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## Numerical study of mechanical behaviour of a polypropylene reinforced with Alfa fibres

Montassar ZRIDA\*1, Hervé LAURENT<sup>2</sup> and Gérard  $\mathrm{RIO}^2$ 

 $^{1}\mathrm{Centre}$ National de Recherches en Sciences des Matériaux, LVMU, Tunisie

<sup>2</sup>Univ. Bretagne-Sud, EA 4250, LIMATB, France

## Abstract

The main purpose of this work consists of the mechanical characterization of a copolymer PolyPropylene (PP) filled with natural Alfa fibres. The elaboration of the PP-based composite reinforced with these natural fibres is explained. The mechanical behaviour of these composite blends has been analysed. Contrary to classical studies of the mechanical characterization of these kinds of materials which focus only on monotonic tensile tests, cyclic loading/unloading tests and loading/unloading tests interrupted by relaxation steps have been investigated. These tests enable us to compare the mechanical response of the virgin PP and the PP filled with Alfa fibres (Alfa/PP). Reinforcement by these natural fibres shows an impact in the improvement of the mechanical properties of this composite. All these experimental results constitute a data base used to identify the material parameters of a phenomenological constitutive Hyperelasto-Visco-Hysteresis (HVH) model. These tests also provide indications about the hyperelastic, viscous and hysteretic stress contributions of the HVH model with these materials during cyclic loading.

## Keywords

Polymer-matrix composites, Polypropylene, Alfa fibres, Mechanical properties, Finite element analysis

<sup>\*</sup>Corresponding author: Montassar ZRIDA, Centre National de Recherches en Sciences des Matériaux, LVMU, BP73, 8027, Soliman, Tunisie. E-mail: montassar.zrida@gmail.com

## $_{1}$ 1 Introduction

<sup>2</sup> Composite materials are increasingly used in daily life due to their low production cost, light <sup>3</sup> weight and efficient performance. Among these materials are the thermoplastics reinforced <sup>4</sup> with natural fibres such as Bamboo, Jute, Kenaf, Flax, Sisal, Hemp, Alfa, etc. Their use <sup>5</sup> has gained significant interest during the last decade because of their biodegradability. These <sup>6</sup> natural fibres offer a number of advantages over other conventional fibres in the form of: abun-<sup>7</sup> dancy, renewability, and relatively low cost. These benefits present an opportunity to make an <sup>8</sup> envorinmentally friendly material with excellent mechanical properties [1, 2].

Nevertheless, the manufacture of these materials is difficult due to poor compatibility between a the hydrophobic polymer matrix and the hydrophilic cellulose fibres. This poor compatibility affects the mechanical properties that strongly depend on the interfacial adhesion between components. Therefore, a number of studies in the modification of the fibre's surface properties were introduced to improve their adhesion with a different polymer matrix [3]. Some of these treatments have a physical nature and some of them are of a chemical nature. These days, more and more natural fibre resources for products are being studied for polymer reinforcement, and the number of the studies and researches that have been published during the last decade reflects the growing importance of these new bio-composites [3, 4, 5, 6, 7]. Since the use of these materials is growing, studying their mechanical properties and behaviour becomes necessary to ensure that their characteristics will be reliable and predictable. 

The aim of the present paper is to study the mechanical behaviour during cyclic tests of a thermoplastic reinforced with chemically treated natural fibres extracted from the Alfa plant. Alfa fibres are available in large quantities in North Africa. They have good mechanical prop-erties compared to other natural fibres [8] and they are mostly used to produce paper. Paiva et al. [9] have studied the tensile properties of an single filament of Alfa fibre, resulting in values of approximately 20 GPa and 250 MPa for tensile modulus and yield strength, respectively. Arrakhiz et al. [10] have investigated the effect of chemical modification (alkali treatment, etherification treatment and esterification treatment) on the Alfa fibre's surface, and its impact on mechanical and thermal properties of composites. However, they only analysed the Young's modulus and tensile strength. To improve the mechanical knowledge of this kind polymer rein-

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forcement, it is important to analyse their mechanical responses during more complicated tests,
such as loading-unloading cyclic tests.

In this study, to characterize the mechanical performance of a PP filled with Alfa fibres, a commercial grade of copolymer polypropylene PPC7712 is used as a matrix. In our previous works, this material was studied in static tensile tests [11], dynamic tensile tests [12] and under multiaxial loading conditions [13].

Monotonic tensile tests, cyclic loading/unloading tests and loading/unloading tests interrupted by relaxation steps (multi-step relaxation test) were carried out to investigate the mechanical behaviour of the Alfa/PP composite. The first objective of these tests is to have a full characterization of the stress-strain responses of the PP and Alfa/PP during the uniaxial tensile cyclic tests. Thus, the second objective of this study is to analyse the mechanical impact of the reinforcement with these natural fibres on these complicated cyclic tests.

