

Sorption studies of few selected raw and nanoclay infused lignocellulosic fibres

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Effect of lumen morphologies of selected raw untreated and nanoclay infused lignocellulosic fibres on the sorption characteristics have been studied. Sisal, kenaf, banana and coir fibres have been selected, and the sorption characteristics, such as adsorption, desorption and absorption properties are evaluated. Nanoclay particles are effectively impregnated into the fibres, and the particles serve as a barrier medium by modifying the fibre-gas/liquid interface. The result indicates that the nanoclay treated fibres result in reduced moisture adsorption with increased fibre surface area. The adsorption-desorption characteristics of untreated and nanoclay treated fibres show sigmoidal isotherm sorption behavior, and the sorption hysteresis depends on crystallinity and nanoclay infusion. It is observed that the absorption characteristics of untreated and nanoclay treated fibres depend upon the nanoclay treatment and lumen phase morphology.

Keywords : Absorption, Adsorption, Coir fibre, Desorption, Lignocellulosic Fibres, Nanoclay

1 Introduction

In recent past, natural fibres have attracted much attention as an important class of reinforcement material for composites due to low cost, higher strength/surface area and availability in abundance^{1,3}. However, the hydrophilic nature of the natural fibres is still a major problem and limits its use. The water uptake causes dimensional changes to the fibres and affects the fibre properties, fibre-matrix adhesion and ultimately the composite properties^{4,5}. Due to this, the mechanical and physical properties of the composites are affected. Properties, such as stiffness, modulus, density and fibre-matrix adhesion, are usually affected^{6,8}. Therefore, moisture uptake is an important characteristic to be considered for a wider application base^{9,10}. In addition, due to the ongoing global trend of using green and natural materials, it is now more relevant to focus on the water barrier properties of the natural fibres.

The raw untreated fibres absorb more moisture and this is limited by the fibre type. The sorption is proportional to lignin and other non-cellulosic phases of the fibre¹¹⁻¹⁵. Chemical treatment of fibres reduces water uptake; however, the amount of water sorption depends on the type of chemical treatment¹⁶⁻¹⁸. The adsorption-desorption properties of natural fibres are

characterized by sigmoidal shape of isotherms with formation of the hysteresis loop between the sorption and desorption curves. The hysteresis is represented by degree of hysteresis (dH), which depends on the fibre quality, fibre type and non-cellulosic phase in the fibre^{19, 20}. Recently, there is a new interest of infusing nanoparticles into the natural fibre surface²¹⁻²⁶. Such process results in a dramatic increase in mechanical (tensile, strength and fibre-matrix adhesion) and thermal properties (decomposition temperature and T_g) of fibres composites. In few reports, improved barrier properties were also reported^{21,23,25}. Since the impregnation of nanoparticles is relatively a new subject, much more understanding on this subject is still required. For instance, the influence of nanoparticles on the cell-wall structure and lumen phase of the fibre is not fully understood. It is well known that these phases constitute major part of the fibre and affects water sorption behavior of the fibre. Therefore, the objective of this study is to investigate the influence of nanoparticle on the lumen and cell wall phase of the fibre and its effect on the water sorption properties. To carry out this study, four different lignocellulosic fibres are chosen, depending on the lumen morphology and geometry. Montmorillonite (MMT) clay is chosen as nanoparticle for infusion into the fibres. MMT clays consist of nanolayered alumina-silica ceramic particle, which serves as a

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high barrier medium to the water vapor and molecules^{27,28}. The outcome of this work will help to understand the role of lumen/cell wall structure in nanoclay infused fibre on fibre-gas/liquid interface sorption.

2 Materials and Methods

2.1 Materials

Banana and coir fibres were extracted from the stem section of locally grown plant/tree by mechanical crushing method. Kenaf fibres were obtained from Agricultural Research Council (ARC) of South Africa. Sisal fibres were obtained from University of Dar es Salaam, Tanzania. All these fibres were extracted at their matured cultivation stage. Na⁺ montmorillonite (MMT) clay was obtained from Southern Clay Products, Inc. USA, supplied under the trade name Na⁺ Cloisite. All the chemicals, to be used for nanoclay infusion were obtained from Merck Chemicals, South Africa.

