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Matieral Properties

# Kapok/cotton fabric–polypropylene composites

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**Abstract**

Kapok/cotton fabric has been used as reinforcement for conventional polypropylene and maleic anhydride grafted polypropylene resins. Treating the reinforcement with acetic anhydride and sodium hydroxide has modified the fabric (fibres). Thermal and mechanical properties of the composites were investigated. Results show that fibre modification gives a significant improvement to the thermal properties of the plant fibres, whereas tests on the mechanical properties of the composites showed poor tensile strength. Mercerisation and weathering were found to impart toughness to the materials, with acetylation showing slightly less rigidity compared to other treatments on either the fibre or composites. The modified polypropylene improved the tensile modulus and had the least toughness of the kapok/cotton reinforced composites. MAiPP reinforced with the plant fibres gave better flexural strength and the same flexural modulus at lower fibre content compared with glass fibre reinforced MAiPP. © 2000 Elsevier Science Ltd. All rights reserved.

## 1. Introduction

The development of a blend of kapok-cotton fabric and its application in thermoset composite manufacture has introduced a new class of cellulose based reinforcement material [1,2]. However, the research and development trend of fibre reinforced composite favours the application of thermoplastic binder materials due to environmental problems associated with thermosets.

Lack of good interfacial adhesion with the polymer phase, due to the inherently poor compatibility and the ability of the hydrophilic cellulose fibres to disperse with the hydrophobic resins, makes the use of cellulose based fibre reinforced composites less attractive. The presence of free water and hydroxyl groups, especially in the amorphous regions, worsens the ability of plant

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fibres to develop adhesive characteristics with most binder materials. High water and moisture absorption of the cellulose fibre causes swelling and a plasticising effect resulting in dimensional instability and poor mechanical properties. Plant fibres are also prone to micro-biological attack leading to weak fibres and reduction in their life span. However, these drawbacks can be overcome by treating the cellulose fibres with appropriate chemicals. For instance, varying concentrations of caustic soda have been applied to cellulose materials for surface and fine structure modifications (Owolabi et al. [3], Sreekala et al. [4], Geethamma et al. [5], and Murkejee [6]).

Cellulose based fibres absorb moisture causing reversible and non reversible swelling. In composite products this can result in undesirable dimensional changes. To arrest this problem, the reinforcing cellulose fibres are subjected to certain modifications. These processes involve either the stabilisation of the cell wall matrix to restrain swelling, reduction of the hygroscopic tendency of the cell wall and bulking the cell wall polymers to maintain the wet volume so that moisture does not cause any additional swelling [7]. Formaldehyde has been extensively applied to create dimensional stability in cellulose materials, whereby it reacts with the cell wall hydroxyl groups bonding together micro-fibril units. The reaction takes place under strong acid catalyst [8].

On the other hand, matrix substrates, which play an important role in filled polymer systems, have similarly been a subject of intensive research studies. These substances when used in their virgin state have been found to produce poor interface with cellulose fibres resulting in poor mechanical properties [9,10]. In order to improve the bonding characteristics, coupling agents are sometimes added and/or grafted onto the polymer backbone [10,11]. The grafted chemical groups can then react with the hydroxyl groups present on cellulose fibres. For instance, maleic anhydride compound is easily grafted onto isotactic polypropylene to obtain a maleic anhydride isotactic polypropylene (MAiPP). This modified polypropylene has been found to have good binding characteristics with cellulose filler materials developing into composites with improved mechanical properties [10]. The matrix is also reported to be more hydrophobic than the conventional isotactic polypropylene [12].

In this article we report on the reinforcement properties of kapok/cotton fabric, using isotactic polypropylene and maleic anhydride grafted polypropylene as the matrices. Bending characteristics of the plant fibre–MAiPP have been compared with that of non-woven glass mat–MAiPP composite. The effect of surface and molecular modification of kapok/cotton fabric by alkali treatment and acetylation on the performance of the composites has been studied. The composites have also been subjected to accelerated weathering to simulate ageing and the mechanical properties determined. Morphological analysis of the fibre–matrix interface and fibre pullout of the composites have been studied by use of a scanning electron microscope (SEM). Fourier transform infrared (FT-IR) has been used to observe the acetyl groups grafted. Thermogravimetry and differential scanning calorimetry have been used to investigate the thermal characteristics of fibres and the composites. Tensile and flexural properties have been evaluated. The toughness of the composites has been determined by evaluating the area under the load–elongation curves.

## 2. Experimental procedure

### *2.1. Materials and methods*

A plain weave kapok/cotton fabric was used together with non-woven glass mat. All the chemicals used including the isotactic polypropylene and MAiPP resins were obtained from the Institute

for Research and Technology on Plastic Materials (IRTeMP) laboratories. A mould measuring 120×120×3 mm length, width and thickness respectively was used to prepare composites for tensile testing, while specimens for flexural properties were produced by using a 65×65×3 mm mould. A 400-mm$^2$ hand operated, electrically heated hydraulic press was used for compression moulding.

