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Influence of Chemically Modified Sisal-Fibre on the Mechanical Properties of Reinforced Homopolymer Polypropylene Composites

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Abstract: *This research investigated the influence of chemical modification on the mechanical properties of soil-retted, sisal-fibre-reinforced homopolymer polypropylene (PP) composites. Sisal fibre was extracted by the soil-retting process, after which parts were treated with selected chemicals—KOH, HCl, NaCl and Ethanol—with varying mole fractions, producing 16 chemically modified sisal-fibre samples. Reinforced homopolymer PP composites were formed by using a compression molding machine to develop samples for mechanical tests—tensile, impact and hardness. From the results, it was revealed that 0.75 M : 0.25 M has higher synergistic effect than others, with 0.75 M HCl + 0.25 M KOH, thus emerging as the best chemical treatment. This treatment gave the best sisal-homopolymer PP composite in terms of hardness, tensile strength and impact strength (in the as notched condition). The chemical treatments were found to be effective in enhancing the properties of sisal-homopolymer PP composites.*

Keywords: *Sisal fibre, Chemical treatment, Homopolymer PP, Mechanical Properties, Reinforcement, Composites*

1. Introduction

The emergence of polymers in the beginning of the 19th century ushered in a new era of using fibres in diverse applications. Because of their superior dimensional and other properties, synthetic fibres gained popularity and slowly replaced natural fibres in different applications. However, the production of synthetic composites requires a large quantum of energy and pollutants are generated during the production and recycling of these synthetic materials. This has renewed interest in natural fibres, spurring research in making natural fibres superior to synthetic ones. There have been tremendous strides in improving the quality of natural fibres, and as such they are fast emerging as the preferred reinforcing material in composites.

Considering the high performance standard of composite materials in terms of durability, maintenance and cost effectiveness, natural-fibre-based composites find extensive use in building and civil engineering applications. In the case of synthetic-fibre-based composites, despite their usefulness in service, they are difficult to be recycled after the designed service life. But, natural-fibre-based composites are environmentally friendly to a large extent. Natural fibres like jute, flax, hemp, coir, and sisal have all been proved to be good reinforcement in thermoset and thermoplastic matrices which are used in the automobile, construction, as well as packaging industries, with a few drawbacks (Mohanty *et al*., 2002; Bledzki *et al.*, 2002; Gross and Karla, 2002; Puglia *et al.*, 2004). Fibre-reinforced polymers have better specific properties compared to conventional materials and find applications in diverse fields, ranging from appliances to spacecraft (Saheb and Jog, 1999).

Most of the composite materials used in different sectors are principally fabricated using thermosetting matrices. However, there are some disadvantages in using thermosets, which include brittleness, lengthy cure cycles and inability to repair and or recycle damaged or scrapped parts. These disadvantages have led to the development of the thermoplastic matrix composite system. Compared with thermosets, composites fabricated from thermoplastic materials typically have a longer shelf life, higher strain to failure, are faster to consolidate and retain the ability to be repaired, reshaped and reused as the need arises (Chand and Hashmi, 1993).

This paper presents the main findings from a research that investigated the suitability of chemically modified soil-retted sisal fibre as reinforcement in a homopolymer polypropylene (PP) thermoplastic material, to enhance the properties of the developed composite for building applications.

2. Materials and Methods

2.1. Materials

Homopolymer polypropylene (PP) which was used for this work was sourced from Sasol, South Africa. Other materials that were used; Sisal plant leaves, Teflon sheet, Silicones, KOH, HCl, NaCl, H_2SO_4, Ethanol, Acetic Acid, Nitric Acid, Benzene, Ether, Distilled Water, Loamy Soil, Stream Water, Sample Bags and Adhesive Glue.

2.2. Methods

2.2.1. Extraction of the sisal fibre material

The extraction of sisal fibre was carried out by soil-retting process using loamy soil as the retting medium. The source of sisal leaves used was from a sisal plantation. The leaves were cut and buried inside soil for 15 days so as to allow fermentation and decay of chlorophyll matter. For extraction to take place normally, the leaves were buried close to a stream of water and are watered daily. The fermented leaves were exhumed and washed thoroughly, after which the resulting fibres were sun dried. Figure 1 shows the sisal plant leaves while Figure 2 shows the extracted sisal fibre.

