Journal of Materials and Environmental Sciences
ISSN : 2028-2508
CODEN : JMESCN

J. Mater. Environ. Sci., 2019, Volume 10, Issue 12, Page 1238-1249

http://www.jmaterenvironsci.com

The Effect of Reinforcing Sisal Fibers on the Mechanical and Thermal Properties of Polypropylene Composites

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Received 06 July 2019,
Revised 01 Nov 2019,
Accepted 02 Nov 2019

Keywords
- ✓ *Natural Fibres*
- ✓ Sisal Powder,
- ✓ Polypropylene
- ✓ Compression moulding
- ✓ Differential Calorimetric Analysis
- ✓ Mechanical properties

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Abstract

The natural fibres are gaining interest among the researchers because of its various properties. The natural fibres can be reinforced effectively in various polymers by different methods. The Natural Fibre Composites (NFCs) have wide range of applications and by using these plastic usages can be minimised. The ability to absorb or to reject heat is an important criterion in electrical and thermal industries. Only a few studies have been done on the powdered form of sisal. In this study an attempt is made to find out the heat absorption/rejection capacities of powder and short fibre sisal composites for different weight fractions through Differential Calorimetric Analysis (DSC) and the corresponding mechanical strengths were studied. To improve the strength of the composites, fibres were treated in Na-OH solution. The reinforced plastics showed better heat absorption and rejection than the pure polymer but less mechanical strength. The powdered form of composite showed better properties than 20 mm fibred composites. The composite with a 10 mm length and 10 percent fibre loading showed best heat absorption/rejection capacities. The treated fibre composites showed improved strength than the untreated fibre. The same composite showed best mechanical properties also. The DSC analysis of the treated fibre showed lesser heat capacities.

1. Introduction

The natural fibres can act as excellent reinforcement for the polymers. In the recent years researchers have proved that natural fibres can replace glass and other manmade fibres in low load applications. The natural fibres may be of different types [1, 2]. It can be derived from animals or plants. Fibres can be extracted from the stems, roots, and leaves of the plant [3]. These natural fibres usually reinforced in thermoplastics or thermosets. Composites produced from natural fibres and plastics are referred as natural fibre composites or NFCs. Compression moulding, hand moulding, extrusion etc. are the different methods of manufacturing thermoplastic composites. [4-6]. The hot compression moulding is one of the most common method to manufacture thermoplastic- NFCs due to its low cost. In compression moulding the ready to press (prepreg) material is pressed between the two dies which are closely matching. The prepreg can be prepared by mixing matrix and reinforcement [7-9]. Sisal is a natural fibre which is extracted from the leaves of plant Agave sisalana. Sisal fibre consists of cellulose, hemicellulose, pectin, lignin and small amount of waxes like other vegetable fibres. Sisal is lignocellulosic fibre its property mainly depends upon the individual constituents. The density of the sisal fibre varies from 1.3 to 1.5 g/cm^3 and strength is around 300-600 MPa. Properties of sisal fibre varies depending upon the manufacturing method, age of the plant, locality etc. Sisal fibre can be reinforced in different forms [7-11]. In earlier studies researchers have reported that the sisal fibre increases the heat capacities of the thermosets (epoxy) than the glass and other manmade fibres. Polypropylene is thermoplastic material looks white in appearance usually available

in pellets. Polypropylene is a versatile material. Its strength varies from 15 MPa to 35 MPa. Polypropylene is mixed with polyethylene to improve its moulding properties which is referred as moulding grade Polypropylene. The density of the PP is around 0.91 g/cm^3. It is hydrophobic material having excellent resistant to most of the chemicals. Its melting point is also high and hence usually used as insulator in electrical devices [12-15].

NFCs consists matrix and reinforcement which are opposite in nature. The fibre and matrix may have different thermal expansion coefficients. The variable thermal expansion results in failure of the composite. Due to the variable cooling, thermal stresses may be induced in the composites. Hence thermal analysis of the composites is very important [16]. The quantity of heat absorbed or released by the composite material can be studied with the help of calorimetric analysis usually Differential Calorimetric Analysis (DSC). The other types of analysis are TGA and TMA. In DSC test, the reference pan and the pan with specimen are heated and cooled in different cycles. The rate of heating and cooling maintained during the test is also an important criterion for the test. Area under the DSC curve gives the thermal capacities of the sample. Thermal analysis gives useful information such as specific heat, co-efficient of thermal expansion, thermal stability, composition etc. So in this study the heat absorption and rejection capacities of the natural fibre composites are determined for different fibre lengths and weights with the help of DSC [17-20, 5].

Moisture absorption is a major problem in natural fibre. The wettability of the natural fibre results in poor bonding between the fibre and the matrix. To overcome this problem fibre treatment is usually done. Heat treatment and chemical modification are two different fibre treatment methods usually used by the researchers [21]. Alkaline treatment, acidic treatment, benzolyzation are the various methods of chemical treatments. Alkanline treatment is a method treating fibre with alkaline solution usually NaOH. The alkaline solution kills the active polar group of the fibre and removes the moisture present in the fibre and results in improved bonding of the fibre and matrix [22-25]. Though several studies have been done on the mechanical and thermal properties of the composites, only a few studies have been done on the powder form of the composites. Hence in the present study, the attempt is made to find out the mechanical and thermal properties of the natural fibre reinforced with powder form and short fibres in the developed composites. Also, mechanical and thermal properties of powder and short fibres were compared with treated form.

2. Material and Methods

2.1. Materials

Sisal fibres are used as reinforcement for this study and is procured from Shrilaxmi Groups, Cherukapalli, AP, India. The fibres of diameter 100 to 300 µm were supplied in two different lengths. i.e. 10 mm and 20 mm. Sisal fibres were procured in powder form also. The size of powder was 100 to 300 micron. The matrix material selected for this study was moulding grade polypropylene of density of 0.9 g/cm^3. When polymer was tested it showed strength of 18 MPa. The polymer pellets were supplied by Mangala Poly-products, Mangalore.

