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Nesrine Bouhamed, Slim Souissi, Pierre Maréchal, Mohamed Benamar, Olivier Lenoir. Effect of Injection Direction in Elaboration of Polypropylene Reinforced with Olive Wood Flour on Ultrasonic and Morphological Properties. Tahar Fakhfakh; Chafik Karra; Slim Bouaziz; Fakher Chaari; Mohamed Haddar. *Advances in Acoustics and Vibration II. Proceedings of the Second International Conference on Acoustics and Vibration (ICAV2018)*, March 19-21, 2018, Hammamet, Tunisia, 13, Springer, pp.292 - 299, 2018, Applied Condition Monitoring, 978-3-030-06885-1. 10.1007/978-3-319-94616-0_30 . hal-03313384

HAL Id: hal-03313384

<https://hal.science/hal-03313384>

Submitted on 29 Jun 2023

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Effect of Injection Direction in Elaboration of Polypropylene Reinforced with Olive Wood Flour on Ultrasonic and Morphological Properties

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Abstract. Although the anisotropy of wood fibers is reasonably well established, the anisotropy of injection molded wood fiber composites is not well understood. For this, fiber distribution is an important parameter in determining the properties of the composite. This work investigates the application of ultrasonic testing in evaluating natural fiber thermoplastic composites reinforced with olive wood flour (OWF). The characterization of sound propagation speed in the composite is intended to be a tool for evaluating the bio-composite namely fiber distribution and the effects of the direction of injection during the elaboration of the composite. The quality of fiber distribution homogeneity can be assessed by mapping the returning signals of the emitted longitudinal ultrasonic wave. This study presents the measured sound speeds for a composite system of OWF and polypropylene (PP) using immersion measurements. It is known that the longitudinal wave velocity is a function of the material property, which in turn is a function of fiber content and adhesion efficiency. Therefore, the aim of this work is to study the feasibility of using the ultrasonic longitudinal sound wave and the time of flight TOF instead of the morphological analysis with the scanning electron microscope, which is much more expensive and complicate.

Keywords: Ultrasonic properties · Bio-composite · Injection direction
Time of flight

1 Introduction

Faced with the growing interest in the correct use of natural fibers, in parallel with glass and carbon fibers because of their low cost, high specific modulus, light weight, high availability and biodegradability [1, 2], the use of natural renewable lignocellulosic materials such as reinforcing fillers in thermoplastic or thermosetting polymers has recently increased in order to preserve environmental resources while improving economic activities. The industrial use of wood-plastic composite (WPC) cross for

several years in many fields like engineering and technology [3], these composites predominate in outdoor deck applications, the automotive field [4], the aerospace industry and others. Concerned thermoplastic polymers reinforced with wood fibers or flour the most widespread matrices are polypropylene (PP) [5] polyethylene (PE) [6], and polyvinyl chloride (PVC) [7, 8], because of their low cost. Furthermore, the processing temperature of these thermoplastics is less than 220 °C, which makes it possible to avoid the degradation of cellulosic fibers. This work will focus on olive wood flour reinforced with polypropylene.

Although several studies have reported the possibility of using wood flour as thermoplastic polymer filler, but no thorough investigation into the incorporation of OWF into a polypropylene (PP) matrix has been conducted. Only a few research studies reported in the literature have addressed the use of olive stones as filler. The use of olive nut flour as a filler for the PP matrix was considered by Amar et al. [1]. Composites based on olive stones and PVC as a matrix and the study of their mechanical and thermal stability were studied by [2, 9].

The applications of the non-destructive control (NDC) for the mechanical characterization materials are more and more numerous [10], and return the experimental approaches destructive mechanical tests.

In particular, the ultrasonic techniques, based on the analysis of the distribution of the elastic waves through a sample, are very used seen the direct connection between the characteristics of the elastic answer of the material and the characteristics of the distribution of the waves [11]. This property can be usefully exploited for the mechanical characterization of innovative and complex materials, as, for example, composites and/or laminated materials, materials anisotropes, biological materials, etc. [12, 13].

There are many advantages of the ultrasonic tests non-destructive: the freedom of choice of the geometry of samples, the possibility of obtaining a high level of precision, the quantity of information, the speed and the low(weak) price(prize) of the experiences (experiments).

