ELECTRICAL AND MECHANICAL PROPERTIES OF MONTMORILLONITE CLAY/POLYESTER NANOCOMPOSITE

[*] **Sumaila, M., Dauda, M. and Aliyu, A.**

Mechanical Engineering Department, Ahmadu Bello University,
P.M.B. 1045, Zaria, Kaduna State, Nigeria
[*]sumailachy@gmail.com +234(0)803 515 2182

ABSTRACT

The dielectric and mechanical properties of montmorillonite clay/polyester nanocomposite at various filler loadings were investigated. Five different samples of the nanocomposite were produced using melt interclation method while varying the percentage composition of montmorillonite clay from 5wt% to 25wt%. The tensile, compressive, and flexural strengths and impact energy on five replicate specimens for each test were analysed. Also the capacitance and dielectric strength of the samples were evaluated. Results obtained showed that whereas a drastic decrease in the impact energy of >100% was observed, the maximum tensile strength and Young's modulus values of 50.27MPa and 8.76GPa respectively were obtained at 10% filler loading. Also the flexural and compressive strengths increased by 48% and 100% at 15wt% and 20wt% filler loading respectively. There was also an increase in capacitance (32%) and dielectric strength (>100%) of the samples with each filler addition up to 25% with 3 times increase in hardness at 25wt% as well.

Keywords: *Montmorillonite, polyester, Dielectric, melt interclation, nanocomposite, Capacitance.*

1.0 INTRODUCTION

Nanocomposites are multiphase materials made of distinctly dissimilar components and mixed at the nanometre scale. It is a new class of composites, with at least one dimension of the dispersed phase at the nanometre range [1]. The matrix may be metallic, ceramic or polymeric (thermoplastic or thermosetting). The nanocomposite mechanics are related to the filler volume fraction and specific surface area rather than filler weight fraction. Thus the extremely high surface area per unit volume of the filler material at nano scale is one of the most attractive characteristics of nanoparticles because it facilitates bonding and creates a great amount of interphase in a composite which leads to remarkable improvement in properties at lower filler loading [2,3,4]. The resulting properties of the nanocomposite are due to the phase interactions that take place between the polymer matrix and the nanoparticles at the interfaces since many essential chemical and physical interactions are governed by surfaces [3,5,6]. Research effort in this direction has been growing exponentially worldwide, motivated by the potential of

improved physical (dielectric, optical, gas and liquid barrierand shrinkage) thermal (flame retardancy, thermal decomposition, coefficient of thermal expansion and thermal stability) and mechanical (toughness, strength and modulus) properties at lower filler concentration compared to pristine matrix [1,4,7-16].

One of the most promising nano composite systems is a hybrid based on an inorganic and an organic layered silicate [17]. This nanocomposite system has been receiving increased attention in both the academia and industry for the past decade, due to its unexpected hybrid properties which are exploited in the synergistic combinatorial advantages of the constituents [1,10,18-21]. Polymeric nanocomposites (PNCs) are a relatively new class of composite materials consisting of organic or inorganic fillers at nanoscale (typically <100μ, Thostenson et al., [22], Bai and Liu, [23]) dispersed in a polymeric matrix [10].

Clay minerals, which are abundant and easy to be exploited are an excellent source of nano-sized inorganic materials used in diverse technological and environmental applications. The use of clay in the polymer–clay nano composite (PCN) technology is considered an important technique to produce materials with enhanced properties and represents one of the most promising and exciting avenues for compact and robust energy storage solutions [11]. Such approaches capitalize on the idea that the amalgamation of inorganic materials of large permittivity with polymer of high breakdown strength may benefit the energy storage capacity of the resulting nanocomposite [24].

Smectites, dominantly montmorillonites, belong to the most important clay minerals in use because of their abundance and special properties mostly connected with their cation exchange capacity and swelling ability. Their structure consists of two fused silica tetrahedral sheets of either aluminium or magnesium hydroxide [21, 25].This structure enhances ion exchange capacity, done using an environmentally benign approach based on direct polymer melt intercalation, and achieved by replacing the ions residing in the interlayer via ion exchange reaction thus rendering the hydrophilic silicate organophilic[12, 21, 26]. This process facilitates homogeneous dispersion and bonding of the filler with the matrix phase [10]. Montmorillonites (MMT) have been used in many different applications varying from thixotropic agents in paints and cosmetics to adsorbents in treatment of contaminated waste streams as well as fillers in polymer nanocomposites owing to their high aspect ratio and unique intercalation/exfoliation characteristics [12, 27].

