

Mechanical and Water Absorption Properties of Sisal-Fibre-Reinforced Polypropylene Composites for Ceiling Applications

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(Received 30 September 2013; Revised 10 February 2014; Accepted 25 February 2014)

Abstract: This work investigated the effect of water on sisal-fibre-reinforced polypropylene composites used in ceiling applications. Sisal-fibre was extracted by soil retting after which specimens were chemically treated with 1 molar solution of KOH, NaCl, HCl and Ethanol, respectively. Both treated and untreated sisal-fibres were used for the reinforcement of homopolymer and copolymer polypropylene. The composites were produced by a compression moulding technique, after which mechanical tests such as: tensile, impact and hardness tests were carried out on the samples. This was followed by water absorption tests using rainwater and distilled water. A Scanning Electron Microscope (SEM) was used to study morphology of the fractured surface of the composites after the test. The results showed that chemical treatment for the modification of the fibre surface can be exploited to enhance the mechanical and water absorption properties of the composites. An optimum result was obtained in KOH treated sisal-fibre-reinforced homopolymer polypropylene composites.

Keywords: Mechanical properties, water absorption, sisal- fibre, chemical treatment

1. Introduction

Growing environmental awareness, increasing concern for the green-house effect and bio-degradation have spurred construction, automotive and packaging industries to develop and or use materials which have the least environmental impact.

Natural-fibers reinforced bio-degradable composites seem a good alternative to man-made fibres, because the source of the fibre is renewable. Natural-fibre composites tend to be light-weight; strong; and stiff. They are better than man-made fibres because of their low cost, low density, recyclability and biodegradability (Frederick and Norman, 2004). Natural-fiber reinforced composites have attracted the attention of the research community because they are an alternative to fibres derived from ever depleting petroleum sources. Additionally, the relatively high cost of synthetic fibres, makes natural fibres an attractive alternative. Natural-fibre reinforced composite matrices are widely used in several technical applications (Rakesh *et al.*, 20011). Hemp and flax, are successfully used as packaging material, interior panels in vehicles, and building components, among others (Hautala *et al.*, 2004).

Sisal (*Agave sisalana*) is a plant that yields stiff fibres traditionally used for rope and twine. It is usually extracted by a process known as *decortications*. Several authors have reported the chemical composition, properties of sisal-fibres and their composites by

incorporating the fibre in different matrices before and after treatment by different methods (Habibi *et al.*, 2008). The mechanical process yields a good quality of fibre compared to the retting process, while the yield is more in the latter case. Hence, there is the need for further research on how the quality of the sisal-fibre derived by the retting process can be improved.

One difficulty that has prevented the use of natural fibres is lack of good adhesion with the polymeric matrix. In particular, the large moisture absorption by natural fibres in a hydrophobic matrix adversely affects adhesion and leads to premature ageing by degradation and loss of strength (Rai and Jaisingh, 1986). There are, however, several separation-of-fibre processing techniques such as mechanical or chemical pulping, whereby lignin is degraded and dissolved, leaving most of the cellulose and hemicelluloses in the form of fibres. This generally has an important effect on both the mechanical and chemical properties of the fibres. Water and dew retting have been employed where there is surplus water and dew respectively, for the extraction of natural fibres. However, the products have not been as good as those from the decortications process.

This work investigated the influenced of the soil retting process and chemical treatment on the mechanical and water absorption properties of sisal-fibre-reinforced polypropylene composites. Due to the difficulty of achieving the best combination of polymer

and natural fibre where the problem of polarity will be minimal, two grades of the polypropylene; homopolymer and copolymer, were used as a solution to this problem. The research focused on reducing environmental hazards caused by advanced processing of materials; and on producing low-cost, strong, durable materials.

2. Materials and Methods

2.1 Extraction of the Sisal-Fibre

Sisal-fibre was chosen for this investigation, because of its availability, ease of extraction, good strength (Oladele *et al.*, 2010) and low density. Sisal-fibre was extracted from the plant leaves using the soil retting method. This was carried out by cutting the leaves and burying them under ground for 15 days so as to allow fermentation to take place.

