

BROOM FIBERS AS REINFORCEMENT FOR THERMOPLASTIC MATRICES

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ABSTRACT : Vegetable fibers deriving from the basts of broom plants have been used as reinforcement for two polypropylenic matrices : a conventional isotactic polypropylene (iPP) and a maleate modified one (iPPMA).

Before mixing the fibers were subjected to two different extraction procedures : a conventional alkaline treatment and an innovative steam explosion process.

Afterwards, the composites obtained by compression moulding were thermally, morphologically, and mechanically characterized. Moreover, water absorption tests, to examine the behaviour of these materials in wet conditions, were also performed.

INTRODUCTION

According to their high specific properties, low cost, biodegradability and renewability, the vegetable fibers can be utilized as reinforcements for plastic materials ^(1,2). The possibility of these fibers to act as good reinforcement agent are dependent on their cellulosic content, fiber size and a strong fiber/matrix interfacial adhesion ⁽³⁻⁵⁾.

In the present work fibers richer in cellulose content, deriving from the branches of broom plant, are tested to reinforce polypropylene matrices. The choice of broom fibers was effected owing to their good mechanical properties and easy availability, especially in mediterranean lands. Moreover the broom needs reduced cultivation care after planting and allows yearly harvesting cycles.

The aim of our work was the achievement and characterization of new composite materials that can be used in those applications where lightness, biodegradability, good properties and low costs are required.

The obtained broom fiber composites have been thermally and morphologically characterized and their mechanical behaviour was compared to that of the homologous composites obtained by using short glass fibers.

Particular attention has been addressed to the study of interfacial adhesion between the matrices and the two different types of extracted broom fibers.

Finally, water absorption tests have been also carried out in order to evaluate the mechanical worsening due to the penetration of water molecules in the composite structure.

RESULTS AND DISCUSSION

The composites were prepared by using two commercial polypropylenes : an isotactic polypropylene (iPP) and a modified one obtained by grafting on the backbone a small amount (~1.6% wt.) of maleic anhydride (iPPMA) ⁽⁶⁾.

To achieve an effective reinforcement action of the broom fibers, they were previously undergone to two extraction procedures :

- a) traditional chemical extraction by using an alkaline solution ;
- b) steam explosion treatment.

The fibers coming from the two different extraction processes were compounded with polypropylenic matrices by melt mixing using a Brabender-like apparatus (Rheocord EC, Haake Inc.) operating at 190 °C for 10 min. The material so obtained was utilized to prepare 1 mm and 3 mm sheets of composites by compression moulding at 190°C.

Different composition of polymer and fiber were utilized to prepare specimens. A complete list of all the samples is reported in table 1.

The thermal characterization of composites was effected by using Differential Scanning Calorimeter (DSC), by using a scan rate of 10 °C/min. The parameters calculated were : the apparent melting temperature (T'_m), the cristallization temperature (T_c), the cristallinity indices (X_c), and the glass transition temperature (T_g).

It was observed a slight increase in T'_m and X_c for the iPPMA-based composites with respect to the neat matrix. This fact can be probably attributed to the strong interactions between cellulosic fibers and iPPMA matrix. As matter of fact, the fibers may "extract" selectively, from the polypropylenic backbone, the maleic anhydride groups (constitutional defects), leading to composites having a matrix characterized, on average, by a higher degree

of constitutional regularity, with respect to the bulk in the neat iPPMA, and consequently higher T'_m and X_c .

Moreover, it was also observed an increase of cristallization temperatures for the same composites with respect to the neat matrix, attributable to the nucleating ability of the cellulosic fiber on the polymeric materials.

Materials	Composition (% wt.)	Code
iPP	100/0	iPP neat
iPPMA	100/0	iPPMA neat
iPP/NaOH extracted fiber	50/50	iPP/Broom 50/50
iPP/exploded fiber	50/50	iPP/Broom SEP 50/50
iPPMA/NaOH extracted fiber	90/10	iPPMA/Broom 90/10
iPPMA/NaOH extracted fiber	70/30	iPPMA/Broom 70/30
iPPMA/NaOH extracted fiber	50/50	iPPMA/Broom 50/50
iPPMA/exploded fiber	90/10	iPPMA/Broom SEP 90/10
iPPMA/exploded fiber	70/30	iPPMA/Broom SEP 70/30
iPPMA/exploded fiber	50/50	iPPMA/Broom SEP 50/50

Tab. 1 : Summary of the prepared composites and their compositions.

Tensile tests were performed on 1mm thickness specimens, by using an Instron machine. The calculated values of tensile parameters for all the studied composites were : Young's tensile modulus (E), tensile strenght at break (σ_b) and percentual elongation at break (ϵ_b) (see tab. 2). The following considerations can be deduced :

- i) iPP-based composites show a tensile Young's modulus comparable to that of iPP homopolymer, on the contrary the tensile strenght at break and the percentual elongation at break strongly decrease with respect to the neat matrix ; these findings are probably due to a weak fiber/matrix interfacial adhesion ;
- ii) on the contrary iPPMA-based composites, reinforced with NaOH extracted fibers exhibit a strong increase of both Young's elastic modulus and tensile strenght at break : these performances can be probably ascribed to a stronger fiber/matrix interface, caused by the interactions between maleic anhydride grafted on polypropylene and -OH groups on cellulosic fibers ;

iii) the iPPMA-based materials reinforced with SEP extracted fibers show only a slight worsening in tensile strenght at break, while elastic modulus remains almost costant respect to the neat iPPMA matrix.

