Actual properties of Epoxy Reinforced with Surface Modified Sisal Fiber

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ABSTRACT

Recently, natural fibers attract the scientific community towards it usage as reinforcement in polymeric composites mainly because of environmental awareness, growing concern and multiple benefits like low cost, huge/easy availability, easy processing. Reinforcing natural fibers is an efficient approach towards reducing the consumption of non-biodegradable plastic material. Though, the major drawbacks of such composites are poor compatibility between the two distinct phases and high rate of water absorption. It has been observed that with this drawback, degradation of different mechanical and thermal properties generally occurs. Keeping this in mind, in present work, the surface of the sisal fibers are modified with aqueous solution of NaOH at different concentration prior to reinforce it in epoxy matrix. Later, water absorption rate of the developed material is evaluated over a period of one week where measurement is taken after every 24 hours. Results obtained indicate that with surface modified sisal fiber, water absorption rate decreases appreciably. Further, it is also determined that rate of water absorption is minimum with 2 Mole NaOH concentrations. Also, the density is measured and evaluated theoretically to know the void content in the fabricated samples

Keyword : - *Polymer matrix composites, Epoxy, Sisal fiber, Surface treatment, Physical Properties, Mechanical properties, Two-body abrasive wear.*

1. INTRODUCTION

Composite, in actual is not a human invention. Wood is a natural composite material consisting of one species of cellulose fibers with good strength and stiffness reinforced in a resinous matrix of another polymer, the polysaccharide lignin. Generally, composites are defined as material which has combination of two or more distinct constituents. Following the above definition, a huge number of materials will come in this category which includes mud bricks with straw used in early age for building construction to modern man-made composite. In recent time, the composite prepared by human being captured a huge market where structure fabrication with low weight in combination with high strength and stiffness is of concerned.

Classification of Composites Materials

Classification of composites can be made in two different categories, first on the basis of matrix materials, in which it can be metal matrix composites, ceramic matrix composites and polymer matrix composites. Metal matrix composites generating a wide interest in research fraternity mainly because of their strength, fracture toughness and stiffness. Added advantage is that it can withstand elevated temperature even in corrosive environment. Ceramics are a solid material which has in general very strong ionic bonding. They also have covalent bonding in few cases. Ceramic matrix is considered to be an ideal choice for high temperature applications that does not give away its properties even at 1500 oC temperature. Ceramics has very high melting point and are also good resistance to corrosion. They have high compressive strength and possess good stability at elevated temperature. Polymers are always considered to be an ideal material to be used as matrix material mainly because of its ease of processing, light weight and desirable mechanical properties. As stated earlier, polymers are further classified in two main category i.e. thermoset and thermoplastic. The basic difference between them arises from the process they convert themselves from pre-polymer to final polymer.

Second classification of composites are on the basis of reinforcement Fibers are considered to be the most useful reinforcement material. The fiber performance as reinforcement depends upon several factors which include length of fiber, cross-sectional shape of fiber, orientation of fiber when reinforced as compared to the direction of loading, composition of fiber and the properties of fiber

Function and properties of reinforcement

- To provide strength to material. Matrix has generally low in strength and reinforcement is used to enhance the strength property of the material.
- Reinforcement generally makes the material stiffer.
- It improves the directional properties of the composites.
- Used in different size can improve packaging density of the material which can improve the overall properties of the material.
- According to the requirement, intrinsic property of the material can be changed by incorporating proper reinforcing material.

Function and properties of matrix

1. The main function of the matrix is to bind the fibers together and holding them in such a way that they will be aligned in the particular direction for which it is designed. It is the reason why load transferred to the fiber from composite.

2. The other important function of matrix is to isolate the fibers from one another. With this the main aim is to make fiber behave as separate entities.

Another very important function of matrix is to protect the reinforcing material from mechanical damage when load is applied over it. Matrix also protects the reinforcement material from ever changing environmental conditions.
When the matrix is ductile, it helps in slowing down the cracks produced due to breakage of fibers when they are within matrix body.

5. Toughness of the material depends upon the grip of matrix on reinforcement material. Better grip provide increased toughness.

