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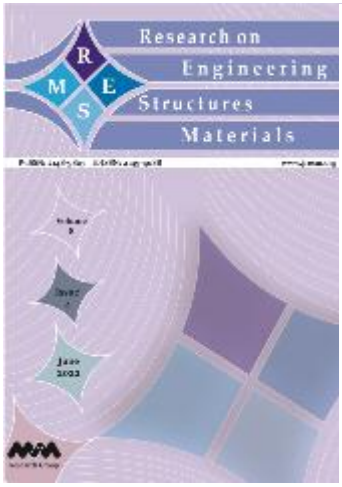

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Research Article

Study of morphology and effect of compression moulding parameters on mechanical properties of nanoclay/polymer nanocomposites sheet moulding compound

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Abstract

Owing to the much larger surface (or interface) area per unit volume, drastic difference in material behaviour can be observed between the conventional and nanostructured materials. A nanostructured material can have totally different properties from a larger-dimension material of the same composition because many of the important chemical and physical interactions are governed by the surfaces. This study considers polymer nanocomposites in the form of sheet moulding compounds. The polymer nanocomposite is formed by adding surface modified Montmorillonite nano-clay as a major nano-material to the SMC composite material so as to enhance the material properties. This study considers fabrication and characterization of polymer nanocomposite by employing the compression moulding technique. Scanning electron microscopy has revealed that though the polymer nanocomposites improve the material properties but its fabrication should be carefully carried out in order to avoid formation of irregularities in the material. The x-ray diffraction results confirmed the retention of crystallinity of the polymer nanocomposites. Upon characterization it has been observed that the material properties like tensile strength, percentage elongation and flexural strength are enhanced. The hardness of the polymer nanocomposite material is slightly improved and the specific gravity is also found to be slightly decreasing making the nano-clay composite lighter.

1. Introduction

1.1 Sheet Moulding Compound (SMC)

The SMC are fiber-reinforced thermosetting semi-finished products, preferably fabricated using compression moulding method [1]. Sheet moulding compounds composites are high-strength, cost-effective composites, with the combination of fillers, fiber reinforcement, thermosetting resin [2], and other additives for special aims that are manufactured by thermo-compression process. The clay (improved surface), calcium carbonate (reduced cost), talc (improved temperature resistance), hollow glass microspheres (weight reduction, thermal insulation), alumina trihydrate (fire retardance), and mica (improved weathering), are the common material used as fillers. The unsaturated polyester (UP), vinyl ester (VE), phenolic or a modified vinyl urethane are mostly preferred for Thermosetting resin [3]. SMC has the major advantage of being able to be manufactured in medium to high volumes [4] but also has many other advantages offered by composites such as light weight, lower tooling costs, and reduced costs by parts integration. The electrical engineering components, construction industry and automotive sector main sections of applications for SMC components. The cabinets, switches and insulating units

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needed in the electrical sector, are produced from SMC. The components like coverings of mudguards, trunk lids, loading areas, and fenders needed for the automotive industry, are made of SMC [5]. SMCs are commonly used in automotive industry for its excellent chemical resistance, long-life service, and most importantly, the light-weight of the structural components [6].

1.2. Polymer Nanocomposites

Owing to the much larger surface (or interface) area per unit volume, drastic difference in material behaviour can be observed between the conventional and nanostructured materials. A nanostructured material can have totally different properties from a larger-dimension material of the same composition because many of the important chemical and physical interactions are governed by the surfaces [7, 8]. For the fiber and layered material, a change in particle diameter, layer thickness, or fibrous material diameter from the micrometer to nanometer range, will affect the surface area-to-volume ratio by three orders of magnitude [9]. So, the larger surface area per unit volume can be observed for the naocomposites that uses very small sizes (in nanometer) of reinforcement material. Nanofibers or nanotubes (1-dimensional) and nanofoils (2-dimensional) [10] facilitates for the high aspect ratios of the particulate constituent which results in good reinforcing efficiency. By adding just enough amount of a low-volume fraction of fillers, the desired performance can be achieved for the polymer nanocomposites and these fillers usually contributes only marginally to the final weight and cost [11].

1.3 Effect of Nanomaterials on Mechanical Properties

The performance of nanocomposites depends on various characteristics of the nanoparticles, such as its size, aspect ratio, specific surface area, and physical/chemical compatibility with the matrix and also on the properties of constituents, composition [12]. Enhanced flexural strength enhances resistance offered to bending and shear loads of a composite material. It has been found by Xu and Hoa [13] that adding a low weight percentage of nano clay to the fiber/epoxy composites improved its flexural strength by 38%. Another study by Shettar et al. has [14] considered the study of tensile and flexural strength of epoxy/nano clay nanocomposites with varying amount of nano clay (1%, 2%, 3%, 4% and 5% wt.) and found that the resistance of the material to tension and bending has been increased after reinforcement of nano clay from 0% to 2% but with reinforcement higher than 3%, the performance got degraded. It has been reported by Pol and Liaghat [15] that using organically modified montmorillonite nano clay (Closite 30B) with 3% wt. (out of 0%, 3% and 5%, 7%, and 10% wt. samples) in glass fiber/epoxy significantly increases the tensile strength and impact strength that decreases upon increasing amount of reinforcement of nano clay beyond 3%. Burmistr et al. [16] investigated the variation of tensile strength of nanocomposites formed by using organo-modified clay as a reinforcement material in the base material of polypropylene, polystyrene, and polyamide, and found that the tensile strength and tensile modulus increases up to certain amount of reinforcement after which the tensile strength decreases. A study by Quigley and Baird [17] showed that adding organic modified nano clay up to 7.6 % wt. (out of 0.9, 3.2, 4.9, 7.6, and 9.2 % wt.) in to nylon 6 using supercritical carbon dioxide (scCO2) as a processing aid, has significantly improved the tensile modulus of the nanocomposite as compared to the base material. Chan et al. [18] considered the study of tensile strength of epoxy/nano clay nanocomposites with varying amount of organomodified montmorillonite nano clay (1%, 3%, 4%, 5%, 7% and 9% wt.) and found that the tensile strength has been increased after reinforcement of nano clay from 0% to 5% but with reinforcement higher than 5%, the performance got degraded.

The abovementioned literature review strongly advocates about using nano-clay as an addition to base material so as to improve several mechanical properties like tensile

strength, tensile modulus, and flexural strength. So, this study will also consider the nano-clay as an important nano-material for improving performance of the material. The readers who are more interested in knowing more about polymer nanocomposites that has higher mechanical properties are encouraged to study articles by Zhade et al. [19] (for tensile strength), Rafiee and Shahzadi [20] (for tensile modulus).

