STIFFNESS PREDICTION IN GREEN COMPOSITES USING HOMOGENIZATION TECHNIQUES

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1 Abstract

Bio-based materials offer interesting solutions to sustainable and eco-friendly industrial applications in the future. In this study micromechanical modeling and Mori-Tanaka mean field homogenization technique is used for stiffness prediction in short flax fiber reinforced PLA biopolymer. The fiber geometry distribution is considered in the homogenization along with the orientation distribution. The concept of multi-phase composite is used to describe the distribution of geometry that is typical for natural fibers. In this concept the fibers were classified into N classes with different geometry. Composites with 20 and 30 wt% of flax fibers were manufactured using compounding followed by injection molding process. Fiber degradation has been tracked and characterized for the implementation in micromechanical modeling and homogenization. Mechanical properties of the constituents are characterized as they provide essential information in micromechanical modeling. The fiber orientation distribution is investigated using Synchrotron tomography and second order orientation tensors are identified for the composites. The homogenization results with the consideration of fiber geometry distribution are compared with experimental results. Consideration of the fiber geometry distribution results in compatible results to experiments.

2 Introduction

A vast amount of research has been focused on injection molded short fiber reinforced composites due to high production rate of the process and major property enhancement in the matrix by fiber addition, especially in the automotive industry. New regulations and environmental concerns have led to the substitution of glass fibers with natural fibers and conventional polymers with biopolymers to promote eco-friendly products and sustainable industrial practices. Besides being ecofriendly, these composites offer also novel properties like high sound and energy absorption because of the hollow structure of the natural fibers which result in also high specific properties. Further efforts are going on to apply the natural fiber reinforced composites in structural components.

Virtual product development is a concept that is implemented to reduce the time to market and increase the product quality while promoting enhanced resource utilization. Finite element analysis (FEA) tools are used, where time effective material models are playing an important role. In this work mean field homogenization techniques, which are based on assumed relations between volume averages of strain in each phase, are used. An advantage of this type of material models is that it is very convenient to apply the material model for other fiber contents, geometry, and fiber type or matrix material.

In this research short flax fiber reinforced PLA biopolymer has been investigated. A recent review on green composites adequate for automotive applications has suggested flax fiber reinforced PLA as the first option considering cost/volume and specific strength [1]. Moreover these composites have the advantage of mass production which is of high interest for example in the automotive industry. PLA biopolymer and flax fibers have been compounded using a double screw extruder in different weight percentage of flax fibers. The effect of using different compounding parameters has been investigated, especially the screw speed on mechanical properties of the composites.

The flax fiber reinforced compounds have been injection molded into plates for investigation of the mechanical properties. In a process like injection molding where high shear rates are present a phenomenon called flow induced orientation (FID) is unavoidable affecting the mechanical properties profoundly. For this reason information on fiber orientation distribution (FOD) are essential for property prediction.

Fiber orientation distribution has been investigated using Synchrotron micro-computed tomography (μCT). Conventional x-rays tomography was not successful in the case of flax fiber reinforced PLA as the density difference between the constituents is not enough to gain reasonable contrast in X-rays tomography. That is the reason why Synchrotron computed tomography was performed on the samples where the higher flux of x-rays in this method provides better contrast. The fiber orientation distribution (FOD) has been considered in homogenization and stiffness prediction of the composites.

In compounding and injection molding of natural fiber reinforced composites severe fiber degradation is dominant where both length and diameter of the fiber bundles are degraded [2-5]. This fiber degradation has been investigated on different matrix materials reinforced with different natural fibers. The wide distribution of the fiber length, fiber diameter and fiber aspect ratio can be considered as multi-phase composite with fibers of different geometry.

