August 9th, 2006

Dr. Christopher C. Ibeh
PSU – REU/RET Director
Department of Engineering Technology
Pittsburg State University
Pittsburg, Kansas 66762

RE: Manufacturing and testing of the Nanocomposites Research Program

Dear Dr. Ibeh:

I am glad to inform you that the formal report for the above project has been completed.

This paper will allow you to see in detail what my report consists of the manufacturing of nanocomposites and testing of the samples. This report will also give you an idea about the different types of mixing techniques used in manufacturing of the nanocomposites, and the procedure for mechanical testing using various equipment.

Sincerely,

Manan Aggarwal

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# Evaluation of flammability, mechanical and impact properties of epoxy-clay nanocomposites

**Abstract**

In this work epoxy matrix nanocomposites with different percentage (2%, 4%, and 6%) of nanoclay are manufactured and the effect of nanoclay filler is studied. The mechanical characteristics, such as tensile strength, strain at break and Young's moduli are evaluated. Flammability characteristics are assessed with cone calorimetry. Izod impact tests are also performed.

## 1. Introduction

In the past decade, a lot of research activities were made on polymer-clay Nanocomposites, which showed great promise for obtaining high performance polymer composites material with increased mechanical strength and stiffness. Polymer clay nanocomposites were first reported in the literature as early as 1961, when Blumstein demonstrated polymerization of vinyl monomers intercalated in to montmorillonite clay. The epoxy resin and the hardener were mixed in the weight ratio 100:44 (volume ratio 2:1). When a small amount of nanoclay is added into epoxy resin, there is a large effect on the mechanical properties including tensile strength, impact strength, and Young's modulus. Nanoclay also affects the flammability properties. The purpose of the study is to evaluate the effect of the clay on the epoxy resin. The main significance of the study is to investigate the effect of nanoclay on the characteristic of the composite material. Epoxy resins find many applications in adhesives, construction materials, composites, aircraft industry and spacecraft shuttles owing to their high strength. Epoxy resin is usually brittle material in their cured state and exhibit poor resistance to crack growth. Thus, they are usually combined with nano clay, which modifies its mechanical properties. The objectives of this study are to synthesize nano composite materials and study the effects of nanoclay on the mechanical properties like impact and tensile strength and flammability properties of the materials. This paper is as an overview of the research on the use of clays dispersed in the polymer for improving the mechanical properties and flammability properties.

## 2. Literature review

On the basis of the study done at Monash University [1], the epoxy/clay nanocomposites have been prepared using diglycidyl ether of bisphenol A (DGEBA) and it is blend with an epoxy functionalized hyper branched polymer (HBP). The improvement in mechanical properties is explained in terms of fracture surface analysis by scanning electron microscopy (SEM). Also incorporation of HBP into the epoxy matrix has little effect on flexural strength but the modest reduction in flexural modulus is observed. They have also found that the DGEBA/HBP and DGEBA/clay blend show greater impact strength compared to the neat DGEBA. They have concluded that the strengthening effect of the clay slightly reduces due to the presence of the HBP. Although both the clay and HBP show a toughening effect, in the ternary blend, the net effect is less then that exerted by the HBP alone.

In a research conducted at Tuskegee University, Alabama [2], a novel technique to fabricate nanocomposite materials was developed. The resin used in this study is a commercially available SC-15 epoxy (obtained from Applied Poleramic, Inc). Consisting of part A (resin mixture of diglycidylether of bispnol- A (DGEBA), aliphatic diglycidylether epoxy toughener) and part B (hardener mixture of cycloaliphatic amine and polyoxylalkylamine). The inorganic clay used in this study was K-10 grade montorillonite (obtained from Sigma-Aldrich Co.,USA) with a surface area 220-270 $m^2/g$. once the samples are prepared by mixing of part A and the part B. The samples were poured in the molds and then were post cured at $100^0$ C for 5 hours in an Oven. Dynamic Mechanical analysis (DMA), Thermogravimetric analysis (TGA) and three-point bending tests were performed on nanocomposites to identify the loading effect on thermal and mechanical properties of the composites. From the DMA and three-point bending test, it was observed that the mechanical properties change with respect to the clay content, however, the TGA results showed that the thermal stability of composite does not change in addition of the clay content. The three-point bending test results indicate that 2 wt. % loading of clay in epoxy showed the highest improvement in flexural strength as compared to others and likewise, DMA studies also revealed that 2.0 wt. % doped system exhibit the highest storage modulus.

In Wang et al. [3], positron annihilation lifetimes have been measured for epoxy resin/organic montomorillonite (OMMT) nanocomposites. Effects of different dispersion states of nano-layered OMMT on the positron annihilation parameters and the mechanical properties were studied. Epoxy resin (diglycidyl ether of bisphenon A, DGEBA) CYD-128, OMMT and methyl four phathalic anhydride (MeTPHTA), 2-ethy-4mehtyl imidazole as a curing agent and accelerator were used to make the epoxy resin nanocomposites using the melting blend technology. The evaluation of layers was done by using X-ray diffraction (XRD) measurement using a Rigaku RAD-B diffractometer with CU Kα radiation generated at 40 ke B and 30 mA and scanning rate of $1.5^0$ / min. The impact and flexural strength tests were carried out using an impact tester (XCJ-400) according to the ASTM-D256 and universal testing machine (ZMGI250, Industrirerk Co. Ltd., Germany).

After performing the test, it is concluded that there is a significant increase in the flexural and impact strengths when compared to that of unfilled epoxy. When the OMMT content is ≤ 2wt%, nano-scale layered OMMT are well dispersed in the epoxy matrix, which leads to the formation of the exfoliated structure. For OMMT content >2 wt%, the exfoliation and intercalation structures coexist, the interfacial interaction between OMMT and matrix is reduced, resulting in poorer mechanical properties. Thus it can be concluded that exfoliated structure enhances the flexural and impact strengths of nanocomposite due to the stronger interfacial interaction between OMMT and epoxy matrix.

The research done at Marquette University [4], polyethylene clay nanocomposites have been prepared using melt blending in brabrender mixer. X- ray diffraction and transmission and transmission electron microscopy were used to characterize the structures of the nanocomposites. The thermal stability was evaluated using the thermogravemetric analysis and the flammability parameters using the cone calorimetry. The melt blending process is used to prepare nano-composites of polyethylene by mixing with clay in the absence or presence of maleic anhydride (MA). Low-density polyethylene was acquired from Aldrich chemical company is used as the material for preparation of the sample. The clay used is of two types:

- No functionality clay: 6A, 20 A or 25 A
- Functionality: 30 B, VB16 and Si18.

It is found that the PE clay nanocomposites have a mixed immiscible intercalated structure and there is better intercalation when maleic anhydride is combined with the polymer and clay to be melt blended. It is also observed that there is reduction in peak heat release rate is 30-40 %.

## 3. Nanocomposites material testing: - nanoclay/epoxy

Diglycidyl ether of bisphenol A type epoxy resin treated with montomorillonite clay is observed in this study. The effects of addition of nanoclay as an impact modifier on thermal and mechanical properties of nanocomposite are investigated by using the izod impact test, tensile test. The flammability properties are also determined by using the cone calorimeter. These tests are performed using the ASTM standards.

### *3.1 Izod impact test*

The izod impact test is based on the rationale that when instantaneous impulse is applied to the nanocomposite sample, it fractures at a certain energy level. The izod impact test measures this energy level for different samples. The ASTM standard used for performing the Izod impact test for determining the impact strength is ASTM D 256-03. **[5]** The standard test for these test method require a specimen made with a mild notch. The notch produces a stress concentration that increases the probability of a brittle, rather than a ductile, fracture. According to the standards the results of all the test methods are reported in terms of energy absorbed per unit of specimen width or per unit of cross sectional area under the notch. Before proceeding with these methods, reference should me made to the specification of the material being tested. If there is no material specification, then default conditions apply. For relatively brittle materials like nanocomposite, fracture propagation energy is small in comparison with the fraction initiation energy. The toss correction (Energy to throw the free end of the broken specimen) obtained in Izod test is only an approximation of toss error, since the rotational and rectilinear velocities may not be the same during the re- toss because store stresses in the specimen may have been release as the kinetic energy.