In the 1990s, the Toyota research group in Japan were the fore-runner of the global trend of researches in nanocomposites. These researchers synthesized polymer nanocomposites based on nylon-6/montmorillonite clay via the in-situ polymerization method. When 5wt% clay was added to Nylon-6 polymer, the tensile modulus increased by 68% and the flexural modulus by 224% [5, 10, 14, 28]. Since then a number of authors [6, 10, 14, 29-32] have successfully synthesized a variety of polymer/clay nanocomposites. Today the concept is being extended in the production of biodegradable polymer nano composite [33-36]. However, these studies have been limited to clay concentration of less than 5wt%. The focus of this present investigation is to extract MMT from clay deposits found locally and formulate same into nanocomposite with polyester as the matrix at high filler concentration for possible application in structural and electrical installations. This will enhance our potential in the exploitation of local resources for industrial use.

2.0 EXPERIMENTAL PROCEDURE
Clay sample collection and preparation
8kg of clay sample containing montmorillonite was obtained from a location in Kano, Kano State, Nigeria, and was crushed using a mortar and pestle so as to remove impurities and obtain smaller particles of the soil. The crushed sample was soaked in water overnight after which it was decanted and sun-dried and further crushed into fine particles. The crushed sample was then sieved through a sieve of nominal aperture of 53µm. The physical properties and chemical composition of the clay samples were studied and are as presented in Table 1.

Table 1: Physical properties and composition of Kano Clay

S/N	Constituent	Composition
1	SiO_2	54.26%
2	Al_2O_3	18.34%
3	Fe_2O_3	10.91%
4	TiO_2	1.25%
5	Others	12.24%
6	Water Soluble Salts	3%
7	Moisture	$0 - 9.0\%$
8	pH(6% solution)	$8.5 - 9.5$
9	Sp. Gravity	2.4
10	Bulk Density	0.6gm\cc

Polyester resin and other reagents
Three litres of polyester resin used for this experiment was purchased from a chemical vendor in Jos metropolis, Plateau State, in liquid form. Also, methyl-ethyl-ketone

(catalyzing agent), Cobalt-naphthalene (accelerator) and Petroleum jelly (debonding agent) were purchased locally from a chemical supplier in Zaria in their usual form.

Montmorillonite clay Modification

The montmorillonite treatment was carried out by melt interclation method as adopted from Narayanan et al. [8] whereby 200g of clay was added to 100ml 0.5M ferric sulphate solution. It was stirred for 4 hours using a magnetic stirrer and kept overnight. This was filtered and made free of SO_4^{2-} ions by washing with distilled water, and then dried in an oven at 150^0C to get Fe^{3+} exchanged montmorillonite. Thereafter, about 10g of the ion exchanged montmorillonite clay was added to 50ml water in a beaker. It was then stirred for 3 hours and kept overnight. The swelled montmorillonite clay suspension was added to 1M HCl aqueous solution and stirred for 1 hour. Aniline was then added to this suspension of clay in the ratio 50ml aniline: 10g of clay. The suspension was again stirred for the intercalation of aniline into the silicate layers and was then kept at 0^oC overnight. The solution was filtered and the substrate was washed with distilled water to remove impurities and excess oxidant and residual aniline, and allowed to dry in an oven at 105^oC for two hours.

Nanocomposite compounding and casting

Measured quantities of the treated montmorillonite clay was first throughly mixed manually with the polyester resin in a beaker until homogeneity was attained to allow for the interclation of the clay with the resin. Thereafter, the catalizing agent and accelerator were added and then mixed further.