### 2.2. Alkali treatment

The fabric was first de-waxed by soaking it in light petroleum at 40°C and was constantly hand agitated for 4 h, thoroughly washed with distilled water and dried. It was then dipped in 2% sodium hydroxide solution for about 48 h and washed with distilled water containing a few drops of acetic acid to neutralise free sodium hydroxide. Finally the fabric was washed again with fresh distilled water and dried.

### 2.3. Acetylation

The fabric was soaked in acetic anhydride for about 1 h at a temperature of 70°C. The fabric was then washed with distilled water and dried.

### 2.4. Fourier transform-infrared spectroscopy

The infrared spectra were obtained using a Perkin–Elmer FT-IR Spectrometer PARAGON 500. About 2 mg of fibres ravelled from kapok/cotton fabric were crushed into powder form in liquid nitrogen. The powdered material was then mixed with KBr and pressed into a small disc 1 mm thick.

### 2.5. Composite manufacture

Fabric pieces were weighed and hand laid alternately with the resin into the mould which was then placed between the platens, electrically heated to a temperature of 190°C. The mould was heated for 6 min without applying any pressure to melt the matrix. It was further heated for another 4 min at a pressure of 10.13 MPa and then slowly cooled to room temperature before releasing the composite.

### 2.6. Weathering

Artificial weathering apparatus was not available during the time of conducting the research. ASTM D, 570-81, which mentions test procedures for water absorption by immersion in boiling water for 2 h, was adopted as a procedure for estimating accelerated weathering.

### 2.7. Thermogravimetric analysis

The loss in mass as a function of temperature of the materials was determined by using a Mettler TG 50 equipped with TA Mettler processor TC 11. Samples weighing between 5 and 10

mg were placed in ceramic crucibles in a furnace and heated in nitrogen between 40 and 800°C at a rate of 10°C/min.

### 2.8. Differential scanning calorimetry

A Mettler DSC 30 with low temperature cell and equipped with a TA controller Mettler TC A/TC 15 was used to investigate the thermal behaviour of the materials. Samples weighing between 5 and 10 mg to ensure sufficient sensitivity for heat capacity measurement were placed in an aluminium capsule. They were then heated in a furnace in nitrogen flowing at a rate of 20 ml/min.

### 2.9. Tensile properties

The tensile testing of rectangular laminated composites was carried out according to ASTM D 3039-82 using an Instron tensile testing machine Model 4505 with an installed computer programme. The machine was run at a cross-head speed of 5 mm/min.

### 2.10. Flexural tests

A three point bending method in accordance with ASTM D 790 Method 1 - Procedure A was applied to determine the flexural properties of the composites. The strain rate was maintained at 1 mm/min, while the span to depth ratio was not more than 16.

### 2.11. Scanning electron microscope examination

A Philips SEM 501 scanning electron microscope was used to study the fibres as well as the fractured surfaces of the composite samples. Prior to the analysis the samples were coated with Au/Pd alloy by means of a Polaron Sputtering apparatus.

## 3. Results and discussion

### 3.1. Alkali treatment

Sodium hydroxide treatment of cellulose fibres leads to the irreversible mercerisation effect, which increases the amount of amorphous cellulose at the expense of crystalline cellulose. The cellulose–sodium hydroxide reaction is thought to be as follows.

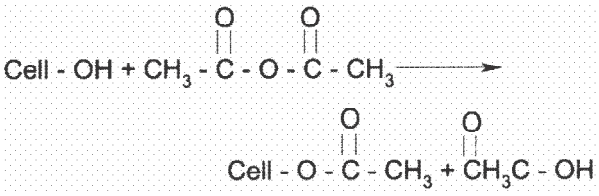
$$\text{Cell}-\text{OH}+\text{NaOH} \rightarrow \text{Cell}-\text{O}^{-}\text{Na}^{+}+\text{H}_2\text{O}+[\text{surface impurities}]$$

Mercerisation improves adhesion characteristics by removing surface impurities, thus exposing the micro-fibrils, which then render the fibre topography with a rough texture. The rough and cleaned surface facilitates mechanical adhesion in addition to improved wetting ability of the resin.

The composites produced from fibres treated with sodium hydroxide are, in principle, expected to have improved mechanical properties but only if there is no increase in the amorphous cellulose.

### 3.2. Acetylation

Acetic acid does not sufficiently react with cellulose so acetic anhydride was applied instead. However, because acetic anhydride is not a good swelling agent for cellulose, in order to accelerate the reaction, cellulose materials are made more accessible at higher temperatures. The rate of reaction is much faster with fibres that have not been mercerised than with mercerised cellulose fibres. This is probably because in mercerised cellulose the hydroxyl groups have already been replaced with the alkali groups. The reaction in this work was carried out in liquid phase and is represented as follows.




