JOURNAL OF MATERIALS SCIENCE (2005)

The effect of fibre content on the mechanical properties of hemp and basalt fibre reinforced phenol formaldehyde composites

SULTAN ÖZTÜRK
Beşikdüzü Technical College,Karadeniz Technical University, Trabzon, Turkey
E-mail: suozturk@ktu.edu.tr

In this study, mechanical properties such as tensile, flexural and impact strengths of hemp/phenol formaldehyde (PF), basalt/PF and hemp/basalt hybrid PF composites have been investigated as a function of fibre loading. Hemp fibre reinforced PF composites and basalt fibre reinforced composites were fabricated with varying fibre loading i.e. 20, 32, 40, 48, 56 and 63 vol%. The hybrid effect of hemp fibre and basalt fibre on the tensile, flexural and impact strengths was also investigated for various ratio of hemp/basalt fibre loading such as 1:0, 0.95:0.05, 0.82:0.18, 0.68:0.32, 0.52:0.48, 0.35:0.65, 0.18:0.82 and 0:1. Total fibre loading of the hybrid composites was 40 vol%. The results showed that the tensile strength and elongation at break increase with increasing fibre loading up to 40 vol% and decrease above this value for hemp fibre reinforced PF composite. Similar trend was observed for flexural strength and the maximum value was obtained for 48 vol% hemp fibre loading. Impact strength of hemp/PF composite showed a regular trend of increase with increasing fibre loading up to 63 vol%. Tensile strength, flexural strength and impact strength values of basalt/PF composites were found to be lower compared to hemp/PF composites. The tensile strength and elongation at break of basalt/PF composite increased by incorparation of basalt fibre up to 32 vol% and decreased beyond this value. Flexural strength of basalt/PF composite decreased linearly with fibre loading. However, the maximum impact strength was obtained for 48 vol% basalt fibre loading. For hemp/basalt hybrid PF composite, the tensile strength decreased with increasing basalt fibre loading. On the other hand, the flexural and impact strengths showed large scatter. The maximum flexural strength value was obtained for 0.52:0.48 hemp/basalt ratio. Corresponding value for impact strength was obtained for 0.68:0.32 hemp/basalt fibre ratio.

1. Introduction

Manufacturing high performance engineering materials from renewable resources is one of the ambitious goal currently being pursued by researchers across the world [1]. Eco-friendly green composites from plant derived fiber and crop-derived plastics are novel materials for the 21th century and can be of great importance to the materials world [2]. Renewable resources in the field of fibre reinforced materials with their new range of applications represent an important basis in order to fulfill the ecological objective of creating closed material circuits [3]. The interest in using natural fibres such as different plant fibres and wood fibres as reinforcement in plastics has increased dramatically during last few years. With regard to the environmental aspects it would be very interesting if natural fibres could be used instead of glass fibres as reinforcement in some structural applications [4, 5]. Plant fibres are renewable and biodegradable natural cellulosic materials. Together with their desirable mechanical properties, they are becoming increasingly useful as raw materials for the preperation of cost-effective and environmentally friendly composite materials [6–8]. The advantages of natural plant fibres over traditional glass fibres are low density, good mechanical performance, unlimited availability, reduced tool wear, low cost, reduced energy consumption and biodegradability [2, 3, 9]. Today, a renaissance in the use of natural fibres as reinforcements in the technical applications is taking place mainly in the automobile industry [10]. Auto companies are seeking materials with sound abatement capability as well as reduced weight for fuel efficiency. Natural fibers possess excellent sound absorbing efficiency and are more shatter resistant and have better energy management characteristics than glass fiber reinforced composites. While natural fibers can deliver the same performance for lower weight, they can also be 25–30 percent stronger for the same weight. Moreover, they exhibit a favorable non-brittle fracture on impact, which is another

0022-2461 © *2005 Springer Science + Business Media, Inc.*
DOI: 10.1007/s10853-005-1103-z

important requirement in automobile industry [2, 11, 12].

Hybrid composites are materials made by combining two or more different types of fibres in a common matrix. They offer a range of properties that can not be obtained with a single kind of reinforcement. Research revealed that the behaviour of hybrid composites appears to be simply a weighed sum of the individual components in which there is a more favorable balance between the advantages and disadvantages inherent in any composite material. It is generally accepted that the properties of hybrid composite are controlled by factors such as nature of matrix; nature, length and relative composition of the reinforcements; fibre-matrix interface; and hybrid design etc. By using hybrid composites made of natural fibres and carbon-fibres or natural fibres and glass-fibres, the properties of natural fibre reinforced composities can be improved further [10–12].

Natural fibres are in general suitable to reinforce plastics (thermosets as well as thermoplastics) due to their relative high strength and stiffness and low density. The use of natural fibres such as hemp will result in significant property advantages as compared to wood-based fibres such as wood flour, wood fibres and recycled paper. Hemp is an important ligno-cellulosic natural fibre and has the best mechanical and thermal properties [2, 6, 10].

The present paper establishes the mechanical performance of the hemp/ phenol formaldehyde (PF) composite, the basalt/PF composite and hemp/basalt hybrid PF composites. Tensile properties such as tensile stress-strain behavior, tensile strength and elongation at break of the composites as a function of volume fraction of the fibres were analysed. The flexural strength and impact strength as a function of fibre loading were also investigated. The tensile and impact fracture mechanism of the composites were studied by scanning electron microscopy (SEM).

