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**Processing and characterization of reinforced polyethylene composites made with  
lignocellulosic fibers from Egyptian agro-industrial residues**

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## ABSTRACT

Lignocellulosic fibers were extracted from Egyptian industrial crops, viz. cotton stalk, rice straw, bagasse, and banana plant waste. The chemical composition of these fibers was determined. Composite materials were processed from these natural lignocellulosic fibers using low density polyethylene and acid stearic as compatibilizer, or maleated low density polyethylene. The thermal and mechanical properties were studied by differential scanning calorimetry (DSC) and tensile tests, respectively. The morphology of processed composites was studied by scanning electronic microscopy (SEM). Better compatibility and enhanced mechanical properties were obtained when using maleated LDPE as compatibilizer. The chemical composition of fibers, in terms of lignin, cellulose and hemicelluloses contents, was found to have a strong influence on the mechanical properties of the composites.

KEYWORDS: Cellulose Fibres (A), polymer-matrix composites (A), fibre/matrix bond (B), interface (B)

## Introduction

Natural fibers are gaining progressive account as renewable, environmentally acceptable, and biodegradable starting material for industrial applications, technical textiles, composites, pulp and paper, as well as for civil engineering and building activities. Natural fibers reinforced composites combine acceptable mechanical properties with a low density. Such composites offer a number of well-known advantages which include low cost, availability of renewable natural resources, biodegradability, etc [1,2].

The utilization of biomass for the processing of novel composites has attracted growing interest because of its eco-friendly and renewable nature. Indeed enormous interest in the development of new composite materials filled with natural fibers has been shown by important industries such as the automotive, construction or packaging industry. Composites have encompassed almost all material domains. All synthetic polymers (thermoplastics, thermosets, and elastomers) can be used as matrices. As fillers, besides inorganic powders, extensive use has been made of inorganic man-made fibers such as glass, silicium carbide, or organic fibers like carbon and aramid, in the form of individual fibers (chopped or continuous), mats, or 2D-3D fabrics [3].

Compared to inorganic fibers, natural fibers present some well-known advantages such as lower density and lower price. They are less abrasive to the processing equipment, harmless, biodegradable, renewable, and their mechanical properties can be comparable to those of inorganic fibers [4-7].

Many studies have been developed based on composites containing natural lignocellulosic fibers from agro-industrial or agricultural residues. Depending on their origin, natural fibers can be grouped into bast (jute, flax, hemp, kenaf, mesta), leaf (pineapple, sisal, henequen, screw pine), and seed or fruit fibers (coir, cotton, oil palm) [8,9]. Cellulose is the main component of natural fibers, and the elementary unit of a cellulose macromolecule is anhydro-D-glucose, which contains three

hydroxyl (OH) groups. These hydroxyl groups form hydrogen bonds inside the macromolecule itself (intermolecular) and between other cellulose macromolecule (intermolecular). Therefore, all natural fibers are hydrophilic in nature.

However, the main disadvantage of natural fibers/plastic composites is the poor compatibility between the mainly hydrophobic polymer matrix and the hydrophilic fibers. This leads to the formation of a weak interface, which results in poor mechanical properties; where the stress transfer at the interface between two different phases is determined by the degree of adhesion. A strong adhesion at the interfaces is needed for an effective transfer of stress and load distribution throughout the interface [10-12]. Thus, in order to improve the mechanical properties of composites, an improvement of the compatibility between the fibers and the matrix has to be made, i.e. a third component, the so-called compatibilizer, has to be used or the fibers have to be surface modified prior to the preparation of the composites [12,13]. Formation of an interphase between the lignocellulosic phase and the polymer matrix phase, with polymer chains bonded onto fibers particles, is probably the best solution to the problem. For such a purpose, polyethylene (PE) was treated with maleic anhydride to form maleated polyethylene (MPE).

Egypt is a country with a large agricultural production and agricultural harvesting residues abundantly available in the River Delta of Northern Egypt constitute a bulky and wet resource of significant quantities. It is estimated that 23 million ton/year of agricultural residues result from cotton and rice. Beside that, there are wastes from sugar-cane and banana plant, which pose major disposal problems. The Egyptian Environmental Ministry most recent legislation regarding particulate matter discharge from burning, will force to dispose wastes by composting or land filling. Consequently, it is important to find uses for these waste materials. The potential to use lignocellulosic fibers extracted from these agricultural residues has been evaluated. Both PE and MPE; that can be bonded on cellulosic fibers under extrusion, were used as polymeric matrices for different lignocellulosic fibers extracted from

Egyptian agricultural wastes, i.e. cotton stalk, rice straw, bagasse, and banana plant waste. The influence of the type of matrix and the type and amount of the fibers on the mechanical properties of the prepared composites was analyzed.

## **Experimental**

### *Materials*

Low density polyethylene (LDPE) (Lacqtene 1008 FE 24, density  $0.924 \text{ g.cm}^{-3}$ ) and maleated low density polyethylene (MLDPE, Polybond 3109 with 0.8% maleic anhydride content, density  $0.926 \text{ g.cm}^{-3}$ ) were supplied by Atofina S.A. and Crompton Corporation, respectively. Stearic acid (98%) was purchased from Aldrich.