2.2. Composite Preparation

The natural fibre composites are prepared by hot compression moulding at The Energy Resource Institute (TERI) Bangalore. For the hot compression moulding prepreg was prepared at Brabender plastograph EC Plus blending machine. The machine consists of twin blades. The blades were rotated at 25 rpm around 150 ^{o}C blending was accomplished. Blended matrix and fibres are also called prepreg. The hot compression moulding machine consists of two closely matching dies of size 150 x 150 x 3 mm^3. The prepreg was pressed between these dies at pressure of 85 bar for 2 hours then allowed to cool till it reaches room temperature. The NFCs were prepared in different fibre weight ratios.

2.3. Test Details

To find out heat absorption and rejection DSC analysis was done. The test specimens were heated then cooled along with the reference pan. The variation of heat flow and the temperature graph is drawn (Fig. 1 and Fig.2). The samples for the mechanical tests are prepared according to the ASTM standard from the composite sheets. DSC and Mechanical testing are conducted at KONSPEC, Mangalore. SEM images were taken for the tensile fractured specimens at Manipal Institute of Technology, Manipal. Fibres were soaked in 10% NaOH solution for 24 hours then dried in sunlight for 7 days.

3. Results and discussions

3.1. DSC Test

For DSC study 6-8 mg of the sample was heated for each weight fraction and fibre length from 30 ^{o}C to 200 ^{o}C. After reaching the temperature it was held for 5 min. The same sample was then cooled from 200 ^{o}C temperature

to 30 ºC and was held for 5 min. To perform second heating cycle the sample was again heated from same 30 ºC temperature to 200 ºC. The maintained heating and cooling rate was 10 ºC/min. The DSC test results are shown in the Table 1.

Fig. 1 and Fig. 2 indicate series of heating and cooling curves for a particular fibre length. Distinct endothermic and exothermic peaks can be observed through these graphs. The exothermic peaks are around 133- 136 ºC for all composites.

Table 1: DSC test results of (a) Powder form (b) 10 mm short fibre and (c) 20 mm short fibre.

Parameters	Pure Polymer	Powder			10mm			20mm		
		10%	20%	30%	10%	20%	30%	10%	20%	30%
Exothermic peak ºC	134.35	134.57	133.53	133.32	134.35	134.65	133.82	136.87	135.54	136.68
Endothermic peak ºC	111.15	114.13	114.87	114.54	114.53	114.71	115.53	114.4	115.66	115.36
Heat released J/g	254.99	284.73	309.07	244.96	351.78	259.05	235.68	187.67	163.15	152.87
Heat absorbed J/g	235.28	268.08	294.81	233.52	331.67	249.24	222.28	181.12	156.76	145.08