Consideration of the fiber geometry distribution like fiber aspect ratio distribution in micromechanical modeling and homogenization techniques is a challenge that has been considered in this research to estimate the stiffness properties of the flax fiber reinforced bio composites. Mechanical and geometrical properties of the fibers are considered as essential information in micromechanical models and homogenization techniques [6-8]. Until now most of the researches have considered a mean value of the geometries. Moreover the number average ($L_N = \frac{\sum N_i L_i}{\sum N_i}$), the weight average ($L_w = \frac{\sum W_i L_i}{\sum W_i}$), Root-Mean-Square average ($L_{RMS} = \sqrt{\frac{\sum N_i L_i^2}{\sum N_i}}$) or the skewed average ($L_s = \frac{\sum N_i}{\sum \frac{N_i}{L_i}}$) of geometries can be used in property prediction of short fiber reinforced composites. In this research the fibers are classified into N classes of volume fraction v_i (where i=1…N) with different geometry. The Mori-Tanaka mean field homogenization technique is used considering the fiber geometry distribution. The homogenization results are compared with number average, root-mean-square and skewed average respectively to find the best match compatible with experimental data.

3 Experimental Procedure

3.1 Material

PLA Ingeo biopolymer 3251D from Nature Works was used as matrix material. This biopolymer has very high flow capability that makes it suitable for injection molding of thin walled parts. Flax fibers were used as strengthening natural fibers. Low twist flax rovings were provided from Safilin in France.

3.2 Processing and Manufacturing

3.2.1 Compounding

A twin screw extruder (Coperion ZSK25 WLE) was used to compound the flax fibers and the PLA biopolymer. PLA granulates and flax fibers were dried at 80°C for 12 hours. PLA granulates and flax rowings were fed into the extruder simultaneously to manufacture the compound. The fiber content into the compound was controlled by the extruder screw speed considering TEX2000 flax fibers. The extruder head screw speed was 300 rpm while the side screw speed was different for different samples. In case of samples with 20 wt% flax fibers the side screw speed was 278 rpm. In case of samples with 30 wt% flax fibers two different side screw speed was selected to investigate the effect of side screw speed on mechanical properties, fiber degradation and viscosity of the compound material. Side screw speed is proportional to production rate. Samples containing 30 wt% flax fibers were processed with side screw speed of 238 rpm (sample 30 wt% A) and 446 rpm (Sample 30 wt% B) respectively. The side screw speed was higher for sample 30 wt% (B) in comparison to sample 30 wt% (A). The temperature profile in extruder was 180/190/200/200/205°C.

3.2.2 Injection Molding

An injection molding machine (Arburg Model 320c) was used to manufacture the samples. Granulates were dried for 12 hours at 80 °C before injection molding. A temperature profile of 150/160/180/190/190°C was selected in the barrel. The injection molded sample was a 120x120x2 mm plate. The mold temperature was set to 40°C and the cooling time was 25 seconds. After injection molding of the plates, dog-bone shaped samples were machined from the plates for the investigation of the mechanical properties.

3.3 Characterization

3.3.1 Fiber Degradation Characterization

To investigate the fiber geometry and fiber degradation during processing of the compound, the PLA was dissolved in Chloroform at 50°C. After separating the fibers, an optical microscope (Olympus Model BH-2) was used to investigate the fiber geometry and imaging. About 150-200 fibers were analyzed for the fiber length distribution, fiber diameter distribution and fiber aspect ratio distribution investigations.

3.3.2 Fiber Orientation Characterization

Synchrotron micro computed tomography was used to image the three dimensional structure of the fiber orientation distribution in the flax fiber reinforced PLA biopolymer composite samples. Synchrotron tomography was performed at European synchrotron radiation facility Grenoble in France at beam line ID19. A spatial resolution of 0.75 µm was used to obtain high quality images. The images obtained from synchrotron tomography have been processed in image processing program Amira 5.3.1 to reconstruct the 3D-structure of the short flax fiber reinforced composite. For quantitate analysis of the 3D structure and determination of the fiber orientation tensor the MAVI 1.4.1 image processing program was used.

