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Mechanical and Thermal Analysis of Basalt/Sisal hybrid Fiber Reinforced Poly (Lactic Acid) Composites

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ABSTRACT

In recent years there is growing demand for the development of fully biodegradable composites materials based on poly (lactic acid) and natural fibers. Natural fibers can be assed in to matrix to improve mechanical and thermal properties of matrix with low cost, low density and high strength and stiffness. The advantage of using hybrid composite is that one of the fibers might complement with another fiber that lacks the desired properties. Hence, we made an attempt to reinforce PLA with basalt / sisal fiber combination. In this study we are carried out the investigation of effect of hybrid fiber content on mechanical and thermal properties of hybrid composites (PBaSi). Blends of alkali-treated sisal fiber and silane treated basalt fibers were used as hybrid fiber reinforcement to obtain hybrid composites with poly lactic acid (PLA) as matrix phase. The composites were prepared in two stages. In the first stage blending of Basalt/Sisal fiber with PLA is carried out using twin screw extruder to form the hybrid compound granules. And in the second stage hybrid composite specimens were prepared according to ASTM standards using injection molding. Then the effects of hybrid fiber content on mechanical properties such as Tensile strength, Flexural strength and Impact strength, have been investigated by means of UTM, as well as thermal stability such as crystallization and melting behavior have been studied by means of Differential Scanning Calorimetry (DSC). Mechanical and thermo – mechanical properties of hybrid composites were studied and compared using Dynamic Mechanical Analysis (DMA). Scanning Electron Microscopy (SEM) conducted on the fracture surfaces of the hybrid composites (PBaSi) reveals superior interfacial linkage between the hybrid fiber and Poly lactic acid (PLA) matrix. The results suggest that the PBaSi-3 composites having 20 wt% hybrid fiber having high mechanical property and thermal stability then the other two combinations of composites. And PBasi-3 is a potential composite among the other natural fibre combinations for automotive seat shell manufacturing; automobile body panels and other structure components to solve the “white pollution” in environmental management.

Keyword: PLA, Basalt/ Sisal Hybrid fiber, Twin screw extruder, Injection moulding process, DSC, DMA, Scanning Electron Microscopy.

1. INTRODUCTION

Due to the environmental consciousness the development of fully biodegradable composites materials based on polylactide and natural fibers gain more and more role. There is an increase demand for environmental friendly materials such as natural fiber composites to replace the traditional fiber (i.e. carbon, glass, and aramid fiber) composites. The reasons are: biodegradability, less emissions to the atmosphere, abundant, renewable, availability and can be produced at low cost in many parts of the developing world [2]. Poly (lactic acid) or poly lactide (PLA) is produced from agricultural base product such as corn, beets, wheat and other starchy product. PLA is a highly versatile biodegradable polymer. Natural fibers are a major renewable resource material. Joshie et.al pointed out that natural fiber reinforced composites are environmentally superior to glass fiber reinforced composites. Karmaker et.al indicated that the biodegradability of natural fibers can contribute to healthy ecosystems and their low cost and mechanical performance are very useful for industries.

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Among the natural fibers Basalt and Sisal fibers are noteworthy and find extensive application in the development of hybrid bio composites wowing to their exceptional combination of properties. Sisal can be used as a reinforcing material in polymeric resin matrices to make useful structural composite materials that offer unusual combination of properties [3]. Basalt is a mineral of volcanic origin; this fiber made of basalt rock is very economical and has excellent mechanical strength, excellent sound and thermal insulator, biologically stable and non flammable and so on. Subramanian and Austin was reported that Basalt as a possible polymer reinforcing material. And it could be applied to polymer matrix composites instead of Glass fiber. Basalt fibers are produced by using basalt volcanic rocks, which can be found in natural and in every country around the globe. It is biologically inert and environmentally friendly in nature. Even though the basalt fiber are not biodegradable, still it is considered as natural fiber, because it is produced from natural volcanic rocks.

Basalt fibers are produced from two different technologies. Short basalt fibers are produced by Junkers method; continuous fibers are produces by spinneret method. In this study Chopped basalt fibers produced with Junkers method from Muktagiri Industrial Corporation were obtained with the company's own treatment, developed for strong adhesion with polyesters. The average diameter of the fibers was 13μ m and the length was 6 mm. It can be used between 200 to 600 ^{0}C without loss of mechanical properties. Basalt fiber surface can be modifies easily, hence it can be used as reinforcement fiber for composites. Sisal Fiber is one of the most widely used natural fiber and is very easily cultivated. It is obtain from sisal plant. The plant, known formally as Agave sisalana. Sisal fibre is exceptionally durable and a low maintenance with minimal wear and tear. Its fibre is too tough for textiles and fabrics. Sisal is a strong, stable and versatile material and it has been recognized as an important source of fibre for composites [4-6]. It's density is 1.45g/cm3 and the diameter is 100-300 µm.

