CONDUCTIVE KENAF/POLYANILINE SHEETS FOR ELECTROSTATIC DISSIPATION AND ELECTROMAGNETIC INTERFERENCE SHIELDING PACKAGING

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Received March 14, 2015

Electrically conductive shielding sheets made up of kenaf fiber and polyaniline (KF/PANI) were successfully prepared *via in situ* polymerization. It was found that the amount of PANI necessary to achieve electrical conductivity transformation was as low as 5 wt%. Thus, the conductive sheet that included 5 wt% PANI in its composition showed rapid charge dissipation at 0.87 seconds and a moderate shielding effectiveness value of -6.1 dB. The mechanical properties of the obtained sheets were retained at a useable level as well. The investigation led to the conclusion that the achieved novel conductive sheet can find interesting applications as electrostatic charge dissipation (ESD) and electromagnetic interference (EMI) shielding material for protecting devices – applications that require materials with mechanical integrity.

Keywords: kenaf, polyaniline, conductive packaging, electromagnetic interference shielding, electrostatic dissipation, mechanical properties

INTRODUCTION

Electrostatic discharge phenomena (ESD) and electromagnetic interference (EMI) are the most undesirable side effects of the explosive growth of electronics and miniaturization of devices.[1,2] ESD arises due to static charge accumulation, which can create significant damages, especially in handling electrostatic sensitive devices, because of their susceptibility towards electrostatic discharge. Similarly, electronic device performance can be impaired by exposure to unwanted incoming or outgoing EM waves.[3-5]

Efforts to handle these issues include the use of plastic sheets with antistatic agents.[6,7] However, this approach has been unsuccessful in dissipating unwanted charge build-up, as plastics are not always electrically conductive. Adding to that, the conductivity of plastics can vary significantly with humidity due to the absorption of atmospheric moisture and salts of the antistatic agents.[8] Another earlier applied method consisted in adding conductive filler, such as carbon black and metallic powders or flakes, to a polymer matrix to make up a conductive composite. The downside however is that metallic powders can be oxidized over time and carbon black might cause contamination in clean room environments. An additional disadvantage of this approach is that high filler loading is needed for electrical percolation to happen.[9]

Polyaniline (PANI) is the most promising inherently conducting polymer due to its good environmental stability, high electrical conductivity, low cost and the ability to modulate its oxidation and protonation level for EMI and ESD applications.[10-13] However, the main drawbacks of PANI are its poor processability[14] and low mechanical properties.[15] Conventionally, the raw materials used for packaging or storing electrostatic sensitive devices are wood-based and electrically insulating. This can be solved by introducing conducting polymers on the surface of lignocellulosic fibers and processing them into packaging sheets.[16] However, simply spraying PANI on natural fiber sheets was found to be insufficient for ESD applications, as charge leaked when laid on a grounded surface.[17] On the other hand, *in situ* coating of natural fiber with PANI has recently gained wide attention for its antistatic, electromagnetic shielding, electrical conductivity and antibacterial properties with a view to

applications as papers and packaging products.[18-22] Though a desirable conductive sheet can be achieved by the addition of PANI, one of the drawbacks of such a combination is that the mechanical integrity of the processed sheet can be easily lost once the synthetic brittle conducting polymer is introduced.[23,24] It is targeted that the desired electrical conductivity be achieved at low PANI inclusion to avoid significant deterioration of mechanical properties.

Growing environmental awareness and depletion of the wood resources worldwide are among the vital factors that fostered the exploration of the potential of agro crops and lignocellulosic materials as alternative sources of fiber material. Kenaf (*Hibiscus cannabinus* L., *Malvaceae*) is a warm season annual fiber crop. The advantages of kenaf fiber (KF) include its low cost, rapid growth of the plant (4-5 months of harvesting time), specific tensile properties comparable to those of synthetic fibers and its high cellulose content.[25,26] Furthermore, kenaf produces an average of 17.8 tonnes fibers per hectare per year, a much higher yield compared to that of wood (2.2 tonnes per hectare per year).[27] Thus, kenaf fiber can be an attractive alternative raw material for pulp and packaging paperboard production.