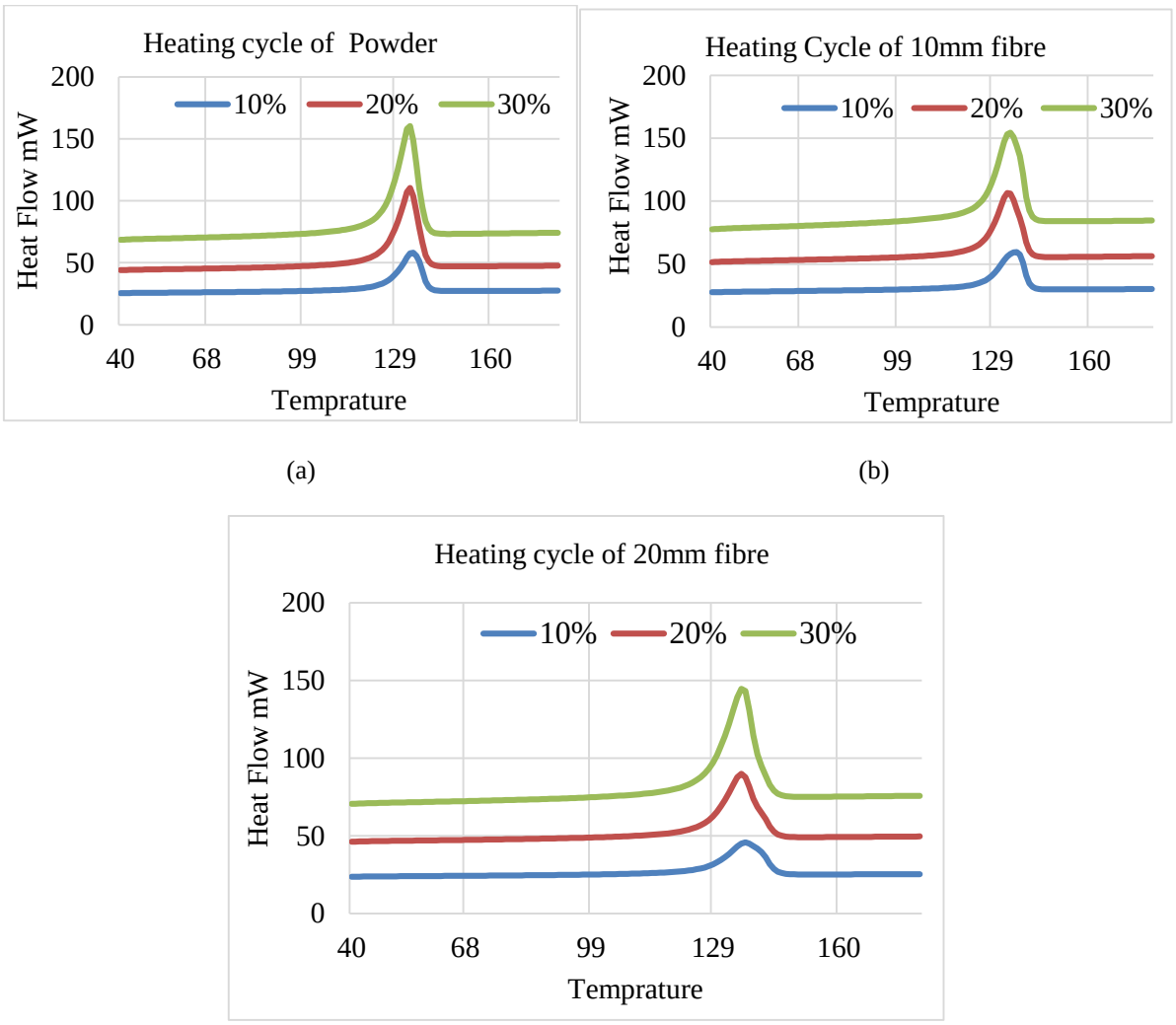


Figure 1: Cooling curves of composite specimens for (a) Powder (b) 10 mm fibre and (c) 20 mm fibre.

The endothermic peak is around 115 °C, the nature of graph is almost similar for all composites but the area under the curve differs. Area under the curve gives the heat capacities of the samples. There is no much change in the other parameters like onset temperature, peaks (endothermic and exothermic). In each graph there are three different lines each line indicates the different percentage of fibre loading. To erase the thermal history of the polymers first heating is done which is not considered while drawing the graphs. The graph is drawn based on the second heating and cooling.

DSC of pure polymer shows the exothermic peak of 134.35 °C and endothermic peak of 111.15 °C. Heat rejected or the area under the curve is 254.99 J/g and heat absorbed is 235.28 J/g. The pure polymer when reinforced with fine powders of 100 to 300-micron size, the heat absorption increases. But there is no much change in the other factors like endothermic peak and exothermic peak, onset temperatures etc. Heat absorbed by 10, 20 and 30 percent fibred composites are 268.08 J/g, 294.81 J/g and 233.52 J/g respectively (Fig. 3(a)). Heat released by these composites in the same order are 284. 73 J/g, 309.07J/g and 244.96 J/g (Fig. 3(b)). These values are much higher than the pure polymer. Sisal has higher melting point than the pure polymer and it contains cellulosic materials which are having higher thermal absorption capacities than the pure polymer which resulted in higher thermal efficiencies than the pure polymer [14, 25].

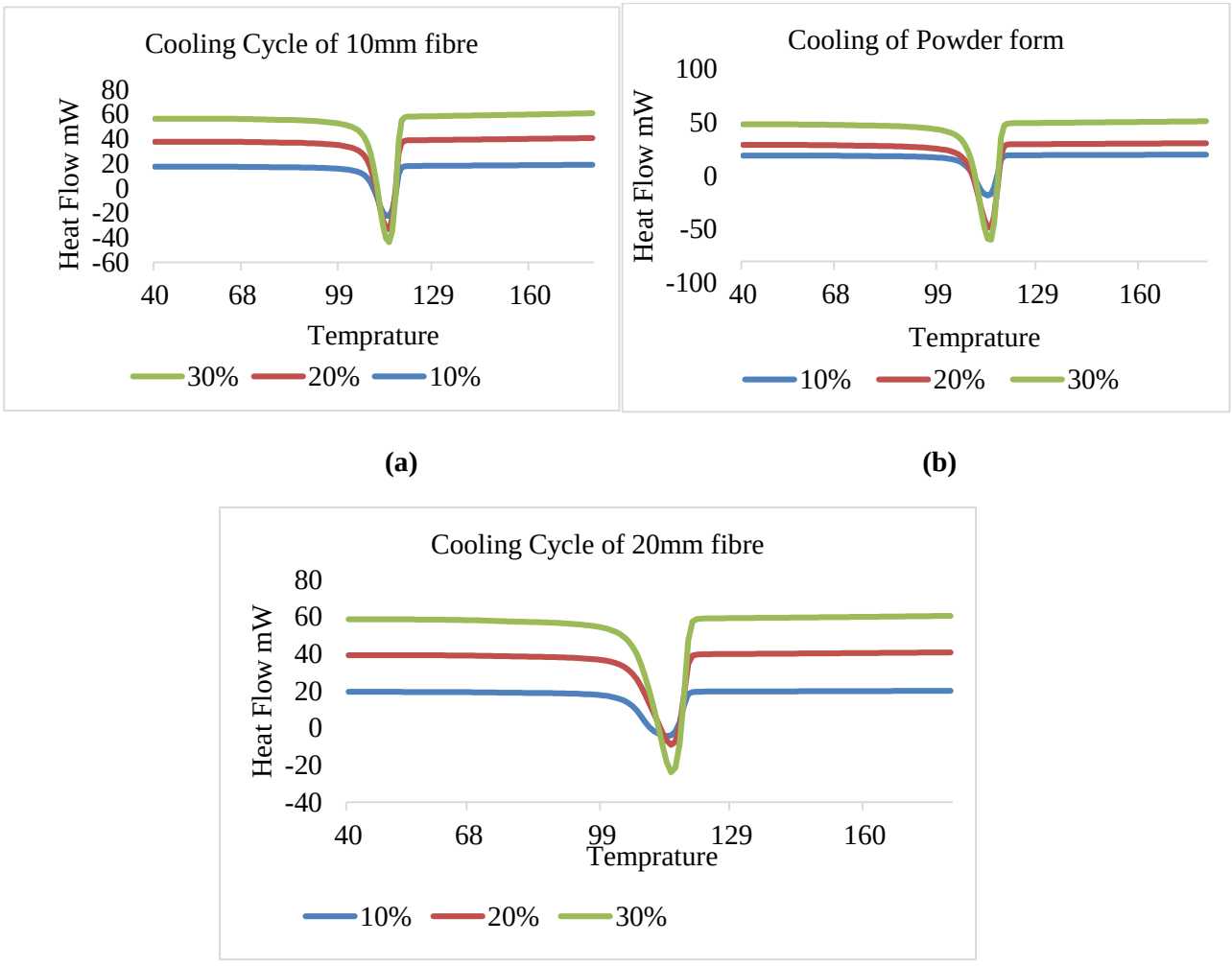
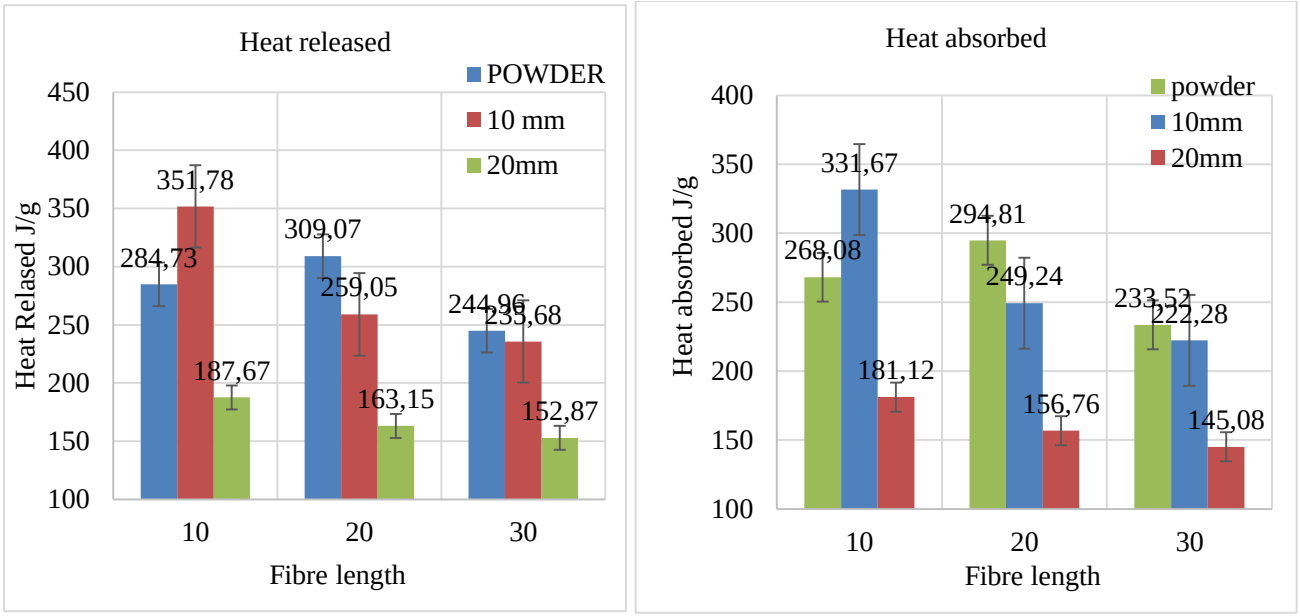


