STUDIA UBB CHEMIA, LV, Sp.Iss., 2010

NEW COMPOSITE MATERIALS PLATES FROM VEGETAL FIBRES

OVIDIU NEMES[a,*], AMALIA MIHAELA CHIPER[a], ANDREEA RAMONA RUS[a], VASILE FILIP SOPORAN[a], OVIDIU TATARU[a], PAUL BERE[a]

ABSTRACT. The first step in obtained new composite materials plates from vegetal fibers is the chemical treatment of those fibers. Chemical treatments carried out on plant fibers lead to changes on their surface, changes that increase the adhesion between fiber and matrix. Treatment of vegetable fibers with alkaline solutions on different concentrations show that the changes on the fiber surface increased significantly with increasing concentration of alkaline solution. The best results were acquired in the case of a chemical treatment with a solution of KOH 10%.

Keywords: *composite material plates, vegetal fibers, chemical treatments.*

INTRODUCTION

Composite materials obtained using wood fibers or other plant fibers may be a potential candidate for partial replacement of the glass fiber or Kevlar fibers used in obtaining polymer matrix composites. Composite plates with wood fibers could provide an excellent eco-friendly solution to real problems regarding fast consumption of natural resources. Composite materials containing wood fiber or other plant fibers are more and more studied.

These new material configurations are attractive both in terms of lower costs but also because of their mechanical properties which recommend them as a new generation of materials [1-3]. Concerning the length and geometry of cellulosic fibers, they are cylindrical with approximately constant diameter and specific area. This is not the case for cellulosic fibers that present many defects caused by processing [4]. These defects are apparent as ‘knees’ at the fiber surface and constitute points where the fiber may fracture more easily. In addition, an important parameter is the aspect ratio (length/diameter), which has an influence on the mechanical properties of the composite. The aspect ratios of wood, including its physical structure, mechanical properties, and density, change

[a,*] *Universitatea Tehnica din Cluj-Napoca, Facultatea de Ingineria Materialelor si a Mediului, B-dul Muncii, Nr. 103 - 105, Cluj-Napoca, 400641, Cluj. România*

from species to species [5]. The fiber aspect ratio is a critical parameter in a composite material. An aspect ratio in the range of 100-200 after composite processing is recommended for high performance short fiber composites. The strength of the unidirectional aligned composites normal to the fiber alignment (transverse) is less than that of the randomly oriented fiber composite. When the fibers are aligned perpendicular to the force direction (transverse), fibers are not reinforcing the matrix to increase the strength of the composite in the direction of applied load [6].

A first step, in obtaining the composites plates, consists in the chemical treatment of fibers. Natural fibers can be chemically treated due to the presence of hydroxyl groups in lignin and cellulose. These OH groups can change the surface energy and polarity of the natural fibers during various treatments. The most common methods of surface treatment are mercerization (alkali treatment) [7], isocyanate treatment [8], acrylation [9], benzoylation [10], permanganate treatment, silane treatment [11] and peroxide treatment [12]. All these treatments can improve adhesion of the fibers with the polymer matrix and leads to higher impact resistance in comparison with samples containing fibers with no chemical treatment.

This paper presents a comparative study regarding the changes at the interfaces in wood and straw fibers treated with alkaline solutions (KOH) in different concentrations. The morphology of the fibers was investigated using scanning electron microscopy SEM.

RESULTS AND DISCUSSION

Chemical treatments can increase the adhesion at the interface between the fiber and matrix and removed the non-cellulosic compounds, which constitute the main objective of the chemical treatments. Therefore, chemical treatments should be considered in modifying the properties of natural fibers.

During the chemical treatment, a part of hydroxyl groups of cellulose will be replaced with units Fiber-O^--K^+.

$$\textbf{Fiber}-\textbf{OH} + \textbf{KOH} \rightarrow \textbf{Fiber}-\textbf{OK} + \textbf{H}_2\textbf{O}$$

This process was also observed in the case of wood fibers treated with NaOH solution [13].

