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# Kenaf–polypropylene composites: Effect of amphiphilic coupling agent on surface properties of fibres and composites

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ABSTRACT

This paper presents an experimental study on the use of zein as a coupling agent in natural fibre composites. Kenaf nonwovens were treated with zein coupling agent, which is a protein extracted from corn. The surface characteristics of untreated and chemically treated kenaf fibres were investigated by FTIR, zeta-potential measurements and Energy Dispersive X-ray Spectroscopy (EDS) mapping. Composites were prepared by compression moulding using nonwovens treated with zein solution. The reinforcing properties of the chemically treated composites were compared with that of untreated composites. The viscoelastic and thermal properties of composites were also determined. Composites containing chemically modified kenaf fibres were found to possess improved mechanical and viscoelastic properties. EDS mapping studies revealed the presence of surface functionalities on treated kenaf fibres.



## 1. Introduction

The use of plant fibre reinforced polymer composites has been the centre of attention of the research community during the last decade (John & Thomas, 2008). The most interesting aspects of natural fibre composites are the high specific properties and the fact that natural fibres are renewable and biodegradable thereby creating a positive environmental impact. Among natural fibre composites, kenaf fibre reinforced composites have found potential applications for mobile phone shells consisting 15–20% kenaf fibres (Iji, 2008). Another example in the automobile industry is the Toyota RAUM, which is equipped with a spare tire cover made of kenaf fibre composites (Anonymous, 2007).

The advantages of using polypropylene (PP) as matrix are their low cost and relatively low processing temperature which is essential because of low thermal stability of natural fibres. Amongst eco-compatible polymer composites, special attention has been given to PP composites, due to their added advantage of recyclability. In an interesting study, Srebrenkoska, Gaceva, Avella, Errico, and Gentile (2008) found that kenaf fibre reinforced polypropylene composites were less sensitive to reprocessing cycles and properties of the composites were unchanged after recycling.

Most of the studies relating to natural fibre reinforced polypropylene composites use maleic anhydride grafted polypropylene as a compatabilizer (Beg & Pickering, 2008; Cantero, Arbeliaz, Lano-Ponte, & Mondragon, 2003). Beckmann and Pickering (2009) investigated the properties of $NaOH/Na_2SO_3$ treated hemp fibre reinforced polypropylene composites containing 4% MaPP. The experimentally obtained tensile strength was found to be one-third of the theoretical prediction. This was attributed to non-axial planar-random orientation of the fibres within the composite. In an earlier study, the authors optimised the concentration of $NaOH/Na_2SO_3$ treatments on hemp fibres and observed that properties of treated hemp fibres were superior to untreated fibres. Thermogravimetric analysis revealed that thermal stabilities of untreated and treated polypropylene composites were similar (Beckmann & Pickering, 2008).

In an interesting study, the effect of hybridization of kenaf fibre and wood flour on the dynamic rheological properties of polypropylene composites was investigated by Ghas Ghasemi, Azizi, and Naeimian (2009). It was observed that storage modulus of the composites increased with filler loading and the Cole–Cole plots revealed that the relaxation times shifted to higher values with the addition of fillers and the longest relaxation times were related to composites with pure wood flour.

Shibata, Cao, and Fukumoto (2006) prepared light weight laminate composites from kenaf and polypropylene fibres. The effects of the number of kenaf layers, heating time and kenaf weight fraction on the flexural modulus of the composite specimen were inves-

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tigated. It was observed that the flexural modulus increased with increasing number of kenaf layers and heating time. The increase of the number of kenaf layers contributed to homogeneous PP dispersion in the composite board. This is because more kenaf layers caused better contact between kenaf and PP and prevented PP fibres from shrinking by heating.

Natural fibres are hydrophilic in nature as they are lignocellulosic, which contain strongly polarized hydroxyl groups and require chemical modification to increase the compatibility and adhesion between fibres and matrix (John & Anandjiwala, 2008). In most of the studies cited in literature, the chemical modifications employed are synthetic and toxic. It would be ideal if the chemicals used for the modification of natural fibres preserve the biodegradable nature of natural fibres. In this study, we have used zein-protein from corn as a coupling agent to see its effect on interfacial adhesion in kenaf fibre reinforced polypropylene composites.