Figure 1. Sisal Plant

Figure 2. Extracted Sisal Fibre

2.2.2. Chemical treatment

To improve the surface morphology of the fibre for good adhesion between fibre and matrix, as well as prevent degradation of sisal fibre due to water absorption, chemical treatment was carried out on 60g samples of sisal fibre as follows:

i. 1M each of selected chemicals

The samples were treated with 1M each of KOH, HCl, NaCl and Ethanol, respectively in a solution of 450 ml inside a shaker water bath maintained at 50 ºC for 4 hours. The treated samples were washed thoroughly with water and finally washed with distilled water. Four samples were prepared during this stage.

ii 0.5 M : 0.5 M Mixture of two selected chemicals

The samples were treated with 1M each from the mixture of 0.5 M: 0.5 M from the following; KOH + HCl, HCl + NaCl, KOH + Ethanol and, HCl + Ethanol, respectively in a solution of 450 ml inside a shaker water bath maintained at 50 ºC for 4 hours. The treated samples were washed thoroughly with water and finally washed with distilled water. Four samples were prepared in this way.

iii. 0.75 M : 0.25 M Mixture of two selected chemicals

The samples were treated with 1M each from a mixture of 0.75 M: 0.25 M of the following; KOH + HCl, HCl + NaCl, KOH + Ethanol and, HCl + Ethanol, respectively in a solution of 450 ml inside a shaker water bath maintained at 50 ºC for 4 hours. The treated samples were washed thoroughly with water and finally washed with distilled water. Eight samples were prepared in this stage.

iv. Control sample

An untreated sisal fibre sample was used as control.

Different cellulose micro-fibrils were prepared using combinations of chemical and mechanical treatments. Sixteen different treatments were carried out on various samples, while some parts were left untreated and served as control samples.

2.2.3. Preparation of sisal fibre and compounding of composite materials

The sisal fibre preparation, determination of fibre diameter and compounding of the materials for the development of composites were carried out in accordance with Oladele *et al.* (2014).

2.2.4. Production of sisal fibre-reinforced polypropylene composites by a compression molding process

Homopolymer PP and sisal fibres were mixed together in predetermined proportions of 3-5 wt% for the development of the composites. To produce these composites, two moulds were used. A tensile test mould and a rectangular mould of dimension 150 x 100 x 3 mm

were used. The filled mould was placed inside a compression moulding machine maintained at 190 ºC and 15 tons for 15 minutes followed by air cooling. Teflon sheet and silicone were used as releasing agents. The unreinforced PP was also produced as neat material.

2.2.5. Testing and structural characterisation of cast samples

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

i. 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. Ten values were taken for each sample: the average is used as the representative value.

ii. Tensile test

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

iii. Impact test

Impact tests were carried out using a Charpy impact testing machine in accordance with ISO 179. Notched and unnotched impact tests were carried out on the samples. To carry out the tests, the samples were cut into the impact test dimension of 80 x 10 x 3 mm maintained at a distance of 60 mm between lines of supports. The notched samples were further prepared by notching them with the notching machine at the center of the samples. The test was carried out by placing the sample horizontally on the machine, taking the initial reading of the gauge and finally releasing a suspended handle that swings and fractures the sample. The final reading was taken after the sample fractured. For the notched samples, the samples were placed with the notched surface opposite to the swinging handle. Three test pieces were tested for each sample and the average value was taken as the representative value.

iv. Scanning electron microscope (SEM) examination

The fractured surfaces of the composites were examined using a JEOL SEM: JEOL JSM-5899 Scanning Microscope, JEOL, Tokyo Japan and a Zeiss SEM: Zeiss Ultra Plus 55 FECSEM, Zeiss, Oberkochen Germany respectively. Before the examination, the samples were prepared by cutting with a hacksaw followed by gluing on sample holder and finally coated with carbon using Carbon Coater: EMITECH K950X, EM Technologies, Kent England.

3. Results and Discussion

3.1. Hardness Properties of the Composites

Figure 3 shows the response of the composites, control and neat samples. From the results, it was observed that improved hardness properties were obtained for most of 5 wt% sisal-fibre reinforced composites compared to 3 wt%. The best results were obtained from the composite developed with sisal-fibre treated with 0.75 M HCl + 0.25 M KOH at 5 wt% sisal-fibre reinforcement which has a value of 75.7 HS, followed by sample from sisal-fibre treated with 0.75 M KOH + 0.25 M Ethanol and sample treated with 1 M HCl which has 75.5 HS and 75.0 HS, respectively.

