EFFECT OF BASALT NANOPARTICLES ON MECHANICAL AND THERMAL CHARACTERIZATION

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Abstract

In this investigative study, composites which are based on basalt, Jute, Polyester and Polypropylene are formulated with epoxy resins and basalt nano particles. An ultrasonic probe with high intensity was used to obtain a homogenous mixture of epoxy resin and nano particles of basalt, the nano particles through sonic cavitation were infused into resin. The measurement of loading effect of nano particles on mechanical properties such as tensile modulus, impact strength and flexural modulus was conducted. Shifting of thermal degradation temperatures of composites with addition of basalt nano particles to higher temperatures was observed in Thermo-gravimetric (TG/DTG) composites. The thermal stability of composites increased considerably by the addition of basalt. A better fiber-matrix interfacial interaction in hybrid composites was observed in the images of tensile fractured sides of composites during observation in images of scanning electron microscope. On the basis of results, it is evident that with reference to performance, prime important factor is miscibility of nanoparticles in resins.

Keywords: Nano composites, basalt fiber, thermal stability, mechanical properties

1. INTRODUCTION

Modern engineering design constitutes of a very important share of polymer composites. Polymer composites can, due to high specific strength, replace metallic components in variety of application. They can provide a reduced weight with same strength. The discovery of nano particles, at the end of 20th century, has provided a new importance to the polymer composites in design engineering. At least one dimension of these nanoparticles belongs to the nanometer dimension. There are many types of nano particles. There are various metal oxides, most typical are TiO_2 and Al_2O. The addition of nanoparticles can help in improvement of properties of fiber reinforced composites [1].

The great asset of polymer composites is their strength whereas main disadvantages involve poor electrical conductivity and low usage temperatures. For ideal applications in structure the materials should be chemically and thermally stable, possess light weight as well as good mechanical properties along with low cost. No real material possess these properties in reality.

In previous decades with the usage of better technology, more advanced fibers were prepared along with new reinforcements out of which, basalt is most promising. Basalt possesses properties in similarity with glass fiber with advantage of simplicity in production process, improved chemical and heat resistance. A simple cleaning and grinding process will make ready for production with just rightly acceptable raw material.
The raw material for basalt fibers is, abundantly present basalt rock which naturally exists. It can be used as reinforcing filler for a wide range of composites such as dispersion reinforced cements and basalt plastics. It has advantages of good thermal stability at higher temperatures, low cost and good resistance to solvents and acids [2].

Basalt fibers, being a relatively recent induction in composites as reinforcement, is yet to be investigated in terms of utility in various areas. A bulk of previous work focussed on epoxy composites and basalt designed to capitalize on thermal stability of basalt. [3-4] with the advantage of low cost compared to carbon/epoxy composites and superiority in terms of strength over E-glass, basalt is presented as a valuable alternative in many applications.

Only a few researchers who managed to create a composite by embedding basalt fiber in a polymer matrix. From previous researches over basalt woven hybrid composites expresses excellent thermal stability and mechanical properties [5]. This research is focused on use of combination of basalt as nano particles with hybrid woven basalt structures for structural applications. The aim of this research was, development of new type of nano particles along with testing and preparation of woven hybrid composites structures of basalt with nano particles. Very few investigation over use of basalt in hybrid structures are carried out. Prepared composites were characterized by their thermal, mechanical and morphological properties.

2. MATERIAL & METHODS

2.1. Material

Basalt(B) was supplied by the company Kamenny Vek (KV). Polyester (PET) and Jute (J) yarn used in this study were available commercially. Polypropylene (PP) yarn was received from company Syntheic. The materials were used as received. The details of yarns are given in **Table 1**. Green epoxy resin CHS-Epoxy G520 and hardener TELALIT 0600 were supplied by Spolek, Czech Republic. It is a low molecular weight basic liquid epoxy resin containing no modifiers, certified by International Environmental Product Declaration Consortium (IEC).Nano particles of basalt were made by ball milling method .For nanoparticle size distribution Zetasizer Nano ZS (Malvern Instruments, UK) was used and particle size was 230 nm. We added 3 wt% nanofiller in the composites produced.