Changes occurring at the fiber interface were analyzed using FTIR spectroscopy. FTIR spectra analysis shows no significant differences between the treatments of wood fibers realized with NaOH or KOH solution at similar concentrations. Specific signals in the case of fibers treated with KOH (5%) appear in the same area as in the case of samples treated with NaOH reported in literature [14, 15] (3990 cm^{-1}, 1720 cm^{-1} respectively). The intensity of signal situated at 3990 cm^{-1} decrease with increasing concentration of KOH solution (8% and 10%), this is correlated with decreased number of -OH groups located on the fiber surface. A similar trend is observed for the signal situated at 1720 cm^{-1} whose intensity decreases when Fiber-OK unit appear.

A usual method to study the morphology of plant fibers is the SEM method. SEM analysis was made on untreated and treated vegetal fibers. The treatment was realized with different concentrations of KOH solution.

SEM microscopy study in the case of wood fibers

In this study we used a mixture of hardwood sawdust. One aspect to be taken into account in the characterization of wood fibers is the size (Figure 1).

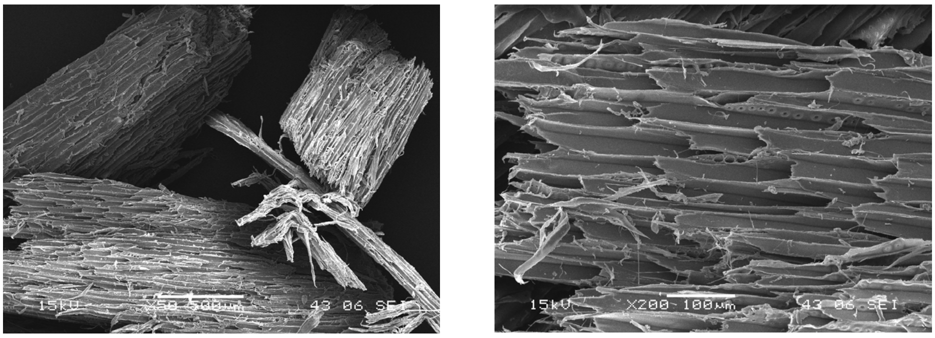


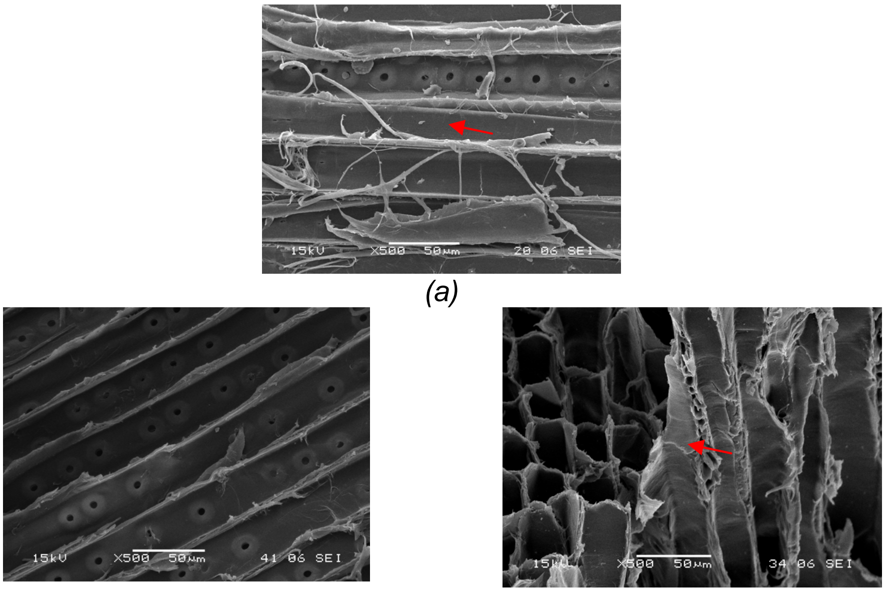
Figure 1. SEM images of wood fiber.

The morphology of the wood fiber, cell size and shape were investigated using scanning electron microscopy (JOEL JSM5510 LV). We observed the sawdust before and after alkaline treatments. In the case of untreated sawdust we can see the normal unchanged cellular structure of wood fiber. The fibers are arranged longitudinally with cellular communication channels (trachea) which transport water, minerals and elaborated sap from root to leaf area.