2. Experimental

2.1. Materials

Phenol formaldehyde novalac type resin was obtained from Cukurova Ltd. Manisa, Turkey. The basic properties of the resin were given in Table I [13]. Hemp fibre was obtained from local sources and were cut to length of 5–10 mm. An approximate chemical analysis of hemp fibre is cellulose (70.2–70.4%), lignin (3.7–5.7%), hemicellulose (17.9–22.4%), pectin (0.9%), wax (0.3%) and moisture (10.8%) [2]. Basalt fibre was obtained from Deutsche Basaltsteinwolle GmbH, Rotedal Germany. An approximate chemical analysis of basalt fibre is SiO_2 (46.2%), Al_2O_3 (13%), Fe_2O_3 (12%), MgO (10%), CaO (10%), TiO_2 (2%), K_2O (1.8%), Na_2O (3.5%) and traces (<1%) [14, 15]. Fig. 1 shows SEM micrographs of hemp and basalt fibres. Important physical and mechanical properties of hemp and basalt fibres are given in Table II [7,16,17].

TABLE I Physical and mechanical properties of phenol formaldehyde novalac resin [13]

Density (kg/m^3)	1.27
Compression strength (MPa)	235–520
Tensile strength (MPa)	34–62
Young's modulus (GPa)	2.8–9.0
Elongation at break (%)	0–2
Melting point (°C)	87
Powder size (μm)	45

TABLE II Physical and mechanical properties of hemp and basalt fibre [7, 16, 17]

Fibre	Density (kg/m^3)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)	Diameter (μm)
Hemp	1.48	550–900	70	1.6	20–200
Basalt	2.70	430	60	1.2	13

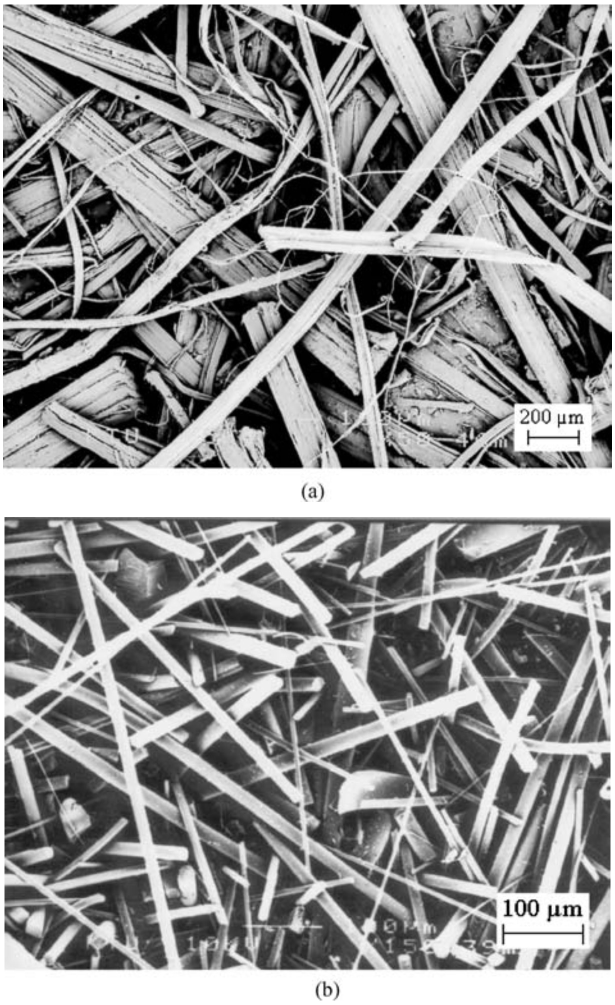


Figure 1 SEM micrographs of fibres. (a) hemp fibre, (b) basalt fibre.

2.2. Sample preparation

Composite sample preparation was performed by mixing the fibres and resin in a mixing chamber for 5 min. The composites were made by pressing the materialin a steel mould at 120 °C for 15 min at 9 MPa pressure. At least five specimens were tested and average value was taken for each composite according to ASTM standards. Hemp fibre reinforced-PF resin composites and basalt fibre reinforced-PF resin composites with various fibre loadings such as 20, 32, 40, 48, 56 and 63 vol% were prepared. Effect of fibre loading on the mechanical properties was analysed. Hybrid composites having various ratio of hemp fibre/basalt fibre loading such as 1:0, 0.95:0.05, 0.82:0.18, 0.68:0.32, 0.52:0.48, 0.35:0.65, 0.18:0.82 and 0:1 were prepared. Total fibre loading for hybrid composites was 40 vol%.

2.3. Mechanical tests

Test samples were cut from composite sheets. Tensile tests were carried out using a Trapezus 2 model tensile testing machine in accordance with ASTM D 638 standard. The samples were tested at a rate of 5 mm/min. Flexural tests were performed using the 3-point bending method according to ASTM D 790 standard. Impact tests were carried out on a charpy impact testing machine and the test method adopted was consistent to ASTM D 256 standard. All the test samples were unnotched. Impact loading was done with a 7.5 J hammer. The impact strength was calculated by dividing the recorded absorbed impact energy with the cross-sectional area of the samples.

2.4. Morphology of fracture surfaces

Hemp fibre, basalt fibre and fractography of the failure surfaces of the composite samples was examined by scanning electron microscope, Jeol 3600 model. The tensile and impact fractographs of the composites were taken to study the fracture mechanism and interface adhesion of the composites.

