Effect of Concentration of NaOH Treatment on Mechanical Properties of Epoxy/Sisal Fiber Composites

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Abstract. In recent years, natural fibers incorporation in polymeric resin has received huge attention among the research community. The reasons of demand are multiple that includes its light weight, environmental friendly nature; non-toxic, low cost, easy availability, low processing cost and most importantly they possess characteristics which are comparable to conventional material. With this approach, present work comprises of fabrication of new category of natural fiber reinforced composites with polymer as base matrix. Sisal fiber is selected as reinforcing phase with epoxy matrix. Four different combination of composites are prepared with sisal fiber loading varies from 2.5 wt. % to 10 wt. % using well-known hand lay-up method. Sisal fibers were treated with NaOH at varied concentration to observe the effect of surface modification and its concentration on the developed material. Three different concentration of NaOH is used i.e. 2 mole, 4 mole and 6 mole for preparing three sets of composites. One set is prepared with raw sisal fiber to make total of four sets of composites. Mechanical properties under investigation are tensile behaviour, compressive behaviour and flexural behaviour. The experimental results obtained are compared for optimizing the concentration of NaOH. From the analysis it is seen that composite with surface modified sisal fiber yield better results and further fiber treated with 2 mole NaOH concentration is superior among their counterpart. The maximum tensile strength, compressive strength and flexural strength obtained are 31.5 MPa, 72.5 MPa and 37.8 MPa respectively. All these values are obtained for fiber treated with 2 mole NaOH aqueous solution.

1. Introduction
In past few years, there has been a strong awareness for products from renewable sources. Environmental friendly products are in demand mainly because of availability of new derivatives for recycling and also due to social influencing. In this regards, composite materials are being established to improve and adapt outdated products and present fresh products in a sustainable and answerable way [1]. Polymer composite materials derived from some sort of natural resources are successfully replacing the non-biodegradable composite material and even various conventional materials. The reasons behind this are the multiple benefits provided by them which include its light weight, low cost, ease and cheap manufacturing, high flexibility, high specific strength, biodegradable and environmental friendly in nature [2]. Apart from this,
natural fibers do not generate any harmful gases during its processing. But the development of high performance composite material derived from natural resources is an ambitious goal which is presently of interest among the scientific community all across the world. The main concern with natural fibers is their hydrophilic nature as it restricts them to be the first choice as reinforcement with polymeric resin. Because of this hydrophilic nature, the composite fabricated had high water absorption rate which has several drawbacks like poor matrix-fiber interfacial adhesion, improper distribution of fiber within polymeric matrix and low stress transfer efficiency [3]. A significant work is in progress to overcome this problem by modifying the surface of the fiber with chemical treatment process. It has been observed that there is tremendous enhancement in various properties of the natural fiber composite material developed by this method [4, 5].

Surface modification of natural fibers results improved adhesion between matrix and fiber and simultaneous reduce the hydrophilic nature of the fiber. The various chemical treatment methods implemented are alkaline treatment [6], silane treatment [7], acetylene treatment [8], benzene treatment [9], peroxide treatment [10], potassium permanganate treatment [11], steric acid treatment [12]. Among them the most widely used treatment methods are alkaline treatment as it is simple, economic and effective. It has been reported that the speed of fiber fragmentation and disaggregation also got increased with this method. During the process of alkanization, hydroxyl group moves away from the structure of fiber and is replaced by –O-Na group. With the removal of hydroxyl group from the surface of the fiber, fiber resistance against moisture increases. Alkaline treatment also cleans the surface of the fiber and makes them uniform which results in enhanced stress transfer capability of the fiber. Proper alkanization reduces the diameter of fiber as well and with reduced diameter, for given weight fraction, surface area of fiber increases which provide better adhesion with continuous phase [13]. Saravanakumaar et al. [14] reinforce alkali treated kenaf fiber in polypropylene matrix and compared the mechanical properties of composite with and without fiber treated composite. They reported to found significant improvement in mechanical properties when treated fiber was used. In more recent work, Asumani et al. [15] treated papaya fibers with NaOH aqueous solution at room temperature and varied the time of treatment. In their study they concluded that treatment time also govern the mechanical property of the developed material.

