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Preparation and properties of HDPE/sugarcane bagasse cellulose composites obtained for thermokinetic mixer

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ABSTRACT

The use of natural fibers as reinforcement for thermoplastics has generated much interest due to their low cost, possibility of environmental protection and use of locally available renewable resources. In this work the mechanical and morphological properties of high density polyethylene/pre-treated and modified residues from sugarcane bagasse cellulose composites were analyzed. Composites were produced by a thermokinetic mixer. The microstructural analyses of fracture surface from composites can be easily evaluated by microscopic techniques. Results showed that the modification of sugarcane bagasse cellulose with zirconium oxychloride was successfully accomplished and that this reinforcement material with high density polyethylene showed tensile strength higher than non-modified sugarcane bagasse cellulose. Modification in the sugarcane bagasse cellulose influenced directly in mechanical properties of the composite material. This can be observed by the fracture surface, which showed that modified cellulose sugarcane bagasse improved interfacial adhesion between fiber and matrix.

1. Introduction

Sugarcane bagasse, an abundant agricultural lignocellulosic byproduct is a fibrous residue of cane stalks left over after crushing and extraction process of the juice from sugarcane. About 54 million dry tons of bagasse is produced annually throughout the world (Satyanarayana, Guimarães, & Wypych, 2008).

The utilization of this biomass for processing of novel composites has attracted growing interest because of ecological and renewable nature characteristic. Indeed enormous interest in development of new composite materials filled with natural fibers has been shown by important industries such as automotive, construction or packaging industry (Bhattacharya, Germinario, & Winter, 2008; Garg, Kaur, Garg, & Sud, 2008; Luz, Gonçalves, & Del'Arco, 2007; Vilay, Mariatti, Mat Taib, & Todo, 2008).

Natural fibers have recently a widely attention of researchers due to advantages over traditional reinforcement materials such as glass fiber in terms of cost, density and specific properties. Others important advantages are biodegradability and nonabrasive attribute to natural fibers, besides readily available and specific properties when compared to traditional fibers used for reinforcements (Facca, Kortschot, & Yan, 2007; Liu et al., 2007).

Natural fibers compared to inorganic fibers, present some disadvantages as poor hydrophobic polymer matrix compatibility, tendency to form aggregates during processing and low resistance to moisture, which provide a potential reduction as reinforcement for polymers (Ma, Yu, & Kennedy, 2005). On the other hand, various treatments are being used to improve fibers/matrix compatibility (Corrales et al., 2007; Liu et al., 2008; Torres & Cubillas, 2005). This process is considered critical as development phase of these materials due to strong interfiber hydrogen bonding, which holds the fibers together. Methods for surface modification can be physical or chemical according to superficial modification approach of the fiber. Others frequently used treatments are bleaching, acetylation and alkali treatment (Karnitz et al., 2007; Liu et al., 2007; Liu et al., 2008).

Some treatments have been studied in order to promote the chemical modification of fibers surface, in this case hydroxyl groups characteristically very reactive and susceptible to chemical reactions, are substituted in direction to obtain an apolar surface. Non-polar groups inserted on fibers, provide a hydrophobic surface characteristic increasing the compatibility of thermoplastic matrices (Ma et al., 2005).

In the recent years, procedures for modification of metallic oxide coated cellulose fibers, Cell/M_xO_y, have been studied (Marques, Trindade, & Neto, 2006; Pavan, Francisco, Landers, & Gushikem, 2005). This type material have been used for specific

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applications: TiO_2 for bactericidal activities (Daoud, Xin, & Zhang, 2005), ZrO_2 for retention and analysis of Cr(VI) (Mulinari, Da Silva, & Da Silva, 2006), Nb_2O_5 for adsorption of phosphate ions (Pavan et al., 2005) and Al_2O_3 for adsorption of some metal halides from ethanol solution (Alfaya & Gushikem, 1999).

The experimental methodology of the fibers coating process depends on the form in which the cellulose is obtained, as fiber or membrane. As fiber form, the treatment of cellulose with a precursor reagent can be made in aqueous or non-aqueous solvent (Gushikem & Toledo, 1999).

Marques et al. (2006) studied titanium dioxide/cellulose nanocomposites prepared through the titanyl sulfate hydrolysis in acidic medium in the presence of cellulose fibers and observed that this material can be used as reinforced fibers in polymer matrix.

