

Enhancement of the Mechanical Behavior of Starch-Palm Fiber Composites

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Abstract. This study discusses the fabrication of starch- based hybrid composite reinforced with chopped randomly oriented flax, sisal, and date palm fibers. The tensile properties, before and after chemical treatment, as well as the morphology of the fibers were evaluated. The hybrid composites were fabricated using hot compaction technique at 5MPa and 160°C for 30min. Fracture surface investigations using field emission scanning microscopy showed a good adhesion between fibers and matrix. The fracture surface revealed the presence of matrix micro cracks as well as fibers fracture and pullout. Hybrid composites containing 20 vf % sisal, and 5 vf % flax at 25 vf % date palm as well as 35vf% sisal, and 5 vf % flax at 10 vf % date palm had the optimum mechanical properties and consequently can serve as competitive eco-friendly candidates for various applications. A finite element (FE) approach was developed to simplify the treatment of random orientation of chopped fibers and predict elastic modulus using Embedded Element technique. Analyses based on rule of hybrid composite (ROHM), COX rule, and Leowenstein rule are presented to validate both experimental and FE numerical results. The FE results compared favorably with the experimental results.

Introduction

The construction of natural fiber bio-composite may have very good applications in the automotive and transportation industry such as car door panels which may save up to 45% from door panel carrier weight, bio-based cushions, the driver's seat back rest, etc. Moreover, reducing cost of bio-composites will be more desirable to industrial economic development [1].

Biodegradable composite materials based on natural fibers and starch had attracted attention over the past several years. Starch is one of polysaccharide matrices. Owing to its low cost, availability as a renewable resource, biodegradable and nontoxic degradation products, it is one of the important raw materials used for packaging, biomedical applications, and in some



automotive parts. Starch, however, has some drawbacks such as poor melting process ability, high water solubility, difficulty of processing, and brittleness. Gelatinization process converts starch to thermoplastic starch (TPS) and improves those draw backs [2-3].

Date-Palm fiber (DPF) is a low cost material with mechanical properties that depend on the place of extraction. DPF can be considered one of the best types of fibers regarding several evaluation criteria such as specific strength to cost ratio if compared to other fiber types [4]. Sisal fiber (SF) is known by its high strength but it has some limitations such as high cost and is not cultivated in Egypt [5]. Flax fiber (FF) has mechanical properties near to SF; however, the cultivation of Flax has been diminished in Egypt as it can be replaced by other imported materials [1, 6].

Several fiber types are incorporated into hybrid composites and such composites can be tailored to meet various design requirements in a more economical way than conventional composites. Their behavior depends on the characteristics and the mechanical properties of the incorporated fibers [7]. Several factors will affect the composite mechanical properties such as fiber type, length, orientation, characterization, resin type, and volume fraction of the reinforcements [8].

The objective of the present work is to study the behavior of starch- based hybrid composites containing three types of fibers, namely, DPF, FF, and SF, and to compare the mechanical properties obtained to flax/date palm hybrid composite at 1:1 matrix/ fiber volumetric ratio. The present work involves both experimental and numerical investigations. Composite preparation stage was performed by using different mixtures of fibers with different volume fraction as shown in Table 1. The composite analysis first stage was based on measuring mechanical properties, examining the fracture surfaces, and applying the morphological characterization of the materials. Finally the finite element analysis (FEA) stage; where different models were implemented using ABAQUS software.

Mixed FE-analytical approaches are suggested for the prediction of the Young's Modulus of reinforced composite having randomly oriented chopped fibers. An attempt is suggested to overcome the difficulty of representing random orientation of chopped fiber across composite in finite element representation. The attempt is based on unidirectional fibers having 33.3% volume fraction to represent the actual fiber volume fraction of randomly oriented chopped fiber in the composite.