We know that the longitudinal sound speed is a function of the material property, which, in his/her turns, is a function of the content in fibers and the efficiency of adhesion. Consequently, in this study, we prepared composites with polypropylene strengthened by fibers of wooden flour of olive tree by studying the feasibility to use sound waves to obtain the mappings of composites to realize one morphological and thermal analysis compare the results obtained with those realized by the microscope with electronic sweeping and tries thermal.

2 Materials and Methods

2.1 Raw Materials

Polypropylene (PP) used in this study is a standardized homopolymer PP H9069. This polymer is a simple flow grade with a melt flow index of 25 g/10 min (230 °C, 2.16 kg) according to ISO 1133 standard in order to facilitate the adhesion of the fibers in the composite material. The tensile strength of this polymer, measured by ISO 527-2,

is 23 MPa, the tensile modulus is 1.6 GPa and the hardness Rockwell is 95 under ISO 2039-2.

The olive wood was from the region of Sfax in the center of Tunisia. It was recovered from the waste wood obtained from the craftwork through an aspirator, it was directly oven dried at 105 °C for 24 h to reduce its moisture content. It was then sieved and stored in plastic bags to protect it against moisture.

2.2 Elaboration of Composites

To eliminate all the absorbed humidity and avoid the formation of agglomerates, the OWF was pre-dried in 105 °C during 24 h before the extrusion. The polymer PP and the OWF were mixed in an extruder mini (DSM Xplore Netherlands) in screws coupled in co-rotation parallel with three zones of heating. The rotation speed of the screw was settled in 100 rpm and the exit (release) of the material was 200 g/h, the diameter of the screw was 10 mm. First, the OWF and the PP were manually mixed and placed in the hopper of extruder. The compound materials crossed the various zones and were extruded through the cylindrical matrix (diameter 1 mm). Then, the extruded were cooled then cut. The granules obtained subsequently underwent injection in order to obtain panels of dimensions $l = 100$ mm, $L = 120$ mm and $e = 4$ mm.

2.3 Ultrasonic Testing

To make the images of C-scan maps, focusing transducers of 0.5" in diameter are used. The water in the tank is maintained at a constant and homogeneous temperature by a double tank system (Fig. 1). The transducer is positioned 43 mm above the plate and a precise X-Y scan is performed (Fig. 2). The equipment used mainly comprises an X-Y-Z rack completed by a control device (MISTRAS) and a signal acquisition and processing software (UTwin from Eurosonics).