Finally, an original phenomenological model, called Hyperelasto-Visco-Hysteretic (HVH) was used to simulate the mechanical behaviour of these materials during these tests. This model, written in 3D and implemented in an in-house code HEREZH++ [14], is based on the super-imposition of three stress components which correspond to linear viscoelastic, hyperelastic and pure hysteresis behaviour. In contrast to previous studies based on the same model [11, 15], a new identification method of material parameters is performed in this paper. This method, pre-sented in [16], but with a fluoro-elastomer, is based only on a cyclic tension test interrupted by relaxation steps (multi-step relaxation test). It does not require an optimization procedure by a least-square method to minimize the experimental data and prediction of the test. The main advantage of this identification method is that, using only this multi-step relaxation test, hy-perelasticity, hysteresis and viscosity contributions of the HVH model are discriminated, which results in the substantially easy and simple identification process of the material parameters. To verify the suitability of the material parameters obtained, experimental tests that were not used for the identification are simulated. A good performance of the proposed model is shown by a comparative analysis between the predictive solution and experimental data. 

## <sup>57</sup> 2 Materials and processing of composites

The composites were prepared using a commercial grade heterophasic copolymer polypropylene, named PPC7712, provided by Total-Petrochemicals. This copolymer is recyclable with good mechanical properties, excellent impact strength and is easy to inject. Some thermo-mechanical characteristics of the PPC7712 are listed in Table 1 [11]. The Alfa fibres used, readily available in the dry region of North Africa, were extracted from plants from the central west of Tunisia. The Alfa plant is the Arab name of "esparto grass" or "stippa tenacissima" plant [9]. These fibres were characterized by good thermal and mechanical properties compared to other natural fibres. Characteristics and the composition of Alfa fibres in comparison to other natural fibres can be found in [8, 9, 10, 17, 18]. 

### 67 2.1 Chemical treatment of the Alfa fibres

To improve compatibility with a hydrophobic polymer and eliminate the majority of its noncellulosic components [19], Alfa fibres were chemically treated according to the standard pro-cedure proposed in [10, 20, 21]. To separate the fibres and clean their surfaces, whole fibres were first immerged in salted water with a salt (NaCl) concentration of 35 g/l at 60° C for a duration of 24 hrs. The fibres were then placed in an aqueous sodium hydroxide solution (NaOH) with a concentration of 1.6 mol/l (corresponding to 64 g/l) for a duration of 48 hrs for each 35 g of fibres, with the objective of eliminating the lignin and pectin elements covering the fibre's surface. Subsequently, to neutralize the sodium hydroxide solution, they were placed in an acetic acid solution (100 ml for each 35 g of fibres). 

Finally, the Alfa fibres were washed with distilled water to remove any excess of sodium hydroxide to obtain a final pH=7. Each step of this procedure was followed by a drying period in an oven at 60° C for a duration of 24 hrs.

#### 2.2 Alfa fibres characterization

The tensile strength and Young's modulus of treated Alfa fibres were determined according to the ASTM D3379-75 standard test method for high-modulus single filament materials. Elemen-

tary fibres were separated from their fibre bundles by hand, and then mounted on 2 mm thick cardboard mounting cards with 10 mm holes punched into them, such that the punched hole diameters determined the fibre gauge length. Fibres were positioned to bridge the hole, and were secured in place with glue. This method follows the procedure proposed by the example in [22]. Mounted fibres were inspected under an Olympus BX60F5 metallurgical microscope to ensure that only a single fibre was present on each card and to determinate the average diameter of the fibre. Thirty fibre samples were analysed. For each fibre, three points were measured: two at the extremities of the fibre and one point on the middle of fibre. The average diameter of the fibres was equal to  $185.2 \pm 39 \mu m$ . The cross section area was also measured for the thirty fibres. The distribution of this experimental value is given in Figure 1. The average cross section area is of 28110  $\mu m^2$  (green line in Figure 1). 

The mounted single fibre was placed in the grips of an MTS tensile testing machine and the supporting sides of the mounting cards were carefully cut using a hot-wire cutter. Tensile tests were performed until failure at a rate of 5 mm/min using a load cell of 50 N at room temperature (20° C). With the average cross section area previously given, an average Young's modulus of  $3.84^{\pm 1.3}$  GPa, a stress and a strain at break of  $173.4^{\pm 5}$  MPa and  $5^{\pm 0.5}\%$  respectively, were found.

## **2.3** Elaboration of the composite material

Composites reinforced with Alfa fibres were prepared by compounding neat polypropylene. Short fibres are of a length between 1 and 2 mm. The fibre percentage in the matrix was fixed at 15 wt.%. The PP and the Alfa fibres were mechanically mixed in, using a single screw extruder at temperature of 190° C. After cooling at room temperature, the strands from the extruder were pelletized. Then, using these mixed pellets, tensile specimens were moulded using a Battenfeld HM 80 injection-moulding machine. The geometric properties of the tensile specimen follow the ISO 3167 norm.