Our earlier work was focused on the optimization of parameters to obtain the maximum achievable electrical conductivity and tear index of conductive PANI modified KF for paper making.[28] Therefore, it is important to evaluate the viability of the developed conductive paper for specific applications, such as electrostatic dissipation and electromagnetic interference shielding packaging. Cost-effective and environment friendly packaging materials that provide adequate protection for electrostatic sensitive devices from ESD and EMI must be an objective. Thus, the main aim of this study has been to prepare an ESD and EMI shielding material based on an electrically conductive KF/PANI sheet and to investigate its mechanical properties.

EXPERIMENTAL

Preparation of KF/PANI sheets

The kenaf bast fiber (KF) was supplied by Everise Crimson (M). The preparation of KF pulp was described in detail elsewhere.[29] The pulp was mechanically disintegrated by a three-bladed mixer for 2 min. *In situ* polymerization of aniline in the presence of KF pulp was performed in formic acid dopant solution of 11.0 *N* (Normality) (Merck). The required amount of aniline monomer (Acros) with respect to KF and oxidant (ammonium persulfate, Merck) was separately dissolved in the dopant solutions. The monomer to oxidant molar ratio was set to 1.5. The blended KF was initially immersed in the reaction mixture containing aniline solution, followed by the addition of oxidant solution to initiate the polymerization process. The temperature of the reaction mixture was maintained at 10 °C in an ice bath. The reaction medium was vigorously stirred with a mechanical stirrer for 4 h and was left for 24 h. The obtained KF/PANI pulp was then washed several times by deionized water until the suspension was clear from excess oxidants and a clear green suspension could be seen. The KF/PANI pulp was formed into sheets by hand, screened via a flat-plate screen with 0.15-mm slits, pressed for 4 min using a hydraulic press and further conditioned at 90 °C for at least 6 h. Following the above mentioned procedures, KF and KF/PANI sheets with varying PANI amount (1, 5 and 10 wt%) were prepared. The conductive sheets were named as follows: KF/1PANI, KF/5PANI and KF/10PANI, which stand for 1, 5 and 10 wt% of PANI, respectively.

Characterization methods

DC electrical conductivities of the prepared sheets were measured using a resistance meter (Advantest) by the four probe method under ambient conditions. Samples were dried for 4 h at 90 °C prior to testing to remove the adsorbed moisture and then stored in a desiccator filled with silica gel. The charge decay time was measured using a JCI unit. Briefly, the sample was sandwiched between two flat metal plates. First, one end of the sample was charged by a high corona discharge voltage (5000 V), using a charged plate monitor, then the other end of the sample was grounded and the voltage of the charged region was monitored as a function of time by the field-meter of the device.[30] Shielding effectiveness (SE_T) was measured using a vector network analyzer (Agilent Technologies) in the frequency range of 12-18 GHz with a power level of -5.0 dBm. The SE_T can be expressed by 10 $\log_{10}$ (P_t/P_i), where P_i and P_t are the magnitudes of incident and transmitted powers through the shield material. The tensile index of the sheet was assessed using an MIT tester according to TAPPI standards and reported as tensile strength (N) × 100/average grammage (g/m^2). Tear test was performed using the Elmendorf Tear method (ASTM D-1922). The tear index was calculated using the following relation: average tearing force (mN) / average grammage (g/m^2), whereby the average tearing force is given by (16 x 9.81 × average scale reading). Bursting test was performed on a Mullen type tester according to ASTM D774. The burst index was calculated by the expression: bursting strength (kPa) / average grammage (g/m^2). Folding endurance of the sheet was estimated using an MIT tester according to TAPPI standards and reported as number of times. Moisture absorption test was done on triplicate samples of 2 x 2 cm placed on top of a wire mesh in five different relative