This study focuses on the reinforcement effects of chemically modified and unmodified kenaf polypropylene composites. The viscoelastic and thermal stability of composites have been investigated. The surface characteristics (qualitative) of untreated and treated kenaf fibres have also been examined.

## 2. Experimental

### 2.1. Materials

Kenaf fibres were procured from Brits Textiles, South Africa. The fibres received in a bale form were opened and cleaned before processing into nonwovens. As the kenaf fibres still contained a lot of woody pith and other particles, it was subjected to a further opening process in the Trusschler and these fibres were used to produce needle-punched nonwovens. The needle-punched nonwovens from 100% kenaf fibres had an area weight of 110–140 g/m$^2$. Polypropylene in sheet form (6 mm thickness), with a density of 0.9 g/cm$^3$ and melt flow index of 1.5 g/10 min was procured from Ampaglas, SA. Zein was obtained from Scientific Polymer Product Company, Ontario, NY. All other chemical reagents used in this study were of analytical grade.

### 2.2. Zein modification of kenaf nonwovens

Zein belongs to the characteristic class of proteins known as prolamines which occur specifically in cereals. The protein products from corn wet milling are corn gluten meal (CGM) and corn gluten feed (CGF) and zein is obtained as a by-product from corn gluten meal (Momany et al., 2006; Shukla & Cheryan, 2001; Wang, Wang, Geil, & Padua, 2004).

2% of zein solution was prepared by mixing with an ethanol/water mixture in the ratio of 80/20. The kenaf nonwovens were immersed in this solution and were allowed to stand for 2 h. The ethanol/water mixture was drained out and the nonwoven was dried in air and then in an oven at 110 °C until completely dry. These nonwovens were used to prepare the modified composites.

### 2.3. Preparation of composites

Composites were prepared from nonwoven kenaf and polypropylene on the basis of varying fibre content. The kenaf nonwoven mats were cut into small uniform squares (30 cm × 30 cm) and then dried in an air oven at the temperature of 110 °C for 7 h. The dried nonwoven mats were placed between weighed polypropylene sheets. This was wrapped in Teflon® sheets and sandwiched between two aluminium plates. These two plates were then placed between the two platens of compression moulding press and cured at a pressure of about 35 bar for 20 min at 210 °C, followed by cooling under pressure for 3 min.

## 3. Analysis

### 3.1. Characterization of fibres

*FTIR*. Infrared spectra of the untreated and treated kenaf fibres were recorded with an FTIR spectrometer [Perkin Elmer Spectrum 100 FTIR Spectrometer with an ATR (Attenuated Total Reflectance) sampling accessory]. The spectra were analyzed over the range of 4000–650 cm$^{-1}$

*Electrokinetic measurements*. Electrokinetic measurements were carried out to determine the zeta-potential ($\zeta$) of fibre surfaces. The electrokinetic analyzer EKA (Anton Paar KG, Graz, Austria) was based on the streaming potential method. An electrolyte solution was forced by an external pressure ($p$) through a bundle of capillaries (fibre plug). The potential ($U$) resulting from the motion of ions in the diffuse layer was measured with respect to the applied pressure. The electrokinetic potential or zeta-potential ($\zeta$) was calculated from the measured streaming potential using Smoluchwski's equation ($\Delta U/\Delta p$). Hence, the calculated zeta-potential is considered as an apparent zeta-potential ($\zeta$ app). The details of the measuring technique are reported elsewhere (Jacobasch, Simon, Werner, & Bellmann, 1992). By measuring the pH dependence of the zeta-potential, the Brønsted acidity or basicity of solid surfaces can be determined qualitatively.

*EDS mapping*. EDS analysis was carried out using a FEI ESEM-EDS Quanta 200 scanning electron microscope. Fibre samples (uncoated) were clamped and sectioned in such a way that a freshly cut surface was presented to the analysing electron beam. The samples were examined at an accelerating voltage of 20 kV and a working distance of 6.6 mm. The horizontal field width (HFW) of the image is 746 μm. The detector (*LN2 Si-Li ED)* was set to the energy of the sodium $K_\alpha$ electrons and the selected area was repeatedly scanned so that an elemental density map was generated.

