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Study of the physical and mechanical properties of a naturally reinforced composite after several recycling processes

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Abstract
Composites based of Poly (vinyl Chloride) (PVC) reinforced with maize flour (MF) at proportion of 30 (%.Wt) were prepared. The objective of this work is to study the effect of the number cycle of the recycling in the physical, chemical and mechanical properties in this composite. Different characterization techniques were used to study the effect of the number of recycling cycles on the mechanical, physical and chemical properties of the materials developed. The results recorded after three cycles of recycling show an increase in tensile strength, in Young's modulus and in water absorption of composites. However, a decrease in hardness is recorded. FTIR analysis showed no change in the absorption bands after recycling.
Keywords: *Composites, recycling, PVC, vegetable filler.*

I. Introduction

The accumulation of plastic waste in the environment has led to overcrowding of landfill sites and pollution of soil and marine environments. A number of solutions have been put in place to reduce their impact on the environment. Like giving these polymers a new lease of life through recycling. So, in the interests of the environment, we need to improve recycling processes and encourage the use of materials derived from abundant, recyclable and renewable natural resources. To this end, numerous environmental standards (ISO 14 001) [1] have been created to contribute to sustainable development goals. These standards have had a major impact on the industry, particularly in the development of new composite materials based on natural fibers. Natural fibers are a new trend in reinforcements and supplements, used to replace synthetic materials and related products. The development of natural fibre-reinforced composites has been one of the notable achievements in materials science over the last century [2-5]. In recent years, composite materials have come to occupy a prominent place in various fields. However, the development of these materials must be accompanied by the introduction of industrial solutions capable of processing production waste and end-of-life products in compliance with the regulatory framework [6].

Polymer matrix composites are used on a large scale in a variety of industrial applications (transport, construction, etc.). However, their recyclability, perceived as difficult or at least perfectible due to their heterogeneity, can be an obstacle to their wider penetration of certain markets. Some users may prefer alternative materials that are easier to recycle to composites. Environmental concerns and regulatory pressure have prompted manufacturers in the composites sector to develop recycling and recovery solutions, whether in terms of materials, heat/energy or chemicals. Against this regulatory backdrop, various industry initiatives are aiming to set up dismantling/recycling/recovery channels for these materials, either by application sector or across the board [7].

The aim of this research is to study the possibility to prepare PVC/maize flour composite and the study of the effect of the number cycle of the recycling in the physical, chemical and mechanical properties in this composite.

II. Material and methods

II.1 Materials

The PVC used is the SE1200 type and has been utilized as a matrix for the development of the different samples. Glycerol was procured from Chemopharma BIOCHEM. The maize flour with a diameter ≤ 300 µm was obtained from an Algerian cereals company, Algeria. The proportion between the maize flour and the glycerol was 70/30 (wt/wt). This percentage is used to obtain flexible materials.

Additives were added for the preparation of the various formulations, including dioctyl phthalate (DOP) as a plasticizer, a Ca/Zn-based thermal stabilizer and stearic acid as a lubricant.

II.2 Methods

Samples preparation

PVC (F0) and PVC/MF composite at ratio of 70:30 (F30) was prepared by two processing methods, namely: calendaring and compression molding. The blend was introduced into a T6HK8 type mixer for 15 min at the rate of

2000 rpm and at the working temperature of 80°C. Before unloading, this mixture cooled at 40°C. Then the blend introduced into a two-roll mixer at a temperature of 140°C. The prepared mixture is introduced into the platens of the brand table press at 170°C with a pressure of 300 kN during 5 minutes. Pieces of hard plastic of 250×250×2 mm^3 are obtained and cooled to room temperature, which will be used for cutting samples in the form of dumbbells.

Recycling process

The PVC and PVC/FM composite are recycled using a brabender. The recycling stage was carried out using a 'Brabender Gmbh & Co KG' internal mixer. The previously prepared plates, which had been cut into small fragments, were poured into the Brabender chamber (hopper) at a temperature of 180°C, a speed of 50 rpm and a residence time of 5 minutes in order to obtain a paste. The Brabender is controlled by a computer running WIN MIX software.

Tensile properties

Measurements of the tensile properties were performed using a Shimadzu tensile testing machine (Model Autograph AGS-X 10kN). Measurements were performed at a 10 mm min^{-1} crosshead speed at ambient temperature. five specimens were tested. The Young's modulus. strain and stress were determined.

Hardness

This technique consists of the application of a force designed to force the pointed steel needle (punch) of the Shore-D durometer onto a 1x6 cm² plate in accordance with standard NF T51-109.

Spectroscopy analysis (FTIR)

FTIR spectra were recorded using an infrared spectrophotometer Fourier Transform Model SHIMADZU FTIR using film samples. The spectra were recorded in a transmittance mode from 4000 to 400 cm^{-1} at a resolution of 4 cm^{-1}.