Researches in biotechnology are projected to make bio-polymers cost-effective with their petroleum based counterparts by 2010 to 2015 [7]. In recent day's natural fiber composites materials are widely used in automotives such as car door panels, car roofs, covers, [8] and structural panels, sandwiched beams for roofs in housing and structural applications [9]. Basalt fiber and Sisal fiber seems to be good alternative to reinforce PLA, however combining of two different natural fibers to reinforce PLA was tried in recent years, but study of sisal and basalt fiber combination (hybrid fiber) is not yet tried by the researchers till now. Hence the present studies comprises the development of basalt/sisal hybrid fiber reinforced PLA composites(PBaSi) and investigation of effect of hybrid fiber content on mechanical, thermal and morphological behavior of "PBaSi" hybrid composites fabricated by injection moulding process.

2. EXPERIMENTAL DETAILS

2.1. Materials

The Basalt fiber used in this work was supplied from Muktagiri Industrial Corporation, Borivali west, Mumbai, India, and it was supplied as silane treated and chopped about 4-6 mm length. Sisal fiber (SF) was purchased from Perfect Fiber Article Manufacture, Sulur town, Coimbatore, Tamilnadu, India. And it was supplied as untreated long fibers.

Poly lactic acid (PLA) Biopolymer 3052 D designed for injection moulding application in pellet form was obtained from Nature-Tec India Pvt Ltd., Chennai, Tamilnadu, India. Sodium hydroxide (NaOH) which is used in surface modification of sisal fiber is purchased from Subra Scientific Company, Puducherry, India.

2.2. Surface treatment of sisal fiber

Almost all the natural fibers contain hemicelluloses, lignin and wax materials, because of this contents Natural fiber (NF) are deliquescent in nature. This property will lead to lack of bonding between the

Figure 1: Surface modified (Alkali treated) long sisal fiber.

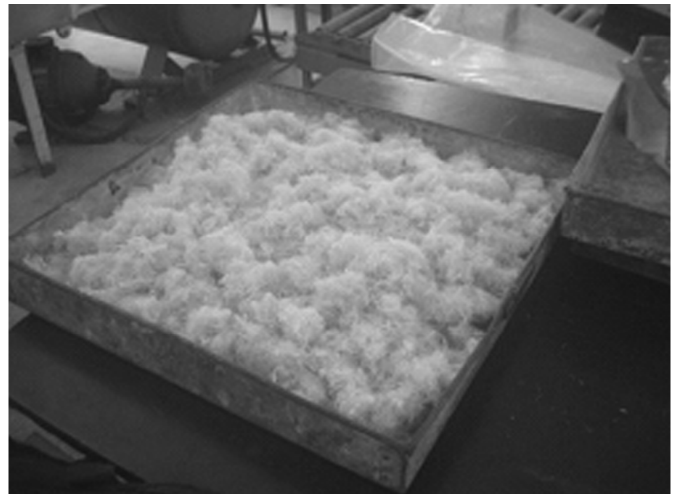

Figure 2: Dried and chopped (3mm- 4mm) short sisal fiber.

fiber and polymer matrices. The presence of wax, oil, and surface impurities provide an insulative layer on the surface of the fiber led to a weak interfacial bond leading to failure [10]. Sisal fiber also contains 70% cellulose, 12% lignin for the diameter of 100 µm to 300, fiber of density 1.45 gm/ cm3.Therefore surface treatment to sisal fiber with NaOH was done to modify the surface of the fiber that enhances adhesion strength to the fiber matrices interface. For surface treatment, sisal fibers were immersed in 5% NaOH solution at room temperature for 2 hrs. Further fibers were washed with distilled water for several times to remove NaOH, which was sticking on fiber surface and then a few amount of acetic acid was added to the fiber to adjust the pH value in between 6-7. After that fibers were taken out and dried for 3 days at room temperature. And then dried at 65 o C for 24 hrs in air circulating oven. Dried and treated long sisal fiber is shown in fig-1.Then the treated fibers are chopped to the length about 3 mm to 4 mm as discontinuous fibers. Chopped short sisal fiber is shown in fig-2. And finally stored in an airtight bags. The surface treatment of Sisal fiber was carried out as per the reported procedure [11, 12].

2.3. Fabrication of hybrid bio composite

Fabrication of Basalt/Sisal hybrid fiber reinforced bio composite consists of two stages. In the first stage blending of Basalt/Sisal fiber with PLA is carried out in Twin Screw Extruder to form the hybrid compound as granules. And in the second stage preparation of hybrid bio composite specimen is carried out using Injection Moulding.

2.3.1. Preparation of hybrid compound

Dried Virgin PLA and dried, chopped and treated Basalt and sisal fibers were mixed in three different combinations named as PBaSi-1, PBaSi-2, and PBaSi-3with the ratio of 90/5/ 5 wt% as PBaSi-1, 85/7.5/ 7.5 wt % as PBaSi-2, and 80/10/10 wt % as PBaSi-3 respectively. And then this mixture is feed in to high speed co- rotating Twin Screw Extruder (as see in Fig-3) for blending and to make homogeneous mix. Screw diameter of 28 mm, L/D ratio of 40, contains 5 different heat zones from feed point to exit point .at various heat temperatures 130°C ,140°C, 150°C and 165°C, respectively. The screw speed is 150 rpm. Homogeneous mixing of PLA matrix and Basalt/Sisal fiber were carried out by twin screw extruder for 15 min and then extruded at the rate of 10 mm/ sec through a 1 mm gauge strands die. Strands were cooled in a water bath and then fed in to pelletizer to make compound pellets. Compounded pellets were dried at 60°C in a vacuum for 12 hrs and stored in an air tight bags.