Among the various natural fibers, sisal fiber is considered to be the best option because of multiple benefits. A sisal fiber composite has various commercial uses mainly due its above average specific values, low density, environmental friendliness and cost-effectiveness. As compared to different NFCs, polymer composites prepared with sisal fiber as reinforcement delivers greater impact strength with reasonable tensile and flexural strength. This makes sisal fiber a potential candidate to be used as reinforcement in polymeric resin and used in applications where improved specific properties are required. But directly usage of raw sisal fiber as reinforcement will not fulfill the need and hence proper surface modification is required prior to use it as reinforcement. In this regards, the work concentrates on modifying the surface of the sisal fiber with aqueous solution of NaOH for fabrication of polymer composites with epoxy as base matrix. The main aim of the work is to present the influence of fiber modification on response to mechanical loading for the fabricated samples. Also the work consists of optimizing the concentration of NaOH to be used with sisal fiber for providing better results as under and over treatment both are undesirable. The different mechanical properties under consideration are tensile strength and extension, flexural strength and compressive strength.
2. Material and Methods

2.1. Raw Material Used
Thermoset epoxy resin LY 556 in combination with triethylene tetramine HY951 is selected as continuous 
phase in present work. Epoxy is readily available at low cost and processing is also quite easy which make
this polymer suitable for present investigation. Further it possesses good mechanical properties as
compared to other polymers and has excellent thermal properties as well. The sisal fiber used as
reinforcement is obtained from the local market as it is used in rural areas for making rope, mat etc.
Among the various natural fibers, sisal fiber is the most promising to be used as reinforcement in polymer
composites as it is easily and cheaply available. Also it possesses reasonably good mechanical properties.
In present investigation, short sisal fiber of approx. 4 mm length with 230 mm diameter is used for
composite fabrication. A NaOH flake is supplied by Rankem Corporation Limited; New Delhi, India is
used for modification of fiber surface.

2.2. Composite Fabrication
Epoxy based composites are prepared by simple hand lay-up method. Sisal fibers are treated with NaOH
solution before processing. The concentration level of NaOH considered in the study is 2 mole, 4 mole
and 6 mole concentration. Epoxy composites with both treated (three sets) and non-treated (one set) sisal
fibers are prepared at four different fiber loading i.e. 2.5 wt. %, 5 wt. %, 7.5 wt. % and 10 wt. %. In total
sixteen sets of composites are fabricated in present work. The list of fabricated composites is shown in
table 1.

| S. No. | SET A | SET B | SET C | SET D |
|-------|-------|-------|-------|-------|
| 1     | Epoxy + 2.5 wt. % raw sisal fiber | Epoxy + 2.5 wt. % sisal fiber (Surface modified with 2 mole NaOH aqueous solution) | Epoxy + 2.5 wt. % sisal fiber (Surface modified with 4 mole NaOH aqueous solution) | Epoxy + 2.5 wt. % sisal fiber (Surface modified with 6 mole NaOH aqueous solution) |
| 2     | Epoxy + 5 wt. % raw sisal fiber | Epoxy + 5 wt. % sisal fiber (Surface modified with 2 mole NaOH aqueous solution) | Epoxy + 5 wt. % sisal fiber (Surface modified with 4 mole NaOH aqueous solution) | Epoxy + 5 wt. % sisal fiber (Surface modified with 6 mole NaOH aqueous solution) |
| 3     | Epoxy + 7.5 wt. % raw sisal fiber | Epoxy + 7.5 wt. % sisal fiber (Surface modified with 2 mole NaOH aqueous solution) | Epoxy + 7.5 wt. % sisal fiber (Surface modified with 4 mole NaOH aqueous solution) | Epoxy + 7.5 wt. % sisal fiber (Surface modified with 6 mole NaOH aqueous solution) |
| 4     | Epoxy + 10 wt. % raw sisal fiber | Epoxy + 10 wt. % sisal fiber (Surface modified with 2 mole NaOH aqueous solution) | Epoxy + 10 wt. % sisal fiber (Surface modified with 4 mole NaOH aqueous solution) | Epoxy + 10 wt. % sisal fiber (Surface modified with 6 mole NaOH aqueous solution) |

2.3. Characterization
Mechanical characterization of the samples includes response of material on tensile loading, flexural
loading, compressive loading and indentation. All the experimentation under consideration are conducted
in computerized Instron 3382 Universal testing machine. Tensile tests were performed in accordance with
For conducting the compressive test, the fixture of UTM needs to be changed. The grip for holding the specimen is changed to 50 mm diameter hardened-steel compression platens. The samples are of cylindrical shape with length to diameter ratio of 2. The compression test is performed as per ASTM D695 standard. The flexural test was carried out as per ASTM D2344-84 standard in the same universal testing machine. The size and shape of the samples used for testing are also as per the ASTM standard.