## <sup>108</sup> 2.4 Determination of the diameter and length of fibres after extru-<sup>109</sup> sion and after injection

To find out the influence of the extrusion and injection processes on the dimensions of the fibres, a microscopic analysis of the Alfa/PP composites after these two steps of elaboration has been performed. Film samples were prepared using a hydraulic press under a pressure of 3 bars for a duration of 2 minutes at a temperature of 190°C, from (i) the pellets obtained after extrusion and (ii) a portion of a tensile specimen after injection. From these film samples, 250 measurements of diameter and length of fibres were recorded. The dimensions (diameter and length) of the Alfa fibre were reduced after each process. Initially, the raw fibres have an average diameter of 185  $\mu$ m which decreases from 70.8  $\mu$ m after extrusion to 40  $\mu$ m after injection step. The raw fibres have an initial length between 1 and 2 mm which decreases from 444.1  $\mu$ m after extrusion to 273  $\mu$ m after injection step. 

## <sup>120</sup> **3** Mechanical characterisation

To compare the mechanical behaviour of the virgin PP and Alfa/PP composite, several tests were performed under tension: monotonic tensile tests, cyclic loading/unloading tensile tests and loading/unloading tensile tests interrupted by relaxation phases (multi-step relaxation tests). The first aim of these tests is to better understand the macroscopic mechanical behaviour of these composites during the cyclic test which is rarely analysed. The second objective is to have a maximum amount of data to compare the simulation results using the HVH model with these experimental tests.

Tensile tests were carried out using the universal testing machine Instron 5560, equipped with a standard load cell of 10 kN and an extensometer with an initial gauge length of  $L_0 = 10$ mm. These tests were performed with an engineering strain rate of  $\dot{\varepsilon} = 4.10^{-4} \text{s}^{-1}$ . For each test, at least three specimens are used to check the reproducibility of the results. The curve that passes at the maximum through the middle of the three experimental curves is chosen as representative of the behaviour.

<sup>134</sup> In this paper, when not stated otherwise, all the experimental curves presented were based

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<sup>135</sup> on the Cauchy stress and Almansi strain. Assuming that the transformation is isovolume, the <sup>136</sup> Cauchy stress is given by:  $\sigma_{Cauchy} = \frac{F}{S_0} \frac{L}{L_0}$ , with L is the final length of the extensometer,  $L_0$ <sup>137</sup> is the initial length of the extensometer, F is the measured load and  $S_0$  the initial specimen <sup>138</sup> section. The Almansi strain is obtained by:  $\varepsilon_{Almansi} = \frac{1}{2} \frac{L^2 - L_0^2}{L^2}$ .

#### <sup>139</sup> 3.1 Monotonic tensile tests

The first experiment performed was the standard monotonic tensile test with a constant strain rate  $\dot{\varepsilon}$ . A comparison of the two material responses is presented in Figure 2. In the case of PP, a break never occurred at a strain of 0.35. Young modulus and the maximum stress have been increased for the Alfa/PP (E=1.103 GPa with PP and E=1.766 GPa with Alfa/PP). However, the Alfa/PP only supports a small deformation (near 0.08) and the break occurs quicky after the maximum stress is reached.

A specimen surface of the Alfa/PP was observed by SEM after the break at the end of a tensile test (see Figure 3). The fibre surface was damaged after the break. This result shows a relatively poor adhesion between the polymer matrix and the fibres at this strain level. This adhesion can be improved with a stronger chemical treatment but it will be in contradiction with the environmental point of view whose the aim is to achieve interesting composite characteristics with low-treated fibres. To compare experimentally and numerically the two materials in the same conditions, the maximum strain imposed in the following tests will be restricted to 0.05.

#### <sup>153</sup> 3.2 Loading-unloading tests with 50 cycles

To study the mechanical behaviour of the two materials under cyclic deformation loads, a further tension test was performed with cyclic loads. During cyclic test presented in Figure 4, the specimen was stretched up to the maximum engineering strain of 4.6% and then unloaded down to zero stress. To clearly compare the mechanical behaviour of PP and Alfa/PP, only the first, the tenth and the last cycle are shown in Figure 4.

<sup>159</sup> With the two materials, the responses obtained during loading and unloading are strongly <sup>160</sup> nonlinear. The remanent strain increases after each cycle. The maximum stress also decreases <sup>161</sup> at the end of each cycle for a given strain level. The area of the hysteresis loop is reduced after

<sup>162</sup> each cycle and is almost similar between the two materials.

#### <sup>163</sup> 3.3 Multi-step relaxation tests

<sup>164</sup> During an loading-unloading test, the test was interrupted at prescribed levels of axial strain <sup>165</sup>  $\varepsilon_r$  for a given holding time of 900 s (see Figure 5). It is assumed that the stress evolution as <sup>166</sup> function of time is much lower after 900 s, because of this the evolution was ignored after 900 <sup>167</sup> S.