### 3.2. Characterization of composites

Tensile and three-point bending tests were carried out using an Instron Universal Testing Machine, model 3369. Tensile testing on rectangular specimens was measured according to ASTM D638 at a crosshead speed of 50 mm/min and a gage length of 50 mm. Flexural testing was carried out in accordance with ASTM D-790, at a crosshead speed of 5 mm/min and a span length of 60 mm.

Charpy impact strength was measured on an Instron Dynatup, according to ASTM D256. Following test conditions have been used; span length 80 mm and drop weight 6.39 kg. During impact, resistive force exerted by the sample on the striker was measured as a function of time.

Five specimens were tested for each test and the average data have been reported.

Dynamic mechanical analysis was carried out using the Perkin Elmer DMA 8000. Samples of dimensions 50 mm × 12 mm × 3 mm were used for testing. The testing temperature ranged from −20 °C to 150 °C and the experiment was carried out at frequencies 0.1, 1, 10 and 100 Hz. The samples were tested under dual cantilever mode at strain amplitude of 0.05 mm.

Thermogravimetric (TGA) studies were carried out using a (Pyris 1 model, Perkin Elmer) in an inert atmosphere at a heating rate of 10 °C/min. The temperature range used for the analysis is 30 °C–700 °C.

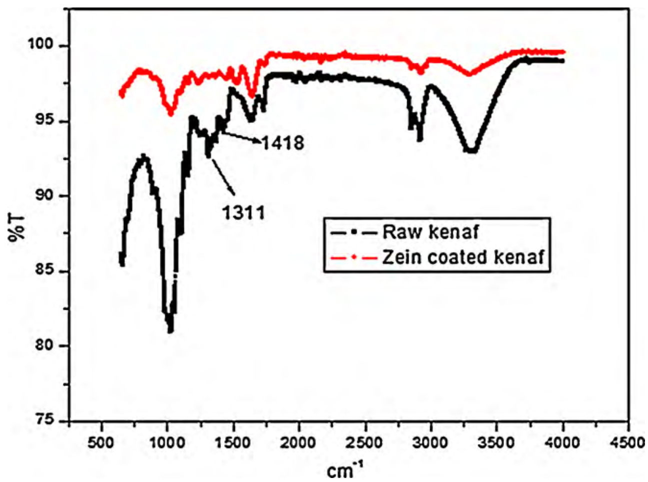



**Fig. 1.** FTIR spectra of untreated and zein coated kenaf fibre.

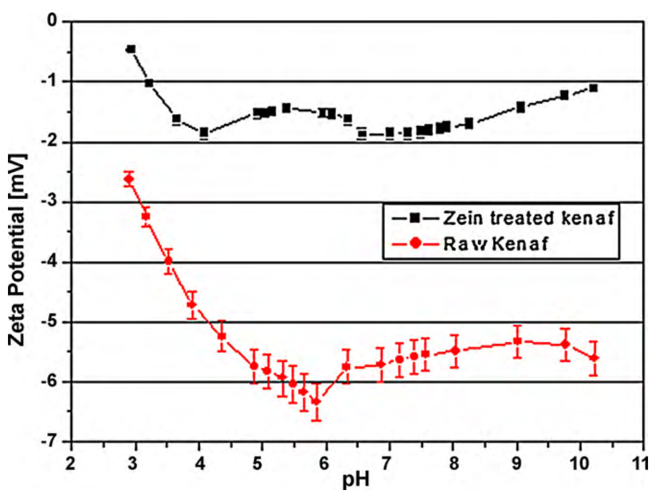



**Fig. 2.** Zeta-potential measurements of raw and zein coated fibres.

## 4. Results and discussion

### 4.1. Surface characterization of kenaf nonwovens

#### 4.1.1. FTIR and zeta-potential studies

The amino acid composition in zein (Di Gioia, Cuq, & Guilbert, 2000) indicates the presence of both polar and non-polar constituents, the major proportion being glutamine. The FTIR spectra of untreated and zein treated kenaf nonwovens are given in Fig. 1. The peaks at 3329 cm$^{-1}$, 1636 cm$^{-1}$ and 1050 cm$^{-1}$ are assigned to –OH stretching, absorbed water and –C–O/C–C stretching vibrations respectively. The peak at 1731 cm$^{-1}$ assigned to –CO stretching is more intense in the treated fibre indicating intermolecular attractions. The emergence of new bands on the zein coated kenaf fibres at 1311 cm$^{-1}$ and 1418 cm$^{-1}$ assigned to C–N stretching are indicative of the fact zein coating has modified the fibre surface.