1.4 Necessity of Reducing Weight

One of the major fields of application of polymer composites is automotive industry and it is important to understand the necessity of weight reduction of the components used in the automotive industry. The use of vehicles is increasing day by day resulting in increasing consumption of fossil fuels. This ultimately contributes considerably to the climate change by increasing the amount of greenhouse gases in the environment. Increased use of lightweight materials decreases the energy consumption and hence greenhouse gas emissions [21]. The lightweight components made of glass-fiber reinforced polymers are shown to outperform their steel counterparts over the full life cycle mainly due to the reduced fuel consumption of the vehicle in the use phase [22]. Light weighting has been identified as a cross cutting technology with promising approach to meet the Corporate Average Fuel Economy (CAFÉ) standards, as 10% reduction in the vehicle weight can result in 6–8% increase in fuel efficiency in case of vehicle with conventional internal combustion engine while in case of a battery-electric vehicle fuel efficiency increases by up to 10% [23]. Similar to automotive industry, several other industries will also benefit by reducing the weight of the components.

The current study has major objective of preparing a polymer nanocomposite SMC material that will be beneficial for many applications which will have many desirable properties that are previously mentioned in this section. To achieve the major objective following sub-objectives are decided first one being fabrication of Nano clay composite SMC using compression moulding technique, second one being sample preparation of Nanocomposite SMC in sizes required as per the ASTM or any other standards for particular mechanical tests, and third being studying mechanical properties of polymer Nanocomposite SMC material. After accomplishment of the third sub-objective suggestions regarding usefulness of this newly formed nanocomposite SMC in the different fields of engineering world can be decided.

2. Methodology of Fabricating Polymer Nanocomposite

2.1 Measurement of Constituents of Polymer Nanocomposite

The important part of fabrication of the nanocomposite SMC is the additive nanomaterial that is responsible for enhancing many properties of the material as compared with the properties of the base material. The nanomaterial considered for addition to the base material is nano-clay. The nano-clay used in this study is Montmorillonite clay-based material which has its with surface modified by using dimethyl dialkyl (C14-C18) amine with content of 35-45 % (on mass basis). The nano-clay has bulk density ranging from 200 kg/m^3 to 500 kg/m^3. The nano-clay has average particle size of 13 μm has Beige coloured appearance.

The thermosetting resin, fillers, fibre reinforcement, and other additives required for simplifying the production process of the nanocomposite SMC material. Unsaturated polyester isophthalic resin (Grade-Polypol 1053) is the thermosetting resin material taken into consideration in this investigation. The Polypol 1053 is a condensation polymer prepared by the reaction of polyols (also known as polyhydric alcohols), organic compounds with multiple alcohol or hydroxyl functional groups, with unsaturated dibasic acids. This resin is based on isophthalic acid which is an organic compound with the

formula $C_6H_4(CO_2H)_2$. The filler material is calcium carbonate, and the reinforcing element is chopped roving of E-glass fibres with random orientation and length of 1" (one inch). For ease of fabrication, additional materials including catalysts, mould release agents, and other added elements are also required. In this investigation, organic peroxide is employed as a catalyst and zinc stearates as a mould release agent. In the matrix material, additional additive components like pigment and inhibitors are also incorporated.

It is necessary to know some important characteristics of the materials utilised for fabricating the nanocomposite SMC before actually proceeding for the fabrication and for accomplishing that, some measurements are required. These measurements include of the measurement of viscosity and moisture content of the resin material and of the filler material. For the Brookfield Viscometer is used for the measurement of the viscosity, while the Karl Fischer Titrator is used for the measurement of moisture content.

2.1.1 Viscosity Measurement

The viscosity measurement is carried out by using the Brookfield Viscometer which works on the principle of the rotational viscometry. The amount of torque needed to rotate an item, such as a spindle, in a fluid is gauged and correlated with the viscosity of the fluid. The torque is applied through a calibrated spring to a disc or bob spindle submerged in the fluid, and the amount of torque is correlated with the viscous drag of the test fluid against the spindle [24]. It is possible to compare the Brookfield viscosities of non-Newtonian fluids, obtained under the same test settings (model, spindle, speed, temperature, test time, container, and any other sample preparation techniques that can have an impact on the behaviour of the fluid). Most of the times the trial-and-error methodology is adopted for choosing right spindle and speeds, when creating a new test technique. The effective test procedure produces a percentage torque reading between 5 to 100. The rheological behaviour of the test fluid can be monitored by using the same spindle at various speeds. But the Brookfied viscometer cannot be used for accurate rheometry because assigning a single shear rate is not possible due to the geometry of fluid around a revolving bob or disc spindle in a big container. The Brookfield Viscometer used in this study can be seen in Fig. 1.

Fig. 1 Digital Brookfield Viscometer

The Digital Brookfield Viscometer of DV2T model is used for this study. The number of spindles available are 4 and the number of speeds available are 18, ranging from 0.3 rpm

to 100 rpm. The range of viscosity that can be measured is from 15 to 2000000 mPa.s (mPa.s is Millipascal×seconds).

The use of second number spindle is recommended for the measurement of slurry viscosity. The spindle speed is kept constant at 20 rpm for 30 seconds to take the reading and then the viscosity running test is started. After 30 seconds, the measured value of viscosity is shown by the Brookfield viscometer.

2.1.2 Measurement of Moisture Content

Karl Fischer Titrator is the equipment used for the measurement of moisture content. The equipment, as the name suggests, works on the principle of Karl Fischer titration, which is a traditional titration technique used in chemical research to identify minute amount of water in a sample using coulometric or volumetric titration. In this titration, the amount of water content in a sample is evaluated by measuring the amount of water consumed during a redox reaction of Sulphur dioxide with iodine. In this simple reaction, exactly one molar equivalent of water gets used. The titration is continued by the addition of Iodine to the solution and is stopped when the amount of Iodine in the solution is overly present, which can be determined using potentiometry. The Karl Fischer titration is the go-to method for determining the amount of water because of its specificity, accuracy, and measuring speed.