3. Results and discussion

3.1. Tensile strength

The stress-strain behavior of hemp fibre/PF composite at varying fibre loading is shown in Figs 2 and 3. It can be clearly seen that tensile strength and elongation at break increased with increasing fibre content up to 40 vol% and decreased above this value. It is well known that [18] the tensile strength of a composite material is mainly dependent on the strength and modulus of the fibres, the strength and chemical stability of the matrix and the effectiveness of the bonding strength between matrix and fibres in transferring stress across the interface. When fibre reinforced composite is subjected to load, the fibres act as carriers of load and stress is transferred from matrix along the fibres leading to effective and uniform stress distribution which results in a composite having good mechanical properties [8]. At low levels of fibre loading, fibres are not capable of transferring load to one another and stress gets accumulated at certain points of composite leading to low tensile strength. At high levels of fibre loading the increased population of fibres leads to agglomeration and stress transfer gets blocked. At intermediate levels of loading the fibres actively participate in stress transfer. In this study maximum tensile strength and elongation at break were obtained at 40 vol% hemp fibre loading. The tensile strength and elongation at break are found to have 121 and 78% increase with increasing hemp fibres from 20 to 40 vol% (Fig. 2). (It can be concluded that the brittle nature of the composite decreases with the addition of hemp fibre.) However, further increase in weight fraction of hemp fibre from 40 % to 63 vol% decreases the tensile strength and elongation at break to about 38 and 6.6%, respectively. This may be attributed to increasing of unwetted hemp fibre by PF and poor fibre-matrix adhesion with increasing fibre loading. However elongation increases again between 56 to 63 vol% fibre loading which may be explained in terms of increasing fibre pull out. The tensile fractograph of hemp/PF composite containing 40 wt% fibre loading was shown in Fig. 4. The wetting of hemp fibre by matrix and the interfacial adhesion between the fibre and matrix were not strong and predominant fracture mechanism was fibre pull-out.

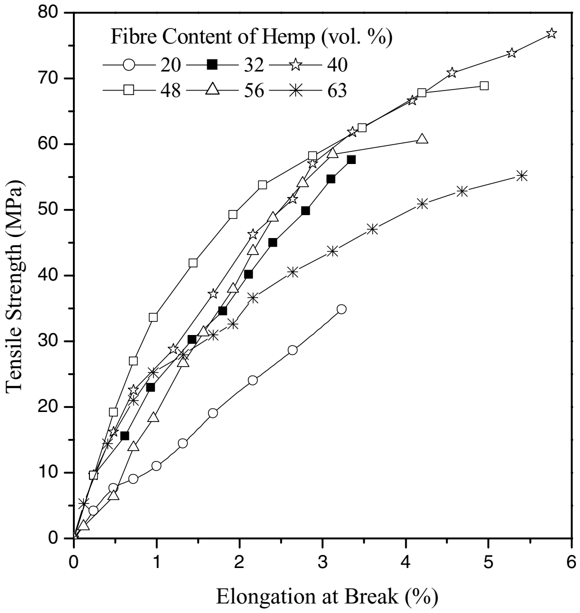


Figure 2 Effect of hemp fibre loading on the tensile strength of hemp/PF composite.

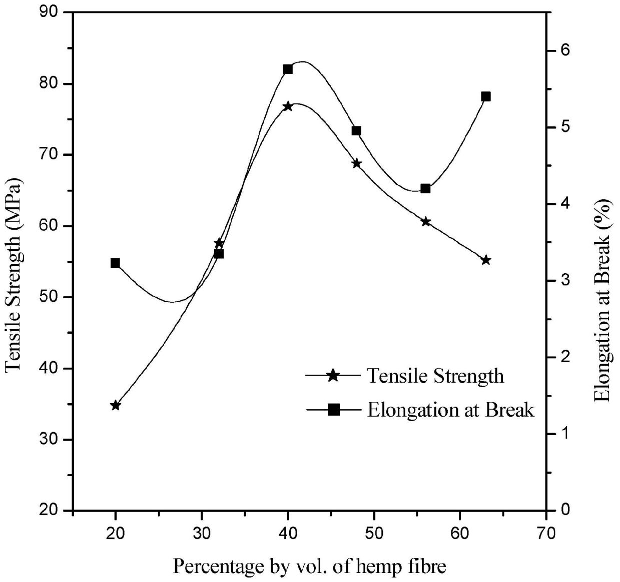


Figure 3 Effect of hemp fibre loading on the tensile properties of hemp/PF composite.

Figure 4 Tensile fracture surface of hemp fibre/PF composite (fibre loading 40 vol%).

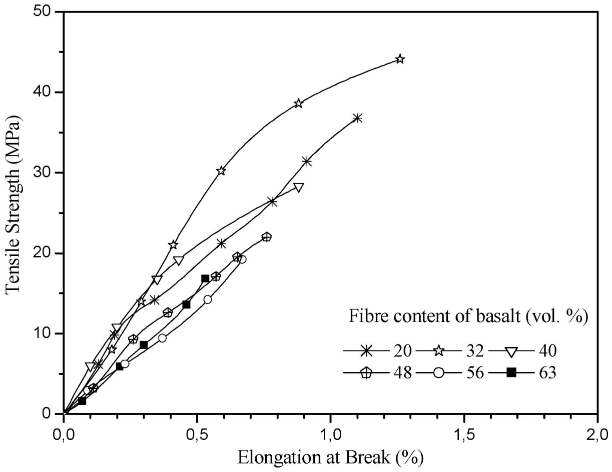


Figure 5 Tensile stress-strain behavior of basalt/PF composites having different basalt fibre loading.

