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MECHANICAL ENGINEERING

Composites with pineapple-leaf fibers manufactured by layered compression molding

INGENIERÍA MECÁNICA

Compuestos de fibra de hoja de piña fabricados mediante moldeo por compresión por capas

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Abstract
Compression molding process was used to manufacture composites reinforced with pineapple-leaf fibers. During manufacturing, four plies of polypropylene with an average thickness of 0.76 mm were used and the fibers were equally distributed between plies to generate a stacked composite with the fibers in external layers transversally oriented to those in the inner layer. Two types of pineapple-leaf fibers were used as reinforcement: untreated fibers and fibers modified in an alkali solution (10% NaOH). Two different fiber contents were also evaluated in order to measure their effect on the mechanical properties of the composite. Ultimate tensile strength, strain at maximum load, Young’s modulus, flexural strength and flexural modulus were measured. Tensile strength was increased 22% and Young’s modulus increased 60%. Flexural strength and flexural modulus increased 19 % and 50% respectively, with fiber content. However, the alkali treatment did not improve those properties. Fractured surfaces of the composites were examined using electron microscopy and failure mechanisms such as fiber pullout and matrix deformation were observed.

Keywords: *Composites, compression molding, mechanical properties, pineapple-leaf fibers (PALF), polypropylene.*

Resumen
Se utilizó el proceso de moldeo por compresión para la fabricación de compuestos de matriz polimérica reforzados con fibra de hoja de piña. Para la fabricación de las muestras se utilizaron cuatro láminas de polipropileno de 0.76 mm de espesor y las fibras se distribuyeron igualmente entre éstas, generando un apilamiento alternado del material de la matriz y las fibras, donde las fibras de las capas más externas se encuentran orientadas transversales a las de la capa interna. Dos tipos de fibras de hoja de piña se usaron como refuerzo: fibras sin tratamiento y fibras modificadas con un tratamiento alcalino en solución de 10% de NaOH. Se evaluaron también dos porcentajes de fibra en el compuesto para determinar su efecto en las propiedades mecánicas del material. Se midió la resistencia a la tensión, el módulo de Young, la deformación a la fractura, la resistencia a la flexión y el módulo de flexión. Al aumentar el porcentaje de fibra en el compuesto, se observó un incremento de un 22% en la resistencia a la tensión y de un 60% en el módulo de Young. La resistencia a la flexión y el módulo de flexión se incrementaron en un 19% y un 50%, respectivamente. Sin embargo, con el tratamiento alcalino no se observó ninguna mejora en dichas propiedades. Las superficies de los compuestos fracturados a tensión fueron examinadas mediante microscopía electrónica y se observaron algunos mecanismos de falla, como el desprendimiento de fibras y la deformación de la matriz.

Palabras clave: *Fibra de hoja de piña, materiales compuestos, moldeo por compresión, polipropileno, propiedades mecánicas.*

1. Introduction

Fibers are a class of hair-like material being discrete or elongated, similar to pieces of thread. They can be spun into filaments or rope and they can be produced by animals or plants. Those materials have been used for centuries in several processes such as clothing and the oldest use found of this material is in the buildings trade (Mohanty et al., 2005). In 2013, fifty six (56) million metric tons of man-made fibers (Tecnon Orbichem, 2014) and about 32 million metric tons of natural fibers were produced worldwide (FAO, 2015).

Currently, researchers have shown an increasing interest in natural fibers for applications in composites. A quick search in a common scientific database, showed an increase from 138 papers in 1985 related to composites with natural fibers to 900 in 2014. Additionally, for promoting natural fibers, 2009 was considered as international year of natural fibers and the composites market of United States has been recorded 2.7–2.8 billion pounds from 2006 to 2007 (Asim et al., 2015). Natural fibers obtained from pineapple leaves are a good option to study because of their high tensile strength and high cellulose content (Kalia & Kaith, 2011).

Fibers obtained from pineapple leaves have been previously studied by several authors (Arib et al., 2006; Asim et al., 2015; Chollakup et al., 2011; Devi et al., 1997; George et al., 1997; Hujuri et al., 2008; Shyamal et al., 2011; Smitthipong et al., 2015). Compared to other natural fibers, pineapple-leaf fibers (PALF) exhibit superior mechanical properties due to its high cellulose content (around 70-82%) and low microfibrillar angle (14°) (Mohanty et al., 2005). PALF are obtained from the leaves of pineapple plant Ananas Comosus, which is a perennial herbaceous plant, widely cultivated in tropical regions of Asia, Central and South America (Asim et al., 2015). After harvesting, the pineapple plant has to be removed, generating a large volume of wastes. Wastes consist of leaves of 30 to 90 cm length (Kalia & Kaith, 2011; Krauss, 1949) and they usually contain herbicides spreading into the air during incineration processes (Kalia & Kaith, 2011). Moreover, postharvest utilization of pineapple waste would be an alternative and renewable source of natural fibers for industrial purposes since it can add value to pineapple cultivation and reduce negative environmental impacts in the field.