Among the agro-industrial residue diverse, sugarcane bagasse is detached to be a residue widely generated in high proportions and contains cellulose (46.0%), hemicellulose (24.5%), lignin (19.95%), fat and waxes (3.5%), ash (2.4%), silica (2.0%) and other elements (1.7%) (Sene, Converti, Felipi, & Zilli, 2002). Bagasse is a vegetable fiber mainly constituted by cellulose, that is a glucose-polymer with relatively high modulus, often found as fibrillar component of many naturally occurring composites (wood, sugarcane straw and bagasse) in association with lignin (Alonso, Garzone, & Cornacchia, 2007; Pandey, Soccol, Nigam, & Soccol, 2000).

Some research works were developed using thermoplastic polymers/sugarcane bagasse fibers, which showed excellent results in mechanical properties (Luz et al., 2007; Stael, Tavares, & D'Almeida, 2001; Vilay et al., 2008). In this paper, mechanical properties of HDPE composites obtained by thermokinetic mixer and injection molded processes will be evaluated employing sugarcane bagasse cellulose and sugarcane bagasse cellulose modified with zirconium oxychloride as reinforcement.

2. Methods

2.1. Materials description

Cellulose from sugarcane bagasse was obtained by pre-treatment with 10% sulfuric acid solution, followed by centrifugation, deslignification with 1% sodium hydroxide solution and bleaching with sodium chloride. Modified cellulose from sugarcane bagasse was obtained by dissolution of 2 g of zirconium oxychloride in 100 mL of aqueous hydrochloric acid solution ($0.5\ \text{mol L}^{-1}$), in which 5 g of cellulose were immersed in this solution. The material was precipitated with ammonium solution (1:3) at pH 10.0, under stirring, filtered under vacuum, exhaustively washed with distilled water for the complete removal of chloride ions (negative silver nitrate test) and dried at 50 °C for 24 h. The resulting material was designated as $\text{Cell/ZrO}_2\cdot n\text{H}_2\text{O}$ (Mulinari & Da Silva, 2008).

2.2. X-ray diffraction

The crystallinity of sugarcane bagasse cellulose fibers treated and non-treated was evaluated by X-ray diffraction. X-ray diffractograms were obtained in a Rich Seifert diffractometer model ISO-DEBYFEX1001. Conditions used were: radiation CuK_α, tension of 30 kV, current of 40 mA and 0.05 (2θ/5 s) scanning from values of 2θ it enters 10 to 70° (2θ).

2.3. FTIR characterization

FTIR allows the measurements of variations of sugarcane bagasse cellulose composition after chemical treatments. The chemical structure of sugarcane bagasse cellulose and sugarcane bagasse cellulose modified with zirconium oxychloride was evaluated by FTIR. FTIR spectra were obtained on an FTIR spectrophotometer (Perkin Elmer). The samples were prepared by mixing the materials and KBr in a proportion 1:200 (w/w). For all spectra, 16 scans were accumulated with a $4\ \text{cm}^{-1}$ resolution.

2.4. TG characterization

Thermal analysis (TG) was performed to understand the stability characteristics of the sugarcane bagasse cellulose and sugarcane bagasse cellulose modified with metallic oxide, which is important during the processing of the fibers into composites. Thermogravimetric fibers parameters were generated in a Shimadzu instrument model TGA-50. The experiments were carried out under continuous nitrogen flow and with a heating rate of $10\ ^\circ\text{C min}^{-1}$.

2.5. Composites preparation

Sugarcane bagasse cellulose and sugarcane bagasse cellulose modified with zirconium oxychloride were mixed with the polymeric matrix (HDPE) in a thermokinetic mixer, model MH-50H, with speed rate maintained at 5250 rpm, in which fibers were responsible for 5 wt% in the composition. After the mixture, composites were dried and ground in mill, model RONE.

2.6. Injection molding

Cellulose (10%)/HDPE composite, $\text{Cell/ZrO}_2\cdot n\text{H}_2\text{O}$ (10%)/HDPE composite and high density polyethylene (HDPE) were placed in an injector camera at 165 °C and $2\ ^\circ\text{C min}^{-1}$ heating rate. The melted material was injected in a required dimensions pre-warm mold (165 °C) to obtain tensile specimen.