During this holding time, the load evolution was stored. The loading phase was interrupted by 3 relaxation phases at strain levels of  $\varepsilon_r = 1$ , 2.8 and 4.6%. The unloading phase is interrupted by 2 relaxation phases at strain levels of  $\varepsilon_r = 3.7$  and 2.8%. The multi-relaxation tests were performed at a strain rate of  $\dot{\varepsilon} = 4.10^{-4} \text{s}^{-1}$ .

The results show that the stress decreases during the holding time in the loading path while an increase is observed during the holding time in the unloading path. Note that, during the holding time, the stress evolves towards a stabilized value which gives the relaxed-response. This kind of test constitutes an attractive way of identifying the material parameters corresponding to the three stress components included in the HVH model (see section 5). The amplitude of the relaxation phase at a given strain level  $\varepsilon_r$  is more large for the Alfa/PP than the PP, as indicated by the distances 1 and 2 in Figure 5(a), which is due of the fact that during the loading step, the relaxation phase is higher with the Alfa/PP, whereas during the unloading step, the relaxation phase is almost similar between the two materials. 

## <sup>181</sup> 4 Hyperelasto-Visco-Hysteresis behaviour model

The macroscopic behaviour of solid materials is most often the result of simultaneous actions, at the relevant scales of reversible physical phenomena and irreversible physical phenomena. To describe the macroscopic properties of the global behaviour related to these physical phenomena, an assumption has been adopted which states that the internal stress power system results from the decomposition of this power in several contributions in stress. Each of these contributions is related to a particular physical phenomenon. From the results of the previous tests, the mechanical response of the two materials studied exhibits three classical main phenomena, namely a reversible elastic phase which occurs at the onset of the loading (hyperelastic response), a strain rate dependent phase which can be described in terms of the viscosity, and an irreversible plastic phase (hysteresis response) which occurs during the loading-unloading cycles.

If the processes of hysteresis, reversibility and viscosity are simultaneous, it is assumed that the stress power or the rate of internal mechanical work  $\mathcal{P}_{int}$ , which describes the response of a material domain  $\mathcal{D}$ , occupying a volume v, at time t, effected by the stress field  $\sigma$  can then be split into three contributions as, the internal stress powers of the hyperelastic  $\mathcal{P}_e$ , viscoelastic  $\mathcal{P}_v$  and hysteresis components  $\mathcal{P}_h$  as follows:

$$\mathcal{P}_{int} = \mathcal{P}_{e} + \mathcal{P}_{v} + \mathcal{P}_{h}$$

$$\int_{\mathcal{D}} (\boldsymbol{\sigma} : \boldsymbol{D}) \, dv = \int_{\mathcal{D}} (\boldsymbol{\sigma}_{e} : \boldsymbol{D}) \, dv + \int_{\mathcal{D}} (\boldsymbol{\sigma}_{v} : \boldsymbol{D}) \, dv + \int_{\mathcal{D}} (\boldsymbol{\sigma}_{h} : \boldsymbol{D}) \, dv \qquad (1)$$

$$= \int_{\mathcal{D}} ((\boldsymbol{\sigma}_{e} + \boldsymbol{\sigma}_{v} + \boldsymbol{\sigma}_{h}) : \boldsymbol{D}) \, dv$$

where  $\sigma$  is the Cauchy stress tensor, D is the strain rate tensor,  $\sigma_e$  is the stress associated with the reversible hyperelastic behaviour,  $\sigma_v$  is associated with the viscoelastic behaviour and  $\sigma_h$ with the non-viscous hysteresis behaviour, always irreversible and of the elasto-plastic type.

The strain rate D is independent on these phenomena and the total stress applied to the material is, therefore, the result of the superimposed hyperelastic, viscoelastic and hysteresis stress components:

$$\boldsymbol{\sigma} = \boldsymbol{\sigma}_{\boldsymbol{e}} + \boldsymbol{\sigma}_{\boldsymbol{v}} + \boldsymbol{\sigma}_{\boldsymbol{h}} \tag{2}$$

This basic assumption gives our macroscopic approach a strong relationship with the physical processes involved. However, it leads to a conceptual break from constitutive and thermodynamic views compared to the classical elasto-plasticity approach, which advocates the decomposition of the total strain  $\varepsilon$  or of the strain rate D. However, this approach interests more and more authors, as the literature shows that is becoming increasingly common, especially for polymer behaviour modelling [23, 24, 25, 26].

The fundamental assumption (eq. 2) which involves three basic contributions  $\sigma_e$ ,  $\sigma_v$  and  $\sigma_h$ sets a framework of a phenomenological model of the Hyperelasto-Visco-Hysteresis type (HVH model). The HVH model has been successfully applied to a fluoro-elastomer [15, 16, 27] and the PPC7712 in previous works [11, 12]. In the following, we will briefly recall the three stress contributions used. One of the main contributions of this paper is to give a new method of identification of material parameters in the HVH model.