Figure 3: Twin Screw extruder–process of hybrid fiber bio composite preparation

2.3.2. Preparation of hybrid composite specimens

The dried and compounded pellets which were produced by twin screw extruder is again processed by Injection moulding at a temperature of 170°C, back pressure of 7 bar, screw speed of 60 mm/ sec and mould temperature of 30°C. Injection moulding machine screw diameter 30 mm with L/D ratio of 20 is used to obtain hybrid composite specimen. Test specimens for mechanical test such as tensile, flexural and impact test specimens were produced as per ASTM D638, ASTM D790 and ASTM D256 standards

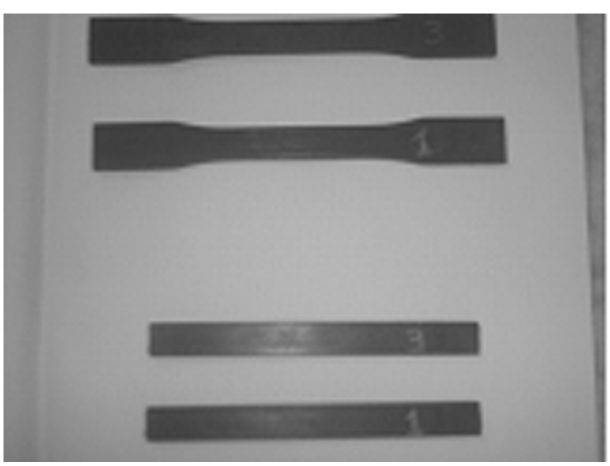

Figure 4: Tensile, flexural and impact test specimens of PBaSi composites.

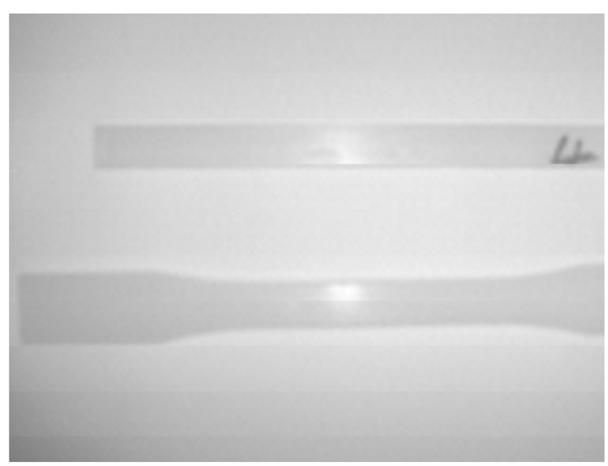

Figure 5: Test specimens of virgin PLA

respectively. The test specimens were subjected to annealing at a temperature of 80°C in an oven for 24 hrs. Tensile, flexural and impact specimens of hybrid composites and virgin PLA were shown in the fig- 4 and fig-5 respectively.

2.4. Mechanical testing

2.4.1. Tensile, flexural and impact test

Test specimens as per ASTM standards for tensile, flexural and impact tests were used. The Tensile properties of PBaSi-hybrid composites were measured for three categories according to ASTM D 638 standards.

The flexural properties of PBaSi-hybrid composites were measured using UTM machine (Instron machine model-5564) according to ASTM D 790 standards. Five samples of flexural specimen of each category were tested and the mean, standard deviation and standard error value were calculated.

The Izod impact strengths of PBaSi-hybrid composites were measured using EMIC pendulum machine according to ASTM D 256 standards. Five samples of impact specimen were tested and the mean standard deviation and standard error values were calculated.

2.5. Thermal Characterization

2.5.1. Differential scanning calorimeter (DSC)

The thermal properties like glass transmission temperature Tg, crystallization temperature Tc, enthalpy of crystallization temperatureH c (J/g), melting temperature Tm, heat of melting H m and degree of crystalinity Xc of hybrid composite were investigated using DSC (TA instrument Q20V 24-11 Build 124) in a Nitrogen atmosphere by purging gas 50ml/min. Sample of about 2 mg of PBaSi-1, PBaSi-2 and PBaSi-3 Hybrid composites were scanned from 40°C to 300°C at the heating rate of 10°C/min. DSC thermo gram of the hybrid composite PBaSi-1, PBaSi-2 and PBaSi-3 are given in fig 9.

2.5.2. Dynamic mechanical analyses (DMA)

Dynamic mechanical analysis (DMA) was performed by DMA Q800 V20.6 Build 24 instrument to examine the usability of the developed composites in the temperature range of 0–160°C and especially in the critical range above glass transition temperature (Tg). The properties measured are storage modulus and loss modulus. Tests were performed using dual cantilever geometry over samples with 35.00 × 13.48 × 3.24 mm dimensions. The distance between the clamps was 10mm and strain of 1Hz frequency and 20μm amplitude was used. Heating ramp from ambient temperature to 180°C was carried at the rate of 10°C/min.