Experimental Work

Materials Preparation, Characterization, and Mechanical Testing: Corn Starch was purchased from Aro Sheri Company in Egypt with an average particle size of 16 μ m. Glycerin with 99.7% purity was used as a plasticizer. Gelatinization process of starch following Ref. [3] methodology is used to form TPS by mixing native starch with 30Wt. % glycerin and 20Wt. % distilled water in temperature range from 60–80°C. Adding glycerin improves process ability and reduces embrittlement by inhibiting the retro gradation process. The TPS was kept in polyethylene bags over night to enhance its flow properties before being used.

Flax and sisal strands were donated by the Egyptian Industrial Center E.I.C. DPFs were extracted from the stem of date palm trees at the American University in Cairo. Sodium hydroxide (NaOH) with molecular weight 40g/mol. was used for alkaline treatment of fibers. The three fibers (DPF, FF, and SF) were chemically treated using the following procedure: 1) Dipping in 5% NaOH for 3 hours at room temperature. 2) Rinsing the treated fibers in cold water. 3) Dipping the fibers in 5% acetic acid to remove any excess NaOH from fibers surface. 4) Rinsing in cold water and oven drying at 120°C for 3hrs. 5) The treated fibers were cut

manually into short fibers with average length varies from 15 to 30mm according to the aspect ratio.

Characterization and testing were performed using the following procedures: 1) Measuring fibers diameter before and after chemical treatment by Leica stereoscopic microscope using 10 samples with a μm divisions scale lens. 2) Measuring density of TPS and fibers using the Mittler Toledo densitometer for 10 samples (Xylene was used as the immersing liquid with relative density is 0.86). 3) Tensile testing using Instron 3382 universal testing machine at 50% RH, 18°C and strain rate of 0.01per min. with a gauge length of 50mm at strain rate of 0.01/min. 4) Fracture surface study of fibers Using ZEISS scanning electron microscope (SEM) operated at a vacuum pressure $1\text{e-}4$ mbar and 8KV.

Hybrid Random Composite Preparation: The different composites were prepared using 1:1 fiber to matrix volume fraction according to Eqs. 1- 4. The DPF was used with 50vf% to 20vf% of fiber at different SF and FF volume fraction percentages. The fiber cutting length was based on fiber aspect ratios. Stearic acid with concentration 98% was used as a mold coating releasing agent. The fiber mixture was uniformly distributed in a die cavity (120X80X10mm) to form ten different fiber volume fractions of hybrid composites as shown in Table 1. The emulsified TPS was poured on the random mixed fibers. The mixture was then pre-heated at $140\pm 3^\circ\text{C}$ for 30min to remove excess water from the mixture. This was followed by hot pressing at 5MPa and 160°C for another 30min then cooling at a rate of about $2^\circ\text{C}/\text{min}$.

$$V_T = V_f + V_m = \frac{Wt_f}{\rho_f} + \frac{Wt_m}{\rho_m} = \left(\frac{\pi}{4} * d_f^2 * l_f * n_f\right) + (W_m * l_m * h_m) \quad (1)$$

$$\sum_i^n v_i = 1 \quad (2)$$

$$v_f = V_f/V_T \quad (3)$$

$$v_m = V_m/V_T \quad (4)$$

Where; v_i is the volume fractional for constituent i, v_f and v_m are fiber and matrix volume fracture, V_f and V_m are fiber and matrix volume, V_T is Total Volume, Wt_f and Wt_m are fiber and matrix weights, ρ_f and ρ_m are fiber and matrix density, d_f is the fiber diameter, l_f is the fiber length, n_f is the number of the different fibers used in a composite, w_m , h_m , l_m are matrix width, thickness, and length respectively.