The SEM images (Figure 2) show the difference between treated and untreated wood fibers. Untreated fiber surface has many large impurities (Figure 2a). Treated wood fibers with KOH 5% solution (Figure 2b) are not affected, there is no fracture and provide a clean surface with few traces of impurities.

The treatment with KOH 10% cleans the fiber surface but affect it causing many fractures (Figure 2c).

The SEM images show the difference between treated and untreated wood fibers.



(a)

(b) (c)

Figure 2. SEM images of wood fiber: (a) untreated; (b) treated with KOH 5%; (c) treated with KOH 10%.

SEM microscopy study in the case of vegetal fibers

SEM analysis examined the surface morphology of untreated and treated fibers. It is expected that the surface morphology of untreated fiber will be different to that of treated fiber particularly in terms of their level of smoothness and roughness. The removal of surface impurities on plant fibers is advantageous for fiber-matrix adhesion and increase fiber strength. Figure 3(a) and 3(b) shows the SEM images of untreated wheat straw and maize stalks, in both figures, there are still a lot of impurites.

SEM image show an improvement in surface morphology after applying a KOH treatment. The improvment can be seen in Figure 3 (c) and (d) which shows that using a KOH 5% solution for treatment, fibers are cleaner but still a large amount of impurities remain on the fiber surface. Figures 3 (e) and (f) shows the absence of impurities on the fiber surface treated with KOH 10%. As compared to the untreated fiber, the KOH 10% treated fiber has a cleaner surface but looks jagged and feels rougher when touched. The changes in morphology are important to predict fiber interaction with the polymer matrix in composites.

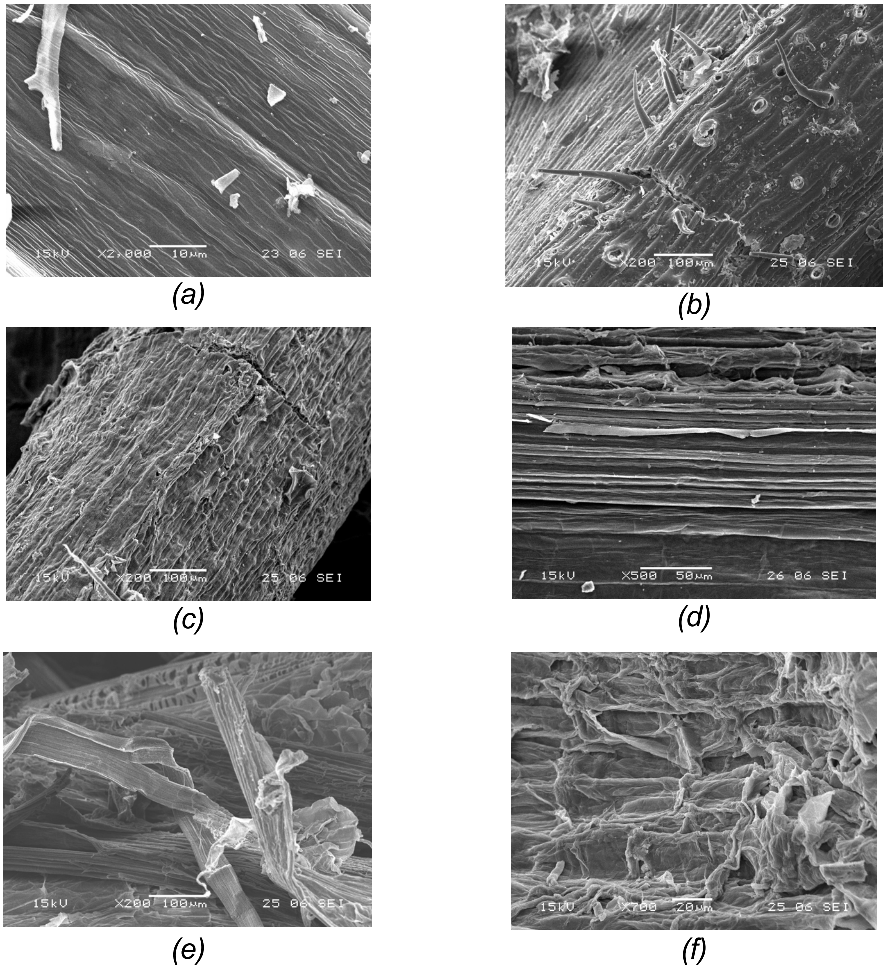


Figure 3. SEM micrograph of (a) untreated wheat straw, (b) maize stalks, (c and d) mixture wheat straw and maize stalks treated with KOH 5%, (e and f) mixture wheat straw and maize stalks treated with KOH 10%.