The non-linear behaviour of the polymer requires the establishment of finite deformation formalism for writing and implementing the constitutive laws. To implement the HVH model in a numerical schema, a 3D finite deformation framework has been developed in a finite element software, called HEREZH++ [14]. An Eulerian formulation, the Cauchy stress tensor, the Almansi strain tensor and the Jaumann time derivative of the stress are chosen for this purpose.

For further details about the Hyperelasto-Visco-Hysteresis model, see [11, 12].

## <sup>223</sup> 5 Identification of the material parameters

In a previous study [11], the identification method of the material parameters of the HVH model was performed using a numerical procedure by inverse analysis through an experimental database consisting of tensile tests, relaxation tests and cyclic loading-unloading tests. This complicated method using all experimental tests simulteanously, leads unfortunately, to several possible sets of material parameters and cannot really differentiate between the different stress contributions of the HVH model. Finally, this method is difficult to use for industrial purposes. In this paper, a new identification method of material parameters is proposed. It is easier to use and only requires the multi-step relaxation tests, presented in Figures 5 and 6. This identification method, proposed in [16] but with a rubber material, is direct and does not require the use of software by inverse analysis. It is performed in two steps. The first identifies the hyperelastic and hysteretic contributions, considering only the end of the relaxation periods corresponding to the equilibrium hysteretic state (as described by [28]), that is independent of the strain rate (cf blue points in Figure 6). In the second step, the viscous parameters are determined from one relaxation phase of the multi-step tests. Analytical identification is then used to obtain the characteristic times of the three Maxwell elements. 

#### <sup>239</sup> 5.1 Hyperelastic and hysteretic contributions

The viscous stress  $\sigma_v$ , hyperelastic stress  $\sigma_e$  and hysteretic stress  $\sigma_h$  components are presented in Figure 6 at a given strain  $\varepsilon_0$ , based on the experimental results shown in Figure 5. The equi-librium state  $\sigma_{\infty} = \sigma_e + \sigma_h$  can be obtained by connecting all the converged asymptotic stress values recorded at the end of the relaxation periods of each corresponding strain level. Assum-ing that the viscous stress  $\sigma_v$  reaches its steady state at the end of these relaxation phases,  $\sigma_\infty$ will be composed of the hysteresis stress component  $\sigma_h$  and the hyperelastic stress component  $\sigma_e$  and will give the blue curve in figure 6. With this assumption of stress decomposition, identification of the material parameters of hyperelastic and hysteresis contributions is direct using a simple manual identification process through several numerical trials with initial values of material parameters initially obtained in [11]. Experimental boundary and loading condi-tions are simulated with HEREZH++ on a single hexahedral finite element to reproduce the assumed homogeneous state at the centre of the specimen. 

The obtained material parameters values are reported in Table 2. The bulk viscosity *K* is fixed to 2000 MPa for the two materials, as previously used in [11]. Figure 7 show the comparison between experimental equilibrium responses and model predictions in the case of PP and Alfa/PP respectively. The identified material parameters are in the same of order of our previous works in the case of PP [11, 12]. The main contribution in the stress-strain response is still controlled by the hyperelastic part for the PP and Alfa/PP.

#### 258 5.2 Viscous contribution

Viscous parameters of the three Maxwell elements are analytically calculated from the second relaxation phase for a strain level of 2.8% (Figure 6). First, the characteristic times of each branch  $\tau_i$  are determined graphically on the experimental relaxation stress-strain curve by removing the loading phase (Figure 8). Each time  $t_i$  of the *i* basic Maxwell elements is taken so that it verifies the relation  $4\tau_i < t_{i+1}$  for  $i \in [1, k-1]$  which ensures that each branch is almost independent. <sup>265</sup> The viscous contribution is the sum of the three Maxwell elements:

$$\sigma_v(t) \approx \sum_{i}^{n=3} \sigma_i(t) \tag{3}$$

Knowing that each Maxwell element satisfies:  $\sigma_i(t) = \sigma_i \exp\left(\frac{-t}{\tau_i}\right)$  and the value of  $\sigma_i$  is given by:  $\sigma_i = \sigma_{0i} \exp\left(\frac{t_i}{\tau_i}\right)$  where each value of  $\sigma_{0i}$  and slope of the characteristic time  $\tau_i$  is obtained graphically from Figure 8.

The stiffness  $E_i$  and the viscosity  $\eta_i$  of each Maxwell element are then obtained from the following equation:

$$E_i = \frac{\sigma_i}{D\tau_i \left(1 - \exp\left(\frac{-t_{ch}}{\tau_i}\right)\right)} \text{ et } \eta_i = E_i \tau_i \tag{4}$$

where  $t_{ch}$  corresponds to the loading time necessary to reach a strain of 2.8% and D is the strain rate during this loading phase.