humidity (RH) environments and conditioned in desiccators in accordance to ASTM E-104 standards. The RH was controlled using saturated salt solutions of LiCl (11%), KCH_3CO_2 (25%), $Mg(NO_3)_2$ (53%), NaCl (75%), and K_2SO_4 (97%). Samples were weighed at an interval of 6 hours until they reached saturation. The equilibrium moisture content at each water activity was calculated on a dry basis. The percentage of moisture absorption M_t (%) was calculated by the relation: $(W_w - W_d)/W_d \times 100$, where W_w and W_d are the weights of the sample before and after exposure to the controlled RH. The sheet morphology was examined using a Leo Supra 50VP scanning electron microscope (SEM). Fourier transform infrared (FTIR) spectra of the sample were obtained using a model 2000 Perkin Elmer spectrometer.

RESULTS AND DISCUSSION

Mechanical performance

The mechanical properties of the prepared sheets (tensile, tear, burst indexes and folding endurance) made from KF as cellulosic material and different concentrations of PANI are shown in Table 1. Briefly, the tensile index indicates the maximum tensile strength of the sheet before failure, the tear index measures the ability of the sheet to withstand tearing force and the burst index represents how much pressure the sheet can tolerate before rupture. The tensile, tear, and burst index values of the KF/1PANI and KF/5PANI sheets show no marked reduction in properties, as compared to those of the unmodified KF sheet. The strength of a cellulosic sheet with randomly oriented fiber is dependent on the strength of the individual fibers, as well as on the strength and number of bonds among them.[31] It was found earlier that the strength of individual kenaf fibers can be maintained even after modification with PANI.[32] Furthermore, the *in situ* polymerization of PANI during the pulping stage renders continuous PANI surroundings, which bonds together the fibers.[33] In this case, the intercalation of PANI between cellulosic fibers is moderate, reaching its conducting percolation state at around 5 wt%. It can be stated that the negative mechanical influence of PANI on the KF, at this particular concentration, is minimal. On the other hand, KF/10PANI revealed a significant reduction in tensile, tear and burst indexes. This is due to the high intercalation and percolation of the brittle PANI. The presence of a high amount of PANI will eventually lead to weak spots, which during the mechanical test will result in fiber rupture induced by the highly doped conductive coating, thus a sharp fall in mechanical properties will occur. The folding endurance test measures the amount of folding that a sheet will endure before its strength falls below a standard value of one kilogram force. The test measures a combination of tensile strength, stretch, and fatigue properties. Contrary to the changes in tensile, tear and burst indexes, the folding endurance decreases nearly by 25% as a result of the modification by only 1 wt% of conducting polymer. At a moderate level of *in situ* coating (1 and 5 wt%), the treated sheets still retain a certain degree of flexibility. The sheets with 10 wt% of PANI recorded a folding endurance of 15, indicating extreme sheet brittleness. The test suggests that the conductive sheet KF/5PANI can be utilized for end uses that require repeated folding durability or resistance to wear over a prolonged period.

Table 1
Mechanical properties of KF and KF/PANI sheets

Sample	Tensile index (Nm/g)	Tear index (mNm^2/g)	Burst index ($kPa \cdot m^2/g$)	Folding endurance (times)
KF	56.1 ± 0.8	22 ± 0.4	7.0 ± 0.6	410 ± 8
KF/1PANI	46.0 ± 0.3	20.5 ± 1.2	6.4 ± 0.9	301 ± 12
KF/5PANI	43.2 ± 0.3	20.9 ± 1.4	5.7 ± 0.8	296 ± 10
KF/10PANI	21.7 ± 1.5	12 ± 0.7	1.1 ± 0.5	15 ± 5