The electrokinetic measurements were used to characterize the acid–base properties of chemically modified fibres. Fig. 2 presents the pH dependence of the apparent zeta-potential. It can be seen that there is a significant difference between untreated and zein treated kenaf nonwovens. For the untreated kenaf nonwovens, the iso-electric point (IEP, pH where zeta-potential is zero) is found at pH = 2.1. The Stern theory of electrochemical double layer (Stern, 1924) relates this point to the number of Brønsted acid surface sites. The increase of pH lowers the zeta-potential indicating the gradual loss of protonated surface groups (Pothan, Simon, Spange, & Thomas, 2006).

The negative zeta-potential also suggests the dissociation of Brønsted acid surface sites. The zeta-potential versus pH curve has a shape that is typical for surfaces with hydrophilic character. It can be observed that there are two distinct plateau phases for the untreated kenaf fibre—first plateau starts at pH ~3.5 and second plateau starts at pH ~6.2. These phases indicate the two different surface charging mechanisms, the dissociation of (Brønsted acid groups) followed by adsorption of $OH^-$ ions on the fibre surface. The IEP of the treated kenaf fibre shifts to higher pH values indicating that the polarity of the fibres has changed.

#### 4.1.2. ESEM and EDS mapping

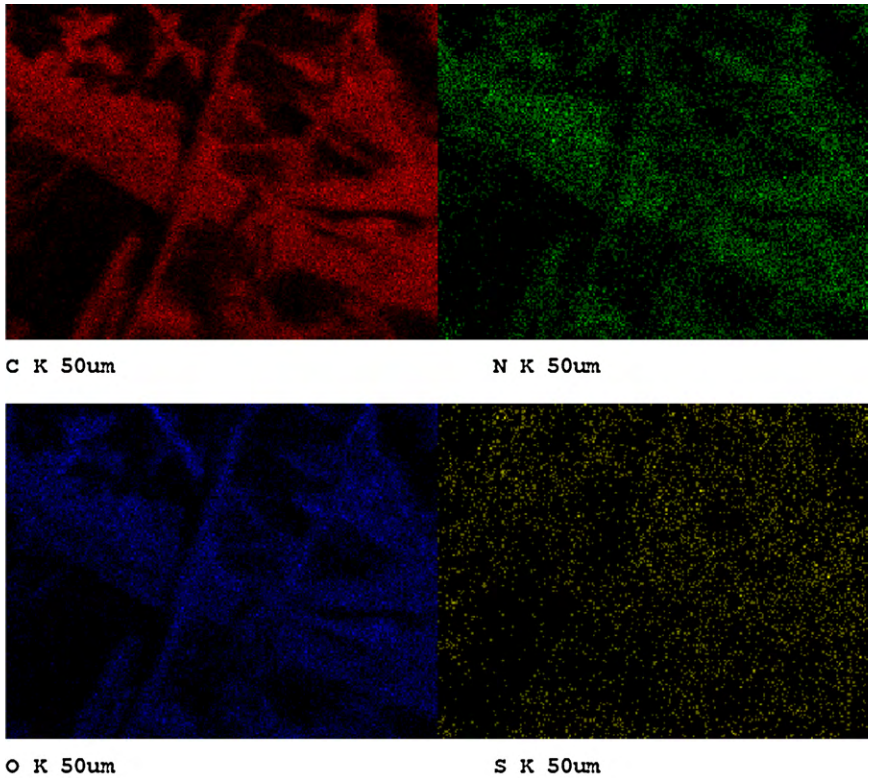
Fig. 3 presents the corresponding density map of EDS spectrum of zein coated kenaf sample and the quantitative elemental analysis of treated kenaf fibres. Natural fibres contain cellulose, hemicellulose, lignin and waxes. As a result, it contains organic matter as carbon and oxygen. Inorganic elements like silicon can also be present. The non-cellulosic constituents in natural fibres include proteins, amino acids and other nitrogen containing compounds. Most of the nitrogenous materials occur in the primary cell wall as well as the lumen of the fibre as protoplasmic residue (Lewin, 2007). This explains the presence of nitrogen in the untreated kenaf fibres. It can be observed that there is an increase in concentration of nitrogen and sulphur for the treated fibre indicating that there is presence of zein on the surface of fibres. EDS mapping also revealed the distribution patterns of nitrogen and sulphur on the zein coated kenaf fibres. The distribution of sulphur seems to be uneven and random but a uniform and higher concentration of nitrogen was detected.