Recently, PALF have been studied by several authors as a reinforcement in thermoplastic materials such as low density polyethylene (LDPE), polypropylene (PP) and starch/poly(lactic acid) (PLA) (Arib et al., 2006; Chattopadhyay et al., 2009; Chollakup et al., 2011; George et al., 1997; Kengkhetkit & Amornsakchai, 2012; Smitthipong et al., 2015). From the results of authors, it can be concluded that properties of fiber-reinforced composites depend on many factors i.e., fiber-matrix adhesion, volume fraction of fiber, fiber aspect ratio, fiber orientation as well as stress transfer efficiency of the interface between the matrix and the reinforcement (Chollakup et al., 2011).

Arib et al. (2006) investigated mechanical properties of pineapple-leaf fiber polypropylene composites as a function of volume fraction. They found that flexural and tensile strength increased slightly from 2.7% to 16.2%, respectively, when the fiber content increased. The authors also observed that, when the fiber content is higher, properties decreased due to the increment in fiber-to-fiber interactions, void content, dispersion and fiber alignment problems. Furthermore, oth er authors have associated the poor dispersion of the fibers and the void content in polymer composites with the hydrophilic character of natural fibers. Strongly polarized hydroxyl groups found inside natural fibers provide its hydrophilic character. They also reduce their compatibility with the hydrophobic polymeric matrix, generate moisture absorption problems in composites and promotes strong fiber-to-fiber hydrogen bonding that holds the fibers together (Li et al., 2007; Hujuri et al., 2008). Therefore, some authors have proposed the use of physical and chemical treatments to increase adhesion of the fiber to the matrix (John & Anandjiwala, 2008; Kalia & Kaith, 2011).

One of the most commonly used chemical treatments to modify the surface of natural fibers is alkaline treatment using sodium hydroxide (NaOH) (Kalia & Kaith, 2011). Alkaline treatment increases the surface roughness of the fibers (Li et al., 2007), it removes lignin, wax and oils of the fiber cell walls and disrupt hydrogen bonding in the network structure. An increment of effective contact area in the fibers creates higher adhesion between the fiber and the polymer matrix, improving the mechanical properties of the composite (George et al., 1997). Alkaline treatment of PALF has been investigated by some authors to improve fiber compatibility with some thermoplastic matrices (Chattopadhyay et al., 2009; George et al., 1997; Hujuri et al., 2008).

In this work, pineapple-leaf-fibers reinforced composites were manufactured using a modified process with several layers of fibers. The pineapple leaves were processed by a decorticator machine to obtain fibers and after that, they were combed and cleaned manually. After extraction, the surface of the fibers was modified with an alkali treatment, and the fibers were finally used as reinforcement in composites. Mechanical properties of composites were evaluated by tensile and flexural tests. This work was performed in order to evaluate if polypropylene composites reinforced with fibers obtained from an agricultural waste from a specific Colombian region, could be successfully obtained. After obtaining the polymer composites, the authors were interested in evaluating if mechanical properties could be improved by using those wastes.

2. Experimental

2.1. Materials

Fibers were extracted from pineapple leaves using a decorticator machine consisting of two rolling cylinders used for stripping the skin of the leaves in order to obtain the fibers. Decortication happens by alternating bending, buckling, breaking, bruising, and softening the stems. The fibers obtained were left overnight in order to perform the fermentation process in the field used for similar fibers such as fique and plantain. After staying overnight, the fibers were cleaned and combed manually. The length of the fibers was between 50 to 70 cm. High-purity (higher than 99.5%) sodium hydroxide (NaOH) was used for alkali treatment and polypropylene (PP) sheets of 0.76 mm thick were used as a matrix for composites manufacturing. Some properties of PALF and PP used in this study are shown in Table 1.

Table 1. *Properties of pineapple leaf fiber and polypropylene.*

Material	Density (g/cm^3)	Tensile strength (MPa)	Young's Modulus (MPa)	Elongation at break (%)
PP	0.96	26	895	--
PALF*	1.30	413-1627	125	0.8-1
PALF used in this work	1.22	499	-	--

**(Chollakup et al., 2011; Hujuri et al., 2008)*

Chemical composition of PALF was measured as follows: extractives (Tappi Method T204 cm-97, 1988; Tappi MethodT207 om-88, 1988), Klason lignin (Tappi Method T222 om-02, 1988), acid insoluble lignin (ASTM E1758-01, 2015), cellulose, hemicellulose (ASTM 1695-77, 2001) and ashes (UNE 57050:2003, 2003), and the properties are shown in Table 2. Thermal analysis (Differential Scanning Calorimetry) of the fibers revealed that at 300 °C they were damaged.