2.7. Mechanical properties

The mechanical strength of sugarcane bagasse cellulose modified and non-modified reinforced HDPE composites was determined using an INSTRON universal-testing machine (model 8801). Tests were carried out according to ASTM standards D638 with $10\ \text{mm min}^{-1}$ crosshead speed. Tensile strength and modulus values are average results of five tested specimens.

2.8. Scanning electron microscopy

Specimens submitted to tensile tests were cut and the composite intact fracture surface was analyzed in JEOL JSM5310 scanning electron microscopy with tungsten filament operating at 20 kV, employing low vacuum technique and secondary electron detector.

3. Results and discussion

The incorporation of hydrous zirconium oxide on to sugarcane bagasse cellulose surface can be described by the reaction Eq. (1):

$$\begin{aligned}&\text{Cell} + \text{ZrOCl}_2.8\text{H}_2\text{O} + \text{HCl} + \text{NH}_4\text{OH}\\&\quad\rightarrow \text{Cell/ZrO}_2.n\text{H}_2\text{O} + \text{NH}_4\text{Cl}\end{aligned} \tag{1}$$

Fig. 1 shows the schematic reaction representation of the Lewis acid which the cellulose (Gushikem & Toledo, 1999). The reaction is carried out under anhydrous conditions in the first step because it is supposed that primarily a donor-acceptor bond is formed between $\text{ZrOCl}_2.8\text{H}_2\text{O}$ and the oxygen of the $\text{C}^1\text{–O–C}^5$ and $\text{C}^1\text{–O–C}^4$ bonds. The attack to the former oxide bond leads to a ring opening and provides a molecule rupture process in order to form micro fibers. Ammonium solution is a required mean to promote the chemical link of metallic oxide with fiber surfaces.

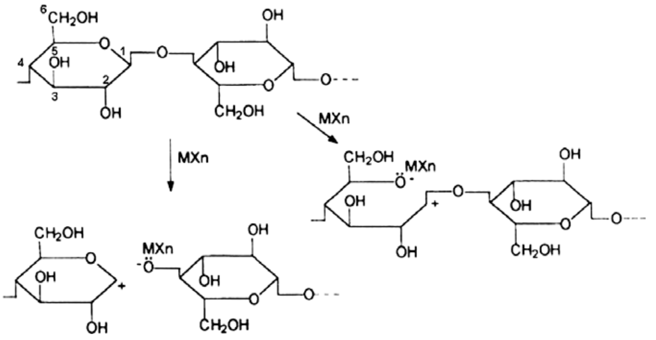

Fig. 1. Reaction of the Lewis acid with cellulose and formation of the donor-acceptor bond.

3.1. X-ray diffraction

Fig. 2 show diffractograms obtained for sugarcane bagasse cellulose fibers non-modified and modified. It possible observes a major diffraction peak for 2θ ranging between 22° and 23°, which corresponds to the cellulose (0 0 2) crystallographic planes.

X-ray diffraction peaks for both materials can be attributed to crystallinity scattering and the diffuse background to disordered regions. Materials tested are semicrystalline.

The spectrum corresponding to the non-modified sugarcane bagasse cellulose fibers shows diffraction peaks at the following 2θ angles: 15.7° and 22.82°. For modified sugarcane bagasse cellulose the same peaks can be observed at 15.9° and 22.82°. The presence of the peaks at 15° and 22° are an evidence of the modification on fiber. The position of these peaks indicates an increase of the interplanar distance in relation to the modified fiber. This behavior occurs due to the generation of disorder when fibers are modified. The projection substituting groups along the axis is associated with an increase in the interfibrillar distance (Rodrigues Filho et al., 2007).

Patterns for both materials are similar; however non-modified fiber is less crystalline than the modified one. The crystallinity index (CI), which is a measurement of the amount of crystalline cellulose with respect to the global amount of amorphous materials, was evaluated using Segal empirical method according to following Eq. (2):

$$\mathrm{CI}(\%) = \frac{(I_{002} - I_{\mathrm{am}})}{I_{002}} \times 100 \tag{2}$$

where I_{002} is the maximum intensity of the 002 lattice reflection of the cellulose I and I_{am} is the maximum intensity of X-ray scattering broad band due to the amorphous part of the sample. According to this method, non-modified and modified fiber presented 47% and 53% of crystallinity, respectively. These values can be attributed to the fibers modification.