4 Theory

The following notations are used during the micromechanical modeling. Boldface symbols denote tensors the order of which is indicated by the context. Dots and colons are used to indicate tensor products contracted over one and two indices respectively. The symbols **1** and ***I*** designate the second and fourth order symmetric identity tensor respectively. The symbol ⟨***Y***⟩ describes the average of the properties over the defined volume.

4.1 Homogenization Concept

Mean field homogenization techniques are based on the volume average of the stress and strain over a representative volume element (RVE). To consider the fiber geometry distribution or aspect ratio distribution the concept of multi-phase composite has been used in this study. In natural fiber reinforced composites the fiber length distribution (FLD) and fiber diameter distribution are wide where the fiber degradation and defibrillation is dominant during the compounding and injection molding of the composite at high melt temperatures and shear rates. Fibers have been categorized into N classes with different aspect ratio (a_r). Each class has been considered as a phase (i). Consider a RVE with N classes of fibers where the volume fraction of the matrix is v_0. Each fiber is characterized with a unit vector **p** and a determined aspect ratio a_r(i). The fibers are classified into N classes of volume fraction v_i where:

$$v_0 + \sum_{i=1}^{N} v_i = 1 \qquad (1)$$

Each class of fiber is characterized by a given constant aspect ratio and a differentiable fiber orientation distribution function (ODF) $1/_i(\mathrm{p})$ such that the probability of finding a fiber belonging to class i and whose orientation is between **p** and **p**+d**p** is $1/_i(\mathrm{p})\mathrm{dp}$. The ODF satisfies the following conditions:

$$1/(\mathrm{p}) = 1/(-\mathrm{p}) \;,\; \oint 1/(\mathrm{p})\, \mathrm{dp} = 1 \qquad (2)$$

The first quality expresses the fact that the fibers oriented at **p** and –**p** are indistinguishable and the second is a normalization condition [9]. Each fiber class in the RVE is decomposed into pseudo-grains. Each pseudo grain ($w_{i,p}$) is a two phase composite containing the matrix material in concentration v_0 and inclusions in concentration (1- v_0) between **p** and **p**+d**p**. Let us to represent p(x) as an arbitrary microfield like micro strain or micro stress. It can be shown that the volume average of p(x) over the RVE can be written as follow [8]:

$$\langle \mathrm{p} \rangle_{\Omega} = \sum_{i=1}^{N} \frac{v_i}{1-v_0} \langle \langle \mathrm{p} \rangle_{w_{i,p}} \rangle_{\mathrm{lJ}_i} \equiv \langle \langle \mathrm{p} \rangle_{w_{i,p}} \rangle_{i,\mathrm{lJ}_i} \qquad (3)$$

Schematically equation (3) can be shown in Figure (1) and Figure (2) respectively. Figure (1) shows the classification of the fibers in to N class of fibers with the same aspect ratio. Figure (2) shows the decomposition of each class of fibers into a set of infinitesimal pseudo-grains with the same orientation. The orientation average of a function $p(p)$ is its ODF-weighted average defined as:

$$\langle p(p)\rangle_{IJ_i} \equiv \oint 1\text{-}(p)\psi_i(p)dp \qquad (4)$$

4.2 Homogenization of the pseudo-grains: First step homogenization

The Mori-Tanaka homogenization model is used as the first step of homogenization. The homogenization technique has been based on the solution of Eshelby for single inclusion. Eshelby results show that if an elastic homogeneous ellipsoidal inclusion is subject to a homogeneous strain E, the stress and strain in the inclusion would be uniform [10].