For the reaction to proceed naturally, the leaves were buried close to stream of water and were watered at daily intervals. The combination of water and sunlight during the day causes reactions to take place which led to the gradual, natural decomposition of the sisal-fibre binder. The fermentation process was monitored by examining the material daily after seven days, so as to determine when best to exhume them. The fermented leaves were disinterred washed and then sun dried for seven days. The extracted sisal-fibre is as shown in Figure 1. The fibre was stronger, due to the absence of stress induction from beating, than decorticated fibres.



Figure 1. Extracted Sisal-Fibre by Soil Retting Process

2.2 Determination of the Sisal-Fibre Diameter

The diameters of the sisal-fibres were determined with the aid of digital vernier calliper. The diameters varied, ranging from 0.08 mm to 0.10 mm. The average diameter was 0.09 mm.

2.3 Determination of the Sisal-Fibre Density

The density of the sisal-fibre was determined using the Archimedes' principle. This was carried out by dropping

a known weight fibre in a measuring cylinder containing water. The water displaced was measured. The test was carried out three times and the average value was used. The density was determined as follows;

$$\rho = M/V$$

where,

$$\rho = \text{Density}$$

$$M = \text{Mass in g}$$

$$V = \text{Volume in cm}^3$$

$$\text{Average Mass of Fibre} = 2 + 2 + 2 = 6/3 = 2\text{g}$$

$$\text{Average Volume of water displaced} = 4 + 4 + 3.8$$

$$= 11.8/3 = 3.98 \text{ cm}^3$$

$$\text{Density} = 2/3.98 = 0.510 \text{ g/cm}^3$$

The density of the polypropylene (homopolymer and copolymer) is 0.905 g/cm³.

2.4 Chemical Treatment

The chemicals selected are readily available and cause little harm to the environment. While hemicellulose is hydrophilic, soluble in alkali and easily hydrolyzed in acids, lignin is amorphous and hydrophobic in nature. Lignin is not hydrolyzed by acids, but soluble in hot alkali. Since these are the unwanted constituents to be removed, acid and hot alkali were used for the treatment of the fibre so as to improve the strength of the fibre and ensure proper bonding of the fibre and the matrix.

Combinations of chemicals and mechanical treatments were carried out on different five samples while one was left untreated. The treatment was carried out by weighing out specimens, each consisting of 60 g of sisal-fibre. 1 M of either KOH, HCl, NaCl or Ethanol was added in a solution of 450 ml inside the shaker water bath and maintained at 50 °C for 4 hours respectively. The treated samples were washed thoroughly with water and finally washed with distilled water to ensure neutrality.

2.5 Compounding of the Composite Materials

Before the fibre was mixed with the polypropylene, it was further prepared by combing and oven drying. The combing was carried out to remove scales that stick to the strands, while the oven drying removed moisture from the fibres. Oven treatment was carried out at 70 °C for 1 hour using a vacuum oven.

Due to the difference in the densities of the polypropylene and the sisal-fibre, the two materials were first mixed and blended together using the compression moulding machine. Each blend that constituted each sample was prepared by mixing 180 g of the polypropylene matrixes with 9.0 g and 5.4 g of the sisal-fibres for 5 wt % and 3 wt % reinforced samples respectively. This was compacted at 190 °C, maintained at a pressure of 10 tons for 5 minutes followed by air cooling. The compounded samples were ground into 8 mm lengths.

2.6 Production of Composites

To test for tensile strength, 3 mm thick composites were produced in dumb-bell shaped moulds. A rectangular shaped mould of 150 mm X 100 mm X 3 mm was used for the production of samples for impact and hardness tests. The moulds were filled with the grounded compounded samples for 3 and 5 wt % sisal-fibre contents, respectively. The filled moulds were placed inside the compression moulding machine maintained at 190 °C and 15 tons for 15 minutes followed by air cooling.

2.7 Testing and Structural Characterisation of Cast Samples

After forming the composites, samples were prepared for tensile, Charpy impact and hardness tests. Scanning Electron Microscope (SEM) was used to investigate the miscibility between the fibre and matrix at the fractured surfaces. These tests were carried out as follows:

1) Tensile Test – The tensile test was carried out on the samples using an Instron Universal Tensile Testing Machine in accordance with ISO / R 527. The test piece which is of a gauge length of 25 mm was fixed at the edges of the upper and the lower grip of the machine. As the test-piece extended, a graph was plotted automatically and important tensile properties data were generated. The load applied was 25 KN at a crosshead speed of 5mm / minute. Three samples were tested. The average value was taken and used as a representative value.

