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INVESTIGATIONS REGARDING DEGRADATION BY HYDRATION-DRYING OF SOME BIOCOMPOSITES REINFORCED WITH NATURAL FIBER

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Abstract: Biocomposite materials with improved properties can be obtained by reinforcing a biodegradable matrix with natural fibers. In order to obtain the biodegradable matrix a viable solution are thermoplastic starch based polymers. A method to avoid the inconveniences of using thermoplastic starch (poor mechanical properties and low resistance to moisture) can also be the reinforcement with natural fibers. The composite material studied was obtained by the reactive extrusion of various mixtures of starch, glycerol, poly (butylene adipate-co-terephthalate) (PBAT) and Miscanthus fibers as reinforcing material. This paper presents the results of water uptake and FT-IR spectroscopy investigations for 4 samples of composite material with thermoplastic starch matrix and reinforced with Miscanthus fibers in different concentrations (up to 20%).
Keywords: composite, thermoplastic starch, Miscanthus fibers, degradation

INTRODUCTION

Extending the use of composite materials in almost all areas of activity is influenced by a multitude of factors such as the need for materials with less weight but with certain mechanical properties, environmental and health concerns, the need to reduce energy consumption, sustainable achievement. etc. [5]

The use of renewable materials both as reinforcement elements and as matrix for composite materials contributes to the mitigation of some environmental pollution problems due to synthetic composite materials. Degradable composite materials are composite materials made of a polymer matrix derived from renewable sources (polysaccharides, vegetable oils) or from fossil sources (synthetic polymers such as polyethylene, polypropylene, polyesters, etc.) with natural fiber reinforcement or by-products from agriculture.

One of the most used and studied bio-composite materials is one that uses starch as a matrix [2.6]. To improve the properties of starch based materials biobased polyesters or synthetic biodegradable polyesters such as poly (butylene adipate-co-terephthalate) (PBAT) [7] can be added.

The use of natural fibers (flax, jute, hemp, Miscanthus) to reinforce the biodegradable matrix ensures improved properties of the composite, good mechanical properties, low weight and certain environmental benefits.[1.3.4].

Generally, the manufacturing technologies of degradable composite materials involve machines and processes for obtaining the matrix, preparing the reinforcement components, impregnating or treating the fibers, cutting the fibers, making the reinforcement (different shapes: network, fabric, braid, etc.) injection molding, compression and extrusion, compression-molding, or other processes.

MATERIAL AND METHOD

The samples in this study were prepared by reactive extrusion with a laboratory twin-screw extruder with co-rotating intermeshing, self-wiping screws ZK 25. Collin. (Germany). The raw materials used were: native corn starch obtained from SC ROQUETTE SA Calafat, Romania, having water content of 12.01 %, a density of 0.561 g/cm^3 and an amylose content of 21%. glycerol purchased from SC Nordic Invest SRL Cluj-Napoca with a concentration of 99.5% and a density of 1.262 g/cm^3. Poly(butylene adipate-co-terephthalate) (PBAT) purchased from BASF Company - product code is Ecoflex F Blend C1200 with mass density of 1.25-1.27 g/cm^3, melting point 110-120 °C and melt flow 2.7 - 4.9 g/10 min. and Miscanthus fibers obtained from Arge Miscanthus Romania.

Table 1 shows the ratio of the components used in the formula for the composite material reinforced with fibers.

Table 1. The components ratio in the composite formula

Composite sample	Starch (%Starch+PBAT+ Glyc)	PBAT (%Starch+PBAT+ Glyc)	Glycerol (%Starch+PBAT+ Glyc)	Miscanthus fibers (%Starch+PBAT+ Glyc))
P0	64	13	23	0
P5	64	13	23	5
P10	64	13	23	10
P20	64	13	23	20

The samples were cut into rectangular pieces (Figure 1) and was added distilled water. For each sample we measure the water uptake using a Partner WLC 0.6/B1 analytical balance with a precision of 0.1 g after the excess water was removed by placing the sample on absorbant paper for 1 minute. The modification induced by hydratio/drying processes could have effects on the vibration of the molecular bonds. FT-IR measurements can reveal this kind of modifications. To study degradation due to hydration-drying processes, the samples were hydrate by submerging them in distilled water for 48 hours (Figure 2), after which they were dried for 6 days at 25°C. The FT-IR absorption spectra were recorded with JASCO FTIR 4100 spectrometer in spectral range 400 - 4000 cm^{-1}, resolution 4 and accumulation 100.

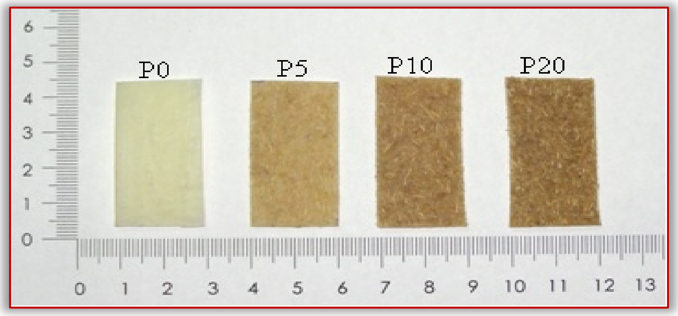


Figure 1 – Composite material samples
with different fibers content

Figure 2 –Samples after 48 h of submerging
in distillate water

RESULTS

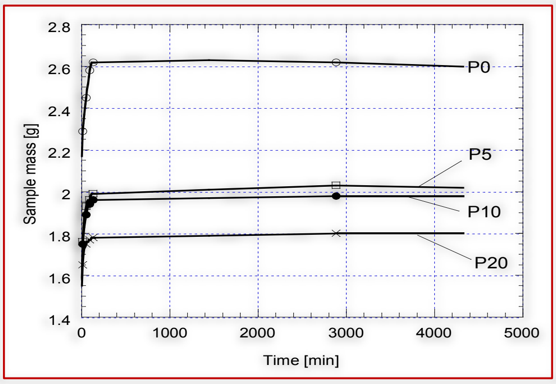
Table 2 presents the samples mass with uptake water absorbed after 24/48/72 h of hydration by four composite materials samples with different formula presented in Table 1. The sample P0 which has the formula with no fibers (0%) reached the maximum water uptake with higher velocity (in 24 h) and that started to degrade.