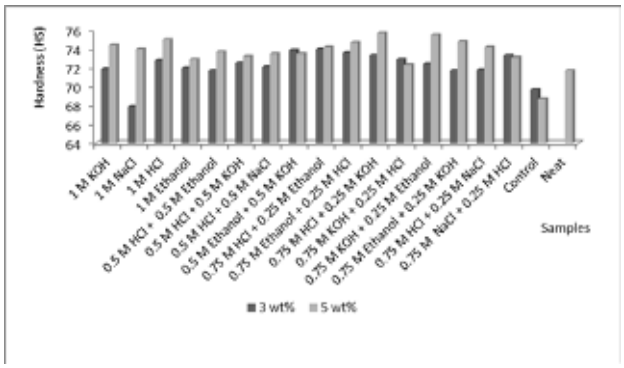

Figure 3. Hardness values of chemically treated and untreated sisal-fiber reinforced homopolymer polypropylene composites and the neat

It was also observed that hardness properties of most of the treated sisal fibre reinforced homopolymer PP composites were higher than neat material with a value of 71.7 HS whereas the reverse was the case for untreated sisal-fibre reinforced polypropylene composites with the best result of 69.7 HS from 3 wt% reinforcement. The observed results were due to the influence of chemical treatments on constituents of fibres. The exposed surfaces of sisal fibres after treatment allow proper binding of fibres and matrixes at their interface, thereby improving the hardness of the materials. Hardness of composites depends on distribution of fibre in the matrix (Premlal *et al.*, 2002; Jamil *et al.*, 2006). Usually, the presence of a more flexible matrix causes the resultant composites to exhibit lower hardness (Jamil *et al.*, 2006).

As shown in Figure 2, the incorporation of treated sisal-fibre into the PP matrix reduced the flexibility of

the matrix resulting in more rigid composites. The hardness of treated sisal-PP composites showed a slightly increasing trend with an increase in the fibre content. The treated sisal-PP composites seem to have much better hardness values than the untreated ones. This may be attributed to better dispersion of fibre in matrix with minimisation of voids and stronger interfacial adhesion between matrix and fibre.

3.2. Tensile Properties of the Composites

3.2.1. Young's modulus of elasticity

Figure 4 shows the plots of Young's Modulus of Elasticity for homopolymer polypropylene composites, control and neat. From the results, it was observed that 3 wt% of sisal-fibre reinforcement enhanced the Young's Modulus of Elasticity of the materials more than 5 wt% sisal- fibre reinforcement in most of the samples produced from treated fibres. The best result was obtained from 3 wt% sisal-fibre reinforced sample treated with 0.75 M Ethanol + 0.25 M HCl with a value of 837.38 MPa followed by sample treated with 1 M KOH with a value of 830.50 MPa. The sample treated with 0.75 M HCl + 0.25 M NaCl at 5 wt% sisal-fibre reinforcement was next with a value of 830.44 MPa. Comparing these with the best from the control at 3 wt% reinforcement and the neat material which have 747.21 and 710.64 MPa, respectively, it becomes obvious that chemical treatment enhances this property.

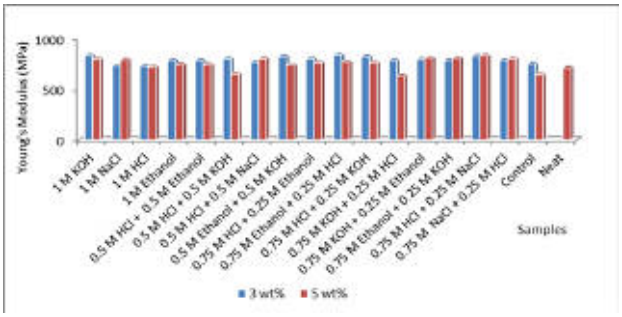

Figure 4. Young's modulus of elasticity for chemically treated and untreated sisal-fiber reinforced homopolymer polypropylene composites and the neat

The importance of natural fibre reinforced composites for polymeric materials comes from substantial improvement of the strength and modulus, this in turn improves the possibility of practical applications for composites. The addition of fibre is expected to increase the modulus of thermoplastic matrix composites (ASTM, 2002; Liu *et al.*, 2005). It is evident from the Figure that treated sisal-PP composites are found to show higher modulus compared to composites of untreated sisal fibre. Usually crystallites possess higher modulus compared to amorphous substances (Karmakar *et al.*, 2007). When sisal is treated with chemicals, crystallisation of the sisal surface probably dominates over its bulk nature, giving a higher modulus of treated sisal-PP composites. Furthermore, incorporation of fibre into the polymer matrix reduced the matrix mobility, resulting in stiffness of the composite.