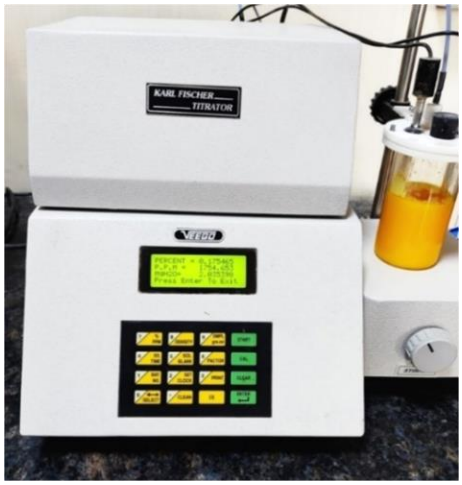


Fig. 2 Karl Fischer Titrator

The Karl Fischer Titrator shows the Karl Fischer factor for the particular sample quantity. After that it sucks the reagent into the glass flask to find out the moisture content in the sample. The Karl Fischer Titrator used in this study can be seen from Fig. 2. The Karl Fischer Titrator uses volumetric method with vessel capacity of 200 ml. The dispensing resolution is 0.01 ml while the measuring range is 100 ppm to 100% with appropriate sample quantity. The titrator has fixed end point confirmation time of 20 seconds.

2.1.3 Measurement of Number of Ends

The filler and thermosetting resin material demand for the measurement of viscosity and the moisture content while only visual inspection is sufficient for the assessment of the reinforcement material. Chopped roving of E-glass fiber is the reinforceing element of the polymer matrix nanocomposite material considered in this study. By doing the visual inspection of glass fiber roving, the number of ends is measured. The chopped roving of E-glass fiber, used in this study can be seen in Fig. 3.

Fig. 3 E-glass fiber Chopped roving of length 1" (inch)

The characteristics of the various materials considered during the fabrication of the polymer matrix nanocomposite material and determined after completing the measurement are found to be as mentioned in

Table 1. Characteristics of raw materials of polymer matrix nanocomposite material **Hata! Başvuru kaynağı bulunamadı.**

After considering the characterization using abovementioned procedure, the constituents of polymer nanocomposite undergo mechanical mixing processing.

2.2 Dispersion of Nanomaterial into Unsaturated Polyester Resin

The mixture of powder and additives is added into the unsaturated polyester resin in order to have proper dispersion of filler material. Then at the operating speed of 700 to 800 rpm, the mixing is carried out for 10 minutes. The nano clay (surface modified Montmorillonite clay) is the nanomaterial used as the principal addition to base material. A high shear mechanical disperser operating at 1250 rpm, is used for 20 minutes to accomplish the dispersion of the nano clay into the unsaturated polyester resin. The dispersion of nano clay in to the unsaturated polyester resin can be seen in Fig. 4.

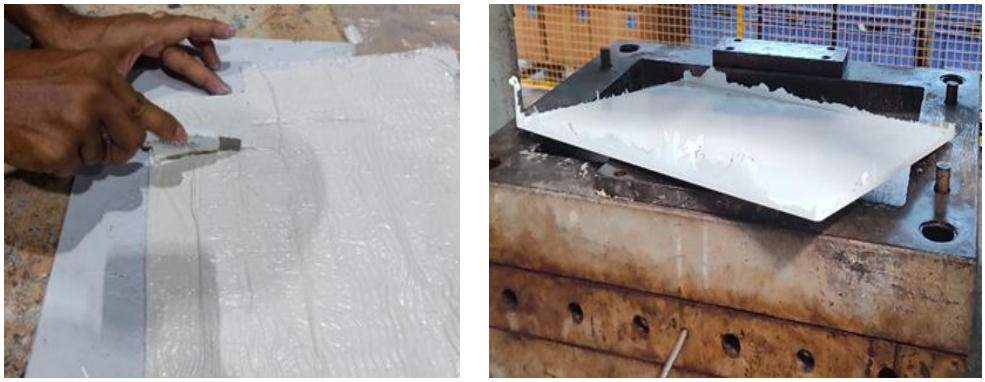
The spreading of matrix material on lower and upper sheet during the formation of SMC of polymer composite material needs to be carefully done so as to have the proper bonding between different materials used for polymer nanocomposite. The wetting characteristics of the material affects the bonding i.e. the highly viscous matrix material will not wet properly which may further result in several manufacturing defects in the polymer nanocomposite material. So, the in-process inspection is very important to monitor the values of viscosity. The viscosity of matrix material is measured by employing Brookfield Viscometer as a part of the in-process inspection during the dispersion process.

Fig. 4 Dispersion of Nano-clay in unsaturated polyester resin

After considering the processing of the constituents, the preparation of polymer nanocomposite SMC is started using compression moulding method.

2.3. Compression Moulding Method

The samples are prepared using compression moulding technique with variation in amount of nano clay, moulding temperature and moulding pressure. There are three stages of compression moulding. The first stage consists of placing the polymer nanocomposite SMC sheet on the lower platen of a hot mould for some time (in seconds) before the mould is closed. Then the sheet is squeezed at a slow closure velocity of (in the range of few millimeters per second) in the second stage [25]. This stage results in considerable deformation of the SMC inside the mould cavity. The mould is kept closed after it gets filled, during a third curing stage for a period of 180 seconds.



(a) (b)

Fig. 5 Sample sheet from compression moulding (a) Charge preparation (b) Moulded plate

The moulding of SMC sheet is carried out using compression moulding machine with capacity of 100 tons. The size of moulded sheet is 350 × 250 × 3.2 mm.

3. Experimentation

3.1 Design of Experiments

For exploring the relationships efficiently and development of greater understanding of the key parameters [26], the design of experiments is very important. The design of experiments also helps in proper planning and execution of the experiments. In this study, the polymer nanocomposite SMC is prepared using compression moulding method by varying different parameters. The number of factors and the levels of those factors affecting the performance of the nanocomposite material (in terms of different properties of materials) are needed to be decided so as to start the procedure of design of experiments. The three important factors affecting the performance, considered in this study are, amount of nano-clay added to base material, moulding pressure and moulding temperature. For these abovementioned three factors two different values of those factors are considered, as mentioned in Table 2.

Table 2. Parameters affecting performance with its amount

Parameter	Amount
Nano-clay (Phr)	0.2 and 0.4
Moulding Temperature (ºC)	150 and 160
Moulding Pressure (kg/cm^2)	120 and 130

The full factorial design consisted of 8 runs with all the possible combination of amount of reinforcement, temperature and pressure applied during compression moulding. The experimentation is carried out as per the design of experiment mentioned in the Table 3.

Table 3. Design of Experiments

Run	Nano clay (Phr)	Temp. (ºC)	Pressure (Kg/cm^2)
1	0.2	150	120
2	0.4	150	120
3	0.2	160	120
4	0.4	160	120
5	0.2	150	130
6	0.4	150	130
7	0.2	160	130
8	0.4	160	130

3.2 Sample Preparation

The samples are prepared as per recommendations for tensile, flexural and hardness test, with variation of different parameters. The samples are prepared from a sheet of the nanocomposite material formed by compression moulding. The geometric layout of the samples needed for different tests can be seen in Fig. 6.