Figure 2: Cooling cycle of various composites for (a) Powder (b) 10 mm fibre and (c) 20 mm fibre

The heat absorbed and heat released changes even in 10 mm fibre reinforced composites. A complete different values are obtained for this fibre length compared to the powder form. Heat absorbed for 10, 20 and 30 percent fibres are 331.67 J/g, 249.24 J/g and 228.28 J/g respectively loading. Heat released for the same fibre loading in the same order are 351.78 J/g, 259.05 J/g and 235.68 J/g (Fig.3 (a) and Fig. 3(b)). The polymer is further reinforced with fibre length of 20 mm, the heat absorbed and released per gram of the sample slightly decreases compared to the other composites of different fibre length. The heat absorbed and heat released are 181.12 J/g, 156.76

J/g,145.08 J/g and 187.67 J/g, 163.15 J/g, 152.87 J/g for 10, 20, 30 fibre weight ratios respectively. These values are less compared to the polymer with no reinforcement. It is noticed that as the weight fraction of the fibre increases in composites heat released decreases for fibre length 10 and 20 mm. The 30 percent fibre weight showed lesser value for almost all cases. When pure polymer is tested it showed heat absorbing capacity of 254.99 J/g but sisal fibre have heat absorption capacity more than the polymer. Natural fibers have better heat capacities than the polymers. Sisal is a natural and lignocellulose fibre contains high amount of lignin [4]. The degradation of the lignin and other cellulosic materials starts above 200 ^{o}C. Cellulose degradation is observed at 383 ^{o}C. Presence of these cellulosic materials increases the heat absorption and rejection capacities of the composites [26, 27].



(a) **(b)**

Figure 3: Heat capacities of different composites (a) Heat released (b) Heat absorbed.

3.2 Mechanical Tests

Mechanical test samples are prepared from the composite sheets which were prepared in hot compression moulding machine. Three specimens were tested according to ASTM standards and the best results were noted. The ASTM standards followed for tensile, flexural and impact tests are ASTM D638, ASTM D790 and ASTM D4812 respectively. The tensile tests are performed on ZWICK ROELL, Z020 with load cell of 20 kN capacity. The strain rate maintained was 1 mm/min. The flexural tests are performed on the same machine with same preload and strain rate. Impact tests are performed on ZWICK/ROELL HIT 50P with a velocity of 3.5 m/s. The mechanical properties of composites are shown in the Table 2.