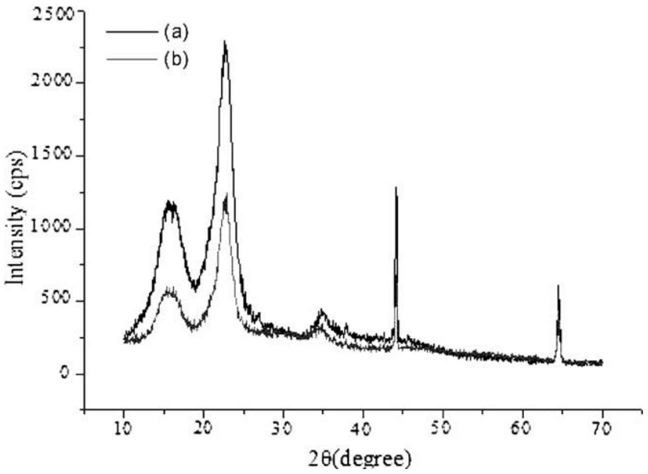

Fig. 2. X-ray patterns for sugarcane bagasse cellulose fibers non-modified (a) and (b) modified.

3.2. FTIR characterization

Table 1 summarizes higher bands observed in the FTIR spectrum of sugarcane bagasse cellulose and their assignments to chemical group vibrations and molecules.

Infrared spectra of sugarcane bagasse cellulose without and with modification are displayed in Fig. 3.

The most visible differences between the spectra of pure cellulose and Cell/$ZrO_2{\cdot}nH_2O$ are the modifications of the signal at 2885 and 1732 cm^{-1}, characteristics of the stretching of symmetrical CH groups and stretching of unconjugated CO groups present in polysaccharides and xylans. Considering the first region, the ratio between intensity of the C–H stretching band (~2900 cm^{-1}) is lower in the spectrum of the Cell/$ZrO_2{\cdot}nH_2O$ material than that observed for the pure cellulose. On the other hand, at the second region it may be observed modifications, especially in the ratio between the intensities of the C=O stretching band (~1730 cm^{-1}).

3.3. TG characterization

TG curves obtained for sugarcane bagasse cellulose and sugarcane bagasse cellulose modified with metallic oxide (Cell/$ZrO_2{\cdot}nH_2O$) at 10 °C min^{-1} are presented in Fig. 4.

Sugarcane bagasse cellulose presents two decomposition steps: the first at 300 °C and the second one at 380 °C. The first step can be assigned to the decomposition of fibers and second corresponds to complete temperature decomposition. The sugarcane bagasse cellulose modified with zirconium oxychloride (Cell/$ZrO_2{\cdot}nH_2O$) shows two different steps compared to cellulose: at 260 and

Table 1
Infrared main transitions for sugarcane bagasse cellulose

Wavenumber (cm^{-1})	Vibration	Source
3300	O–H linked shearing	Polysaccharides
2885	C–H symmetrical stretching	Polysaccharides
1732	C=O unconjugated stretching	Xylans
1650–1630	OH (water)	Water
1335	C–O aromatic ring	Cellulose
1162	C–O–C asymmetrical stretching	Cellulose
670	C–OH out-of-plane bending	Cellulose

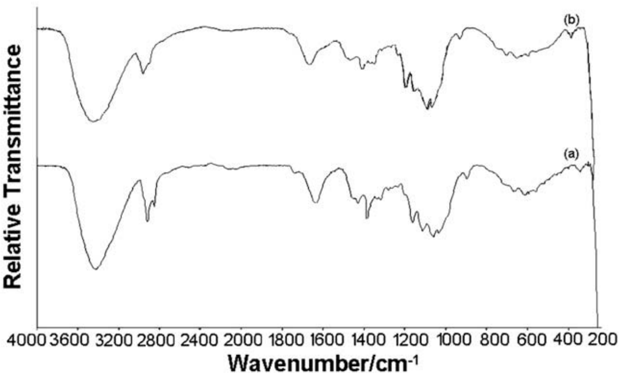

Fig. 3. FTIR spectra: (a) sugarcane bagasse cellulose and (b) sugarcane bagasse cellulose with zirconium oxychloride (Cell/$ZrO_2{\cdot}nH_2O$).

