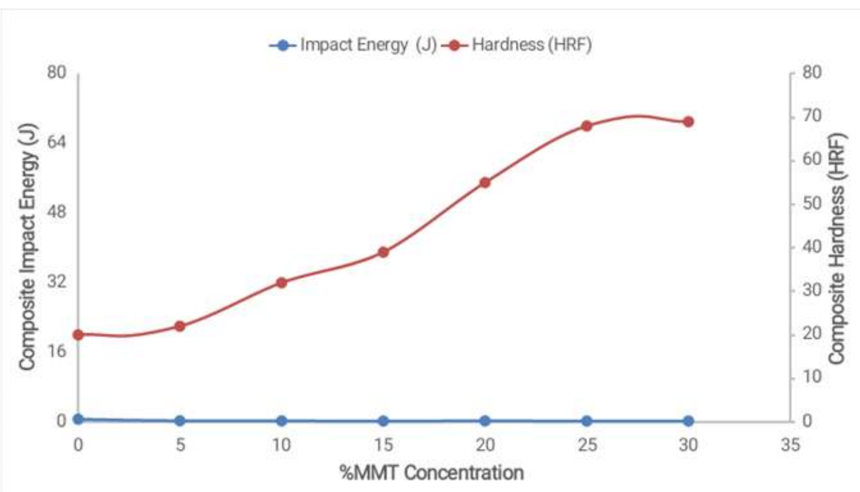

Figure 1- Effect of MMT concentration on hardness and impact energy of MMT. polyester Nano composite

Figure 1 also shows the effect of the MMT nano clay addition on the impact energy behaviour of MMT clay/polyester nano-composite. As displaced in the figure. the addition of MMT clay has an adverse effect on the impact energy of the produced composite. A drastic decrease in impact energy at 5% filler concentration when compared to at no addition of MMT clay and keep decreasing up to 25 wt%. a similar tendency was reported where the impact strength increasingly reduces as filler content increases although at lower filler concentration of 3%. It can therefore be concluded that higher filler concentration is undesirable for applications subjected to impact loading.

The effect of the MMT nano-clay addition on the tensile stress and strain at breakage of MMT clay/polyester nano composite is shown in Figure 2. From the figure it is observed that tensile strength increases from 0% weight of 38.41 MPa to its peak of 43.29 MPa at 10% weight. Further increase in the filler concentration results in decline in the tensile stress at breakage. An increase in viscosity was observed with increasing concentration during composite compounding making degassing difficult, which could aid the entrapment of air thus, forming voids within the mixture causing poor dispersion of the MMT. The reduction in the tensile strength may be ascribed to the increase in MMT content beyond the critical value as written by (26, 31) which may be ascribed to the stress concentration due to filler particles and the entrapment of air at higher loading of filler.

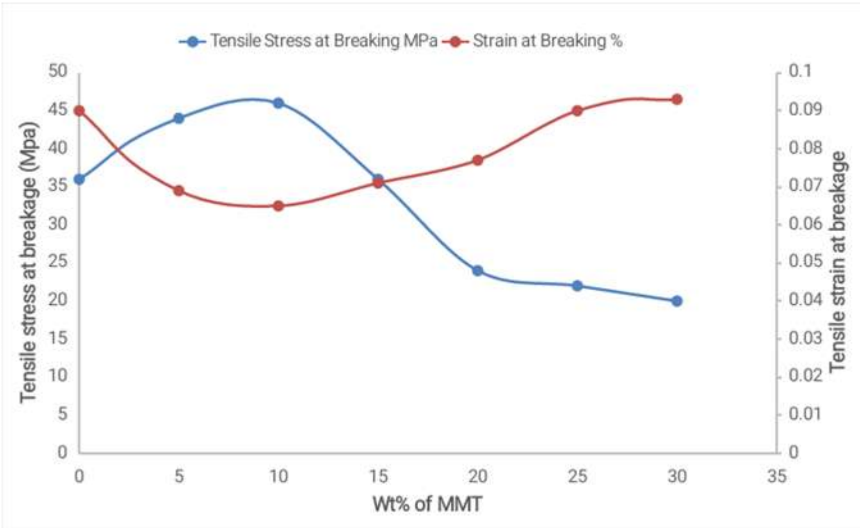

Figure 2- Effect of MMT concentration on tensile stress and strain at breakage of MMT. polyester nano composite.

Figure 3 shows effect of the MMT nano clay addition on the tensile extension and young's modulus of MMT clay/polyester nano-composite. The figure shows an increased in the tensile modulus at 10 wt% filler concentration after which a decline was observed in the tensile modulus with an increased in the filler concentration of up to 25 wt%. reported an increase of 42% in polypropylene/clay nano-composite with clay concentration of up to 7 wt%, after which there was decreased in the in the modulus. They ascribed the decrease to the possibility of layered silicate orientation as while as molecular orientation which might have contributed to the lowering of the stiffness at the clay contents above 5 wt% concentrations.

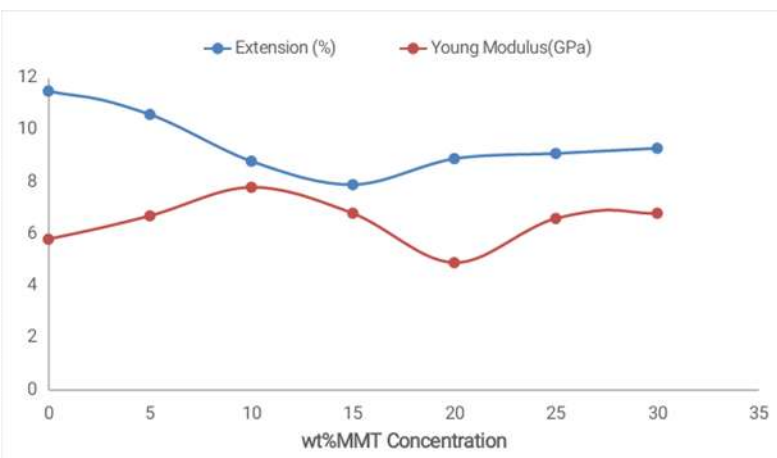

Figure 3 - Effect of MMT concentration on tensile extension and young modulus of MMT. Polyester nano composite.