Table 2. *Chemical composition of pineapple leaf fiber.*

Chemical compositions	Percentage
Extactives	6.49
Moisture	9.40
Klason Lignin (acid insoluble)	8.31
Acid insoluble lignin	10.46
Cellulose	66.74
Hemicellulose	17.45
Ashes	0.71

2.2. PALF characterization

Wet pineapple leaf fibers were fractured in liquid nitrogen and then observed to study its transverse section in a Field emission Scanning Electron Microscope (FEG-SEM JEOL 7100F microscope) at the microscopy laboratory of Instituto Tecnológico Metropolitano. Additionally, the fibers were also observed using a Stereomicroscope Leica EZ4 D. Every fiber was randomly selected and measured in ten different points along its length.

2.3. Chemical modification of fibers

Dry PALFs were soaked into a 10wt% NaOH solution at 30°C, maintaining a liquor ratio of 1:10. Fibers were kept immersed for 2h into the alkali solution and afterwards, they were washed with distilled and fresh water several times to remove any traces of NaOH (Maniruzzaman et al., 2012). After washing, the fibers were dried in an oven at 80°C for 24h.

2.4. Composites manufacturing

Composites were manufactured using pineapple-leaf fibers by layered compression molding. Two different fibers were used to reinforce composites: raw and surface modified fibers. After extraction both fibers (raw and surface modified) were cut into 6 mm length. The length of the fibers was selected according to the results reported in a previous work by other authors (Chattopadhyay et. al, 2009). Composites were obtained by seven alternated layers (0.76 mm thickness) ofcommercial homopolymer polypropylene (PP) from Ideplast company and partially oriented fibers. The properties of the polypropylene can be found elsewhere (Ideplast, 2013). Before manufacturing the composites, PP layers and fibers were kept into an oven at 80° C to avoid problems related to moisture during processing. The PP layers and fibers were positioned into a custom-made mold as shown in the scheme in Figure 1. Fibers were manually oriented and dispersed. Three layers of fibers were located in the mold. Outer layers of fibers were positioned in the main direction of tensile stresses in mechanical tests and those in the inner layer were perpendicular to the main stress direction. Once the fibers and the polymer laminates were located in the mold, they were preheated during 3 minutes at 220°C. After that, a molding pressure of 10 MPa was applied to the plies during two minutes at the same temperature. Subsequently, the upper mold was released to allow trapped gases to escape and then, the molding pressure was applied again for one more minute. Samples were cooled during 20 minutes with the molding pressure being applied in order to avoid deformation of laminates.

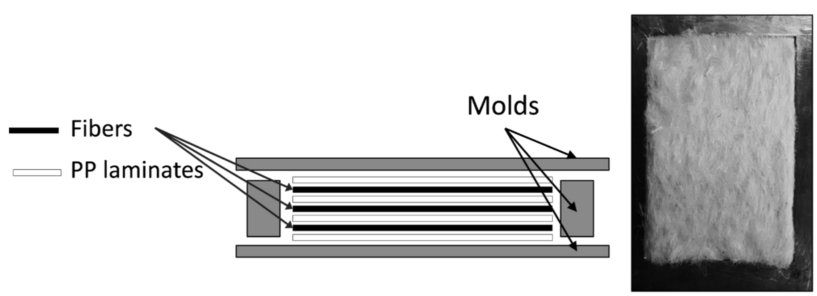

Figure 1. *Left: Scheme of PP/PALF plies arrangement in the steel frame. Right: PALF ply of partially oriented fibers.*

An experimental design was used for test samples. A factorial design with two factors: fiber content and surface modification was used. Two levels of fiber content were defined considering the values of this factor previously studied by Arib et al. (2006) and Hujuri et al. (2008). Factors and levels are shown in Table 3; three samples were tested for every treatment.