The viscous parameters of the three Maxwell elements are presented in Table 3. In all simulations, the Poisson coefficients, necessary for 3D formulation, are arbitrarily selected as  $\nu_1 = \nu_2 = \nu_3 = 0.4$ . Comparison between parameters in Table 3 shows that the second and third viscous parameters are different between the two materials. The results of the viscous parameter identification are presented in Figure 8.

## <sup>278</sup> 6 Simulations using the HVH model

Using the hyperelastic and hysteretic parameters of Table 2 and the viscous parameters of Table 3, comparisons between experimental and model predictions for the tension tests interrupted by relaxation steps are presented in Figures 9 and 10 in the case of PP and Alfa/PP, respectively. The results of the identification correlated well with the experimental data. A good corre-lation can be observed during the relaxation steps during the loading and unloading phases. However, especially in the case of the Alfa/PP, after the first loading and relaxation phase, a discrepancy is observed between the experimental and numerical results during the two loading steps. This is probably due to the fact that the viscosity in this material is strongly non-linear 

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whereas in the HVH model, the viscous contribution, using a generalised Maxwell's model is
not dependent on the level of strain or stress.

To verify the pertinence of the HVH model under various loading conditions, based on the parameters herein identified in the case of the PP and Alfa/PP materials, simulations of experimental tests previously presented in section 3 are performed. The numerical simulations of the complex loading test are carried out in the HEREZH++ software. Experimental boundary conditions are reproduced in a single hexahedral element by imposing experimental loading.

Firstly, the numerical results of the monotonous tensile tests are compared with experimental data in Figure 11. In these figures, the effect of the hyperelastic, viscous, and hysteresis contributions are presented. The hysteresis contribution is low whereas the hyperelastic and viscous contributions are the highest. This monotonous tensile test shows that the effect of the viscous part is non-negligible in this kind of test. The discrepancy with the experimental results at the end of the loading is due to the limitations of the linear viscoelastic contribution with Maxwell branches used in the HVH model.

The loading/unloading test with 50 cycles is also numerically simulated. Comparisons with experiments are shown in Figure 12. Good agreement was found to exist between the experimental and numerical data obtained in these cyclic tests. In the case of the Alfa/PP, some significant differences were observed during the loading steps, as already observed in the monotonous tensile test, however the hysteresis loops are relatively well described for all tests with the two materials.

The identification method developed here has shown its efficiency to be able to simulate the mechanical behaviour in simple and cyclic tensile tests. This method is simple and rapid in comparison with the previous used in [11]. However, it appears that an improvement of the viscosity contribution in the HVH model is necessary to enhance the results obtained.

## Conclusion

In this paper, a semi-crystalline polymer (PP) reinforced with natural Alfa fibres was studied. The elaboration and the treatment to improve the adhesion of fibres/matrix in the Alfa/PP blend were presented. Several mechanical tests for different loading targets (monotonic tensile

tests, cyclic tensile tests and multi-step relaxations tests) were carried out. The experimental results showed a good adhesion between the polymer matrix and the Alfa fibres and a strong influence of the presence of fibres on the mechanical behaviour of PP. These tests were subse-quently used as an experimental database to identify the material parameters of a Hyperelasto-Visco-Hysteresis model. A new identification method, rapid and simple, already used for the simulation of the elastomers behaviour, has shown its efficiency in simulating the mechanical responses of the PP and Alfa/PP during all the experimental cyclic tests. Some limitations in the viscous part with a generalised Maxwell's model were observed during the loading phases. The improvement of the viscous part of the HVH model seems important due to the fact that the viscosity of polymer material is strongly non-linear as a function of the strain or stress level. 

## References

- [1] Le Duigou A, Davies P, Baley C. Environmental Impact Analysis of the Production of Flax
   Fibres to be Used as Composite Material Reinforcement. Journal of Biobased Materials
   and Bioenergy. 2011 Mar;5(1):153–165.
- Joshi SV, Drzal LT, Mohanty AK, Arora S. Are natural fiber composites environmentally
   superior to glass fiber reinforced composites? Composites Part A: Applied Science and
   Manufacturing. 2004;35(3):371 376.
- [3] Faruk O, Bledzki AK, Fink HP, Sain M. Biocomposites reinforced with natural fibers:
   2000-2010. Progress in Polymer Science. 2012 Nov;37(11):1552–1596.
- [4] Bledzki AK, Gassan J. Composites reinforced with cellulose based fibres. Progress in
   Polymer Science. 1999;24(2):221 274.
- [5] Yu L, Dean K, Li L. Polymer blends and composites from renewable resources. Progress
   in Polymer Science. 2006;31(6):576 602.
- [6] Shalwan A, Yousif BF. In State of Art: Mechanical and tribological behaviour of polymeric
   composites based on natural fibres. Materials & Design. 2013;48(0):14 24. Materials,
   Design and Tribology.

#### **Journal of Composite Materials**

[7] Ku H, Wang H, Pattarachaiyakoop N, Trada M. A review on the tensile properties of natural fiber reinforced polymer composites. Composites Part B: Engineering. 2011;42(4):856
- 873.