2.6. Morphological study of Hybrid composite.

2.6.1. Scanning Electron Microscope: SEM Analysis

To investigate the distribution of Basalt/Sisal Hybrid fiber in the polymer (PLA) matrix and to find the nature of interaction with each other, the Scanning Electron Microscope (SEM - JEOL JSM 6610 LV) was used. The samples were coated with thin layer of gold before scanning observation, to avoid electrostatic charging and to increase the conductivity of the samples during scanning. The results of SEM micrograph for hybrid composites PBaSi-1, PBaSi-2 and PBaSi-3 are depicted in Fig: 12-14 respectively.

3. RESULT AND DISCUSSION

3.1. Effect of Basalt/Sisal hybrid fiber content on Mechanical properties of "PBaSi" composites

It is inferred from the Table-1 the flexural strength of PBaSi-3 composite (having 10 wt% of sisal) is 59.10 MPa, which is significantly higher than the value of 30 wt% sisal with UF combination of composites, made by Yihe Zhang et.al in his research on "Mechanical and Thermal properties of basalt fiber reinforced poly (butylenes) composites(see Table-2). Hence in this present study we are achieved saving of 20 wt % of sisal fiber in PBaSi-3 composite. This result suggests that high strength with low weight and low cost hybrid composite PBaSi-3 is suitable for automotive products. The flexural strength of PBaSi composites are shown in fig 6.

Tensile strength of PBasi-3 hybrid composite is noted from the Table-1 is 55.70 MPa. It is often observed that the increase in fiber loading leads to an increase in tensile properties [13-14].

Kuruvilla Joseph et.al (See Table-2) in his study on "A Review on sisal fiber reinforced polymer composites" he noted that for 10 wt% of sisal fiber with polymer composite gives maximum tensile strength of 36 M Pa. Hence our present study has significantly improved tensile strength of hybrid composite PBaSi-3.Hence it is clear that hybrid fiber reinforcement of PLA composites have high mechanical performance with low weight and low cost of product. The tensile strength of PBaSi composites are shown in fig 7.

The izod impact strength of PBaSi composites are shown in fig 8. When PLA matrix reinforced with 20% wt of basalt/sisal hybrid fiber, the impact strength is 2.63 k j/m2 further reduction of hybrid fiber content, decreases the impact strength of the composites. This is due to the addition of basalt /sisal fiber to PLA matrix increases the toughness.

Table 1
Mechanical Properties Of Basalt/Sisal Hybrid Fiber Reinforced "Pbasi" Composites.

Hybrid Bio-composite	*Tensile strength (M Pa) ASTM D 638*	*Flexural strength (M Pa) ASTM D 790*	*Impact strength (KJ/m2) ASTM D 256*
PBaSi-1	21.02	43.70	2.14
PBaSi-2	33.42	56.50	2.54
PBaSi-3	55.70	59.10	2.63

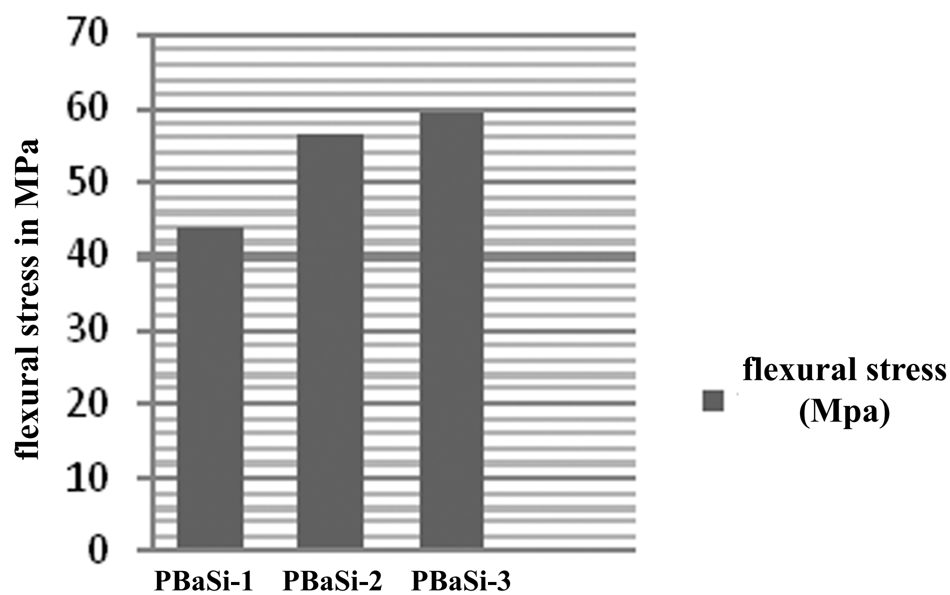

Figure 6: Analysis of flexural stress for different hybrid fiber content.