The position of the pendulum holding and releasing mechanism shall be such that the vertical height of fall of the striker shall be 24.0 ± .1 inches. This will produce the velocity of the striker approximately 11.4 ft /s.

$$V = (2gh)^{1/2}$$

Where

*V*= velocity of the striker at the moment of impact (m/s).

*g* = gravitational acceleration (m/s2)

*h* = vertical height of fall of striker

The effective length of the pendulum shall be between12.8 – 16.0 in. so that the required elevation of the striker is obtained. During an Izod impact test, the specimen is held as a vertical cantilever beam. It is broken by a single swing of the pendulum. The line of initial contact is a fixed distance from the specimen clamp as well as from the centre line of the notch and is on the same face as the notch.

### *3.2 Tensile tests*

The tensile test is performed on the INSTRON universal testing machine model- 4467 using the standard ASTM D – 638. For tensile test, dog bone shaped samples is used. Samples are clamped in the holders from the end. Holder stretches the sample in the opposite direction until the crack is initiated and the sample breaks. The Young modulus, strain at break, strain at peak load and strain at peak stress are calculates using the standards. The Young modulus is the very important factor and it is measured in mega Pascal (MPa) Young's Modulus (also known as the *Young Modulus*, modulus of elasticity, elastic modulus or tensile modulus) is a measure of the stiffness of a given material. It is defined as the ratio, for small strains, of the rate of change of stress with strain. Young's modulus, *Y*, can be calculated by dividing the tensile stress by the tensile strain:

$$Y = \text{tensile stress} / \text{tensile strain} = F/A_0 \;/\; \Delta l/l_0 = F l_0 / A_0 \Delta l$$

where *Y* is the modulus of elasticity, measured in pascals; *F* is the force applied to the object; $A_0$ is the original cross-sectional area through which the force is applied; Δl is the amount by which the length of the object changes; and $l_0$ is the original length of the object. The strain is calculated as given that strain results in the deformation of a body, it can be measured by calculating the change in length of a line or by the change in angle between two lines (where these lines are theoretical constructs within the deformed body). The change in length of a line is termed the *stretch*, absolute strain, or extension, and may be written as $\delta\ell$. Then the (relative) strain, $\epsilon$, is given by

$$\epsilon = \frac{\delta\ell}{\ell_0}$$

where $\ell_o$ is the original length of the material. The extension ($\delta\ell$) is positive if the material has gained length (in tension) and negative if it has reduced length (in compression). Because $\ell_o$ is always positive, the sign of the strain is always the same as the sign of the extension. Strain has no units of measure because in the formula the units of length are cancelled.

### *3.3 Flammability test*

The rationale for flammability test is to burn a sample by introducing a heat flux by using the cone calorimeter. The cone gives the sufficient heat flux to increase the temperature of the sample so that the sample can burn by just initiating the spark. The time to ignite, heat release rate and smoke release rate can be determined. The cone calorimeter used for the test is of Fire Testing technology Ltd. Charles wood road, UK. The FTT Cone Calorimeter has been produced to meet all existing Standards (including ISO 5660, ASTM E 1354, ASTM E 1474, ASTM E 1740, ASTM F 1550, ASTM D 6113, NFPA 264, CAN ULC 135 and BS 476 Part 15).

# 4. Methodology

In this section, we are going to discuss method of mixing the clay in the epoxy resin. The structure and properties of the clay are also discussed. The equipment used for the testing of the samples and their working and calibration is also illustrated.

## 4.1 Materials used

The epoxy resin used was liquid diglycidil ether of bisphenol A (DGEBA) and it is purchased from the System Three Inc. Silvertip Laminating Resin is a medium-low viscosity, liquid epoxy resin system that has been optimized for coating and reinforcing fabric. It has superior wet-out characteristics with little tendency to foam or trap air. Both the resin and hardener are nearly colorless and are used in an easy 2:1 volumetric ratio. Silvertip Laminating Resin cures to a brilliant blush-free film with either the fast or slow hardener eliminating secondary bonding concerns as experienced with other epoxies. The two-part A and part B are mixed in the volume of 2:1.epoxy is highly branched aliphatic polyester backbone with average of 11 epoxy groups per molecule.

The clay used for synthesis of nanocomposite was commercially treated clay manufactured by Nanocor, which contains Montomorillonite. Montomorillonite is classified as the magnesium aluminum silicate, which can be used to make a new class of clay/polymer. Montomorillonite has a sheet type or platy structure. Although their dimensions in length and width directions can be measured in hundred of nanometers, the mineral thickness is only one nanometer. As a result individual sheets have aspect ratios (L/W) varying from 200-1000, with a majority of platelets in the range 200-400 after purification.

The structure and the formula of the montmorilliate is shown in the figure 1.

**$M+y(Al_{2}\text{-}y\ Mg_{y})(Si_{4})\ O_{10}(OH)_{2}*nH_{2}O$**

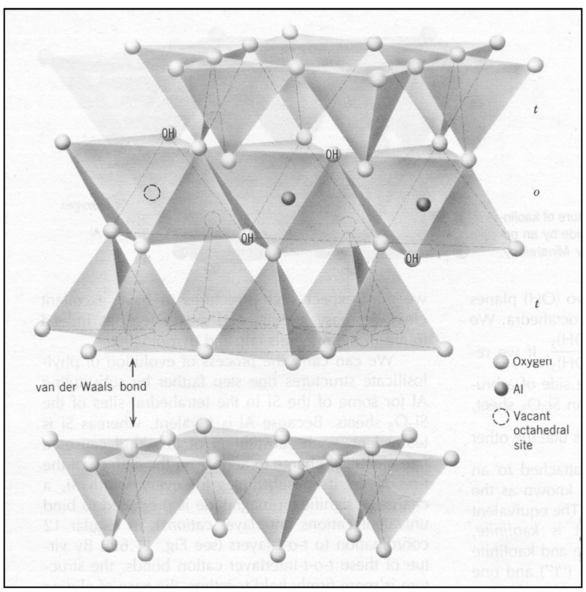


**Figure 1: Formula and structure of Nanoclay**

## 4.2 Equipment used

### *4.2.1 Impact tester*

The monitor impact tester is used to perform the impact test on the samples. The main parts of the machine comprise of the swinging pendulum, vice to clamp the samples. The monitor in whom the parameters like thickness is entered and the measurements are noted.

**Figure 2: monitor impact tester**

### *4.2.2 Flammability test*

To determine the flammability properties of the nanocomposite, the samples are tested on the cone calorimeter. With the aid of this machine valuable quantities like heat release rate, time to ignite and mass loss rate can be calculated. Before testing of any material on the cone calorimeter the calibration of machine is necessary. The cone calorimeter used for the test is manufactured by Fire testing technology, UK.

**Figure 3: cone calorimeter**

### *4.2.3 Tensile test*

The instron universal testing machine is used to perform the tensile test on the samples. The model of the machine used is INSTRON 4467 and the serial number is C1115.

**Figure 4: Instron universal testing machine**

## 4.3 Procedure

### *4.3.1 Preparation of the epoxy-nanocomposite*

The first step in the preparation of the composite is to dry the nanoclay in oven at 80 degrees for 24 hours. After drying the clay, the part A of the epoxy is heated in the oven for 30 min at 65˚C so that the viscosity of the part A reduces and the clay can be dispersed more easily. After removing the resin from the oven, nanoclays in the desired percentage (2%, 4%, 6%) are mixed in the epoxy part A by mechanical mixing for 24 hours using a mechanical drill head stirrer.