Fiber reinforced polymer matrix composites

Fiber reinforced polymer composites consist of fibers of high strength and modulus embedded within the polymeric matrix body. The two phases have distinct interface or in simple language we called it as boundaries separated them. In this combination, both the fiber and matrix does not lose their physical and chemical identities. Rather, the combination produces the properties which are impossible to attain by the individual phase. As already discussed in previous sections, fibers act as a principle load carrying member, whereas, main function of the matrix is to provide fixed location and desired orientation to the fiber.

Fiber reinforced polymer matrix composites: Applications

Fiber reinforced polymer composites has wide variety of industrial and commercial application. Though the list of application is very long and in this section, major application of fiber reinforced polymeric composites which include military, aircraft, automotive, sporting goods, medical and marine applications. Rather than enumerating only the areas in which polymer based composites are used, a few examples have been taken from each industry.

Sisal fiber: A well-known natural fiber

Sisal fibers are extracted from the leaves of the sisal plant. Botanical name of the sisal plant is Agave sisalana. It is an herbaceous monocotyledonous plant from the Agavaceae family. This plant is found all over the world, but largely in tropical and sub-tropical region. This region comprises of North America, South America, Complete Africa, West Indies, Brazil, Tanzania and India. Though, Tanzania and Brazil are the two main producing countries among all. Generally, the sisal plants are not systematically cultivated grows wild in the hedges of fields and railway tracks. Soil rich in calcium, magnesium, potassium, nitrogen and phosphorous helps in fast and proper growth of sisal plant. A study shows that around 45 Lakhs tons of sisal fiber produced per year all over the world.

Introduction to the present work

A well-known natural fiber i.e. sisal fiber can used as reinforcement in polymer composites in different forms. The various forms in which the fiber can be used are long or continuous or unidirectional sisal fiber, bi-directional sisal fibers, short sisal fibers or woven sisal fibers. The usage of sisal fiber in short form had various advantages as discussed but the main advantage is that it provides isotropic material. Usage of short sisal fiber as reinforcement in epoxy matrix and fabrication of samples by hand lay-up method is an important part of present research work. The drawback of using natural fiber as reinforcement is its hydrophilic nature and its poor adhesion with matrix. Both the drawback can be minimized by selecting proper surface modification method. Proper selection of surface modification method of natural fiber and optimizing the concentration of chemical used is also a part of present research. By proper experimental investigation in term of evaluating physical and mechanical for different concentration of surface modifier is a part of present study.

LITERATURE REVIEW

Daniel, G et.al 2003 [1] The continuously increase in the world population require intensified effort to fulfill the need of material both socially and economically. This requires that the conventional metals and synthetic fibers need some replacement for the betterment of our mother earth. It is high time to reduce the usage of petro-chemical by-products so that their impact on environment can be reduced. Also, guideline shows that the fossil fuels which are been used will get extinct within next fifty year

Faruk et.al 2012 [2] On the basis of their origin, natural fibers are subdivided in three different categories i.e. plant fibers, animal fibers and mineral fibers. The three main categories are further divided in different sub-categories. Like, plant fibers are majorly divided into eight different groups. The first and biggest group is of bast fibers in which fibers are collected either from the skin or from the bast around the stem. The fibers which are in this group and are of interest among the scientific community are jute, flax, soybean, vine, ramie, rattan, hemp, banana and kenaf. Among all, jute is majorly used because of its various favorable properties. The second group is named as leaf fibers as they are collected from the leaves of the plant. Abaca, banana, sisal and pineapple are the main members of this group. Fibers obtained from seed of plant are in the third category and are called seed fibers. Cotton, coir and kapok are the main family member of this group. Next group is grass fiber with family member corn, bamboo, wheat rice and barley. Corn and wheat stalks collected from the stalks of the plants comprises fifth group and named as core fibers. Fifth group is named as wood pulp fibers followed by root fibers as sixth group with family member luffa, swede and cassava. Here, the fibers are extracted from the root of the plant. Last group is fruit fibers which has tamarind, banana and coir as their family member.

Mohanty et.al 2005 [3] Natural fibers generally shows rigid behavior but still does not fractured during its processing. Most of the plant fibers mentioned earlier shows comparable properties with glass fibers in term of specific strength and stiffness.