Acetylation of the hydroxyl groups swells the plant fibre cell wall, greatly reducing the hygroscopic nature of the cellulose fibres. This consequently results in dimensional stability of the composites, as any absorbed water will not cause further swelling nor shrinkage on the composite material [7]. The presence of the acetyl groups has been confirmed by infrared analysis which shows a decrease in absorbency at 3430 $cm^{-1}$ of the acetylated fabric indicating a decrease in H-bonded hydroxyl groups stretching due to the reactions with acetic anhydride. The increase in intensity at 1735-1740 $cm^{-1}$ is clear evidence of the presence of acetyl groups grafted on the cellulose molecular structure. Quantitative analysis was not carried out to determine the actual amount of acetyl groups. Acetylated fibres also show decreased intensity of C-H stretching and bending at 2900 $cm^{-1}$ and 1384 $cm^{-1}$ respectively (Fig. 1).

### 3.3. Thermal analysis

Fig. 2 shows the thermal degradation pattern of iPP matrix reinforced with untreated, acetylated and alkali-treated kapok/cotton fabric together with that of weathered composites. It also gives thermal characteristics of kapok/cotton fabric–MAiPP composites. Kapok/cotton–MAiPP and weathered kapok/cotton–iPP composites show similar rates of loss in mass up to about 360°C. Other composites also show a similar loss in mass up to 260°C. The loss in mass below these temperatures may be attributed to the presence of low molecular weight chemical groups and/or impurities, which may have been trapped in the composites during the moulding process. The weathered kapok/cotton–iPP composite shows the highest degradation temperature followed by kapok/cotton–MAiPP composite. Dehydration temperature under TG is below 100°C as seen in Table 1.

Acetylated kapok/cotton fabric–iPP composites have a higher onset temperature of degradation than the weathered composite and mercerised kapok/cotton fabric–iPP composite, but lower than

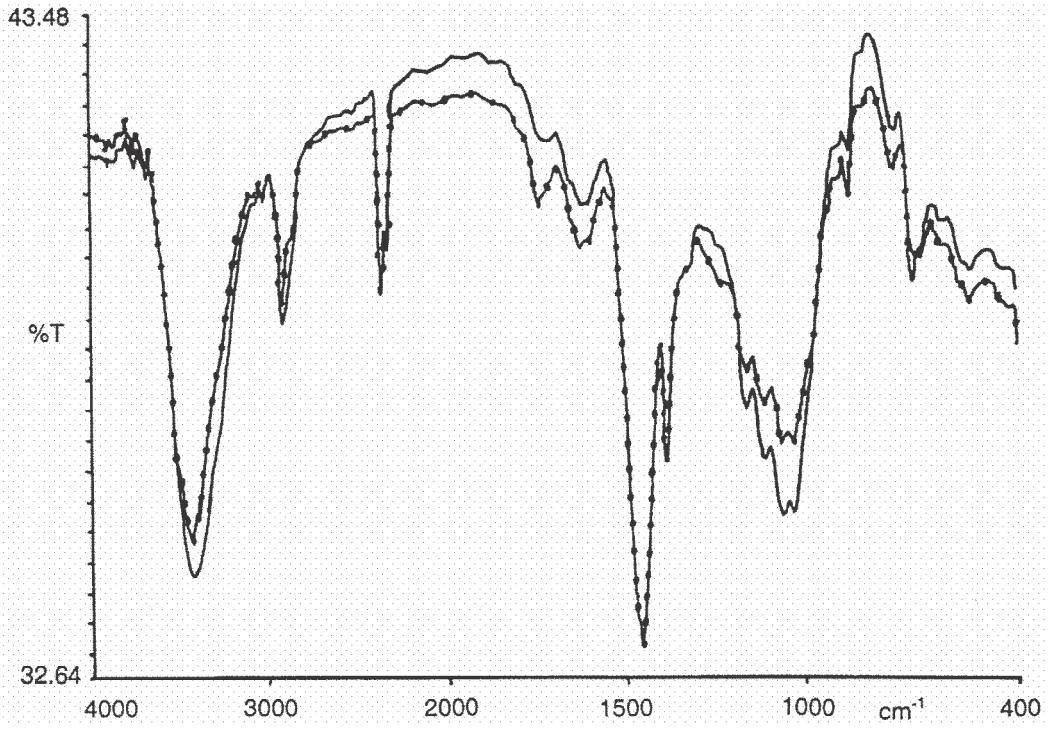



Fig. 1. Infrared spectrograph of (a) untreated kapok/cotton fabric (———) and (b) acetylated kapok/cotton fabric (-●-).