It has been shown that a KOH 5% solution treatment removes partial the impurities, while after a KOH 10% solution treatment the cleanest fibers surface was obtained. Therefore, chemical treatments can be considered as an important step in modifying the properties of natural fibers used for composite materials manufacturing.

CONCLUSIONS

Development of bio-composites as an alternative to petroleum based materials addresses the dependence on imported oil, reduces carbon dioxide emission, and generates more economical opportunities for the agricultural sector.

The morphological changes were examined using scanning electron microscopy. It has been shown that KOH 5% remove partial the impurities and KOH 10% treatment showed the cleanest fiber surface. The main objective of the chemical treatments is to increase the interface adhesion between the fiber and matrix, and to remove non-cellulosic compounds from the fiber surface. Therefore, chemical treatments can be considered in modifying the properties of natural fibers. The treated fibers show smaller values of moisture content, which could indicate a change in their hydrophobic surface.

Thus, in order to develop composites with good properties, it is necessary to improve the interface between the matrix and the lignocellulosic material.

EXPERIMENTAL SECTIONS

Materials

In this study we used a mixture of hardwood sawdust (wood fiber). Fiber dimensions affect the quality of the interfaces that appear in the composite material, and its default properties. Grain size of the sawdust used was between 0,44 and 0,8 μm.

The non-wood fiber, wheat straw (*Triticum aestivum*) and maize stalks *(Zea mays L.)* were the sources of fibers used in this study. The major constituents of wheat straw are 71,24% cellulose and hemicellulose, 23,22% lignin and 5,54% ash and for maize stalks are 38 – 40% cellulose, 7 – 21% lignin, 28% hemicellulose and 3,6 – 7% ash [16]. Wheat straw and maize stalks were collected from local farmers and then chopped in a knife mill Grindomix GM 200. In the production of experimental panels, polyester ortophtalic resin Lerpol TIX 3603 was used as binder with a density 1110 – 1120 kg/m^3.

Wood-fiber treatments

The fibers surficial treatment is an important step in the manufacturing process of obtaining composite materials with sawdust. These chemical treatments are designed to modify the fiber surface. This operation removes impurities, increase the roughness and ensure a high mechanical adhesion to the matrix. The most effective chemical treatments for natural fibers are the alkaline treatments. To achieve the fibers treatment operation, we used a solution of potassium hydroxide (KOH) of different concentration of 5%, 8% and 10% respectively.

Treatment was done by immersing the sawdust fibers in KOH solution with known concentrations for 3 hours at room temperature, being achieved the fiber surface degreasing. There followed a fiber washing with distilled water for 30 minutes. After this the fibers are immersed in a solution of acetic acid (CH_3COOH) for 30 minutes and washed again with distilled water for neutralization. Following this operations, sawdust was placed in the oven for 24 hours at a temperature of 105 °C.

Non-wood fiber treatments

The fiber were first placed in solution of potassium hydroxide (KOH) with a concentration 5%, 8% or 10% for 3 h at room temperature. Afterward, the alkalized fibers were washed with distilled water, followed by neutralization with 20 mL of acetic acid solution. Wheat straw and maize stalks were then washed with distilled water again and dried at 105 °C for 24 h.

ACKNOWLEDGEMENTS

This work was supported by CNCSIS-UEFISCSU, project number PNII-IDEI ID_1100/2007. O.N. thank the project "Progress and development through post-doctoral research and innovation in engineering and applied sciences – PRiDE – Contract no. POSDRU/89/1.5/S/57083", both co-funded from European Social Fund through Sectorial Operational Program Human Resources 2007-2013

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