Water absorbance test

The water uptake is determined in standard ASTM-7031-04. The measurement consists of submerging the specimen into distilled water at room temperature with agitation. The samples are then weighed at intervals of 24 hours until the weight have stabilized. The rate of water uptake is measured using the following equation:

$$\Delta m(\%) = \frac{m-m_0}{m_0} \times 100 \qquad (1)$$

With:

m_0: Sample weight before immersing.

m: Sample weight after immersing.

III. Results and discussion

Tensile properties

Figure 1 shows the change in stress at break of PVC/MF composites as a function of the number of recycling cycles. It can be seen that recycling has not affected this property for virgin PVC, but an increase in stress is recorded for composite after the first recycling cycle compared with before recycling. This may be due to the reinforcement of the matrix by the filler after recycling and the increase in bond strength between the filler and the matrix [8]. After the second and third recycling cycles, we did not record any significant change in this parameter, which means that recycling does not affect this property.

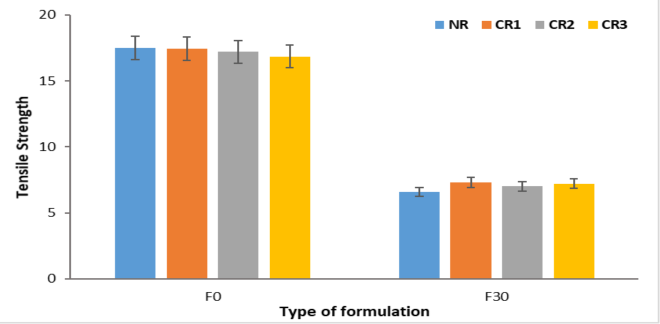

Figure 1. Tensile Strength of pure PVC and PVC/MF composite, before and after recycling.

The variation in Young's modulus of PVC and composites at 30% load is shown in Figure 2. The results show that the Young's modulus increases with the incorporation of the MF into the PVC matrix. This behavior is attributed to the rigid phase of the dispersed fillers, which imparts high rigidity to the PVC matrix. This increase could also be explained by the increased crystallinity of the material after addition of the filler. Sahi et al. [9] have indicated that Young's modulus generally increases with increasing cellulose filler content. However, it was found that the Young's modulus of PVC and the PVC/MF composite for a filler content of 30% increased with the number of recycles.

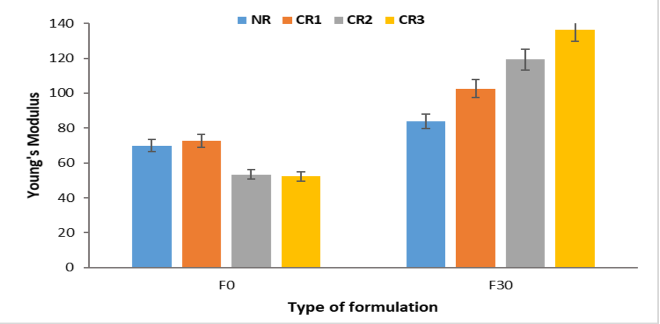

Figure 2. Young's Modulus of pure PVC and PVC/MF composite, before and after recycling.

Hardness

The variation in hardness is shown in Figure 3. It can be seen that hardness increases after the introduction of the filler compared with virgin PVC. This increase can be explained by the fact that adding an organic filler to a material can strengthen its structure and improve its hardness [10, 11]. For the recycled PVC matrix, the hardness increased after the first cycle, then a decrease was recorded from the second cycle onwards. On the other hand, for the various composites, the hardness decreased continuously with the number of cycles. This can be explained by the fact that the recycling process can introduce impurities or defects into the crystalline structure of the material, and recycling can lead to heating cycles that can alter the microstructure of the material, thus influencing its hardness [12].

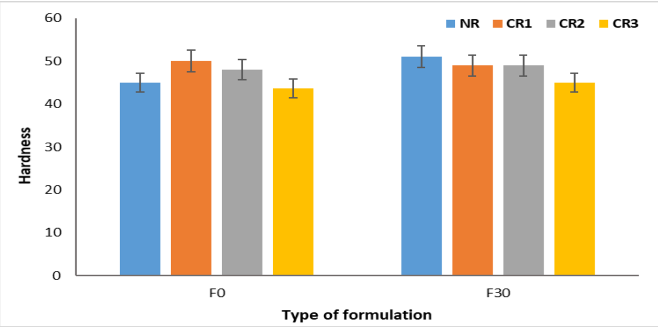

Figure 3. Hardness of pure PVC and PVC/MF composite, before and after recycling.