Hybrid Composite Characterization and Mechanical Testing: Density measurement of composite using the densitometer for five samples per each hybrid composite is very important as indicator for measuring void fraction percentage. Voids can be as a result of an imperfection from the processing of the material and is generally deemed undesirable. Its presence can affect the mechanical properties and lifespan of the composite. Voids can also act as a crack nucleation site as well as allow moisture to penetrate the composite [9]. Void percentage was calculated using Eq. 5, and Eq. 6.

$$\rho_{theoretical} = \rho_c = \rho_f V_f + \rho_m (1 - V_f) \quad (5)$$

$$void\ fraction = \frac{\rho_{theoretical} - \rho_{exp}}{\rho_{theoretical}} \quad (6)$$

Tension tests were carried out at a strain rate of 2mm/min for 5 samples for the different volume fraction composites. Test specimens were cut manually in the form of rectangular bars 80x8x2mm according to ASTM D5083-10 with working gauge length equals 50mm.

In case of analytical calculation for UTS; Kelly and Tyson [10] proposed an approach to deal with discontinuous unidirectional fiber and to overcome the problem of unequal strain in fibers and matrix using Eq. 7 and Eq. 8. It is accomplished by assuming perfect bonding between fibers and matrix, besides both fibers and matrix behave as linear elastic material so the stress on the fibers start from zero at ends to reach the maximum stress within fiber length. If the fibers are shorter than critical length, they cannot be loaded to their failure stress.

$$L_c = \frac{\sigma_f d_f}{2\tau} \quad (7)$$

$$\sigma_c = \sum \left(\frac{3}{8} \left(1 - \frac{L_c}{2L} \right) \sigma_f V_f \right) + \sigma_m V_m \quad (8)$$

Where; L_c is the critical length, τ is the shear strength of fiber/matrix bond which is set to be about 0.5 of matrix strength, and L is the fiber length.

In case of Elastic modulus prediction; ROHM [11] was used to present the elastic modulus for random fibers oriented composite (E_c) by substituting Eq. 10 and Eq. 11 in Eq. 9. Cox [12] implemented an analytical equation, where its concept is based on averaging the elastic constants over all possible orientations by integration as shown in Eq. 12. Leowenstein [13] governing rule is as shown in Eq. 13. Also Leowenstein rule [13] is based on ROHM [11] criteria, whereas; the most effective volume fraction is fiber. These predictions are only good at very low fiber volume fractions. At high fiber volume fractions, the predicted modulus is much higher than measured.

$$E_c = \frac{3}{8} E_1 + \frac{5}{8} E_2 \quad (9)$$

$$E_1 = E_f V_f + E_m V_m = E_f v_{ff} + E_s v_{fs} + E_D v_{fd} + E_m v_m \quad (10)$$

$$E_2 = E_m \left(\frac{1 + \xi (\sum \eta_f V_f)}{1 - (\sum \eta_f V_f)} \right) \quad (11)$$

$$E_c = \frac{1}{3} E_1 \quad (12)$$

$$E_c = \frac{3}{8} E_1 \quad (13)$$

Where; η_f is the fiber stress portioning parameter in transverse direction, and ξ is the curve fitting parameter.

Finite Element Proposed Mixed Technique

In the present work finite element analyses (FEA) using ABAQUS 6.12 commercial software were conducted to predict the Elastic Modulus of fiber reinforced composites using embedded element technique. In order to make an adequate prediction of Elastic Modulus for natural fiber reinforced composites using FEM, it is essential to carry out an appropriate analysis which requires both the correct mechanical characterization of the materials and composite structure.

Analytical or numerical micro mechanical analysis of fiber reinforced composites had involved the study of the Representative Element Volume (RVE).

In an attempt to avoid the difficulties associated with modeling randomly oriented chopped fibers, two simplified FE approaches were used. In the first approach, a mixed FE / ROHM model was developed for the prediction of Elastic Modulus based on Eq. 9 to represent final hybrid composite Elastic Modulus. Fibers were placed in the matrix using array as longitudinal beam element in unidirectional and with approximate equal spaces. The load was applied in longitudinal and transverse directions. The output data E1 and E2 are substituted in Eq. 9. Based on this analysis, mixed FE/Cox and mixed FE/Leowenstein was made by substitution of E1 results in Eq. 12 and Eq. 13. The results were compared to those obtained from the mixed FE/ROHM model.