[8] Ben Brahim S, Cheikh RB. Influence of fibre orientation and volume fraction on the tensile
 properties of unidirectional Alfa-polyester composite. Composites Science and Technology.
 2007 Jan;67(1):140–147.

[9] Paiva M, Ammar I, Campos A, Cheikh R, Cunha A. Alfa fibres: Mechanical, morphological and interfacial characterization. Composites Science and Technology. 2007
 May;67(6):1132–1138.

[10] Arrakhiz F, Elachaby M, Bouhfid R, Vaudreuil S, Essassi M, Qaiss A. Mechanical and
 thermal properties of polypropylene reinforced with Alfa fiber under different chemical
 treatment. Materials & Design. 2012 Mar;35:318–322.

- I11] Zrida M, Laurent H, Rio G, Pimbert S, Grolleau V, Masmoudi N, et al. Experimental
   and numerical study of polypropylene behavior using an hyper-visco-hysteresis constitutive
   law. Computational Materials Science. 2009;45(2):516–527.
- I2] Zrida M, Laurent H, Grolleau V, Rio G, Khlif M, Guines D, et al. High-speed tensile tests
   on a polypropylene material. Polymer Testing. 2010 June;29:685–692.
- <sup>358</sup> [13] Guitton E, Rio G, Laurent H. A new multiaxial loading test for investigating the mechan <sup>359</sup> ical behaviour of polymers. Polymer Testing. 2014 Jun;36:32–43.
- <sup>360</sup> [14] Rio G. HEREZH++. certification IDDN-FR-010-0106078-000-R-P-2006-035-20600;
   <sup>361</sup> http://www-lg2m.univ-ubs.fr/~rio.

[15] Vandenbroucke A, Laurent H, Hocine NA, Rio G. A Hyperelasto-Visco-Hysteresis model
 for an elastomeric behaviour: experimental and numerical investigations. Computational
 Materials Science. 2010 May;48(3):495–503.

[16] Laurent H, Vandenbroucke A, Rio G, Hocine NA. A simplified methodology to identify material parameters of a hyperelasto-visco-hysteresis model: application to a fluoro-elastomer.
 Modelling and Simulation in Materials Science and Engineering. 2011 Dec;19(8):085004.

[17] Arrakhiz FZ, Malha M, Bouhfid R, Benmoussa K, Qaiss A. Tensile, flexural and torsional
 properties of chemically treated alfa, coir and bagasse reinforced polypropylene. Compos ites Part B: Engineering. 2013;47(0):35 – 41.

<sup>371</sup> [18] Bessadok A, Roudesli S, Marais S, Follain N, Lebrun L. Alfa fibres for unsaturated
<sup>372</sup> polyester composites reinforcement: Effects of chemical treatments on mechanical and
<sup>373</sup> permeation properties. Composites Part A: Applied Science and Manufacturing. 2009
<sup>374</sup> Feb;40(2):184–195.

- [19] El-Abbassi F, Assarar M, Ayad R, Lamdouar N. Effect of alkali treatment on Alfa fibre as
  reinforcement for polypropylene based eco-composites: Mechanical behaviour and water
  ageing. Composite Structures. 2015 Dec;133:451–457.
- <sup>378</sup> [20] Le Troedec M, Sedan D, Peyratout C, Bonnet J, Smith A, Guinebretiere R, et al. In<sup>379</sup> fluence of various chemical treatments on the composition and structure of hemp fibres.
  <sup>380</sup> Composites Part A: Applied Science and Manufacturing. 2008 Dec;39:514–522.
- [21] Le Troedec M, Rachini A, Peyratout C, Rossignol S, Max E, Kaftan O, et al. Influence of
   chemical treatments on adhesion properties of hemp fibres. Journal of Colloid and Interface
   Science. 2011 Dec;356:303–310.
- Beckermann GW, Pickering KL. Engineering and evaluation of hemp fibre reinforced
   polypropylene composites: Fibre treatment and matrix modification. Composites Part A:
   Applied Science and Manufacturing. 2008;39(6):979 988.
- [23] Miehe C, Keck J. Superimposed finite elastic-viscoelastic-plastoelastic stress response with
   damage in filled rubbery polymers. Experiments, modelling and algorithmic implementa tion. Journal of the Mechanics and Physics of Solids. 2000;48(2):323–365.

#### **Journal of Composite Materials**

<sup>390</sup> [24] Lin RC, Schomburg U. A finite elastic-viscoelastic-elastoplastic material law with dam <sup>391</sup> age: theoretical and numerical aspects. Computer Methods in Applied Mechanics and
 <sup>392</sup> Engineering. 2003;192(13-14):1591–1627.

<sup>393</sup> [25] Besdo D, Ihlemann J. A phenomenological constitutive model fo rubberlike materials and
 <sup>394</sup> its numerical applications. International Journal of Plasticity. 2003;19:1019–1036.