Materials	E (GPa)	σ_b (MPa)	ϵ_b (%)
iPPMA neat	1.2	18.3	2.0
iPPMA/Broom 90/10	1.4	22.9	2.1
iPPMA/Broom 70/30	1.8	27.6	2.2
iPPMA/Broom 50/50	2.5	29.2	1.5
iPPMA/Broom SEP 90/10	1.2	15.0	1.6
iPPMA/Broom SEP 70/30	1.4	15.1	1.3
iPPMA/Broom SEP 50/50	1.5	11.2	0.8
iPP neat	1.0	16.5	5.8
iPP/Broom 50/50	0.8	9.5	1.6
iPP/Broom SEP 50/50	1.3	3.2	2.2

Tab. 2 : Young’s elastic modulus (E), tensile strenght at break (σ_b) and percentual elongation at break (ϵ_b) of all the samples.

To explore the possibility for a more diffuse utilization of broom fiber composites in substitution of short glass fiber materials ⁽⁷⁾, a comparative analysis about properties of these two kinds of composites was performed, and the results reported in figures 1a and 1b. In these graphics Young’s modulus and tensile strenght at break, normalized respect to the materials density are reported respect to the fiber content for these two series.

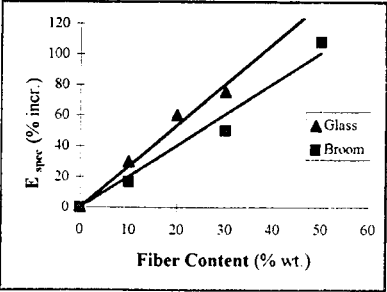


Fig. 1a : Tensile tests - Percentual increase in Young’s elastic modulus for glass reinforced and NaOH extracted broom fiber reinforced composites both based on a polypropilenic matrix

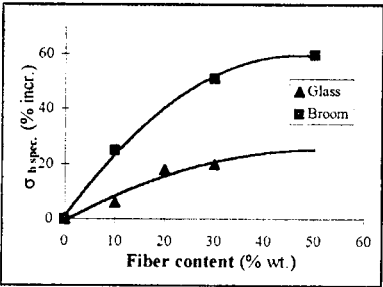


Fig. 1b : Tensile tests - Percentual increase in tensile strenght at break for glass reinforced and NaOH extracted broom fiber reinforced composites both based on a polypropilenic matrix

The results of the mechanical behaviour can be interpreted on the basis of the surface analysis performed by Scanning Electron Microscopy on the notched specimens.

Figs. 2(a, b) show the SEM micrographs relative to the fracture surfaces of the composites reinforced with 50% of broom fibers having as matrix iPPMA. From these figures the following can be noted that the iPPMA based composites show a good interfacial adhesion between the two phases for both types of treated fibers. In particular, the SEM micrograph relative to the iPPMA/broom SEP (fig. 2b) seems to present a higher degree of interaction between matrix and fiber with respect to that found in the case of iPPMA/broom NaOH extracted (fig. 2a): infact there is no evidence of pulled-out fibers, wich appear to be well embedded inside to the plastic matrix ; a different feature is observed for iPPMA/broom NaOH extracted, where some pulled-out phenomena from the surface of material during the fracture are detected.

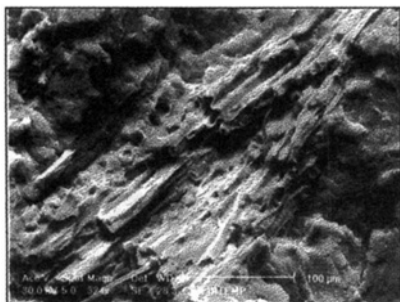


Fig. 2a : SEM micrographs - Fracture surface of iPPMA/broom 50/50 (324×).

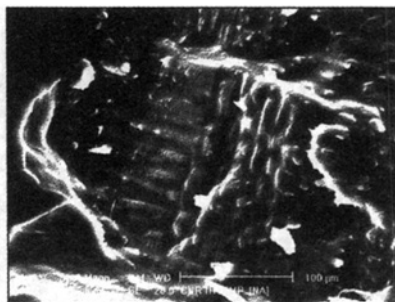


Fig. 2b : SEM micrographs - Fracture surface of iPPMA/broom SEP 50/50 (325×).

Finally, water absorption tests have been also carried out to evaluate the mechanical worsening due to the penetration of water molecules in the composite structure. In fact, one of the most undesirable properties of vegetable fibers is their dimensional instability due to moisture absorption. This phenomenon is mainly caused by the hydrogen bonding between water molecules and the hydroxyl groups present in the cellulose structure. Clearly, a strong fiber/matrix interfacial adhesion can avoid, almost partially, the water penetration reducing the hygroscopicity, and, consequently, the worsening in the mechanical performances of materials.