$$O_i = \langle O\rangle_i \text{ and } E_i = \langle E\rangle_i \qquad (5)$$

Where index i refers to the inclusions. The uniform strain in the inclusion is related to the applied strain according to the following equation [11]:

$$_i = H : E \qquad (6)$$

Where the single inclusion concentration tensor has the following form [11]:

$$H = \{/ + S: [(C_0)^{-1}: C_1 - /]\}^{-1} \qquad (7)$$

Here **S** is referred to Eshelby tensor and depends only on the material properties of the matrix material (**c**$_0$) and on the aspect ratio (a=L/D) of the inclusions or fibers. The strain average per phase is related by strain concentration tensor B as below [11]:

$$\langle \ \rangle_{W_1} = B : \langle \ \rangle_{W_0} \qquad (8)$$

And per phase average strains are related to the macro strain ⟨ ⟩ as below [11]:

$$\langle \ \rangle_{W_0} = [v_1 B + (1 - v_1)/]^{-1} : \langle \ \rangle \qquad (9)$$

$$\langle \ \rangle_{W_1} = B : [v_1 B + (1 - v_1)/]^{-1} : \langle \ \rangle \qquad (10)$$

The Mori-Tanaka assumes that each particle sees the far field strain equal to the average strain in the matrix or [7]:

$$B = H \qquad (11)$$

Finally the macro stiffness can be calculated according to the following equation for M-T homogenization technique [7]:

$$C = [v_1 C_1 : H + (1 - v_1)C_0] : [v_1 H + (1 - v_1)/]^{-1} \qquad (12)$$

The stiffness tensor of a composite consisting of fibers with the same orientation has been identified until now. In a process like injection molding where high shear rates are dominant, the fibers could arrange differently along the thickness of the plate. Therefore the orientation averaging of the properties is essential as equation (4). The reconstruction of the orientation distribution function with second order tensor has been performed according to [9]. Each class of fibers is divided into a defined number of pseudo-grains with the specific orientation **p** and the reconstruction of the fiber orientation distribution is performed with the second order tensor a_{ij} which has been specified by synchrotron imaging.

4.3 Second step homogenization: Voigt model

The second step of the homogenization is the homogenization of the pseudo-grains. It has been reported that the two step homogenization following the Voigt model leads to better results [12]. However theoretically it is possible to perform other homogenization techniques like Mori-Tanaka as the first step but it might result in unacceptable predictions [8]. Mori-Tanaka can be applied in first step successfully where all fibers have the same orientation and aspect ratio. In this step the homogenization of the pseudo-grains with different orientation has been performed using Voigt model over all N fiber classes. The basic assumption of the Voigt model is that the strain field is uniform over the RVE and the stiffness can be calculated for the multi-phase composite over different aspect ratio ($a_{r(i)}$, i=1-N) as below where $C_{i,p}$ shows the stiffness of one pseudo-grain with fibers in class i:

$$C = \langle (C_{i,p})\rangle_{i,\mathbb{N}1_i} \qquad (13)$$

5 Results and Interpretation

The mechanical and geometrical information of the constituents are essential information for micromechanical modeling and homogenization. Flax fibers that are processed differently result in different mechanical properties. Other parameters like flax fiber diameter, growing and weather conditions could also affect the mechanical properties of flax [13, 14]. Epoxy-Flax unidirectional (UD) composites were manufactured to back calculate the Young's modulus of the fibers using the rule of mixture. Epoxy plates were made also with the same Epoxy that has been used for vacuum infusion of the Epoxy-Flax UD composites. Figure (3) shows the stress-strain behavior of the pure Epoxy and Epoxy-Flax UD composites. Using rule of mixture as below:

$$E_c = E_f V_f + E_m V_m \qquad (14)$$

Where V_f=0.52 and E_c and E_m are known from figure (3) the Young modulus of the flax fiber could be back calculated about 36051 MPa. Using rule of mixture it is also possible to back calculate the poisson's ratio of the flax fiber. Fiber density was measured also by measuring the density of the pure matrix and composite and back calculation of the fiber density. All results concerning mechanical properties of the constituents are presented in table (1) and were used in micromechanical modeling and homogenization.