Cotton stalk, rice straw, sugarcane and banana fibers were collected from Egypt. These raw materials were obtained from the farmers after harvesting, i.e. we got the rice straw after harvesting the rice and the same for the cotton stalk we got it after harvesting the cotton and so on. The fibers used in this study were chemically characterized. The chemical characterization consisted in the determination of the carbohydrates, the Klason lignin and the ash content.

### *Fibers preparation*

All the fibers were ground for a few minutes in a mechanical grinder (Fristch Pulverisette, Germany) with speed 3000 rpm and sieved on sieves with various mesh sizes (18, 35 and 60 mesh) to produce homogeneous fractions.

### *Fiber length measurements*

The diameter and length of fibers were measured automatically with the MorFi LB-01 fiber analyzer (Techpap, France) by a computer analysis of images of the suspension flowing through a flat cell observed by a digital CCD video-camera.

### *Composites preparation*

The samples of fiber powders were extruded with the polymeric matrix at 75 rpm with a Micro 15-cc Twin Screw Compounder system (DSM Research, Geleen, The Netherlands) at 130°C for 10 min. The LDPE or MLDPE was first added to the mixer and after the thermoplastic matrix was melted, the fiber powders were added and the mixing maintained for additional 10 min. For LDPE based composites, stearic acid was added as compatibilizer (2% of weight of fibers content) with fibers. The resulting composites were chopped and then molded into films 0.5 mm thick, using compression molding at 130°C and 200 bar for few minutes.

#### *Differential Scanning Calorimetry (DSC)*

DSC experiments were performed using a DSC Q100 differential scanning calorimeter (TA Instruments, USA). Around 10 mg of samples were placed in a DSC cell in glove box. Each sample was heated from -100 to 200°C at a heating rate of 10°C min<sup>-1</sup>. The melting temperature  $T_m$  was taken as the peak temperature of the melting endotherm.

#### *Tensile tests*

The mechanical behavior of the composites prepared with different fibers was analyzed with an RSA3 (TA Instruments, USA) with a load cell of 100 N. Experiments were performed with a cross head speed of 10 mm.min<sup>-1</sup> at room temperature, 25°C. The sample dimensions were 10 × 5 × 0.25 mm<sup>3</sup>, and the results were averaged over five measurements.

#### *Scanning Electron Microscopy observations*

Scanning electron microscopy (SEM) was used to investigate the morphology of the different types of materials and the filler/matrix interface by using an ABT-55 microscope. The specimens were frozen under liquid nitrogen, fractured, mounted, coated with gold/palladium and observed using an applied tension of 10 kV.

## **Results and discussion**

### *Fibers characterization*

The chemical characterization revealed the proportion of each component of the fibers from agricultural residues. Results are collected in Table 1. Important differences were found depending on the different origin of the fibers, especially in regard to lignin, cellulose and minerals contents. Cotton stalk and banana plant waste fibers exhibited the highest lignin content, in contrast with the cellulose being the lowest. Comparing these further, these fibers were taken from the plant trunk, which presents high rigidity and hardness, indicating its high content of lignin. An important amount of cellulose and lower lignin content were observed for bagasse and rice straw fibers. One can observe a higher amount of ashes in banana plant waste and rice straw fibers showing their high content in minerals. The wax content was identical for the different fibers.

All the fibers were grounded and sieved with different sieves to produce homogenous fractions. The 18 mesh sizes retained fraction is the main fraction for all fibers. It was used to process the composites. Both the average diameter and length of all fibers were calculated using MorFi analysis. Results are reported in Table 2 and it was found that they were short fibers. The fiber length used in the preparation of composites is critical. It should not be too long, otherwise the fibers may get entangled with each other [14]. This will cause problems with fiber dispersion. If fibers are too short, the stress transfer area will be too small for the fibers to offer effective reinforcement. According to Derringer [14], commercially available fibers such as nylon, rayon, and polyester need to be cut into lengths, of approximately 0.4 mm for best dispersion. However, it is worth noting that values reported in Table 2 refer to the initial fiber length before extrusion and this initial fiber length will not be retained in the final composite. The fibers undergoes severe attrition in the extrusion barrel due to extreme shear and the fiber distribution is probably much shorter coming out of the extrusion machine.

*LDPE or MLDPE-based composites*

First, composite materials were prepared using either LDPE or MLDPE as matrix. For LDPE-based composites stearic acid was used as compatibilizer. It is known to improve the compatibilization between the hydrophobic LDPE matrix and hydrophilic fibers without any covalent chemical bonds between them. Raj and Kokta [15] investigated the influence of using various dispersing aids (stearic acid and mineral oil) and a coupling agent (maleated ethylene) in cellulose fiber reinforced polypropylene composites. Both the tensile strength and modulus of the composites studied were found to increase with the fiber content when either stearic acid or mineral oil (1 wt% by weight of fiber) were added as processing aids during the compounding. Stearic acid was found to perform better in improving the fiber dispersion compared to mineral oil. In contrast, anhydride maleic groups in the MLDPE act as coupling agents and covalent bonds can be created between fibers and matrix (Figure 1). Many studies were concerned with the effectiveness of maleic-anhydride-polypropylene copolymers as a coupling agent. Miecz et al. determined increased shear and tensile strengths of about 100% and 25% respectively for flax-polypropylene composites, when the coupling agent was applied to the flax fibres before the composite was processed [16-17]. The chemical bonding between the anhydride- and the hydroxyl groups caused a better stress transfer from the matrix into the fibres, leading to a higher tensile strength.