### 4.2. Effect of zein modification

Figs. 4 and 5 exhibit the tensile and flexural properties of untreated and zein modified kenaf composites at 30% fibre loading. It can be seen that after modification flexural strength increased by 7% while tensile strength did not register a significant increase. It may be noted that the authors had observed a higher percentage of increment in the case of zein treated flax-PP composites (John & Anandjiwala, 2009). This can be attributed to the physical nature of kenaf fibres. Kenaf stem contains two types of fibre, bast fibre and woody core fibre. The bast fibres need to be separated from the woody core fibre before being used in composites (Lips, Iñiguez de Heredia, Op den Kamp, & van Dam, 2009). In the present case it was observed that the bast fibres contained a lot of woody particles and pith that were not completely removed even though the fibres were subjected to intensive cleaning process. As a result it is most probable that zein solution was not able to coat the kenaf fibres uniformly.

Impact strength (Fig. 4) is seen to decrease due to the modification of kenaf fibres with zein protein. The energy dissipation mechanisms operating during impact fracture are matrix and fibre fracture, fibre–matrix debonding and fibre pull out. Fibre fracture dissipates lesser energy compared to fibre pull out. The main failure mechanism in these composites is fibre fracture (as there is not significant interfacial adhesion), resulting in lower energy dissipation and hence impact strength decreases.

Zein is neither soluble in pure water nor in alcohol but requires a high percentage of alcohol-aqueous system for dispersion There are mainly four types of zein ($\alpha$,$\beta$,$\chi$,$\delta$) which are classified according to their solubility properties. The isoform $\alpha$-zein, which accounts for ~85% of zein in the corn kernel, has a unique amino acid sequence containing more than 50% non-polar amino acids. The secondary and tertiary structure of zein was reported as having a possible configuration containing 9 or 10 $\alpha$-helix segments folded upon each




| Fibres | Element (Wt %) | | | |
|---|---|---|---|---|
| | C | O | N | S |
| Untreated kenaf fibre | 62.5 | 35.27 | 1.01 | - |
| Zein treated kenaf fibre | 55.5 | 25.1 | 18.6 | 0.6 |