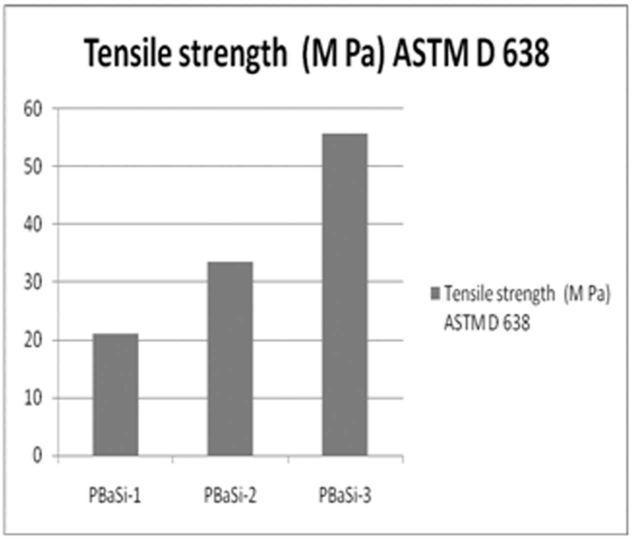

Figure 7: Analysis of tensile stress for different hybrid fiber content.

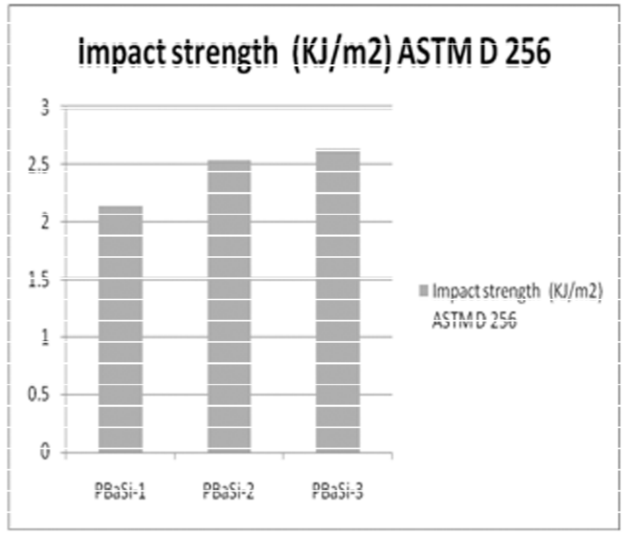

Figure 8: Analysis of impact strength for different hybrid fiber content.

Table 2
Mechanical Properties of Polymer Composites With Different Fiber Content.

Fiber content %	*Composite type*	*Tensile strength (MPa)*	*Flexural strength (MPa)*	*Impact strength (KJ/m2)*	*Reference*
Sisal 10 wt %	PP PS PE	36.0 21.3 15.61			Kuruvilla Joseph et.al on "A Review on sisal fiber reinforced polymer composites".
Basalt 3 wt % 15 wt %		31.0 46.0			Yihe Zhang et.al on "Mechanical and Thermal properties of basalt fiber reinforced ply (butylenes) composites.
Basalt 3 wt %	PBS		18.0		
15 wt %			71.0		
Basalt 3 wt %				2.9	
5 wt %				4.0	
Sisal 30 wt %	UF		58.58		J.B. zhong et.al on hid "Mechanical properties of sisal fiber reinforced urea formaldehyde resin composite.

3.2. Effect of hybrid fiber content on Thermal Properties of "PBaSi" composites: – DSC

Thermal properties such as glass transition temperature Tg (°C), crystallization temperature Tc (°C), enthalpy of crystallization temperature ΔHc (J/g), melting temperature Tm (°C), heat of melting ΔHm (J/g) and degree of crystallinity Xc (%) of hybrid bio composites obtained from the DSC test were tabulated in Table 2. The degree of crystallinity was calculated based on the following equation (1)

$$X_c\% = \frac{\Delta Hm}{f\,\Delta H_m^o} \times 100 \tag{1}$$

Where ΔHm is the heat of melting (J/g) of test specimen composites and ΔHmo is the heat of melting for 100% crystalline PLA taken as 93.1 J/g and 'f' as weight fraction of PLA from the literature [15, 16]. The results of DSC scans of hybrid fiber composites "PBaSi-1, PBaSi-2 and PBaSi-3"were illustrated in table 3.

Table: 3
Thermal Properties From The DSC Scan For Basalt/Sisal Hybrid Fiber Reinforced "Pbasi" Bio Composites:

Specimens	Tg(o C)	Tc(o C)	ΔHg(J/g) exo	Tm(o C)	ΔHm(J/g) endo	Xc(%)
Virgin PLA	60	–	–	160	36	38
PBaSi-1	67	72	17	160	59	79
PBaSi-2	65	71	65	159	69	80
PBaSi-3	63	68	20	164	72	89

From the thermo gram fig. 9 for PBaSi-1 composite we can observe that three peaks corresponding to Tg, Tc and Tm at temperatures 67°C, 72°C, 160°C, where as for PBaSi-2 composite exhibited three peaks corresponding to Tg, Tc and Tm at 65°C, 71°C, 164°C,.and for PBaSI-3 t is 63°C, 68°C, 159°C.