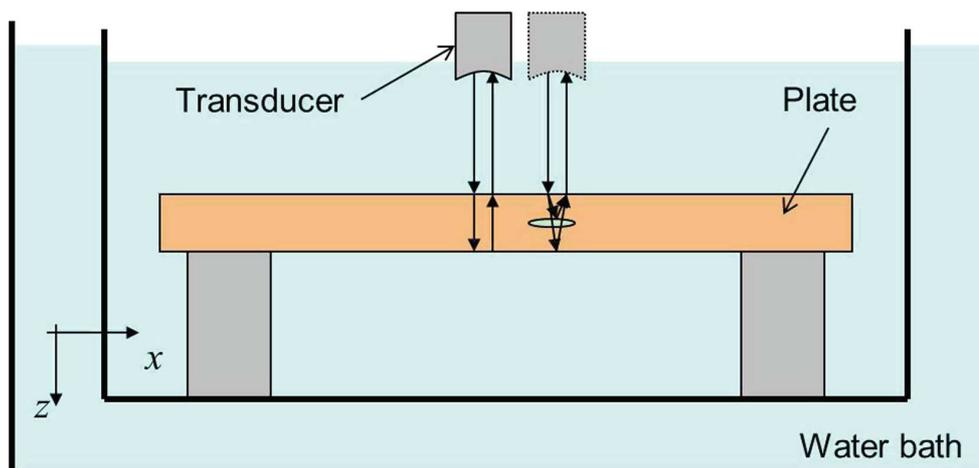


Fig. 1. Explanatory diagram of the equipment used for the ultrasonic measurement

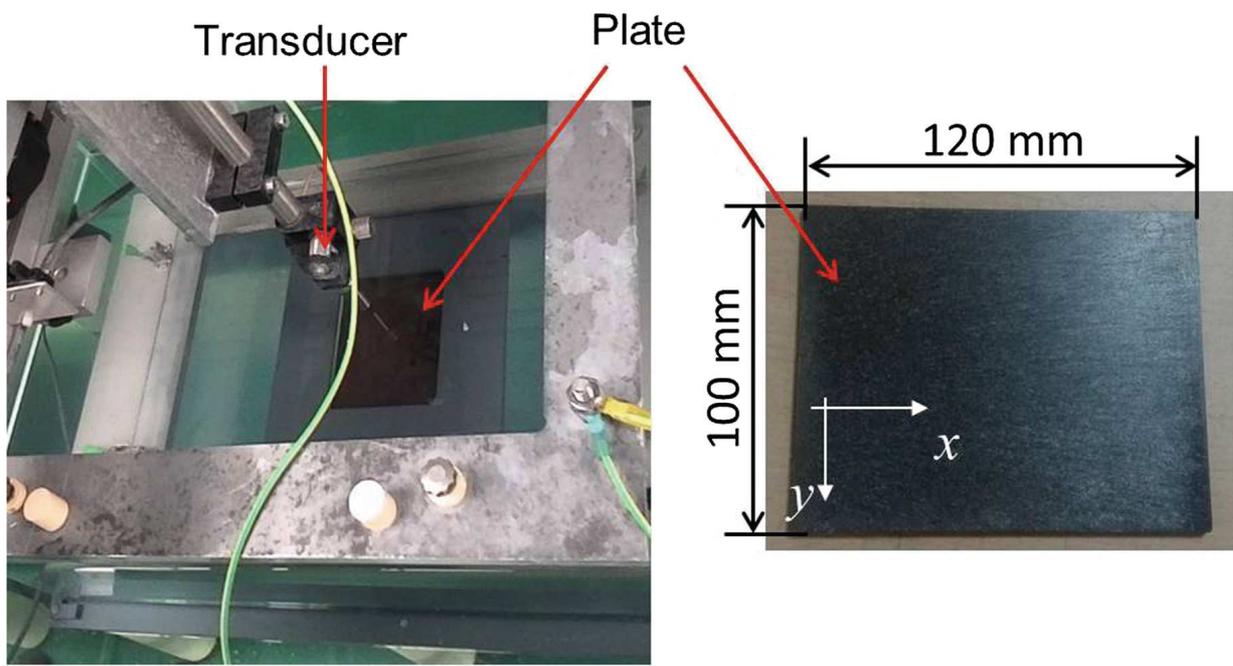


Fig. 2. Immersion control of the plate using the ultrasonic method

2.4 Acquisition of Data

The mappings obtained are used to inform about the surface state as well as the homogeneity of the samples, it was also possible to interpret echoes based on these maps as shown in Fig. 3, the echoes obtained allows us to evaluate the properties composite by determining its longitudinal velocities (C_L) and its attenuation (Att_L).

Signals were processed by Matlab software using two estimation methods: the time of flight and the spectral method. The calculation of the speed of propagation of the wave in a material by flight time measurements is made from two echoes.

By time of flight and amplitude ratio measurements (Fig. 3b), properties such as speed and attenuation in the time domain can be evaluated from two echoes.

The propagation longitudinal speed C_L given by the Eq. (1) and the ultrasonic attenuation att_L by Eq. (2) are deduced from the transfer function:

$$C_L = \frac{2e_p}{\Delta t} \quad (1)$$

$$att_L = \frac{1}{2e_p} \ln\left(\frac{A_2}{A_1}\right) \quad (2)$$

Where:

$$\Delta t = T_2 - T_1$$

- T_1 : is the projection on the time axis of the maximum of first signal
- T_2 : is the projection on the time axis of the maximum of second signal
- A_1 : is the amplitude of the first emitted signal