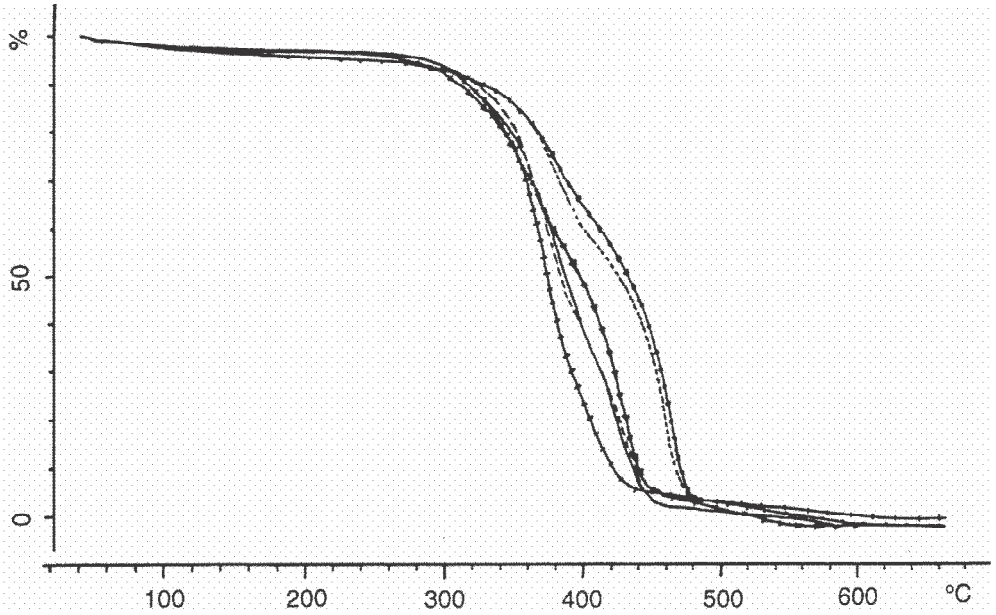



Fig. 2. Thermograms (TG) of composites. (a) (▶) Kapok/cotton–iPP composite; (b) (-+-) mercerised kapok/cotton–iPP composite; (c) (– – –) acetylated kapok/cotton–iPP composite; (d) (-●-) weathered kapok/cotton–iPP composite; (e) (- - -) kapok/cotton–maleic anhydride iPP composite; (f) (———) weathered kapok/cotton–maleic anhydride iPP composite.

Table 1
Thermogravimeter (TGA) analysis of composites

| Properties | Alkali-treated kapok/cotton–iPP composite | Acetylated kapok/cotton–iPP composite | Unweathered kapok/cotton–iPP composite |
|---|---|---|---|
| Dehydration temperature (°C) | 74.1 | 59.3 | 76.3 |
| Onset temperature (°C) | 332.3 | 339.5 | 316.4 |
| Degradation temperature 1 (°C) | 372.8 | 372.63 | 370.4 |
| Degradation temperature 2 (°C) | — | 431.7 | 428.2 |
| Decomposition temperature (°C) | 656.7 | 655.3 | 656.4 |
| Properties | Unweathered kapok/cotton–MAiPP composite | Weathered kapok/cotton–iPP composite | Weathered kapok/cotton–MAiPP composite |
| Dehydration temperature (°C) | 77.5 | 71.8 | 71.8 |
| Onset temperature (°C) | 339.5 | 318.5 | 339.2 |
| Degradation temperature 1 (°C) | 381.9 | 380.6 | 384.2 |
| Degradation temperature 2 (°C) | 459.1 | 423.7 | 462.5 |
| Decomposition temperature (°C) | 657.9 | 658.7 | 655 |

MAiPP composites reinforced with kapok/cotton fabric. The maleic anhydride is likely to be the possible source for the improved thermal properties of polypropylene as also evidenced by the rise in the degradation temperature of the composites. These findings concur with the general concept regarding the effect of grafting on the thermal properties of polymeric materials and the resulting composites. Kessira et al. [13] also report similar results in their study on the thermal behaviour of bagasse and wood pulps at a weight loss of below 18% of cellulose.

Mercerisation of fibres, which is known to result in reduced crystalline cellulose, has been found to decrease the thermal properties of the kapok/cotton fabric–iPP composites. This is because amorphous cellulose contains hemicellulose molecules, which have low thermal resistance. It has also been found, with the exception of mercerised kapok/cotton fabric–iPP, that all composites subjected to TG analysis exhibit two degradation temperatures (Table 1). It is possible that the first degradation temperature is due to the depolymerisation of the cellulose materials. The breakdown of the matrices into monomers and/or the decomposition of the levoglucosan cause the second degradation temperature, for which Peters et al. [14] report that its magnitude is dependent on the quantity of the crystalline cellulose.

Fig. 3 shows the DSC thermograms of the composites obtained in the first run. Both fibre and composite treatment appear to have a significant effect on the segmental movement during the elastic and plastic transitions. The endotherms appearing below 100°C are mainly due to water desorption and their intensity is indicative of the amount of water present in the composite. The endotherm due to the glass transition temperature of the composites appears to have been overshadowed by the endotherm caused by the removal of water or moisture content.

