

ELASTOPLASTIC BEHAVIOR OF POLYBUTYLENE TEREPHTHALATE POLYESTER BIO LOADED BY TWO SUSTAINABLE AND ECOLOGICAL FIBERS OF ANIMAL ORIGIN WITH TWO NUMERICAL METHODS

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Abstract- Research in composites area is moving towards green composites or so-called bio composites thanks to their friendly properties towards the environment. They present a light density and more advantages for the economy, recycling and sustainable development. In this study, a numerical mechanical study of two keratinous animal bio-loads, cow and sheep horn fibers is carried out in order to mix them with the polybutylene terephthalate (PBT) polymer widely used in the industry and which is classified in the group of semi-crystalline thermoplastic polyesters which has the best performance in terms of rigidity, toughness and environmental resistance, etc. It is used in automotive, electronics and electrical components. Homogenization is achieved by two numerical methods, Mori-Tanaka and the finite element method which have shown more advantages in modeling more complex composites. Optimization of horn content, aspect ratio and orientation of cow and sheep horn fibers in the polybutylene terephthalate polymer were simulated and the best mechanical results are obtained using cow horn fibers for a content of 10 Wt% by the Mori-Tanaka method and 8.9 Wt% by the finite element method with an orientation of 0° and high aspect ratios.

Keywords: Green Composites, Sustainable Development, Environment, Animal Horn Fibers, Numerical Modeling.

1. INTRODUCTION

Large ecological requirements encourage technologies that try to reduce the use of chemicals that have a dangerous effect. The loads meeting the environmental and energy requirements have attracted the attention of researchers in the field of bio composites unlike derivatives of petroleum products. Therefore, the current trend is towards the use of protein-based fillers such as gelatin, albumin, collagen and keratin for the enhancement of naturally derived biomaterials [1]-[4].

Pool et al. has shown that regenerated protein-based fibers are best for the environment [5]. Natural biomaterials have better mechanical properties.

Jammoukh et al. in 2018 showed the good effect of bio-loading on polyvinyl chlorides morphology [6]. The use of keratin is increasingly studied thanks to its biodegradability, biocompatibility and its sustainable development [7]. The notion of keratin is of Greek origin "kera" which means horn. It is used around 1850 to describe materials that are made by hard fabrics such as horns and hooves.

Shi-Zhen Li [8] used it in the field of medicine in the 16th century and was the beginning of its use as a biomaterial for medical applications. Xiao Chun et al studied the chicken feather for the production of a keratin film for use as a natural load [9]. The extraction of the protein can be done from the hair, nails and wool [10]. Several works have been done in recent years to characterize keratinous materials.

The field of composites and nonwoven fabrics has begun to choose keratin for current applications [11]. This protein is classified after collagen, the best biopolymer found in animals. Comparing the elastic properties of keratin with those of other biological fibers shows that keratinous products derived from horns are among the materials that exhibit significant young modulus as well as larger stresses at break.

The present work will focus on the comparison of the elastoplastic properties of the Polybutylene terephthalate (PBT) reinforced by horns of two domestic animals that are the cow and sheep that have attracted the attention of the latest research in the field of biomaterials Jammoukh et al. 2018 [6] and Zhang et al. 2019 [12].

2. MATERIALS AND METHODS

2.1. Materials

2.1.1. Bio Loads

We will carry out a comparative study between the mechanical properties of cow and sheep horns. Keratinous materials are studied in terms of α and β keratins. Their classification can be made in α pattern, β pattern and amorphous pattern. They are obtained by the intracellular synthesis of keratins [14].

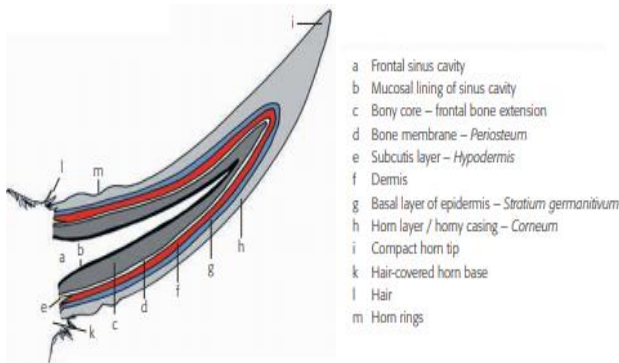


Figure 1. Horn structure of the keratinous biomaterial [13]

