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# *Some Properties of Hemp Fibre and Hemp Fibre Filled Natural Rubber Composites*

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*Hemp fibre filled rubber were prepared using industrial hemp fibre mats with rubber latex. Three types of fibres, namely, untreated, alkaline treated and digested fibres were used in the present study. Prior to the preparation of the fibre filled rubber, thermal and surface morphological analyses indicated certain changes in the treated fibres in contrast to untreated fibres. Fibres treated with a concentrated alkaline solution at high temperature demonstrated a higher level of surface roughness and resistance to thermal degradation than untreated fibres. The higher level of surface roughness observed in the treated fibres was also observed in the resulting properties of the fibre filled rubber. The fibre filled rubber exhibited poorer strength than the rubber control which can be attributed to inappropriate adherence between the rubber and fibres.*

**Keywords:** hemp fibre; natural rubber composites; fibre filled rubber; thermal; surface morphology

The use of natural fibres for making products ranging from textile to paper has existed for a long time. Recently, there has been renewed interest in utilising natural fibres for industrial, automotive and many other related applications, in particular to reduce the dependence on petrochemical derived materials and to utilise more sustainable and renewable resources.

Natural fibres such as industrial hemp fibres (*Cannabis Sativa L.*) have long been valued for their strength and used extensively in the fabrication of thermoplastics composites[1–3], paper[4] and textiles[4–7]. Nonetheless, utilisation of hemp fibres and rubber to make suitable flexible materials is rarely described.

Incorporation of natural fillers such as plant fibres in rubbers is not a novel concept. It has attracted considerable attention and much research in this area has been done[8–13]. Among the advantages anticipated for fibre filled rubber composites[14] were improvements in thermo-mechanical properties, reduced raw material cost and ability to withstand extreme service conditions. Apart from that, mere usage of natural fibres could be deemed as environmentally beneficial due to their natural occurrence and renewable nature.

In the present investigation, a simple and cost efficient approach is adopted to prepare fibre filled rubber composite. These materials were prepared using hemp fibre mats

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which were impregnated with rubber latex. Subsequently, the relevant properties of the resulting fibre filled rubbers were characterised and the effects of differently treated industrial hemp were also investigated.

## MATERIALS AND METHODS

### Fibres Treatments

*Untreated*. Retted industrial hemp fibre (*Cannabis Sativa L.*) supplied by Hemcore (UK) was used as received. The retting process is a common technique to ease the separation of natural fibres by subjecting the fibres to natural rotting.

*Mild alkaline treatment.* Retted hemp fibres were treated with 0.24% NaOH in aqueous solution for 48 hours at room temperature. The alkaline treated fibres were then washed sufficiently with water in a pulp washer. The fibres were dried at 60ºC.

*Fibre Digestion.* Retted hemp fibre was digested with a 5% NaOH and 2% $Na_2SO_3$ in aqueous solution[15]. The digestion process was carried out at a temperature of 120ºC and pressure of 2 bars, for 20 minutes. The digested fibres were then sufficiently washed with water followed by drying at 60ºC as it was done for the mild alkaline treatment.

### Preparation of Fibre Filled Rubber

Randomly oriented non woven fibre mats (10 cm × 15 cm) were prepared from unchopped technical hemp fibres with an average length of less than 18 cm using a fibre carding machine. The prepared fibre mats exhibited average thickness in the range of 2.5 mm to 3.5 mm. The mats were then impregnated with prevulcanised natural rubber latex (MR latex, Revertex, Malaysia) followed by heating at 70ºC under compression of 0.5 kN until sufficiently dried. The resulting fibre weight percentage content in the fibre filled rubber samples were in the range of 17% – 23%. Three types of rubberised fibres comprising (1) untreated fibres (2) alkaline treated fibres (3) digested fibres were prepared. Rubber without fibres incorporation was taken as the control sample.

### Characterisations

*Differential scanning calorimetry (DSC)*. Differential thermal analysis was performed on the fibres using a Shimadzu DSC 60 instrument. DSC measurement was conducted in a temperature range of 30ºC to 500ºC with a heating rate of 5ºC $min^{-1}$ in static air.

*Atomic Force Microscopy (AFM)*. The surface morphology analysis of the fibres and the fibre filled rubber were conducted using an Asylum Research MFP-3D Atomic Force Microscope in the AC-Mode. In the present analysis using AFM, all samples were cut into test pieces and placed onto specimen glass slides. An epoxy adhesive was used to attach the samples to the glass slide. Height and phase imaging were performed simultaneously in the AC mode with scan size and rate of 10.00 μm and 0.45 Hz, respectively.