It was observed that the Tg values of PBaSi hybrid composites were increased to 63°C, 65°C and 67°C compared to the Tg of virgin PLA-60°C. Sandi et al. reported that a higher glass transition temperature for lower fiber content was obtained due to better adhesion[17].This interaction had been enhanced by hybrid fibers, which in turn caused an increased value of Tg for PBaSi composites. This substantiates PBaSi composites further reduces the macromolecular mobility through strong adhesion between PLA and hybrid fibers. Resulting in the greater improvement in thermal properties.

This was well supported by SEM micrographs. For all above composites, the melting endothermic peaks indicate that the inclusion of hybrid fiber in virgin PLA change the melting temperature significantly. The Tm of the PBaSi-3 bio composite slightly shifted to a higher temperature, indicating its effective heat transfer. It was also interesting to observe that the % of crystallinity of "PBaSi" composite was significantly increased compared with vergin PLA. Mathew et al. reported similar observation in which the fibers promoted crystallinity in PLA as a consequence of crystalliite growth being initiated at the treated fiber surface. [12-14]

Crystallinity of PBaSi-3composite is 89% which is 13% higher than other PBaSi composites. This is probably due to the enhance fiber matrix interaction and improve fiber stiffness. Hence PBaSi-3 composites have improved thermal stability and high stiffness than other natural fiber composites.

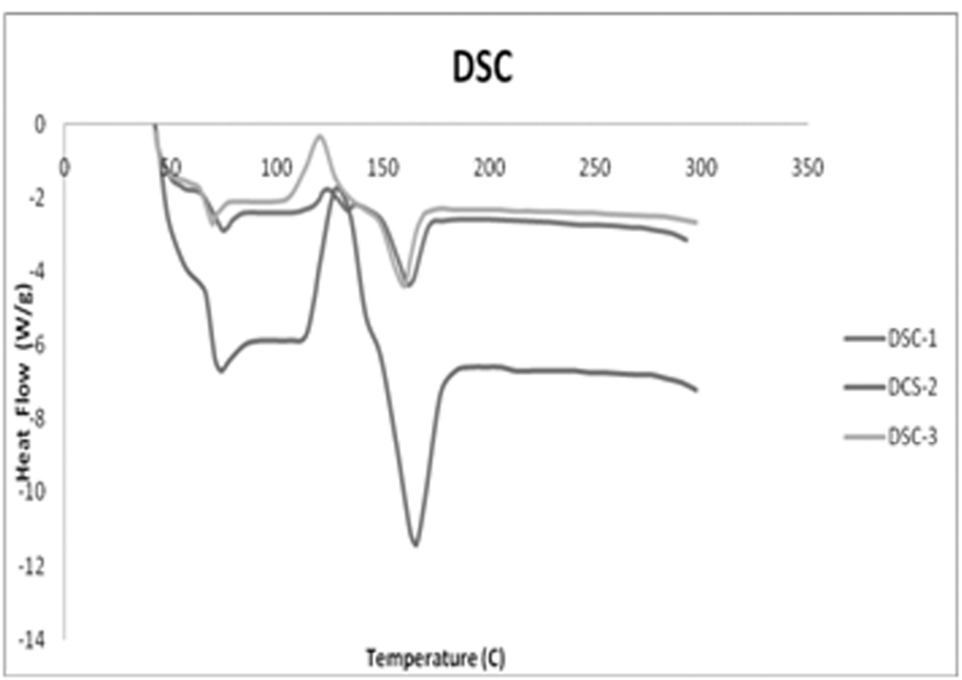

Figure 9: DSC results for hybrid fibers reinforced PBSi composite.

3.3. Effect of hybrid fiber content on Thermal Properties of "PBaSi" composites: – DMA

Dynamic mechanical analysis (DMA) was performed to examine the usability of the developed composites in the temperature range of 25–170°C and especially in the critical range above Tg. Due to the slow crystallisation of PLA, it remains mostly amorphous during injection moulding, thus the dramatic drop in storage modulus at Tg. Naturally the basalt fibres increase the storage modulus. Table 4 shows the storage modulus, loss modulus and glass transition temperature (Tg) of hybrid composites.

It is inferred from the fig-10 storage modulus of PBaSi-3 having 20 %wt basalt /sisal fiber shows 2779 MPa, where as it reduces in PBaSi-2 and PBaSi-1 having lowest basalt/sisal fiber weight percentage. High storage modulus is noted in the temperature range of 45 - 55°C. The storage modulus of PBaSi-3 increases about 16% when compared to PBaSi-1.This shows PBaSi-3 having better mechanical properties with respect to rise in temperature.

The loss modulus is more in PBaSi-1 and PBaSi-2 compared to PBaSi-3,which indicates less energy loss occurred on PBaSi-3 compared to other composites. Based on this DMA analysis it is suggested that PBaSi-3 hybrid fiber composites are having better mechanical properties and less energy losses with respect to temperature.