Zhu et.al 2013 [4]Sisal fibers are extracted from the leaves of the sisal plant. Botanical name of the sisal plant is Agave sisalana. It is an herbaceous monocotyledonous plant from the Agavaceae family. This plant is found all over the world, but largely in tropical and sub-tropical region. This region comprises of North America, South America, Complete Africa, West Indies, Brazil, Tanzania and India. Though, Tanzania and Brazil are the two main producing countries among all. Coconut fiber is used by the scientific community from last one decade only. The coconut fiber is obtained from the husk of the coconut. Coconut fiber is thickest among the family of plant fibers. Climate of tropical region is best for the growth of coconut tree.

Lemos et.al 2017 [5]The problem faced by natural fiber as their utilization as reinforcement in polymeric resin can be solved either by physical modification technique or by treating the fibers with some chemicals which is known as chemical modification technique. Physical modification implies improving the mechanical adhesion between the matrix and reinforcing phase without changing the chemical properties of the fiber.

Gassan et.al 2000 [6] Physical modification improves the surface of the fiber so that it provides proper bonding between the two phases. There are various physical modification methods on which research has been conducted. The most common methods are corona, ultraviolet, plasma, fiber beating and heat treatment. Different methods had different impact on the surface of the fiber. Corona treatment and plasma treatment improves the surface energy of the fiber to establish improved bonding between the different phases.

Campilho, et.al 2017 [7] Alkaline treatment generate amorphous region and because of that a change in crystalline cellulose order is observed where the micro-molecules of the cellulose got separated and water molecules filled the generated space.

Bledzki, et.al 2015 [8] Silane treatment of fiber is an effective way and as compared to NaOH treatment, it was noted that, silane modification provide better result. Though, silane treatment is much costlier process as compared to alkaline treatment as soct of silane coupling agent is high. When fibers are treated with silane, a microsphere coating is formed over the surface of the fiber. This microsphere coating as act as a chemical link between the fiber and matrix body. This chemical linkage is given a name called Siloxane Bridge.

Tserki et al. 2005 [9] used acetylation over flax, wood and hemp fiber and found that treated fiber possess less moisture absorption as compared to untreated fiber. The treated fiber were observed under scanning electron microscope and revealed that treatment of fiber remove the non-crystalline constituent from the surface of fiber. This removal of non-crystalline substance has great impact on the properties of material as it changes the characteristics of the surface topography\

Zhang et al. 2005 [10] found that proper benzylation treatment can convert wood flour into thermoplastics. By the changing the concentration of chemical, authors found that that the properties of the material can be changed successfully. The developed material in their work is considered to be an alternative to petro-based material which can found applications in structural applications.

Li et al. 2007 [11] studied the work performed by the various researcher on peroxide treatment compiled the work and concluded that peroxide is an effective treatment method when combine with alkaline treatment and increases the adhesive property which in turn improves mechanical properties.

Khan et al. 2006 [12] in which they used a combination of NaOH and KMnO4 for treating natural fiber and observed improved results in mechanical properties. The natural fiber used in their investigation is coir fiber.

Wismon et al. 1996 [13] studied the effect of voids on inter laminar shear strength of the fiber reinforced polymer composites. They have studied the effect of length of voids on inter laminar shear strength rather than content of voids. They reported that when length of voids increases from 0.28 mm to 3.00 mm, ILSS decreases by 23 % and the decrement is proportional to the length.

Kakakasery et al.2015 [14] established dependency of residual flexural strength of composites with void content. They reported that the residual strength is slightly higher for a high-voidage laminate because large voids enhance the resistance to delamination, which is the predominant failure mode in post-impact flexural loading.

Jumaidin et al. 2016 [15] studied water absorption behavior of sugar palm fiber reinforced thermoplastic composites and found that with increase in the content of fiber, water absorption rate increases.

Nayak and Mohanty 2020 [16] investigated the erosive wear behavior of benzoyl chloride modified areca sheath fiber reinforced polymer composites. Specimens with 0, 10, 20, 30 and 40 wt. % of short fiber were prepared by injection molding process. Erosive response of the composites was examined using silica sand particles ($200 \pm 50 \mu m$) as erodent, sticking with different impact velocities of 48, 70, 82 and 109 m/s at impact angles of 15, 30, 45, 60 and 90°. It was found that composite with 10 wt. % fiber loading exhibits minimum erosion rate.