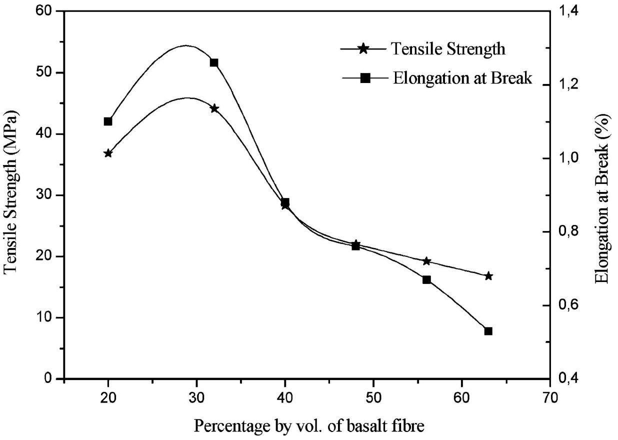


Figure 6 Effect of basalt fibre loading on the tensile properties of basalt/PF composites.

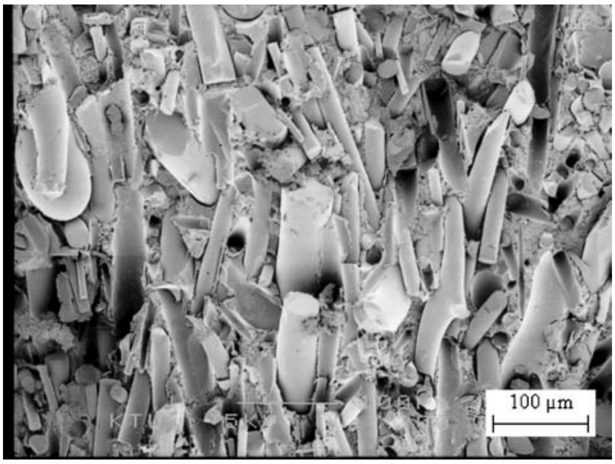


Figure 7 Tensile fracture surface of basalt/PF composite (fibre loading 40 vol%).

Figs 5 and 6 show the variation of tensile strength and elongation at break with basalt fibre loading in basalt/PF composite. Fig. 5 clearly shows that the tensile strength and elongation at break of basalt/PF composite increased by incorporation of basalt fibre up to 32 vol%. The tensile strength of the composite at 32 vol% basalt fibre loading is found as 44.1 MPa. However higher strength values were expected considering the strength of the basalt fibre itself. This may be due to poor bonding between inorganic basalt fibre and PF matrix. This is illustrated in SEM images (Fig. 7) which shows fibre-PF matrix debonding and clean fiber surface. In fact these fibers were manufactured for the purpose of brake pad manufacturing. The tensile strength and elongation at break values are observed to decrease by addition of basalt fibre from 32 to 63 vol%. For the 63 vol% basalt fibre loading, the tensile strength of composite is found as 16.8 MPa. This is probably due to lost of integrity of the matrix due to high fibre loading. Botev *et. al.* [14] reported that decreasing tensile strength with addition 10, 20 and 30 vol% basalt fibre to polypropylene matrix. Similar behavior for the percentage elongation in basalt/PF composite was observed as shown in Fig. 6. The values of elongation at break also decreased with the increasing of fibre loading.

The tensile fractograph of basalt/PF composite containing 40 vol% fibre loading is shown in Fig. 7. As can be seen, fibre-fibre contact points and fibre pull out are clearly observed. The clear surface of fibres shows poor wetting of fibres by matrix and poor interfacial bonding between fibres and matrix. Predominant fracture mechanism is fibre pull out.

The hybrid effect of hemp and basalt fibres on the tensile strength and elongation at break of hemp/basalt hybrid PF composite having 40 vol% fibre loading is presented in Figs 8 and 9. As can be seen from figures, increasing weight fraction of basalt fibre resulted negative effect on the tensile properties of hemp fibre but the values found higher compared to basalt/PF composite. The properties of any hybrid composite are mainly dependent on the modulus and percentage elongation at break of the individual reinforcing fibres. The modulus and elongation at break of hemp fibre are comparatively higher than that of basalt fibre (see Table I and II). This suggests that the tensile properties of hemp/basalt hybrid PF composite are dependent more on the weight of hemp fibre rather than basalt fibre. Tensile strength and elongation at break values of the hybrid composite at 5 vol% basalt fibre loading are 65.3 MPa and and 4.6%, respectively. Corresponding values at 82 vol% basalt fibre loading are 33.5 MPa and 2.7%, respectively. Fig. 10 shows SEM micrographs of tensile frac-

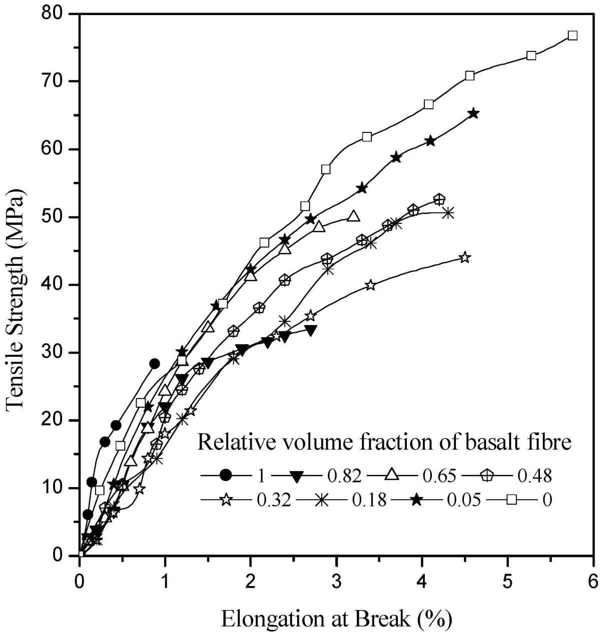


Figure 8 Effect of basalt fibre loading on the tensile strength of hemp/basalt hybrid PF composite (total fibre content: 40 vol%).