3.2.2. Ultimate tensile strength (UTS)

The ultimate tensile strength is a measure of the maximum stress a material can withstand before it fails. Figure 5 shows variations of the ultimate tensile strengths for varous samples where it was observed that the strength of the materials was enhanced with 3 wt% of sisal fibre reinforcement than with 5 wt% in most of the samples produced from treatments. The best results were obtained with 3 wt% reinforcement with samples developed from sisal-fibre treated with 0.75 M HCl + 0.25 M KOH with a value of 34.53 MPa followed by unreinforced homopolymer polypropylene with a value of 33.88 MPa and a sample from sisal-fibre treated with 0.75 M HCl + 0.25 M NaCl with a value of 33.86 MPa.

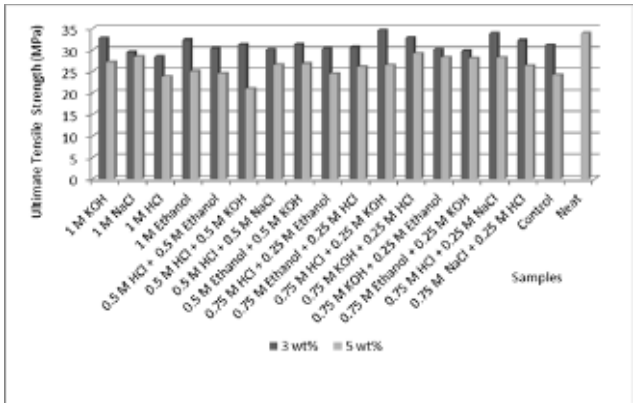

Figure 5. Ultimate tensile strength of chemically treated and untreated sisal fiber reinforced homopolymer polypropylene composites and the neat

The tensile strengths were found to decrease with increasing fibre loading. As fibre load increased, weak interfacial area between the fibre and matrix increased, this consequently decreased the tensile strength (Yang *et al.*, 2004; Lou *et al.*, 2007). An increase in fibre content increases the micro spaces between fibre and matrix, which weaken filler–matrix interfacial adhesion. As a result, the values of tensile strength show a decreasing trend with increasing fibre content in the composite. The presence of hydroxyl groups in cellulose of raw sisal is responsible for its inherent hydrophilic nature. As a result, it becomes difficult to compound hydrophilic sisal with hydrophobic PP, resulting in inefficient composites with weak interfacial bonding.

In order to improve mechanical properties of composites, sisal was chemically treated. Of the three

hydroxyl groups present in a cellulose anhydro glucose unit, one is primary hydroxyl group at C6, while the other two are secondary hydroxyl groups at C2 and C3 positions. Although the primary hydroxyl group is more reactive than the secondary ones, the chemical treatment breaks some of the OH groups thereby reducing the hydrophilic nature of the sisal. Due to the replacement of most of hydroxyl groups by compound groups upon chemical treatment of sisal, interfacial bonding between fibre and matrix increased in the resultant composites. This in turn enhanced the tensile properties of the developed composites compared to untreated sisal-PP composites as shown in Figures 4-5. The observed improvement may be attributed to the effect of chemical treatment on the interfacial bonding between the matrix and the fibre. This indicates the efficacy of the chemical treatment of sisal in improving the interfacial adhesion between sisal and PP leading to increased stress transfer efficiency from the matrix to the fibre with a consequent improvement in the mechanical properties of the composites.

3.3.3. Stress at 0.2% yield

Yield stress is defined as the stress at which a material will undergo an increase in strain at a constant stress. At this point, the microstructure of the material will be distorted, that is, there will be a change at microstructural level. Figure 6 shows the stress at 0.2 % yield for the composites and neat. The results show that stress at 0.2 % yield were enhanced at both weight fractions.