2.2. Methods

Hybrid fabric samples were developed with Plain weave from Basalt/Jute (B/J),Basalt/Polyester(B/PET) and Basalt/Polypropylene(B/PP) yarns. Non- hybrid Basalt/Basalt (B/B) fabrics were also developed. All fabrics were made on the CCI sample loom with the same density for all fabrics, 12 threads/cm in warp and 8 threads/cm in weft .All the fabric variants were measured, according to standardized procedures. Yarn tensile properties were measured as presented in **Table 1**.

Table 1 Properties of fibers and yarn

Properties	Basalt	Polyester	Polypropylene	Jute
Diameter of fibers (micron)	12	22	34	18
No. of filaments	890	900	300	-
Linear density of yarn (Tex)	295	250	292	296
TPM (Twists/m)	20	24	30	180
Tensile strength (N)	92.75	88.91	57.44	41.43
Tensile elongation %	1.29	12.55	12.27	1.39
Tenacity (N/tex)	0.315	0.305	0.23	0.139
Modulus M Pa	9.378	1069	721	3.741

Preparation of Nanocomposites:

The Basalt hybrid and non-hybrid composites were fabricated using hand layup method with single ply of the reinforcing fabric. Epoxy resin was used as matrix (matrix comprising of both resin and nanoparticles) with a hardener at weight ratio of 100:32 (by weight) according to manufacturer recommendations. The composite layup along with Teflon sheets were sandwiched between a pair of steel plates and cured at 120°C for 1.0 h in mechanical convection oven with predetermined weight. The fiber volume fraction (Vf) of all composites was around 0.4. Specimens of various dimensions were then cut from the sheets for various testing.

Mechanical testing:

Tensile tests were performed by a TIRATEST universal tensile tester computer controlled according to standards EN ISO 527-5.From the force-elongation curves the tensile strength and the elongation at break values were determined. The tensile modulus was calculated from the slope fitted to the initial portion of the stress-strain curve.

Three point bending tests were performed on a computer controlled, TIRATEST type universal tester according to the EN-ISO 14125standard. The deformation rate was 2 mm/min.

Impact strength (Charpy) was carried out in an impact tester EN-ISO 14125 rectangular using CEAST RESIL 5.5 with a force of 22 J at a velocity of 2.9 m/s. The width and thickness of the specimen were measured and recorded. The work of fracture/impact strength values were calculated by dividing the energy in J recorded on the tester by the cross-sectional area of the specimen.

The average values of 5 specimens for each sample in each direction have been reported.

Thermal Properties:

The Mettler Toledo **TGA/SDTA85**1[e] instrument was used to study the thermal gravimetric behaviour (thermal stability and degradation) of the composite. Thermo gravimetric analysis was performed under dynamic nitrogen atmosphere. The samples were heated from 25°C to 700°C at a heating rate of 10°C/ min to yield the decomposition temperature and mass loss.

Morphology:

Morphology analysis was done by SEM. The SEM photographs composites were taken using a scanning electron microscope TS5130-Tescan SEM at 20 kV accelerated voltage. The surfaces of the samples were coated with gold by means of a plasma sputtering apparatus prior to SEM investigation and were investigated at 2,000 × magnification to observe the fiber matrix adhesion. The microstructure of fracture surfaces was examined by Scanning Electron Microscope (SEM) techniques in order to identify the relevant fracture mechanisms involved. Prior to analysis the fractured samples were also coated with gold and samples were examined with different zoom level for obtaining high resolution image.