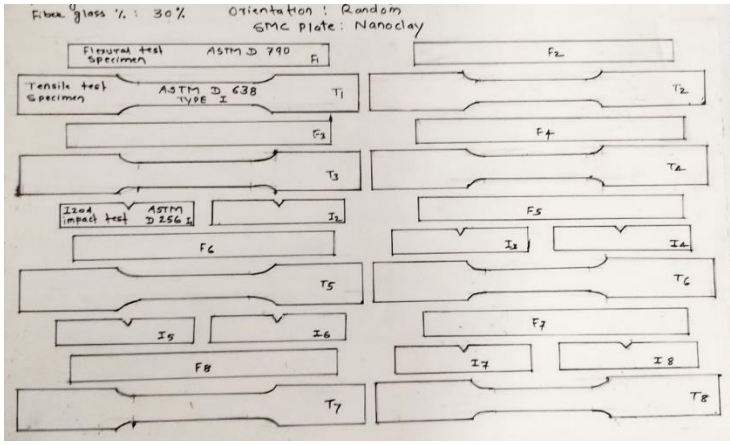


Fig. 6 Layout of the samples from a sheet needed for tensile (T) and flexural (F) tests

4. Characterization of Material Properties

4.1 Morphological Characterization

The Scanning Electron Microscopy (SEM) is one of the major primary diagnostic methods employed for the visualization of the morphology of the surface of the substances like polymers or its composites. In SEM, scanning of the cross-section of the polymer sample is carried out to obtain an image, using a focused electron beam. The atoms and the electrons in the cross-section interact with each other and lead to a variety of detectable signals that reveal details about the surface topography and composition of the sample. The electron beam is typically scanned in a raster scan pattern, and an image is created by fusing the position of the beam with the detected signal [27].

This study is carried out using NOVA NAMOSEM 450 model of Field Emission Scanning Electron Microscope (FE-SEM) that has the Magnification capacity of 20X to 1,000,000X and the images are processed using xT microscope Server. Understanding and locating the irregularities due to agglomeration, void formation and crack formation is the main motive of carrying out the morphological study using SEM.

4.2 X-ray Diffraction Analysis

In order to create polymer nano composites, various nanomaterials in the form of platelets, fibres, and spheroids have been used. Nanomaterials are frequently used in the form of nano clays (mainly layered silicates), nano silica, nanotubes, and nanofibers. The surface chemistry of nanoparticles is frequently further optimized to influence the performance of nanocomposites, resulting in improved nanoparticle dispersion, stronger polymer-particle interactions, and improved polymer adherence to the nanoparticle [28]. X-ray diffraction spectroscopy (XRDS) commands an important position in characterizing and optimizing these interactions.

Utilizing the fundamental tool of X-ray diffraction (XRD), standard studies of the crystallographic materials, including polymers, have been conducted for material characterization. Traditionally, XRD has been used to investigate the crystalline and amorphous structure of polymers, composites, and fillers. The XRD facilitates to the analysis of material using different types of characterizations like phase composition, composition variations, crystallite size and shape, orientation, lattice distortions and faulting, in-situ structure development of nano materials. XRD technique has earned

accolades and has been seen as an attractive tool owing to its simplicity, reliability, non-destructive nature and the quantitative information that can be collected.

4.3 Characterization of Tensile Strength

The tensile strength of the sample is checked using the tensile test carried out using A H25KS model of Universal Testing Machine. For selecting the specimen size and for conducting the tensile test, ASTM D 638 Type I standards for tensile test are followed. The specimen samples size is decided to be 165 × 13 × 3.2 mm. The moulded specimen sample tested at head displacement speed of 2 mm/min.

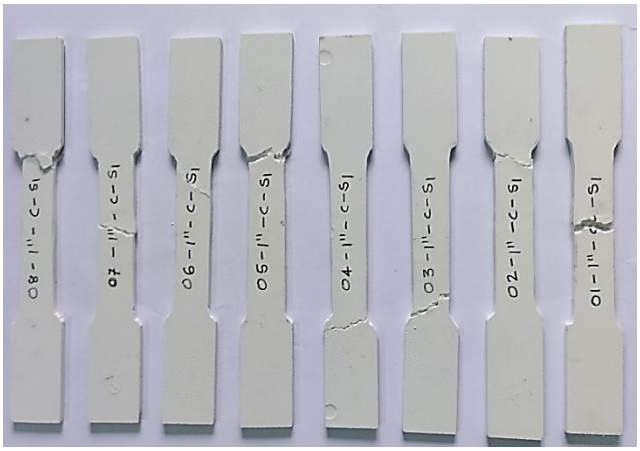


Fig.7 Samples used for the tensile test

4.4 Characterization of Flexural Strength

The flexural strength of the sample is checked using Universal Testing Machine. For selecting the specimen size and for conducting the tensile test, ASTM D 790 standards for flexural test are followed. The specimen samples size is decided to be 127 × 12.7 × 3.2 mm. The moulded specimen sample tested at head displacement speed of 2 mm/min.

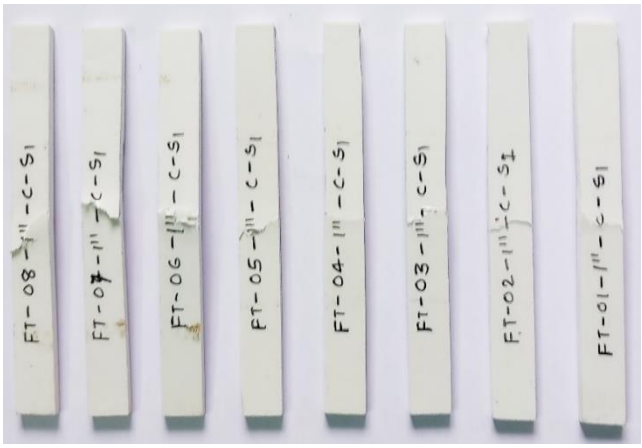


Fig.8 Samples used for the flexural test

The Universal Testing Machine used for the flexural test has specifications similar to that is being used for the tensile test.

4.6 Characterization of Hardness

The moulded specimen samples are tested according ASTM D 2583-07 standard. The specimen samples size is decided to be 25 × 25 × 3.2 mm. The total four samples are prepared for the test. The hardness of the sample is checked using the hardness test carried out using Barcol hardness impressor.