Table 2 shows that treatment on the fabric and the composites increases the glass transition temperature of the composites, with weathered MAiPP based composites giving the highest value. The melting temperature obtained by the DSC method (Table 2) does not seem to differ signifi-

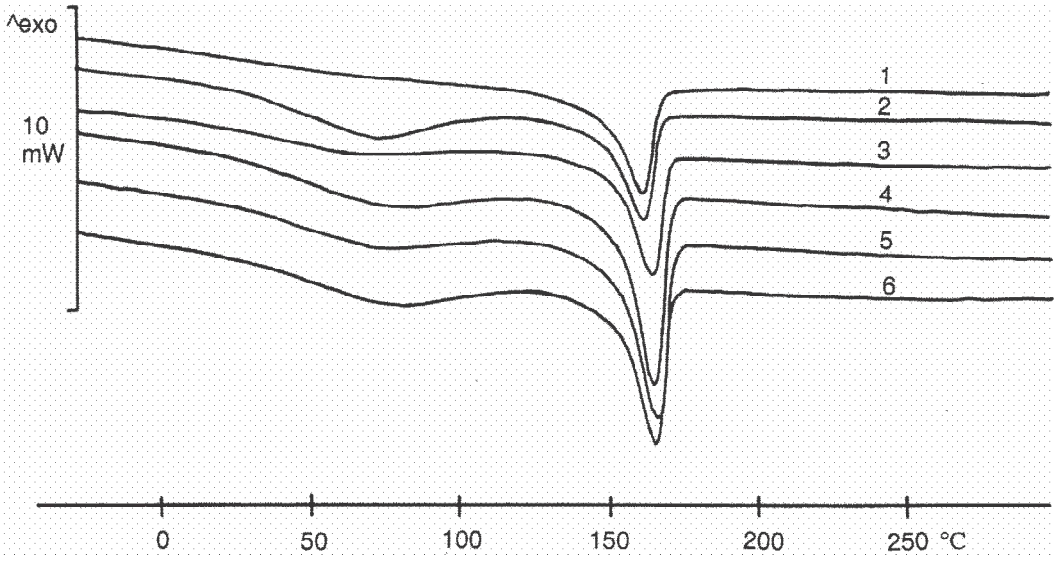



Fig. 3. DSC thermograms of (1) untreated kapok/cotton–MAiPP; (2) weathered kapok/cotton–MAiPP; (3) untreated kapok/cotton–iPP; (4) mercerised kapok/cotton–iPP; (5) acetylated kapok/cotton–iPP; and (6) weathered kapok/cotton–iPP composites.

Table 2
Differential scanning calorimeter (DSC) analysis

| Properties | Alkali-treated kapok/cotton–iPP composite | Acetylated kapok/cotton–iPP composite | Unweathered kapok/cotton–iPP composite |
|---|---|---|---|
| Glass transition temperature (°C) | 41.9 | 40.9 | 37.9 |
| Dehydration temperature (°C) | 95.1 | 93.3 | 81.8 |
| Melting temperature (°C) | 164.2 | 163.7 | 164.4 |
| Crystallisation temperature (°C) | 115.7 | 114.5 | 112.8 |
| Decomposition temperature 1 (°C) | 352.5 | 351.24 | — |
| Decomposition temperature 2 (°C) | 464.4 | 464.5 | 463.9 |

| Properties | Unweathered kapok/cotton–MAiPP composite | Weathered kapok/cotton–iPP composite | Weathered kapok/cotton–MAiPP composite |
|---|---|---|---|
| Glass transition temperature (°C) | 46.0 | 44.8 | 52.4 |
| Dehydration temperature (°C) | 95.9 | 93.0 | 73.0 |
| Melting temperature (°C) | 161.7 | 164.9 | 161.4 |
| Crystallisation temperature (°C) | 116.94 | 114.2 | 117.3 |
| Decomposition temperature 1 (°C) | 363.06 | 362.4 | — |
| Decomposition temperature 2 (°C) | 467.4 | 466.5 | 467.5 |

cantly. However, MAiPP composites show slightly lower melting temperatures compared with the iPP based composites. Fabric treatment lowers the melting temperature of the iPP composites while composite treatment gives a slight increase in its melting temperature. Crystallisation temperature is lowest in unweathered iPP based composite and is highest in the weathered MAiPP based composite. The first and second decomposition temperature represent the responses of the cellulose in the composite to the heat; whereas the former is an indication of crystalline regions the latter may be indicating traces of lignin and/or other monomeric compounds produced by the degrading cellulose.

### *3.4. Mechanical properties*

#### *3.4.1. Stress–strain*

Fig. 4 shows stress–strain curves of selected test specimens from the composite samples. They are not curves obtained by averaging groups of test specimens. The MAiPP reinforced with untreated fibres have less strain at break compared with composites with treated fibres and weathered composites. This indicates the characteristic brittleness of the MAiPP. This is also seen on the SEM micrographs (Fig. 10b) where MAiPP shows cracks on the fractured surface. Untreated kapok/cotton fabric–iPP shows less stress resistance compared with composites with mercerised kapok/cotton fabric. This indicates that mercerisation forms a ductile interface, together with good fibre–matrix interfacial adhesion. Weathered kapok/cotton fabric–iPP composite reduces the interfacial bond strength thus producing composites with low stress resistance.