3.4. Morphological study of fractured surface of PBaSi composites

Surface morphology of hybrid composites are analyzed with fractured flexural specimen and fractured impact specimens.SEM photograph of flexural and impact fractured composites are shown in Fig12-15 respectively.

Table 4
The Effect of Fiber Content (WT %) on Storage Modulus of Hybrid Fibers Reinforced PBSI Composites

Sl. no	*Composite specimen*	*Glass transsion temperature (Tg)*	*Storage modulus (M Pa)*	*Loss modulus (M Pa)*
1	PBaSi-1	71.00	2334	385.9
2	PBaiS-2	71.00	2616	391.5
3	PBaSi-3	71.88	2779	325.2

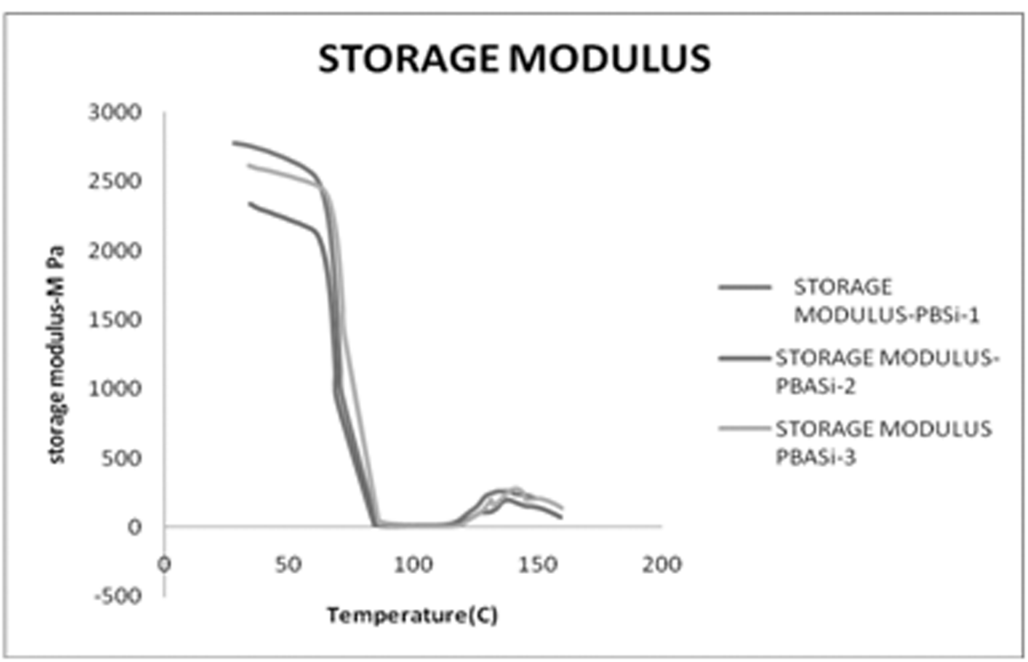

Figure 10: The Effect of fiber content on Thermal Properties of "PBSi" composites: – DMA:

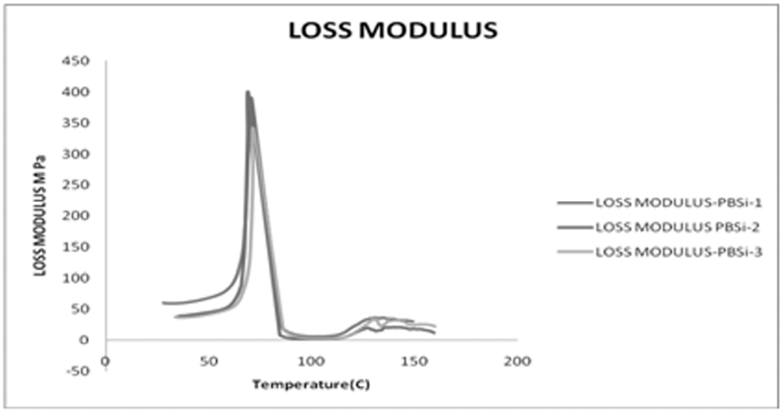

Figure 11: The effect of fibre content (wt%) on loss modulus of " PBaSi" composites

Flexural fracture surface SEM micro graph of PBaSi-1, PBaSi-2 and PBaSi-3 composites are given in Fig: 12-14respectively. These micro graphs show that the fibers were well dispersed in the PLA matrix. The fibers were covered with a thin layer of matrix linking the fiber surface to the matrix and thus better stress transfer could be expected. This effect reflected in the mechanical properties ofPBaSi -3 composite.