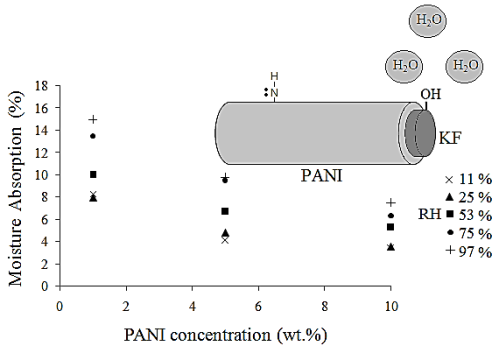

Figure 1: Moisture absorption of KF/PANI sheets with varying PANI content

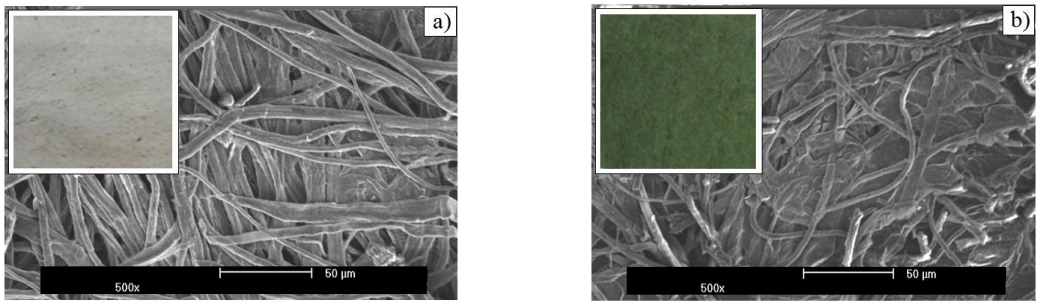

Figure 2: SEM images of a) KF and b) KF/5PANI sheets

Moisture absorption

Figure 1 presents the moisture uptake of the unmodified and modified KF sheets. The percentage of moisture absorption of the unmodified KF sheet is around 17% at an RH of 53%, and reaches up to 21% at an RH of 97% (not shown in graph). It can be seen from Figure 1 that the PANI modified KF samples exhibit progressive reduction of moisture absorption with increasing PANI content. This reduction can be explained by the hydrophobic features of the PANI component that surrounds the KF surfaces. In addition, the moisture absorption of all KF/PANI samples increases with an increasing RH, but is still much lower than that of unmodified KF at all levels of RH. The interaction between water molecules and the KF/PANI is shown in the insert of Figure 1. The compact manner in which the PANI is bound to the KF enables it to act as a barrier and prevent the contact between water molecules and KF surface hydroxyl groups.[33] Such modification could reduce the degree of water uptake, which in turn will preserve the mechanical properties and the ESD/EMI criteria of the conductive sheets.

Morphological analysis and FTIR

Figure 2a presents the electron micrograph of the KF sheet, including its photograph. The sample exhibits typical surface features of lignocellulosic fibers. Figure 2b reveals that after the *in situ* modification of the KF with PANI, the fibrous sheet morphology is still intact and no fiber rupture or damage can be observed. This is a positive indication of the feasibility of the applied method, which thus makes it possible to obtain conductive sheets, while preserving fiber characteristics. The PANI provides conductivity to the sheet due to percolating conducting PANI domains, which is preferable for antistatic and EMI shielding applications. The corresponding photograph in Figure 2b shows the green color of the sheet, which essentially indicates the presence of the PANI component in its doped state.