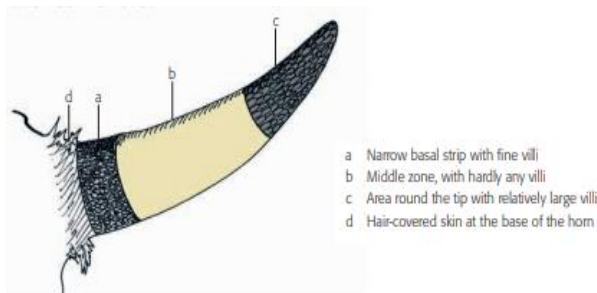


Figure 2. Dermis of the keratinous biomaterial [13]

Table 1. Sources of α and β keratin

Type of keratin	Origin
α keratin	Wool, hair, horn, hooves
β keratin	Avian beaks, claws
α and β keratin	Reptilian, epidermis, pangolin scales

2.1.2. Polybutylene Terephthalate (PBT)

The Polybutylene terephthalate (PBT) classified in the group of polyesters is designed as synthetic thermoplastic semi crystalline which has the same properties as the polyethylene terephthalate.

This polymer has a best performance and stiffness. It is used in automotive, electronics and electrical components where the highest strength, chemical resistance and stability is needed. It is also used for their advantages characteristics like the toughness, machining properties, environmental resistance etc. [15].

2.2. Methods

2.2.1. Experimental Evaluation

2.2.1.1. Experimental Evaluation of Cow Horns

Figure 3 and Table 2 present the specimen intended for tensile test.

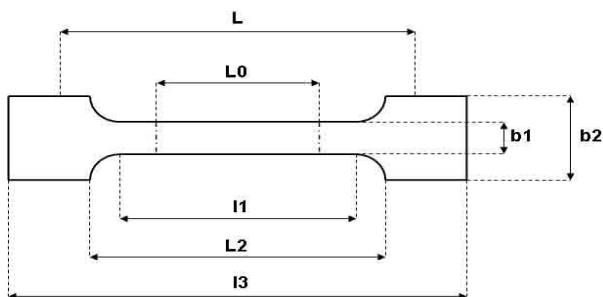


Figure 3. Normalized specimen for the tensile test

Table 2. Normalized values in mm of samples

I.S.O	527:2
L_3	>74
Type	1/BA
B_2	10
L_1	30
H	> 2
L_0	25
B_1	5
L	$L_2=58$
Form	Halter

2.2.1.2. Experimental Evaluation of Sheep Horns

The horn sheep and the keratin sheaths where the tensile sample is cut are shown in the Figure 4. All the samples were cut from the central region of the horn with a longitudinal direction [12]. The dimensions in mm of the samples are shown in Table 3.

Table 3. Dimensions of the samples

Dimensions	Values
Length	15
Width	3
Thickness	0.3
Gage length	10

The tests were performed using a machine equipped with a loading cell of 500 N (Instron 3342). The crosshead speed is 0.0001 mms^{-1} which corresponds to $1 \times 10^{-3} \text{ s}^{-1}$ strain rate [12].

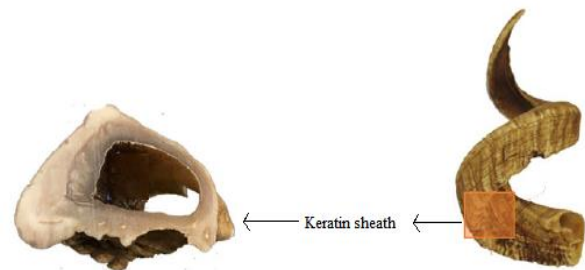


Figure 4. Keratin sheath intended for tensile samples [12]

2.2.2. Numerical Modeling of Virgin Cow and Sheep Horns

2.2.2.1 Incrementation

A maximum number of increments of 100 were used with minimum and maximum times of 0.001 and 0.1 respectively. The equation is solved using the iterative Casi method with a tolerance of 1×10^{-8} .

2.2.2.2 Mesh

The choice of the type of mesh is important in order to have quality results as well as to decrease the times of computations and to solve the nonlinearities. We chose the Tetra Quadratic element which is efficient in modeling complex mechanical behaviors. The total number of elements is 26.800 with a number of nodes of 40.667.

The size of the element plays an important role in correctly predicting the behavior of our two biomaterials. We chose a size of 0.05 with a minimal size of 0.01. The mesh obtained is shown in the Figure 5.

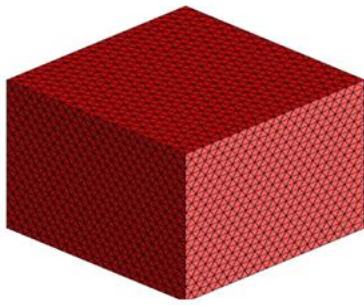


Figure 5. Mesh of the two biomaterials

2.2.3. Numerical Homogenization by the Mori-Tanaka Method

We will simulate the mechanical behavior of Polybutylene terephthalate reinforced by the two types of horns by two numerical methods of homogenization, the Mori Tanaka model and the finite element method which showed more advantages in the homogenization of even complex behaviors [16], [17].