In the second approach; two modified mixed FEMs were examined. The first modified mixed model followed Leowenstein approach in which fibers RVE in the model constitute 3/8 from the fibers actual volume fractional. The second modified mixed FE model followed Cox approach in which fibers RVE in model constitute 1/3 of the fibers actual volume fractional. The results of two modified mixed FEMs were used directly to predict the Elastic Modulus of randomly oriented chopped fiber hybrid composite.

In all analyses, the composite structure was assumed to be a rectangular prism consisting of fibers surrounded by matrices in which both were created as 3-D elements. The matrix structure was 3D deformable solid model and was shaped to the required thickness based on the RVE. The fibers were modeled as 3D deformable wires lay in a plane, as the wire represented the beam elements immersed inside matrix. The fiber length must be equal to the length of the hosting matrix to avoid the end point factor. The area of matrix is controlled by matrix volume fraction in which thickness and width are assumed to be equal. In the present model; the structure of meshing used in case of fiber was beam element with 2 nodes but the matrix was eight-node hexahedral element shape. .

Results and Discussion

Physical and Mechanical Properties of Fibers and Matrix: The experimental measurements for FF, DPF, and SF diameter ranges were 9-20 μm , 19.6-30 μm , and 27-187.5 μm after chemical treatment stage, and the average densities were 1.44, 1.352, and 1.43 gm/cm^3 , respectively. The ultimate tensile strength (UTS) for FF, DPF, and SF were 351 MPa, 195 MPa, and 565.7 Mpa. And Young's moduli were 21.5 GPA, 8.81 GPA, and 41.2 GPA, respectively.

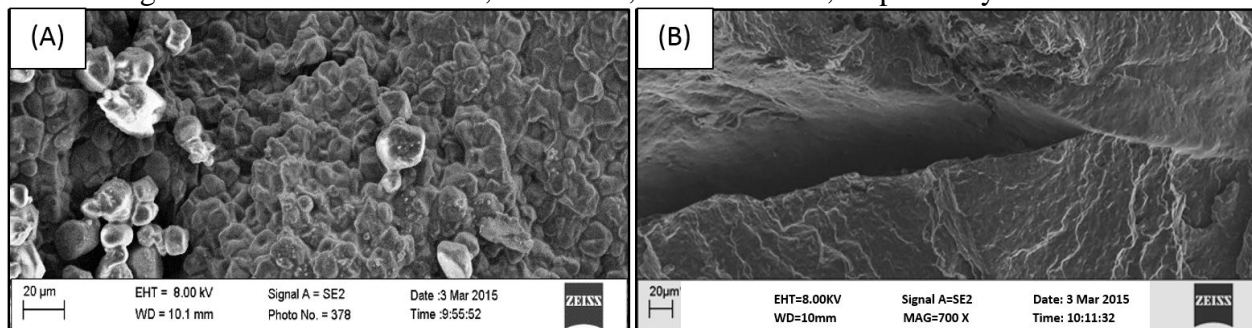


Fig. 1: (A) SEM investigation for plasticized corn starch; agglomerated and connected corn starch particles, (B) SEM fracture surface investigation of hot-pressed matrix (TPS emulsion technique); smooth.

The matrix granules size investigated by SEM varied from 4.9 to 33 μm with average size being about 16 μm . The presence of dimpled structure in TPS blend increased the ductility of

material as shown in Fig.1A. This effect was more noticeable in composite fracture SEM in Fig.1B. The hot pressed TPS matrix had an average density of 1.445 gm/cm³, UTS of 3.6 MPa, and Young’s modulus of 378 MPa.

Physical Properties of Composites: The densities of the implemented hybrid composites with different volume fractions of DPF are summarized in Table 1. The void percentage ranged from 1.2% to 4.6%. The variation in diameter along the length of the fibers had an effect on the void contents as well as acting as stress concentration sites that may weaken the final composite mechanical properties and this difference in diameters was more obvious in Fig. 2.

