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FABRICATION OF CONDUCTIVE POLYMER COMPOSITES FROM TURKISH HEMP-DERIVED CARBON FIBERS AND THERMOPLASTIC ELASTOMERS

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ABSTRACT: In this study, carbon fibers filled flexible conductive polymer composites were fabricated. Turkish hemp was used to produce conductive carbon fibers. In order to do this, hemp fibers were carbonized under different conditions. After this step, flexible conductive composites were fabricated by using poly[styrene-b-(ethylene-co-butylene)-b-styrene] matrix and hemp-based carbon fibers. Composite films were produced by combination of solvent casting and hot pressing. Various levels of carbon fibers were used in order to determine the percolation behavior of the composites. Morphological and electrical properties of the composite films were analyzed. Electrical resistivity of the samples decreased by increasing the filler ratio.

Keywords: Hemp fibers, Turkish hemp, carbonization, carbon fibers, thermoplastic elastomers, flexible electronics, conductive polymer composites

TÜRK KENEVİRİNDEN ÜRETİLEN KARBON LİFLERİ VE TERMOPLASTİK ELASTOMERLERDEN İLETKEN POLİMER KOMPOZİT ÜRETİMİ

ÖZET: Bu çalışmada, karbon lifleri içeren esnek iletken polimer kompozitler üretilmiştir. İletken karbon liflerinin üretiminde Türk keneviri kullanılmıştır. Bunu yapmak için, kenevir lifleri farklı koşullar altında karbonize edilmiştir. Bu aşamadan sonra, poli[stiren-b-(etilen-co-butilen)-b-stiren] matris ve kenevir esaslı karbon lifleri ile esnek iletken kompozitler üretilmiştir. Kompozit filmler çözeltiden dökme ve sıcak presleme yöntemleri ile üretilmiştir. Kompozit filmlerin perkolasyon davranışını belirlemek için farklı oranlarda karbon lifleri kullanılmıştır. Kompozit filmlerin morfolojik ve elektriksel özellikleri analiz edilmiştir. Kompozitteki karbon lifi miktarı arttıkça, elektriksel özdirenç düşmüştür.

Anahtar Kelimeler: Kenevir lifleri, Türk keneviri, karbonizasyon, termoplastik elastomerler, esnek elektronikler, iletken polimer kompozitler

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1. INTRODUCTION

Hemp is one of the oldest natural polymers used by human being not only because of its high production yield but also for its uncountable benefits in many areas. It is adaptable to various climate conditions, with high growing rates, it can be harvested more than 1 time (indoor) annually and it does not require any pesticides. In addition to these advantages, environmentally friendly nature and low carbon footprint; recently it has been accepted as one of the most sustainable biopolymers in the world. Hemp can be used in many industries in different forms including dust stem, fiber and hurd. The hemp plant basically consists of long cellulosic fibers that are found together with hemicellulose, lignin and pectin. The content of hemp and comparison with other common cellulosic fibers can be from Table 1. Since fibers are cellulosic and aligned in the stem direction, they show high strength and elastic modulus. However, all fibers do not show the same properties throughout the plant stem. Depending on the fiber length, hemp fibers can be evaluated as bast and core fibers. From the outer skin of the plant many layers can be peeled off and separated, and these fiber bundles are called ribbons. The ribbons obtained from the outer layers of the stem skin are longest fibers and they show better properties compared to core fibers [1-5].

Table 1. Composition of various cellulosic fibers

	Hemp	Flax	Jute	Kapok	Sisal
Lignin	2.9-13	27.3	12.9	15.1	4-20
Pectin	0.8-18	NR	NR	NR	0.8-10
Hemicellulose	2-22.4	21.9	NR	NR	10-15
Cellulose	55-78.3	33.3	59.4	43.2	43-88
Other	0.8-7	0.5	NR	NR	1.15-6
References	[1]	[6]	[7]	[7]	[8]

Hemp can be used for many applications including textiles [9], architecture [10], insulation [11], paper [12] and polymer industry [13-15] for many years. In addition to these recent studies were focused on hemp-derived carbon-based products. Although various polymers can be used as a carbon source, hemp is of significance in terms of its affordable price and sustainability. In the literature, hemp dust [4], hemp bast fibers [16, 17], short and entangled hemp fibers [18] hemp hurd, retted hemp hurd [19], hemp stems [20], hemp canes [21] were used and carbon based products including char [4], activated carbon [16, 19, 20], carbon monoliths [21], carbon fibers [18, 22-24] were synthesized by various methods including hydrothermal synthesis [4, 16], carbonization under N_2 gas [18, 21] and so on.