The thermal properties of both LDPE and MLDPE-based composites reinforced with each kind of fiber were investigated by DSC. The thermograms (not shown) obtained for the neat matrix, as well as for all fibers composites were conventional and very similar among them. The temperature position of the endothermal peak corresponding to the melting and degree of crystallinity of all composites obtained from DSC curves are given in Table 3. For neat matrices, it was found that both the melting point and degree of crystallinity were higher for MLDPE compared to LDPE. No significant change of the melting temperature was reported when adding lignocellulosic fibers

regardless the nature of the matrix. It remains roughly constant around 110-112°C and 121-124°C for LDPE- and MLDPE-based composites, respectively. The degree of crystallinity of MLDPE-based composites was roughly constant compared to the unfilled matrix. On the contrary, it seems to globally slightly increase when adding lignocellulosic fibers to the LDPE matrix.  $\chi_c$  values were found to be higher for cotton stalk-based materials compared with other fibers. This may be due to the high amount of lignin and hemicelluloses in this fiber, which are amorphous and display strong molecular interactions with the compatibilizer.

The evolution of the mechanical properties (Young's modulus, strength and elongation at break) as a function of fiber content for both sets of composites is shown in Figures 2, 3 and 4. The Young's modulus (Figure 2) of the unfilled matrix was found to be higher for MLDPE compared to LDPE as a consequence of its higher degree of crystallinity. It more or less continuously increased when adding lignocellulosic fibers, being independent on the nature of the matrix. However, it is found to remain higher for MLDPE-based composites compare to its LDPE-based counterparts because of the higher degree of crystallinity of the former. However, there is a slight increase in crystallinity and much of the increase is probably due to crosslinking and covalent adhesion of fiber to anhydride-modified matrix. The strength (Figure 3) clearly increased as the fiber content was increased in the case of MLDPE, and in contrast it tends to decrease in the case of LDPE, the value for the unfilled matrix being similar. These results show the beneficial effect of the copolymer and that using maleated PE certainly improved the tensile strength owing to better adhesion between the filler and matrix and better stress transfer from the matrix to the filler. The elongation at break (Figure 4) of the unfilled matrix is higher for LDPE than for MLDPE. This is most probably due to the lower degree of crystallinity of the former. Amorphous domains being in the rubbery state at room temperature are more able to undergo the strain. The elongation at break decreased upon fiber addition for both sets of composites regardless the nature of the fiber. The lignocellulosic fibers are

responsible for the decrease of the deformation capability. The PE matrix provides ductility whereas the lignocellulosic fibers exhibit brittle behavior with a subsequent loss of toughness of the composite material. The elongation at break mainly depends on the fiber content, even though MLDPE based composites show slightly higher values at high filler content.

The fractured surfaces of the composite materials filled with 30 wt% of lignocellulosic fibers were examined using SEM. Some other compositions were examined as well (i.e., 20 and 40 wt% fibers), but the results were found to be most apparent for the 30 wt% fiber content level. For LDPE-based composites (Figures 5A<sub>i</sub>) fiber pull-out as well as holes resulting from fiber delamination were observed regardless the nature of the fiber. The fractured surfaces of each composite revealed extensive interfacial delamination between the fiber and the matrix. It indicates that lignocellulosic fibers were hardly wetted by the LDPE matrix with a gap developing around the fibers. The fibers also stay intact after fracture. Pulled-out fibers and gaps are generally observed when the adhesion between fibers and matrix is not sufficient, and this suggests poor fiber-matrix compatibility. These observations are in agreement with the low strength values reported for these materials.

In contrast to LDPE-based composites, fibers in MLDPE-based composites were found to be uniformly coated by the matrix (Figures 5B<sub>i</sub>). This indicates that the fibers are more compatible with the MLDPE matrix. The fibers were also severely fractured after the fracture suggesting that the fibers did get strained up to their maximum stress values. This suggests improved interfacial adhesion between the fibers and the MLDPE matrix. This must be attributed to the covalent bonds created between the fiber and the matrix and which were ensured by the maleate groups.

#### *LDPE and MLDPE blends-based composites*

In order to improve the mechanical properties of LDPE-based composites, blends of LDPE and MLDPE, at different proportions, were used as matrix for composite materials reinforced with 30

wt% of lignocellulosic fibers. The thermal properties (melting point and degree of crystallinity) of resulting composites are reported in table 4. For each type of fiber, the melting point increased when increasing the MLDPE content. Compared to pure LDPE- or MLDPE-based composites, the degree of crystallinity of the composites obtained from the blend of these two polymers was found to be significantly lower. This is probably due to the disturbance of the crystalline structure. For each blend-based composite, the mechanical properties were estimated and the results are reported in Figure 6. The strength (Figure 6B) continuously increased as the MLDPE weight ratio in the matrix increased. This suggests improved interfacial adhesion between the fibers and the matrix which must be attributed to the covalent bonds created between the fiber and the MLDPE moieties. Except for the LDPE:MLDPE 0.75:0.25 blend-based composites, the Young's modulus also displays a continuous increase upon adding of MLDPE in the blend, whereas the elongation at break remains roughly constant. The LDPE:MLDPE 0.75:0.25 blend-based composites, low values of the Young's modulus and high values of the elongation at break were observed. These phenomena are very clear for the composites containing lignin-rich fibers, i.e. cotton stalk and banana plant waste (open diamond-shape and filled circles in Figures 6A and 6C). Lignin moieties, which are amorphous and behave like a co-compatible or plasticization agent, display strong molecular interactions with MLDPE and probably chemical bonds can be easily created and firstly between them.