Table 1: Different implemented hybrid composites at 10% and 25% volume fractions DPF and void percentage results.

Composites	Fibers volume fractional			Theoretical Density results (gm /cm ³)	Experimental Density results (gm /cm ³)	Void (%)
	DPF (%)	FF (%)	SF (%)			
SFD0520R	25	5	20	1.422	1.357	4.6
SFD1015R		10	15	1.423	1.405	1.3
SFD1313R		12.5	12.5	1.423	1.404	1.3
SFD1510R		15	10	1.423	1.405	1.2
SFD2005R		20	5	1.424	1.404	1.4
SFD0535R	10	5	35	1.435	1.413	1.5
SFD1525R		15	25	1.436	1.373	4.3
SFD2020R		20	20	1.436	1.374	4.3
SFD2515R		25	15	1.437	1.377	4.1
SFD3505R		35	5	1.437	1.383	3.7

Mechanical Behavior of Composites: The surface fracture morphology of the implemented hybrid composites is shown in Fig. 2. Fig. 2A shows the presence of good adhesion in matrix/fibers and also good wettability. The brittle matrix surface cracking behavior and voids may be as result of the relatively fast cooling rate of composite after compression molding.

Fig. 2B shows fibers failure and unexpected bad adhesion at the matrix/fibers interface, due to the presence of untreated fibers lignin. The fracture of a SF in tension as a result of the inhomogeneity in fibers crosses section would lead to unequal stress distributions. Shear took place across the fiber due to the lateral strains produced accompanied with the axial stresses.

Good fibers wettability is also obvious in Fig.2C, unfortunately; the presence of brittle matrix surface cracking is also detected. This was all due to difficulty of distribution of mixture across composite.