In one of these studies Dicke *et al.*, carbonized the hemp dust at 230 °C for 6h in distilled water in a reactor by a hydrothermal route [4]. In another study, Hossain *et al.* synthesized mesoporous active carbon by using hemp bast fibers. In the first, step biochar was synthesized and in the second step active carbon was obtained at 750 °C for 1 h under argon atmosphere [16]. In addition to fibers, hurd was also used as a carbon source. Liu *et al.*, used hemp hurd and retted hemp hurd and used different activation agents and carbonized the biomass. Obtained activated carbon was used in order to investigate the CO_2 capture and type of hurd and activation agent were found significant in terms of CO_2 adsorption. Another interesting study was carried out by Rosas *et al.* In the study hemp-derived carbon monoliths were obtained from the canes of the hemp stems [21]. In addition to active carbon fabrication, carbon fibers were also fabricated from hemp. In a study carried out by Mijailovic *et al.*, short and entangled hemp fibers were carbonized at 700 and 1000 °C and performed an activation process to obtain activated carbon fibers. The capacitive properties of the carbon fibers were determined, carbonization temperature, time and activation process were found significant in terms of electrical properties of the samples [18]. In another study hemp based activated carbon fibers were prepared and oxidation resistance of the samples were evaluated [22]. Another interesting study was based on the investigation of pesticide absorbency of carbon fibers. As reported in the study, hemp based activated carbon fibers could be used as solid-phase sorbents [23].

As mentioned above hemp-derived carbon materials can be in particle, monolith or fiber form and they are generally fabricated as activated carbon. The hemp-derived carbon based products were used for some applications including soil conditioner [4], gas adsorption [19, 25], catalyst for waste water treatments [17], water vapor adsorption (desiccants) [21], Cr(VI) removal from waste water [26], capacitor electrodes [18], pesticide sorbent [23, 27]. To summarize, in the literature polymer composites of hemp-derived carbon fibers and their electrical characterization were not studied.

In this study, carbon fibers were fabricated by using Turkish hemp fibers. Carbonization was carried out under different conditions in order to optimize the thermal treatment process. Following that flexible conductive composites were fabricated by using hemp-based carbon fibers and SEBS matrix for first time in the literature. Composite films were fabricated by solvent casting and hot pressing. Morphological, electrical and mechanical properties of the samples were analyzed.

2. MATERIALS AND METHODS

2.1 Materials

Poly [styrene-b-(ethylene-co-butylene)-b-styrene] (SEBS) was used as a polymer matrix and supplied from Kraton, G1643. The triblock copolymer with a styrene/ethylene-butylene ratio of 19/81 and a melt flow index of 75 g/10 min (5 kg load at 200 °C). Toluene (99.9% purity) was used as a solvent, supplied from Merck.

Hemp fibers were obtained from a local supplier at Samsun, Turkey. The plants were grown in Narlısaray, Vezirköprü, Samsun. As given in Fig. 1 hemp fibers were obtained from the skin of the plant. They were peeled of and collected. As known, these fiber bundles (skin, bark) were separated from the core of the plant and they are called ribbons because of their geometry. The ribbons had 2-3 mm width and 170-200 mm length. Ribbons were cut into small pieces with the length of 15-20 mm.

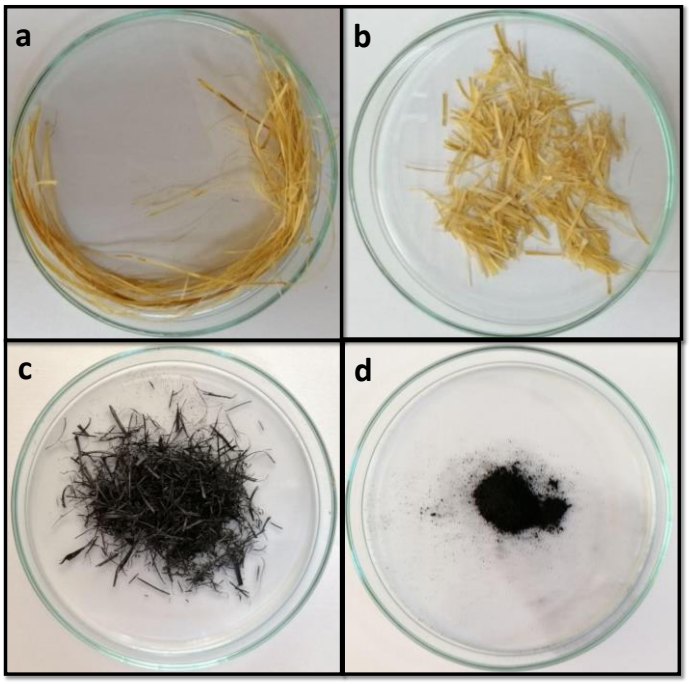

Figure 1. Digital images of a) long hemp ribbons, b) short hemp ribbons, c) carbonized hemp fibers, and d) powderized carbon fibers

2.2 Carbonization of Hemp Fibers

Carbonization of hemp fibers was performed in two steps, known as stabilization and carbonization, as shown in Fig. 2. The process was carried out by using a tube furnace (OTF-1200X, MTI). The stabilization step started at 25 °C and reached to 220 °C with an increment rate of 5 °C min^{-1}. Samples were kept at 220 °C for 5 h to complete the stabilization process. In the second step, temperature was raised to 900 °C by a rate of 2 °C min^{-1}. Fibers were carbonized at 900 °C for 30, 60, 90 and 120 min under argon atmosphere. Carbonization time and carbon yield of the hemp-derived carbon fibers (HDCFs) can be seen from Table 2. During carbonization, hemp fibers turned into electrically conducting carbon fibers [28]. At the end of this process 4 different HDCFs were obtained. After the carbonization HDCFs were grounded by a ceramic mortar and pestle (Fig. 1d). 0.2 g of sample was put into the mortar and powderized by hand for 2 min. This was repeated for all samples. After this, samples were sieved, pelletized and resistance values were determined.