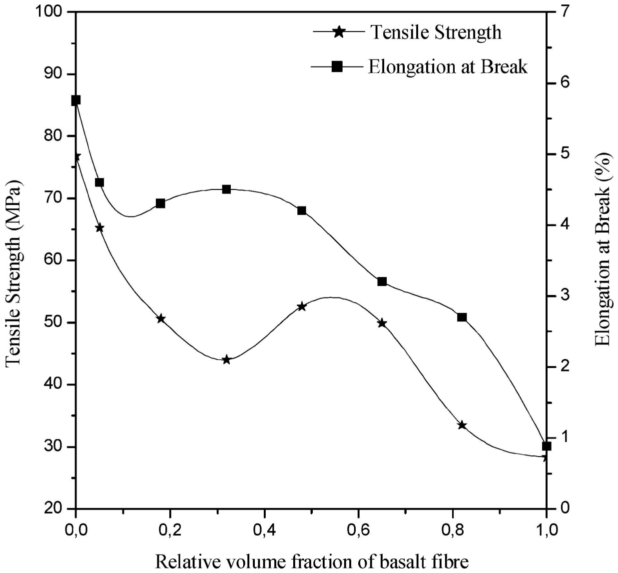


Figure 9 Effect of basalt fibre loading on the tensile properties of hemp/basalt hybrid PF composite (total fibre content: 40 vol%).

tured surface of hemp/basalt hybrid PF composites for varying hemp/basalt ratios.

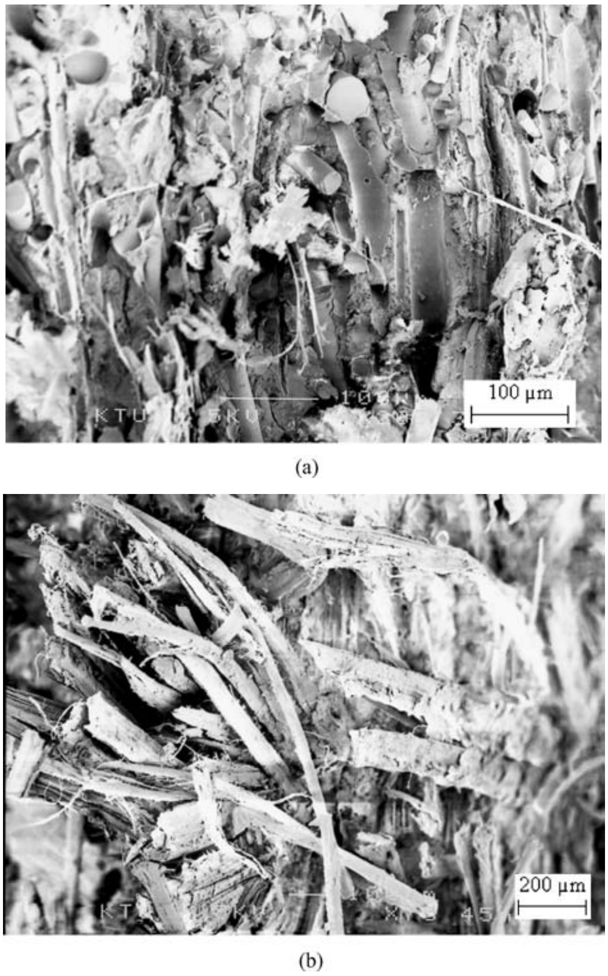


Figure 10 SEM micrographs of tensile fractured surfaces of hemp/basalt hybrid PF composite. (a) Hemp/basalt ratio: 0.18:0.82, (b) hemp/basalt ratio: 0.95:0.05.

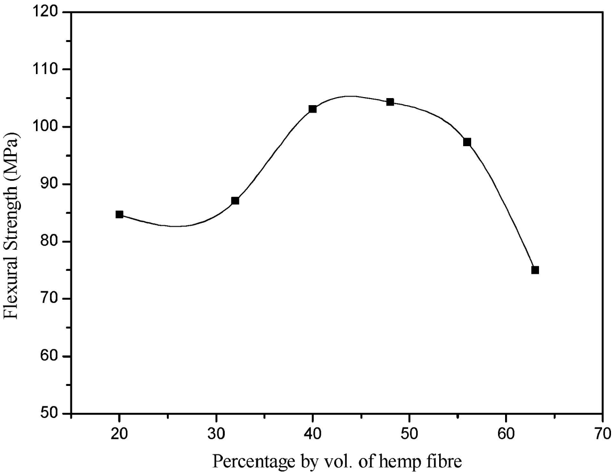


Figure 11 The flexural strength of the hemp fibre/PF composite as a function of fibre loading.