3. Results and Discussion

3.1. Tensile Strength

Ultimate tensile strength of all sets of material developed in present work is presented in figure 1. Figure represents the change in tensile strength of the prepared composites at varied fiber content and concentration of NaOH used for fiber treatment. It is clear that inclusion of fibers in small fraction i.e. 2.5 wt. % reduces the tensile strength drastically irrespective of untreated or treated fibers. Later, when loading of fiber increases, increase in value of tensile strength is noticed. The trend is similar for all composites under investigation. Ultimate tensile strength of neat epoxy is 27 MPa as per the measurement. With the addition of raw sisal fiber, the value reduces to 16.4 MPa for a combination of epoxy with 2.5 wt. % raw sisal fiber.

With sisal fiber added further, tensile strength increases with fiber content and maximum tensile strength of 24.5 MPa is obtained with 10 wt. % of sisal fiber. Tensile strength of the composites increases with fiber content due to reinforcing effect of sisal fibers. But still the maximum value obtained for raw sisal fiber with maximum fiber content is less than that of the tensile strength of neat epoxy. This can be explained due to lower interfacial adhesion and compatibility between the two phases. Further, surface modification of sisal fiber using aqueous solution of NaOH results in improvement in tensile strength of the fabricated composites. Alkaline treatment of sisal fiber removes wax and impurities present over the surface of the fiber. Alkalization also helps in removing some hemicellulose from the fiber surface. This results in generation of rough surface on fiber followed by fiber fibrillation. With this, effective surface area of fiber increases drastically to adhere with epoxy matrix. Hence, adhesion between fiber and matrix
improves via mechanical interlocking and thus for same fiber content, alkaline treated sisal fiber/epoxy shows improved tensile strength as compared to raw sisal fiber/epoxy.

Tensile strength also varies with concentration of NaOH used. Fibers surface modified with 2 mole NaOH aqueous solution delivers better results irrespective of content of fiber as compared to their counterparts. Maximum tensile strength of 31.5 MPa is obtained when 2 mole concentration of NaOH treated 10 wt. % sisal fiber is added in epoxy. The improvement is around 16 % over neat epoxy but 28.5 %, 20.2 % and 33.7 % over set A, set B and set C composites respectively. It is observed that when the concentration of NaOH for treatment increases, tensile strength decline. It can be explained as when concentration of NaOH is low i.e. 2 wt. %, than the surface material present over sisal fiber dissolves completely. Due to this rough surface is presented for epoxy resin for better mechanical interlocking, thus increases the tensile strength. When the concentration of surface increases, sisal fibers were severely corroded. This damage the surface of the fiber and severe fiber fibrillation occurs which reduces the mutual integrity of the fiber. Further, excessive fiber treatment partially removed the cellulose from fiber surface. Hence, limited concentration treatment of sisal fiber is suggested for better output.

Figure 2. Elongation at break of samples under study.

Figure 2 shows the dependency of extension at break variation with fiber content and concentration of fiber treatment. Epoxy is highly brittle in nature with extension of only 0.88 mm before it breaks. This limited the application of epoxy as highly brittle nature is not desirable in many applications especially where repeated loading took place. It can be seen that incorporation of raw sisal fiber changes its behavior from being highly brittle to slightly ductile where extension at break increases as the fiber loading increases. Elongation at break increases to 4.08 mm with 10 wt. % of raw sisal fiber. This is an increment of 363.6 %. The similar trend is obtained when sisal fiber are surface modified. Though, raw sisal fiber imparts more ductility as compared to treated fiber. It is mainly due to the improvement of single sisal fiber after treatment with NaOH. Also, elongation at break reduces as the concentration of treatment increases. This is due to sudden decreases in strength and severe fibrillation with excessive fiber treatment.
3.2. Compressive Strength