Durshan and Suresha 2019 [17] determined the effect of control factors on the abrasive wear behavior of waste silk fiber reinforced epoxy biocomposites using Taguchi method. Four parameters (sliding velocity, normal load and abrading distance) with five levels were considered for the study. L25 orthogonal array was used for experimental

run. Based on ANOVA analysis, authors concluded that abrading distance played a critical role on specific wear rate followed by fiber loading, normal load and sliding velocity.

Costa et al. 2015 [18] performed the similar study of sisal fiber with polyester matrix. The composite were fabricated by manual moulding technique. They evaluated the tensile strength of the composites and found different behavior from the obtained earlier. They found that tensile strength increases with fiber length.

Kumar et al. 2014 [19] used similar combination of sisal fiber and polyester resin and studied the effect of fiber loading and fiber length on tensile strength, flexural strength and impact strength. They used the length of fiber as 3 mm, 4 mm and 5 mm and varied the content of fiber from 30 wt. % to 50 wt. %. They reported that the various mechanical properties depend upon the length of fiber and fiber loading as well.

Rao et al. 2010 [20] fabricated sisal fiber reinforced polyester composites by varying the content of fiber from 10 volume % to 40 volume %. They used hand lay-up method for composite fabrication and evaluated the tensile and flexural strength of the composites.

Mahato et al. 2014 [21] used sisal fiber with vinyl ester polymer and fabricated the samples using hand lay-up method. They used sisal fiber of length 50 mm in their investigation. NaOH is used by them for modifying the surface of sisal fiber. They fix the concentration of NaOH used of 2 % and varied the time of treatment.

Naushad et al. 2017 [22] also used the combination of PP and sisal fiber and they used NaOH with maleic anhydride grafted PP and found that the tensile and flexural properties increases. MAPP supported the increment in properties. When they evaluated the mechanical properties of the material by reinforcing 40 wt. % of untreated fiber in PP matrix, they not found noticeable increment in mechanical properties. The mechanical properties evaluated by them are tensile and flexural properties.

Taguchi and Konishi 1987 [23] advocated the use of orthogonal arrays and Taguchi devised a new experimental design that applied signal-to-noise ratio with orthogonal arrays to the robust design of products and processes. In this procedure, the effect of a factor is measured by average results and therefore, the experimental results can be reproducible. This method is used by different researcher in the field of engineering. Implementation of Taguchi method for optimization of process parameter for studying the tribological behavior of is performed by many. But, usage of this technique for studying the tribological behavior of natural fiber reinforced polymeric composites is still rare. Few latest work done were discussed in this section. In a recent work, abrasive wear behavior of surface modified jute fiber reinforced epoxy composite were studied by Swain and Biswas.

Swain et.al. 2017 [24]. This method is used by different researcher in the field of engineering. Implementation of Taguchi method for optimization of process parameter for studying the tribological behavior of is performed by many. But, usage of this technique for studying the tribological behavior of natural fiber reinforced polymeric composites is still rare. Few latest work done were discussed in this section. In a recent work, abrasive wear behavior of surface modified jute fiber reinforced epoxy composite were studied

Paturkar et al. 2018 [25] performed dry sliding wear test for jute fiber reinforced epoxy composites. They also used well-established Taguchi method for design optimization. Four control factors selected by them are fiber loading, sliding velocities, applied load and sliding distance. The level of their experiment was set to three.

METHODOLOGY

Raw material used

In present work, short fiber reinforced polymer composites are fabricated. The polymer used is a thermoset polymer named epoxy resin and the fiber used is sisal fiber which is a natural fiber.

Epoxy

Matrix materials are the base of composite fabrication. The presently used matrix is a thermoset polymer epoxy. The epoxy resin Lapox-12 is used in the present work. Bisphenol-A-Diglycidyl-Ether (commonly abbreviated to DGEBA or BADGE) is the common name of the presently used epoxy resin and its molecular chain structure is shown in Figure 3.1. It provides a solvent free room temperature curing system when it is combined with the hardener tri-ethylene-tetramine (TETA) which is an aliphatic primary amine with commercial designation HY 951 (Figure 3.2). It is a liquid, unmodified epoxy resin of medium viscosity which can be used with particular hardener for making composites.