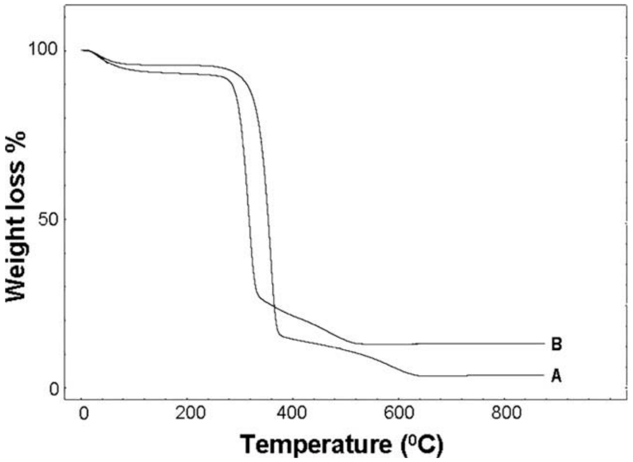

Fig. 4. TG curves: sugarcane bagasse cellulose fibers non-modified (A) and (B) modified.

338 °C. This progressive decrease in temperature in comparison to sugarcane bagasse cellulose can be attributed to the separation of the polymer chains caused by the dispersed oxide particles. Toledo, Gushikem, and Castro (2000) studied the antimony (III) oxide film on a cellulose fiber surface and observed similar results.

3.4. Mechanical properties

Mechanical properties of studied composites are summarized in Table 2. Modification of the sugarcane bagasse cellulose with hydrous zirconium oxide presented higher tensile strength results than without modification.

The amount of added reinforcement contributes to variation of the tensile modulus as indicated in Table 2. Fibers insertion can contribute to the modulus increase, because the Young's modulus of the fibers is higher than the thermoplastic modulus. However, to obtain a significant increase, a good interfacial bond between fiber and matrix is necessary. The tensile strength exhibited a good interaction between fiber and matrix, with increases of 24.1% and 31.8%, respectively, in relation to the high density polyethylene.

Cell/$ZrO_2 \cdot nH_2O$ (10%)/HDPE composite presents higher average values for tensile strength and tensile modulus in comparison to high density polyethylene. This increase in tensile modulus occurred as a consequence of the oxide treatment.

Experimental results in Table 2 may be explained by the interaction observed between fiber and matrix during the mixture process. The modified cellulose presented better tensile strength and adhesion between fiber and matrix than the non-modified cellulose, confirming that cellulose modification with zirconium oxychloride improves the adhesion between fiber and matrix.

Table 2
Mechanical properties of the materials obtained by injection molding

Materials	Elongation at break (%)	Tensile strength (MPa)	Tensile modulus (MPa)
High density polyethylene (HDPE)	2.20 ± 0.2	1.54 ± 0.13	732 ± 90.6
Cellulose (10%)/HDPE composite	1.74 ± 0.04	1.54 ± 0.13	897 ± 27.5
Cell/$ZrO_2 \cdot nH_2O$ (10%)/ HDPE composite	1.54 ± 0.13	18.2 ± 1.06	1233.1 ± 118.4

Reinforcement in wt.%.

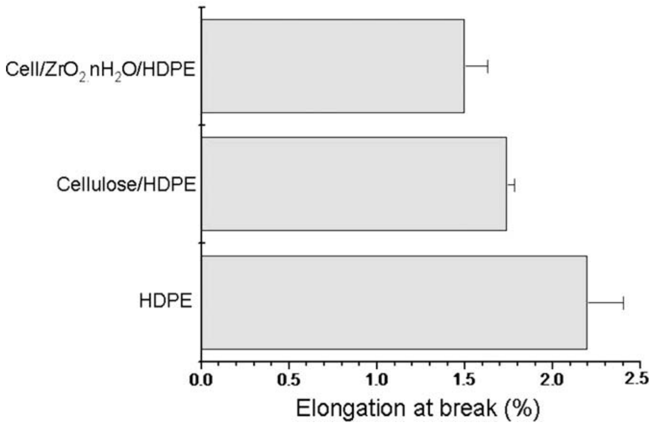

Fig. 5. Elongation at break (%) of composites specimens.

Composites presented lower elongation in comparison to the high density polyethylene (Fig. 5), which confirms the reinforcement in the matrix. However the reinforcement of the non-modified cellulose was not effective enough to increase resistance.