<sup>395</sup> [26] Hasanpour K, Ziaei-Rad S. Finite element simulation of polymer behaviour using a three dimensional, finite deformation constitutive model. Computers & Structures. 2008;86(15 16):1643-1655.

[27] Laurent H, Rio G, Vandenbroucke A, At Hocine N. Experimental and numerical study on
 the temperature-dependent behavior of a fluoro-elastomer. Mechanics of Time-Dependent
 Materials. 2014;18(4):721-742.

<sup>401</sup> [28] Lion A. A constitutive model for carbon black filled rubber : Experimental investiga<sup>402</sup> tions and mathematical representation. Continuum of Mechanics and thermodynamics.
<sup>403</sup> 1996;8:153–169.



Figure 1: Distribution of the cross sectional area as function as the fibre number (in green the average value).



Figure 2: Comparison of PP and Alfa/PP material responses in the monotonic tensile test.



Figure 3: Scanning Electron Microscopic analysis on a specimen surface after the break of a tensile test.





Figure 4: Comparison of PP and Alfa/PP material responses in the loading/unloading test with 50 cycles (only the first, the tenth and the last cycle are presented).





Figure 5: Comparison of PP and Alfa/PP material responses in the multi-step relaxation test.(a) Stress-strain response. (b) Stress-time response.



Figure 6: Example of estimated viscous  $\sigma_v$ , hyperelastic  $\sigma_e$  and hysteretic  $\sigma_h$  contributions from the loading/unloading test interrupted by relaxation phases.







Figure 7: Identification of the hyperelastic and hysteresis contributions from the end points of relaxation phases in the case of : (a) PP and (b) Alfa/PP.



Figure 8: Identification of the slope of the three Maxwell elements and final identification of the viscous contribution during the relaxation phase in the case of : (a) PP and (b) Alfa/PP.



Figure 9: Comparison between experimental and identification in the loading/unloading test interrupted by relaxation steps in the case of the PP. (a) Stress-strain response. (b) Stress-time response.



Figure 10: Comparison between experimental and identification in the loading/unloading test interrupted by relaxation steps in the case of the Alfa/PP. (a) Stress-strain response. (b) Stress-time response.

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Figure 11: Numerical simulation of the monotonous tensile test in the case of : (a) PP and (b) Alfa/PP. Influence of the hyperelastic stress contribution  $\sigma_e$ , viscous stress contribution  $\sigma_v$  and hysteresis stress contribution  $\sigma_h$ .



Figure 12: Numerical simulation of the loading/unloading test with 50 cycles in the case of : (a) PP and (b) Alfa/PP.

Properties	Values
Melt flow index $MFI(g/10 min)$	13
Molecular weight Mw (g/mol)	229527
Crystallinity ratio (%)	34.2
Tensile modulus (MPa)	1280
Yield stress (MPa)	19
Charpy impact strength $(kJ/m^2)$	7

Table 1: Thermo-mechanical characteristics of the PPC7712 copolymer at 20° C[11].

	Hyperelastic parameters (in MPa)	Hysteretic parameters (in MPa)
PP	$Q_{0r} = 3.9$	$n_p = 0.9$ (-)
	$\mu_0 = 120$	$\mu_h = 160$
	$\mu_{\infty} = 27$	$Q_0 = 3.5$
Alfa/PP	$Q_{0r} = 4.5$	$n_p = 2.5$ (-)
	$\mu_0 = 300$	$\mu_h = 170$
	$\mu_{\infty} = 17$	$Q_0 = 4.5$

Table 2: Hyperelastic and hysteresis parameters identified in the case of PP and Alfa/PP.

	$1^{st}$ Maxwell branch	$2^{nd}$ Maxwell branch	$3^{rd}$ Maxwell branch
PP	$E_1 = 277 \; (MPa)$	$E_2 = 72 \text{ (MPa)}$	$E_3 = 82 \text{ (MPa)}$
	$\eta_1 = 2764 \text{ (MPa.s)}$	$\eta_2 = 6043 \; (MPa.s)$	$\eta_3 = 32942 \text{ (MPa.s)}$
	$\tau_1 = 9.97 \; (s)$	$\tau_2 = 83.93 \text{ (s)}$	$\tau_3 = 401.73 \; (s)$
Alfa/PP	$E_1 = 283 (\mathrm{MPa})$	$E_2 = 16.7 \; (MPa)$	$E_3 = 163 \; (MPa)$
	$\eta_1 = 2812 \; (MPa.s)$	$\eta_2 = 1537 \; (MPa.s)$	$\eta_3 = 43184 \text{ (MPa.s)}$
	$\tau_1 = 9.93 \; (s)$	$\tau_2 = 92.03 \ (s)$	$\tau_3 = 264.93 \text{ (s)}$

Table 3: Viscous parameters of three Maxwell branches obtained in the case of PP and Alfa/PP  $(\nu_1 = \nu_2 = \nu_3 = 0.4).$