The mixture can also be sonicated for 30 min at 65°C. The amplitude is set at 35% and the energy supplied was set to 6% using the ultrasonic sonicator. A temperature probe (which is provided with the sonicator) is inserted in the mixture to know the temperature of the mixture. A sonication result in increase in temperature of the mixture, the maximum temperature limit is set to 50°C, once the temperature rise to the limit the sonication process stops. To speed up the sonication process the mixture is placed in a water + ice beaker so the heat dissipates into the surroundings at the faster rate. Then the mixture is placed in the vacuum oven and degassed it for the 2 hours at 70° (depend upon the content of clay). The degassing is required so that the bubbles trapped inside the mixture come to the surface and escape. Mixture is then placed at the room temperature to cool down and part B (hardener) is added to the mixture in amount to 44% in weight (50% in volume) of the part A. The part B is vigorously mixed for 2-3 minutes, taking care that bubbles are not formed, and the mixture is poured in the aluminum molds. Before pouring the mixture, the mold must be clean and a mold release must be applied. The mixture is left curing for 24 hours at room temperature. After 24 hours the samples are removed from the molds and placed in the oven for post curing at 100 degrees for 2 hours. The samples are then cooled down at room temperature, before testing them.

### *4.3.2 Procedure for impact testing*

To determine the impact strength of the composites a monitor impact-testing machine is used. Monitor impact tester readily determines the impact energy required to break plastic, ceramic and light metal samples. We have used the Izod impact test to test the impact strength of the samples by using the ASTM D 256-06 standards. During an Izod impact test, the specimen is held as a vertical cantilever beam. It is broken by a single swing of the pendulum. The line of initial contact is a fixed distance from the specimen clamp as well as from the centre line of the notch and is on the same face as the notch. The Izod impact specimen holder is mounted in the centre of the base of the impact tester. It is secured by two Allen cap screws. The weight of the pendulum used is 5 lbs. According to the ASTM standards the units for the tests are selected as foot-pound per inch (FTLB/IN). When ISO standard are selected the units must be changed to kilojoules per square meter (KJ/SQM).

**Figure 5 : cutting machine**

The samples for the Izod impact test shall conform to the dimensions and geometry. The thickness of the sample plays an important role, the length of the specimen must be 18 in and the thickness is between .3 to .35 in. the preparation of the notch in the izod specimen serves to concentrate the stress, and direct fracture to the part of the specimen behind the notch. Notching can be done on a milling machine, lathe or any other cutting tools. The notch angle must be 22 ½ ° in angle.

To test the specimen on the impact tester, the specimen is notched on the cutting machine. The sample is placed vertically in the clamp. Make sure the notch is placed in the middle. Press the test button on the monitor impact machine and release the pendulum. There can be four types of breaks:

1. Complete break.
2. Partial break.
3. Hinge break.
4. No break.

The results of all tests are reported in terms of energy absorbed per unit of specimen width.

### *4.3.3 Flammability test procedure and calibration*

Characterization of the flammability properties of a variety of polymer clay nanocomposite, under fire like conditions, using cone calorimeter has revealed the significant results of the effect of clay on the epoxy. The cone calorimeter measures fire relevant properties such as heat release rate (HRR), Time to ignite, Time to flame out, Heat distortion temperature, mass loss rate and carbon monoxide and carbon dioxide production during the burning of the materials. Heat release rate, in particular peak HRR has been found the most evaluating parameter to evaluate fire safety. The cone calorimeter used for the test is of Fire testing technology cone calorimeter with Xentra gas analyzer (for oxygen only) and laser smoke system.

The calibration of the cone calorimeter is required every time before performing the test. This includes several steps, as follows:

The steps below are based on the assumption that the cone calorimeter has been shut down in the prescribed manner the previous day and that no problems were noted in any of the equipment. If the unexpected does occur, some trouble shooting and corrective measures will be necessary.

To start with the calibration, the first step is to open the cold tap to drain the water, which is accumulated in the machine due to the condensation. Turn the system ON; turn ON the Data logger (data acquistation AGILINT). Turn the computer ON and check that the power to all of the following is ON:

1. Analyzer must be ON.

2. Laser (smoke) power must be ON.
3. Cold trap refrigerator must be on main power must be ON.
4. Cone power must be ON and check the temperature in the lower part shows zero.
5. Load cell must be ON.

*Overall main power should remain ON permanently under normal conditions.*

After switching ON all the above check for the filters and drying agents. The drying agents used are Drierite and Ascarite. These agents are used for drying the air so that there is no moisture present in the air. Also check for the primary balston filter and secondary hepa vent are clean, if necessary change it this will give us the flow rate of 3.5 l/min. After this set the duct flow, adjust the speed on exhaust control to set the volume flow rate to 24 l/s (approximately 30 g/s at room temperature). The duct flow exhausts the gases in to the atmosphere. Smoke calibration is done by inserting the blank card between the laser and the compensating photo diode and check on the computer screen that it shows zero. Once the smoke system is calibrate, then we need to calibrate the gas analyzers for this turn on the sample pump and check for the leaks and check the flow should be 3.5 l/min. calibration of the gas analyzer involve checking the zero, using nitrogen, then setting the valve at that for dry ambient air, namely 20.950% for the oxygen analyzer. The most important and critical calibration is of the C factor, it is calculated by placing the burner on the load cell and turn the methane on, adjust the methane flow to obtain approximately 5 KW. The acceptable range for the instrument is .04-.045. Measurements on two successive test days shall not differ by more than .002.such difference indicates the malfunction, which requires rectification before testing is continued. Before testing a set of materials check that the scale of the load cell output is appropriate for the masses of the specimen that will be tested. To set the load cell output, firstly determine the maximum mass, M max of the samples and choose the range slightly higher than the largest mass. For example, if your sample weighs 23 g, select 50 g as a full scale load. This value has to be entered in the new port controller (note that it is very important that the mass does NOT exceed the M max).

In the end set the heat flux, the setting of heat flux is required to set the amount of heat flux ensure that the cold water to heat flux meter is flowing. Set the heat flux meter below the cone make sure that it is 25 mm from the cone and then set the temperature controller to give approximately the required heat flux (770 degree Celsius for 50 w/m2). When the temperature get stabilized look at the heat flux reading in the computer (irradiance). When stable remove the flux meter and check that the copper end of the heat flux meter is cold and then turn the water flow off.

Before performing the test check the system one more time, when the machine is ready for the test, place the sample on the sample holder on the load cell and open the shutters and press the start key when ready.

The basic principle of cone calorimeter is to burn the specimen by radiating heat in it without any flame, just by the spark. The sample is placed on the load cell and just above the cell the cone is their whom temperature is set approximately to 770°c. Due to this high temperature of the cone when the sample is exposed to the cone the heat from the cone is radiated in to the sample and the temperature of the sample increase so much that it starts burning only by giving the spark. Once the sample get ignited spark turn off and the time to ignite and time to flame out is noted.

### *4.3.4 Procedure for tensile test*

Tensile test is done on the specimen to know the resistance of the specimen before it fails. Tensile strength measures the force required to pull something such as rope, wire, or a structural beam to the point where it breaks. Specifically, the tensile strength of a material is the maximum amount of tensile stress that it can be subjected before failure. Tensile test (ASTM D-638) are performed at room temperature and 50% mean relative humidity using INSTRON -4467 tensile testing machine with a crosshead speed of 10 mm / min. the tensile samples are of dog bone shaped and is clamped to the sample holder vertically. The test is performed to calculate the break extension, peak extension, peak stress and the Young modulus. The break extension can be measured as strain at break (mm/mm). The peak extension is the highest point of the extension before the sample breaks. The young modulus is the very important factor and it is measured in mega Pascal (Mpa) Young's Modulus (also known as the *Young Modulus*, modulus of elasticity, elastic modulus or tensile modulus) is a measure of the stiffness of a given material. It is defined as the ratio, for small strains, of the rate of change of stress with strain. This can be experimentally determined from the slope of a stress-strain curve created during tensile tests conducted on a sample of the material. It can also be calculated on the basis of the graph and it is the difference between peak load and the minimum load divided by the difference between the highest and the lowest extension.