Besides mechanical properties of the constituents the geometrical properties of the fibers are also important as it has been mentioned in equation (7). Figure (4) and figure (5) show the flax fiber length distribution and flax fiber aspect ratio distribution in case of PLA reinforced with 20 wt% flax fibers. The flax fiber aspect ratio distribution has been implemented according to figure (5) in homogenization of PLA with 20 wt% flax fibers. High temperature and high shear forces during compounding and injection molding cause the fiber degradation. Fiber diameter degradation is mostly because of the defibrillation of the fiber bundles to elementary flax fibers [15]. Figure (6) and Figure (7) show the fiber length and diameter distribution in case of PLA reinforced with 30 wt% flax (A) samples. The compounding and injection molding parameters in samples with 20 wt% flax and 30 wt% flax (A) fibers are mostly the same however the (number) mean value of fiber length has been reduced from 161µm to 149µm. This could be because of the higher viscosity of the melt for the compound with 30 wt% flax fibers which result in more shear forces and fiber degradation [3, 16]. Another attempt was made also to investigate the influence of screw speed in compounding process on the flax fiber degradation and consequently the mechanical properties. In case of PLA reinforced with 30 wt% flax (B) the screw speed was higher than case (A). Figure (8) and (9) show respectively the fiber length and aspect ratio distribution in case of PLA reinforced with 30 wt% flax (B). The fiber average length was decreased from 149µm to 124µm in comparison to sample (A) because of the higher screw speed during compounding process. The fiber aspect ratio and Young's modulus is slightly higher than sample (A). The fiber aspect ratio distribution according to figure (9) was used for the homogenization of the sample 30 wt% flax (B).

The higher screw speed is directly proportional to production rate but more energy consumption. The question is to find an optimum compromise between mechanical properties, energy consumption and production rate. A similar research has been published recently on bamboo fiber reinforced PLA, where the screw speed doesn't affect mechanical properties significantly [17].

As mentioned in the introduction section of this paper number average of the aspect ratio are used mostly for micromechanical modeling and homogenization. It is also possible to calculate RMS and skewed average with the same data available for the calculation of number average and according to the equations mentioned in introduction section of this paper. These averaging methods have been used also in this study to compare the result of micromechanical modeling with fiber aspect ratio distribution consideration. Table (2) shows briefly the result of different averaging methods. Another averaging method which is common in glass fiber reinforced composites is weighted average where the weight of fiber with different geometry is measured after burning of the polymer. This averaging method is not applicable in case of natural fiber reinforced polymer feasibly as glass fiber reinforced composite as both constituents are burnable.

As it is mentioned the mechanical properties are highly orientation dependent. For this reason it is important to have information on the fiber orientation distribution in the samples. However imaging and 3D tomography of flax fiber reinforced PLA is challenging. The density similarity of the natural fibers and PLA biopolymer along with the base structural element (carbon) similarity makes it difficult to have good contrast between the constituents. The same problem exist for modern carbon fiber reinforced composites. The good signal to noise ratio in synchrotron micro-computed tomography lead to higher contrast between constituents in a resolution about 1µm [18]. A single fiber in short fiber composite can be represented by a unit vector **p** along the fiber. One set of orientation tensors can be defined by forming dyadic products of the vector **p** and then integrating the product of these tensors with the distribution function over all possible directions [19]. The second order tensor is defined as below:

$$a_{ij} = \oint p_i \, p_j \mathrm{l}/\mathrm{J}(p)dp \quad (15)$$

The diagonal components of the second order tensor, which is determined with the image processing of the Synchrotron images is presented in table (3) for the PLA reinforced with 20 wt% flax and 30 wt% flax. This information is used for orientation averaging according to equation (4).