#### *3.4.2. Tensile strength*

The tensile strength of iPP matrix reinforced with untreated kapok/cotton fabric is higher than all other composites tested, followed by composites reinforced with alkali treated fabric, then acetylated fabric kapok/cotton–iPP composites. Caustic soda is well known to reduce the crystal-

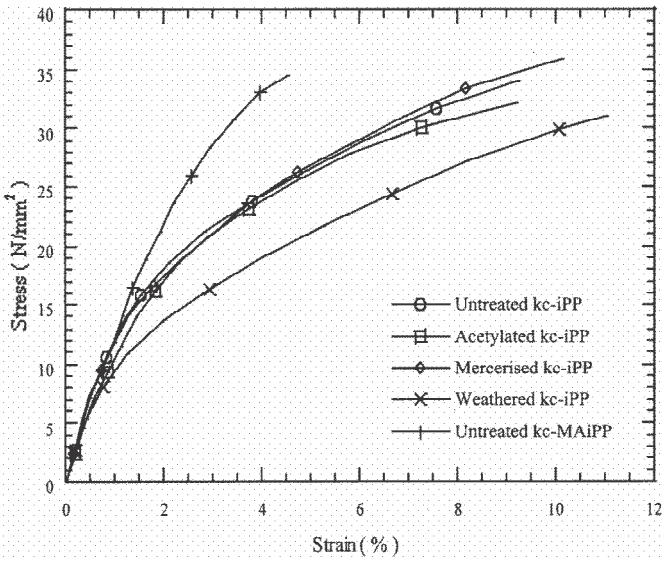



Fig. 4. Stress–strain curves of composites.

line cellulose hence the resulting low tensile strength of the composites reinforced with alkali treated fabric. Earlier work by the author showed a similar effect of alkalisation whereby increased caustic soda concentration resulted in reduced tensile strength of the kapok/cotton–polyester composites [2]. Lower tensile properties of the acetylated fabric composites implies also that acetylation lowers the crystalline content of the cellulose. However, at around 23% the weathered composite has a tensile strength second to the untreated kapok/cotton–iPP composites (Fig. 5). Kapok/cotton–MAiPP composites have the lowest tensile strength compared to other composites and this is due to the brittle characteristics of the MAiPP matrix causing poor load transmission.

### *3.4.3. Stiffness*

The stiffness or modulus of the untreated iPP composites increases with an increase in fibre volume fraction but drops slightly beyond about 23% fibre volume fraction. On the other hand, the addition of the same amount of fibre causes a significant increase of the initial tensile modulus of the MAiPP reinforced with untreated kapok/cotton fabric. It also has the highest stiffness properties at higher fibre content (Fig. 6). This may be attributed primarily to the enhanced interfacial adhesion resulting from the presence of a matrix with increased polarity that may react or interact favourably with hydroxyl groups on the fibre surface. Results of the treated fibre reinforced iPP composite show that mercerised fibre improves the elastic modulus better than acetylated fibre composites. Weathered iPP composites have the least elastic modulus except at around 23% where it is slightly higher than that of the acetylated kapok/cotton fabric–iPP composites.

### *3.4.4. Flexural properties*

The flexural properties of iPP and MAiPP–kapok/cotton fabric composites are shown in Figs. 7 and 8 together with glass fibre–MAiPP composites. The increase in the cellulose reinforcements on the iPP and MAiPP based composites show a similar trend of the increases in their flexural strength and modulus. The maleated polypropylene fibre reinforced composite gives superior flexural properties compared with the conventional polypropylene composites. The glass fibre reinforced composites exhibit lower flexural strength at 18% fibre volume fraction, but higher

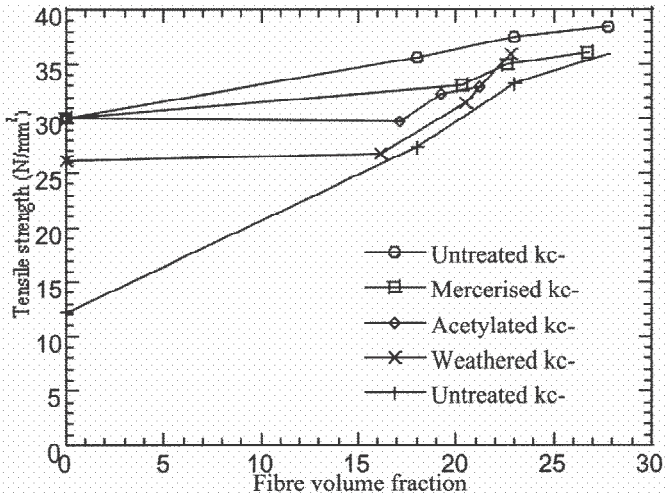



Fig. 5. Tensile strength of the composites.

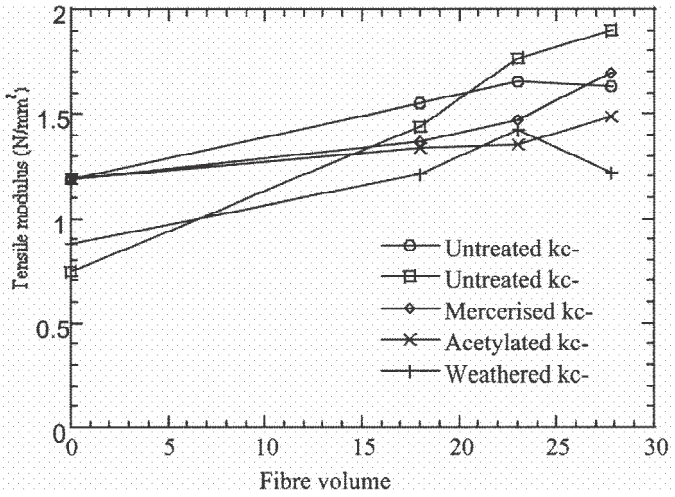



Fig. 6. Tensile modulus of the composites.