*Surface friction coefficient measurements*. Surface friction of the fibre filled rubber samples against a wavy glass surface were measured using a Plint TE 75P friction meter at 60 ± 3% relative humidity. The frictional force between sample and the wavy glass surface was measured by attaching the sample over a rubber hemisphere with a diameter of 37 mm and hardness 40 IRHD. The sample was fastened to a sample holder which traversed to and fro over a distance of 20 mm at a speed of 1 mm/s. The load perpendicular to the sample holder was fixed at 2 N. The load was kept

fixed because the friction coefficient is load dependent[16]. Friction coefficients were taken as the displacement force: *F*, divided by the applied load: *P (Equation 1)*. A typical scheme for rubber surface friction measurement is illustrated in *Figure 1*.

$$\text{Surface friction coefficient, } \mu = \frac{F}{P} \qquad \ldots 1$$

*Tensile Strength Measurements*. Dumb bell test pieces suitable for the samples were prepared using the type 2 die according to *ISO 37* (Rubber: Determination of tensile stress-strain properties). Measurements of tensile properties were done on an Instron 5565 tensile tester at 25ºC and 65% relative humidity and a crosshead speed of 500 mm/min. Values were taken as the average of three readings.

## RESULTS AND DISCUSSION

### Surface Morphology of Fibres

AFM images for the fibres are shown in *Figure 2*. These images were further analysed to acquire the values of surface roughness and skewness of the fibres as tabulated in *Table 1*. The results indicate that different types of fibre treatment produced different levels of roughness. The effect of treatments such as alkaline treatment, in influencing fibres surface roughness has been described by several researchers[17–19]. In general, in the present study, it is shown that fibre treatment attained by means of digestion gives rise to a higher level of surface roughness compared to alkaline treatment.

The skewness value can be derived from the surface roughness (*Equations 2* and *3*) as described by Méndez-Vilas *et al*.[20]. Where, $Z_i$, is the height of the $i^{th}$ point, z is the average height and N is the total number of points within the image, while $R_q$ is the root mean square (RMS) roughness. The skewness value provides quantitative asymmetry of the height distribution of the samples. A positive skewness may indicate that the surfaces have peaks protruding out of a fairly flat average.

$$\text{RMS roughness, } R_q = \sqrt{\frac{\sum_{i=1}^{N} (Z_i - \overline{Z})}{N}} \qquad \ldots 2$$

$$\text{Skewness} = \frac{1}{NR_q^3} \sum_{i=1}^{N} (Z_i - \overline{Z}) \qquad \ldots 3$$

It is shown in *Table 1* that there is an observable difference between alkaline treated and digested fibres. For the alkaline treated fibres, roughness is extending out of the surface with a skewness value of 0.07 and the acquired average surface roughness is slightly higher than the untreated fibres. The digested fibres, on the other hand, show the highest roughness values and the value of skewness is negative, indicating that the occurrence of roughness is probably due to furrows in the surface of the fibre.

The surface roughening with positive skewness as shown by the alkaline treated fibres is possibly caused by bulging and swelling of the fibres surfaces in the mild alkaline environment. When digestion is carried out at higher temperature, the alkaline concentration and pressure are believed to be able to affect the fibres surface by eliminating most of the surface chemical components such as pectin, hemicelluloses and lignin. It is likely that surface related artefacts, such as furrows and crazes, were left behind as the surface chemical components were removed. The roughening effect may only be concentrated at a certain point or extent at the surface of the fibres. It should be recognised however, that digestion of fibres at high temperature and pressure can be suggested as a good treatment to introduce a higher level of surface roughening in natural fibres.