Numerical simulations will also consist of the optimization of horn content, the aspect ratio and the orientation of cow and sheep horns in the polybutylene terephthalate polymer.

2.2.3.1. Incrementation

The incrementation values are listed in Table 4.

Table 4. Incrementation time values

Time	Values
Final	1
Maximum	0.1
Minimum	0.01

2.2.3.2. Tolerance

The control of tolerance can be used as presented in the Tables 5 and 6.

Table 5. Homogenization control

Parameters	Values
Admissible tolerance	1E-005
Target tolerance	1E-006
Maximum-iterations	25

Table 6. Loading-control

Parameters	Values
Admissible-tolerance	1E-005
Target- tolerance	1E-006
Tolerance threshold	1E-005
Maximum iterations	25

2.2.3.3. Orientation

The control of orientation is used as presented in the Table 7.

Table 7. Orientation parameters

Parameters	Values
Angle increments	6
Trace tolerance	0.1

2.2.4. Numerical Homogenization by the Finite Element Method

2.2.4.1. Position of Horn Fibers

The position of the horn fibers in the geometry is presented in the Figure 6 and Table 8 in the three planes.

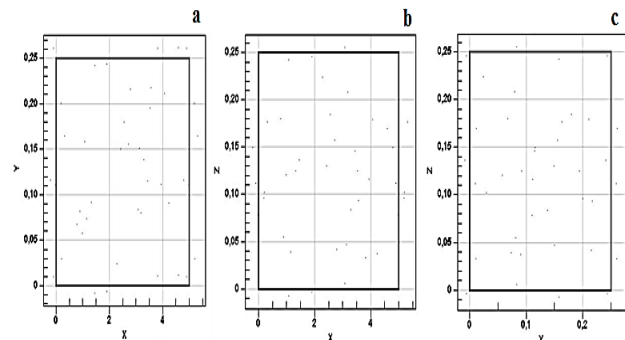


Figure 6. Position of cow and sheep horn fibers in the PBT geometry in the three planes (xy, xz and yz)

Table 8. Position of cow and sheep horn fibers

Statistical values	Minim	Maxim	Mean	Std-Dev
x	-0.2	5.34	2.59	1.67
y	-0.008	0.262	0.126	0.08
z	-0.007	0.256	0.119	0.07

2.2.4.2. Nearest horn distance

The Figure 7 shows nearest horn fibers distance distribution.

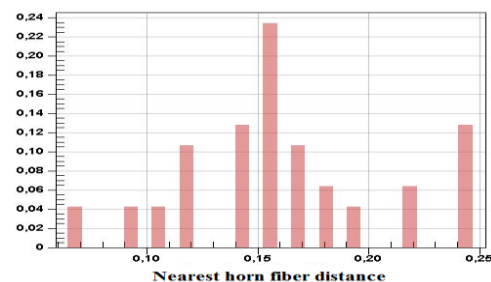


Figure 7. Nearest cow and sheep horn fiber distance

The maximum increments used in the finite element method are 100 increments. The other parameters of incrementation are listed in the Table 9.

Table 9. Incrementation table

Time	Number
First	0.1
Last	1
Minimal	0.01
Maximal	0.1

3. RESULTS

3.1. Virgin Cow and Sheep Horn Fibers

3.1.1. Mechanical Behavior of Cow and Sheep Horns

The tensile tests are carried out in the longitudinal direction which has the best elastic properties [12], [18]. The tensile curves obtained at the longitudinal direction for both types of horns are presented in the Figure 8.

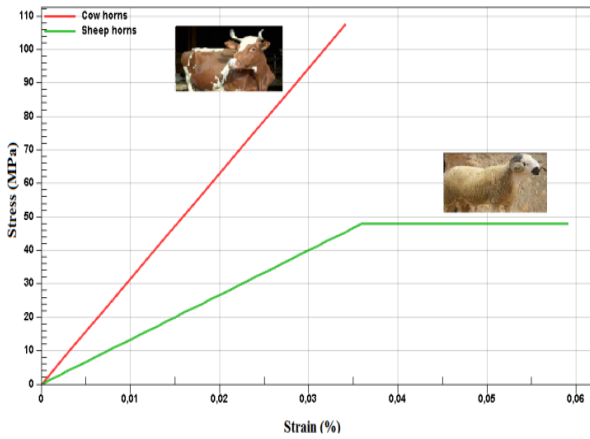


Figure 8. Tensile curves in longitudinal direction

Table 10. The mechanical properties using numerical modelling

Horn type	Stress - Break [MPa]	Elongation - Break [%]	Young - Modulus [MPa]
Cow	106.92	3.41	3148.4
Sheep	47.9	5.9	1333