3. RESULTS AND DISCUSSION

3.1. Mechanical testing:

Measurement of tensile properties is the most common mechanical measurement. It is used to determine the behavior of a sample while under an axial stretching load. The tensile properties of the composites material is dependent on tensile properties of fiber, matrix and interfacial bonding of resin and reinforcement material. Normally in fiber reinforced composites, the modulus of a composite material is dependent on the reinforcing fiber properties. The tensile properties of nanocomposites are shown in **Figure 1**. Superior mechanical properties are observed in warp direction, compared with weft direction in all nanocomposites. The composites

fabrics contains more number of yarns in warp direction than in weft direction. Due to its higher yarn at warp direction composite having lower density might be the reason why its performance shows contrast with weft direction of composite.

From **Figure 1** it can be observed that after 100 % basalt composites, structures which have polyester has highest modulus, followed by PP composites. As polyester has more filaments, less liner density and low twist so after relaxation warp come closer so high level of crimp in warp direction which make them stiffer**.**

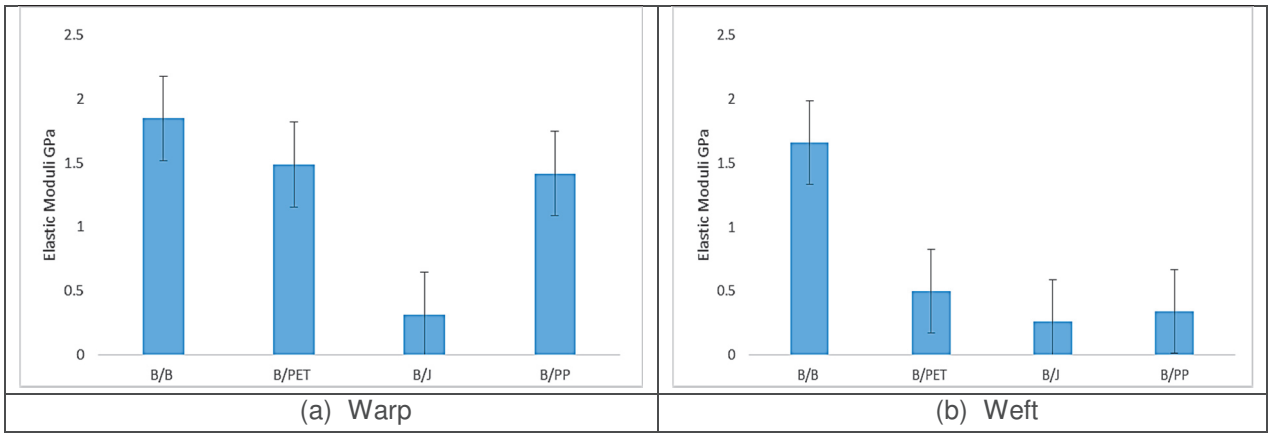


Figure 1 Comparison of modulus of composites

The flexural modulus is a measure of the resistance to deformation of the composite in bending. Very similar result is observed in the flexural strength of the composites which is shown in **Figure 2**. It can be seen that, B/B composites has higher bending properties as bending properties also dependent on fiber properties so it follow the same trend as tensile properties. As like tensile properties, flexural behavior of composite is found higher in case of warp direction than weft direction. Less yarns in weft direction of composite limit their tensile stress dispersion. Therefore, as the tensile stress tries to propagate upwards, delamination failure occurred thus reducing its flexural strength. Moreover, our research work found the highest flexural properties compared to the tensile properties for different fiber orientation. The strengths from the flexure test look much larger than that from the tension test, although the material failed in same way i.e. tension. It is also evidence that our material also follows theory and experiment that has been proposed by others [6].This is due to the size effect, which is well documented. The size effect is the decrease of mean strength with increasing flaw-sensitive volume under stress.