## *5.* Results

Addition of the nanoclay in the epoxy resin affects the impact properties and the flammability properties of the polymers. All the results are made under the 90% error confidence. Confidence intervals are random regions that contain a Statistic with some probability. For example, “correlation is between 0.85 and 0.98 with
90% probability”. The margin of error expresses the amount of the random variation underlying a survey's results. This can be thought of as a measure of the variation one would see in reported percentages if the same poll were taken multiple times. The larger the margin of error, the less is the confidence. Assume that the sample distribution for our stastic is normal. The following graph shows the error confidence for 90%.[5]. The 90% error confidence is taken in the consideration to plot the results of the tests. To calculate the error we need to multiply the standard deviation by 1.65.

$$E = 1.65 * \sigma$$

Where: $E$= 90 % error confidence

$\sigma$ = standard deviation

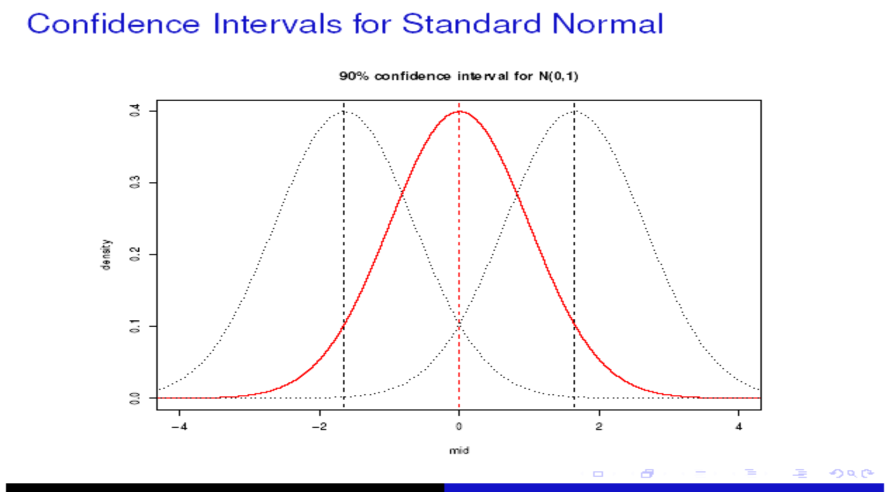


**Figure 6: error graph for 90% confidence**

The orange and black color bars indicate the results of post-cured samples at 100° C and samples without post curing respectively.