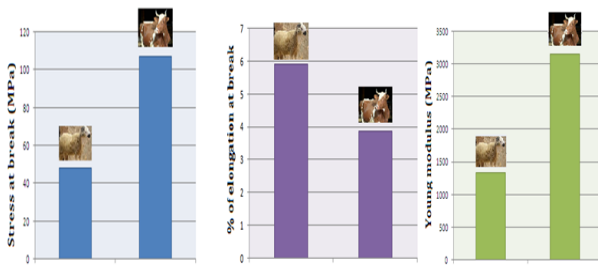


Figure 9. Mechanical properties of cow and sheep horn fibers

The mechanical properties for both types are mentioned in the Table 10. The Figure 9 shows that the cow bio load has a rigid elastic behavior where the deformation is in elastic mode. For the other sheep bio load, the behavior is rather tenacious with elastic properties less than the first load.

The behavior of the two bio loads is anisotropic [6], [12]. The rigidity at 0° is better for both. The stress at break is very high for the case of the cow horns with a relatively small displacement and a large Young's modulus showing the rigidity of the material.

3.1.2. Von Mises Criterion and Displacements

Figures 10 and 11 present the Von Mises stresses values obtained numerically using the finite element method. They are below the elastic limit (106.92 MPa) for cow horns and above this limit (47.9 MPa) for the case of sheep horns, which confirm that the deformation is made elastically for the first bio load and plastically for the second type.

The Figures 12 and 13 present the displacement obtained. A reading of these figures shows that the cow horns displacements are smaller than of the sheep horns.

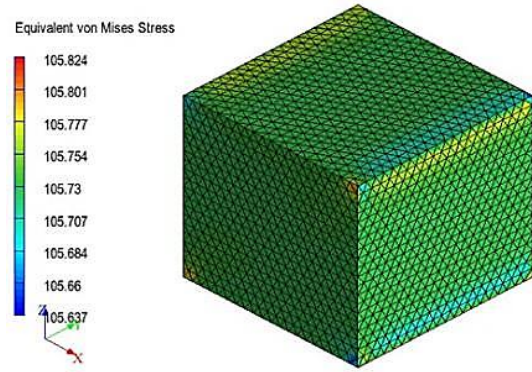


Figure 10. Von Mises stress of cow horns

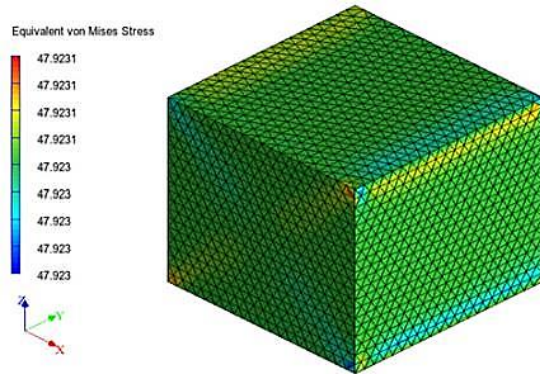
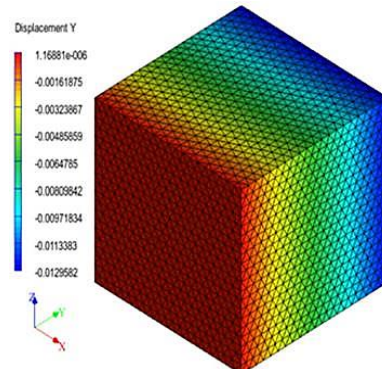
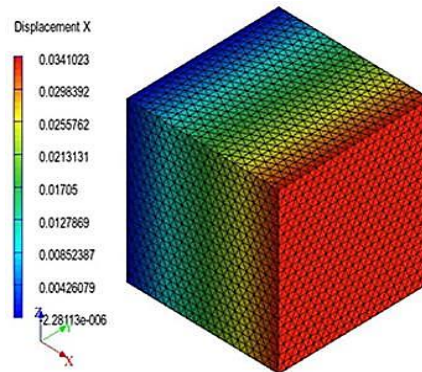