The reinforcement in matrix with cellulose modification presented lower elongation than non-modified cellulose, due to interfacial adhesion, confirming that fibers modification improves the fiber and matrix adhesion. This can be confirmed by the fracture surface analysis.

3.5. Scanning electron microscopy

SEM micrographs of the fractured surface for the composite specimens are shown in Fig. 6. It corresponds to the surface of fractured cellulose/HDPE composite (Fig. 6A) and Cell/$ZrO_2 \cdot nH_2O$/HDPE composite (Fig. 6B).

Without modification, obvious separation between HDPE and the cellulose fibers occurred, as shown in Fig. 6A, because of the incompatibility between hydrophobic matrix and hydrophilic fibers.

The modified cellulose improved significantly the compatibility between the matrix and fibers, and the matrix was well bonded to the fibers (Fig. 6B). The cellulose modification improved the compatibility between HDPE and fibers more than the non-modified cellulose.

In this way both, the modification of sugarcane bagasse cellulose and injection molding were adequate for thermoplastic composites preparation.

4. Conclusions

The feasibility of utilizing the agro-residue as alternative reinforcement in thermoplastics was studied. Chemical modification of sugarcane bagasse cellulose was studied to demonstrate the effect of modification on the mechanical properties of the composites and to study the practicability of processing these agro-residue with thermoplastics. The modification of sugarcane bagasse cellulose with zirconium oxychloride was successfully accomplished and it was verified that effectively improves the tensile strength in comparison to non-modified sugarcane bagasse cellulose.

The modification of sugarcane bagasse cellulose reduced the composites elongation 15% in comparison to non-modified sugarcane bagasse cellulose; on the other hand, the tensile modulus increased 38%. Thus agro-residue modification was adequate for thermoplastics composites preparation.

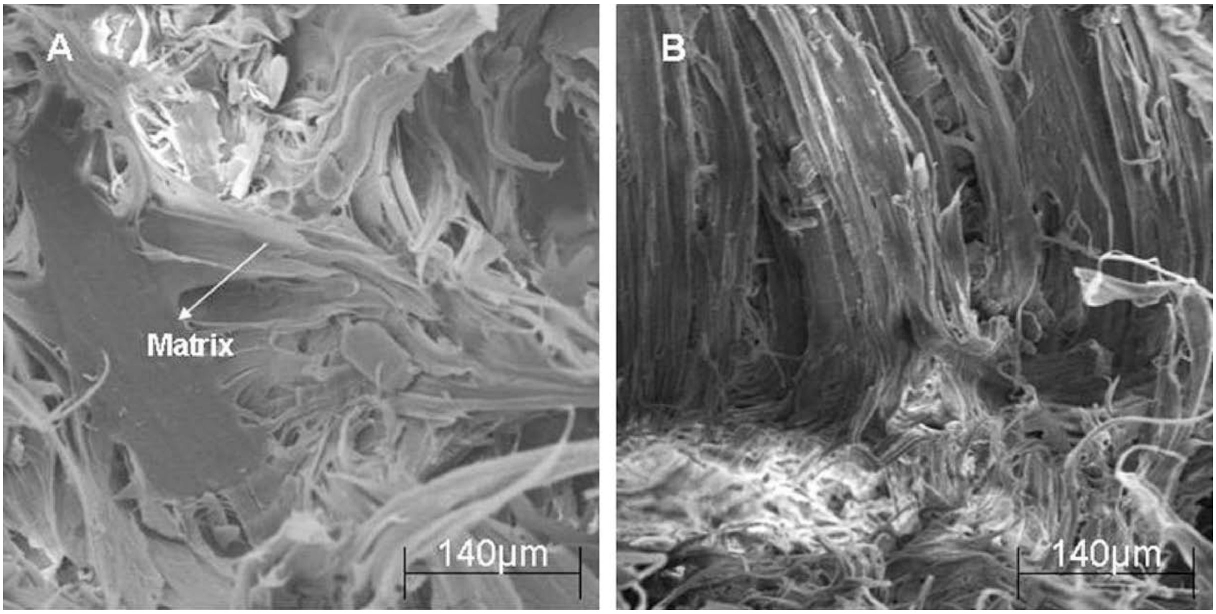

Fig. 6. SEM of fracture surface of composites: (A) Cellulose/HDPE; (B) Cell/$ZrO_2 \cdot nH_2O$/HDPE.

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