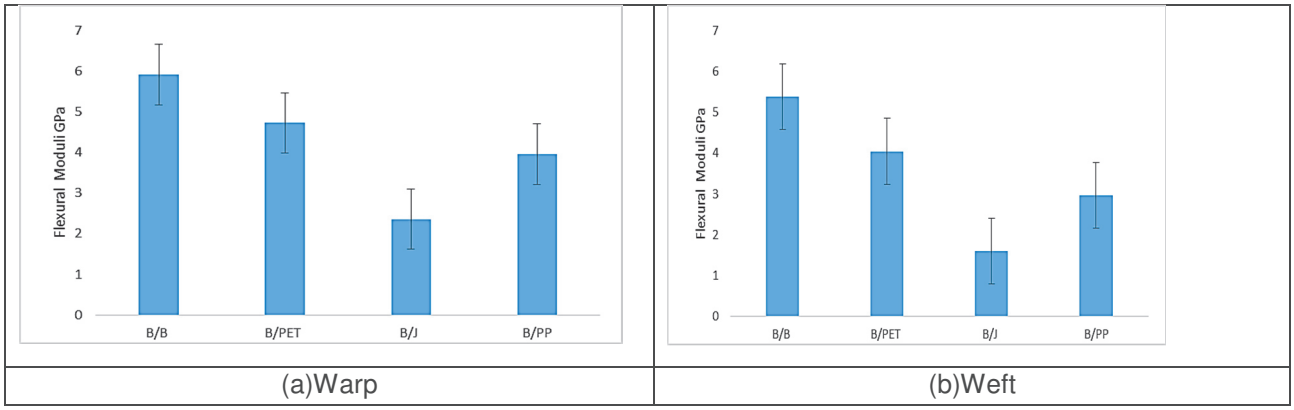
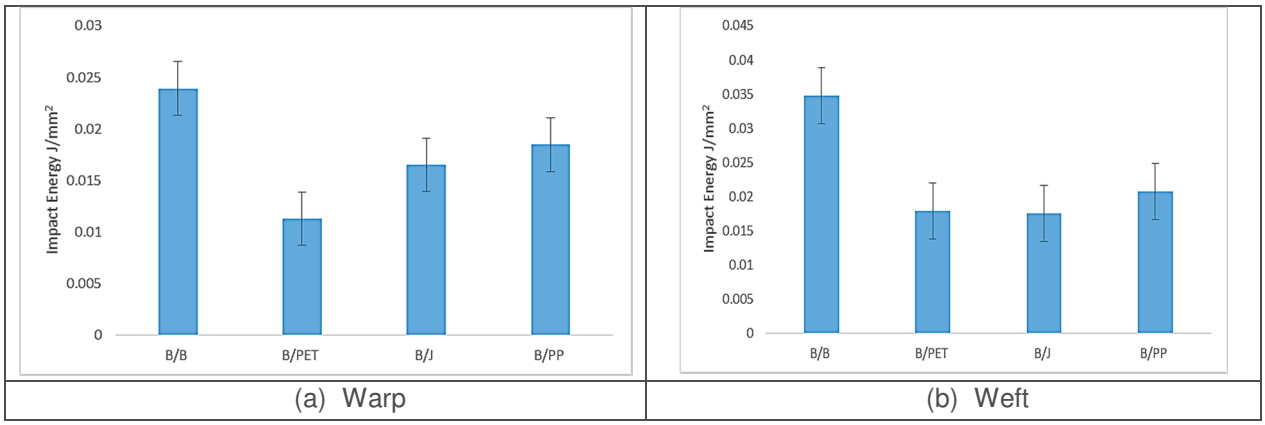


Figure 2 Comparison of Flexural modulus of composites

Impact Strength of textile reinforced composites is a measure of the ability of the composites to resist the fracture failure under stress applied at high speed and is directly related to the toughness of the composites.

Fibers play an important role in the impact resistance of composites as they interact with the crack formation and act as stress- transferring medium. Fiber absorbs part of the energy during an impact, but they also distribute some of the load internally. This excess energy can induce cracking and delamination. Next to fiber, another important factor that influences the impact energy is the fiber-matrix interfacial shear strength. It can vary depending on the adhesion between fiber and matrix. It can be observed from **Figure 3**, that non-hybrid basalt structures has highest impact strength followed by B/PP and B/J structures respectively. The increase in the impact strength with the basalt fibers content can be attributed to the higher energy dissipation at the fiber/matrix interface in order to detach the fibers from the matrix. It can be described to the fact that PP yarn decreases the stiffness and rigidity of the composite, thus causing an increase of impact resistance.