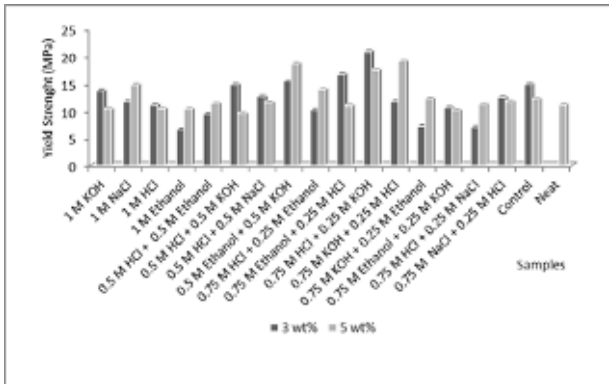

Figure 6. Yield strength (0.2 % proof stress) of chemically treated and untreated sisal fiber- reinforced homopolymer polypropylene composites and the neat

However, highest stress value was obtained at 3 wt% for a sample developed from sisal fibre treated with 0.75 M HCl + 0.25 M KOH with a value of 20.88 MPa followed by 5 wt% sisal fibre reinforced samples that are treated by 0.75 M KOH + 0.25 M HCl and 0.5 M Ethanol + 0.5 M KOH having 19.09 and 18.64 MPa respectively.

3.3.4. Strain at peak

Figure 7 shows the variation of strain at peak for homopolymer polypropylene composites and the neat. From the result, it was observed that 3 wt% sisal-fibre reinforcement enhanced strain at peak in almost all samples produced than that of 5 wt%. The best result was obtained from a 3 wt% sisal-fibre reinforced sample treated with 0.75 M HCl + 0.25 M NaCl which has a value of 16.11 mm/mm followed by the neat (unreinforced homopolymer polypropylene) with a value of 13.83 mm/mm and 3 wt% sisal fibre reinforced sample treated with 0.75 M HCl + 0.25 M Ethanol with a value of 12.38 mm/mm.

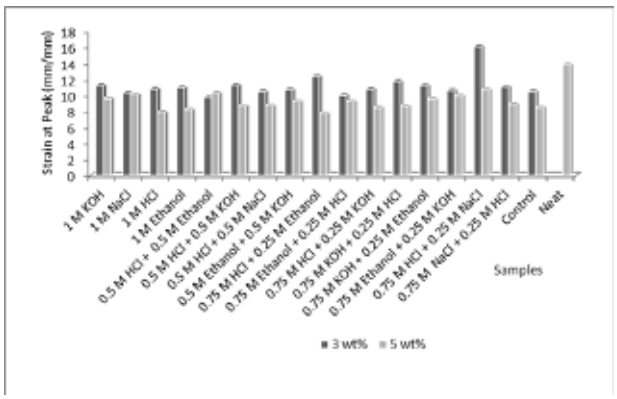

Figure 7. Strain at peak for sisal fiber-reinforced homopolymer polypropylene composites and the neat.

3.3.5. Impact properties of the composites

Variation of impact strengths for notched and unnotched homopolymer polypropylene matrix composites and the neat are shown in Figure 8. Samples were prepared in these forms so as to investigate the effect of sudden loading on the presence of fracture in case of notched samples while the unnotched accounts for the effect of sudden loading on thermoplastic materials where there is no fracture but there exists curve or bend. From the results, it was observed that both 3 wt% and 5 wt% sisal-fibre reinforcement enhanced the impact strength of the composites but were more enhanced with 3 wt%. The unnotched samples were noticed to possess better impact strength than the notched samples.

Considering notched samples, composites developed from 3 wt% sisal fibre reinforcement treated with 0.75 M HCl + 0.25 M KOH with a value of 0.016 J/mm^2 followed by 5 wt% sisal fibre reinforced sample treated with 0.5 M HCl + 0.5 M Ethanol having a value of 0.014 J/mm^2 gave better performance than the neat material. The neat follows with a value of 0.010 J/mm^2. This shows that in the as notched condition, reinforcement with chemically treated sisal fibre gave

promising results compared to the control and the neat materials.