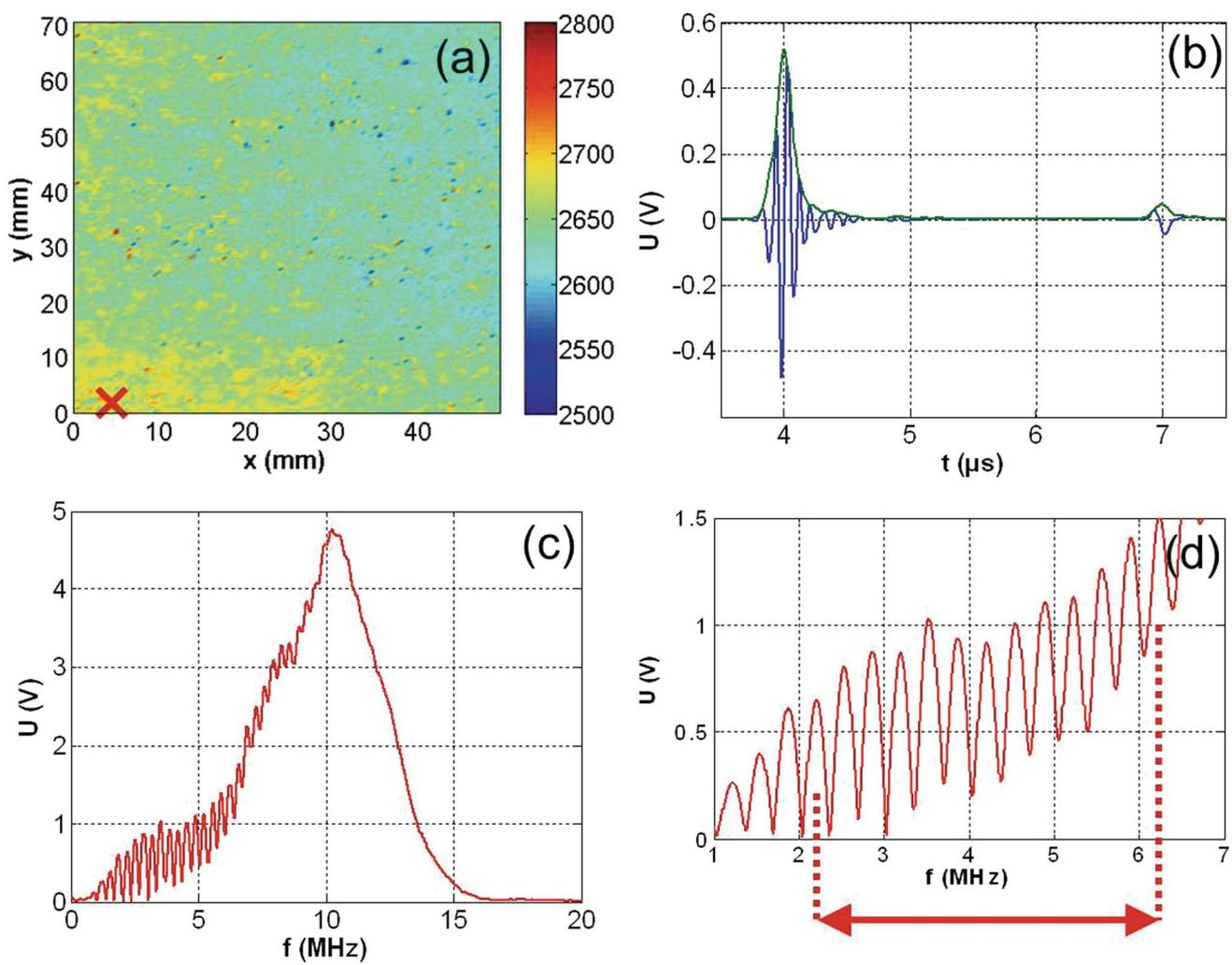


Fig. 3. Methods for calculating C_L in a marked point of the cartography (a) by the calculation of the time of flight (b) and the spectral method (c, d)

- A_2 : is the amplitude of the second emitted signal
- e_p : is the thickness of the measuring point

The spectral method is more complicated, it consists in expressing the temporal signal (Fig. 3c–d) according to the spectral ratio of two echoes indeed, by applying the Fourier Transform (FT) via the Matlab signal processing software, we transform the real time signal $s(t)$ in a complex frequency signal called spectrum $\underline{S}(f)$. The spectral method consists in expressing these properties according to the spectral ratio of two echoes, in the case where frequency evolutions of speed and attenuation can be observed. This method allows finding the ultrasonic dispersion properties with the frequency. The spectral method allows us to calculate the longitudinal speed propagation C_L in the plate from the Eq. (3):

$$C_L = 2e_p \cdot \Delta f \quad (3)$$

2.5 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) was performed with a JEOL JSM-5400 Microscope operate in gat 15 kV. Prior to analysis, all samples were coated with a layer of gold to avoid sample charging under the electron beam.