The water absorption tests on our samples were performed according to the ASTM D570 method, by immersion in aqueous middle of a 1 mm sheet for 1 month, periodically measuring the increasing in weight of the samples.

The results, shown as the percentual weight increase in function of immersion time, are summarized in figure 3. The following can be deduced :

- i) as expected, the homopolymers (iPP and iPPMA) show the lowest water absorption ;
- ii) the two iPP-based materials (iPP/Broom 50/50 and iPP/Broom SEP 50/50) are characterized by the shortest saturation time : as matter of fact, the maximum water absorption was obtained after an immersion time of about 200 hr. ; moreover, for these samples, the highest saturation percentage (~12% wt.) was found ; clearly these findings can be ascribed to the poor fiber/matrix adhesion as already shown by their mechanical behaviour ;
- iii) the two iPPMA-based composites reinforced with NaOH extracted and SEP treated fibers show different amount of water absorbed. In the case of SEP extracted fiber composites a lower quantity of water penetrates with respect to the NaOH extracted fiber materials. This finding is probably due to the lower cristallinity index of these latter fibers with respect to the exploded ones ⁽⁸⁾. Moreover, the good fiber/matrix interfacial adhesion, obtained by using SEP extracted fibers as reinforcements for iPPMA matrix, can also contributes to reduce the water absorption, as reported elsewhere ⁽⁹⁾.

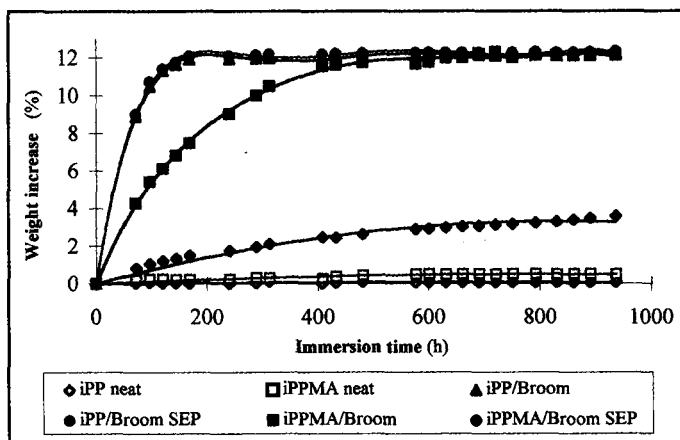


Fig. 3 : Water absorption tests - Weight increase of the samples in function of time.

Tensile tests, both on wet samples and dried (50 °C for 24 hr. in oven) were performed. They confirm that a strong interface between cellulosic fiber and polypropylenic matrix, leads to a reduction of the penetration of water, allowing only a slight worsening of tensile performances. In fact, the materials that show a low water absorption, such as composites reinforced with SEP fibers, presents only a slight decrease in tensile performances, and the subsequent treatment in oven seems to restore the initial properties, indicating only a superficial water absorption. On the other hand, materials that are characterized by a large amount of absorbed water, such as iPP-based composites, show also a large reduction in mechanical properties ; these features are not restored after oven treatment, indicating a deep penetration of water in composite structure through the cellulosic fiber.

CONCLUSIONS

In this paper the performances of iPP and iPPMA reinforced with broom fibers, extracted by using both a conventional alkaline treatment and an innovative steam explosion process, were studied.

The following conclusions from the results above reported can be drawn :

- a) broom fibers obtained by steam explosion seem to produce a better fiber/matrix interfacial adhesion with respect to those extracted by alkaline treatment, as clearly shown by SEM analysis and water absorption tests. Nevertheless, the mechanical behaviour of composites reinforced with NaOH extracted broom fibers is superior with respect to that of the SEP fiber composites ; this findings can be probably attributed to the minor destructuration produced by the chemical treatment that allows to obtain longer and bundled fibers ;
- b) the utilization of an iPPMA matrix as alternative to a conventional polypropylene, as previously shown ⁽⁹⁾, produces composites with good final properties. This occurs because of the presence of maleic groups that are able to create a strong interface between matrix and cellulosic material ;
- c) the utilization of SEP fibers as reinforcement for iPPMA-based composites contribute to reduce the penetration of water in the structure with respect to the NaOH extracted fibers : this effect can be due both to the higher crystallinity of the SEP fibers, that reduces the water diffusion through the amorphous region, and to a better fiber/matrix interfacial adhesion ;

d) the NaOH extracted fiber iPPMA-based composites present specific mechanical properties comparable to those of analogous short glass fibers reinforced materials ; this can lead, in a next future, to an effective possibility for broom fibers to be used as alternative materials to the standard synthetic reinforcement, also owing to their biodegradability, lightness and low cost.

In this way it was demonstrated the possibility, for treated broom fibers based biocomposites, to be used as alternative material when light, inexpensive and partially biodegradable products are requested.

Finally, the utilization of vegetable fibers can upgrade cultivations deriving from rural industry today largely underestimated.

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