2) Hardness Test – a hardness test was carried out on the samples using a Shore D hardness tester in accordance with ISO 868. The test was carried out by indenting the sample with the instrument for about 5 seconds before taking the reading that displayed on the gauge. Ten values were taken each for each sample of which the average is used as the representative value.

3) Impact Test - An impact test was carried out on the sample using a Charpy impact testing machine in accordance with ISO 179. Samples were cut into the impact test dimension of 80 mm x 10 mm x 3 mm and notched at the centre. Samples were placed horizontally on the machine, maintaining a distance of 60 mm between lines of supports. The initial reading of the gauge was taken and then a suspended handle that swings and fractures the sample was released. The final reading was taken after the sample has fractured. For each sample, three test pieces were tested. The average value was taken as the representative value.

4) Water Absorption - Water absorption tests were carried out in accordance with ISO 175. Two media were selected and used for the absorption tests. The media used were, distilled water and rainwater. These media were chosen because the polymer composite is likely to encounter them in its service. To carry out the test, 250 cm³ of water media were poured into clean, plastic containers. The initial weight of each of the

sample was taken using chemical weighing balance; FA2104A Model which has a precision of ± 0.0001 g. Readings were taken every day for seven days. To take the readings, the samples were brought out, cleaned with white cloth and weighed. The data collected was used to determine the % water absorption using the formula:

$$\% \text{ Water Absorption} = \frac{\text{Final Weight} - \text{Initial Weight}}{\text{Initial Weight}} \times 100$$

3. Results and Discussion

3.1 Response of the Composites to Young's Modulus of Elasticity Test

Young's Modulus of Elasticity measures the stiffness of materials. Figure 2 shows the response of the various samples using Young's Modulus of Elasticity.

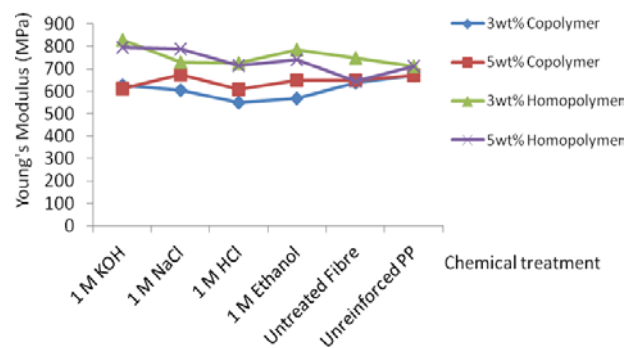


Figure 2. Variation of the Young's Modulus of Elasticity against Samples

1 M KOH treated sample in homopolymer 3 wt% fibre fraction gave the best Young's Modulus of Elasticity value of 831 MPa, followed by 5 wt% fibre fraction of the same sample with a value of 795 MPa and 5 wt% fibre fraction of sample treated with 1 M NaCl with a value of 789 MPa. The ability of the composites to bear the load efficiently is due to the fact that the bonding between the treated sisal-fibres and the homopolymer was greatly enhanced by the chemical treatment of the fibre. The emergence of KOH treated sisal-fibre as the best source of reinforcement in homopolymer may be due to the increase in the possible reaction sites as a result of the extraction process adopted: the soil retting process permits the extraction to proceed naturally in a controlled mode.

3.2 Response of the Composites to Ultimate Tensile Strength Test

The Ultimate Tensile Strength (UTS) of each composite is shown in Figure 3. A 1 M KOH-treated sample was used in a homopolymer 3 wt% fibre fraction gave a value of 32.70 MPa and a 1 M ethanol-treated sample in homopolymer 3 wt% fibre fraction gave a value of 32.33 MPa. The un-reinforced homopolymer marginally

exceeds having a value of 33.88 MPa. From the tensile test, KOH treated sisal-fibre-reinforced composite gave the best result.