The sample P5 which has the formula with lower fibers content (5%) absorbed the highest amount of water (~28% of sample mass) in 48 h and then started to degrade. The lowest quantity of water (~16% of sample mass) was absorbed by the sample P20 (with 20% Miscanthus fiber) in 48 h.

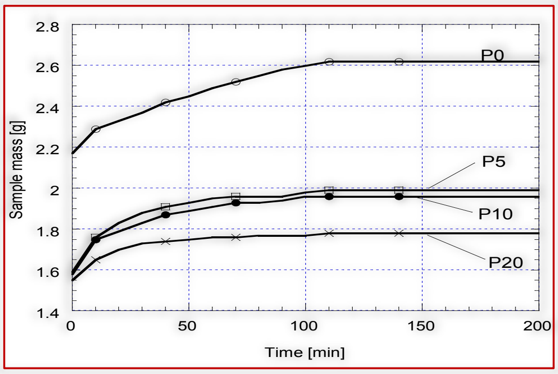
Table 2. The sample mass after hydration and water uptake

Sample	P0	P5	P10	P20
Dry sample [g]	2.17	1.59	1.58	1.55
Distilled water [g]	20	20	20	20
Sample after 24 h	2.63	2.01	1.97	1.79
Sample after 48 h	2.62	2.03	1.98	1.80
Sample after 72 h	2.60	2.02	1.98	1.80
Water uptake [g]	0.46	0.44	0.40	0.25

As can be seen from Figure 3. B, all samples regardless of the fiber content absorb most of the water in the first 3 hours, but the samples reinforced with Miscanthus fibers absorbed the highest amount of water slower than the sample with no fibers (P0).



a)



b)

Figure 3 –Sample mass with water uptake after 72 h – a;
detailed view for the first 3 hours – b

Also the sample with higher fiber content absorbed the lowest amount of water. The samples reinforced with different ratio of fibers keep their integrity for 72 h but the sample P0 (no fibers reinforcement) start to degrade after 24 h (Figure 3.a) Also the sample with higher fiber content absorbed the lowest amount of water. The samples reinforced with different ratio of fibers keep their integrity for 72 h but the sample P0 (no fibers reinforcement) started to degrade after 24 h (Figure 3.a)

In order to study the samples by FTIR spectroscopy we recorded and compared the IR spectra of samples P0-P20 before and after hydration-drying. Due to the observation regarding water uptake, we analyzed the spectra of samples P0 (no fiber content). P5 (5% fibers absorbed the highest amount o water) and P20 (20% fibers absorbed the lowest amount o water).

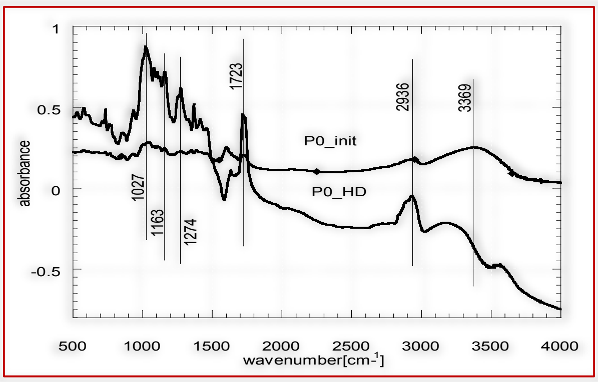


Figure 4 – FT-IR spectra of P0 sample: initial state-P0_init and after hydration-drying

CONCLUSIONS

- Biocomposite materials with improved properties can be obtained by reinforcing a biodegradable matrix with natural fibers.
- Samples were prepared by reactive extrusion with a laboratory twin-screw extruder.
- Raw materials used are: native corn starch, glycerol, PBAT and Miscanthus fibers.
- All the samples regardless of the fiber content absorb most of the water in the first 3 hours.
- Miscanthus fiber content decrease the speed of water uptake.
- The sample with higher fiber content absorbed the lowest amount of water.

Note

This paper is based on the paper presented at ISB-INMA TEH' 2017 International Symposium (Agricultural and Mechanical Engineering), organized by University "POLITEHNICA" of Bucharest – Faculty of Biotechnical Systems Engineering, National Institute of Research-Development for Machines and Installations Designed to Agriculture and Food Industry – INMA Bucharest, Scientific Research and Technological Development in Plant Protection Institute (ICDPP), National Institute for Research and Development for Industrial Ecology – INCD ECOIND, Research and Development Institute for Processing and Marketing of the Horticultural Products "HORTING" and Hydraulics, Pneumatics Research Institute INOE 2000 IHP, University of Agronomic Sciences and Veterinary Medicine of Bucharest (UASVMB) – Faculty of Horticulture and Romanian Society of Horticulture (SRH), in Bucharest, ROMANIA, between 26 – 28 October, 2017.

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