### **Conclusion**

The potential to use lignocellulosic fibers extracted from Egyptian agricultural wastes, viz. cotton stalk, rice straw, bagasse, and banana plant waste has been evaluated. It was found that cotton stalk and banana plant waste fibers exhibited the highest lignin content, in contrast with the cellulose being the lowest. A high amount of minerals was reported for banana plant waste and rice straw fibers. After grounding, fibers around 300-500  $\mu\text{m}$  long and 40  $\mu\text{m}$  in diameter were used to process composite

materials using low density polyethylene (LDPE) or maleated LDPE (MLDPE) as matrix. The degree of crystallinity of LDPE-based composites was found to slightly increase when adding lignocellulosic fibers, mainly in the case of lignin- and hemicellulose-rich cotton stalk fibers. A significant increase of the strength was reported upon fibers addition when using MLDPE as matrix showing the beneficial effect of this copolymer as compatibilizer, as confirmed by microscopic observations;

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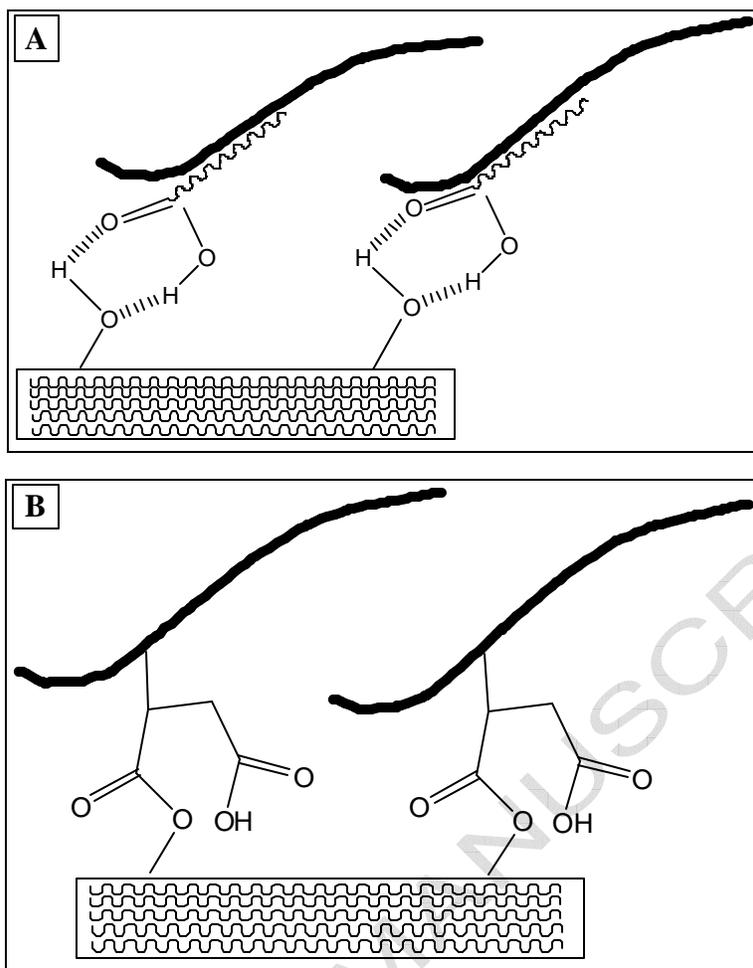


Figure 1: Adhesion mechanism for (A) LDPE-stearic acid and (B) MLDPE based composites reinforced with lignocellulosic fibers.