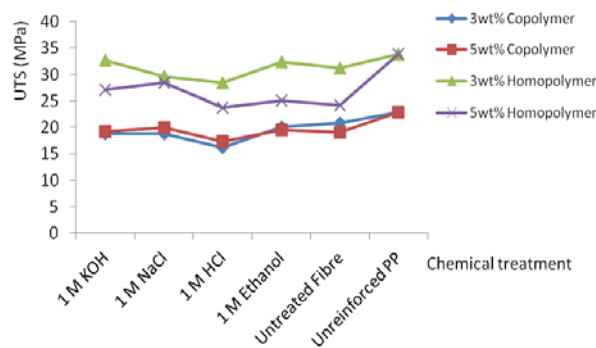


Figure 3. Variation of the Ultimate Tensile Strength against Samples

The result of the tensile test shows that the KOH treated sisal-fibre-reinforced composite sample with 3 wt % in homopolymer gave the best product with respect to the unreinforced and other treated composite samples. This may likely be due to the increases in the possible reaction sites as a result of the extraction process that precedes the treatment. Since lignin, which is amorphous, is readily soluble in hot alkali, the treatment was able to de-lignify the sisal-fibre, thereby enhancing the crystallinity and strength of the fibre (Oladele et al., 2010). Tensile properties are usually enhanced by crystallinity of the materials. Also, by obtaining the best result from 3 wt % shows that, the fibre was able to mix properly with the homopolymer matrix which allow adequate wetting of the fibre. This led to less contact between fibres which in many cases is usually responsible for weak interfacial bonding and strength in composites due to the inability of the matrix to transfer load to the fibres.

3.3 Response of the Composites to Hardness test

Figure 4 shows the variations in hardness of the samples. Reinforcement from the chemically treated fibre samples at 5 wt % gave the best results.

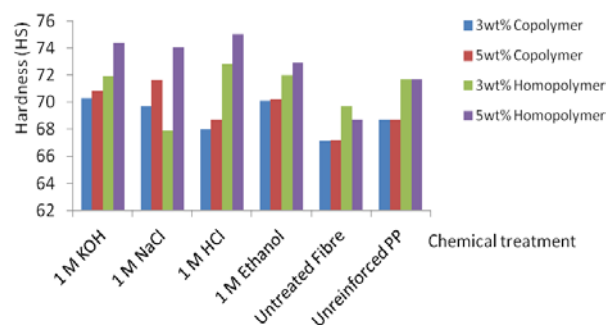


Figure 4. Bar Chart showing the Variation of Hardness Property against Chemical Treatment

Homopolymer matrix based composites gave good hardness values. This was due to the fact that homopolymer matrix is harder than copolymer matrix. The best result was obtained in 5 wt% HCl treated sisal-fibre-reinforced-homopolymer composite with a value of 75.0 HS, followed by 5 wt% KOH treated sisal-fibre-reinforced-homopolymer composite with a value of 74.4 HS.

Here, the hardness of the materials increases with an increase in the mass of the fibre used in both matrixes, because as the fibre increases, the density also increases. Higher density enhances higher material hardness. Treatment with HCl led to hydrolysis of the hemicellulose leaving most of the lignin, which is amorphous and this usually enhances hardness. The treatment, in general, increases the possible reaction sites between the fibre and the matrix. This is most likely encountered in homopolymer matrix based composites, and forms the basis for obtaining better results in this matrix.

3.4 Response of the Composites to Impact Test

Figure 5 shows the response of the sisal-fibre-reinforced polypropylene matrixes composites to the impact test that was carried out by the Charpy impact testing machine in accordance to ISO 179. The 3wt% sisal-fibre-reinforced copolymer samples treated with 1 M Ethanol had an average value of 0.0357 J/mm² and the 1 M KOH samples produced an average value of 0.0329 J/mm² was next to the unreinforced copolymer polypropylene matrix that has the best impact strength value of 0.0768 J/mm². Ethanol is a weak acid but has the tendency to produce moderate dissolution of the hemicelulose and lignin from the fibre. The treated fibre gave good impact strength as reinforcement in the copolymer matrix. The impact strength was enhanced in copolymer matrix because it is more ductile than homopolymer matrix.