2.2 Nanoclay Infusion Treatments

Nanoclay was infused into the fibres by a two-step process. In the first step, the raw fibres were alkaline treated (NaOH treatment) and in second step these treated fibres were infused with nanoclay particles using a shear induced mechanical mixing process.

In the NaOH treatment, 1 mol/L of NaOH solution was taken and the equivalent weight of 5 cm long chopped untreated fibre (sisal, banana, kenaf and coir) was soaked in solution for 4 h. The soaked fibres were then taken out, and rinsed with 0.01 mol/L dilute acetic acid solution for 30 min. The fibres were then washed with distilled water and air dried at 23°C±1°C for 48 h.

In the second step, the alkaline treated fibres were infused with MMT clays by shear induced mechanical mixing process. Initially 750 mL of distilled water was taken in a beaker, placed on a temperature controlled magnetic stirrer (Heidolph MR Hei: Standard, Labotec, South Africa) and then heated to 80°C. Ten grams of clays was then introduced into the water medium and the clay/water solution set at 80°C was stirred for 30 min at 500 rpm. Thereafter, 10 g of short alkaline treated fibres (sisal, banana, kenaf and coir) were introduced into the clay/water solution (80°C) and stirred for 4h at 500 rpm. The stirred fibre-clay solution was then placed in an ultrasonic agitation bath (MRC laboratory Equipment, UK. Model: DC-150H, operating at 40 kHz with ultrasonic power 150W) for 1 h. The nanoclay treated fibres

were then removed from the ultrasonic bath and air dried for 48 h.

2.3 Characterization

The longitudinal and cross-sectional surface morphologies of all untreated and nanoclay infused fibre series (sisal, banana, kenaf and coir) were analyzed using scanning electron microscope (SEM). The cross-sectional surfaces of fibre specimens were prepared using cryo-fracture method. In this method, the fibre was dipped into a liquid nitrogen bath medium, then taken out from bath and immediately cut along the cross-section. The longitudinal surface analysis was carried out as such without any type of specimen preparation. The fibre surface was analyzed using Zeiss Environmental SEM (ESEM: model EVO HD 15 operating at controlled atmospheric conditions at 20 kV). Before carrying out SEM studies, all specimens were gold sputter coated using Quorum-150R ES model thin film coating equipment.

X-ray diffraction (XRD) was performed on MMT clay and on the untreated and nanoclay infused fibre series, to study the phase morphology, crystalline index (CI) and presence of nanoclay onto the treated fibre surface. XRD was carried out using Philips PW1050 diffractometer at a scanning rate of 0.5°/min with CuK α radiation ($\lambda = 1.5401 \text{ \AA}$) operating at 30 kV and 15 mA. In measuring the clay content, the weight of treated fibre and Cloisite MMT clays were kept same for all XRD measurements.

The chemical constituents (α – cellulose, hemicellulose and lignin) of untreated and nanoclay infused fibres were determined using chemical methods as reported elsewhere²⁹⁻³¹.

2.4 Barrier Properties

2.4.1 Adsorption-desorption

The sorption of water by fibres at various vapor pressures (or relative humidity) was determined gravimetrically using an analytical balance (Gunt Hamburg, ET 605 model), at 23 ± 0.3°C. Five milligrams of fibres were placed in the chamber and relative humidity (RH) was controlled by steam humidifier. At the start of each moisture sorption cycle, the fibres were dried at 0% RH until the weight change was stabilized to be less than 0.1%. After stabilization, the moisture adsorption cycle was started and the humidity was increased stepwise, with steps of 10% RH. It was observed that the equilibrium moisture content (EMC) weight occurred from 4th to 15th day, depending upon the

fibre type. After this period, the fibres were in equilibrium with atmosphere. The desorption isotherm, from 95 RH to 5% RH was also recorded. The RH at 0% and 100% were extrapolated based on the trend of the sigmoidal sorption-desorption curve. The adsorption-desorption curves form a hysteresis loop, and were measured by degree of hysteresis (dH), as per the following equation at various RH levels:

$$dH = \frac{EMC_{desorption} - EMC_{adsorption}}{EMC_{adsorption}} \times 100 \quad \dots (1)$$

where $EMC_{desorption}$ and $EMC_{adsorption}$ are the equilibrium moisture content during desorption and adsorption respectively.