Figure 11. Von Mises stress of sheep horns



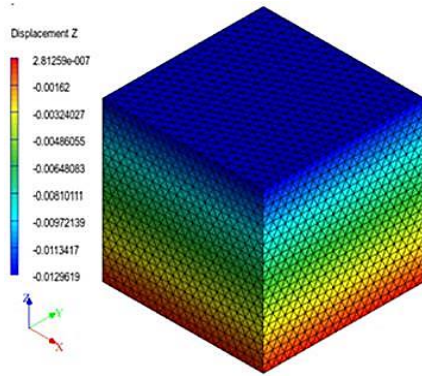


Figure 12. Displacements x , y and z of the cow horns

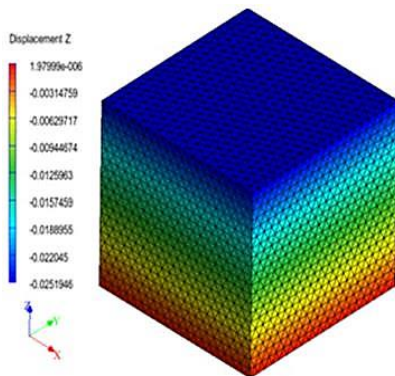
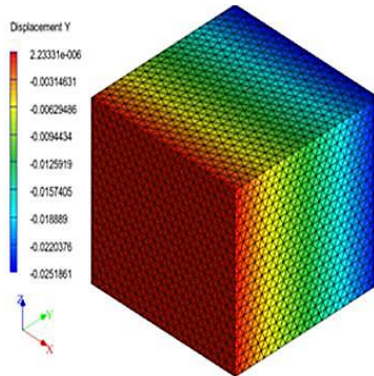
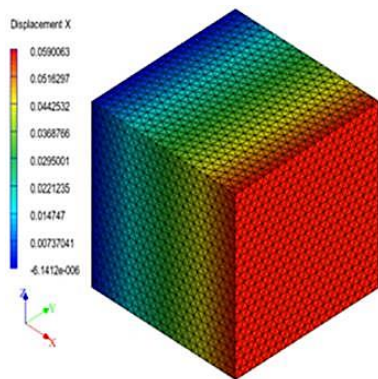


Figure 13. Displacements x , y and z of the sheep horns

3.2. Numerical Behavior of Pure Polybutylene Terephthalate Polymer

In order to obtain the numerical behavior of Polybutylene terephthalate polymer, we chose the plasticity J_2 model and the exponential and linear law for

the isotropic hardening model. The behavior of PBT is elastoplastic as it is illustrated in the figure 14. The numerical mechanical properties of PBT are listed in Table 11.

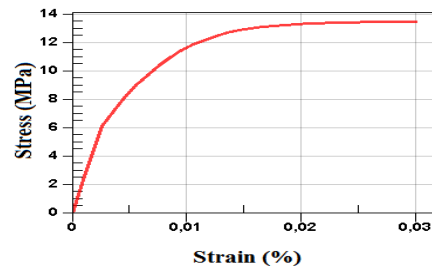


Figure 14. Elastoplastic curve of the polybutylene terephthalate

Table 11. Numerical mechanical properties of PBT

Properties	Values
Density	1.13 g/cm ³
Young modulus	2352.6 MPa
Poisson's ratio	0.37
Yield stress	6.1 MPa
Stress at break	13.5 MPa
Haredening modulus	7.31 MPa
Linear haredening modulus	2 MPa
Haredening exponent	275.41

3.3. Numerical Homogenization by the Mori-Tanaka Method

3.3.1 Fiber Content

The elastoplastic behavior of different content of cow and sheep horns reinforced PBT polymer is presented in Figures 15 and 16.

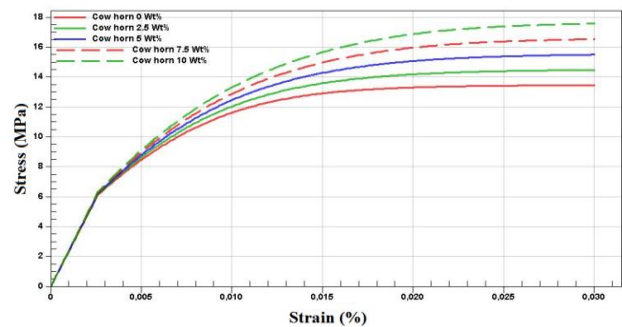


Figure 15. Elastoplastic curves of the cow horns reinforced PBT at different content

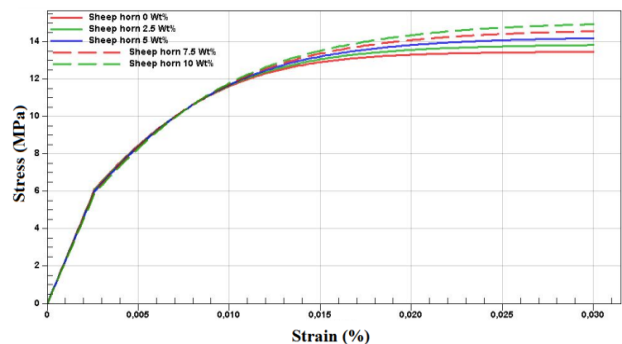


Figure 16. Elastoplastic curves of the sheep horns reinforced PBT at different content

The Table 12 present the values of the elastoplastic parameters obtained numerically for the cow horns reinforced PBT (CH-PBT) and sheep horns reinforced PBT (SH-PBT).