The FTIR spectrum of the KF sheet in Figure 3a exhibits the OH band of free cellulose, that of kenaf at 3400 cm^{-1} and a C-H stretching peak at 2900 cm^{-1}. The peak at 1734 cm^{-1} represents the acetyl group of hemicellulose and the ester linkage of lignin. The 1734 and 1233 cm^{-1} peaks are assigned to the C=O and C-O stretching of the acetyl groups after fiber treatment. The FTIR spectrum of the KF/5PANI sheet sample (Fig. 3b) clearly shows the presence of benzoid at 1480 cm^{-1} and quinoid ring vibrations at 1560 cm^{-1}, indicating the oxidation state of the emaraldine salt of PANI. The strong band around 1140 cm^{-1} is the characteristic peak of PANI conductivity and serves as the measure of the degree of delocalization of electrons.[34] The absence of the C-H stretching peak at 2900 cm^{-1} might

suggest that the surface hydroxyl groups of the KF have been dominated by the PANI component. This can be associated with the overlapping of the C-H stretching of KF by the broad absorption of PANI N-H stretching. Furthermore, the spectrum shows no significant band for C=O stretching. Presumably, an interaction between π electron clouds of carbonyl and benzoid/quinoid has taken place.[29] This can be due to the dopant ions that act as bifunctional molecules: 1) acting as charge moieties to promote electron conduction to the PANI backbone and 2) forming π interactions with the acetyl groups of KF. The interactions between components are depicted in the insert of Figure 3.

DC conductivity

The results obtained for the DC conductivity, charge decay time and shielding effectiveness of the sheet samples are given in Table 2. The room temperature electrical conductivity of uncoated KF sheet was measured in the range of 10^{-13} S·cm^{-1}. The conductivity of KF/1PANI increases about 10^2 times, compared to that of uncoated KF. Even so, the sheet still remains at its insulating level. This signifies that the doped PANI component is still in its unpercolated state and no significant electrical pathways have been created at this particular concentration. Percolation or meeting of electrical junctions is an important requirement for charge transport[35] and, in this case, charge dissipation and EM shielding. KF/5PANI shows a leap in conductivity up to 10^{-3} S·cm^{-1}, indicating that a transformation of the sheet from an insulating state to a conducting one had occurred. This conductivity level conforms to the range specified by the Electronic Industries Association (EIA) standards for ensuring safe, rapid and efficient dissipation of electrostatic charges. [12,30,36] At the percolation threshold concentration (5 wt% PANI), 3D networks of PANI junctures are formed throughout the sheets, thus creating electrical pathways. This electrical percolation is also assisted by the PANI coated surface fiber-fiber contact.[37] No further rise in conductivity was observed as the PANI concentration was increased to 10 wt%.

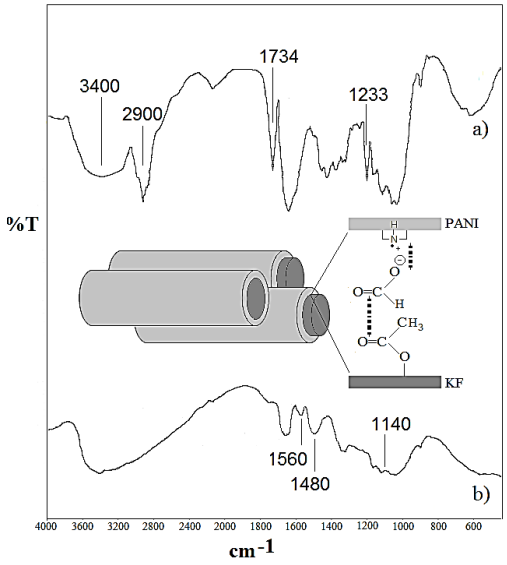

Figure 3: FTIR spectra of a) KF b) KF/5PANI

Table 2
DC conductivity, charge decay time and shielding effectiveness of KF and KF/PANI sheets

Sample	DC Conductivity, σ (S·cm^{-1})	Charge decay time, τ (s)	Shielding effectiveness, SE_T (dB)
KF	10^{-13}	4.12	-1.0
KF/1PANI	2.0 x 10^{-11}	2.65	-1.2
KF/5PANI	8.8 x 10^{-3}	0.87	-6.1
KF/10PANI	5.5 x 10^{-2}	0.94	-1.6