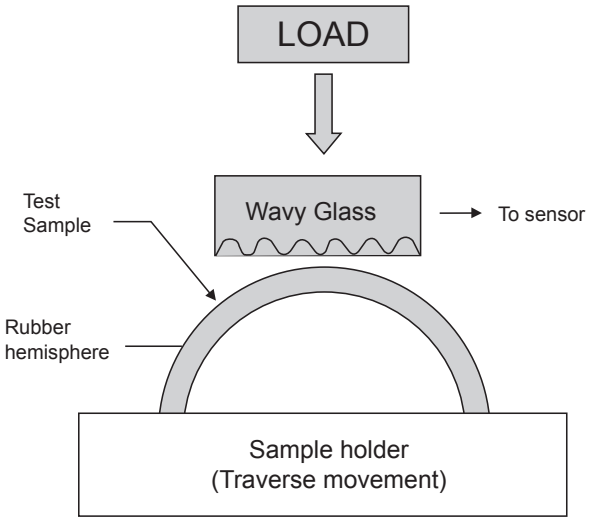


*Figure 1. Schematic of surface friction measurements.*

TABLE 1. SURFACE ROUGHNESS OF FIBRES

| Samples | Surface roughness (RMS) (nm) | Skewness (nm) |
|---|---|---|
| Untreated fibre | 214 | -0.04 |
| Alkaline treated fibre | 274 | 0.07 |
| Digested fibre | 414 | -0.10 |

**Thermal Analysis of the Fibres**

The objective of conducting thermal analysis on the fibre samples was to determine the effect of treatment on their degradation profile. Thermal analysis of hemp fibre using DSC was reported by Troedec *et al*.[21] and Aziz *et al*.[22]. It was described that three distinct exothermic regions were exhibited in thermal decomposition of hemp fibre, namely, in the region of approximately 320ºC to 370ºC, 390ºC to 420ºC and a region higher than 420ºC. These exothermic regions were attributed to cellulose depolymerisation, decomposition of hemicelluloses and pectins, and the decomposition of lignin residues respectively. The exothermic peaks of the fibres obtained from DSC measurements in the present study are shown in *Table 2*. Deviations from the reported values were observed and these are likely to be due to different fibre treatment and the specific DSC measurement parameters used in the present study.

The most obvious differences in the DSC thermograms obtained for the fibre samples