The compounded mixture, which appears molten at this stage, was then poured into a prepared two-piece open mould and allowed to cure for 8hours at room temperature and further cured at 80^oC in an oven for 2hours. The cured samples were then ejected from the mould, trimmed and stored away in a plastic bag from which the test specimens were cut out.

Table 2: MMT/Polyester nano composite formulation Composition

SN	MMT Clay (wt%)	Polyester Resin (cm³)	Methyl-ethyl-Ketone (cm³)	Cobalt Naphtalene (cm³)
1.	5	235	2.4	2.4
2.	10	235	2.4	2.4
3.	15	235	2.4	2.4
4.	20	235	2.4	2.4
5.	25	235	2.4	2.1

In all, five specimens from each sample of the composite for each test were produced. The composition of each sample is as shown in Table 2.

Determination of Capacitance

The capacitance of the samples was measured using a digital multimeter (Model DT9205A(CE), precision of ±0.0001). From this, the dielectric constant (ε') was calculated using the equation 1 as given by Pothan et al. [37];

$$\varepsilon' = \frac{Ct}{\varepsilon_0 A} \qquad (1)$$

Where C = capacitance of the material (ρF), t = thickness of the sample (mm), ε_0 = permittivity of free space ($8.85 \times 10^{-12} CNM^2$) and A = area of sample under electrode (mm²)

Tensile strength Test

The test was conducted according to ASTM D638 standard with a gauge length of 25mm. It was carried out using a Hounsfield tensometer, Model H12 KW, by manual application of load through the turning handle. The width and thickness of the guage area of the specimens were first measured using a vernier caliper before being mounted and properly gripped in the machine.

Load application commenced thereafter by turning the handle of the tensometer until test piece failure. The maximum load and extension at breakage were then recorded. This was repeated on five replicate specimens of each sample.
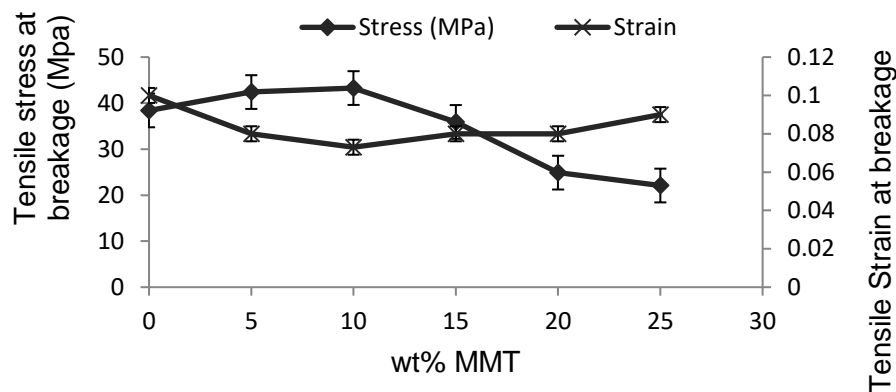
*Figure 1: Effect of MMT concentration on the tensile stress
and strain at breakage of MMT/polyester nanocomposite*

Hardness test

An Indentec universal hardness testing machine (model 8187.5LKV) was used for the hardness test. A cuboid specimen was cut from each sample and was placed on the machine. The machine used a standard 1/16″ diameter hardened steel ball as the indenter. During the experiment, a minor load of 10 kg was first applied on the specimen, to give a small initial penetration to seat the indenter and remove the effects of any surface irregularities. Then, the dial is set to zero and the major load is applied. Upon removal of the major load, the depth reading is taken while the minor load is still on and the hardness number for the specimen can then be read directly from the scale. The hardness test was carried out at three different spots on each specimen. The machine computed and displayed the average of the three readings it recorded.

Compression test

The test was carried out in accordance with ASTM D 695-02a on specimens of dimension 5x5x10mm.
The test specimens were placed between the plates of the Houngsfield tensometer and compression load was applied until failure.

The compression test was carried out at 32ºC with 28 ± 2% relative humidity on five replicate samples for each test.

Flexural Strength Test

Three point bending test was performed in accordance with ASTM D790M test method I, procedure A to measure flexural properties of the composite samples. The samples measured 100x8x5mm. In the test the outer rollers were 64mm apart at and testing was conducted at 0.5mm/min strain rate on five replicate specimens for each sample.