Table 3. *General factorial design.*

Factor	Levels	
	1	2
Fiber content	8%	16%
Alkali treatment	Yes	No

2.5. Composites characterization

Before mechanical testing, the samples were inspected; size, color, porosity and deformation were registered. Tensile tests according to ASTM D3039 (ASTM, 2014) were performed in a Shimadzu AGX100 universal tester in Polymers Laboratory of Instituto Tecnológico Metropolitano. Flexural tests according to ASTM D790 Procedure A (ASTM, 2010) were also performed. From tensile tests, ultimate tensile strength, strain at maximum load (as defined in ISO 527-1 Standard) and tensile chord modulus of elasticity (Young's modulus) were calculated; a strain range of 0.003 to 0.005 mm/mm was used for chord modulus calculation. Data obtained from the mechanical measurements were statistically assessed using analysis of variance ($P < 0.05$). Furthermore, failed samples from tensile tests were analyzed by using microscopic techniques (Scanning-Electron Microscope) in order to determine the failure mechanisms.

3. Results and discussion

3.1. Pineapple leaf fibers characterization

The histogram of diameter measurements obtained for PALF by considering 6 diameter intervals is shown in Figure 2. Generally speaking, the diameter of PALF was found from 41 to 155 µm.

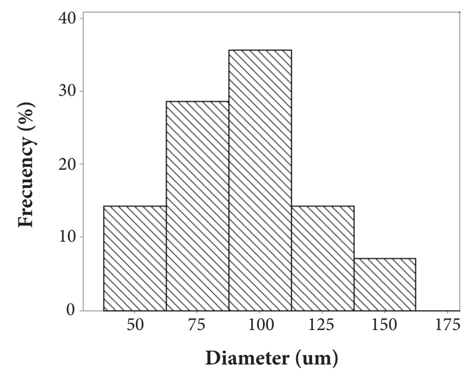

Figure 2. *Histogram of fibers diameter within six conventional diameter intervals.*

Micrographs obtained in electron microscope of pineapple leaf fibers are shown Figure 3. From these micrographs it can be seen that PALF, similarly to other vegetable fibers, are composed of many fiber-cells bonded by lignin, hemicellulose and waxes (Alves et al., 2013). It is also observed that PALF have a smooth surface, commonly associated with a waxy coating called cuticle (Hujuri et al., 2008).

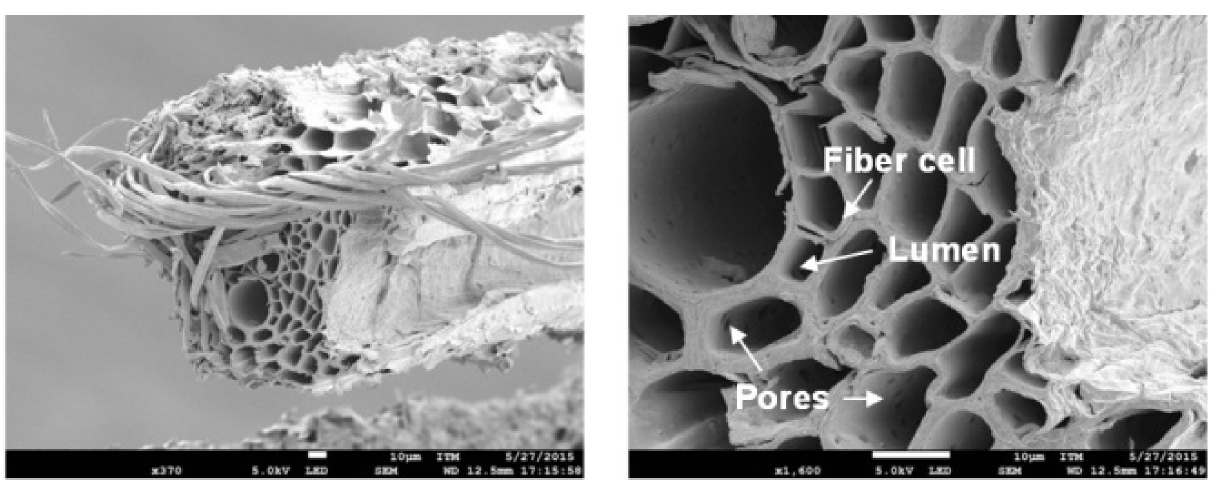

Figure 3. *SEM micrographs of untreated pineapple leaf fiber.*

Pineapple leaf fiber-cells are formed by a lumen and a cell wall with a polygonal shape. Fiber cells of PALF have pores at the walls. Since fiber tissues in pineapple leaves are concentrated around xylem and phloem tissues (Krauss, 1949), it is believed that those irregularities in cell-wall structure may have an important role in the transport of water and nutrients in the plant.