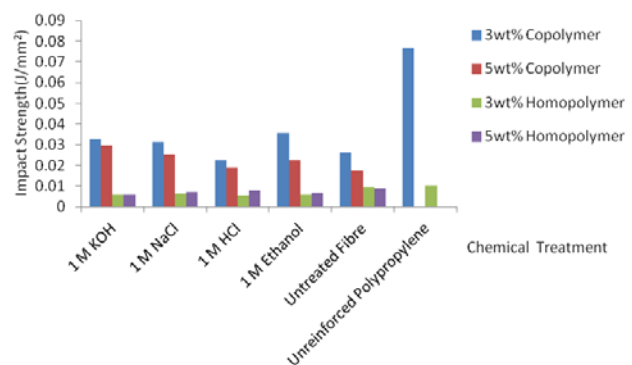


Figure 5. Variation of impact Strength with Chemical Treatment

The impact strength is found to decrease with an increase in fibre content due to poor interfacial bonding that induces micro-spaces at the fibre-matrix interface.

These micro-spaces cause micro-cracks when impact occurs, resulting in crack propagation and giving a decrease in the impact strength of the composites. The impact strengths of the treated sisal-PP composites are found to be higher than those of the untreated ones, indicating that better interfacial bonding between the matrix and the fibre has occurred upon chemical treatment. As a result, the chemically treated sisal polypropylene-PP composites are capable of absorbing higher amounts of energy, compared to the untreated ones thereby reducing crack propagation.

3.5 Response of the Composites to Water Absorption Properties

Figure 6 shows variations in water absorption for the homopolymer based composites in rain water medium (HR), copolymer based composites in rain water medium (CR), homopolymer based composites in distilled water medium (HD) and copolymer based composites in distilled water medium (CD) respectively. This test ascertained the suitability of the material under leakage conditions that usually occur in some buildings. From the results, both the composite and the unreinforced polypropylene materials exhibit higher resistance to water absorption in rainwater medium (R) than distilled water medium (D). There is progressive increase in water absorption from 3 to 5 wt % in most of the results. The results show that unreinforced homopolymer and homopolymer based composites (H) exhibit higher resistance to water absorption than unreinforced copolymer and copolymer (C) based composites. By and large, the unreinforced polypropylene matrixes have the best resistance to water absorption than their composites.

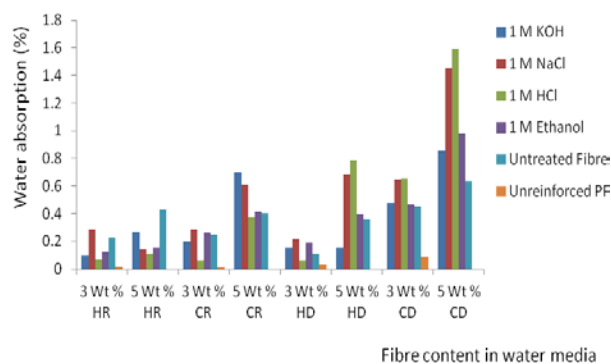


Figure 6. Variation of Water Absorption Properties against Fibre Content in Rain (R) and Distilled (D) Water Media for the Composites

The results showed that, HCl-treated fibre-reinforced composite samples gave the overall best performance in rain water medium, while its performance was poor in distilled water medium, except at 3 wt % homopolymer based composites where its

performance was also good. It was observed that, KOH-treated fibre-reinforced composite samples gave the overall best performance in both media and in both matrixes followed by the untreated fibre-reinforced samples. These chemically-treated fibre-reinforced composites possess high resistance to water due to the removal of hemicelluloses which is hydrophilic. Also, the untreated fibre-reinforced composites show good results which justify the use of sisal-fibre as reinforcement in polymers.

The hydrophilic nature of fibre surfaces is due to the presence of hydroxyl and polar groups of cellulose. It leads to swelling and agglomeration of fibres within the matrix when composites are formed. Furthermore, there is high moisture content which results in poor interfacial adhesion between fibres and matrix. These factors significantly decrease the mechanical properties of the composites and the use of these composites for various applications becomes less attractive (Parida *et al.*, 2013). By carrying out the above stated treatments, these problems are overcome.

From the mechanical test results, KOH treated sisal-fibre enhanced the properties of most of the composites, coupled with high resistance to water under different conditions. For ceiling sheet applications, KOH treatment of soil-retted sisal-fibre proves to be the best treatment to develop strong and light-weight from reinforced homopolymer and copolymer matrixes based composites.