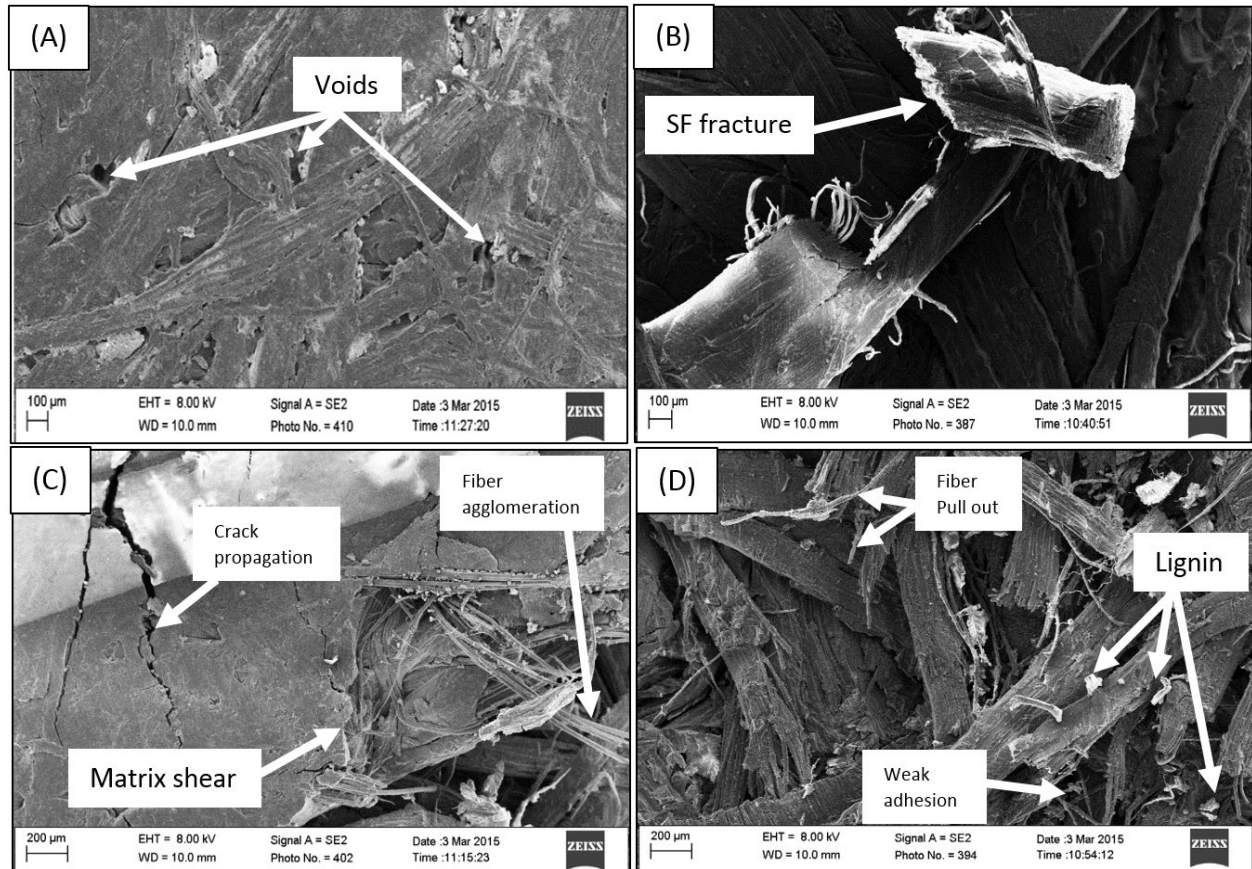


Fig. 2: A) SFD3505R hybrid composite - Matrix surface voids, B) SFD0520R hybrid composite - Fiber cracking, C) SFD2515R hybrid composite – Matrix crack propagation as a result of stress concentration as well as fiber agglomeration, D) SFD2020R hybrid composite-Multiple failure sources.

Fiber aspect ratio, orientation, and agglomeration are other sources of premature failures. With increasing SF vf% at low DPFs vf%, the composite suffered from a combined matrix and fiber shear. The agglomeration of date palm fibers acted as internal stress raisers where cracks were initiated and consequently propagated across the composite as shown in Fig. 2C. In Fig. 2D, fibers pull out and fracture is also detected. The fiber pull out were more obvious with the increase of the FF vf%.

Table 2 shows the UTS results achieved with 25vf% and 10 vf% DPF content in comparison with Kelly and Tyson [10] analytical results. It was found that increasing FF coupled with decreasing SF volume fractional led to the decrease of composite strength. The increase of SF will increase the strength and consequently the cost since SF is more expensive than FF. Knowing that the SF strength is the highest among the three fibers, the experimental results were unexpected. This can be as result of miss-distribution of SF in the composite and/or fabrication problems leading to a large void percentage in the composite. Hybrid composite of 5vf% FF and 35vf% SF shows the highest strength for 25vf% and 10vf% Date-palm. Based on strength and Young's modulus results, this composite is considered to be the best amongst other compositions in the study. Ibrahim et al. [3] observed an average strength of 26.77 MPa using FF and DPF chopped randomly hybrid composite with equal weight fractions. The strength trends show good

agreement to that of Kelly and Tyson results. At equal volume fractions of SF and FF with 10vf% Date-palm; the composite behaves differently than the rest of composites.

Table 2: Experimental UTS results versus Kelly and Tyson analytical results.

Composites	UTS (Mpa)	
	Experimental results	Kelly and Tyson rule [10]
SFD0520R	16.37	62
SFD1015R	8.83	59
SFD1313R	6.68	57
SFD1510R	6.51	56
SFD2005R	6.1	53
SFD0535R	18.56	79
SFD1525R	14.86	73
SFD2020R	18.13	70
SFD2515R	10.12	67

Table 3 shows a comparison of Young’s modulus results using ROHM, Leowenstein and COX. The empirical analytical results show higher values than experimental ones. This can be due to the effect of void percentage, compression molding technique, and the difference in fibers diameters on the composite elastic modulus. The Cox results are the closest results to experimental ones.