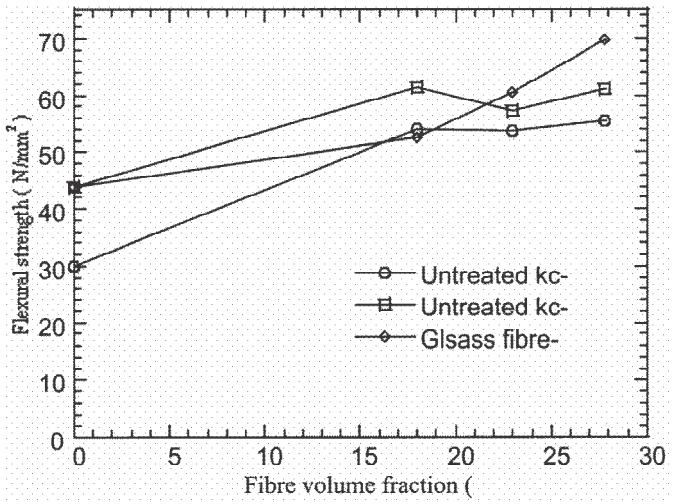



Fig. 7. Flexural strength.

values at increased fibre volume fraction than the conventional and maleic grafted polypropylene composites. The flexural modulus of glass fibre–MAiPP was found to be superior compared to those of the cellulose based composites especially above 18% fibre volume fractions.

### *3.4.5. Toughness*

Fig. 9 shows the work done or toughness of the untreated and treated fibre reinforced iPP and MAiPP composites measured by the area under the stress-strain curves up to breaking point. MAiPP–kapok/cotton reinforced composite shows an increasing trend of toughness with an increase in fibre volume fraction. The overall toughness is, however, much lower than that of the fibre reinforced iPP composite. This is mostly due to the brittle characteristics of the MAiPP matrix. The slight increase in toughness of acetylated fabric composite can be attributed to the plasticisation effect of the acetylation process on the fibres. The decrease at higher fibre volume

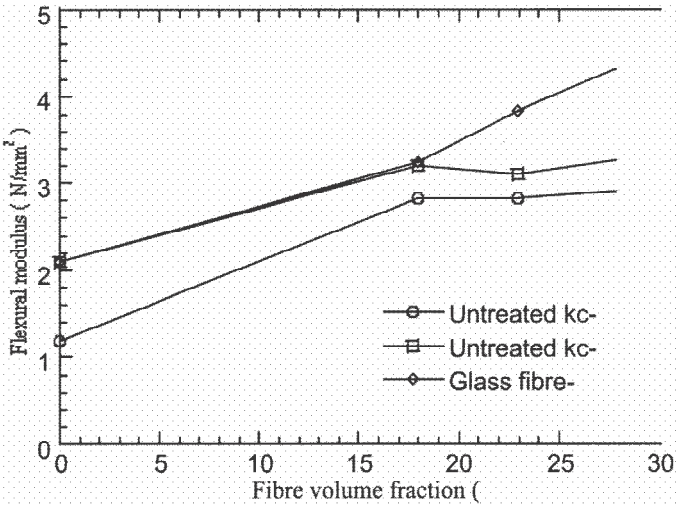



Fig. 8. Flexural modulus.