The specific surface area of untreated and nanoclay infused fibre samples was measured by BET (Brunauer, Emmett and Teller) method, using a particle analytical (Gemini V Micromeritics 2020 series) analyzer, and nitrogen gas purging.

2.4.2 Absorption

Water absorption test of all the fibre series was conducted at $23^{\circ}C \pm 1^{\circ}C$ as per ASTM D570-98(2005) test procedure. Initially three fibre specimens of 5 cm length from each series were chosen and then oven dried at $60^{\circ}C$ for 4 h to remove moisture content in the sample. The samples were dried and immediately fully dipped into a distilled water bath. The water dipped fibre samples were taken at different times, wiped with a paper towel to remove surface water, and weighed with an electronic balance until the increase in weight of water attained equilibrium level (i.e. no more or negligible water uptake by test sample). The mole per cent (mol. %) of water uptake (Q_t) was calculated from the water mass uptake by the sample, using the following equation :

$$Q_t = \frac{M_t(W)/M_r(W)}{M_i(S)} \times 100 \quad \dots (2)$$

where $M_t(W)$ is the mass of water uptake at a given time 't'; $M_r(W)$, the relative molecular mass of water; and $M_i(S)$, the initial mass of the sample.

The swelling property of fibre was examined by measuring the change in diameter of the fibre before and after water immersion. The % swelling diameter (D_s) of the fibre sample was calculated using the following equation equation:

$$D_s = \frac{\text{Diameter after equilibrium water absorption}}{\text{Diameter before water immersion}} \times 100 \quad \dots(3)$$

Three samples were used for measuring average D_s .

3 Results and Discussion

3.1 Structure and Morphology

Figure 1 shows the XRD pattern of untreated fibre series, nanoclay and nanoclay infused fibre series. All untreated fibres show two broad peaks at 2θ value of $\sim 15^{\circ}$ and $\sim 25^{\circ}$ values. These diffraction peaks are due to the cellulosic crystalline phase of fibre, which corresponds to 110 and 020 reflection planes of α -cellulose crystalline planes³². Besides this crystalline cellulosic phase, each fibre consists of non-cellulosic phases, such as lignin, wax and amorphous polymer at various levels based on their fibre characteristics³³. Nanoclay infused fibres show three broad diffraction peaks. Two peaks are similar to that of cellulose phase of the corresponding fibre and the third peak occurs at the 2θ reflection angle (7.58°) as that of MMT clay. The presence of clay peak in XRD spectrum suggests that the clay has been infused into the fibre. The clay content on the fibre is in the range of 2.7 - 3 wt.% for all the fibre series (Table 1). The clay content in the treated fibre was measured by taking the fraction of XRD peak intensity values of clay in treated fiber to that of peak intensity of neat MMT clay. Figure 1 also shows that the crystalline fraction of fibre increases due to reduction of amorphous area in XRD pattern. The crystalline fraction is measured by crystalline index (CI) (Table 1). The CI is measured by taking ratio of the areas of the amorphous (or crystalline) region to