3.2. Mechanical properties

Accordingly to ANOVA test, fiber content is the only factor that had a significant effect on tensile strength (p-value: 0.007) and modulus of elasticity (p-value<0.0001). The results of ANOVA test are in a good agreement with those observed in Figure 4 and allow concluding that tensile properties of PP increase with fiber addition. Moreover, it can be seen that for raw fibers, when the fiber content increased from 8% to 16%, tensile strength increased around 22 % and elastic modulus increased around 60 %. On the other hand, for treated fibers when the average value is compared, tensile strength and elastic modulus increased 23% and 32%, respectively. Moreover, it can be concluded that, for composites reinforced with raw fibers, the standard deviation for average of ultimate tensile strength is lower than that of treated PALF composites. This behavior can be associated to some factors that were not controlled during the experiment such as fiber agglomeration.

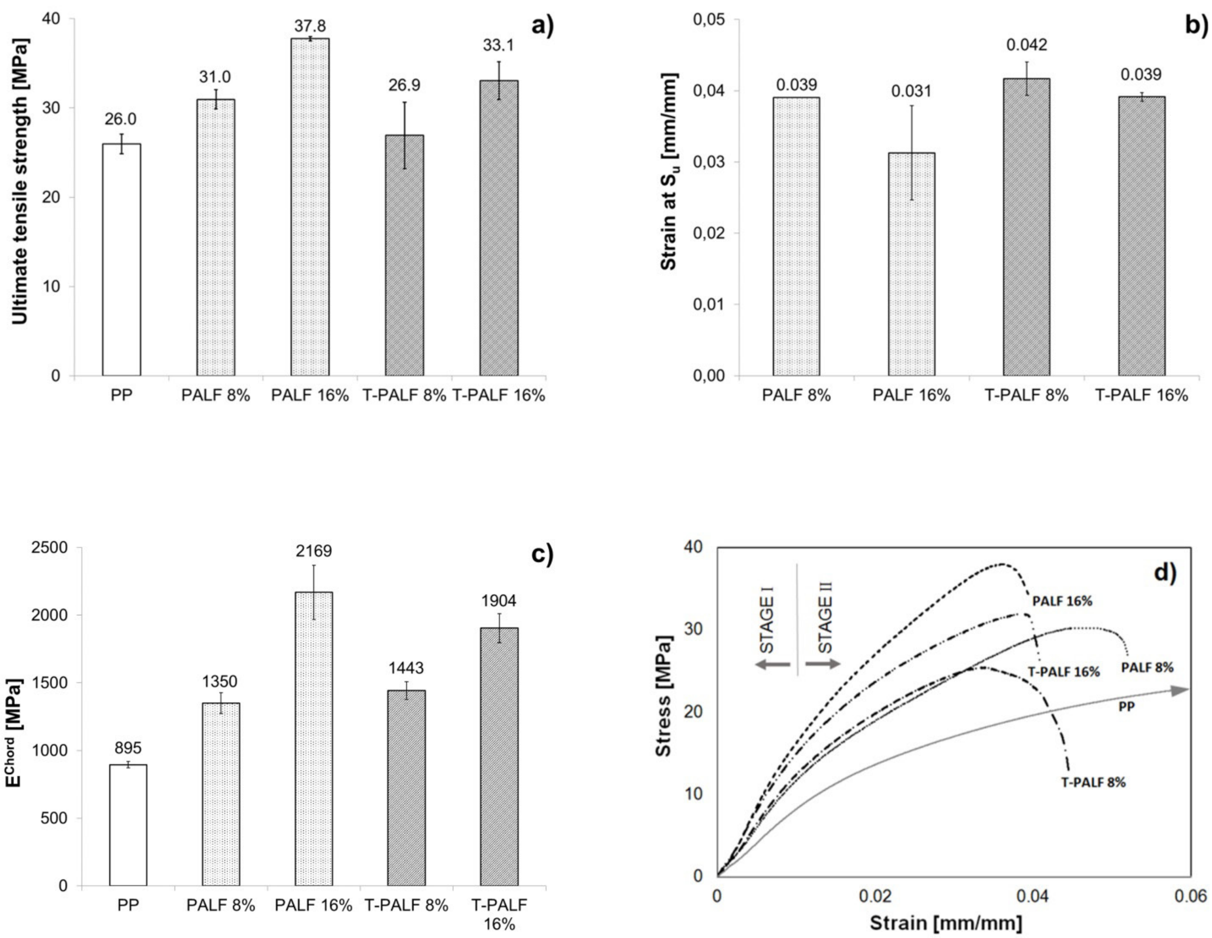

Figure 4. *Tensile properties of raw fibers (PALF) and treated fibers (T-PALF) polypropylene composites.* ***a)*** *Ultimate tensile strength* ***b)*** *Strain at ultimate tensile strength (Su),* ***c)*** *Tensile chord elastic modulus (EChord),* ***d)*** *Tensile Stress-Strain curves of various samples. Error bars represent the standard deviation around the expected value.*

Curves in Figure 4 (d) show a transition strain (around 1%) and it is a common response for fiber aligned composites (Callister & Rethwisch, 2010). Stage I in Figure 4 (d) corresponds to a strain range in which both fibers and matrix suffer elastic deformation and in the tensile plot it is observed as a straight line. Stage II is a region in which the fibers deform elastically while the polypropylene deforms plastically. After that, fibers show a brittle failure because of their low ductility.