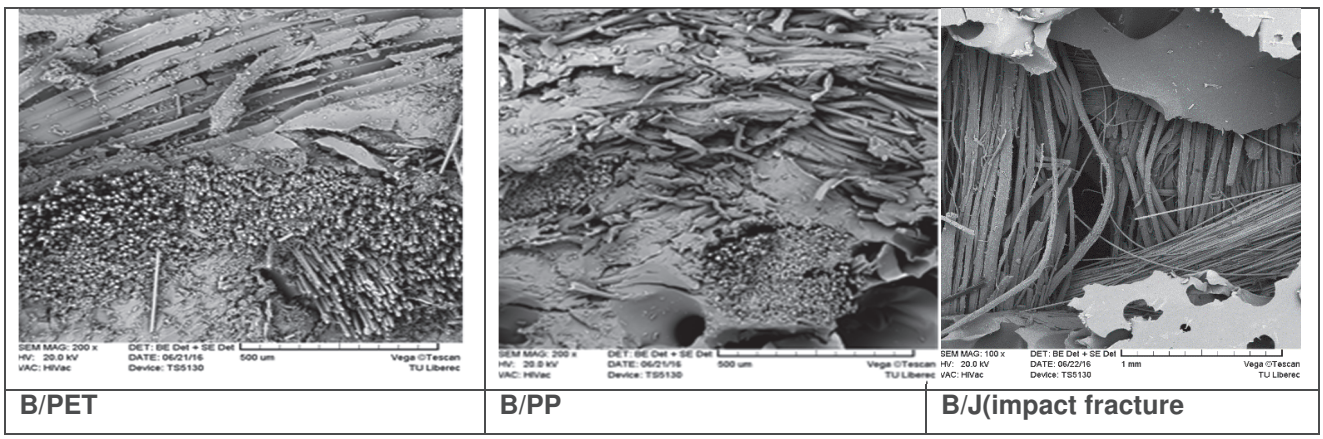


(a) Warp (b) Weft

Figure 3 Impact strength of composites

3.2. Interfacial Properties of the Composites

Interface is the key region which determines, to a great extent, the set of properties of all heterogeneous systems, including composite materials. To find out the fiber matrix adhesion inside the composites, SEM studies were carried out. SEM images of the composites are presented in Figure 4. Photography of damaged sample confirms the following failure modes in composites such as fiber breakage, matrix shear failure, shear failure between fiber and matrix, and breakage in interfiber cleavage area. A composite fail in tension, it can be due to brittle failure or fiber pull-out. This is clearly indicated for B/PP composites that fiber pull-out is quite higher and the bonding between fiber and matrix is not good. Small gaps are evident in the matrix near to the fibers However, for B/B B/J and B/PET composite interface suggested better fiber matrix adhesion which is supported by low fiber pull-out. The data presented above for the mechanical properties of composites, also supported the SEM observation.



B/PET **B/PP** **B/J(impact fracture**

Figure 4 SEM images

3.3. Thermo gravimetric analysis (TGA)

Thermogravimetric analysis (TGA) is used to study the thermal stability of composites.