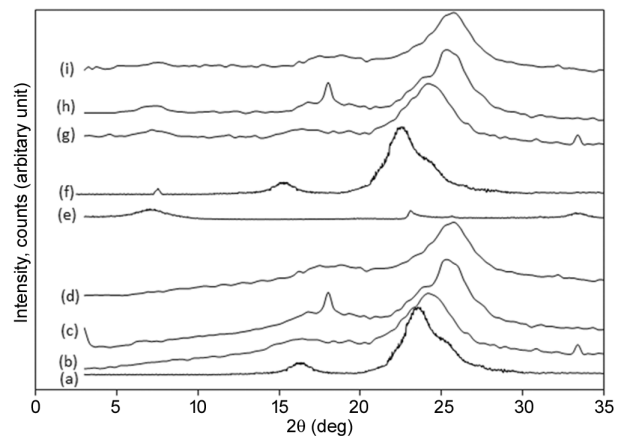


Fig. 1 — XRD pattern of untreated (a) sisal, (b) coir, (c) kenaf & (d) banana fibre; (e) MMT nanoclay; and nanoclay infused (f) sisal, (g) coir, (h) kenaf & (i) banana fibre

the total area of the XRD spectrum. The CI of nanoclay infused fibres is increased by ~ 14 - 42% in different fibres. The increased CI of nanoclay infused fibres may be attributed to the elimination of lignin and amorphous phases during NaOH/clay infusion treatment. The alkaline chemical used in nanoclay infusion treatment can eliminate lignin, hemicellulose and non-cellulosic phase of fibre¹⁶⁻¹⁸, leading to the increased CI values. The chemical constituents of untreated and nanoclay infused fibre series are measured, and the result are shown in (Table 1). The result shows the reduction in lignin and hemicellulosic phases after fibre treatment. The extent of lignin removal depends on the concentration and duration of chemical treatment. Almost complete removal of lignin and hemicellulosic phases has also been reported elsewhere³⁰.

The morphology of untreated and nanoclay infused fibres has already been examined by SEM and reported in our earlier studies^{21,25}. However, selected SEM images of cross-sectional and longitudinal morphologies of untreated and nanoclay infused coir fibres are shown in Fig. 2. The cross-sectional image of untreated fibre [Fig. 2(a)] shows the spherical shaped lumen phase in the cell wall structure of micro fibril. The dark spherical phase is the lumen (arrow 1) and bright phase (arrow 2) is the cell wall phase of the micro fibril. The purpose of selecting these four lignocellulosic fibres is based on the geometrical arrangement of lumen phase in the micro fibril and varying content of cellulosic phase among these fibres. The size and number of lumen phases in these fibres are found different. This different size and number of lumen phase affects the sorption behavior of fibres. Moreover, the concentration of cellulosic/non-cellulosic phases also contributes to the sorption behavior of fibres. Hence, the sorption behaviour of these different lumen phase of fibers has been examined in this work. Sisal, kenaf and banana fibres consist of coarse lumen phase with almost the same lumen size (diameter) as observed from earlier SEM study. However, sisal fibre has only one lumen phase, followed

by few in kenaf fibre and more in banana fibre. On an average, banana fibre has ~ 3 - 20 more lumen phases in a micro fibril than that of kenaf. Also, the size of lumen phase varies from ~ 3 μ m to ~ 30 μ m within a micro fibril. The coir fibre has a finer lumen phase with highest numbers among all the fibres. The cell wall phase resembles a composite type of structure, which consists of cellulosic and non-cellulosic fibrous structure embedded into a lignin matrix. The amount of these cell wall phases also varies among the micro fibril³⁴. Added to this, the size (cross-section) of micro fibril itself is not consistent within a plant fibre type, and varied in the order of ~ 10¹ - 10² μ m^{35,36}. This results in difficulty when analyzing the fibres due to large scattering of experimental results and this study also addresses these issues and finds an optimized effect of sorption on natural fibres.