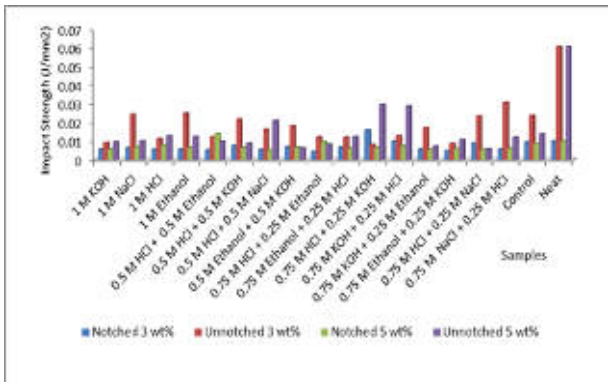

Figure 8. Notched and unnotched impact strength values for sisal fiber-reinforced homopolymer polypropylene composites and the neat

The neat material, in unnotched condition, had the best impact strength with a value of 0.061 J/mm^2 , followed by a composite developed from 3 wt% sisal-fibre reinforced sample treated with 0.75 M NaCl + 0.25 M HCl with values of 0.031 J/mm^2 , and then a 5 wt% sisal- fibre reinforced sample treated with 0.75 M HCl + 0.25 M KOH with a value of 0.029 J/mm^2.

The impact strength of a material provides information regarding the energy required to break a specimen of given dimensions; the magnitude of which reflects the ability of the material to resist a sudden impact. There is a diminishing effect of fibre on impact strength due to a drastic decrease in break elongation, because fibre bridges the crack and increases the resistance of crack propagation (Liu *et al*., 2005; Sanadi *et al*., 1997). 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 decreased impact strength of the composites.

The impact strength of the treated sisal-PP composites were found to be higher than those of the untreated ones, indicating that better interfacial bonding between the matrix and the fibre occurred upon chemical treatment. As a result, the chemically treated sisal-PP composites are capable of absorbing a higher amount of energy, stopping crack propagation compared to the untreated ones.

3.3.6. Surface morphology

Different approaches have been applied to change the fibre/matrix adhesive properties in natural fibre-reinforced composites: chemical or physical modifications of the matrix, fibre or both the components. Mohanty *et al.,* (2000) studied the effect of alkali treatment, cyanoethylation and grafting of jute fabrics in jute/biopol composites and found a 50 % enhancement in tensile strength and 30 % in bending strength compared to the untreated fibre-reinforced composites. Pothan *et al.,* (2002) examined the mechanical properties of various silane treated and mercerised banana fibre-reinforced polyester composites and concluded that alkali treated composites have better mechanical properties due to the better packing of the cellulose chains after dissolution of lignin, the cementing material.

Rout et al. (2001) studied the effect of alkali treatment on the performance of coir–polyester composites and found that as the concentration of sodium hydroxide increased, the mechanical properties decreased due to the cell wall thickening, which lead to poor adhesion with polyester resin. Guduri, (2006) and co-workers proved that an alkali treatment of the lignocellulosic natural fabric Hildegardia Populifolia is a good method to improve the fibre/matrix interaction. Various physical methods such as corona treatment (Belgacem et al., 1994), plasma treatment (Felix et al., 1994) and heat treatment (Sapieha et al., 1989) have been reported to affect the compatibility in natural fibre composites; in most cases positively.

Using the SEM Morphology of the fractured surfaces of sisal fibre/homopolymer PP composites, it was observed that proper wetting of the sisal fibres occur in the developed composites, which was likely to be one of the reasons for the improved mechanical properties. Figure 9 shows the SEM image of untreated sisal fibre, while Figure 10 show sisal fibre with 0.75 M HCl + 0.25 M KOH treated.

Wetting of the fibre by the matrix is what aids proper binding between the fibre and the matrix. And, this is responsible for the transfer of load from the matrix to the fibre, which is a critical factor/issue in the production of composite materials. The morphology of the fracture surface shows the face information reflecting the reasons why the mechanical properties of the composites fabricated under different conditions are different. The solid white strand parts represent the fibre while the dark parts represent the matrix. Also seen from the surfaces are voids caused by trapped gases during compaction. These voids account for the porosity of some materials during production. Too much porosity in most cases adversely affects the mechanical properties of the materials as shown in both figures.