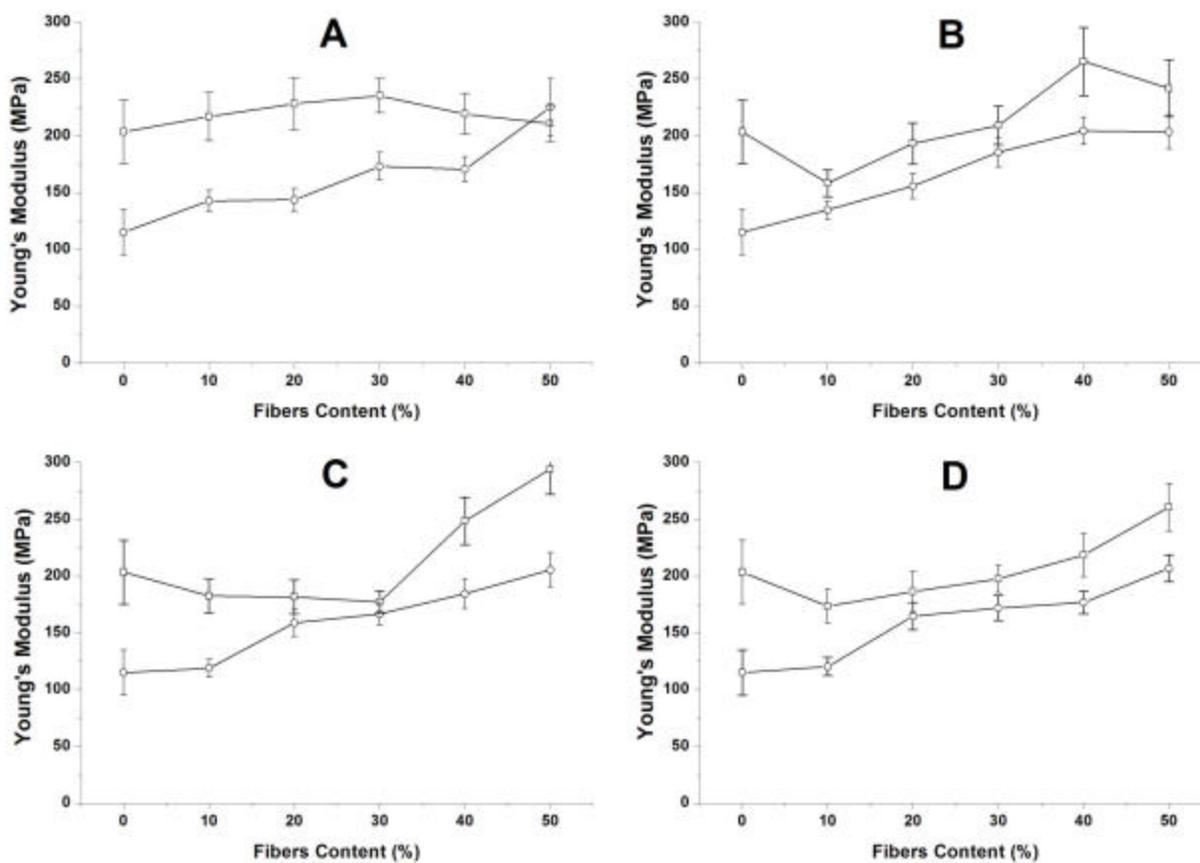


Figure 2. Young's Modulus versus fibers content for LDPE- (○) and MLDPE- (□) based composites: (A) Bagasse, (B) Banana, (C) Cotton Stalk and (D) Rice Straw

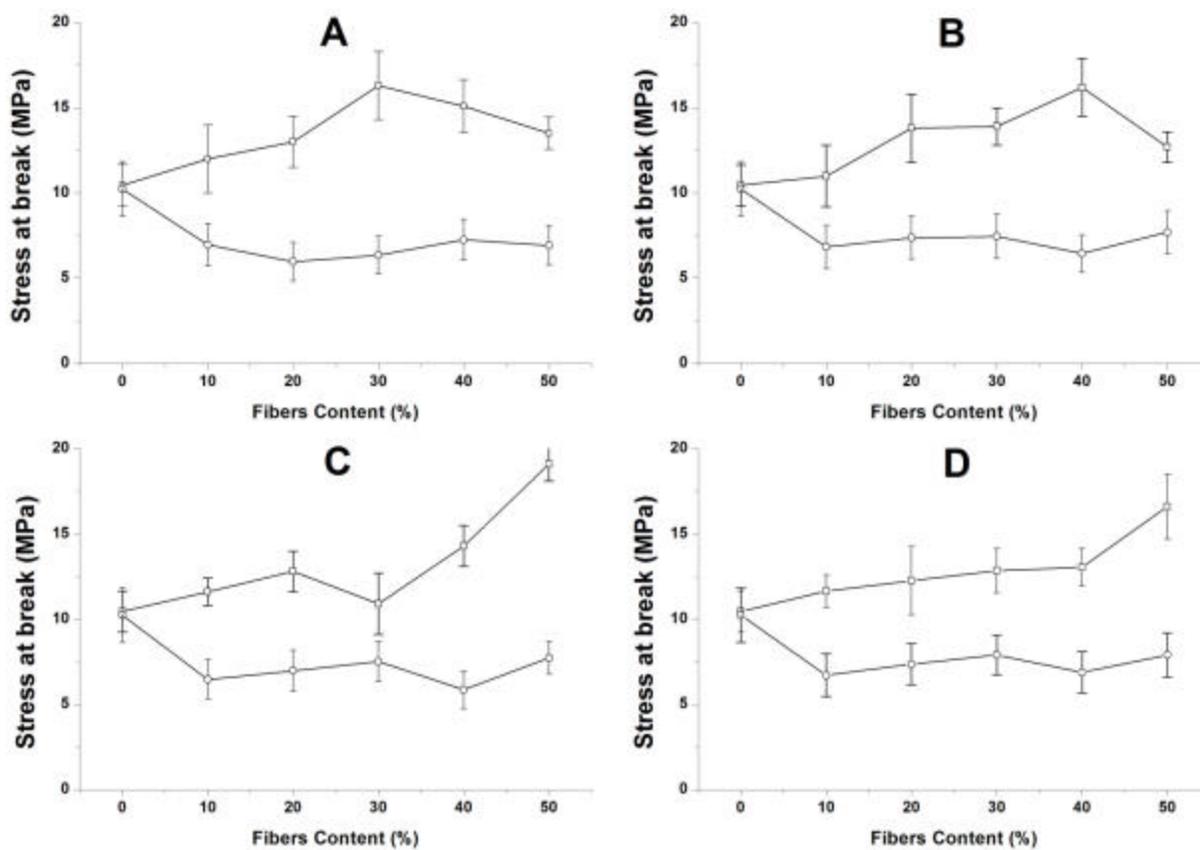


Figure 3. Strength versus fibers content for LDPE- (○) and MLDPE- (□) based composites: (A) Bagasse, (B) Banana, (C) Cotton Stalk and (D) Rice Straw