Table 12. Elastoplastic properties of the bio composites in MPa

Fiber content	0 [%]	2.5[%]	5 [%]	7.5 [%]	10 [%]
Young modulus CH-PBT	2352.6	2370.3	2388	2405.9	2423.9
Young modulus SH-PBT	2352.6	2330.3	2307.8	2285.2	2262.5
Stress at break CH-PBT	13.5	14.5	15.5	16.5	17.5
Stress at break SH-PBT	13.5	13.75	14.2	14.75	15.2

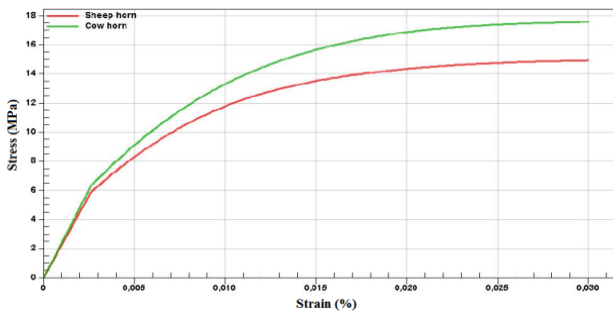


Figure 17. Comparative curves of the cow and sheep horns reinforced PBT at 10 Wt%

These results are obtained for an aspect ratio of 20 to simulate the behavior of short fibers and a size of 0.777 mm. The morphology of the cow and sheep horn fibers is cylindrical. According to the Table 12 and the Figures 15 and 16, we can say that the bio composites mixed with the cow and sheep horns have very good elastoplastic characteristics in rigidity compared to pure PBT at 0 Wt. The more the content of fibers is added the more the stress at break increases at 17.5 MPa for cow horns and 15.2 MPa for sheet horns at 10 Wt%. We therefore note that the values of the stress at break are greater for cow horns than those of sheep.

This is also noticed for the Young's module of bio composites; we notice that the more the content of cow horn fibers is added the more the Young's module becomes improved from 2352.6 MPa in the pure state up to 2423.9 MPa using this bio loading at 10 Wt%. On the contrary, the addition of sheep horn fibers leads to a decrease in the Young modulus values of the bio composites obtained from 2352.6 MPa in a pure state up to 2262.5 MPa by using bio loading with this type of horns. It can be deduced that the bio loading with cow horns is better for Polybutylene terephthalate polymer.

3.3.2. Fiber Orientation

The orientation of the fibers is an important parameter in obtaining most optimal results. We simulated three orientations for the two types of horns (0°, 45° and 90°). We tried to fix the others parameters of the numerical study to have more reliable results. The content of the horns is 10 Wt% for all orientations. The aspect ratio is 20 with cylindrical fiber morphologies of 0.777 mm in

size. The results of the numerical homogenization are illustrated in the Table 13 and the figures 18 and 19 respectively for bio composites based on cow horns and the sheep horns.

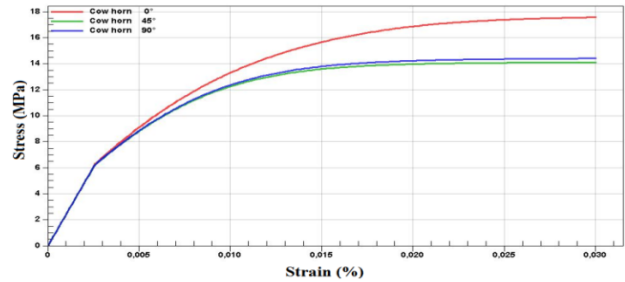


Figure 18. Elastoplastic curves of cow horns reinforced PBT at different orientations

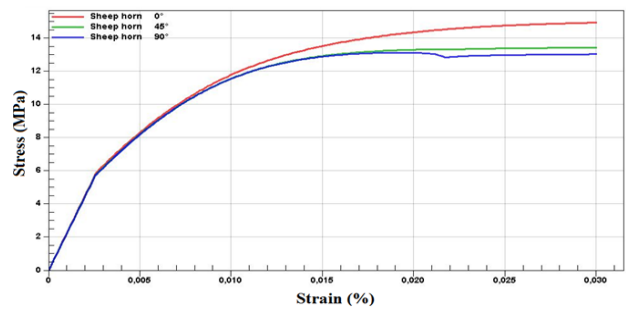


Figure 19. Elastoplastic curves of sheep horns reinforced PBT at different orientations

Table 13. Elastoplastic properties of the bio composites at 10 Wt%

Fiber content	0°	45°	90°
Young modulus CH-PBT	2423.9	2423.9	2423.9
Stress at break CH-PBT	17.5	14	14.3
Young modulus SH-PBT	2262.5	2262.5	2262.5
Stress at break SH-PBT	15.2	13.4	13

The Young modulus values for the two types of bio composites are not affected by the orientation of the fibers from 0° to 90°. The 2262.5 MPa for the sheep horns and 2423.9 MPa for the cow horns while the stress at break vary depending on the orientation of the fibers.