The SEM images of the untreated sisal-PP composites show a number of pullout traces of fibre with smooth surfaces and micro-voids as well as agglomeration of the fibre in the PP matrix as shown in Figure 9. These features suggest weak interfacial bonding between the fibre and the matrix. On the other hand, chemically treated sisal-PP composites show better dispersion of the fibre into the matrix, which

results in better interfacial adhesion between the fibre and the matrix.

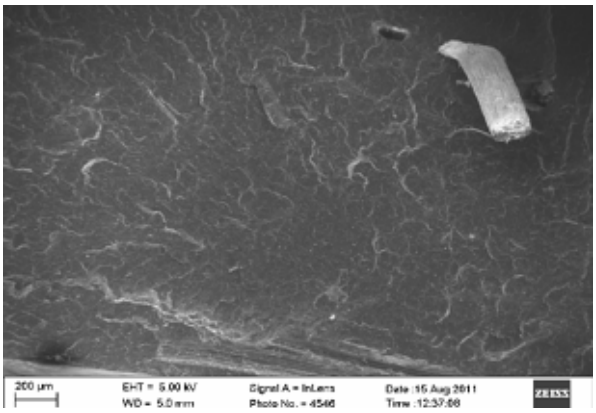

Figure 9. SEM image of untreated sisal fibre

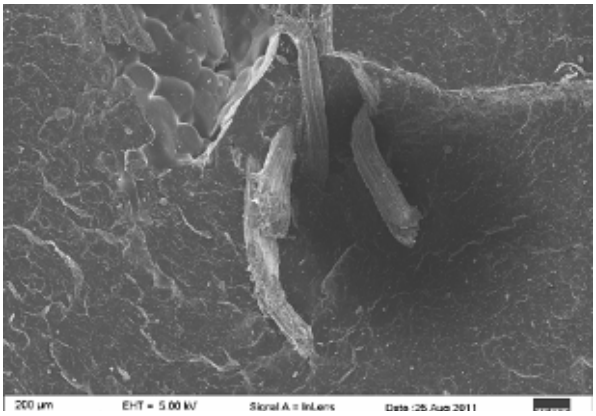

Figure 10. SEM image of sisal fibre with 0.75 M HCl + 0.25 M KOH treated

As clearly seen in the micrograph in Figure 10, both fibre pull-out traces and the agglomeration of sisal in the matrix have substantially reduced in the treated sisal-PP composites, suggesting that interfacial bonding between the treated fibre and the matrix is much more favorable compared to that of the untreated one. The outcome of the better interfacial bonding between the fibre and the matrix is reflected in the improvement of the mechanical properties of the treated sisal-PP composites.

4. Conclusion

Several deductions can be made from the research. Firstly, in all samples where sisal-fibre reinforced homopolymer PP composites perform better than the unreinforced homopolymer PP or compete favourably, chemically treated samples happened to be the best compared to the untreated samples. Treated sisal-fibre reinforced homopolymer PP composites had the best hardness, tensile strength and impact strength in the as notched condition properties than both untreated sisal-fibre reinforced homopolymer PP composites and unreinforced homopolymer PP. This may be attributed to better dispersion of the fibre into the matrix with minimisation of voids and stronger interfacial adhesion between the matrix and the treated fibres. The improved mechanical properties of the treated sisal-fibre reinforced homopolymer PP composites are further supported by SEM images of the fracture surface that show better matrix/fibre interaction compared to those prepared from untreated sisal-fibre.

Secondly, low weight fraction (fibre content) gave the best properties except in hardness. Tensile and impact strengths are found to decrease with increasing fibre loading. As the fibre load increased, the weak interfacial area between the fibre and the matrix increased, this consequently decreased these strengths. An increase in the fibre content increases the micro spaces between the fibre and the matrix, which weaken the fibre/matrix interfacial adhesion and allow moisture absorption. As a result, the values of these strengths show a decreasing trend with increasing fibre content in sisal-fibre reinforced homopolymer PP composites. Conversely, the presence of a more flexible matrix causes the resultant composites to exhibit lower hardness.

Considering the mixing ratios studied, 0.75 M: 0.25 M has higher synergistic effect than that of others. The treatment that has the highest best performance was 0.75 M HCl + 0.25 M KOH. The treatment gave the best result obtained in treated sisal-homopolymer PP composites for hardness, tensile strength and impact strength in the as notched condition properties.

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