3.2. Flexural strength

The effect of hemp fibre loading on the flexural strength of hemp/PF composite is given in Fig. 11. There is maximum flexural strength at 48 vol% hemp fibre loading. The flexural strength at 20 vol% fibre loading is found as 84.7 MPa. By addition of 48 vol% hemp fibre, the flexural strength increased to 104.3 MPa. Further addition of hemp fibre from 48 to 63 vol% decreased flexural strength. In flexural testing various mechanism takes place simultaneously. By application of flexural force, the upper and lower surface of the sample under three point bending load is subjected to compression and tension and axisymetric plane is subjected to shear stress. So there are two failure modes in the materials; bending and shear failure. The sample fails when bending or shear stress reaches the corresponding critical value [4]. The loading of 48 vol% hemp fibre leads the composite having a good flexural strength.

The flexural strength of basalt fibre/PF composite is shown in Fig. 12. As the content of basalt fibre raised from 20 to 63 vol% the flexural strength of the composite decreases with sharp decrease above 48 vol%. The

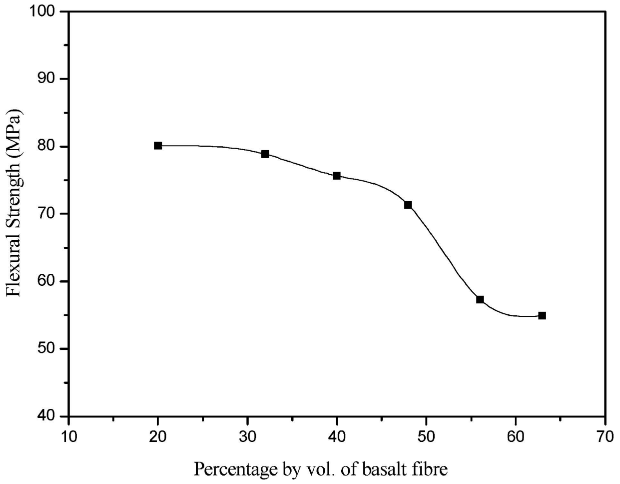


Figure 12 Effect of basalt fibre loading on the flexural strength of basalt/PF composite.

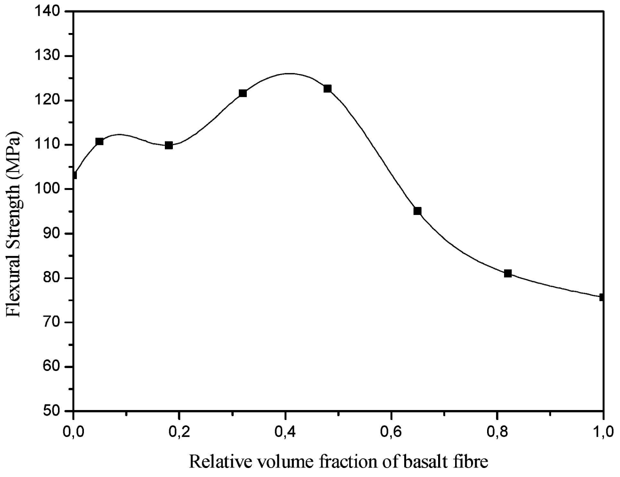


Figure 13 Variation of flexural strength of hemp/basalt hybrid PF composite with basalt fibre loading.

flexural strength values at 20, 48 and 63 vol% basalt fibre loadings are 80.1, 71.3, and 54.9 MPa, respectively.

Variation of flexural strength upon hybridisation of the hemp and basalt fibre is given in Fig. 13. The basalt fibre loading is varied from 0 to 100 vol%. As the content of basalt fibre is raised, the flexural strength of the hybrid composite increases. There is the maximum flexural strength at 48 vol% basalt fibre loading. The flexural strength of the hybrid composite at 0 and 48 vol% basalt fibre loadings are 103.1 and 122.6 MPa, respectively. Flexural strength of hemp/basalt PF hybrid composite exceeds the flexural strength of basalt/PF composite.

3.3. Impact strength

The impact failure of a composite occurs by factors such as fibre/matrix debonding, fibre and/or matrix fracture and fibre pull out. Fibre fracture dissipates less energy compared to fibre pull out. The former is common in composites with strong interfacial bond while the occurence of the latter is a sign of a weak bond. The applied load transferred by shear to fibres may exceed the fibre/matrix interfacial bond strength and debonding occurs. When the stress level exceeds the fibre strength, fibre fracture occurs. The fractured fibres may be pull out of the matrix, which involves energy dissipation [5, 11, 12]. Fig. 14 shows the variation of impact strength of hemp/PF composite with hemp fibre loading. The impact strength of hemp/PF composite increased almost linearly with hemp fibre loading up to 56 vol%. However further increase in fibre loading (up to 63 vol%) caused a moderate increase in impact strength.

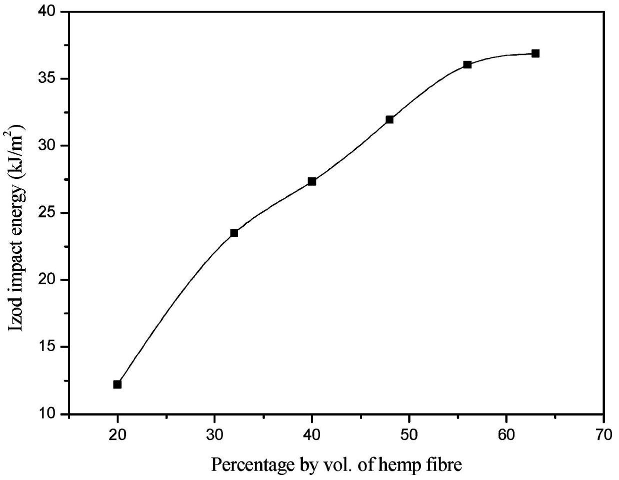


Figure 14 Impact strength of hemp/PF composite as a function of fibre loading.