Table 2: The mechanical properties of composites

Sl no	Fibre Type	Tensile ASTM D638			Flexural ASTM D790			Impact ASTM D4812		
		10%	**20%**	**30%**	**10%**	**20%**	**30%**	**10%**	**20%**	**30%**
1	Powder	16.733	10.933	8.55	28.5	23.8	20.30	116.50	115.60	80.66
2	10 mm	17.73	15.23	12.20	32.56	29.93	26.46	128.14	122.67	101.33
3	20 mm	9.75	13.30	13.70	29.40	25	23.20	110.00	99.00	94.00

Optimum mechanical properties are observed for 10 mm and 10% fibre loading. Though powder form properties are less than the 10 mm fibre, but can be comparable with 20 mm fibre. When fibres are in the powder form its surface to volume ratio will be high. Due to the more surface area the chances of moisture absorption is also more. The mechanical strength of the composite depends on several factors like type of matrix material, type of reinforcement, distribution of fibre in matrix and interfacial strength [28]. Comparison mechanical strengths for (a) Tensile strength (b) Flexural strength (c) Impact strength (d) Tensile Modulus (e) Flexural modulus is shown in Fig. 4.

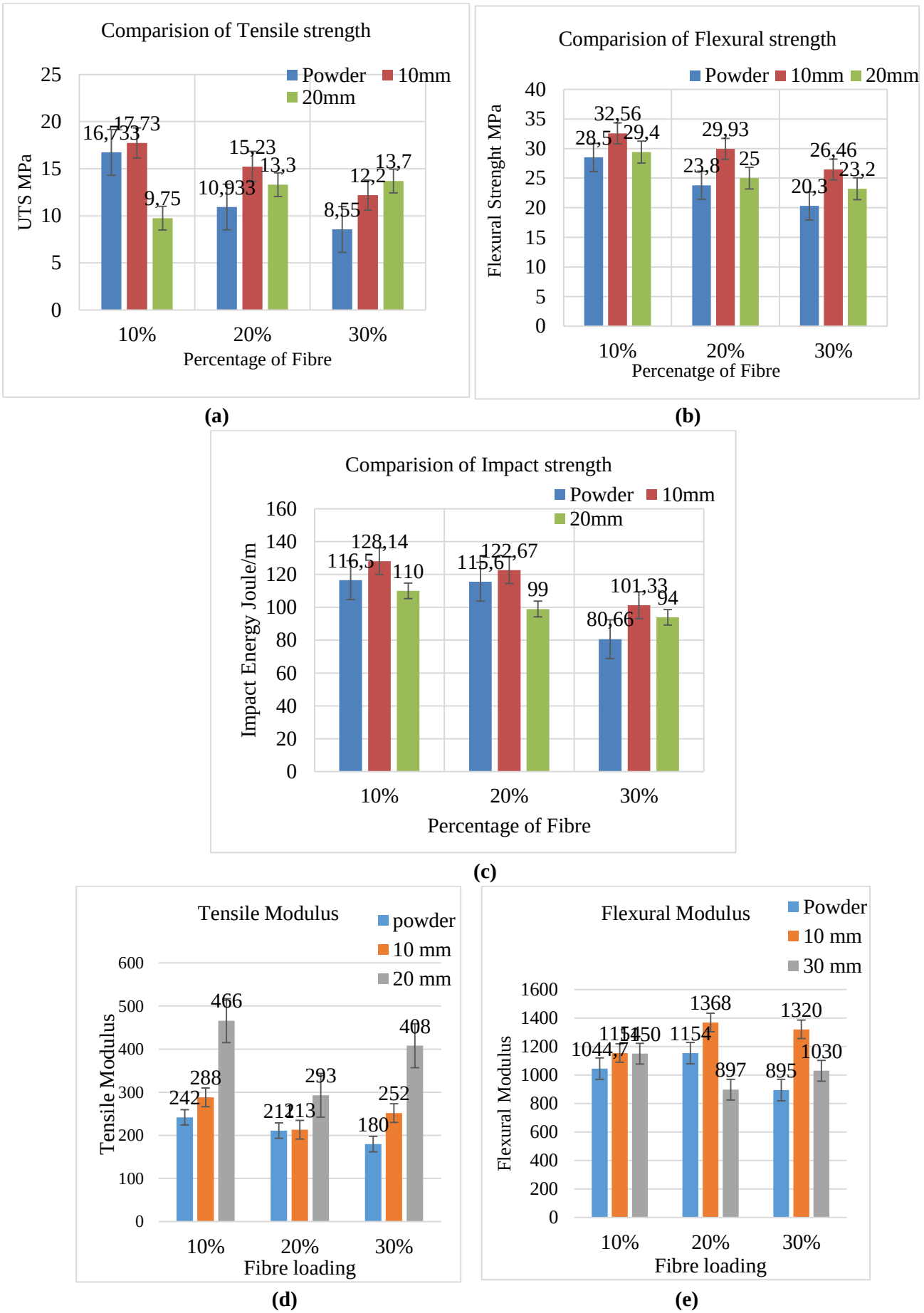


Figure 4: Comparison mechanical strengths for (a) Tensile strength (b) Flexural strength (c) Impact strength (d) Tensile Modulus (e) Flexural modulus.

Subsequently, reduction of strength is observed in all the cases. It is mainly because of the poor bonding strength of the fibre and matrix [7]. As the length of the fibre increases number of weak links increases, so beyond 10 mm may be due to the more number of weak links the decreased strength is observed in composites [26].
Polypropylene is hydrophobic material and sisal is hydrophilic material. The bonding between these materials are difficult. Thus, as the weight fraction of the composite increases the strength of the composite decreases. Though Reinforcing the fibre increases the heat capacities but decreases the mechanical strength of the polymer but by doing the fibre treatment the mechanical properties can also be improved [6, 18]. Powdered form of fibre is having better heat capacities and mechanical strengths compared to 20 mm fibre. Hence powdered form of fibres can be effectively used as a reinforcement. But while using the powdered fibres one should take care of moisture absorption.