Water absorbance test

Figure 4 shows the evolution of the water absorption of un-recycled and recycled PVC and PVC/MF composite as a function of immersion time in water. It can be seen that virgin PVC (F0) does not absorb water. This is due to the fact that PVC is a hydrophobic polymer which often records very low water absorption [13]. After recycling, it can be seen that for F30 formulations, an increase in water absorption was recorded from the second recycling cycle. It increased from 11.07% for the F30 NR formulation to 11.27%, 15.87% and 20.80% for the F30 1RC, F30 2RC and F30 3RC formulations respectively. These results can be attributed to the structural changes produced by recycling, which can make PVC/FM composites more permeable to water.

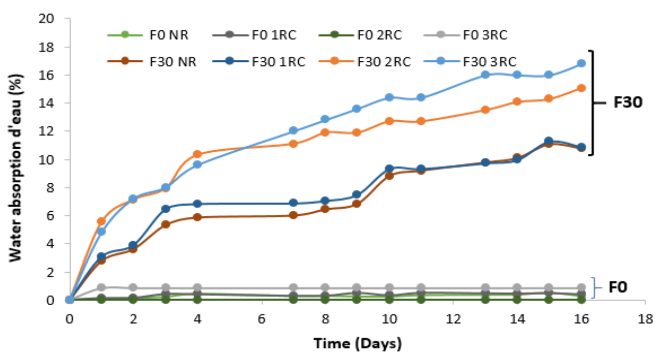

Figure 4. Water absorption of PVC and PVC/MF composites, before and after recycling.

FTIR analysis

The FTIR spectra of corn is shown in Fig. 5. The spectra of maize flour have characteristic profiles to native starch. According to the literature [9, 14]. the chemical functions for each absorption band which appears on the FTIR spectra of starch are given as follow:

There are three characteristic bands of starch between 990 cm^{-1} and 1160 cm^{-1}. attributed to C-O bond stretching. The bands at around 1150 cm^{-1}. 1080 cm^{-1} were characteristic of C-O-H in starch. and the band between 990 cm^{-1} and 1030 cm^{-1} was characteristic of the anhydroglucose ring O-C stretch. The band at 1655 cm^{-1} is attributed to the water adsorbed in the amorphous region of starches. The band at 2920 cm^{-1} is characteristic of C-H stretch. An extremely broad band due to hydrogen-bonded hydroxyl groups appeared at 3400 cm^{-1} which ascribed to the complex vibrational stretches coupled with free. inter and intramolecular bound hydroxyl groups. which made up the gross structure of starch.

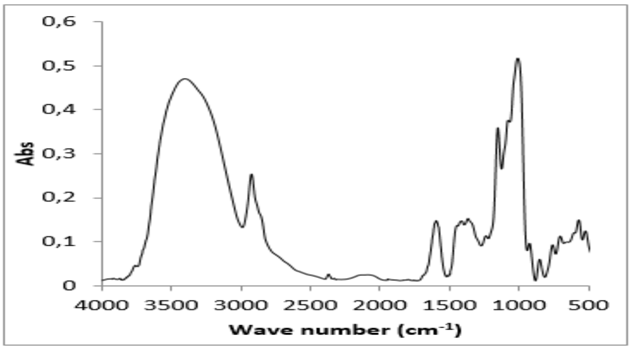

Figure 5. FTIR spectra of MF.

The FTIR spectra of recycled (1R, 2R and 3R) and non-recycled of PVC and PVC/MF composites with a 30% filler content are shown in Figures 6 and 7 respectively. It can be seen from all the spectra that recycling causes neither the disappearance nor the formation of new absorption bands. However, there was an increase in the intensity of certain bands during recycling, namely those centered at 1720 cm^{-1} relating to carbonyls groups.

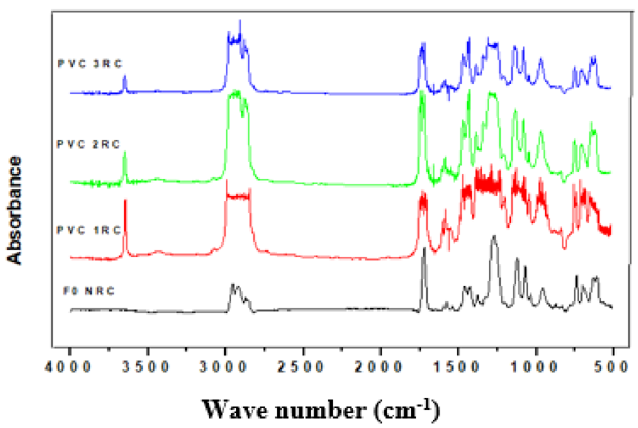

Figure 6. FTIR spectra of PVC before and after recycling.