The result of the micromechanical modeling and homogenization for samples with 20 wt% flax is presented in figure (10). The simulations were performed using ABAQUS FE-Code and incorporation of a user material subroutine. The Young's modulus determined experimentally is presented along with the homogenization results considering fiber aspect ratio distribution and homogenization with the number average, RMS average and skewed average of the aspect ratio respectively. Figure (11) and Figure (12) show simulation results for PLA reinforced with 30 wt% flax (A) and (B) samples. The stiffness prediction error is summarized in table (4) for all three samples. It can be seen that using the skewed average of the aspect ratio of the fibers in homogenization gives more compatible results to experiments in all samples or the lowest error. Using number average of aspect ratio overestimates the stiffness and gives stiffer results in comparison to homogenization with consideration of fiber aspect ratio distribution. The results of stiffness prediction with different averaging methods including fiber aspect ratio distribution consideration could be summarized as below:

$$C^{\text{skewed.AV}} \leq C^{\text{FLD,FOD}} \leq C^{\text{N.AV}} \leq C^{\text{RMS.AV}}$$

6 Conclusions

Fiber geometry and orientation distribution has been integrated in micromechanical modeling and applied to flax fiber reinforced composites. Property and geometry variation in natural fibers are inevitable as they are of natural origin. Geometrical and mechanical properties of the constituents were investigated and identified. Noticeable fiber length degradation identified during compounding and injection molding along with fiber diameter degradation due to fiber bundle defibrillation. Orientation distribution of the fibers investigated with Synchrotron tomography and the orientation tensors are identified. Orientation investigations showed that not all fibers are oriented in flow direction. Consideration of fiber geometry distribution resulted in more compatible results to experiments as the commonly used number average of the geometry in homogenization and micromechanical modeling. Using skewed average of the fiber aspect ratio, which gives higher weight to shorter fibers, resulted in the best stiffness predictions.

Tables and Figures

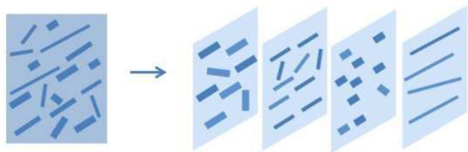

Fig. 1. Decomposition of the fibers into N class with different geometry

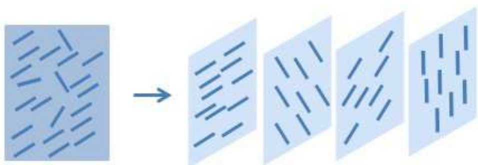

Fig. 2. Decomposition of the class i of fibres with the same aspect ratio into a set of pseudo-grains with the same orientation

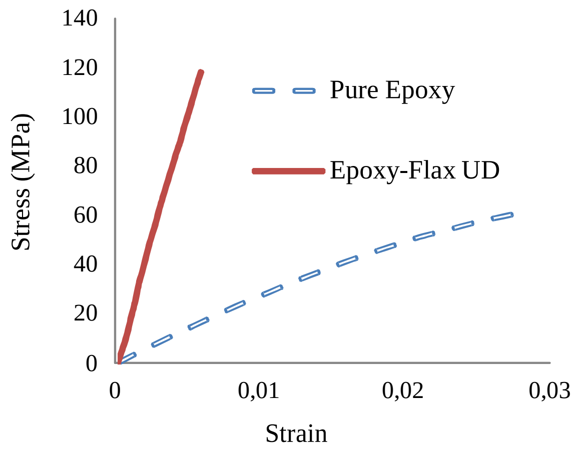

Fig.3. Stress strain diagram of the Epoxy-Flax UD laminates and pure Epoxy (V_f=0.52)

Table.1. Material properties of the constituents used for micromechanical modeling (Flax fiber reinforced PLA)

Fiber Young's Modulus	36051 MPa
Fiber Poisson's ratio	0.312
Fiber density	1.49 gr/cm^3
Fiber weight fraction	20 and 30 wt%
Matrix Young's Modulus	3450 MPa
Matrix Poisson's Ratio	0.336
Matrix Density	1.23 gr/cm^3