Fig. 5 shows the SEM images of fractured specimens indicated the short fibred composites are failed due to its fibre pull-out not due to the fibre breaking. In powdered fibre composites, the dark patches of polypropylene are indicating improper distribution of the sisal powder in polypropylene. The agglomeration of the sisal powders results in improper distribution of fibre in matrix. The micrographs of short fibre composites shows that the fibres are randomly distributed in the matrix. The composites are failed to withstand excess load due to the fibre pull out nor due to the fibre breaking. The composite strength varies depending upon the direction of the fibres [18]. In the direction of fibre composite has maximum strength and if the fibres are distributed in random direction the strength may decreases.

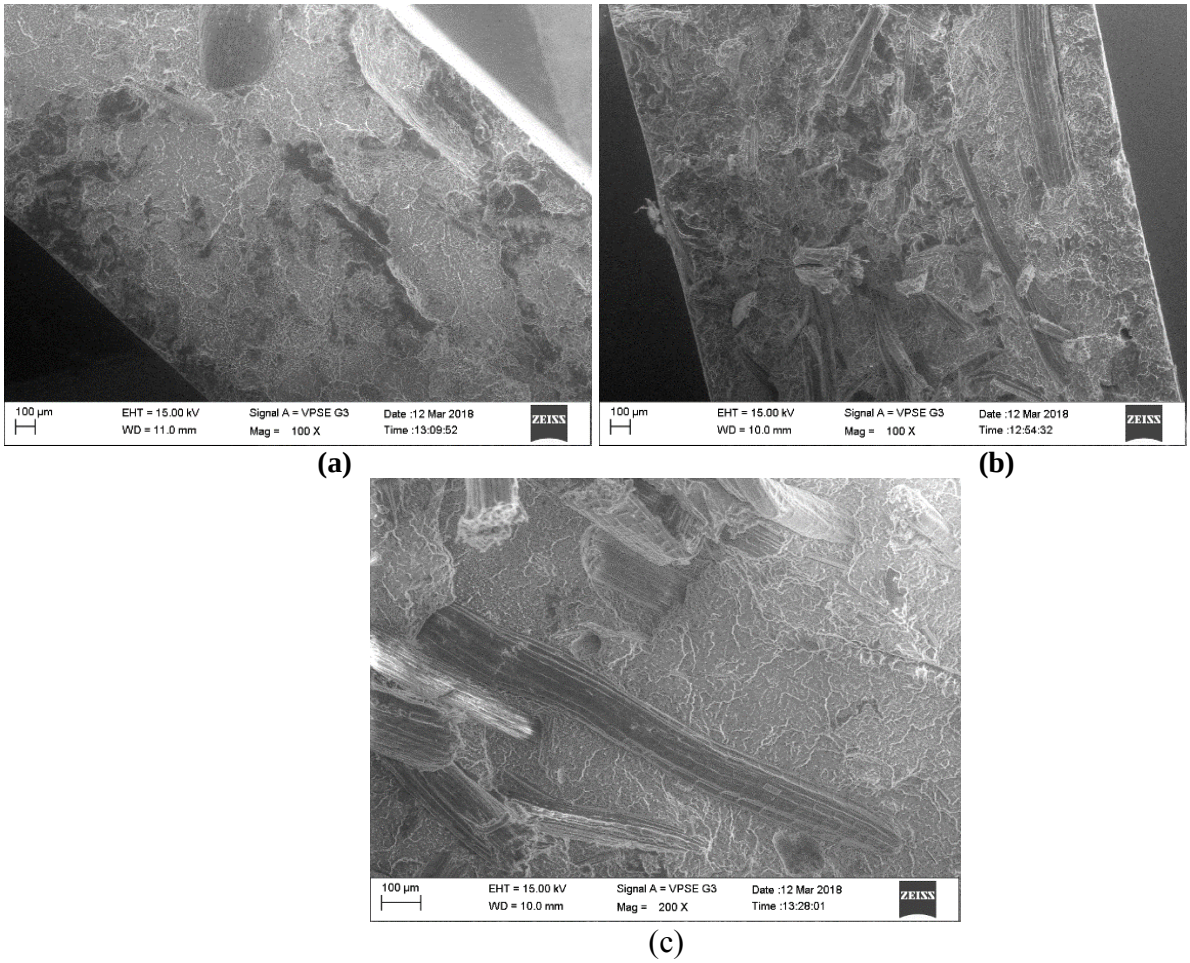


Figure 5: SEM images of tensile fractured specimens of (a) Powder (b) fibre of 10 mm (c) fibre of 20 mm

3.3 Fibre Treatment

Sisal fibres when treated in an alkaline solution it showed improved strength. The alkaline solution kills the OH group in the fibre and reduces moisture absorption. The fibre texture changes and washing away of unwanted wax exposes more lignin material to the matrix which improves the bonding between the fibre and matrix results in improved strength of the material [23, 28]. Mechanical Strengths of treated fibres for (a) Tensile strength (b) Flexural strength (c) Impact strength (d) Tensile Modulus (e) Flexural modulus is shown in Fig. 6.