3.6 SEM of Fracture Surfaces of the Composites

Alkaline treatment (mercerization) is one of the most used chemical treatments given to natural fibres. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fibre cell wall. It also depolymerises cellulose by disrupting hydrogen bonding in the network structure of cellulose and exposes the short length crystallites (Mohanty *et al.*, 2001). The alkaline treatment reduces the aggregation of fibre in the matrix and increases surface roughness. This results in better mechanical interlocking between fibre and matrix (Gomes *et al.*, 2007). The number of possible reaction sites also increases with an increase in the amount of cellulose exposed on the fibre surface. It leads to better adhesion between fibre and matrix (Dipa *et al.*, 2001).

Figure 7 shows the SEM for the fractured surfaces for the composites. Due to the increase in the number of exposure sites and possible reactions, as a result of increase in exposed cellulose, composites developed from the treated fibres produce the best products. The fibres (white) were more wet in homopolymer (black) as shown in Plates (c-d) than in copolymer (black) as shown in Plates (a-b), hence, better properties were achieved in the homopolymer matrix than in copolymer matrix based composites. These show that the reactions between homopolymer and the treated sisal-fibres were

more likely than that of the copolymer and the fibres, particularly in KOH treated fibres that gave the best results.

These are the reasons for the results that were obtained for mechanical and water absorption tests, where the properties were more enhanced in homopolymer matrix than in copolymer matrix based

composites. The chemical treatment was able to expose the surface of the sisal-fibre for proper bonding with the polypropylene, particularly, in the homopolymer matrix. These exposures were as a result of the removal of hemicelluloses and lignin which are mainly responsible for water absorption and weak interfacial bonding strength, respectively.

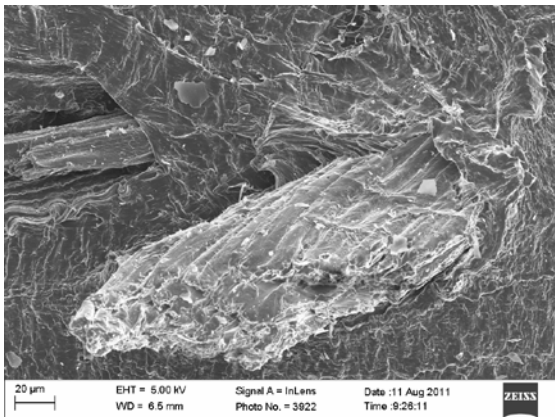


Plate a. Untreated Sisal Reinforced Copolymer Composite

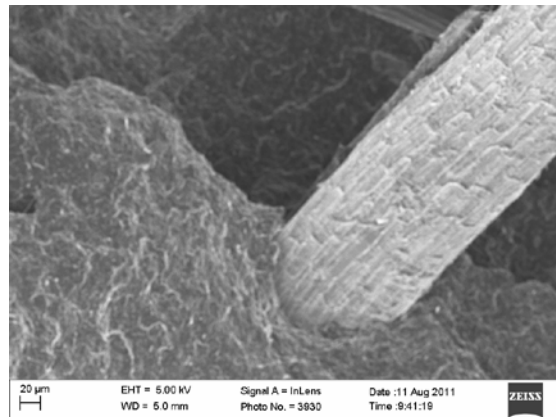


Plate b. KOH Treated Sisal Fibre Reinforced Copolymer Composite

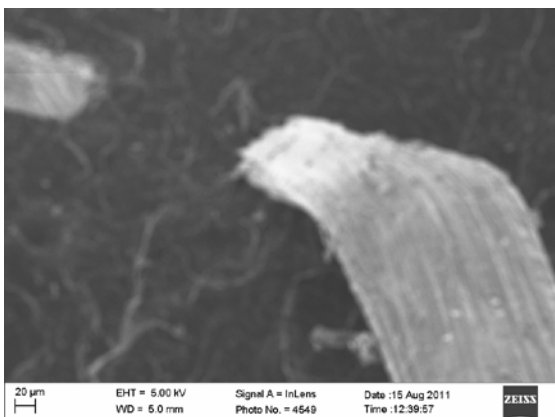


Plate c. Untreated Sisal Reinforced Homopolymer Composite

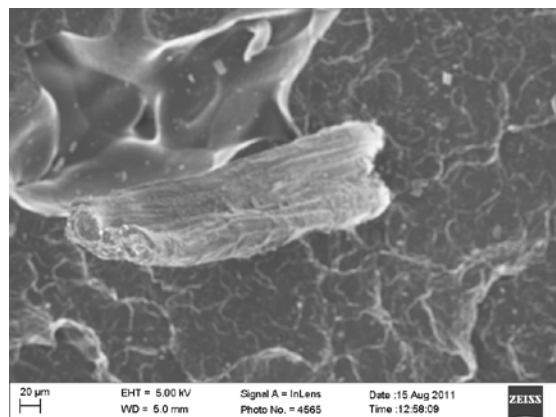


Plate d. KOH Treated Sisal Fibre Reinforced Homopolymer Composite

Figure 7. SEM (a-d) of the fractured surfaces of Sisal- Fibre-Reinforced Copolymer and Homopolymer Composites (Magnification 20 μm)