Fig. 9 Barcol hardness impressor

5. Results and Discussion

5.1 Scanning Electron Microscope Imaging

In this study, the specimens failed in the tensile test are used for the SEM image analysis. Fracture surface of tensile test sample coated with 5 nm of platinum to avoid the charging effect.

5.1.1 Agglomeration of Filler

The degree to which the material properties of a polymer nanocomposite are strengthened depends on the filler's distribution over the polymer matrix. Material scientists concentrate on enhancing the polymer-filler interaction, or bonding between fillers and the polymer basis, while making composites. However, because the fillers prefer to bind with one another rather than the polymer matrix, the fillers may gather into clusters or clouds. This kind of bonding is referred to as filler-filler interaction. Increased filler-filler interaction degrades a polymer composite's material quality. When viewing a greyscale SEM image, as can be seen in Fig. 10, the agglomerates are clearly visible in white colour.

5.1.2 Void Formation

The majority of polymer nanocomposite materials exhibit volume dilatation when subjected to forces that cause elongation. At small strains, the material reacts by deforming between its segments in an elastic and inelastic manner. At higher stresses, cavitation-induced volume dilatation is seen [29]. Due to the fact that it reduces a polymer nanocomposite specimen's ability to support loads, the void formation phenomena has received extensive research. Voids (between crystal lamellae) are easily generated in uniaxial tension because tie chain molecules disentanglement and/or rupture are

encouraged, leading to inter-lamellar slip, which prevents significant strain hardening. [30]. When viewing a greyscale SEM image, as can be seen in Fig. 10, the micro voids, are clearly visible in dim grey colour with its dimensions mentioned in green colour.

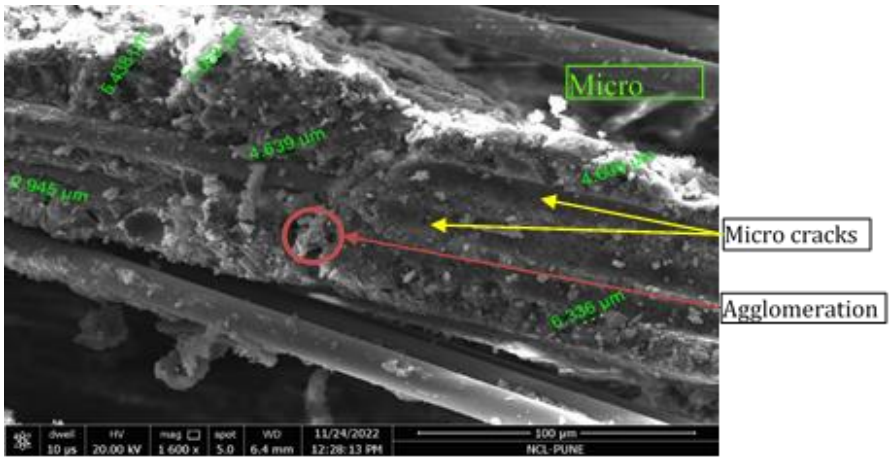


Fig. 10 SEM image showing various irregularities

5.1.3 Crack Formation

Before the start of crack initiation, a number of physical and mechanical phenomena, such as the presence of interfaces and interfacial layers around particles that cause changes in material properties, the triggering of fibrillation and crazing due to the formation of voids, and scaling or confinement effects brought on by the small sizes of the constituent structural elements, occur in nanostructured polymers. In order to prevent void coalescence, the size of the voids should be smaller (with dimensions of few nano meters) in case of unavoidable occurrence of void formation [31]. If there is formation of larger voids upon cavitation and – by coalesce of voids formed in closely connected polymer nanocomposite particles – give rise to crack formation and premature fracture. Two of the micro cracks that are formed in the sample can be observed in Fig. 10.

5.2 X-ray Diffraction Analysis

A single XRD reflection consists of various independent basic parameters and each of them possesses its own physical meaning [2]. The Bragg' s diffraction angle (the angle between the incident rays and the diffracting planes) graphically exhibits the peak position. The size of sub-micrometer crystallites in a solid is related to the width of a peak in a diffraction pattern by the Scherrer particle size. So, in getting the dimensions of the particles, the width of an XRD peak plays a pivotal role. The peak shape comprehensively issues the effects of both crystalline size and lattice strain. The sharp peaks are often observed in case of powder XRD which can be seen in Fig. 11. Peak intensity is a reflectance of both absorption and amount of phase in the mixture. The diffraction angle, geometry of instrument, phase diffraction feature, and crystal structure affects the value of peak intensity.

The X-ray diffraction of powder of nano-clay composite SMC weighing 5 gm is carried out at different diffraction angle (2θ) from 10º to 80º. The results, as indicated in Fig. 11, shows highly crystalline peaks for the sample with 0.2 phr observed at diffraction angles of 23.40º, 29.72º, 36.29º, 39.75º, 43.50º, 47.85º, 48.85º with highest peak having intensity of 6558 at 29.72º. For the sample with 0.4 phr observed at diffraction angles of 23.19º,

29.57º, 36.12º, 39.59º, 43.33º, 47.72º, 48.67º with highest peak having intensity of 6075 at 29.57º. As per the Bragg' s equation, peak position (diffraction angle) is a function of the distance (d_{hkl}) between reflection planes (hkl) and in this case the distance between reflection planes varies from 3.7967 Å to 1.8620 Å. The equation known as "Scherrer's equation" is frequently used to calculate the size (diameter) of crystallites from recorded diffraction peak profiles by substituting the FWHM (full width at half its maximum intensity) for the integral width for the corresponding peak. In the current study the diameter of the crystallite is found to be 316 Å.

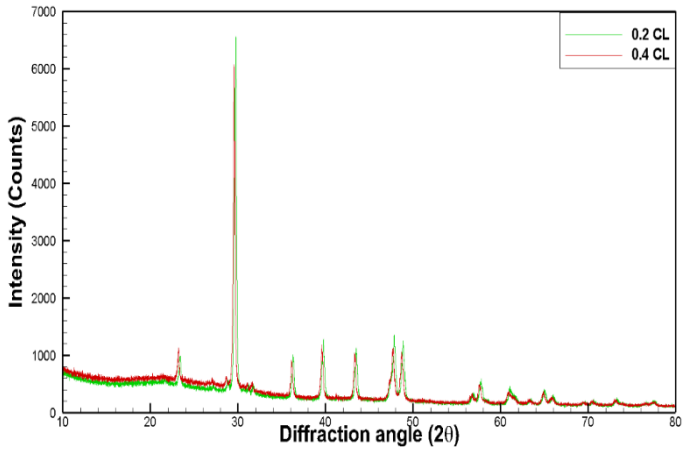


Fig. 11 X-ray diffraction diffractogram of nanocomposite formed using nano-clay

These multiple peaks indicate that the polymer nanocomposite is crystalline [31], however, the intensity at the peak decreases when the amount of nano clay is increased from 0.2 phr to 0.4 phr. The crystallinity is proof that there exists a strong bonding between the several additives used in the formations of polymer nanocomposite.