Figure 1 Epoxy resin and corresponding (Fabio Bignotti at.el. 2011)

Characteristic Property	Values	Units	
Density	1.13	g/cm ³	
Tensile strength	27	MPa	
Tensile modulus	6850	MPa	
Extension at break	0.8796	Mm	
Flexural strength	27.3	MPa	
Flexural modulus	1545	MPa	
Compressive strength	82.5	MPa	

Micro-hardness	0.087	GPa		
Specific wear rate				
Table 1 Important properties of epoxy resin (M.Ramesh at.el. 2013)				

Sisal Fiber

The Sisal fiber used in present work was obtained from the local market as it is used in rural areas for making cord, met etc. The fiber was extracted from the leaf of the plant Agave-Sisalana which is available in plenty in the Southern part of India.



Figure 2 Long and short sisal(A.C.H.Barreto at.el.2011) fiber

Sodium Hydroxide

A NaOH flake is supplied by Rankem Corporation Limited; New Delhi, India is used for modification of fiber surface in present work. Aqueous solution is prepared with particular concentration NaOH for treating fibers.

Composite fabrication

Simple hand lay-up technique is used in the present investigation for fabrication of epoxy based composites. This method is considered as the simplest technique for composite fabrication. Using this method, different sets of composites are fabricated with different sisal fiber loading. In present work, four fiber loading levels i.e. 2.5 wt. %, 5 wt. %, 7.5 wt. % and 10 wt. % are selected for preparation of composites.

Surface modification of sisal fiber

Sisal fibers received are long sisal fiber. First step is to cut the fibers with an approximate size of 4 mm. Sisal fiber were cut with the help of scissor. Distilled water is used to prepare aqueous solution of NaOH. Three different concentration value solutions are prepared with 2 moles NaOH, 4 moles NaOH and 6 moles NaOH. All the solutions are kept in different vessels to treat the sisal fiber separately. Sisal fibers are than added in solution prepared in three different beakers. The duration of immersion is made constant to 2 hours as the time of immersion is decided by the condition of fiber at various times during the treatment. Also, the ambience conditions are kept constant to atmospheric.



Figure 3 Surface modification of sisal fiber treatment with NaOH aqueous(A.C.H.Barreto at.el.2011)

Density and void content

The experimental density (ρ_{ce}) of composites under study is determined by using Archimedes principle using distilled water as a medium (ASTM D 792-91). The relationships between the mass of the body, the volume of the body and the density of solid body immersed in liquid as described by Archimedes form a basis for the determination of the density of substances. According to the principle, if density of the liquid is known and the volume of the liquid displaced is measured, the apparent loss of weight is calculated and hence density of the composite is obtained.

$$\rho_{ce} = \frac{\rho_w W_a}{W_a - W_w} \tag{3.1}$$

Here ρ_{ce} is the measured density of composite, ρ_w is the density of water, W_a is weight of sample in air and W_w is weight of sample in water. The theoretical density (ρ_{ct}) of composite materials in terms of weight fractions of different constituents can easily be obtained using rule of mixture model

$$\rho_{ct} = \frac{1}{\left[\left(\frac{w_f}{\rho_f}\right) + \left(\frac{w_m}{\rho_m}\right)\right]}$$
(3.2)

The volume fraction of voids in the composites is calculated by following equation:

$$V_{v} = \left(\rho_{ct} - \rho_{ce}\right) / \rho_{ct} \tag{3.3}$$

Here V_{ν} gives the amount of voids present in the composite body.

Water absorption behavior

Water absorption rate of the composites is investigated as per ASTM D 570. The specimens were submerged in water in room temperature for study the kinetics of water absorption. The samples were taken out periodically i.e. after each 24 hours and weighted immediately. The content of water absorption by sample was found out using a precise weighing machine. The test duration is not fixed at it is depended upon the time span in which the saturation level reach i.e. further intake of water by composites ceases. The percentage of water absorption is given by:

Water absorption (%) =
$$\frac{w_2 - w_1}{w_1} \times 100$$
 (3.4)

where w_1 is weight before soaking into water (g) and w_2 is weight after soaking into water (g).

Taguchi Method

The Taguchi experimental design was developed by Dr. Genichi Taguchi while working at the Japanese telecommunications company NTT in the 1950s and 1960s. He tried to utilize the experimental methodology to attain low-cost as well as high quality design solutions. The Taguchi experimental design is one of the best method used to determine minimum number of experiments to be performed within the permissible limit of levels and parameters.