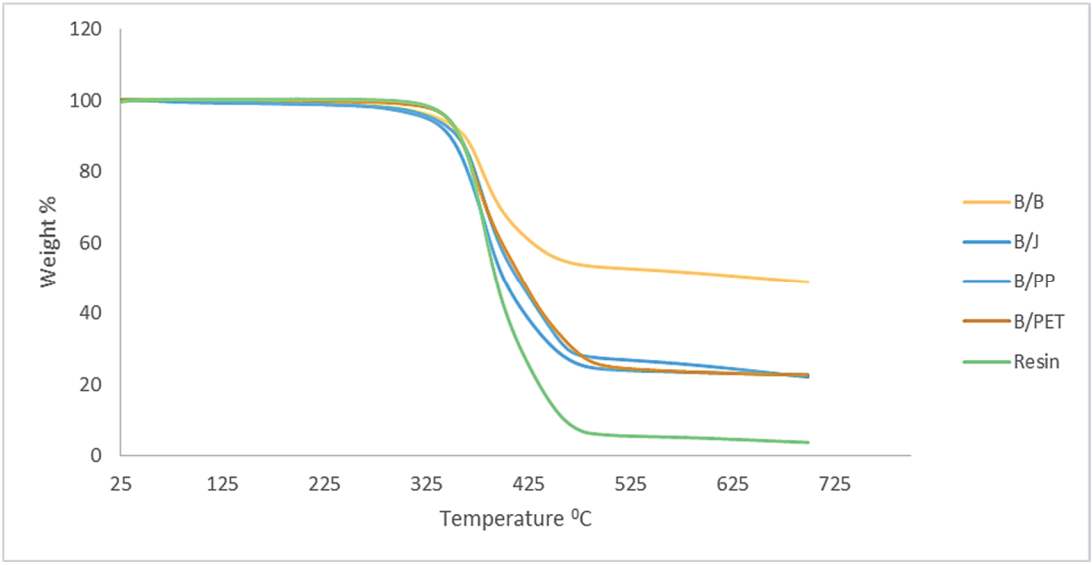


Figure 5 Combined TGA graph of all materials

The TGA curve of the neat green epoxy resin shows a gradual weight loss with increasing temperature which started around 350°C. From thermogram it is observed that a degradation starts around 350°C with a maximum degradation at 420°C. The curves presented indicate the existence of only one main mass-loss region, always located between 350 and 420°C which is higher than pure epoxy as mention in previous research [5]. This region can be attributed to the thermal decomposition of the polymer matrix.

It can be viewed that degradation of B/B composites starts around 370°C due to degradation of epoxy. Overall mass loss is less than epoxy composites [5].

In B/J hybrid composites, two stage degradation occurs: First stage is responsible for jute fabric and second stage is for Nano Epoxy. **Figure 5** shows that maximum degradation temperatures of B/J composite for first and second stage are 320°C and 430°C respectively. 100 % weight loss is not possible due to basalt fiber. During thermal decomposition of lignin, relatively weak bonds break at lower temperature whereas the cleavage of stronger bonds in the aromatic rings takes place at higher temperature. Corresponds to the thermal decomposition of hemicellulose and the glycosidic links of cellulose.

In B/ PP decomposition is a two-stage process characterized by a first step in the temperature range 347°C-380°C which may be attributed to degradation of resin, followed by second weight loss at 478°C and almost completely depleted. Polypropylene is liable to chain degradation from exposure to heat, Oxidation usually occurs at the tertiary carbon atom present in every repeat unit.

It can be noticed from **Figure 5** that the polyester degradation follows a two-step reaction scheme characterized by a first step in the temperature range of 350°C-420°C followed by a second decomposition step located in the range 470°C-600°C. This behavior is determined by random scission of the polyester backbone (ester linkage) and to the oxidation and the breakage of the secondary bonds.

The thermal analysis has illustrated that nanocomposites are stable until 350°C. From the thermogram TGA, it can be viewed that the maximum degradation temperature T_{max} has significantly improved for B/PET fabrics and the thermal stability of the composites have been improved, which justifies the development of strong fiber-matrix interface in composite

4. CONCLUSIONS

The performance of the nanocomposites was evaluated. This article compares and discusses the mechanical properties/ thermal properties in the warp and weft direction of polymer composites reinforced by basalt woven fabric .This study shows that basalt/epoxy composites reinforced with basalt nanoparticles present higher mechanical performance than conventional basalt fiber reinforced epoxy matrix composites. A series of nanocomposites was developed. SEM micrographs revealed that well-dispersed and non-agglomerated nanocomposite systems were produced. These nanocomposites are a promising candidate for developing structural applications.

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