4. Conclusion

This work investigated the effect of chemically treated sisal-fibre on the mechanical and water absorption properties of sisal-fibre-reinforced-polypropylene composites targeted for use in ceiling applications.

The results revealed that samples developed from chemically treated sisal-fibres in a homopolymer matrix were the best in all the properties that were investigated, except for ultimate tensile strength (UTS) and impact properties.

Water absorption and mechanical properties (tensile and hardness) of the homopolymer matrix based

composites were better compared to the copolymer matrix based composites, while the impact property was better enhanced in copolymer composites. This was due to the fact that a copolymer matrix is more ductile than a homopolymer. It was showed that the use of these treated fibres in homopolymer matrix to develop its composites is a good advancement. Hence, KOH treatment was considered as the best treatment for the soil-retted sisal-fibre, since it has the greatest overall effect in all the properties considered for the composites.

Decortications have been the most widely used methods of extraction for sisal-fibre due to the advantages over water or due retting products. However,

in this work, soil retting used for the extraction shows that this can be used to extract sisal-fibre with good tensile properties and enhanced mechanical properties in polymer composites. The process was an improvement on the water retting process. It is an environmentally friendly process that can be used where there is enough soil.

Acknowledgements:

The authors wishes to thank members of the Polymer Composites Group, Tshwane University of Technology, CSSIR, Pretoria South Africa, for their technical assistance. Production and characterisation aspects of this work were carried out in South Africa.

References:

- Rakesh, K., Sangeeta, O. and Aparna, S. (2011), "Chemical modifications of natural fiber for composite material", *Pelagia Research Library*, Vol.2, No.4, pp. 219-228.
- Frederick, T.W. and Norman, W. (2004), *Natural Fibers Plastics and Composites*, Kluwer Academic Publishers, New York.
- Hautala, M., Pasila, A. and Pirila, J. (2004), "Use of hemp and flax in composite manufacture: A search for new production methods", *Composite Part A: Applied Science and Manufacturing*, Vol.35, pp.11-16.
- Habibi, Y., Heux, L., Mahrouz, M. and Vignon, M.R. (2008), "Morphological and structural study of seed pericarp of opuntia ficus: Indica prickly pear fruits", *Carbohydrate Polymers*, Vol.72, No.1, pp. 102-112.
- Rai, M. and Jaisingh, M.P. (1986), *Advances in Building and Construction Materials*, Central Building Research Institute, Roorkee.
- Oladele, I.O., Omotoyinbo, J.A. and Adewara, J.O.T. (2010), "Investigating the effect of chemical treatment on the constituents and tensile properties of sisal fibre", *Journal of Minerals and Materials Characterisation and Engineering*, Vol. 9, No.6, pp.569-582.
- Parida, C., Dash, S.K. and Das, S.C. (2013), "Effect of fibre treatment on mechanical and thermal properties of luffacylindrica-resorcinol composites", *International Journal of Fiber and Textile Research*, Vol.3, No.1, pp. 13-17.
- Mohanty, A.K., Misra, M., and Drzal, L.T. (2001), "Surface modifications of natural fibers and performance of the resulting biocomposites: an overview", *Compos. Interface*, Vol.8, pp. 313-343.
- Gomes, A., Matsuo, T, Goda, K and Ohgi, J. (2007), "Development and effect of alkali treatment on tensile of curaua fibers green composites", *Composite Part A, Applied Science and Manufacturing*, Vol.38, No.12, pp.1811-1820.
- Dipa, Ray, Sarkar, B.K., Rana, A.K. and Bose, N.R. (2001), "Effect of alkali treated jute fibres on composite properties", *Bulletin of Materials Science*, Vol.24, No.2, pp.129-135.

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