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Experiment No.	Fiber Loading	Normal	Sliding	Sliding Velocity
	(wt.%)	Load (N)	Distance (m)	(m/s)
1	0.0	10	500	1

2	0.0	20	1000	2
	0.0	20	1000	_
3	0.0	30	1500	3
4	0.0	40	2000	4
5	0.0	50	2500	5
6	2.5	10	1000	3
7	2.5	20	1500	4
8	2.5	30	2000	5
9	2.5	40	2500	1
10	2.5	50	500	2
11	5.0	10	1500	5
12	5.0	20	2000	1
13	5.0	30	2500	2
14	5.0	40	500	3
15	5.0	50	1000	4
16	7.5	10	2000	2
17	7.5	20	2500	3
18	7.5	30	500	4
19	7.5	40	1000	5
20	7.5	50	1500	1
21	10.0	10	2500	4
22	10.0	20	500	5
23	10.0	30	1000	1
24	10.0	40	1500	2
25	10.0	50	2000	3

where *n* indicates the number of observations, *y* denotes the observed data, \vec{Y} denotes the mean and *S* represents the variance. The *S/N* ratio for specific wear rate comes under 'smaller-is-better' characteristic, and can be determined using equation 3.6. One of the important steps in Taguchi experimental design is selection of process parameters. found that fiber loading, normal load, sliding distance and sliding velocity affects the wear characteristics of fiber reinforced composites. In the present work, the effect of four parameters with five levels were examined using L₂₅ (4⁵) orthogonal array. The test parameters and their levels for abrasion test are shown in Table.

The optimal parameter settings for specific wear rate are determined based on Taguchi orthogonal array. The parametric combination for each experiment is show in table. All the tests are performed at room temperature. A full factorial experiment having four parameters and five levels requires 45 = 256 runs, whereas it reduces to 25 runs only, on using the Taguchi orthogonal array. The results obtained from experiment are converted into S/N ratio. These S/N ratios act as the objective function for optimization.

RESULTS AND DISCUSSION

Physical Characteristics

Scanning Electron Microscopy

The properties of the composites are strongly affected by the compatibility between the matrix and filler phase. In order to assess this fiber-matrix interaction, the dispersion of fiber in the matrix body and bonding between the fiber and the matrix are observed under scanning electron microscope (SEM). Prior to that, the effect of chemical treatment on the surface of sisal fiber is studied.



Figure 4 Micrograph of sisal fibers (a) Raw sisal fibers, (b) 2 Mole NaOH treated sisal fiber, (c) 4 Mole NaOH treated sisal fiber, (d) 6 Mole NaOH treated sisal fiber

Effect of Normal Load on Specific Wear Rate of Composites

Represents the effect of normal load on the specific wear rate of treated sisal fiber reinforced epoxy composites keeping remaining parameters constant (i.e. at sliding distance: 1500 m and sliding velocity: 3 m/s). It is clear from the figure that the specific wear rate of composites increases with increase in normal load. Similar observation has been reported by Kumar and Panneerselvam in case of glass fiber reinforced nylon composites. The increase in specific wear rate at higher load may be due to thermal softening. At higher load, contact temperature between sisal fiber reinforced epoxy composite and disk increases and easy detachment of soften layer from the surface of material occurs. This is indicating the improvement in wear behavior with addition of fibers in epoxy. However, composites with 10 wt. % of fiber loading exhibits minimum specific wear rate at different normal load when compared to other specimens.

CONCLUSIONS

- When sisal fibers are added in epoxy resin, the wear resistance of the developed material increases as compared to the neat epoxy.
- From Taguchi design of experiment it is found that actor combination of A5B3C4D5 i.e. fiber content 10 wt.%, normal load 30 N, sliding distance 2000 m and sliding velocity of 5 m/s will result in minimum specific wear rate.
- Confirmatory test showed that the predicted and experimental values are in good correlation. A difference of 4.39 % is observed in between predicted and experimental values.
- Specific wear rate of composites increases with increase in normal load. Pure epoxy shows maximum specific wear rate irrespective of different normal load. Composites with 10 wt.% of sisal fiber content exhibits better wear resistance compared to other fabricated composites.
- From the above investigation it is seen that modification of surface of sisal fiber using aqueous solution of NaOH improves the various properties of composite and best result is obtained when fiber surface are modified with 2 mole NaOH aqueous solution.

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