3 Results and Discussion

Following the observation of the ultrasonic speed mapping (Fig. 4), we noticed a color difference from left to right depending on the injection direction of the bio-composite plate. The distribution of colors varies from red (2750 m/s) at the beginning of injection followed by a great heterogeneity of colors (blue, green, yellow from 20 mm from a variation of ultrasonic speed. From the 90 mm length of the panel the colors going more and more towards low speeds (dominance of the blue color).

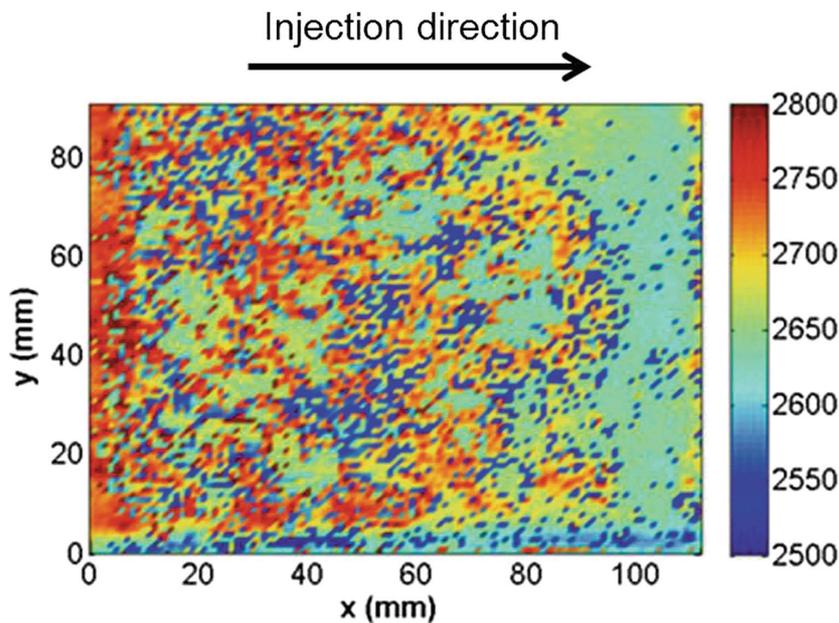


Fig. 4. C-scan maps of C_L of the PP /20% OWF plate.

This large variation between the two ends of the bio-composite panel according to the direction of injection and due to the poor distribution of the olive wood flour. The accumulation of red at the beginning of the panel shows the agglomeration of the OWF on this part.

This is confirmed after cutting and measuring the density of the noted samples (P1 and P2) as shown in Fig. 5, we observe a decrease in the density value from 0.963 (part P1) to 0.931 (part P2). This explains that the density of the plate decreases according to the direction of injection. This variation in density is accompanied by the presence of visually remarkable porosity on the cutting face (Fig. 5), this porosity increases with the direction of injection of the panel.

To be able to determine the adhesion rate between the fibers and the matrix on the surface of the plates according to their directions of injection, we could cut a piece of the lower part of the bio-composite plate as shown in the Fig. 5. We then made