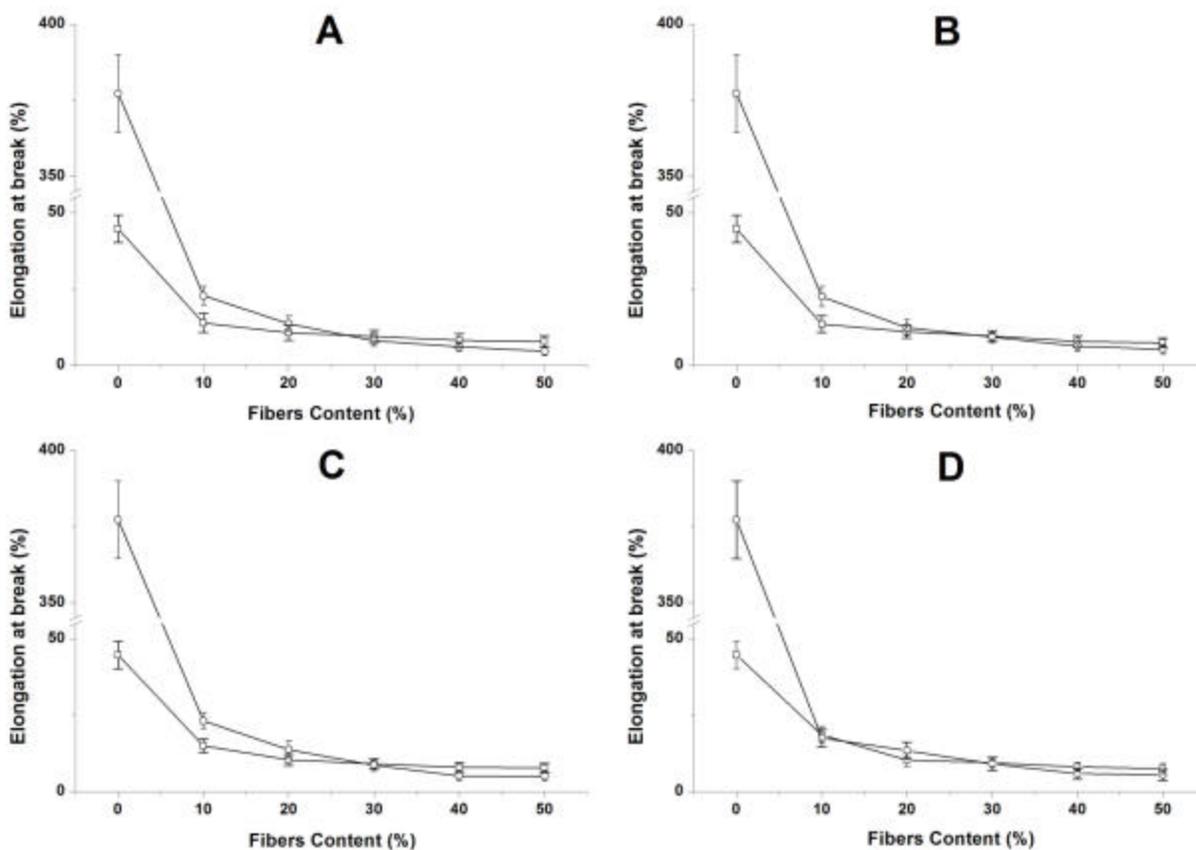


Figure 4. Elongation at break versus fibers content for LDPE- (○) and MLDPE- (□) based composites: (A) Bagasse, (B) Banana, (C) Cotton Stalk and (D) Rice Straw.