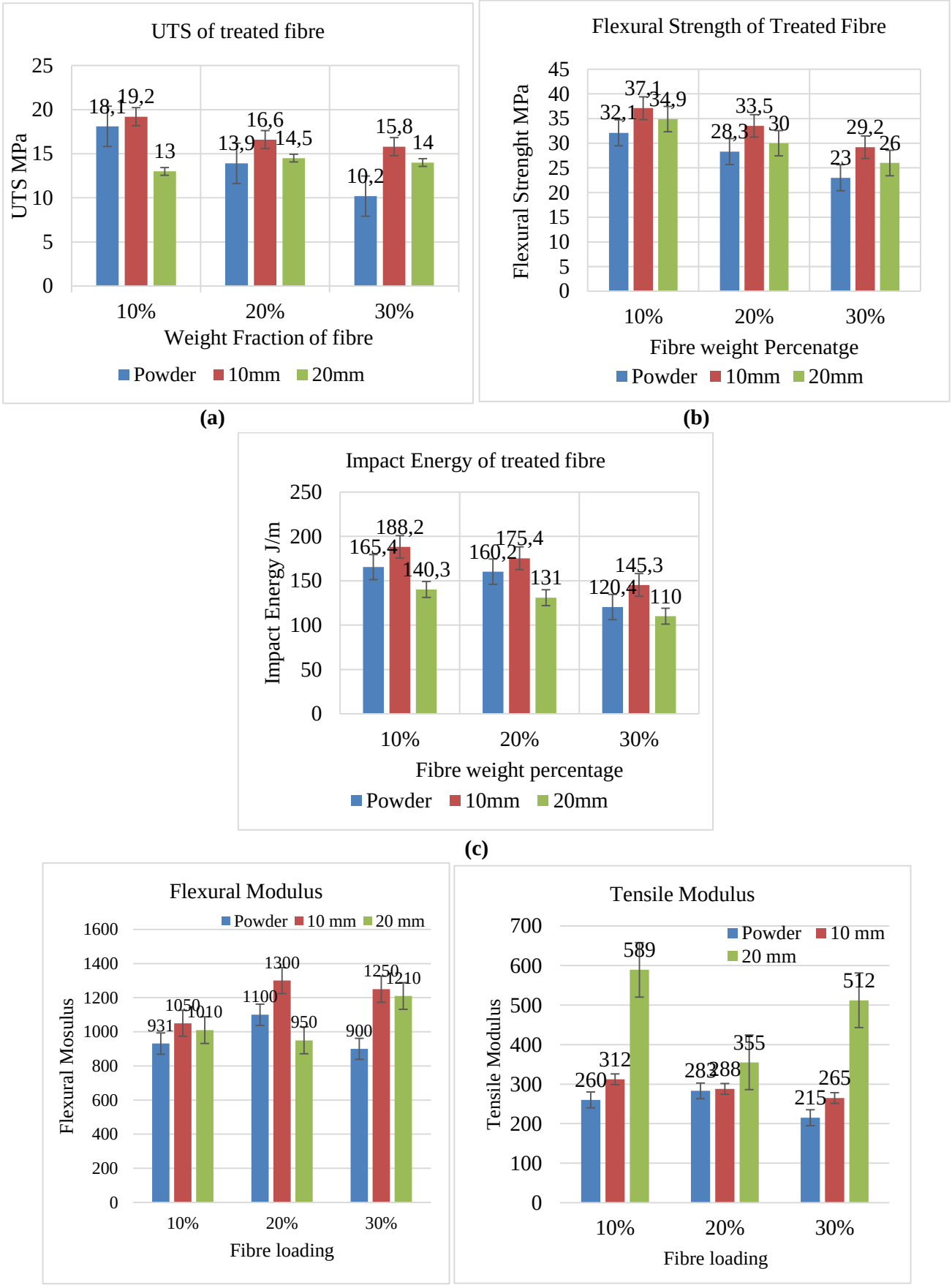


Figure 6: Mechanical Strengths of treated fibres for (a) Tensile strength (b) Flexural strength (c) Impact strength (d) Tensile Modulus (e) Flexural modulus.

The tensile strength of the 10 mm untreated fibre for 10% fibre weight was 17.73 MPa and for the treated fibre it is 19.2 MPa. So the total improvement of 8.29%. Similarly, the improvement for the powder fibre for same weight fraction is 8.19% and for 20 mm fibre the improvement is 33%. The improvement in strength is observed more in 30 percent fibre weight ratio composites for all fibre length. Due to the proper bonding between fibre and matrix the improved strength is observed in flexural strength also. Here also the percentage of improvement for different fibre weight and for different fibre lengths are in the range of 11 to 20 percentage. Though the values are higher in the 10mm fibre but the percentage of improvement is more in powder and 20 mm fibre than the 10 mm fibre. Improvement in the strength is in the range of 15 to 45 percent for the different fibre weight ratios and lengths. The 10mm 10 percent fibre showed an improved strength of 188.2 J/m which is almost 45 percent improvement. Sisal fibre consists of cellulose in the range of 47-78%, and lignin in the range of 7-8% hemicellulose 10-24% small amount of ash and wax [6]. The lignin improves but wax minimises the adhesion property of the sisal. The treatment of the fibre with alkaline solution removes the unwanted wax material. This results in the better adhesion of sisal with polymer and hence the improved strength can be clearly seen in almost all composites [6, 27].