**Fig. 3.** Density map of EDS-spectrum of zein coated kenaf sample.

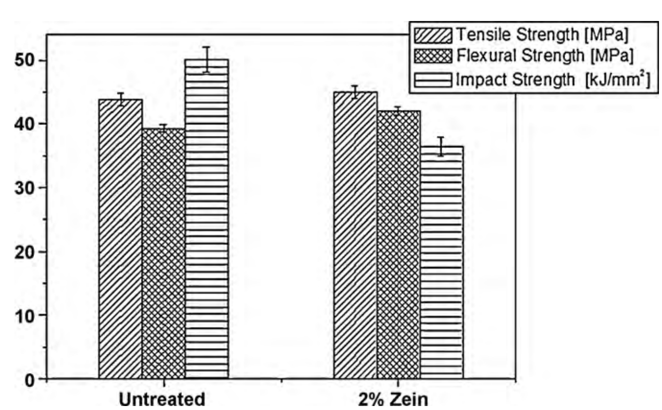



**Fig. 4.** Variation in tensile, flexural and impact strength of untreated and treated composites.

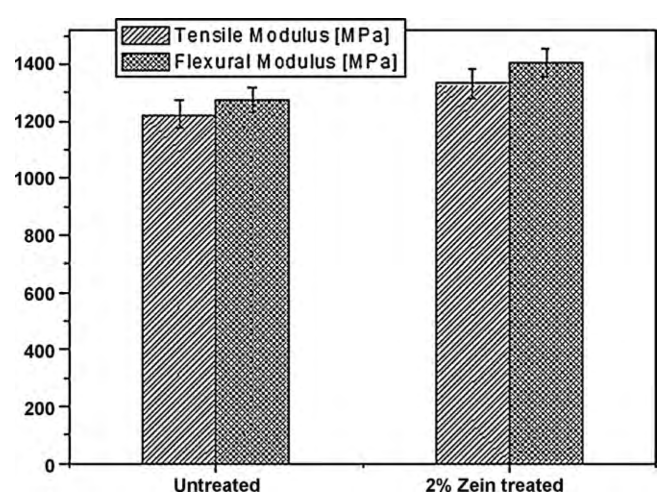



**Fig. 5.** Variation of tensile and flexural modulus of untreated and treated composites.

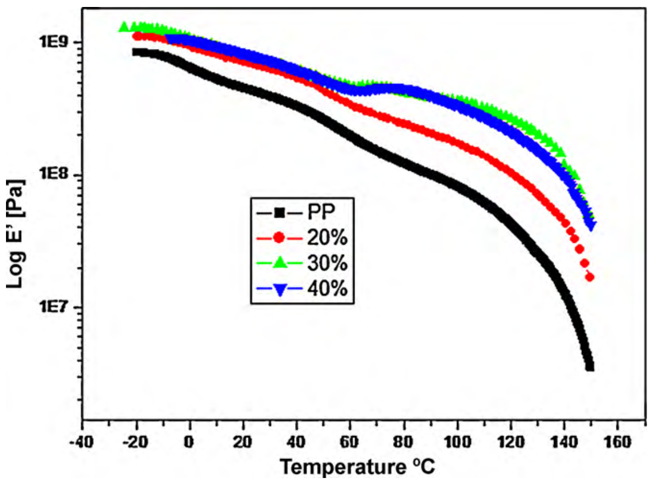



**Fig. 6.** Variation in storage modulus with temperature as a function of fibre loading.

other in a nonparallel fashion. According to the model proposed by Argos, Pedersen, Marks, and Larkins (1982) helical segments are arranged in a ring of "pencils" held together, side-by-side, by hydrogen bonds and linked at each end by glutamine-rich turns or loops. The exterior of the helical segments forming the lateral faces have a hydrophobic character, whereas the top and bottom surfaces containing the glutamine-rich loops are hydrophilic. Therefore, zein is amphiphilic in nature having affinity for both polar and non-polar groups. This characteristic allows it to bind itself between the polar kenaf nonwovens and non-polar matrix results in enhanced mechanical properties.

### 4.3. Dynamic mechanical analysis

#### 4.3.1. Effect of fibre loading

*4.3.1.1. Storage modulus.* Storage modulus ($E'$) provides valuable input into the stiffness of composites and measures the elastic response of a material. The variation of storage modulus with temperature (measured at 1 Hz) at different kenaf fibre loading is given in Fig. 6. It can be seen that storage modulus increases with increasing kenaf content at all temperatures when compared to the polypropylene. When fibres are incorporated in the polypropylene matrix, the stiffness of the composite increases resulting in high storage modulus. Also, the addition of fibres allows effective stress transfer at the interface, which consequently increases the storage modulus.

*4.3.1.2. Loss modulus and damping properties.* Loss modulus is a measure of the viscous response of a material. Table 1 shows that loss modulus (at 20 °C) increases with increase in fibre loading. The increase in loss modulus is attributed to the increase in energy absorption caused by the addition of fibres. It can be observed that upon incorporation of kenaf nonwovens, tan $\delta$ decreases. Incorporation of nonwovens acted as barriers to the mobility of polymer chains, leading to lower degrees of molecular motion and hence lowers damping characteristics. The glass transition temperature does not seem to have a significant co-relation with fibre loading.

**Table 1**
Loss modulus, $T_g$ and peak temperatures of composites.

| | $E''$ [Pa], 20 °C | tan $\delta$ | $T_g$ [°C] | Temperatures [°C] | |
|---|---|---|---|---|---|
| | | | | Peak I | Peak II |
| PP | $5.23 \times 10^7$ | 0.1280 | 1.2 | – | 501.8 |
| 20 | $5.78 \times 10^7$ | 0.1275 | 2.6 | 411.37 | 524.07 |
| 30 | $6.51 \times 10^7$ | 0.1247 | 0.61 | 415.60 | 535.5 |
| 40 | $6.81 \times 10^7$ | 0.1002 | 4.8 | 421.84 | 533.4 |
| 2% zein | $7.434 \times 10^7$ | 0.1183 | 1.4 | 380.10 | 519.4 |