## 5.1 Impact test

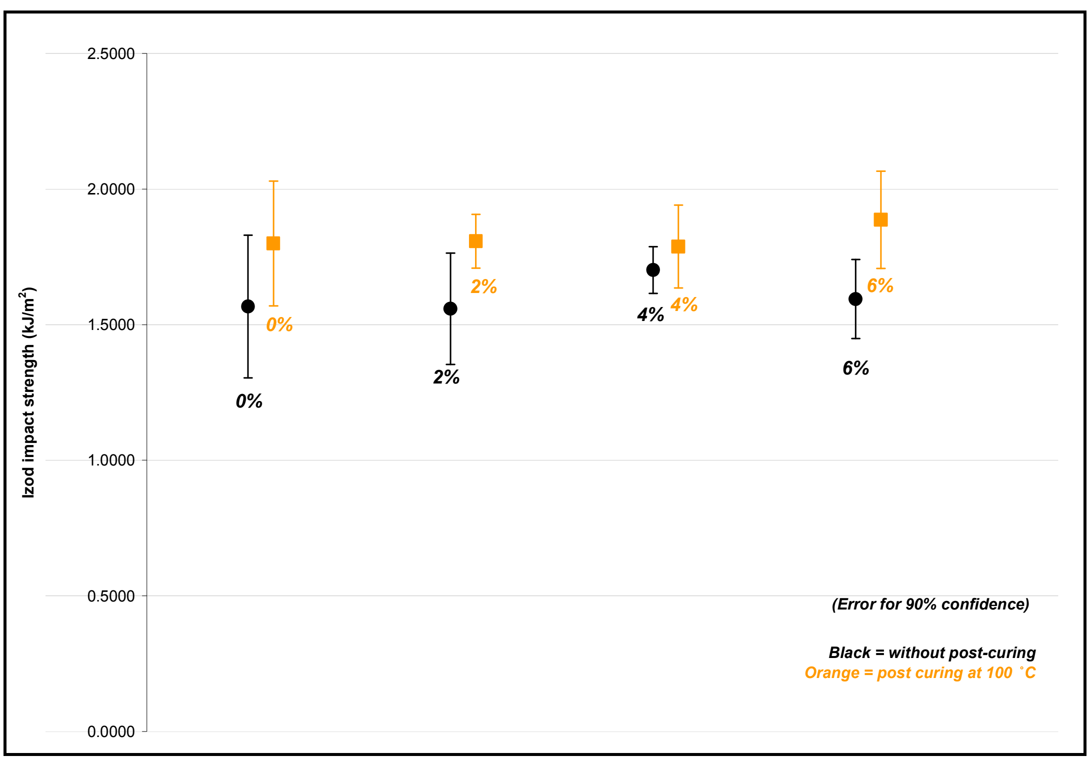


**Figure 7: Izod impact fracture toughness of nanoclay reinforced epoxy nanocomposite**

## 5.2 Flammability test

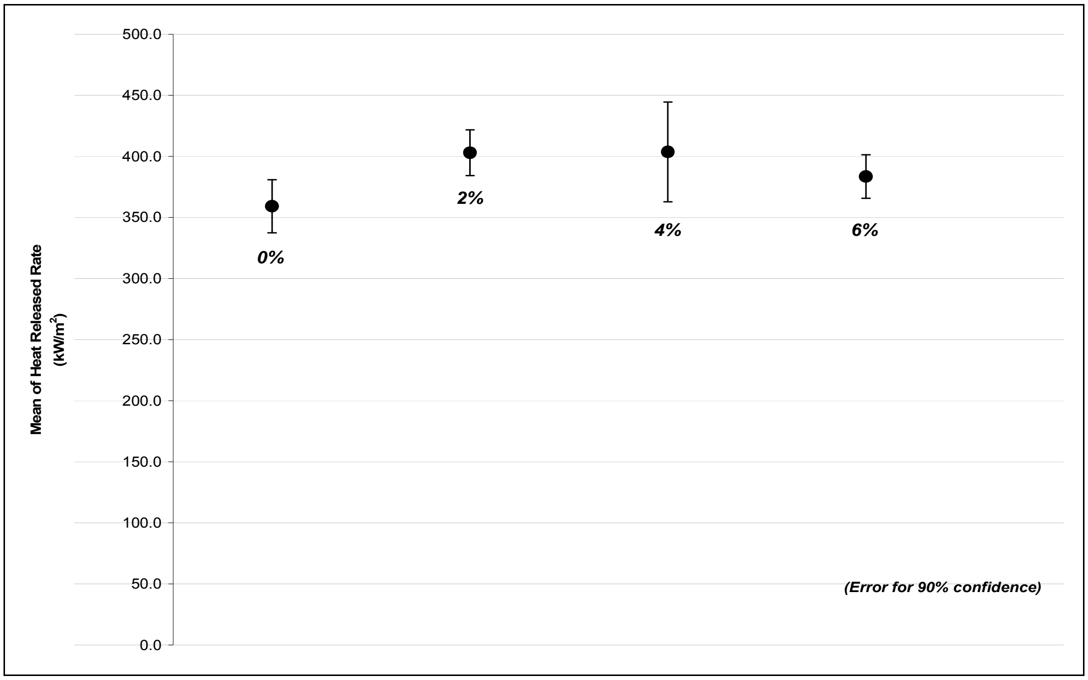


**Figure 8: mean of heat release rate**

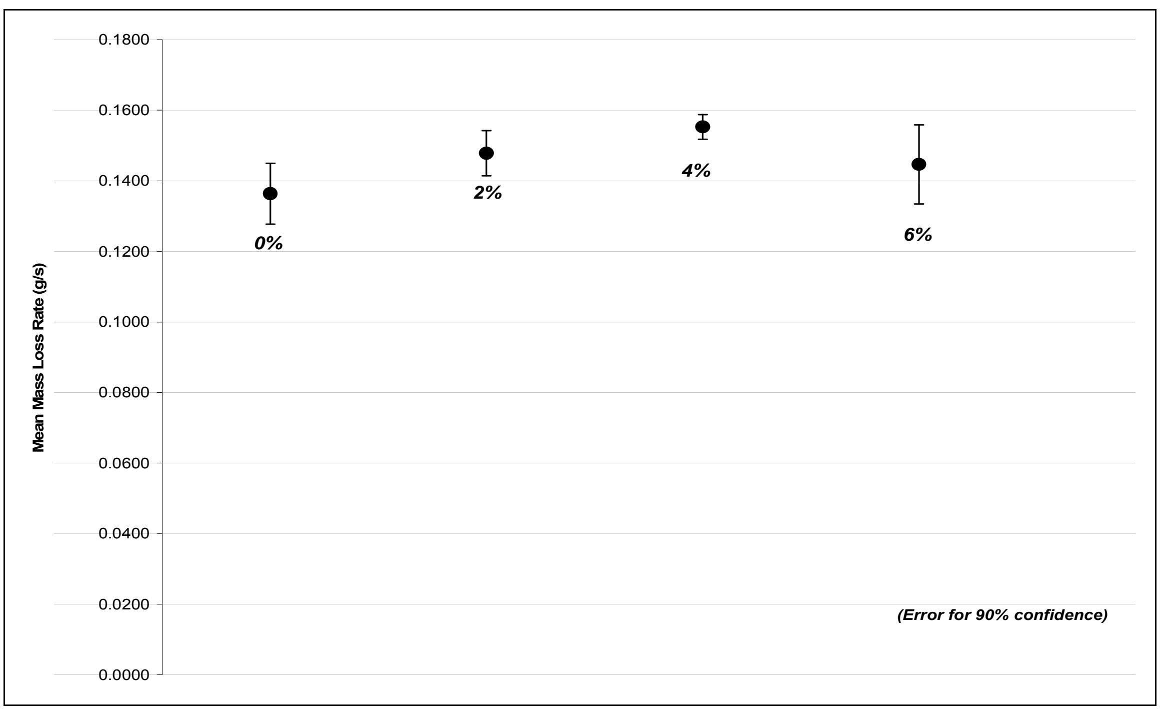


**Figure 9: mean mass loss rate (g/s)**

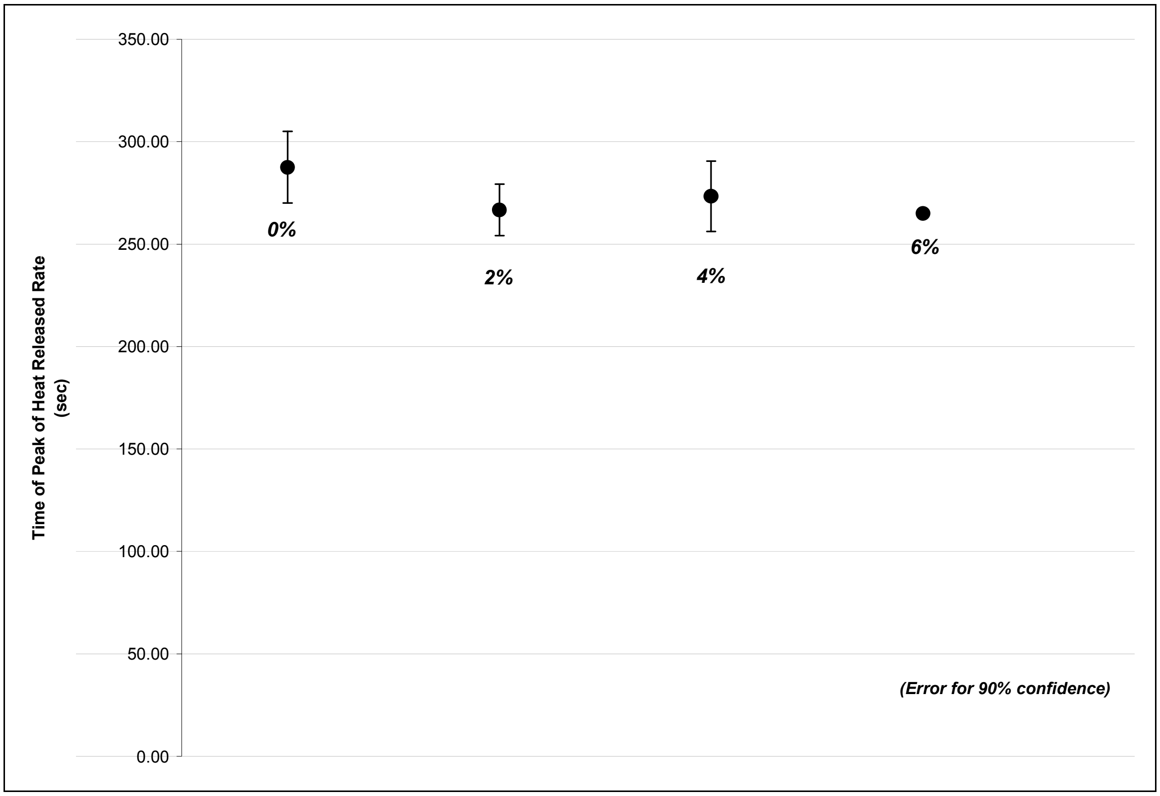


**Figure 10: time of peak heat release rate (sec)**

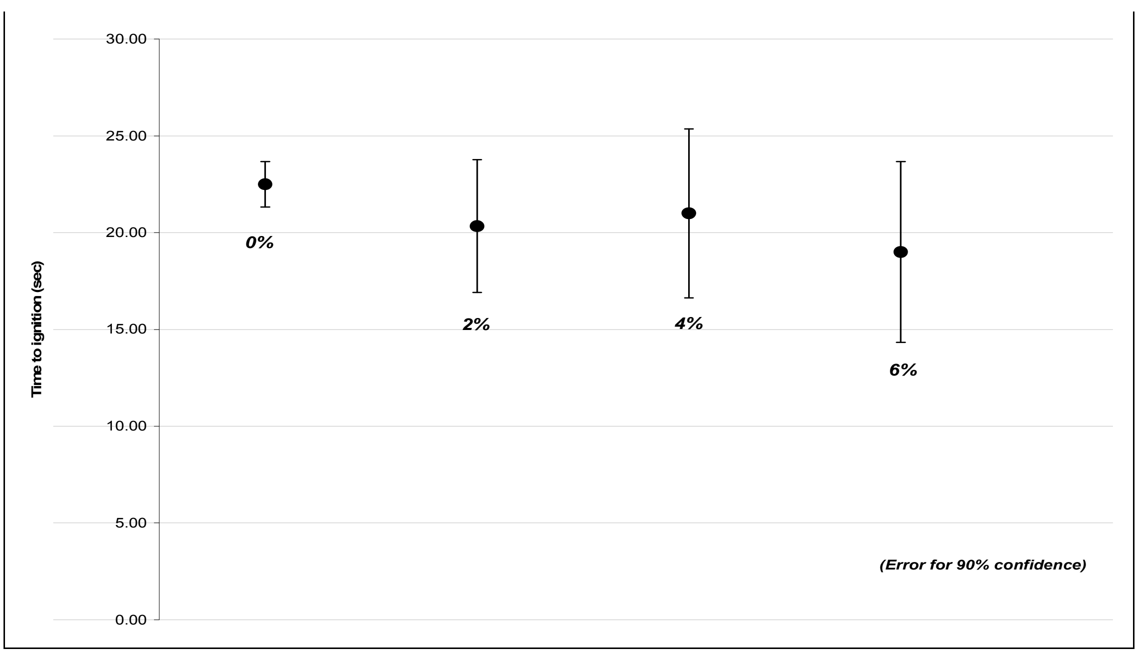


**Figure 11: time to ignition of various percentages of nanoclay**

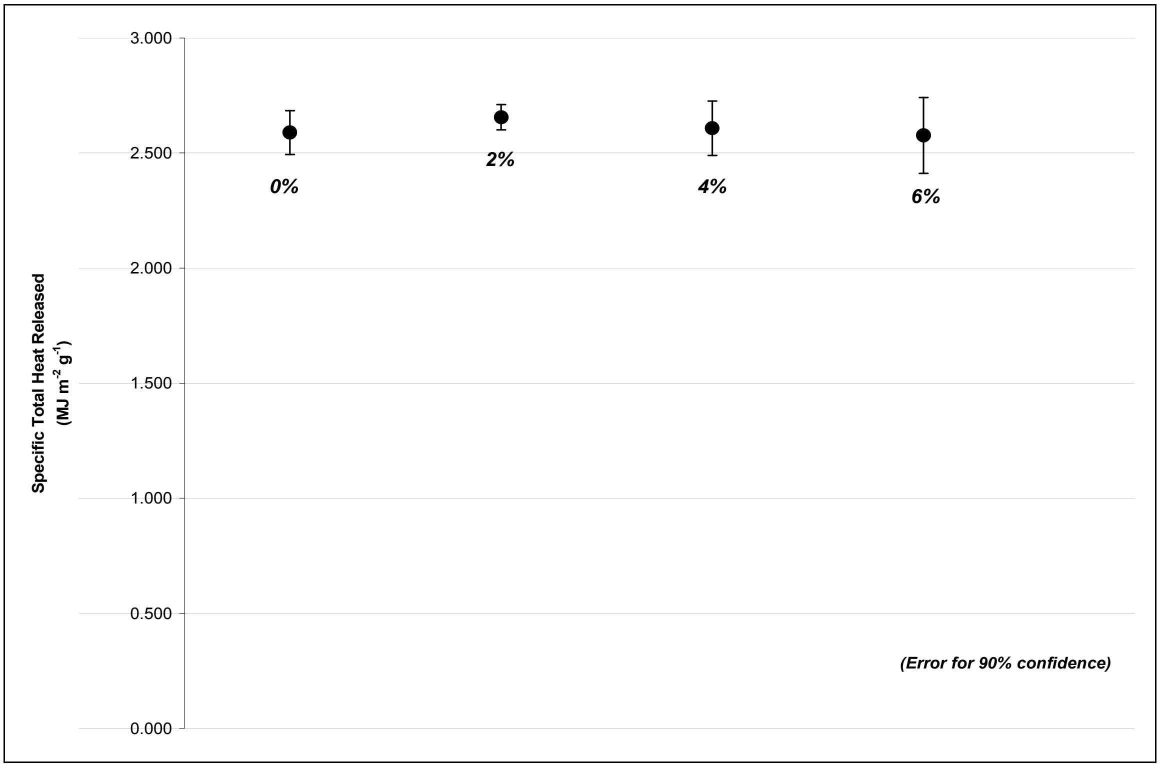


**Figure 12: specific total heat release (MJ m2/g)**

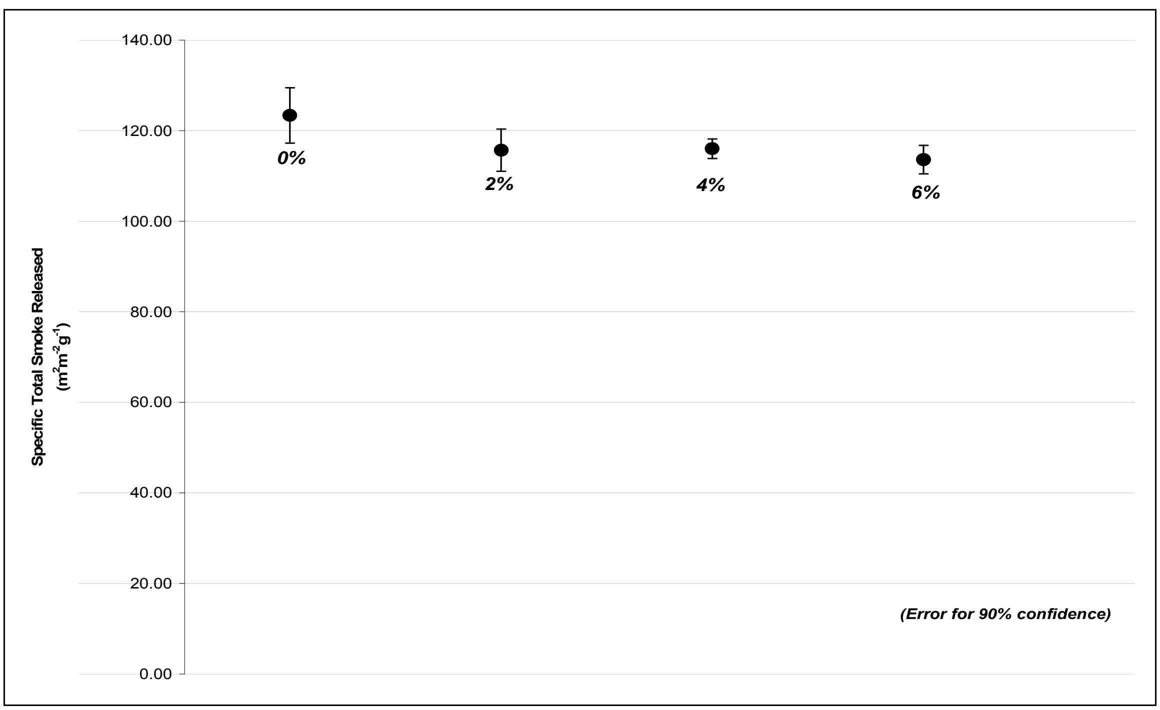

**Figure 13: specific total smoke release rate**

## 5.3 Tensile tests

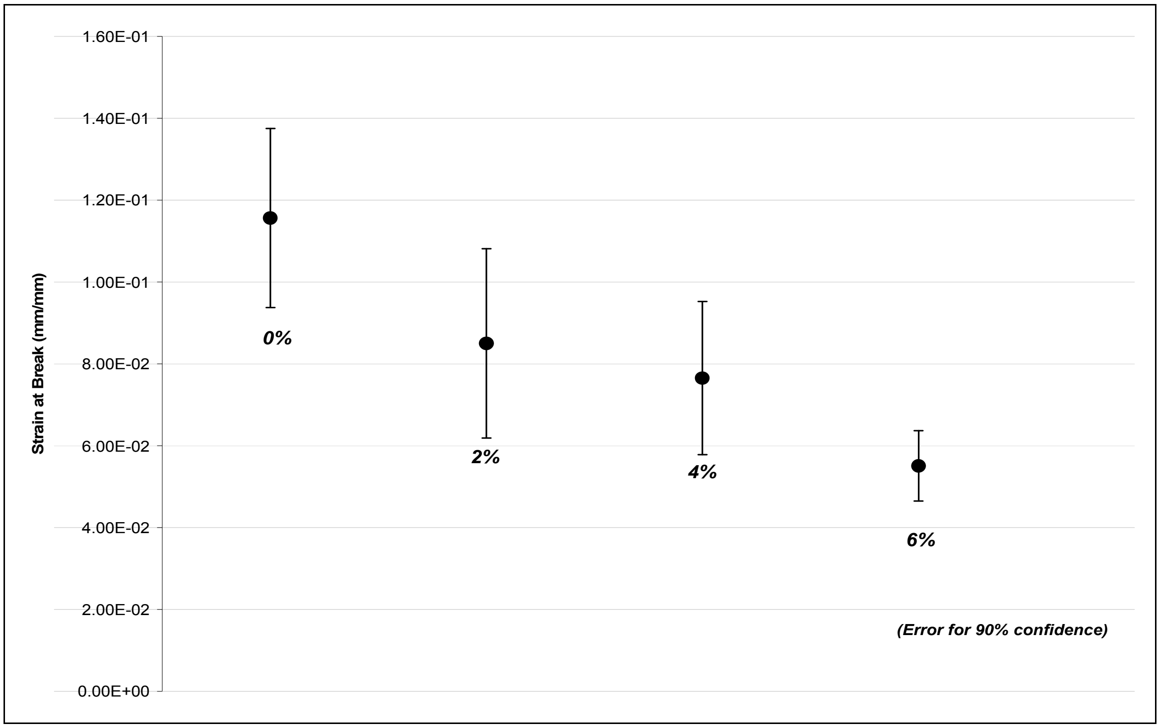


**Figure 14: strain at break for tensile toughness of nanocomposite**

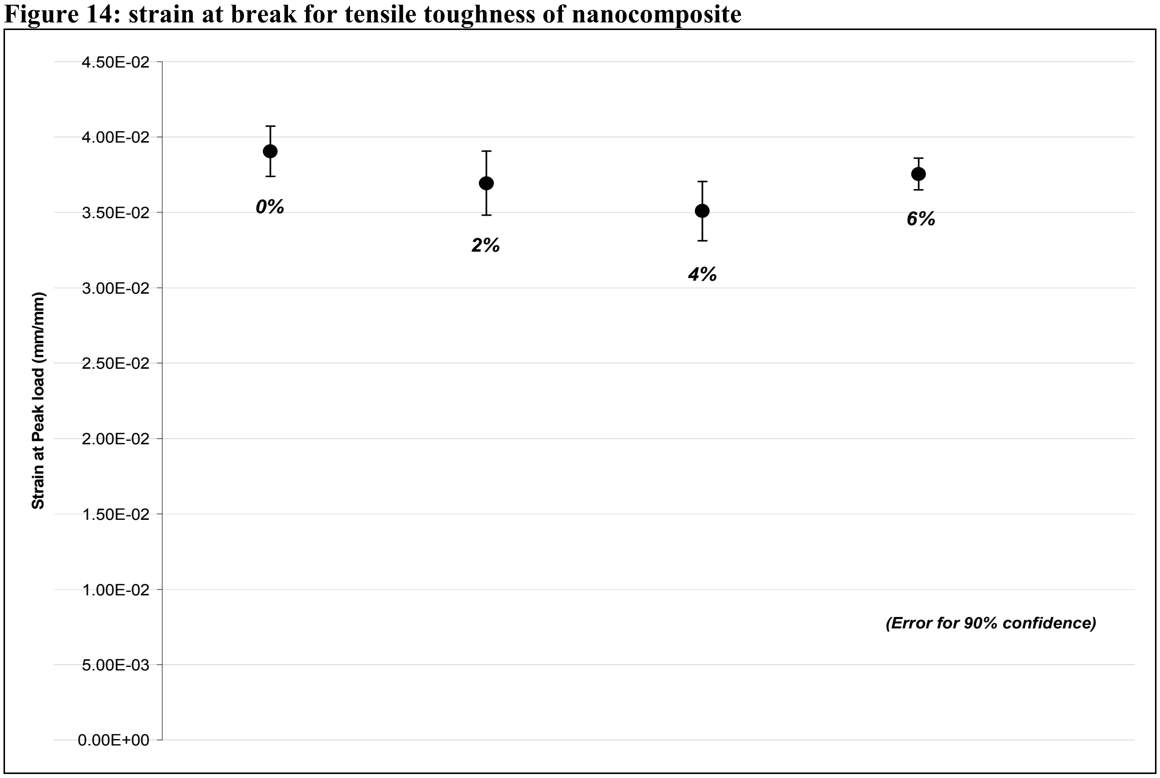


**Figure 15: strain at peak load (mm/mm)**

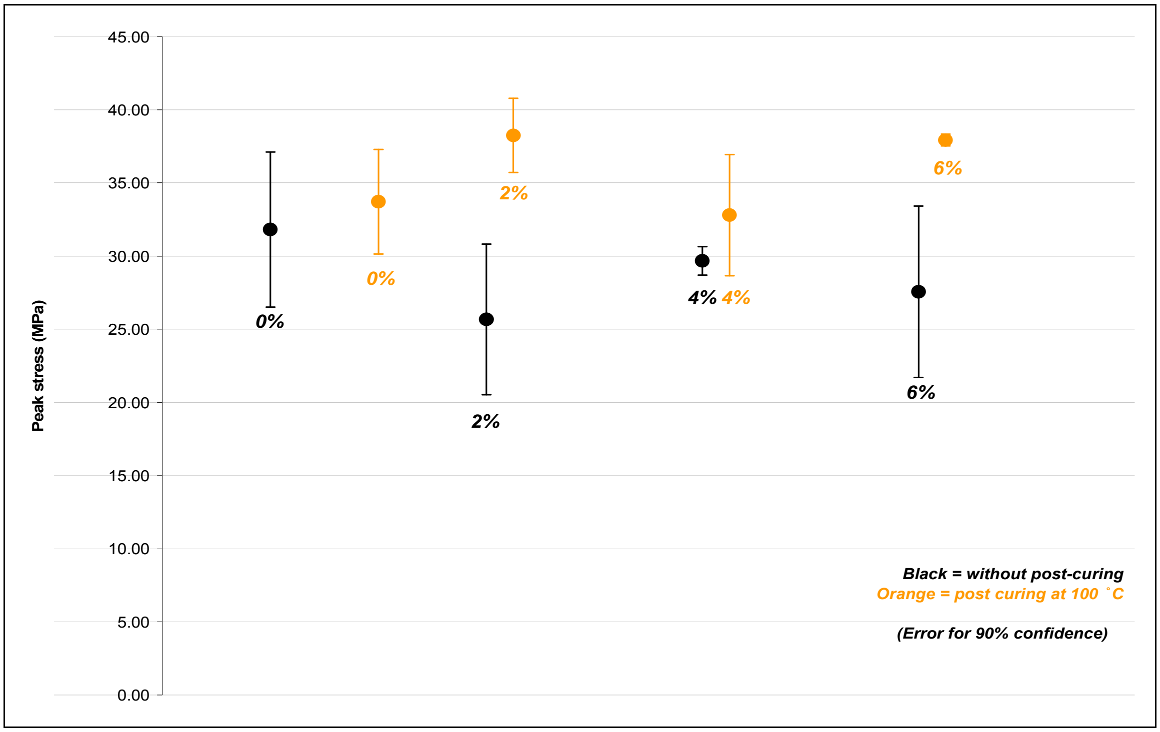


**Figure 16: peak stress (Mpa)**

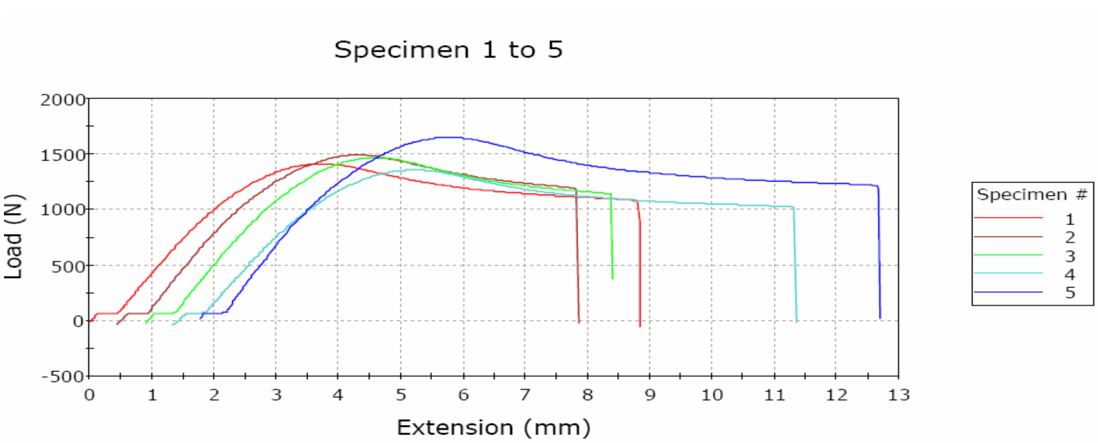


**Figure 17: 4% wt nanoclay**

# 6. Discussion of the results

## 6.1 Impact tests

After the testing of the samples on the monitor impact tester, the Izod impact strength of the material is calculated in $kJ/m^2$. The results are plotted in figure 7. The *x*-axis data are labeled for 2%, 4% and 6%. The y-axis shows the impact strength of the materials. The results shown in orange points are representative of post cured samples at 100° C, while the black points represent samples that are not post cured. A minimum of 10 samples is tested and the readings are taken. The average, standard deviation and the 90% error confidence are calculated.

From figure 7, we observe that addition of the nanoclay in the epoxy resin does not drastically affect the impact strength of the materials. However there is small increase in the 4% for not post cured samples. 4% always shows the better results due to the proper amount of clay content. The uniform distribution of the clay leads to exfoliation structure. The most important is the effect of post curing. After post curing the impact strength for all samples in all percentages increases. For 0% for example, the increase is from 1.60 to 1.80 $kJ/m^2$. Due to post curing the impact strength of the 6% also increases significantly. By studying the above graph we came to the conclusion that the nanoclay in the epoxy resin does not affect the impact properties, however the post curing process has positive effects on the properties.