The bonding between the matrix and the fiber mainly govern the compressive strength of the composite material. The dependency of compressive strength with varying content of sisal fiber is plotted in Figure 3. It is clear from graph that, compressive strength of the material decreases with increase in fiber content. The decreasing trend in the value of compressive strength with increase in fiber content is for all sets of composites irrespective of treated or untreated fiber. Compressive strength of neat epoxy is measured to be 82.5 MPa. When raw sisal fiber incorporated in it, compressive strength reduces to 73.7 MPa with 2.5 wt. % of fiber. This decrement continuous and compressive strength reduces to 56.8 MPa when 10 wt. % of sisal fibers are added. This is a decrement of 31.15 %. Similarly, when 10 wt. % of treated fibers are incorporated in epoxy resin, compressive strength reduces to 72.5 MPa, 74.9 MPa and 67.4 MPa for 2 mole, 4 mole and 6 mole NaOH concentration respectively. The decrement is limited to 12.1 %, 9.2 % and 18.3 % for 2 mole, 4 mole and 6 mole NaOH concentration treated fibers respectively against the decrement of 31.15 % with raw sisal fibers. The phenomenon of decrease of compressive strength can be explained as addition of sisal in epoxy increases the brittleness of the material which has been seen in figure 3. This reduces the deformation of material significantly under similar loading condition. With sudden change of behavior, relative deformation reduces which allow early rupture of material and result in reduction in the value of compressive strength. Again, treated fibers show comparatively better results as compared to raw fibers. This is mainly because of the enhanced compatibility between the fiber and matrix post treatment of fibers.

![Compressive Strength Graph](image)

**Figure 3.** Compressive strength of samples under study.

3.3. Flexural Strength

The flexural strength of samples with and without surface modification of fibers is shown in figure 4. The trend obtained with treated fibers is different from those with untreated fibers when flexural strength is under consideration. Flexural strength of neat epoxy is 27.3 MPa. When raw sisal fibers are added in epoxy resin, flexural strength of composites increases when small quantity of fibers are added. When 2.5 wt. % of fibers are added, flexural strength increases to 28.1 MPa. With further addition of raw sisal fiber, flexural strength reduces with raw sisal fiber content. When 10 wt. % of raw sisal fibers are added, flexural strength reduces to 26.9 MPa. Though the decrement is very less but decreasing trend is obtained with raw sisal fibers. First, the flexural strength increases with fiber content due to proper wetting of fibers...
and when loading of raw fibers increases, low interfacial adhesion and improper fiber wetting decreases the flexural strength of the material. Against that, when treated sisal fibers are incorporated in epoxy resin, flexural strength of the composites increases and the trends continue as the fiber content increases. Similar trend is obtained with all treated fibers irrespective of the concentration used. The enhancement in flexural strength with treated fiber is due to strong fiber-matrix interfacial bonding. With surface modification, hydrophilic nature of the fiber reduces as seen from the result obtained from water absorption test. With reduction in hydrophilic nature, load bearing capacity increases.

Figure 4. Flexural strength of samples under study.

With this, material withstand high bending load as compared to its counterpart. Further, sisal fiber treated with mole NaOH concentration yield better result as compared to other similar sets. It is again due to damage of fiber when over treatment occurs. Maximum flexural strength of 37.8 MPa is achieved with 2 mole NaOH treated fiber at 10 wt. % of sisal fibers. The appreciable increment of 38.5 % is observed with this combination. Again, when 4 mole NaOH treated fiber is used, 35.1 MPa of flexural strength is obtained and when 6 mole NaOH treated fiber is used, 32.5 MPa of flexural strength is noticed. This is an increment of 28.5 % and 19.2 % respectively.

4. Conclusion

The surface modification effect of sisal fiber with aqueous solution of NaOH on various mechanical properties under consideration is studied and following conclusion is drawn from the investigation.

1. With the addition of sisal fiber, tensile strength of composites reduces for a combination of epoxy with 2.5 wt. % sisal fiber. With further addition of sisal fiber, tensile strength increases.
2. Tensile strength also varies with concentration of NaOH used for alkaline treatment of fibers. Fibers treated with 2 wt. % NaOH aqueous solution delivers better results.
3. Incorporation of raw sisal fiber changes epoxy behavior from being highly brittle to slightly ductile where extension at break increases with increase in fiber content and hence extension at break decreases with increase in fiber content. Further, elongation at break reduces as the concentration of treatment increases.
4. Compressive strength of the material decreases with increase in fiber content. The decreasing trend in the value of compressive strength with increase in fiber content is for all sets of composites irrespective of treated or untreated fiber.
5. When raw sisal fibers are added in epoxy resin, flexural strength of composites increases when small quantity of fibers are added. With further addition of raw sisal fiber, flexural strength reduces with raw sisal fiber content. Against that, when treated sisal fibers are incorporated, flexural strength increases and the trends continue as the fiber content increases.
6. From the above investigation it is seen that modification of surface of sisal fiber using aqueous solution of NaOH improves the various mechanical properties of composite and best result is obtained when fiber surface are modified with 2 mole NaOH aqueous solution.

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