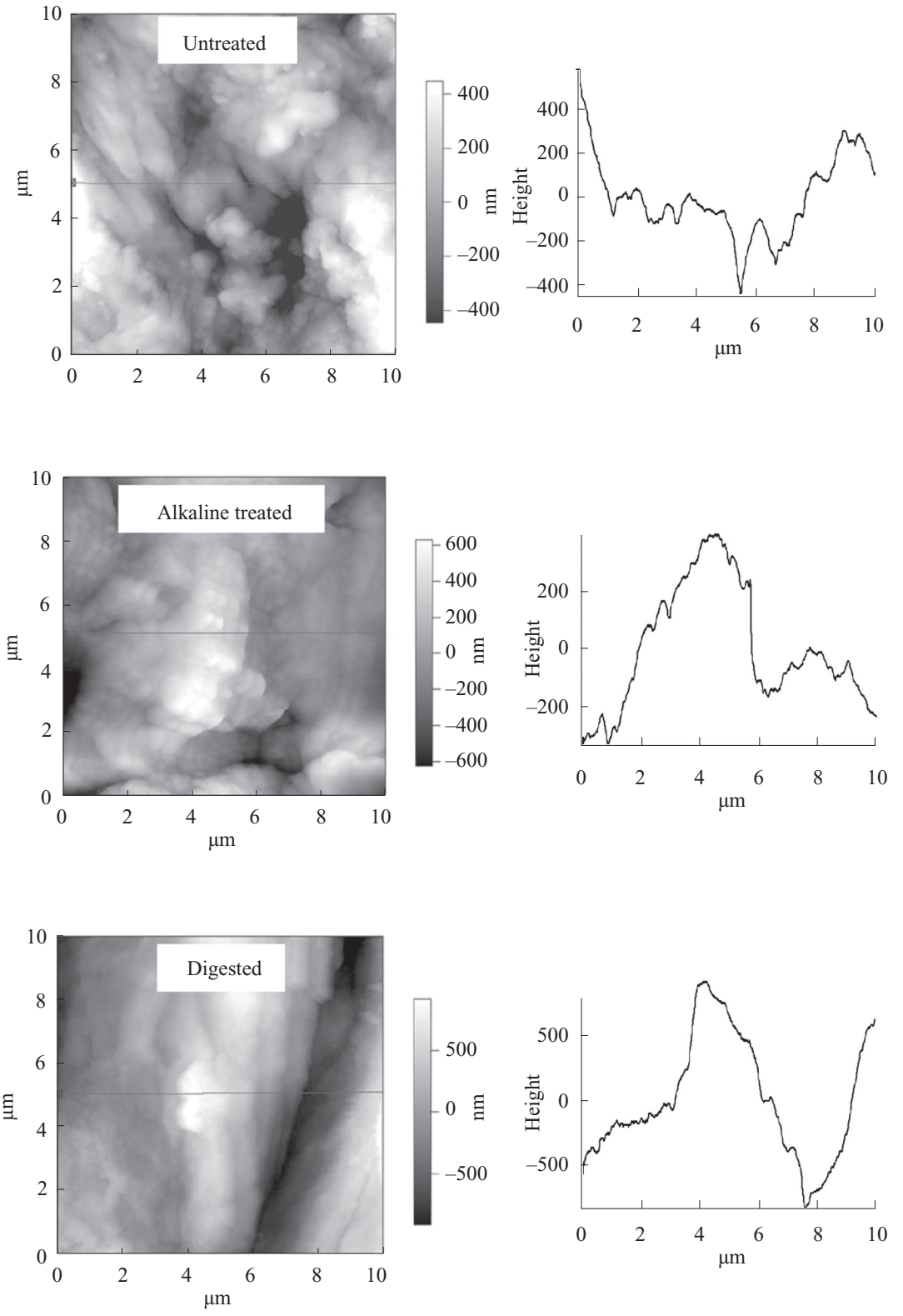


*Figure 2. AFM images of fibres.*

TABLE 2. EXOTHERMIC PEAKS OF FIBRES

| Samples | Cellulose depolymerisation Peak/ºC | Hemicellulose decomposition Peak/ºC | Cellulose decomposition Peak/ºC |
|---|---|---|---|
| Untreated fibre | 335 | 375 (351) | 445, (431) |
| Alkaline treated fibre | 317, (346) | 376, (367) | 443, (460) |
| Digested fibre | 325 | 380 | 468 |

Values in parentheses are obtained from references 21 and 22

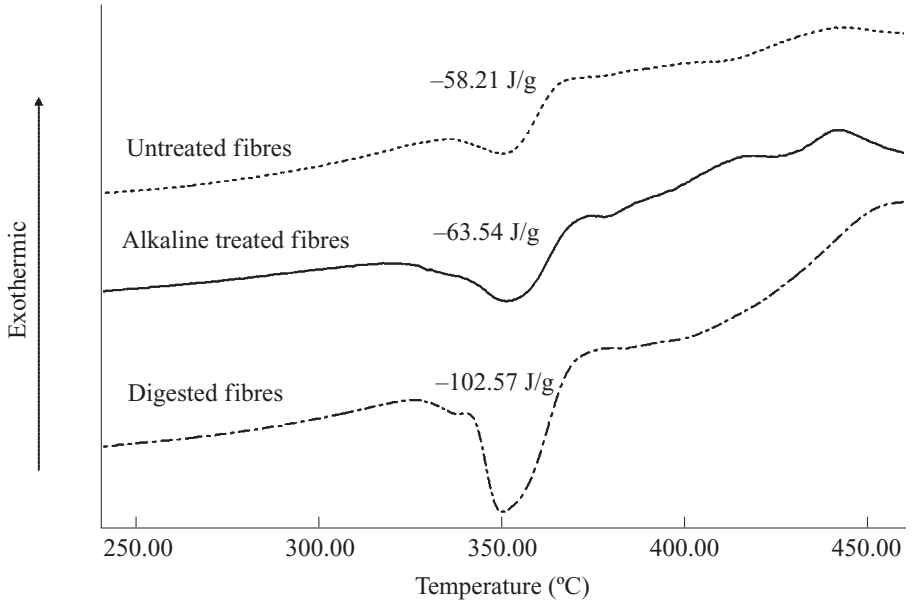


*Figure 3. DSC Thermograms of fibres.*

were found in the region of *ca*. 350ºC. *Figure 3* shows the difference in heat absorbed in the endothermic peaks. The DSC thermogram of the digested fibres displays a relatively larger amount of heat adsorbed prior to the decomposition at higher temperatures. This is related to the amount of energy required to disrupt crystalline domains of cellulose[23], because it is believed that cellulose is the major component left in the fibres after the digestion.

**Fibre Filled Rubber Surface Friction**

In the present study, a glass surface was pressed against a sample on a rubber hemisphere. For such surface according to the classical theory of Hertz[24,25], the friction coefficient, μ, can be written as in *Equation 4*.

Coefficient of friction,

$$\mu = \pi\tau\,(9R/16E)^{2/3}\,W^{-1/3} \quad \ldots 4$$

In the equation, $R$ is the slider radius, $\tau$ is the interfacial shear strength, $W$ is the load and $E$ is the Young's modulus. The interfacial shear strength depends upon factors such as the presence of contaminations and rubber visco-elasticity, while the area of contact relates to the hardness and surface roughness of the material in contact.