The nanoclay infused fibre shows a different type of cross-sectional morphology [Fig. 2(b)]. The micro fibril appears to be elongated and stretched with irregular lumen morphology. The lumen phase is either folded, elliptical, elongated or distorted from its original spherical morphology. This change in micro fibril and lumen phase could be due to the shear induced force generated during the nanoclay infusion process. The nanoclay is infused using shear mixing, and this might have generated force on the fibre surface. This force could have strained the fibre surface, and hence the lumen phase appears distorted. The other possible reason could be dissociation of lignin and other non-cellulosic phase from cell-wall backbone during chemical treatment (NaOH) which might have distorted lumen phase. Figures 2(c) and (d) show the longitudinal morphology of untreated and nanoclay infused coir fibre surface. The untreated fibre [Fig. 2(c)] show several discontinuous phase (arrow 3) embedded on the continuous phase (arrow 4). This discontinuous phase could be from non-cellulosic phases such as hemicellulose and lignin. This discontinuous phase is found absent in the nanoclay infused coir fiber [Fig. 2(d)] and also other

Table 1 — Chemical and physical characteristics of untreated and nanoclay infused fibre series

Fibre	Crystalline index, %		Clay content in treated fibre, wt. %	α -cellulose, wt. %		Hemicellulose, wt. %		Lignin, wt. %		BET surface area, m ² /g	
	Untreated	Nanoclay infused		Untreated	Nanoclay infused	Untreated	Nanoclay infused	Untreated	Nanoclay infused	Untreated	Nanoclay infused
Sisal	59	71	2.7 \pm 0.2	60	72	21	16	19	12	73	97
Kenaf	65	74	2.9 \pm 0.3	65	75	20	17	15	8	79	101
Banana	53	62	3.0 \pm 0.3	54	63	26	21	20	16	81	108
Coir	43	61	2.8 \pm 0.2	43	62	21	14	36	24	86	103