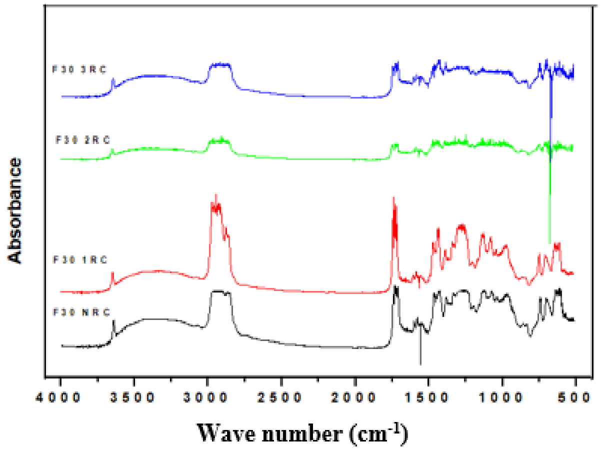

Figure 7. FTIR spectra of PVC/MF before and after recycling.

IV. Conclusions

Study consists on the incorporation of maize flour in PVC matrix and to study the feasibility of its recycling.

Analysis of the experimental results recorded before and after three recycling cycles has enabled us to draw the following main conclusions:

- The stress at break of PVC and PVC/FM composites was not affected by recycling.
- The Young's modulus of PVC decreased after the second and third recycling cycles. However, a continuous increase in stiffness was observed after each recycling cycle at a loading rate of 30%.
- PVC hardness showed an increase after the first recycling cycle, followed by a slight decrease after the second and third recycling cycles. For the filled material, an increase in hardness was observed after each cycle.
- The water absorption test revealed that the water absorption of PVC showed a negligible increase after the three recycling cycles and that an increase in this parameter was recorded after the third and second recycling for the material filled with 30% MF.
- Analysis of the FTIR spectra enabled us to determine the main changes induced by the three recycling cycles for PVC and the PVC/MF composite.

Conflict of interest

The authors declare that they have no conflict for financial interests or personal relationships that how can influence the work reported in this paper.

References

[1] D. Triebert, H. Hanel, M. Bundt, K. Wohnig, Solvent-based recycling. In Circular Economy of Polymers: Topics in Recycling Technologies. ACS Symposium Series 1391. 33-59. 2021.

[2] L. Avérous. Biodegradable multiphase systems based on plasticized starch. Journal of macromolecular Science. Part C: Polymer Review 44. 231-274. 2004.

[3] M. Kim. Evaluation of degradability of hydroxypropylated potato starch/polyethylene blend films. Carbohydrate Polymer 54. 173-181. 2003.

[4] D. Ray. B. K. Sarkar. N. R. Rose. Impact fatigue behavior of vinylester matrix composites reinforced with alkali treated jute fibres. Composites: Part A 33. 233-241. 2001.

[5] Y. Cao. S. Shibata. I. Fukumoto. Mechanical properties of biodegradable composites reinforced with bagasse fibre before and after alkali treatments. Composites : Part A 37. 423-429. 2006.

[6] W. Sikorska, M. Musioł, Zawidlak- B. Węgrzyńska, J. Rydz. End-of-Life Options for (Bio)degradable Polymers in the Circular Economy. Advances in Polymer Technology 6695140, 18 pages. 2021.

[7] P. Krawczak. Recyclage des matériaux composites : Etat de l'art et perspectives. Journée Technique "Le recyclage et la valorisation des matériaux composites", May 2015, Méaulte, France.

[8] R. Scaffaro, A. Di Bartolo, N. Tz Dintcheva. Matrix and Filler Recycling of Carbon and Glass Fiber-Reinforced Polymer Composites: A Review. Polymers 13. 3817. 2021.

[9] S. Sahi, H. Djidjelli. Utilisation de l'amidon comme charge biodégradable dans les matériaux composites polyéthylène basse densité/amidon. Annale de Chimie et Science des matériaux 38 189-202. 2013.

[10] M. B. Uctasli, H. D. Arisu, L. V. Lasilla, P. K. Valittu. Effect of preheating on the mechanical properties of resin composites. European Journal of Dental 2 263-268. 2008.

[11] D. Tantbirojn, S. Chongvisal, D. G. Augustson, Versluis. A Hardness and postgel shrinkage of preheated composites. Quintessence International 42. 51-9. 2011.

[12] I. Ben Amor, O. Klinkova, M. Baklouti, R. Elleuch, I. Tawfiq. Mechanical Recycling and Its Effects on the Physical and Mechanical Properties of Polyamides. Polymers (Basel) 15. 4561. 2023.

[13] A. Asadinezhad, M. Lehocký, P. Sáha, M. Mozetič. Recent Progress in Surface Modification of Polyvinyl Chloride. Materials (Basel). 5. 2937-59. 2012.

[14] S. Sahi, H. Djidjelli, A. Boukerrou. Biodegradation study of bio-corn flour filled low density polyethylene composites assessed by natural soil. Journal of Polymer Engineering 36. 245-252. 2016.