*Table 3* shows the surface roughness from AFM analysis for the fibre filled rubber composites and surface friction values obtained from friction measurements. It is observed that the friction coefficients for the rubber filled with untreated fibres are fairly similar to the rubber control sample. In the present investigation, a fairly large reduction in friction values is observed in the fibre filled rubber comprising digested fibre. This is most likely due to the higher level of surface roughness and skewness values.

Thus, in the present study, it can be concluded that to acquire low surface friction for the composites, digested fibres can be suggested to be a better material in comparison to alkaline treated fibres.

**Fibre Filled Rubber Tensile Strength**

*Table 4* shows the mechanical properties of the fibre filled rubber samples. It is observed that the elastic modulus at 300% elongation increased in comparison to the rubber control sample. Marked reductions in tensile strength in comparison with the rubber control sample are seen for all fibre filled rubbers. The elongation at break is reduced by 5.6% in the samples comprising digested fibres, while the fibre filled rubber consisting of untreated fibres and alkaline treated fibres are reduced by approximately 20%.

These results can be compared to wood filled natural rubber[26] where a 50% reduction in tensile strength was observed when wood powder was added at more than 10 parts per hundred rubber (p.h.r.). It is assumed that tensile failures in elastic materials such as

TABLE 3. SURFACE FRICTION VALUES OF FIBRE FILLED RUBBER COMPOSITES

| Samples | Mean Friction (N) | Coefficient of Friction (μ) | Average Surface Roughness/rms (nm) | Skewness (nm) |
|---|---|---|---|---|
| Rubber control | 4.42 | 2.21 | 122 | -1.00 |
| Untreated fibres | 4.25 | 2.13 | 274 | 0.04 |
| Alkaline treated fibres | 3.94 | 1.97 | 274 | 0.17 |
| Digested fibres | 3.60 | 1.80 | 283 | 1.48 |

TABLE 4. MECHANICAL PROPERTIES OF RUBBERISED FIBRES

| Samples | Tensile stress at max load (MPa) | M300 (MPa) | EB (mm) |
|---|---|---|---|
| Rubber control | 22.9 | 1.0 | 940 |
| Untreated fibres | 7.6 | 1.3 | 753 |
| Alkaline treated fibres | 5.8 | 1.5 | 747 |
| Digested fibres | 4.4 | 1.6 | 887 |

rubber are initiated from flaws or stress-raisers. These flaws can be in the form of physical discontinuities such as furrows, crazes or scratches in the surface or other particles embedded in the material. Any inhomogeneous phase in the composition or structure of the rubber can also represent a flaw. Hence, the incorporation of natural fibres into rubber devoid of reinforcing filler particulates, introduces a higher level of inhomogeneity and this eventually leads to the lowering of the rubber strength. The fibre filled rubber samples comprising digested fibres exhibit the lowest strength, yet they retained their high elasticity modulus and gave quite a large value of the elongation at break under tensile stress. It is most likely that the fibres were appropriately impregnated with rubber latex, but without an appropriate amount of coupling agent, hence the adherence of the fibre to the rubber was poor. It is expected that the strength of the composite can further be improved by using a few layers of fibre mats in the preparation of the fibre filled rubber composites.

Even though the fibre demonstrated poor adherence to rubber as shown by tensile strength measurements, they may offer additional options over other conventional rubber materials. Such an application, for instance is a soil protective cover where strength in the service application is not a requirement. The fibre filled rubber exhibited better protection against soil erosion in comparison to conventional plastic canvas in simulation experiments described elsewhere[27].

## CONCLUSIONS

Preparation of fibre filled rubber composites utilising industrial hemp fibres impregnated with rubber was shown. In the present study, the fibres consisted of untreated, alkaline treated and digested hemp fibres. Characterisation of the fibres using DSC and AFM indicated certain changes as results of fibre treatments. Digested fibres exhibited higher resistance to thermal degradation and had higher levels of surface roughness relative to untreated and alkaline treated fibres. The occurrence of a higher level in surface roughness was also reflected in the properties of the resulting composite. In the present study, fibre digestion is suggested as the most suitable process to increase the fibre surface roughness. Even though the fibre demonstrated poor adherence to rubber, as shown by tensile strength measurements, they may offer additional options over other conventional rubber materials. Such an application, for instance, is a soil protective cover where strength in the service application is not a requirement.

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