The effect of the MMT nano clay addition on the flexural and compressive strength of MMT clay/polyester nano-composite is shown in Figure 4. The figure shows an increased in compressive strength with filler concentration of 15 wt% after which there was a decreased in strength with increase in filler concentration. The increase in the compressive strength could be ascribed to the strong interaction between the matrix and filler under compressive loading. The highest flexural strength of MPa as obtained at wt% filler concentration after which the flexural strength begins to decrease. The reduction in the flexural strength may be ascribed to the increase in MMT content beyond the critical value as advanced which may be ascribed to the stress concentration due to filler particles and the entrapment of air at higher loading of filler causing the sample to fail at relatively low stress. The formation of micro voids is less, in lower concentration of filler since dispersion is more uniform, hence, leading to strength improvement. An increase of 70% was reported by Reddy et al., 2010 at 2 w% of clay filler and 30 wt % of wheat straw concentration in polypropylene/wheat straw-clay composite. They authors also wrote that intercalated clays performed less in flexural loading than exfoliated clays. This might be the reason for the moderate increase obtained of the flexural strength in this study, because the samples were melted intercalated.

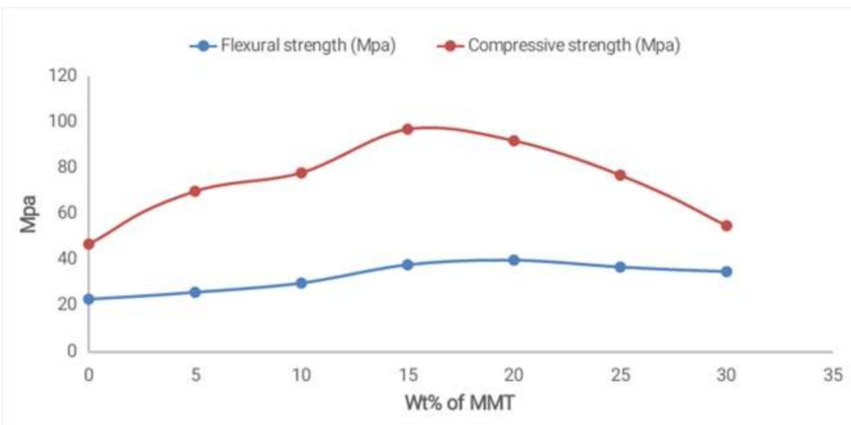

Figure 4 - Effect of MMT concentration on Flexural and compressive strength of MMT. polyester nano composite with percent loading of MMT

3.3 Results of electrical properties of the composites

The effect of the MMT nano clay addition on the capacitance and dielectric constant of MMT clay/polyester nano composite is shown in Figure 5. As displaced in the figure, the increase in capacitance of the samples with increase in filler concentration. Also from the figure, a sharp increase in dielectric constant with MMT clay concentration of up to 10 wt% was observed after which there is gradual increase of dielectric constant up to 25 wt% filler concentrations. Hence, it can be concluded that addition of MMT clay in polyester resin improves both its capacitance and dielectric constant. A similar trend was reported by Pothan et al., 2010 in modified banana fibre polyester composite. This could be attributed to higher permittivity clay than polyester.

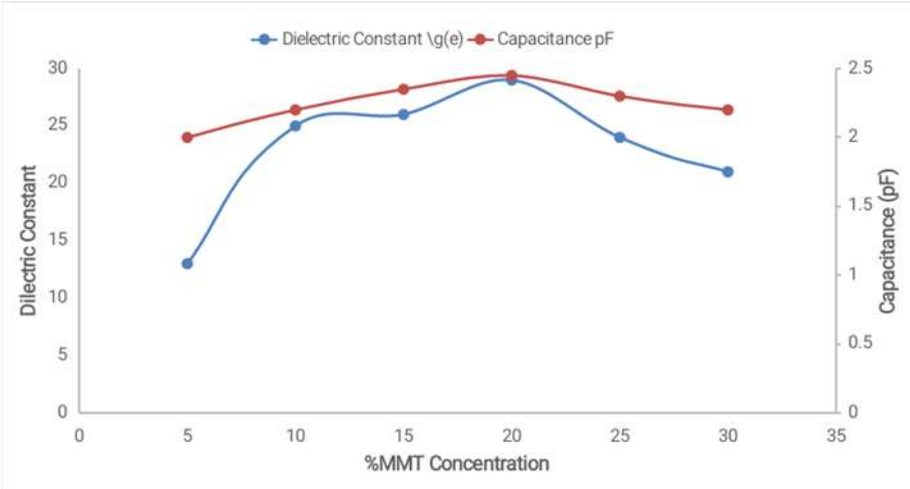

Figure 5 - Effect of MMT concentration on capacitance and dielectric constant of MMT. Polyester nano composite

4 Conclusion

The mechanical and electrical behaviour of the overview of MMT clay in the polyester nano-composite was studied. The results from the studies showed that the tensile stress, tensile strain, elongation at breakage and modulus of the nano composite were less than those of the base polyester at higher filler concentration. There was a sharp decline in the impact energy and compressive strength and becomes progressively less as the filler content increases. The flexural strength of the composite is not affected significantly by filler content. The capacitance, dielectric constant and hardness of the produced nano composite increases up to 25wt% of the filler concentration.

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