It can be concluded that, since strain at ultimate tensile strength (strain at maximum load, Su) does not change significantly between treated and raw fibers, as can be seen in Figure 4 (b), the alkali treatment has a low effect in fibers ductility and their cellulosic structure.

Alkali treatment did not improved mechanical properties of composites. Contrary, UTS decreased when a surface modification treatment was performed. Composites showed some fiber agglomerations after alkali treatment (see Figure 5 for details). Fiber agglomeration was difficult to control and finally derived in a low aspect ratio and a less effective contact area of some fractions of the reinforcement material, increasing the amount of stress concentrators in the composite, reducing the adhesion of the polymer to the surface of the fibers and therefore caused an ineffective load transfer from the matrix to the fiber (Chollakup et al., 2011).

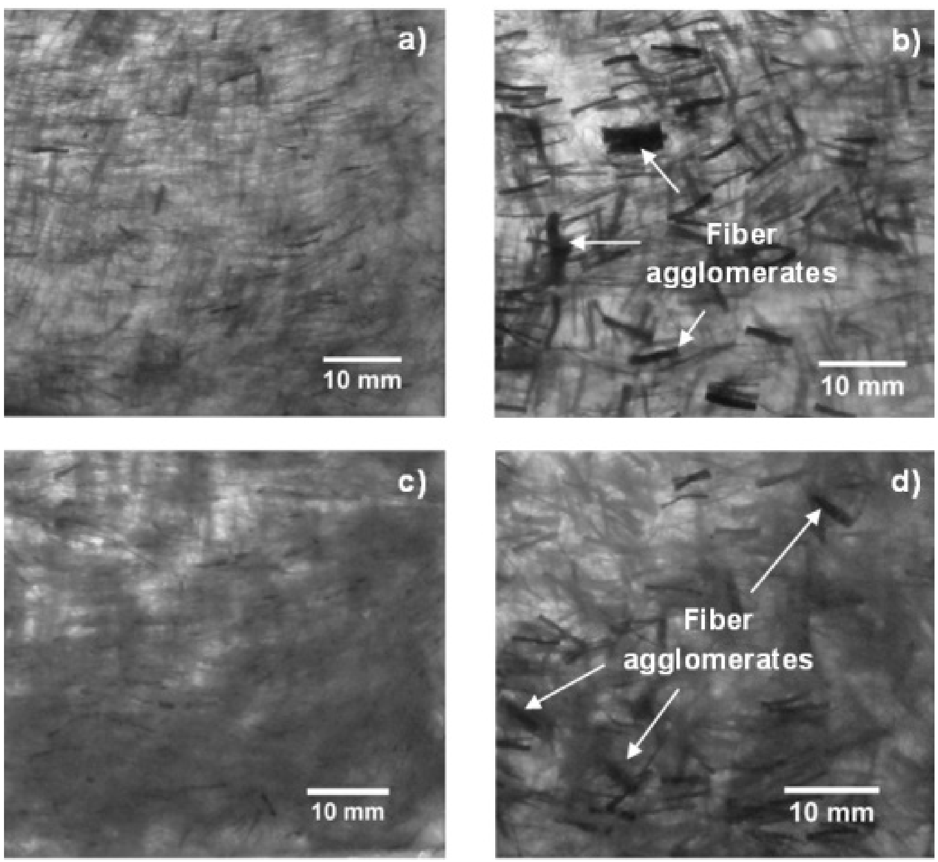

Figure 5. *Photograph of PP-PALF composites by Transmission optical stereomicroscope* ***(a)*** *8% PALF,* ***(b)*** *8% T-PALF,* ***(c)*** *16% PALF and* ***(d)*** *16% T-PALF.*

Results from three-point bend tests (flexural tests) are shown in Figure 6. As found for tensile tests, ANOVA test indicated that fiber content was the only factor that had a significant effect in flexural modulus (p-value: 0.003). Moreover, flexural modulus was higher than modulus of elasticity in tensile tests. This behavior was expected for flexural tests, since the stacking sequence of layers during manufacturing provided a rigid structure to the composite. In flexural samples, the fibers were oriented parallel to the tensile stresses during the test and they were located away from the neutral axis, increasing the stiffness of the composite.