5.3 Effect of Amount of Reinforcement on Mechanical Properties

The detailed result on properties of nanocomposites formed by varying different parameters like amount of nano clay, temperature and pressure applied during compression moulding, can be seen in Table 4.

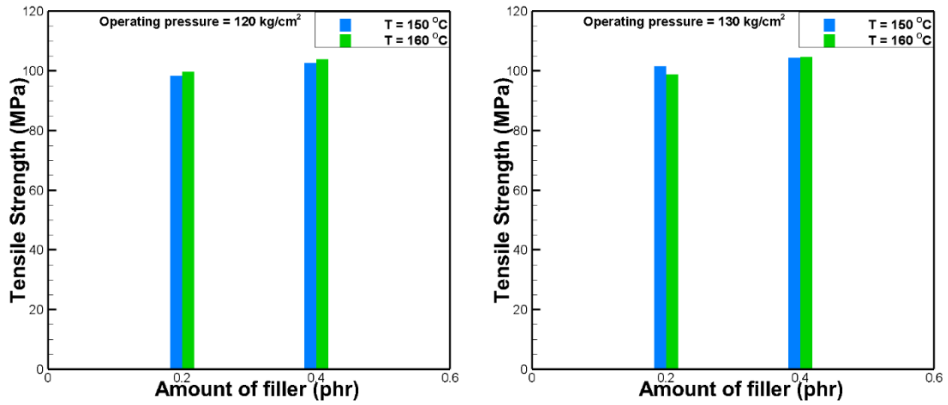
Table 4. Tensile and flexural properties of the samples

Run	Nano clay (Phr)	Temp. (ºC)	Pressure (Kg/cm^2)	Tensile strength (MPa)	Flexural strength (MPa)	Elongation (%)
1	0.2	150	120	98.2	191.4	5.17
2	0.4	150	120	102.6	195.4	6.28
3	0.2	160	120	99.6	191.8	5.68
4	0.4	160	120	103.8	198.3	7.11
5	0.2	150	130	101.5	193.5	5.48
6	0.4	150	130	104.3	198.7	7.66
7	0.2	160	130	98.8	192.6	5.24
8	0.4	160	130	104.6	199.4	7.91

By comparing the results of the samples of 2nd, 4th, 6th, and 8th run with the results of the samples of 1st, 3rd, 5th, and 7th run, respectively, it can be concluded that for all the values of temperature and pressure of compression moulding, the tensile strength, the flexural strength, and the percentage elongation of the nanocomposite increases with increase in amount of nano clay material. This means that the polymer nanocomposite with more amount of reinforcement offers more resistance to the tensile and bending load than that with low amount of reinforcement which ultimately means that the ductility of the polymer nanocomposite with more amount of reinforcement is higher than that with low amount of reinforcement. This underlines the importance of formation of polymer nanocomposite from the base material so as to improve the performance against tensile and bending load.

5.4 Effect of Temperature of Compression Moulding

The performance enhancement of the nanocomposites in terms of the tensile strength, as can be seen from Fig. 12, improves negligibly with increase in temperature for all the samples except the samples with 0.2 Phr of nano-clay and 130 kg/cm^2 of operating pressure. The maximum improvement in the tensile strength is found to be 1.4 %.



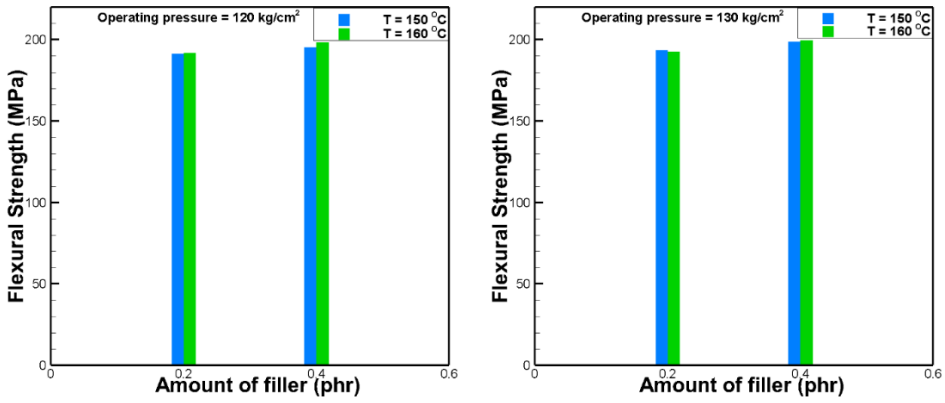
(a) (b)

Fig. 12 Effect of compression moulding temperature on tensile strength of nano-clay nanocomposite at operating pressure of (a) 120 kg/cm^2 (b) 130 kg/cm^2

The performance enhancement of the nanocomposites in terms of the flexural strength, as can be seen from Fig.13, improves negligibly with increase in temperature for all the samples except the samples with 0.2 Phr of nano-clay and 130 kg/cm^2 of operating pressure. The maximum improvement in the flexural strength is found to be 1.5 %.

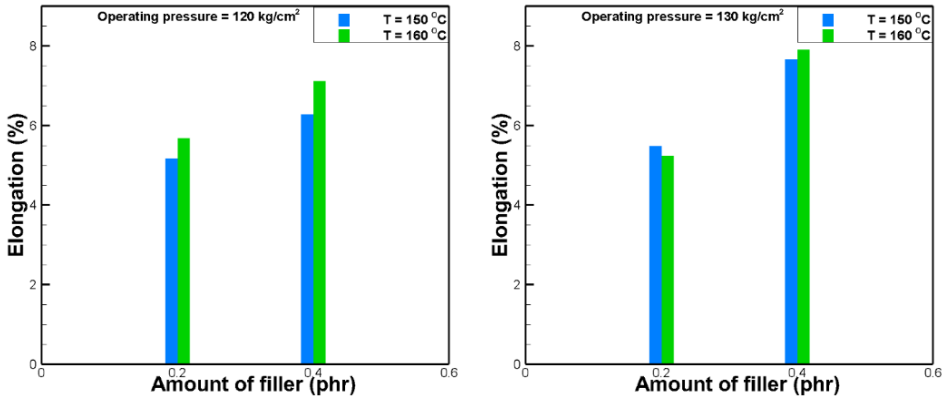
The performance enhancement of the nanocomposites in terms of the percentage elongation, as can be seen from Fig.14, improves with increase in temperature for all the samples except the samples with 0.2 Phr of nano-clay and 130 kg/cm^2 of operating pressure. The maximum improvement in the percentage elongation is found to be 13.2 %.