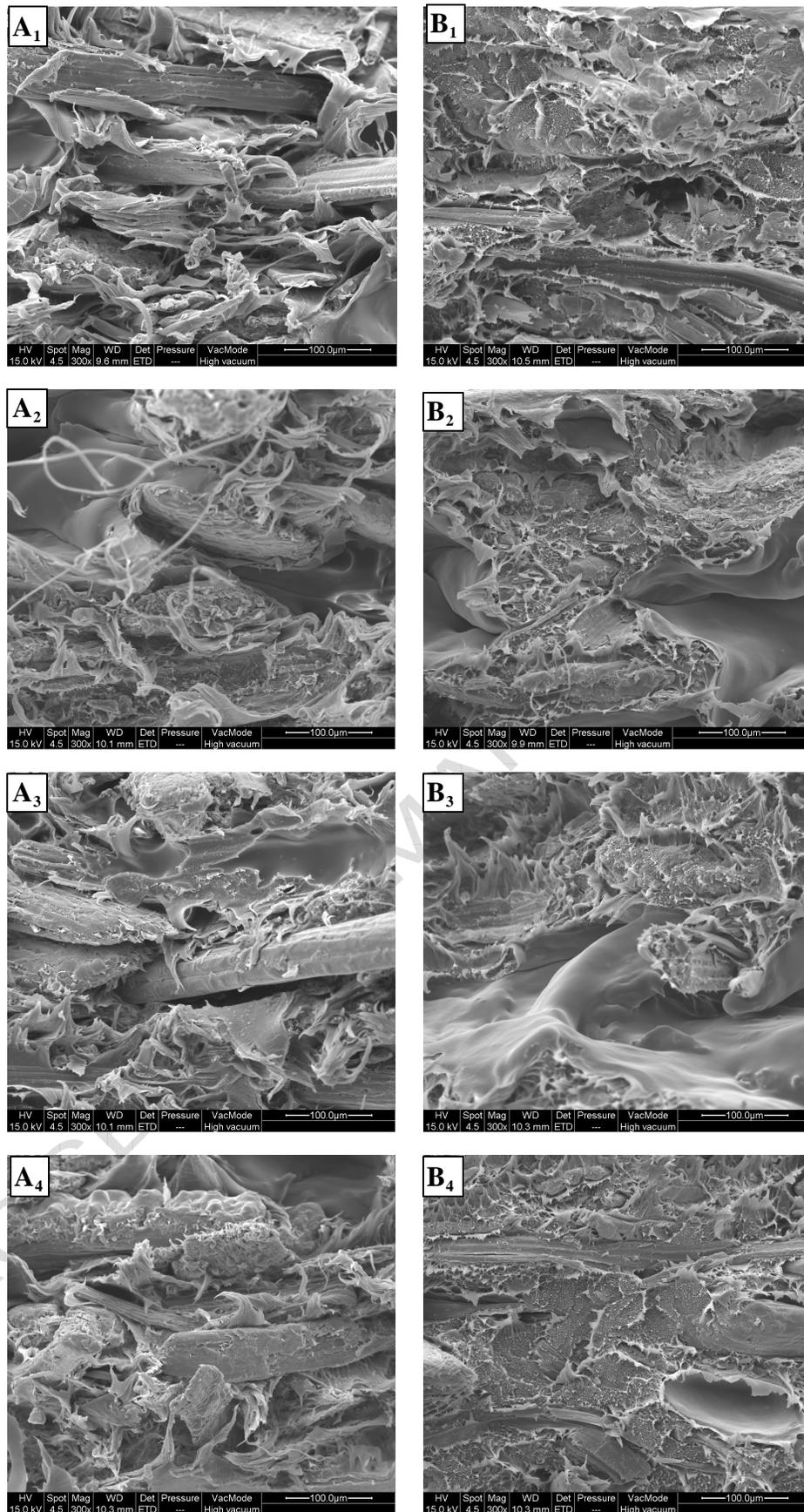


Figure 5. SEM micrographs of the fractured LDPE- (A<sub>i</sub>) and MLDPPE-(B<sub>i</sub>) based composites reinforced with 30 wt% of lignocellulosic fibers (1: Bagasse, 2: Banana, 3: Cotton Stalk, 4 Rice Straw).