For bio composites based on sheep horns, the stress at the break is great (15.2 MPa) in longitudinal orientation (0°). The more we are far from 0°, the more the stress at break decrease (13.4 MPa at 45°, 13 MPa at 90°). For cow horns, the rigidity is better at 0° (17.5 MPa), is slightly good at 90° than at 45° (14 MPa at 45° and 14.3 MPa at 90°).

We can deduce that the optimal properties are obtained using a longitudinal orientation of cow and sheep horns. The stress at break is better for bio composites based on cow horns than those of bio composites based on sheep horns.

3.3.3. Fiber Aspect Ratio

The aspect ratio of fibers also plays an important role in numerical homogenization and obtaining optimal results. We simulated bio composites for aspect ratios of 20, 30 and 40 by fixing the other parameters of the study (content: 10 Wt% and orientation: 0°).

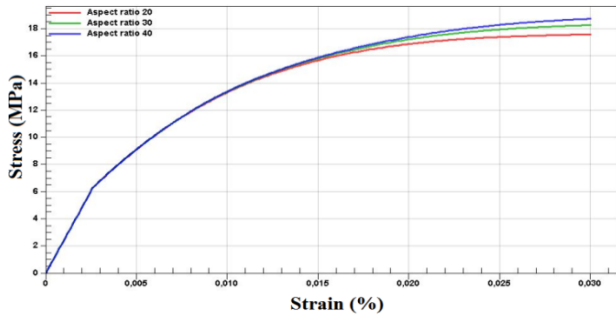


Figure 20. Elastoplastic curves of the cow horns reinforced PBT at different aspect ratios

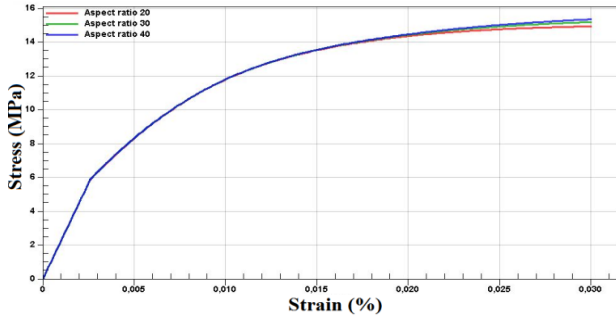


Figure 21. Elastoplastic curves of the sheep horns reinforced PBT at different aspect ratios

The Figures 20 and 21 respectively illustrate the results obtained for the two types of bio composites. We can deduce that the more the aspect ratio is increased the more the stress at break becomes large with better properties for bio composites based on cow horns than those of sheep horns. 18.8 MPa at an aspect ratio of 40 for the PBT reinforced by cow horn and 15.4 MPa for the PBT reinforced by the sheep horns.

3.4. Numerical Homogenization by the FEM

In order to validate results obtained by the Mori-Tanaka method, we carried out a second numerical method widely known for its power in the numerical homogenization of complex behaviors and the reliability of its results. It is the finite element method largely used.

The Figure 22 present the cow and sheep horn fibers reinforced the PBT with 0.0896 as the fraction in geometry obtained by this numerical method for the same cylindrical morphology as that carried out previously by Mori Tanaka method at a longitudinal orientation, aspect ratio 20 and a fiber size of 0.777 mm.

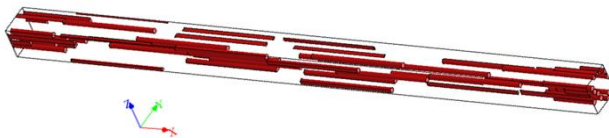


Figure 22. Horn fibers distribution in the PBT geometry

We used the tetrahedron element in our numerical homogenization view the flexibility, accuracy and fast convergence demonstrated by this type of element. The number of elements in the horn fibers reinforced PBT is

15625, the nodes are 17576. The Figure 23 present the mesh used for the modeled bio composite and the table 14 present the fraction obtained in geometry and mesh with the number of elements used.