It is noteworthy that the performance of the samples with 0.2 Phr of nano-clay and 130 kg/cm^2 in terms of tensile strength, flexural strength and the percentage elongation, deteriorates with increase in operating temperature. This means that with lower amount of filler and applied pressure, the increase in temperature may be causing weakening of bonding between the nano-clay and the base material resulting in decrement in the resistance offered by nanocomposite to the tensile and bending load.



(a) (b)

Fig.13 Effect of compression moulding temperature on flexural strength of nano-clay nanocomposite at operating pressure of (a) 120 kg/cm² (b) 130 kg/cm²



(a) (b)

Fig.14 Effect of compression moulding temperature on percentage elongation of nano-clay nanocomposite at operating pressure of (a) 120 kg/cm² (b) 130 kg/cm²

Also, when the composite does not completely melt or flow at low mould temperatures, glass fiber impregnation is decreased. If the heating temperature is below a certain level during the moulding process, the resin cannot completely melt or flow, resulting in excessive flow viscosity and insufficient saturation; if the heating temperature is above a certain level, the resin will degrade, reducing its mechanical performance. A composite material can never revert to its initial state once it has solidified. Cross-links, which are three-dimensional molecule chains, may have formed as a result. In order to prepare the composite to be extremely strong and thermally resilient, the curing temperature needs to be raised. Beyond the critical mould temperature, the matrix starts to degrade and the fibers start to disorder, leading to brittleness and reduced strength. As it has been found that the increment in moulding temperature enhances the mechanical properties of the composite, it can be safely said that the operating temperature used in this study is less than the critical mould temperature.

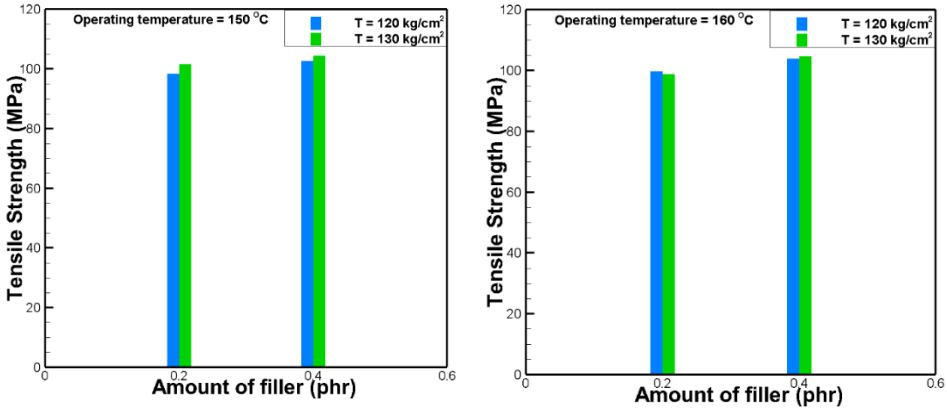
Table 5. Barcol hardness and specific gravity of the samples

Run	Nano clay (phr)	Temp. (ºC)	Pressure (Kg/cm²)	Barcol hardness	Specific gravity
2	0.4	150	120	47	1.711
4	0.4	160	120	48	1.694
6	0.4	150	130	48	1.699
8	0.4	160	130	49	1.690

The detailed results of Barcol hardness test and measurement of specific gravity for fixed amount of filler of 0.4 phr, and variable temperature and pressure applied during compression moulding, can be seen in Table 5. For applied pressures of 120 kg/cm² and 130 kg/cm², the performance of nano-clay composites, in terms of Barcol hardness and specific gravity, slightly enhances with increase in temperature (decrement in specific gravity is desirable).

5.5 Effect of Applied Pressure of Compression Moulding

The performance enhancement of the nanocomposites in terms of the tensile strength, as can be seen from Fig. 15, improves negligibly with increase in pressure for all the samples except the samples with 0.2 Phr of nano-clay and 160 ºC of operating temperature. The maximum improvement in the tensile strength is found to be 3.4 %.

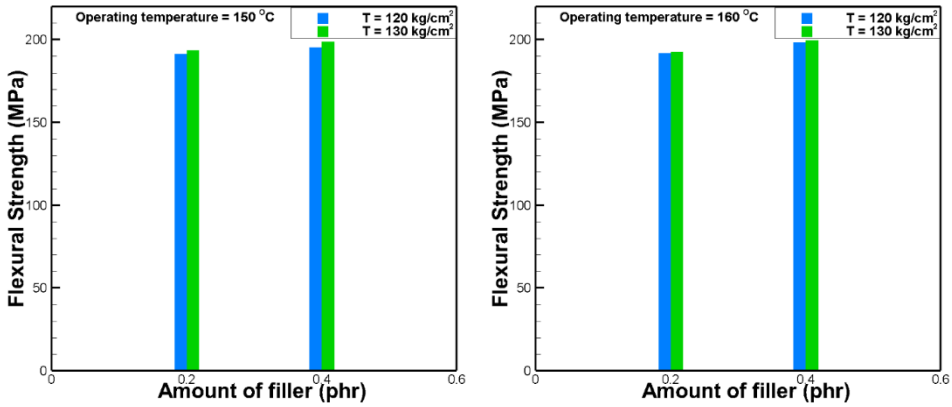


(a) (b)

Fig. 15 Effect of compression moulding pressure on tensile strength of nano-clay nanocomposite at operating temperature of (a) 150 ºC (b) 160 ºC

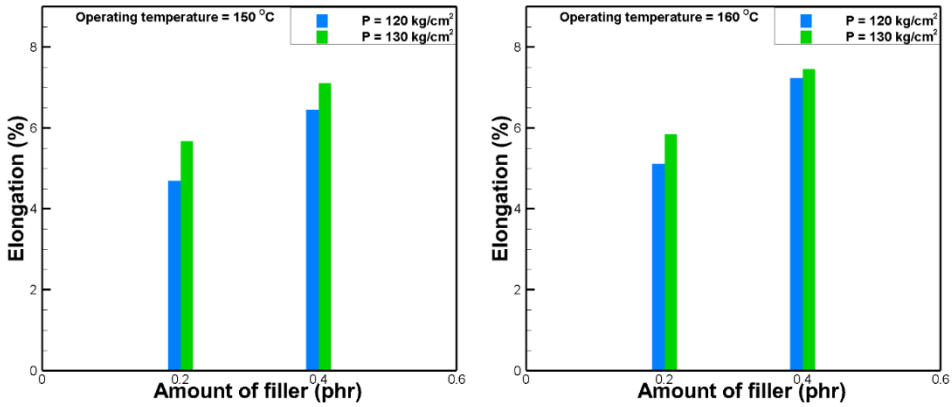
For samples with 0.2 Phr and 0.4 Phr of nano-silica and 150 ºC and 160 ºC of operating temperature, the performance of the nanocomposites in terms of the flexural strength, as can be seen from Fig. 16, improves negligibly with increase in pressure. The maximum improvement in the flexural strength is found to be 1.7 %.