The effect of basalt fibre loading on impact strength of basalt/PF composite is depicted in Fig. 15. The impact strength of basalt/PF composite was not changed with increasing fibre content between 20–48 vol%. But impact strength decreased sharply with further increase of fibre content. It is thought that at higher basalt fibre loading, the failure mechanism will be fibre pull out due to increasing unwetted fibres by PF matrix. It can be explained that unwetted areas act as crack propagation notches in the matrix. This reduces impact strength at higher fibre loadings.

SEM micrographs of impact-fracture surfaces of hemp/PF and basalt/PF composites containing 40 vol% fibre are shown in Figs 16 and 17. It can be seen from the fracture surface that the hemp fibres adhere well to

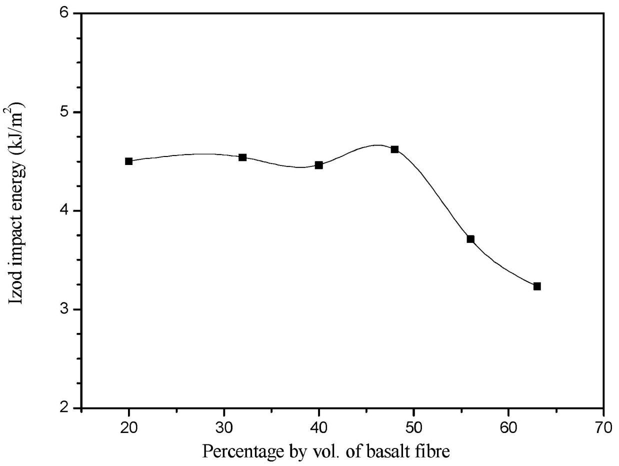


Figure 15 Variation of impact strength with fibre loading for basalt fibre reinforced composite.

Figure 16 Impact fracture surface of hemp/PF composite (fibre loading 40 vol%).

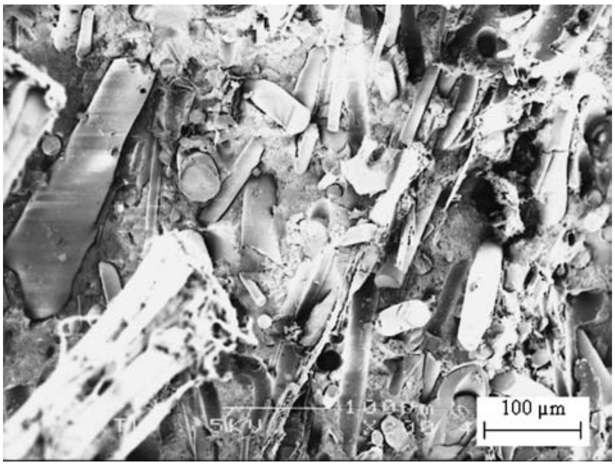


Figure 17 Impact fracture surface of basalt/PF composite (fibre loading 40 vol%).

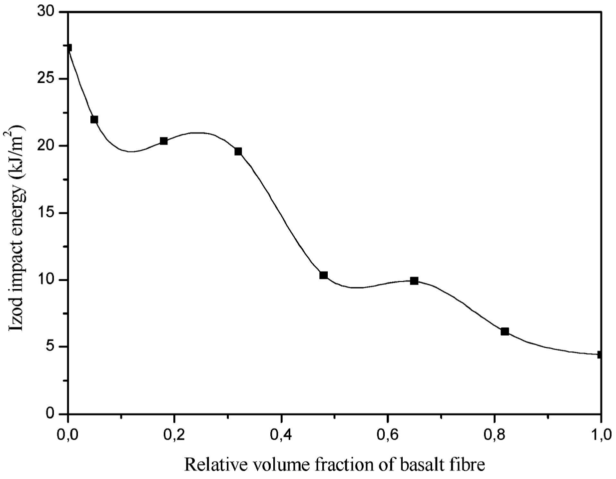
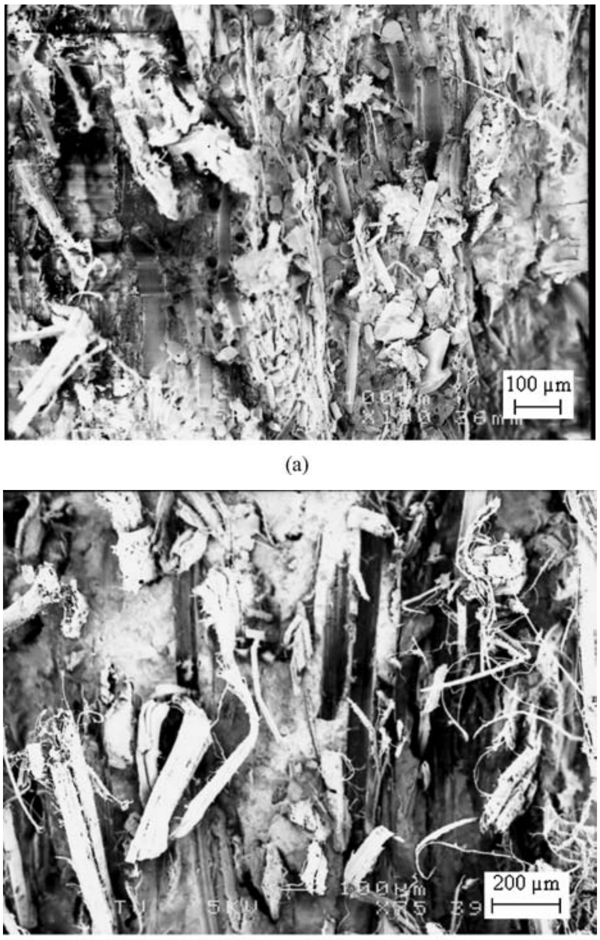