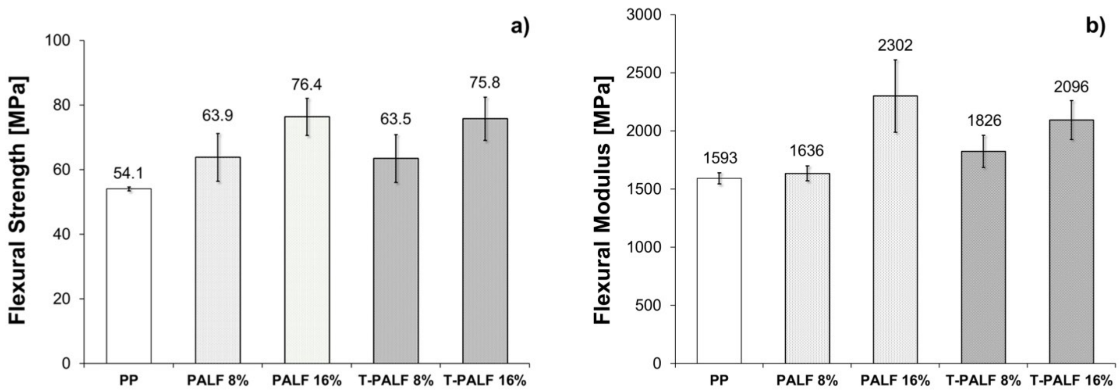

Figure 6. *Results from flexural tests of PALF-PP composites.* ***a)*** *Flexural strength* ***b)*** *Flexural modulus. Error bars represent the standard deviation around the expected value.*

Alkaline treatment changed morphology and structure of the pineapple fibers. When NaOH is used, the rupture of cellulose and hemicellulose bonds is increased and the lignin and extractives are partially removed. Usually, an alkaline treatment is performed before other surface treatments in order to improve the efficiency of additional chemical agents. However, the results from the mechanical tests revealed that NaOH did not improve the adhesion between the fibers and the polymeric matrix. Probably, if the polymer is functionalized, adhesion among fibers and polymer could be increased. Additionally, the same behavior described in tensile strength related to agglomeration of fibers was also observed is flexural test samples.

3.3. Morphology of tensile fracture surface

Figure 7 shows the fracture of the test samples after tensile tests. The observation was performed with two aims: analyze the defects induced during composites manufacturing and identify the failure mechanisms in order to improve the process by subsequent treatments of fibers, polymer or to modify the processing parameters. Failed surfaces showed in most of the cases sound samples. However, some defects were found in several samples. However, it must be emphasized that data (mechanical properties) obtained from failed samples with pores was discarded and the samples were analyzed just to identify problems during processing. The defects were identified as pores, as can be seen at the top of Figure 7 (d). Since the pores were located in the zone in contact with the low mold, it is believed that they were induced in test samples during manufacturing process by inadequate removal of gas because of mold design or inadequate molding pressure (Berins, 1991). Further analysis must be done to identify the causes of porosity in the composites. Additionally, inspection of zones with fibers showed that the polymer filled effectively the mold and the fibers were embedded in the polymeric matrix (see Figure7 e, f and g).

On the other hand, in failed test samples obtained in this work, fiber pullout and matrix failure were observed. Brittle fracture (i.e. broken fibers) of the reinforcement is expected by the low ductility of fibers (Figure 7 f and g). Fiber pullout was observed (Figure 7 e) since it is a common mechanism in materials with a poor fiber/matrix bond. Composites with in fibers with a surface modification showed the same failure mechanism (fiber pullout) but in a lesser extent and a slight increase in terms of the failure mechanism was identified. Accordingly, it can be concluded that surface modification did not improved significantly the adhesion between the fibers and the matrix. It is believed that a new treatment must be performed to the fibers or polymer in order to improve the interaction between the fiber and the polymer. Additionally, it is important to pair the differences of ductility between the fibers and the polymer matrix to improve fiber pullout.