## 6.2 Flammability results

The dispersion of the clay in the epoxy resin affects the overall flammability properties. There are three different types of structures noticed due to the dispersion of the clay in the materials. The first is the exfoliation dispersion of the clay in which the clay particles are evenly distributed all over the epoxy resin. The second structure we come across is the intercalated. In this there is uneven distribution of the clay and due to non-uniform distribution the clay the small groups formed in the epoxy resin and the curing is done only in some parts. Also, sometimes the nanoclay particles agglomerate in the epoxy resin, which leads to the formation of large groups. This is an undesired affect because it causes a negative effect in the material properties.

In figure 8, we can observe that the mean of heat release rate is higher for nanoclay-epoxy composites than for pure epoxy. This is due to the fact that nanoclay particles in this grade of epoxy work as heat conductor. As a result heat is transferred faster through the thickness and the material burn faster. We can also observe that there is a maximum for percentage of clay near 4%, which corresponds to the amount of clay that better link with epoxy, as evaluated from other tests performed on the same samples. Anyway the same clay might behave differently, according to the resin used. We can argue that type of resin and processing conditions produce different microstructures and bonds, which affect the properties of the overall composite produced.

From the graph in figure 9, we see that there is an increase in the mean mass loss rate due to the addition of the nanoclay in the epoxy resin. The mean mass loss rate is calculated in g/sec. The Mass Loss Rate behaves like the Heat Released Rate, showing a maximum for percentage of nanoclay close to 4%. Nanocomposites with 4% of clay burn faster, due to better conduction of heat through the thickness.