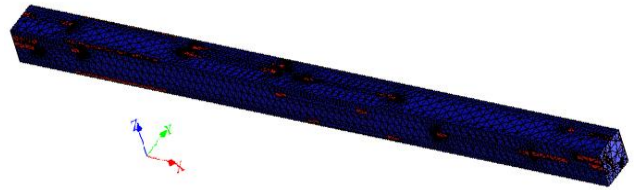


Figure 23. Mesh of cow and sheep horn fibers reinforced PBT

Table 14. Fraction obtained in geometry and mesh with the number of elements used

Bio composite	Elements	Fraction in geometry	Fraction in mesh
Horn fibers	1398	0.0896	0.0894
PBT	14227	0.9103	0.9105

The Table 14 show that the optimal fraction is around 8.9 Wt% of horn fibers which is almost the value obtained by Mori-Tanaka (10 Wt%). The following figures illustrate the comparison of the results of elastoplastic behaviors obtained for cow and sheep horns reinforced PBT by the F.E.M. compared to M-T model.

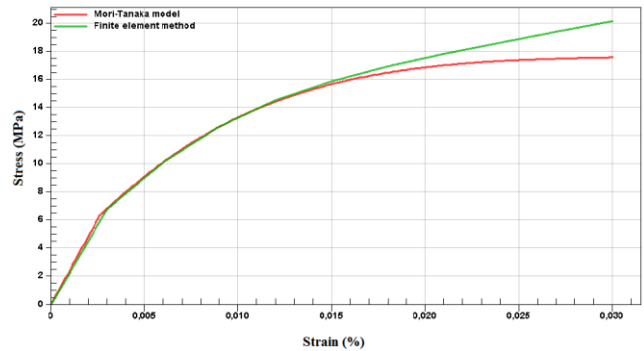


Figure 24. Elastoplastic curve of the cow horns reinforced PBT by the F.E.M. compared to the M-T model

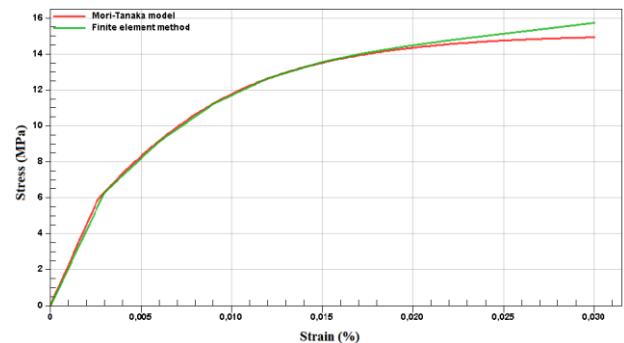


Figure 25. Elastoplastic curve of the sheep horns reinforced PBT by the F.E.M. compared to the M-T model

We can deduce from the figures 24 and 25 that the results of the F.E.M. for the cow & sheep horns reinforced PBT are almost the same as those obtained by M-T model. The two curves trace the same paces of the rigidity of the Young's modulus and the point of the yield

stress with a small offset in terms of the stress at break. This shows the power of these two methods in numerical homogenization of our bio composite studied.

4. CONCLUSION

In this work, a comparative study of the elastoplastic properties of two bio composites based on cow and sheep horn fibers was carried out. The bio load derived from the cow horns has a rigid behavior where the deformation is made in an elastic regime with a very high tensile stress. For the other bio load derived from sheep horns, the behavior is rather tenacious with less elastic properties than the first one.

The behavior of the keratinous bio loads is anisotropic and the rigidity in the longitudinal direction is better for both. The polymer studied is the polybutylene terephthalate polyester designed as synthetic thermoplastic semi crystalline having better mechanical and environmental resistance properties. It is used in automotive, electronics and electrical components.

We validated the results of the mechanical behavior of Polybutylene terephthalate reinforced by the two types of horns by two numerical homogenization methods, M-T model and the finite element method which showed more advantages in the homogenization of even complex behaviors, having a fast convergence, best flexibility and accuracy results.

We can deduce that the polymers mixed with the cow and sheep horn fibers have very good rigidity characteristics compared to the pure polybutylene terephthalate polymer at 0 Wt%. The optimal content is 10 Wt% for the two types of horns by the Mori-Tanaka method and 8.9 Wt% by the finite element method. In terms of fiber orientation, the best properties are obtained at 0° for the two types of bio loads. The aspect ratio is also an important factor to take into consideration since the larger it is, the more the PBT is more rigid.

It can be deduced that bio loading by cow horn fibers is better for polybutylene terephthalate than by sheep horn fibers and this keratin derived from these bio loads offers more advantages thanks to its biocompatibility, biodegradability and its sustainable development.

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BIOGRAPHIES



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