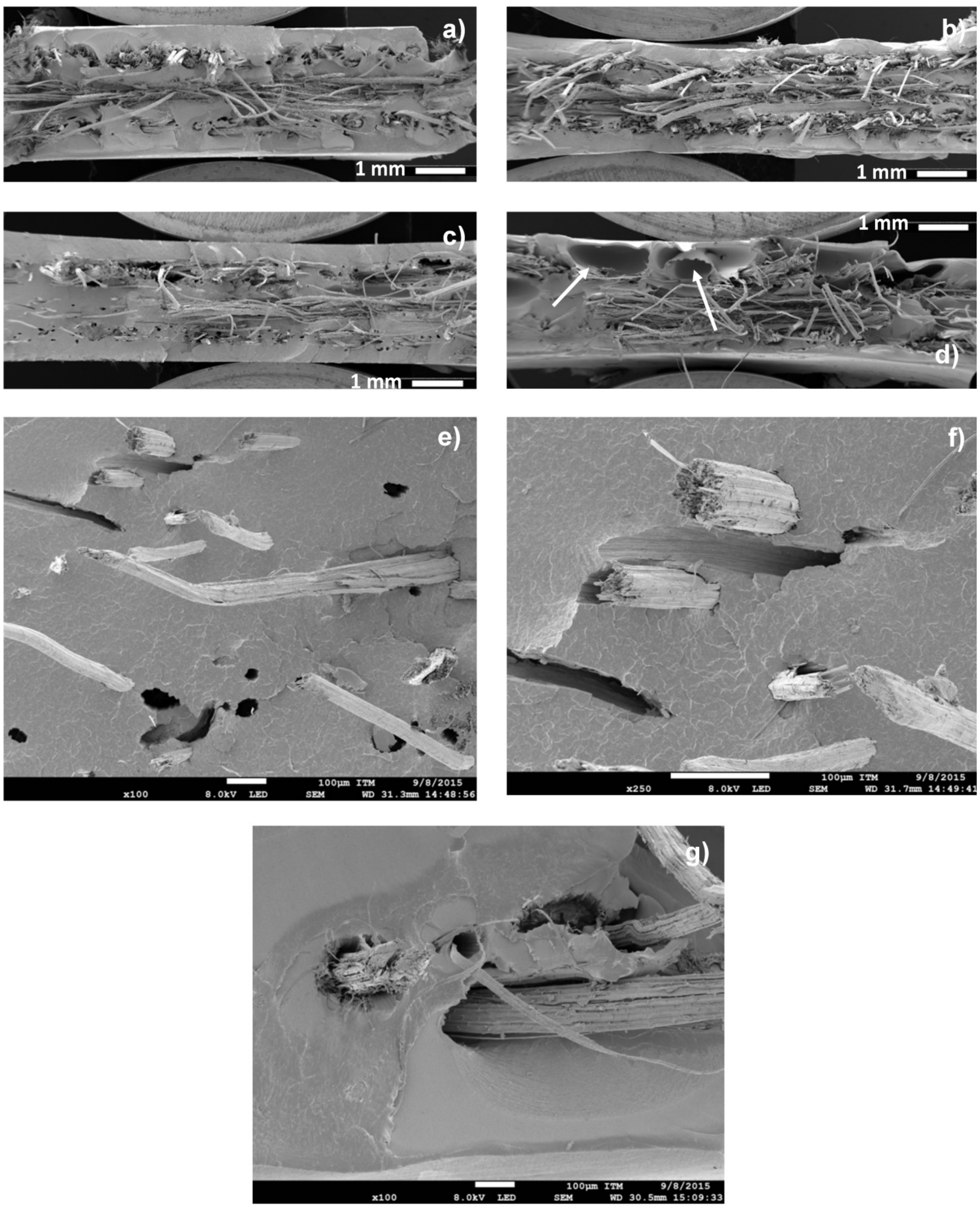

Figure 7. *SEM micrographs of fractured surface of PP-PALF composites.* ***(a)*** *and* ***(g)*** *8% PALF,* ***(b)*** *8% T-PALF,* ***(c)*** *16% PALF,* ***(d)***, ***(e)*** *and* ***(f)*** *16% T-PALF.*

4. Conclusions

Composites with pineapple-leaf fibers were successfully manufactured by layered compression molding. The composites showed in some cases pores related to mold design and a low pressure during molding.

Mechanical properties of the polymer were significantly improved by the addition of pineapple-leaf fibers obtained from agricultural wastes. In the case of raw fibers, it was observed that tensile strength was improved around 22% and elastic modulus was improved around 60%, when the fiber content increased from 8% to 16%. Orientation of fibers and piling sequence provided enhanced stiffness of laminates as observed by modulus of elasticity in flexural tests.

Surface modification treatment performed to pineapple-leaf fibers did not improve mechanical properties of composites because of the formation of agglomeration of fibers inside the composite.

The most important failure mechanisms were fiber pullout and matrix failure. Brittle fracture (i.e. broken fibers) of the reinforcement was observed. It is important to improve the adhesion of fibers by pairing the differences of ductility between the reinforcement and the polymer matrix.

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