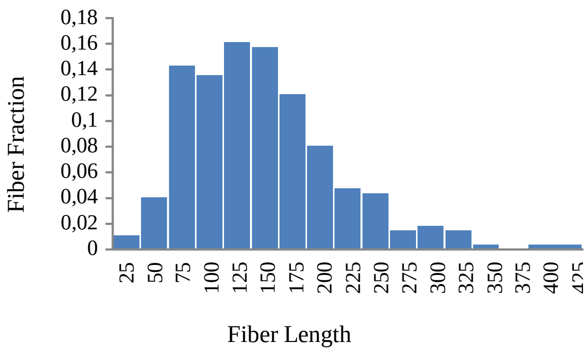

Fig. 4. Fiber length distribution for PLA+20Wt% Flax fibers applied in homogenization

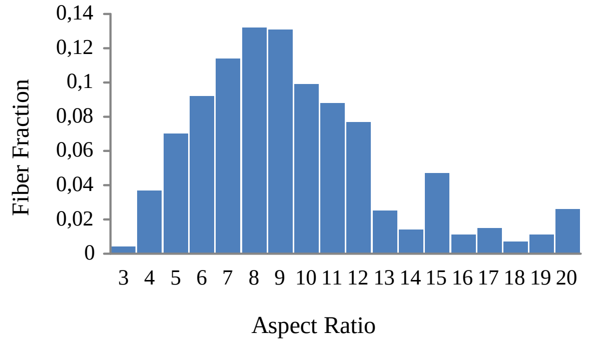

Fig. 5. Fiber aspect ratio distribution for PLA+20wt% Flax fibers applied in homogenization

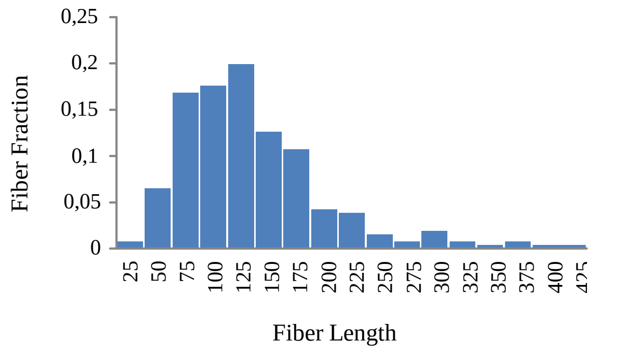

Fig.6. Fiber length distribution for PLA+30wt% Flax (A) applied in homogenization

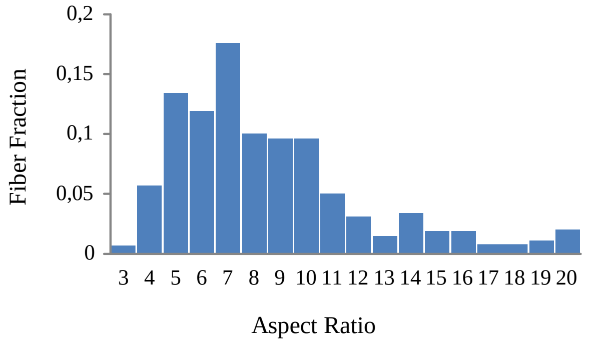

Fig.7. Fiber aspect ratio distribution for PLA+30wt% Flax (A) applied in homogenization

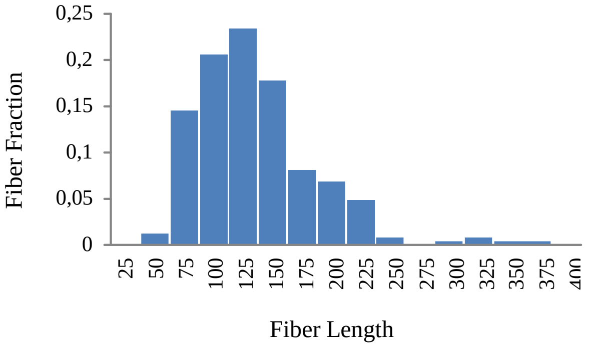

Fig.8. Fiber length distribution for PLA+30wt% Flax (B) applied in homogenization