In the figure 10, we concluded that the time of peak of heat release rate is higher for 0% and for rest of the percentages it's same. It is the time to reach a peak value of heat release rate. Due to the presence of the nanoclay particles the heat dissipated to the atmosphere reduces and hence the peak reduces and the material become less flammable. As we also see the results for 6% has no errors.

As we can see from the graph 11, there is not much change in the time to ignition the material by adding the nanoclay in the epoxy resin. The results of the time to ignition has very large errors, This is due to the human errors while pressing the ignition button on the machine during the test. Every time while performing the test, the person has to press the ignition button and if in case there is a delay its going to affect the readings. As the result we cannot rely on this graph and some other results are taken in to consideration. However there is not large change in the time to ignite for the various percentages of nanoclay.

The total specific heat release rate is measured in $MJ/m^2/g^{-1}$. As from the graph 12, we come to know that there is not much effect of the nanoclay on the specific total heat release rate. The case for Specific Total Heat Released is different.

The changes between different percentage of clay and pure epoxy are minimal. Nanoclay seems not to affect the total heat released.

The Specific Total Smoke Released instead is lower for the nanocomposite with clay than for pure epoxy. For the good material properties the smoke produced by burning the material should be less. We have observed that due to addition of clay the smoke producing characteristic of the nanocomposite reduces.

## 6.3 Tensile results

To determine the tensile strength the strain at break, strain at peak and the load at peak is calculated. After observing the tensile curves which is plotted between the extension on x-axis and load on y – axis. The approximate thickness of the sample is between 3.5 – 4.0 mm and width is 14mm. In the figure 16, the four samples of 0% nanoclay are tested. It is observed that the peak load is approximately around 1650 N, the post cured samples produce better results then without post cures ones. The samples with very low peak load curve are discarded. For the 2% nanoclay there is the significant increase in the peak load. The peak load is increased to approx. 