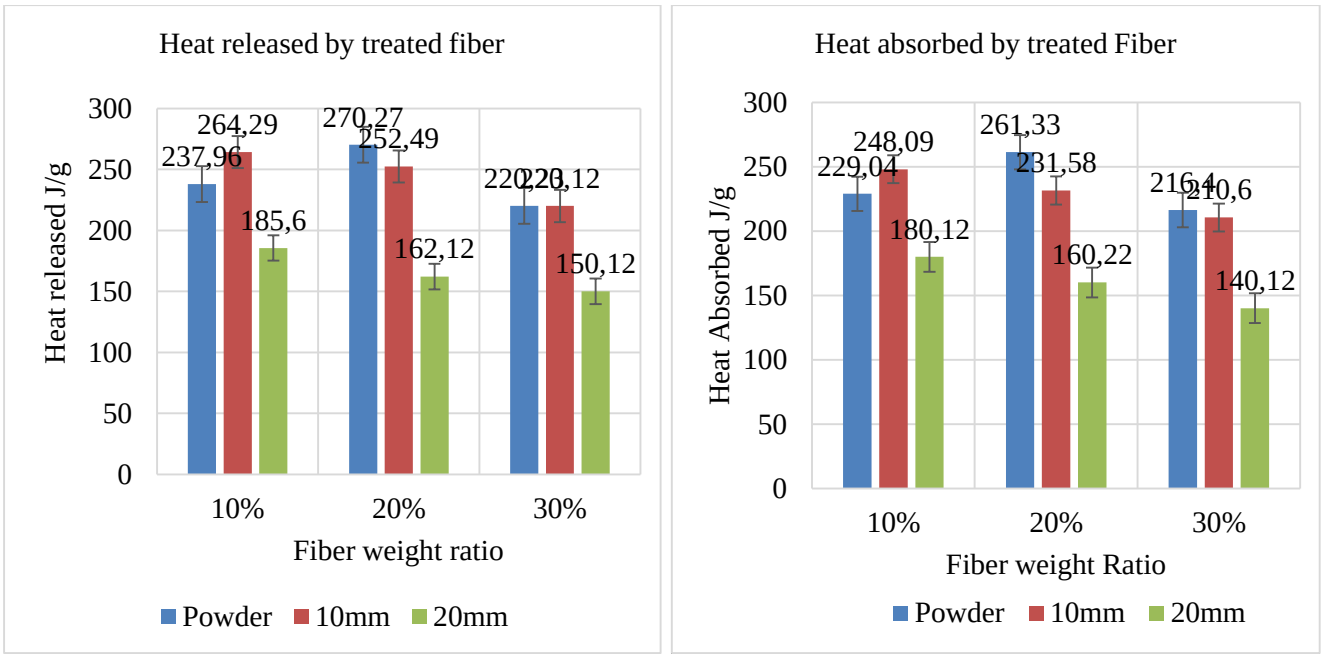
3.3 DSC of treated fibre

The treated fibres when analysed through DSC, it showed lesser heat absorption and rejection capacities than the untreated fibres (Table 3). Though the mechanical strength of the treated fibre increased drastically, but there is decrease in thermal capacities of the composites. The heat capacity of treated fibre are shown in Fig. 7. The nature of graph is almost same but the area under the curve has changed. Every sample showed distinct endothermic and exothermic peak. The heat absorbed for powdered 10% untreated fibre composite was 268 J/g and for treated fibre it has become 237 J/g. The reduction in heat capacity is 19.65%. The same treated powder composite for the different fraction like 20% and 30% showed reduced heat capacities of 14.36% and 11.23%. Similar trend is observed in other fibre lengths also. The 10% fibre weight and 10mm length composite which has shown best mechanical property and better heat capacities is also showed down trend and the reduction is around 33%. The maximum reduction is shown in this composites compared to others. The treated fibre of 20 mm fiber length which has shown lesser mechanical strength and heat capacities showed lesser drop in heat capacities. The drop is around 1 percent which can be neglected. Similar down trend is observed in heat released also the maximum drop is in 10 mm 10 % fiber weight and least drop is in 20 mm fiber.

Table 3: DSC test results of treated fibre

Parameters	Powder			10mm			20mm		
	10%	20%	30%	10%	20%	30%	10%	20%	30%
Exothermic peak ^{o}C	135.19	133.79	133.12	135.04	134.67	134.8	134.28	136.2	136.88
Endothermic peak ^{o}C	114.33	114.25	114.2	113.64	114.21	115.23	114.26	115.81	115.46
Heat released J/g	237.96	270.27	220.23	264.29	252.49	220.12	185.6	162.12	150.12
Heat absorbed J/g	229.04	261.33	216.4	248.09	231.58	210.6	180.12	160.22	140.12

The maximum decrease in heat capacities are observed in powder form. Due to the more surface area the treatment was more effective in powder form. The effective removal of moisture resulted in reduced heat capacities of the composites. Hence in powder form of composite the heat capacities reduced more than the other composites [14]. The majority of the hemicellulose is removed after the treatment the treated fiber shows less heat capacity than the untreated fiber. The untreated fiber contains water the heat capacity of the water is around 4180 J/kg/K. Water content is more means the heat capacity of the composite is also more. Sisal is natural fiber and like other natural fibre it has tendency to absorb moisture. In the presence of moisture, the heat capacities of the composite increases [13, 28-31]. Hence, the treating fiber has greater impact on 20 mm fiber and powder when mechanical properties are considered. The treatment influence is maximum for 10 mm fiber when heat capacities are considered. Hence when better mechanical properties are required then Na-OH treatment can be done on the fibers but when better heat capacities are required fibre treatments can be avoided [32].



(a) **(b)**

Figure 7: Heat capacities of treated fibres (a) Heat released by treated fibre (b)) Heat absorbed by treated fibre.

Conclusions

NFCs were developed in powder form and compared with short fibres reinforced composites. NFCs composites have better heat absorption and rejection capacities than the pure polymers. However, these composites showed lesser mechanical strength when compared to the pure polymer. The thermal analysis through differential calorimetric analysis (DSC) shows that there are no much appreciable changes in the endothermic peak and the exothermic peak irrespective of fibre loading. The powdered form and 10 mm fibre composites have showed better heat capacities and mechanical strengths when compared to the 20 mm fibre. The thermal results indicated that, heat absorption capacity of the composite is maximum for 10 mm and 10 percent of fibre loading. The untreated fibre composites showed lesser strength and the reduction in heat capacities is up to 33%. It indicates that the elimination of water resulted in lesser heat capacities. It can be concluded that as the weight fraction of the fibre increases in the matrix the strength of the composite decreases in a particular length of the fibre. The 20 mm fibre showed poor mechanical strength and lesser heat capacities. Though presence of sisal fibre increases the heat capacities of the polymer but mechanical strength should be compromised. The microstructural analysis results showed that polypropylene matrix with powder form and short sisal fibres of 10 mm improved the adhesion strength and tensile strength of the composite.

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