The experimental results showed favorable agreement with those of Cox and Leowenstein approaches and show lower agreement than the ROHM approach based on both longitudinal and transverse solutions. It must be noticed also that the voids effect is not taken into consideration in analytical analyses.

The results of the suggested FE/ROHM mixed model show good agreement with the analytical results, but are higher as compared to experimental results. This is expected in view of the presence of large void percent and the lower strength of the fabricated composites compared to that of Ibrahim et al [3] as discussed in the previous section.

Both mixed FE/ Leowenstein and FE/Cox models show a higher results compared to experimental results, with the mixed FE/Cox approach showing closer results to the experimental ones. Modified Cox FEM shows closer agreement to experimental results than Leowenstein based model. In fact, such results are consistent with the fact that less fraction of fibers are used in the Cox approach leading to lower predictions of the Elastic Modulus. Generally speaking, the FEM results compare well with most of the analytical solutions.

In view of the results of the various attempted models, the modified mixed FEM model based on Cox approach was adopted to predict Elastic Modulus of various hybrid composites.

Table 3: Comparison between experiment, analytical and FE Young’s Modulus results.

Composites	Young’s modulus (GPa)									
	Experimental results	Analytical Results			1 st approach			2 nd approach		Final FE adopted approach
		ROHM rule [11]	Leowen-stein rule [13]	Cox rule [12]	FEM mixed ROHM	FEM mixed Leowen-stein rule	FEM mixed COX	FEM based on Leowen-stein rule approach	FEM based on COX approach	Adopted Modified FEM/COX
SFD0520R	2.1-2.69	5.23	4.39	3.90	4.9	4.5	4.05	4.02	2.59	2.59
SFD1015R	1.3-1.91	4.84	4	3.6	4.35	3.99	3.55	2.29	1.6	1.6
SFD1313R	1.16-1.22	4.68	3.84	3.4				3.24	2.29	2.29
SFD1510R	1.74-2.33	4.48	3.64	3.2						3.62
SFD2005R	1.06-1.32	4.10	3.27	2.91						3.75
SFD0535R	4.2-4.7	7.12	6.24	5.54				4.69	4.62	4.62
SFD1525R	2.9-3.26	6.4	5.5	4.9	4.91	4.54	4.038	4.28	4.13	4.13
SFD2020R	2.27-2.68	6.00	5.12	4.6						4.46
SFD2515R	2.03-2.13	5.62	4.75	4.22						4.83
SFD3505R	1.2-2.6	4.87	4.00	3.56						4.33

Concluding Remarks

It should be emphasized that the experimental results generally show lower Elastic Modulus values as compared to both the analytical and the FEM results. This could be attributed to the manual compression molding process used in the present work leading to high void percentage, and the lower efficiency in preparing the composite with no guarantee that the actual volume fraction of fibers is necessarily equal to that in the RVE.

Furthermore, as detailed in Ref. [14] and indicated previously in microscopic and SEM investigations, fiber cross sectional area changes along the fiber length, and fiber lengths are not necessarily cut to equal lengths which could affect the strength of the implemented composite mixture. This is not the case in analytical and FEM solutions where the fibers are assumed to be of uniform cylindrical cross section of equal lengths to overcome the end point effect.

However, it should be noticed that a perfect mixing and bond between matrix and fibers are assumed in analytical and the mixed FEMs leading to results of strength and Elastic Modulus that are generally higher and could be out of range of realistic experimental work. The suggested

Mixed FE results indicate favorable agreement with those of Cox and Leowenstein approaches and show lower predictions than the ROHM approach based on both longitudinal and transverse solutions.

Ref [14] discussed in details the relative cost of the implemented hybrid composites under considerations, and that DPFs are observed to be the cheapest among the three fibers in both markets. The cost of the hybrid composites based on the local market increases with an increase in SF volume fraction.

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