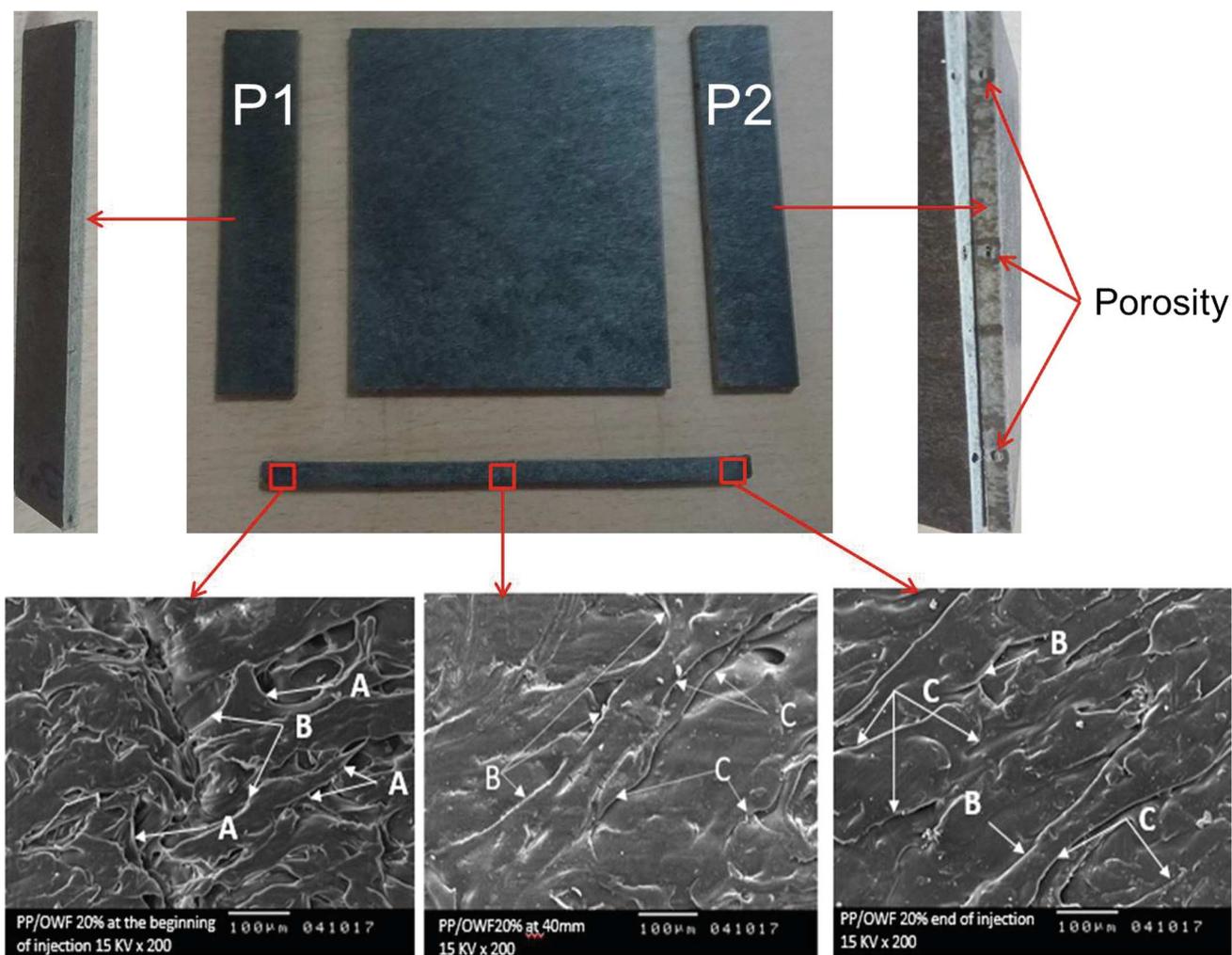


Fig. 5. Scanning electron micrographs at 200x enlargement at the surface of the samples made with 20% of fibers (legend: A = no close contact/good wetting, B = macro-fibrils, C = close contact/good wetting)

observations with the SEM on three different zones (begin of injection, sample medium and end of injection), the observations obtained show a difference in adhesion between the OWF and the PP according to the inspect area.

SEM micrographs showed variations in wetting at the fiber-matrix interface among the different sample area (Fig. 5). For example, at the beginning of the plate (first injected zone) fibers are not in close contact with PP (noted A) whereas at 40 and 120 mm length of the plate (middle and end of injection) we find some fibers completely wetted with PP (noted C), other fibers are not completely wet-ted either. SEM micrographs also showed variations in mechanical adhesion and interlocking at the fiber-matrix interface among the different sample area investigated. The three areas observe have macro-fibrils at the surface interlocking with the polymer matrix, thus increasing fiber reinforcement (noted B). The wetting and interlocking phenomena suggest a superior stress transfer in the case of fibers in the end-injected zone. It explained the better performance of this area among the three zone observe in the present study.

4 Conclusion

The direction of injection during the manufacture of bio-composite plates has a huge influence on the characteristics of the latter such as the density, the porosity and the adhesion ratio between fiber and matrix. It can be concluded therefore that moving away from the injection nozzle the ultrasonic speed decreases, consequently, the density of the composite decreases because of the agglomeration of the charge at the beginning of the plate. We also noticed the formation of pores at the end of the plate (end of injection). Regarding the adhesion between fiber and matrix it much better at the end of injection because of the lack of charge rate in this area.

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