The performance enhancement of the nanocomposites in terms of the percentage elongation, as can be seen from Fig. 17, improves with increase in pressure for all the samples except the samples with 0.2 Phr of nano-clay and 160 ºC of operating temperature. The maximum improvement in the percentage elongation is found to be 22 %.



(a) (b)

Fig. 16 Effect of compression moulding pressure on flexural strength of nano-clay nanocomposite at operating temperature of (a) 150 ºC (b) 160 ºC

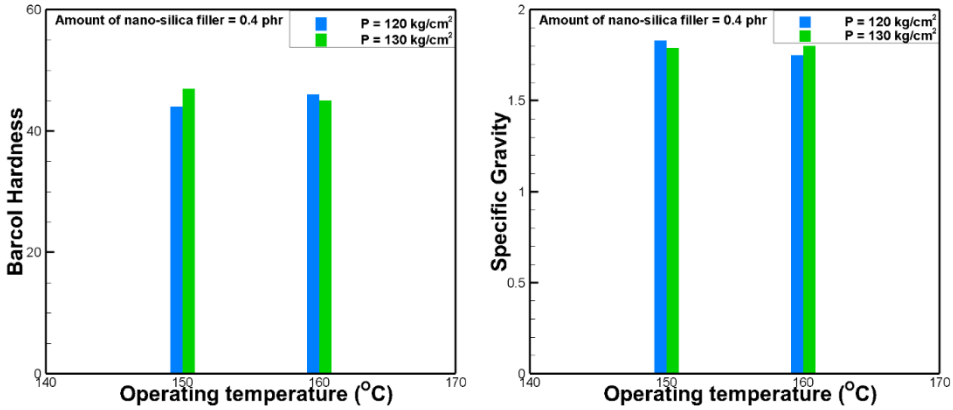


(a) (b)

Fig. 17 Effect of compression moulding pressure on percentage elongation of nano-clay nanocomposite at operating temperature of (a) 150 ºC (b) 160 ºC

The melting point of the polymer is raised by the pressure used during compression moulding. More of the initial crystallinity is preserved in the polymer when the melting point rises. The polymer consolidates better under the higher pressure. Various mechanical properties, like tensile and flexural properties, of the moulded part are directly impacted by the consolidation of the part. When compared to weakly consolidated components, well-consolidated parts offer higher resistance to the various types of mechanical loads.

The results of Barcol hardness test and specific gravity measurement for fixed amount of nano-silica filler of 0.4 phr, and variable temperature and pressure applied during compression moulding, can be seen in Fig.18.



(a) (b)

Fig.18 For the samples with the amount of nano-clay filler to be 0.4 phr, effect of compression moulding pressure on (a) Barcol hardness (b) Specific gravity

For temperature of 150 ºC the performance of nano-clay composites, in terms of Barcol hardness and specific gravity, slightly enhances with increase in pressure. The opposite trend in the performance in terms of Barcol hardness and specific gravity, is observed at the operating temperature of 160 ºC.

5.6 Performance Comparison of Nano Clay Composite And Base Material

The tensile strength, flexural strength and % elongation of the base material is 81.2 MPa, 171.3 MPa, and 5.06% while from Table 4, it can be seen that the maximum value of the tensile strength, flexural strength and % elongation for the Nano clay composite is 104.6 MPa, 199.4 MPa and 7.91%. This means that the tensile strength, flexural strength and % elongation of the base material can be enhanced by 28.82 %, 16.40 % and 56.32 %, respectively by reinforcement of nano clay. The Barcol hardness and specific gravity of the base material is 47 and 1.715 while from Table 5, it can be seen that the maximum value of the Barcol hardness is 49 and the minimum value of the specific gravity is 1.69, for the Nano clay composite. It has to be noted that the reduction in the specific gravity is the desirable result. This means that the performance of the base material in terms of Barcol hardness and specific gravity can be slightly enhanced by 4.26 % and 1.46 %, respectively by reinforcement of nano clay.

6. Conclusions

This study has considered characterization in the form of various mechanical tests and analysis methods. The major conclusions that can be drawn from the study are as mentioned below.

- The irregularities like agglomeration of filler material, formation of micro voids and micro cracks, that can be clearly seen from the SEM images, are needed to avoided during the fabrication of polymer nanocomposites, in order to enhance the performance of the nonocomposites.
- X ray diffraction results has shown multiple peaks which are indirect indication of the crystalline nature of polymer nanocomposite. Also, the intensity of the peaks got decreased after increasing the amount fnano clay from 0.2 phr to 0.4 phr.

- The tensile strength, flexural strength, and the percentage elongation of the nanocomposite material enhances with increase in amount of reinforcement material, at any moulding temperature and pressure.
- At lower moulding pressure, the increase in temperature enhances the of tensile strength, flexural strength, and the percentage elongation for both the values of amount of reinforcement whereas at higher moulding pressure and amount of reinforcement, the increase in temperature decreases the of tensile and flexural strength.
- At lower moulding temperature, the increase in pressure enhances the of tensile strength, flexural strength, and the percentage elongation for both the values of amount of reinforcement whereas at higher moulding temperature and lower amount of reinforcement, the increase in pressure decreases the of tensile strength, flexural strength, and the percentage elongation.
- The increase in the moulding temperature and pressure, increases the Barcol hardness test and decreases the specific gravity (which is desirable), but by very small amount.
- It has been revealed that the polymer nanocomposite becomes more ductile (by virtue of enhancement of tensile strength and the percentage elongation by 29% and 56%) and offers more resistance to bending (by virtue of enhancement of flexural strength by 16%), as compared to its base material.
- Barcol hardness test and measurement of specific gravity has revealed that there is improvement in the performance of the polymer nanocomposite as compared to that of base material, but by very less amount.

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