Impact Energy Test

The impact strength of the composite panels was carried out using the izod impact testing machine according to ASTM D 256 standard. Samples were tested in replicates of five each at room temperature by a single swing of the pendulum hammer using ATS FAAR impact tester (Model no. 16.1, capacity of 25 joules). The specimen size was 65 x 12.5 x 3mm with depth under notch of 1.2mm.

3.0 RESULTS AND DISCUSSION
Composite Tensile strength

Figure 1 gives the variation in tensile stress and strain at breakage of the nanocomposite samples at various filler loading. It can be observed that the tensile strength increased from 38.41MPa at 0%wt to 43.29MPa at
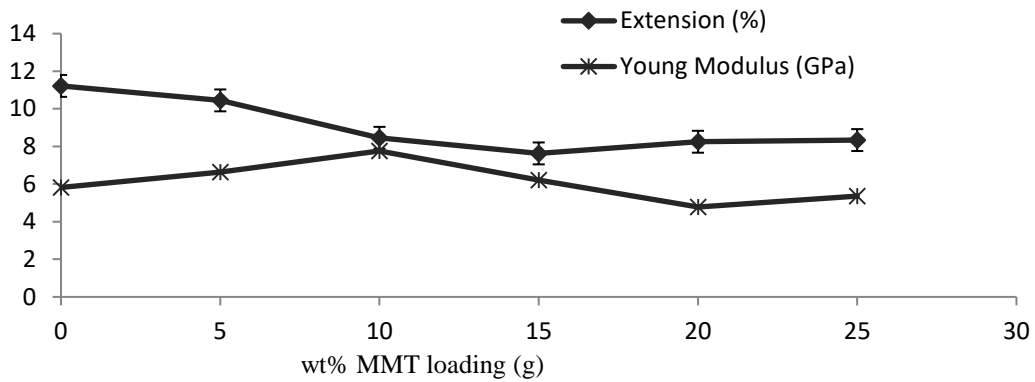
Figure 2: Effect of MMT concentration on the tensile extension and Young's Modulus of MMT/polyester nanocomposite

10wt% MMT concentration where it reaches its peak. Subsequent increase in filler concentration levels showed a decline in the tensile stress at breakage to 22.09MPa. It was observed that during composite compounding, there was an increase in viscosity with increasing level of concentration which made degassing difficult.

This may facilitate the entrapment of air thereby forming microvoids within the blend causing poor dispersion of the MMT. [6, 30, 36] noted that the decrease in tensile strength may be attributed to the increase in MMT content beyond a critical value (10% in this case) which could be attributed to the stress concentration as a result of aggregation of the filler particles and air entrapment at higher filler loading. A decline in the tensile strain of about 27% was recorded for samples with 10% loading from where an increase in the tensile strain up to 0.09 was observed at 25% filler loading. This result agrees with works done by Kaynak et al. [6] and Yasim et al., (2006) where a decline in strain at breakage of up to 3wt% filler concentration was reported. This was attributed to the presence of non-interclated particles and their aggregates acting as stress raisers at higher filler levels.

The variation in percentage tensile extension and the elastic modulus of the nanocomposite at various filler concentrations is presented in Figure 2. It can be seen that the percentage tensile extension was reduced by 26% from 0wt%

to 25wt% meaning that the samples lost some ductility with increase in filler concentration. Hwang et al. [37] and Alexandre et al. [15] reported similar trends, although at filler concentration of <3wt% where a significant decrease in ductility was observed.

Also, from Figure 2, an increase in tensile modulus of 33% was observed at 10wt% filler concentration. Thereafter there was a decrease in the modulus to 5.36GPa with increase in filler levels up to 25wt%.

Liu and Wu [21] reported improvement in modulus of 42% in polypropylene/clay nanocomposite with clay loading up to 7wt%, beyond which there was a decrease. In their analogy, they attributed the decrease to the possibility of layered silicate orientation as well as molecular orientation which might have contributed to lowering of stiffness at clay contents above 5wt% loading.