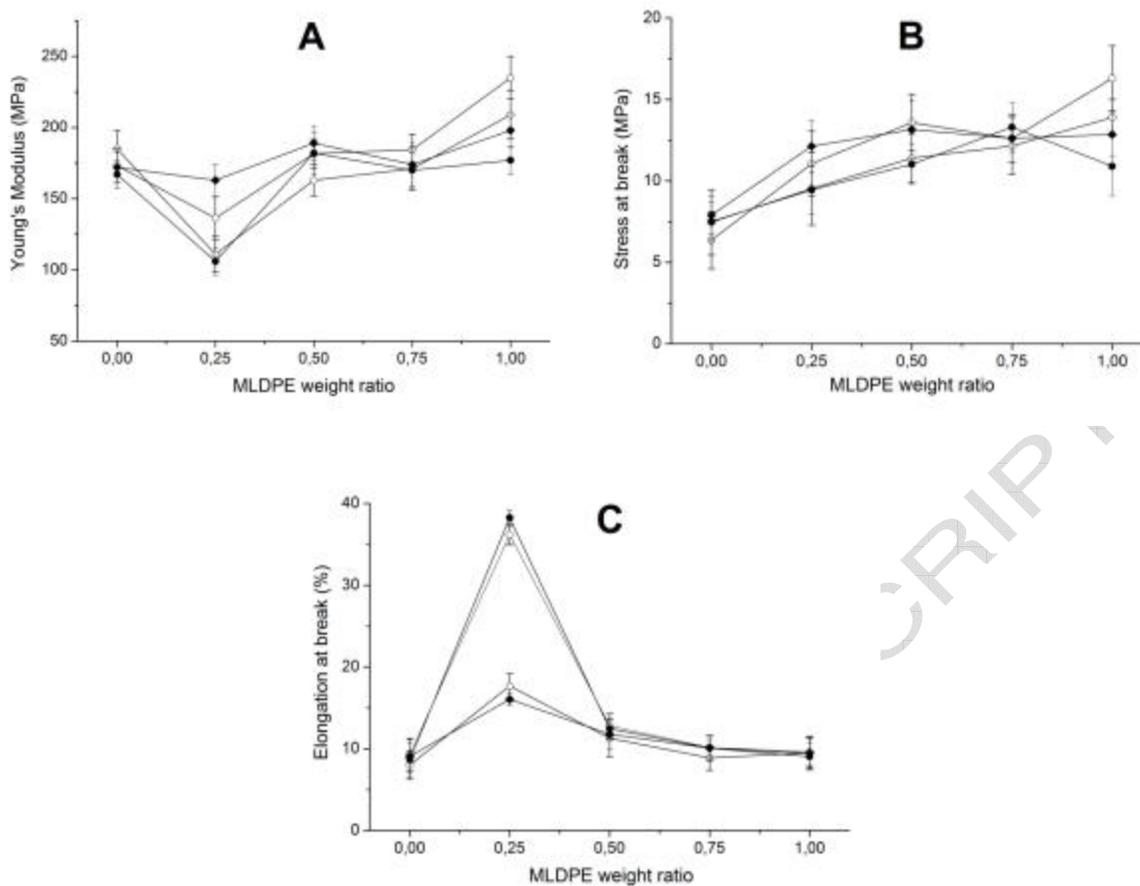


Figure 6: Young's Modulus (A), strength (B) and elongation at break (C) vs MLDPE weight ratio for composites reinforced with 30 wt% of bagasse (○), banana (◇), cotton stalk (●) and rice straw fibers (◆).

Table 1. Chemical composition of the agricultural residues\*

Raw material	$\alpha$ -cellulose	Hemicellulose	Lignin	Ash	Wax
Bagasse	69.4	21.1	4.4	0.6	5.5
Cotton stalk	50.6	28.4	23.1	0.5	5.1
Banana plant waste	43.5	31.7	16.9	9.9	6.1
Rice straw	59.1	18.4	5.3	13.7	6.3

\* As percentage of dry raw material

Table 2: Diameter and length of the fibers

Fiber	Length ( $\mu\text{m}$ )	Diameter ( $\mu\text{m}$ )
Bagasse		
Cotton stalk		
Banana plant waste	380	38.5
Rice straw		

Table 3: Melting characteristics of LDPE- and MLDPE-based composites reinforced with lignocellulosic fibers obtained from DSC curves: melting temperature ( $T_m$ ), associated heat of fusion ( $\Delta H_m$ ) and degree of crystallinity ( $\chi_c$ ).

Sample	Fibers Content (%)	LDPE			MLDPE		
		$T_m$ (°C)	$\Delta H_m$ (J.g <sup>-1</sup> )	$\chi_c^a$	$T_m$ (°C)	$\Delta H_m$ (J.g <sup>-1</sup> )	$\chi_c^a$
PELD	0	112.0	98.4	0.34	-	-	-
MPELD	0	-	-	-	122.4	121.1	0.42
Bagasse	10	112.2	104.2	0.40	122.5	129.8	0.50
	20	112.0	99.2	0.43	123.6	98.1	0.42
	30	112.3	88.9	0.44	124.8	83.5	0.41
	40	112.1	74.3	0.43	122.7	66.5	0.38
	50	112.1	65.6	0.45	124.6	64.6	0.44
Banana	10	112.0	92.7	0.35	122.2	110.3	0.42
	20	112.0	88.2	0.38	124.2	97.0	0.42
	30	110.8	86.3	0.42	122.4	99.6	0.49
	40	111.2	79.2	0.45	123.7	79.7	0.46
	50	111.9	58.9	0.40	120.2	63.0	0.43
Cotton Stalk	10	112.2	95.8	0.37	123.7	124.2	0.47
	20	112.0	92.1	0.40	123.6	96.1	0.41
	30	111.6	90.4	0.44	123.7	90.7	0.45
	40	111.6	91.6	0.53	123.7	51.0	0.29
	50	112.0	81.0	0.56	122.5	57.3	0.40
Rice Straw	10	112.1	84.2	0.32	124.4	114.2	0.44
	20	111.4	101.2	0.43	123.0	94.1	0.40
	30	111.9	76.0	0.37	123.8	88.3	0.44
	40	111.5	81.2	0.46	122.8	73.1	0.42
	50	110.4	74.6	0.50	121.3	62.2	0.43

<sup>a</sup>  $\chi_c = \Delta H_m / w \Delta H_m^\circ$  where  $\Delta H_m^\circ = 288,83$  J/g is the heat of fusion for 100% crystalline Polyethylene [18] and

$w$  is the weight fraction of polymeric matrix material in the composite.

Table 4: Melting characteristics of LDPE and MLDPE blends-based composites reinforced with lignocellulosic fibers obtained from DSC curves: melting temperature ( $T_m$ ), associated heat of fusion ( $\Delta H_m$ ) and degree of crystallinity ( $\chi_c$ ).

Fiber	Weight Ratio LDPE : MPELD	$T_m$ (°C)	$\Delta H_m$ (J.g <sup>-1</sup> )	$\chi_c^a$
Bagasse			88.9	0.44
		112.5	47.6	0.24
		116.7	46.4	0.23
		123.0	60.4	0.30
		124.8	83.5	0.41
Banana		110.8	86.3	0.42
		113.0	59.5	0.30
		115.5	47.6	0.23
		120.0	49.8	0.24
		122.4	99.6	0.49
Cotton Stalk		111.6	90.4	0.44
		113.9	39.8	0.20
		120.1	63.5	0.31
		121.9	57.4	0.28
		123.7	90.7	0.45
Rice Straw		112.1	76.0	0.37
		113.6	50.3	0.25
		117.7	51.3	0.25
		119.2	54.5	0.27
		123.8	88.3	0.47

<sup>a</sup>  $\chi_c = \Delta H_m / w \Delta H_m^\circ$  where  $\Delta H_m^\circ = 288,83$  J/g is the heat of fusion for 100% crystalline Polyethylene and  $w$  is the weight fraction of polymeric matrix material in the composite.