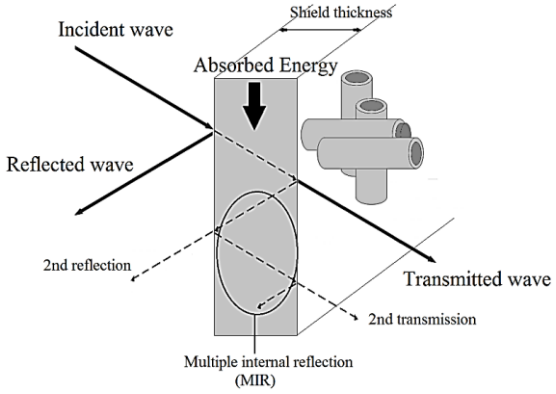

Figure 4: EMI shielding mechanism

Charge decay time

The measurement of antistatic charge decay time involves the application of corona voltage of 5000 V and recording the time required for the decay of a maximum accepted voltage to 10% of its initial strength, τ (10% cut-off limit).[12,30] An antistatic material suitable for any commercial applications should present a 10% cut-off decay time of less than 2 seconds.[38] The neat KF sheet exhibited a peak at 146 V, which indicates that the uncoated sheet accepted only 2.92% of the applied voltage of 5000 V. The accepted voltage (146 V) decayed to 10% cut-off in 4.12 seconds. This was higher than the limiting time of 2 seconds. Such a low dissipation rate can be caused by the inherent insulating nature of natural fiber, as can be observed from its corresponding conductivity level. The KF/1PANI sample accepted 106.8 V (out of 5000V), which decayed to 10% cut-off within 2.65 s. Though the charge acceptability and retention capability significantly decreased, the decay time of the sample was still high for ESD applications. This suggests that the amount of the PANI component in the KF sheet was not sufficient for carrying or mobilizing the charges efficiently. However, the PANI percolated sample (KF/5PANI) achieved a decay time of 0.87 s with maximum voltage acceptance of 45.4 V. The rapid charge dissipation and good antistatic response of the sample are due to the feasibility of the charges to move along the created conductive pathways of PANI junctions. The accepted voltage and decay time of KF/5PANI would ensure efficient ESD performance of the sheet under service conditions. The KF/10PANI showed no further significant enhancement compared to that of KF/5PANI, with a decay time of 0.94 at a voltage acceptance of 43.8 V.

Shielding effectiveness

A schematic representation of EMI shielding, showing reflection, absorption and multiple reflection steps, is illustrated in Figure 4. The higher the magnitude of the SE_T, the lesser energy passes through the sheet. KF and KF/1PANI show low SE_T values (Table 2), which can be attributed to their relatively low conductivities. KF/5PANI, on the other hand, reveals a more acceptable SE_T. The PANI component induces a favourable multiple reflection mechanism, which enhances the absorption. Furthermore, the adhered percolated PANI exhibits nano-sized fibril features, as reported earlier,[32] which promotes high surface/interface for multiple internal reflections, which, in turn, contributes towards cumulative absorption. The SE_T value of the KF/10PANI sheet reduced considerably, reaching those of uncoated KF and KF/1PANI. This can be explained by the fact that, though percolated 10 wt% of PANI is present in the sheet, the poor sheet formation, fiber rupture and relatively large inter-weave spaces caused by the high content of PANI reduce an effective absorption.

CONCLUSION

It has been demonstrated that the conductive KF/5PANI sheet achieved desirable electrical and mechanical properties. The results obtained for DC conductivity, charge decay time and SE_T showed good compliance with electronic device requirements and, additionally, the sheet presented no major reduction in mechanical properties (tensile, tear, burst indexes and folding endurance). This combination of properties is advantageous for end use. The moisture uptake of the conductive sheet was quite low compared to that of the unmodified sheet. Moreover, the SEM images revealed no fiber

damage incurred by the *in situ* modification. The vibrational analysis indicated the presence of the doped PANI component and good surface interaction between KF and PANI.

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