Composite Flexural and Compressive Strength

Figure 3 shows the result of the effect of MMT clay filler concentration on the compressive and flexural strengths of the MMT/polyester nanocomposite samples. A 100% increase in compressive strength with filler concentration of 15wt% was obtained after which there was a decline in strength to 64.22MPa at 25wt% filler loading. The increase in compressive strength may be attributed to the strong interaction between the filler and the matrix under compressive

loading. Also, the highest value of the Flexural strength of 37.95MPa was observed at 20wt% filler loading representing an increase of 48%, beyond which the flexural strength decreased.

The decrease may be as a result of the reasons advanced by Chung et al. [36] and Qi et al., [30] as mentioned ealier. This in turn resulted in sample failure at relatively low stress. In contrast, with a lower loading of nanoclay, the potential of the formation of micro-voids is less, and the dispersion is more uniform which both lead to strength improvementt. Reddy et al. [30] reported increase in flexural modulus of up to 70% at

30wt% wheat straw and 2wt% clay filler loading in polypropylene/wheat straw-clay composite.

They also noted that exfoliated clays performed better in flexural loading condition than interclated clays. This may explain the moderate increase in flexural strength in the case here, since the samples were melt interclated.

Composite Impact Energy and Hardness

Figure 4 shows the effect of MMT nanoclay addition on the impact behaviour of MMT clay/polyester nanocomposite. As shown the presence of MMT nanoclay had an adverse effect on the impact energy of the resulting composite.
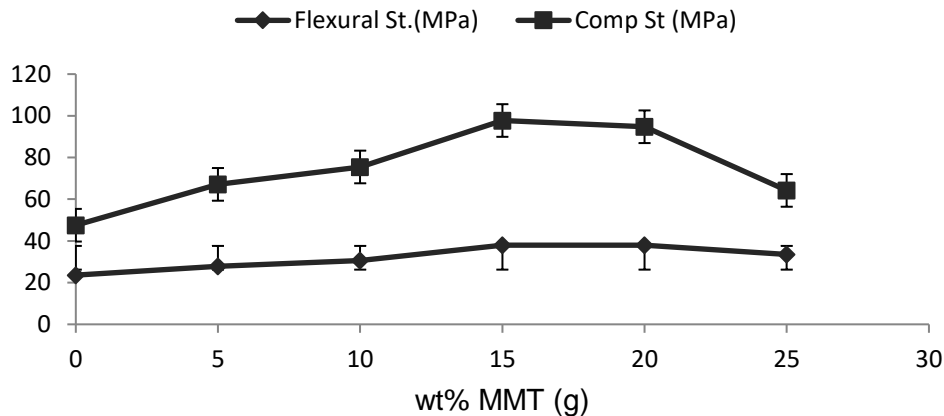

Figure 3: Effect of MMT concentration on Compressive and Flexural strength of MMT/polyester composite with percent loading of MMT
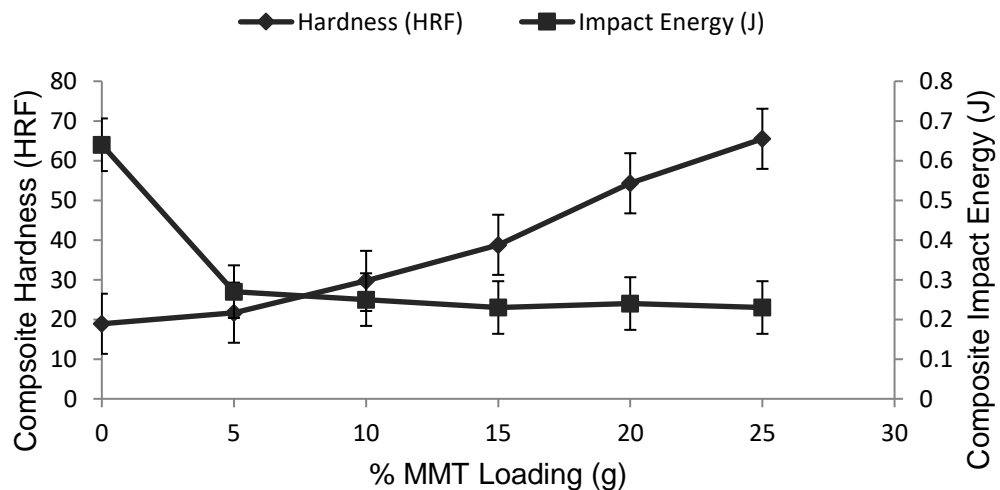