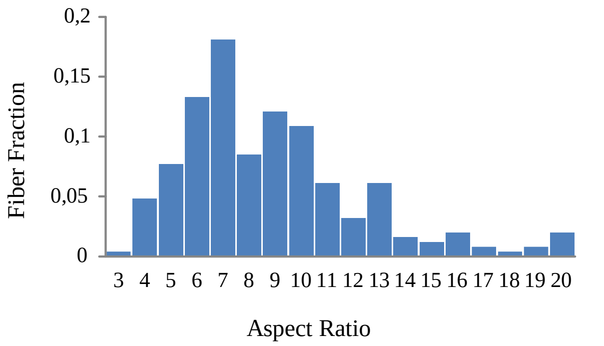

Fig.9. Fiber aspect ratio distribution for PLA+30wt% Flax (B) applied in homogenization

Table.2. Different averaging data of the fiber aspect ratio distribution

Sample	Number Average	RMS Average	Skewed Average
20 wt% Flax	9.65	10.38	8.24
30wt%Flax(A)	8.46	9.34	7.06
30wt%Flax(B)	8.9	9.6	7.7

Table.3. Orientation tensor of short flax fiber reinforced PLA obtained from Synchrotron tomography

Sample	a_{11}	a_{22}	a_{33}
30wt%Flax	0.5	0.26	0.24
20wt%Flax	0.53	0.25	0.22

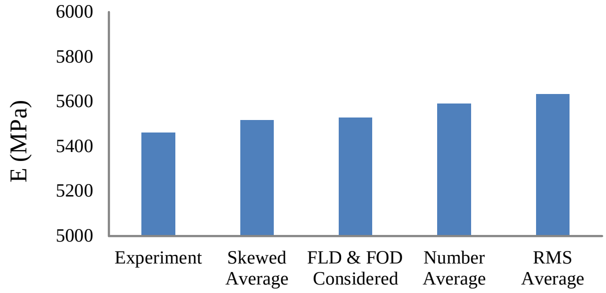

Fig.10. Stiffness prediction using homogenization technique in comparison to experiment for PLA+20wt% Flax

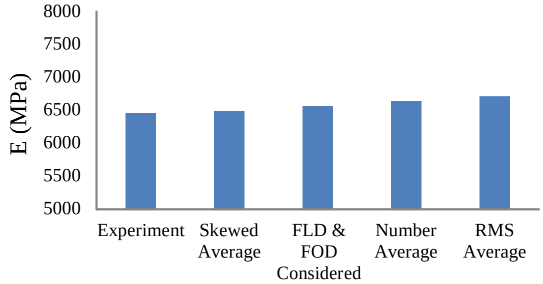

Fig.11. Stiffness prediction using homogenization technique in comparison to experiment for PLA+30wt% Flax (A)

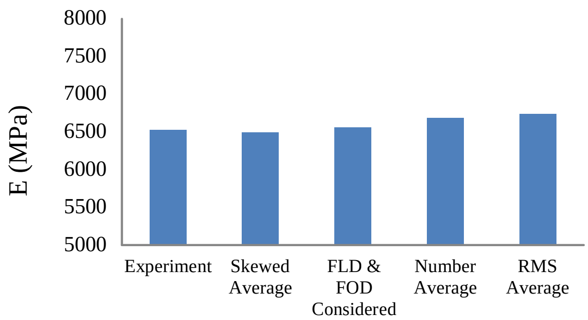

Fig.12. Stiffness prediction using homogenization technique in comparison to experiment for PLA+30wt% Flax (B)

Table.4. Stiffness prediction error percentage with fiber geometry distribution consideration and different averaging methods in comparison to experiment

Sample	FLD&FOD	Number Average	RMS Average	Skewed Average
20% Flax	1.2	2.4	3.2	1
30% FlaxA	1.6	2.8	3.9	0.5
30%FlaxB	0.5	2.4	3.2	-0.5

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