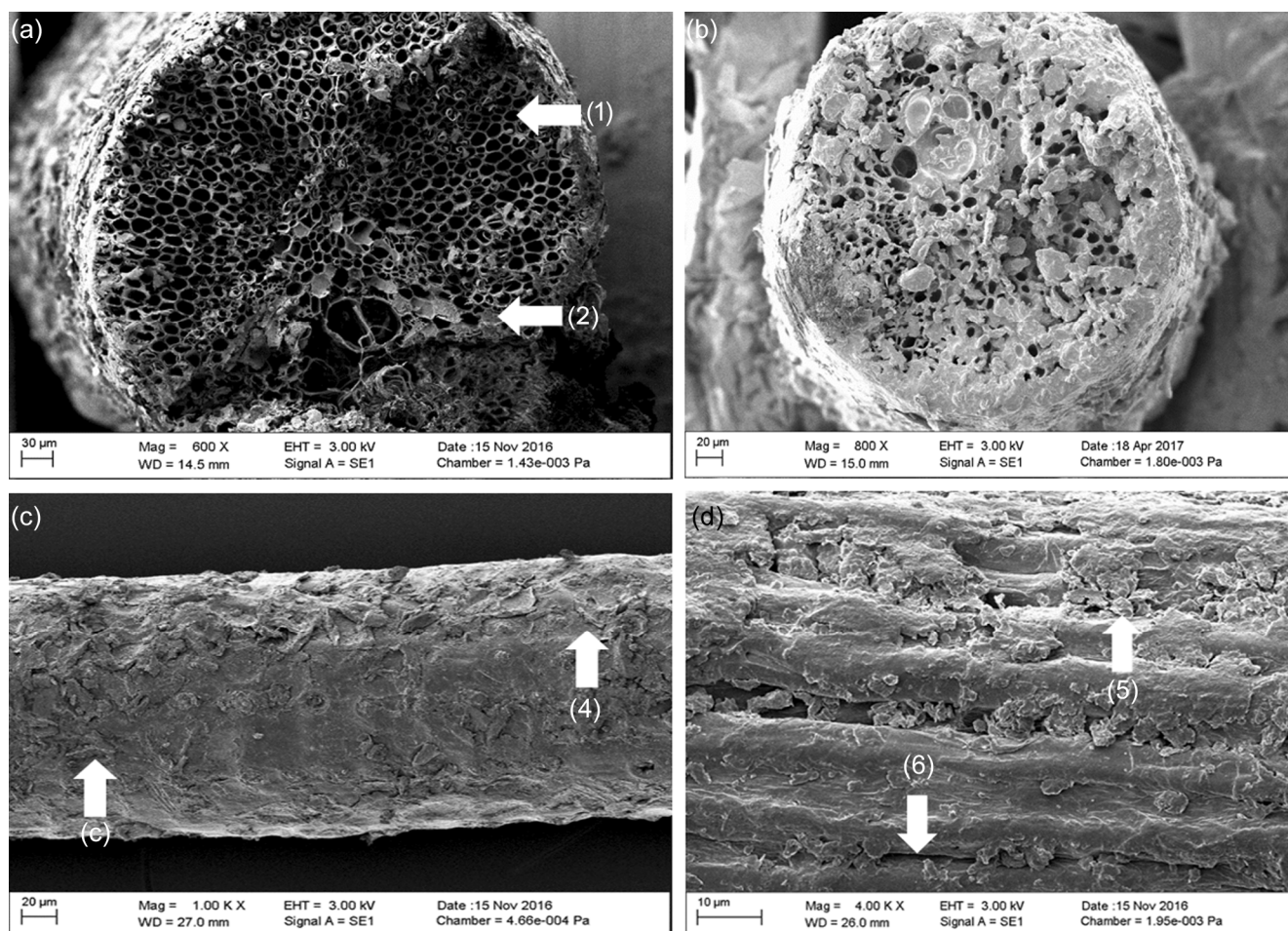


Fig. 2 — SEM images of coir fibre cross-sectional view (a) untreated and (b) nanoclay infused and longitudinal view (c) untreated and (d) nanoclay infused.

fibre series, suggesting the effective removal of non-cellulosic phase of the fibre. The nanoclay distribution is seen in the treated fibre (the nanoclay particles are indicated by arrow 5). The nanoclays are seen as spherical bright phase particles distributed uniformly on the fibre surface. The nanoclay may have bonded to fibre surface with hydroxyl group²², as shown below :



The longitudinal surface of treated fibre also shows fibrillation marks (arrow 6). Fibrillation is the process in which the packed untreated fibre gets split into small fibres after treatment. Fibrillation occurs in other nanoclay infused fibre series (banana, sisal and kenaf). It is reported^{21,24,25} that the chemical treatment and nanoclay infusion in the fibre leads to the fibrillation. The shear force

applied during the nanoclay infusion might have induced fibrillation of fibre. Fibrillation of fibre also increases cellulose content, surface area and CI. This, in turn, results in improved thermal, physical and mechanical properties of fibres, since cellulose is primary load bearing phase. Equation (4) also shows the removal of the hydroxyl group of clays. The nanolayers (or few nanolayers of clay tactoids) attach on the surface/sub-surface of fibres. These nanolayers of clay are an impermeable medium and protect fibre against sorption.

3.2 Adsorption

Adsorption may be defined as adhesion of atoms or molecules from a gas or liquid to a surface. Figure 3 shows adsorption curve of untreated sisal fibre at various pressure levels. Adsorption curve of sisal fibre and all other fibre series curves resembled, showing an S-shaped sigmoidal type isotherm curve. According to IUPAC classification, this is a type II-

degree of hysteresis (dH) values of all untreated and nanoclay infused fibre series. The result indicates dH values are higher in the untreated fibres. In particular, dH is higher in the fibres having more amorphous content (coir and banana). The moisture easily occupies the amorphous region and changes the physical state of the material, resulting in increased dH ^{19, 20}. The nanoclay treated fibre results in decreased dH values as compared to untreated fibre. The dH values are almost equal for all nanoclay treated fibre series at the respective vapour pressures. The result also indicates that at lower and higher relative vapor pressures, the dH values are lower possibly due to the constrained physical medium, as the water vapor forms monolayer and condensation onto the fibre surface. The relatively smaller difference between the moisture content of adsorption and the desorption might also be a factor of decreased dH at lower and higher vapor moisture content. At low vapor pressure (< 15%), monolayer water adsorption takes place, whereas at an intermediate vapor pressure level from (15 - 70%), polylayer water adsorption forms. The polylayer water adsorption increases dH . Above 70% vapor pressure, the adsorption is primarily governed by condensation mechanisms^{19, 20}. The presence of nanoclay reduces the formation of polylayer moisture adsorption at the fibre surfaces at intermediate vapor pressure (RH) values, which results in reduced dH .