Figure 4:   Effect of MMT concentration on the Impact energy and Hardness of MMT/Polyester nanocomposite

A drastic reduction in impact energy absorbed by >100% at 5wt% filler concentartion compared to prestine polyester was observed. And it became progressively worse with filler addition up to 25wt%.

Alamri et al., [14] and Alamri and Low [29] also reported similar trend in nanoclay-recycled cellulose fibre epoxy composite where the impact strength progressively decreases as filler content increased although at lower(<3wt%) filler concentration. It can therefore be inferred that higher filler concentration is undesirable for applications subjected to impact loading.

The nanocomposite hardness on the other hand improved throughout the filler addition from 21.7RHF at 5wt% to 65.5RHF at 25wt% filler concentration representing an increament of over 3 times. Ha et al. [9] reported increase in hardness as a function of filler addition in clay/epxoy nanocomposite. 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.

Composite Dielectric Constant and Capacitance

The effect of MMT clay concentration on the dielectric constant and capacitance of the MMT clay/polyester nanocomposite samples is presented in Figure 5. It can be seen that there is a sharp increase in the dielectric constant of the samples of >100% with MMT clay concentration of up to 10wt% to polyester and progresses gradually thereafter to 26.92. Also, the capacitance of the samples increased from 1.92pF at 5wt% to 2.44pF at 25wt% filler concentration representing about 32% increament. It can therefore be said that incorporation of MMT clay in polyester resin improves both the capacitance (32%) and the dielectric constant (2 times).

This is expected since clay has higher electrical permittivity than polyester [24].

Pothan et al. [37] reported simillar increament in dieletric constant in modified babana fibre polyester composite.
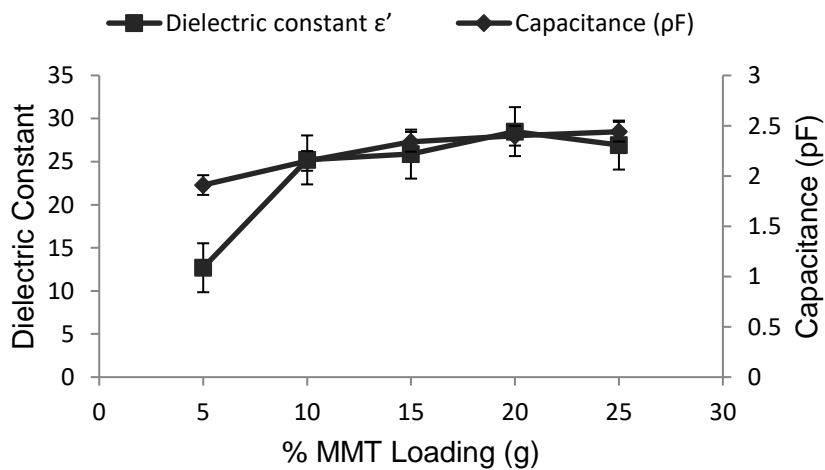

Figure 5: Effect of MMT concentration on the dielectric constant and Capacitance of MMT/Polyester nanocomposite

4.0 CONCLUSION

The effect of high MMT nanoclay concentration on the mechanical and dielectric behaviour of MMT clay/polyester composite was investigated and the following conclusions can be drawn:

i. The tensile properties (tensile stress and strain, elongation at breakage and modulus) of the nanocomposite were less than those of the base polyester at higher filler concentration.

ii. The flexural strength of the composite is not significantly affected by the level of filler concentration.

iii. A sharp decline in the impact energy (>100%) and compressive strength (34%) was observed, and it became progressively less at higher filler concentration levels.

iv. The hardness, dielectric constant and capacitance of the nanocomposite samples improved consistently throughout up to 25wt% filler concentration.

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