Figure 18 Effect of basalt fibre loading on the impact strength of hemp/basalt hybrid PF composite (total fibre content is 40 vol%).

the PF matrix and the composite is considered to fracture primarily in ductile manner (Fig. 16). On the other hand, Fig. 17 shows poor interaction between basalt fibres and PF matrix. The composite is thought to fracture in a brittle mode which is in aggrement with the experimental results.



(a)

(b)

Figure 19 Scanning electron micrographs of impact fractured surfaces of hemp/basalt hybrid PF composites. (a) Hemp/basalt ratio: 0.18: 0.82, (b) hemp/basalt ratio:0.95:0.05.

The effect of basalt fibre loading on the impact strength of hemp/basalt hybrid PF composite is indicated in Fig. 18. The impact properties of hemp/basalt hybrid PF composite show large scatter with increasing basalt fibre content. The impact strength of hemp fibre at 40 vol% fibre loading (without basalt fibre) is found to be 27.3 kJ/m^2. The impact strength values are found as 21.9 and 6.2 kJ/m^2 for 5 and 82 vol% basalt fibre loadings, respectively. When examining the impact fracture surfaces of hemp/basalt hybrid PF composites, the decreased adhesion between basalt fibre and PF matrix is evident for the hemp/basalt ratio of 0.18:0.82 Fig. 19.

4. Conclusions

In this paper, mechanical properties such as tensile, flexural and impact strengths of hemp/PF, basalt/PF and hemp/basalt hybrid PF composites have been investigated. The following results can be obtained:

1. The tensile, flexural and impact properties of hemp/PF composites improved by increasing fibre loading up to 40 vol%. However, higher loadings of hemp fibre than 40 vol% resulted in a drop of mechanical properties.
2. Tensile strength of basalt/PF composite increased by incorporation of basalt fibre up to 32 vol% and decreased beyond this value. Flexural strength decreased

linearly with fibre loading. However, the maximum impact strength was obtained for 48 vol% basalt fibre loading.

3. Addition of basalt fibres decreased the tensile strength of the hemp/ PF composites. Flexural and impact properties of hemp/basalt hybrid PF composite show large scatter with increasing basalt fibre content.

4. The tensile, flexural and impact properties of hemp/PF composites are higher than that of basalt/PF composites for the same fibre loading.

References

1. L. A. POTHAN, Z. OOMMEN and S. THOMAS, *Compos. Sci. Technol.* **63** (2003) 283.
2. A. K. MOHANTY, A. WIBOWO, M. MISRA and L. T. DRZAL, *Compos. Part A-Appl. S* **64** (2004) 363.
3. B. WIELAGE, TH. LAMPKE, H. UTSCHICK and F. SOERGEL, *J. Mater. Process. Tech.* **139** (2003) 140.
4. S. JOSEPH, M. S. SREEKALA, Z. OOMMEN, P. KOSHY and S. THOMAS, *Compos. Sci. Technol.* **62** (2002) 1857.
5. P. WAMBUA, J. IVENS and I. VERPOEST, *ibid.* **63** (2003) 1259.
6. L.Y. MWAIKAMBO and M. P. ANSELL, *ibid.* **63** (2003) 1297.
7. S. H. AZIZ and M. P. ANSELL, *ibid.* **64** (2004) 1219.
8. M. JACOB, S. THOMAS and K. T. VARUGHESE, *ibid.* **64** (2004) 955.
9. M. RICHARDSON and Z. ZHANG, *Reinf. Plast.* (2001) 40.
10. A. K. BLEDZKI and J. GASSAN, *Prog. Polym. Sci.* **24** (1999) 221.
11. M. S. SREEKALA, J. GEORGE, M. G. KUMARAN and S. THOMAS, *Compos. Sci. Technol.* **62** (2002) 339.
12. S. MISHRA, A. K. MOHANTY, L. T. DRZAL, M. MISRA, S. PARIJA, S. K. NAYAK and S. S. TRIPATHY, *ibid.* **63** (2003) 1377.
13. CUKUROVA KIMYA Ltd. Manisa, Turkey.
14. M. BOTEV, H. BETCHEV, D. BIKIARIS and C. PANAYIOTOU, *J. Appl. Polym. Sci.* **74** (1999) 523.
15. DBW GmbH&Co. KG Rotedal 40. Bovenden, Germany.
16. J. S. SZABO and T. CZIGANY, *Polymer Testing* **22** (2003) 711.
17. Catalog, Basalt, Physical and Chemical Properties, Deutsche Basaltsteinwolle GmbH, Rodetal 40 D-3406 Bovenden, 1, Germany (1993).
18. T. M. GOWDA, A. C. B. NAIDU and R. CHHAYA, *Compos. Part A-Appl. S* (1999) 277.

Received 11 February
and accepted 22 March 2005