3.4 Absorption

Absorption is a physical or chemical process in which atoms or molecules enter into bulk phase of solid (or liquid, gas) material. Absorption differs from adsorption, as molecules undergoing absorption are taken by volume, and not by the surface (adsorption). Natural fibres are subjected to high water absorption due to their hydrophilic and swelling characteristics^{13, 40-43}. During water absorption, the natural fibres swells. The swelling of fibres results in dimensional change and affects the fibre and fibre-matrix adhesion. The influence of nanoclay on the absorption and swelling characteristics has been examined and the results are presented in Table 3. The swelling increase is also proportional to the coarse size and number of lumen phase, amorphous content and non-cellulosic phase of fibres. The swelling of nanoclay infused fibres is considerably reduced than that of corresponding untreated fibre series. The nanoclay infused fibre imparts dimensional stability against absorption and results in

reduced swelling. The effect of nanoclay infusion for reducing swelling on high lumen phases fibres (coir and banana) is higher than that of less lumen phase fibres (kenaf and sisal). In nanoclay infused fibre, hard nanolayers of clay would have protected the fibre from swelling by resisting the expansion of fibres, thereby maintaining dimensional stability.

The absorption of fibres is also measured by water uptake. The time at which water uptake becomes constant is called as an equilibrium water uptake [$M_e(W)$], i.e. no more or negligible increase of water mass by sample. The molar mass of water corresponding to $M_e(W)$ is called as Q_∞ . The equilibrium molar water uptake (Q_∞) values of nanoclay infused banana, coir, kenaf and sisal fibre are 42%, 42%, 36% and 18% lower than that of their corresponding untreated samples (Table 3). This reduced water mass uptake could be due to the presence of nanoclay at the surface/subsurface of the fibre phase. The free capillary movement of water molecules in the lumen phase may have also induced absorption. Since the lumen phase is distorted in the nanoclay infused fibres, the possibility of capillary movement of water molecules in lumen phase might have been restricted, resulting in reduced water mass uptake. Table 3 also shows that water uptake increase in fibre correlates to lumen structure and morphology. As banana and coir fibres have a large number of lumen and amorphous phase, the water uptake is higher. However, in sisal and kenaf, single or very few lumen phases are present, resulting in reduced capillary flow of water molecules into the fibre. The sorption values of fibre series are different among the fibres, suggesting that the water sorption

Table 3 — Transport properties of untreated and nanoclay infused fibre series

Fibre	Q_∞ mol %	Q_t mol %	Swelling % increase
Untreated			
Banana	0.25	0.16	24
Coir	0.22	0.19	23
Kenaf	0.18	0.15	16
Sisal	0.15	0.14	11
Nanoclay infused			
Banana	0.14	0.13	9
Coir	0.12	0.12	9
Kenaf	0.11	0.10	8
Sisal	0.11	0.11	9

is dependent on the fibre architecture and the nature of the sorption mechanism occurring in that fibre.

4 Conclusion

Sorption characteristics of selected lignocellulosic fibres have been examined in this work. The fibres are chosen based on their lumen phase size and distribution in the cell wall microfibril. The influence of this lumen phase and nanoclay particles infusion on sorption characteristics (adsorption, desorption and absorption) of the fibres are examined. Nanoclay treatment affects lumen phase, and increases fibrillation, surface area and crystallization. Nanoclay attaches onto the fibre surface with hydroxyl bonding, and serves as a barrier medium for moisture and water uptake. The adsorption-desorption result indicates that the moisture uptake forms a sigmoidal curve. The *sdH* values of fibres are reduced in nanoclay treated fibre due to the decreased polylayer water formation. The absorption result indicates reduced swelling and water uptake in the nanoclay infused samples, thereby showing induced dimensional stability to the fibres.

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