In Fig 15 the impact fracture surface SEM micrograph of PBaSi-3composite shows that the surfaces of the basalt fiber become very smooth and the PLA matrix surface also very smooth .we observe that there is

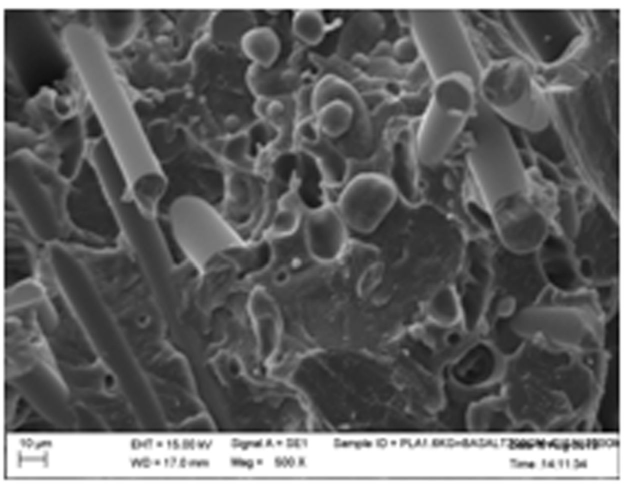

Figure 12: Flexural specimen PBaSi-1 fracture surface

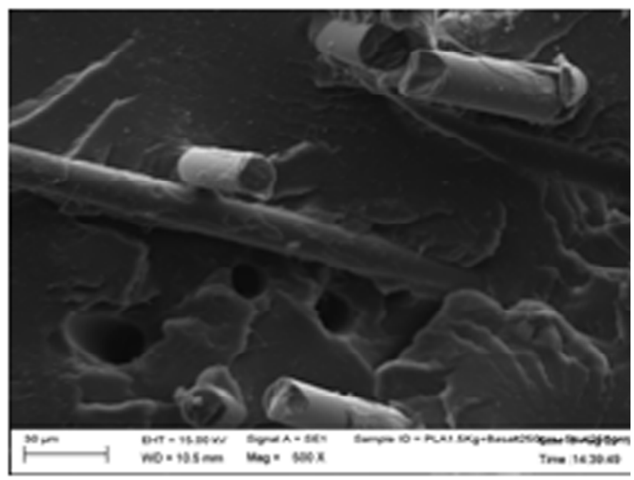

Figure 13: Flexural specimen PBaSi-2 fracture surface

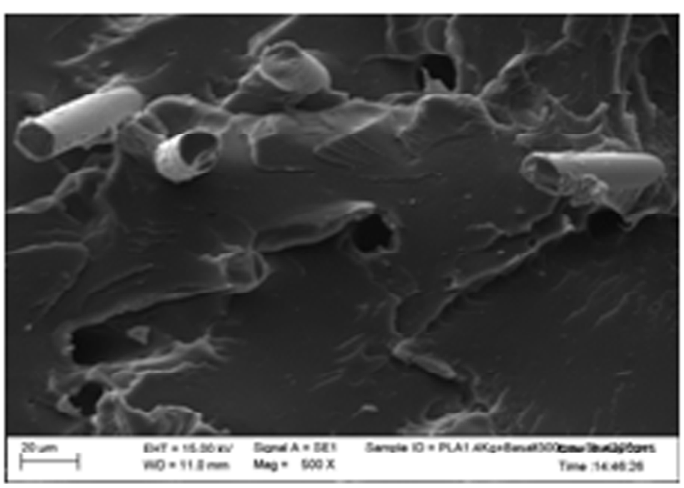

Figure 14: Flexural specimen PBaSi-3 fracture surface

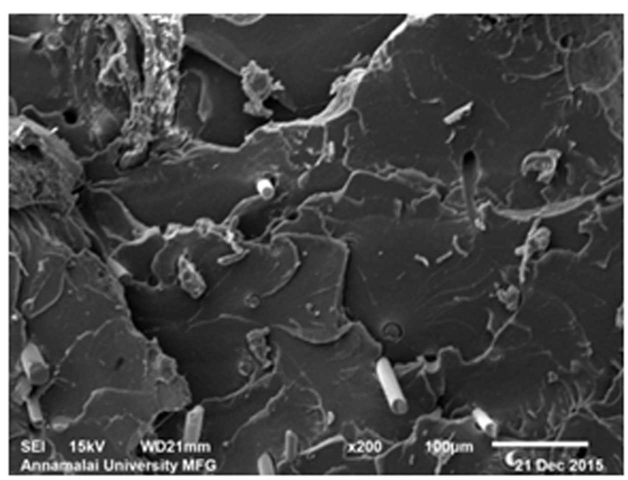

Figure 15: Fracture surface of impact specimen PBaSi-3

no gap between the fiber and the matrix. This may offer excellent fiber-matrix interface adhesion and improve stress transfer. These will give rise to improvement in mechanical properties.

4. CONCLUSIONS

This study concludes that treated Basalt/sisal fibers and its subsequent reinforcement with PLA matrix reveal sensible mechanical properties and gave higher values tensile, flexural and impact strength for "PBaSi" composites. The presence of 20 wt% Basalt/sisal hybrid fiber in the composite PBaSi-3 has significantly augmented mechanical and Thermal properties. The SEM micrographs of "PBaSi" composites clearly indicate that the nature of bonding established between fiber and matrix was physico - chemical. Due to the low density and high specific properties of these hybrid fiber composites it may have very good implication in the automobile and transportation industry.

Thus, we conclude that the systematic and protracted analysis of this present study may pave an avenue to an increased scope and future for basalt/sisal reinforced PLA composite for intended end use of applications.

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