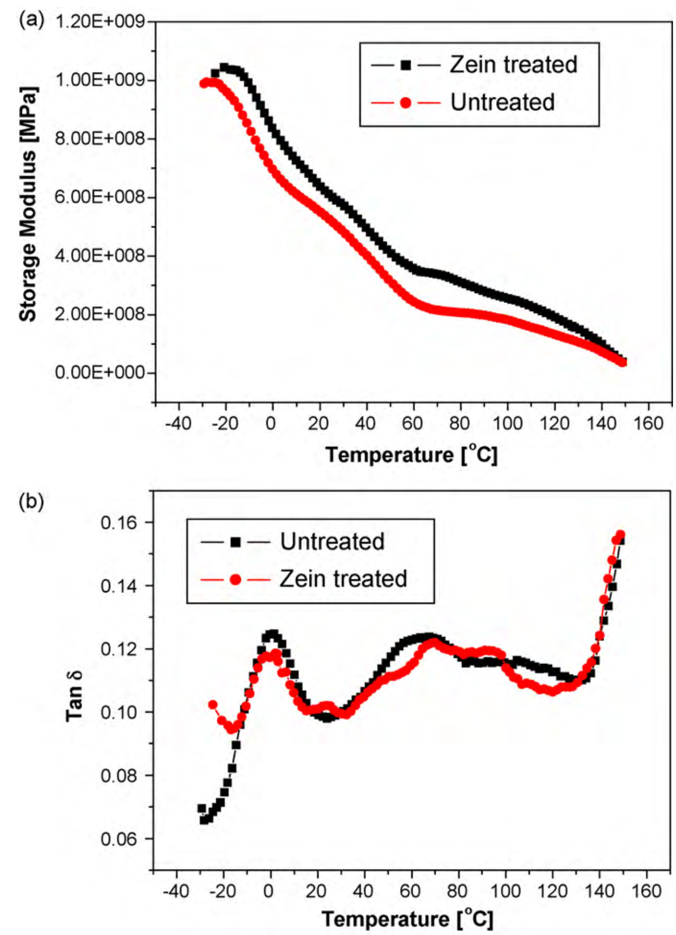



**Fig. 7.** Variation in storage modulus and tan $\delta$ of untreated and treated composites.

#### 4.3.2. Effect of zein coating

*4.3.2.1. Storage modulus and tan $\delta$.* The variation of storage modulus and tan $\delta$ with zein coating for 30% kenaf fibre composites is given in Fig. 7(a) and (b). It can be observed that storage modulus of the treated composites shows an increase. This can be attributed to the better reinforcing effects which increase the thermal and mechanical stability of the material at higher temperatures. It must be noted that the increment is prominent between 60 and 80 °C temperature ranges. In Fig. 7(b) the position of β-relaxation was found to be shifted to higher temperatures and magnitude of tan $\delta$ was seen to decrease for chemically modified composites. This was attributed to a more compact structure in treated composites leading to further hindrance of molecular motions which consequently reduced tan $\delta$.

#### 4.3.3. Thermal analysis

*4.3.3.1. Effect of fibre loading.* Table 1 presents the peak temperatures obtained from derivative thermograms of all the composites. The degradation of polypropylene is a one step process and the major peak is observed around 501.8 °C. In the composite two peaks were obtained; a minor peak at 410 °C due to hemicellulose and α-cellulose degradation and the major peak at 524.1 °C indicating higher thermal stability for the composites. The addition of kenaf nonwovens results in an increase of degradation temperatures which could be attributed to consolidation effects (Araujo, Waldman, & De Paoli, 2008). On comparing the stability of the untreated and treated composites it was seen that thermal stability of composites containing zein coated flax nonwovens decreased when compared to the untreated sample.

## 5. Conclusions

This study focused on the effect of using a chemical modification that preserves the renewable and biodegradable character of natural fibres. Zein coating of kenaf nonwovens was found to enhance the flexural and viscoelastic properties of composites. The storage modulus increased for composites containing zein coated kenaf fibres indicating increased stiffness in treated composites. Chemical modification of kenaf nonwovens resulted in a slight decrease of thermal stability. Surface characterization of raw and chemically modified kenaf nonwovens revealed the presence of surface functionalities. Energy dispersive X-ray analysis confirmed the qualitative and quantitative evidence of nitrogen and sulphur on the surface of the zein coated fibres.

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