1900 N for addition of 2 % clay. We also concluded that strain at break reduces as the percentage of the nanoclay increases (refer figure 14). The 0% has the maximum strain at break and it decrease linearly. In figure 15, there is not much affect on strain at peak load, due to increase in percentage of nanoclay. For 0% it is the highest and then it decreases for 2 % and 4%, but there is a small increase for 6 %. The reason for this increase is unknown. The peak stress for post-cured samples provides improved properties then the uncured samples. The post curing results in increase in peak stress for 2% as compared to 0%. There is the also the significant increase in the peak stress for 2% post cured samples as compared to the without post cure samples. However the peak stress is same for 2% and 6%.

## 7. Conclusions

The addition of nanoclay in to the epoxy matrix by mechanical stirring method affects the various mechanical properties such as impact and tensile. The change in flammability properties is also observed. It is found that not all the results we recorded have positive effects on the material. For the flammability test we did not achieve good results, as the data we got from the flammability test is not consistent, it varies from sample to sample, maybe due to the change in the samples manufacturing methods along the way. For the impact tests we come to know that the nanoclay makes the material more brittle. Any way we are able to improve the resistance of our materials by adopting the new method, which we refined during the preparation of the samples. The method by which we made the last samples of nanocomposites shows better properties as compared to the initial results.

## 8. Recommendations

We test few samples produced using a sonicator in the mixing process, and we found better results. Mixing by ultrasonic waves produces a better dispersion of clay into the epoxy resin, than the mechanical stirring. The results achieved with sonication are better in terms of mechanical and flammability properties. It is advisable consider sonication in the next future. Also it will be interesting to compare the effect of nanoclay into the epoxy with the effect of other nanoparticles, such as carbon nanofibers, and carbon nanotubes.

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