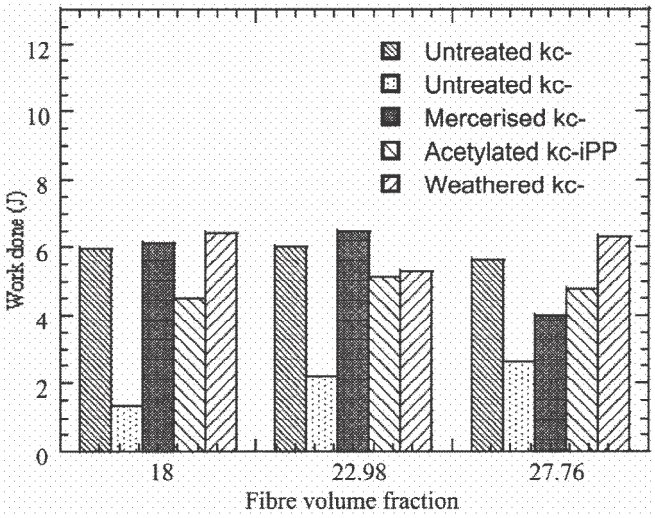
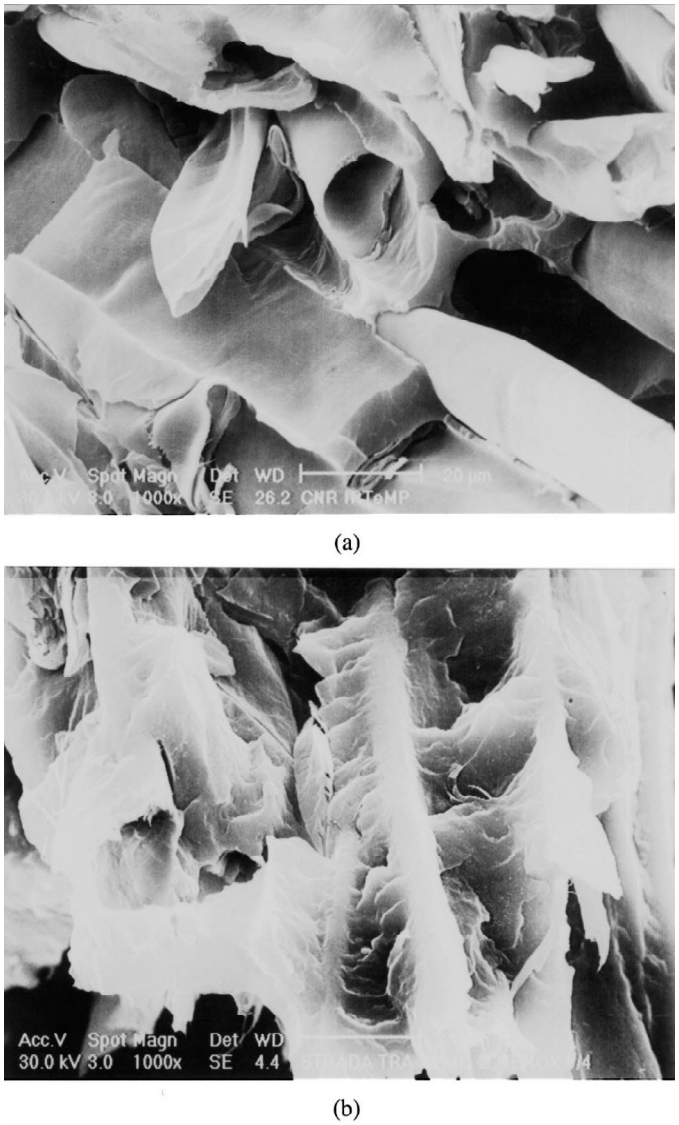



Fig. 9. Work done to break (toughness) the composites on tensile loading.

fraction could be due to the increase in fibre content and hence voids rather than being an effect of acetylation treatment. Weathering seems to show no significant effect on the composite toughness between 18% and 28% fibre volume fraction indicating that the small deviations at 22.98% fibre volume fraction could have resulted from either testing error or material preparation. However, the increase in toughness of mercerised fibre and weathering composites compared to acetylated fibre composites is a strong indication of an increase in plasticisation and hence ductility of the fibre and the composites.

### 3.5. Morphology

Fig. 10 gives information about the adhesive properties of the unmodified and maleic grafted polypropylene respectively. It was observed that MAiPP has a good interface with the cellulose




(a)

(b)

Fig. 10. SEM micrographs of tensile fracture surface of (a) kapok/cotton fabric–iPP composite and (b) kapok/cotton fabric–MAiPP composite.

fibres compared with conventional iPP and it also appears to have a much better interaction with cotton fibre than with kapok fibres. However, MAiPP exhibits a brittle characteristic as shown in Fig. 10b, where cracks are clearly seen in the matrix phase.

## 4. Conclusion

Alkalisation and acetylation of the kapok/cotton fabric results in low tensile strength of iPP composites compared to the untreated fabric composite. Similar effects of caustic soda treatment on the same reinforcement material on the polyester matrix have been reported by Mwaikambo and Bisanda [2]. It is believed that caustic soda and acetylation reduces the amount of crystalline

cellulose, which is largely responsible for the mechanical properties of plant fibres. The 2% caustic soda treatment on the cellulose fibres lowers the energy on tensile loading at higher fibre content while acetyaltion seems to favour applications where the polypropylene composites are supposed to exhibit stable energy absorption with the advantage of using more fibre than the matrix. Acetylation would also be more favourable in environments where the thermoplastic composites are subjected to high thermal applications, than would be composites with alkali treated plant fibres.

The incorporation of maleic anhydride on the polypropylene molecule improves the fibre–matrix interfacial adhesion and composite stiffness as observed by SEM (Fig. 10) and the increase in the tensile modulus at higher fibre content shown in Fig. 6. A comparison between the glass reinforced-MAiPP composites and the polypropylene composite shows that glass fibre gives higher flexural properties on the MAiPP matrix than kapok/cotton fabric, especially at 23% fibre volume fraction (Figs. 7 and 8). The results obtained in this work indicate the potential of using plant fibres for reinforcement with thermoplastic matrices for end use that would not require extreme mechanical properties.

## Acknowledgements

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