Table 2. The carbonization conditions, carbon yield, average diameter and resistance of the fibers

Sample Code	Process Time (min)	Carbon Yield (%)	Fiber Diameter (µm)	Fiber Resistance (Ω)
S1	30	14.82	16.98	6.98E+1
S2	60	12.73	15.31	1.12E+1
S3	90	26.17	14.82	1.01E+1
S4	120	24.97	12.73	9.95E+0

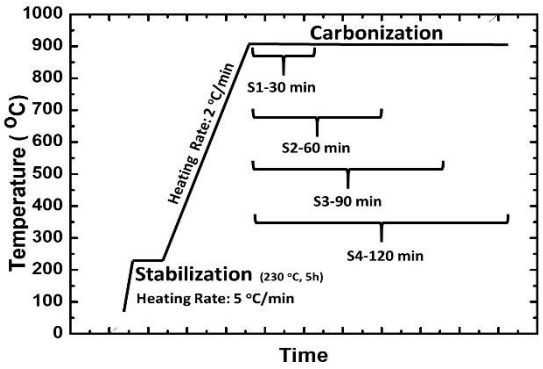

Figure 2. Thermal treatment conditions of the hemp fibers

2.3 Composite Film Fabrication

Composite film fabrication steps can be seen from Fig. 3. The SEBS/toluene solution was prepared with the weight ratio of 1:3 by a magnetic stirrer (Wisd, MSH-20A, Daihan). HDCFs were incorporated into the SEBS solution and this mixture was further mixed for 5 min. Following that, the mixture was casted into a petri dish and then, dried in an oven (Wisd, WOV-20, Daihan) at 40 °C for 12 h to remove the solvent. Casted composites were hot pressed between Teflon coated metal plates at 180 °C under 1 MPa for 120 s. Filler concentration was determined as 0, 35, 40, 45, and 50 wt %.

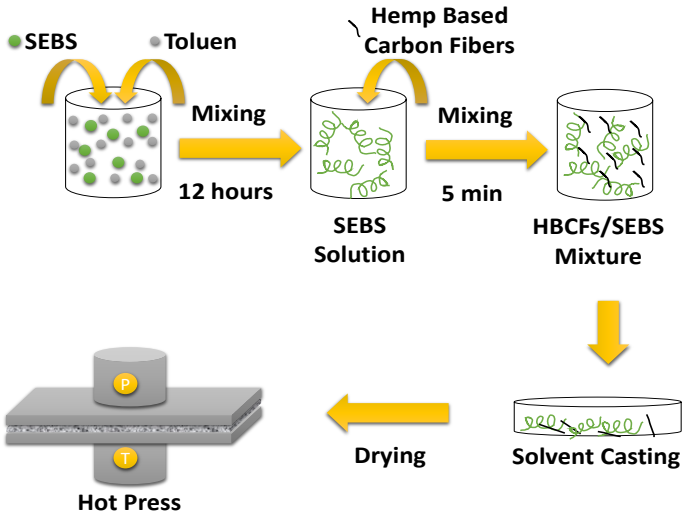

Figure 3. HDCFs/SEBS composite fabrication

2.4 Characterization

The morphology of the carbon fibers and composites were analyzed by an optical microscope instrument (BX51M, Olympus) at 50x (wavelength: 450-700 nm).

Carbon yield was calculated based on the weight change %. A_1: Weight of hemp fibers, A_2: Weight of carbon fibers. Carbon yield % = $(A_2/A_1)*100$.

The resistance of the fibers was determined by a 4-probe set-up consisted of a current source (Keithley 6221) and a nanovoltmeter (Keithley 2182A) under input current of 1 µA.

Thickness of the samples was determined by a digital thickness meter (Asimeto). The average thickness of the samples was determined around 0.5 - 0.6 mm.

The volume resistivity of the samples was measured by considering ASTM D257 standard by an electrometer (6517B Keithley) and a resistivity chamber (8009 Keithley). 5 measurements were performed, and average was calculated.

Mechanical properties of the composite films were characterized Devotrans, DVT GPU/RD testing system with the strain rate of 100 mm/min.

3. RESULTS and DISCUSSION

In order to determine the fiber morphology of carbonized hemp fibers, optical microscope was used (Fig. 4.). As obvious from the images, under all conditions, composites were successfully prepared. Small spherical features are carbon particles that were formed during the grinding step. Diameter distribution histograms can be seen from Fig.5. The average fiber diameter values were determined as 16.98, 15.31, 14.82, 12.73 μm for S1, S2, S3 and S4, respectively. Although the difference was not very drastic S4 had the lowest average fiber diameter value because of higher carbonization time. As known, the most important factors for the weight loss are carbonization temperature and process time.

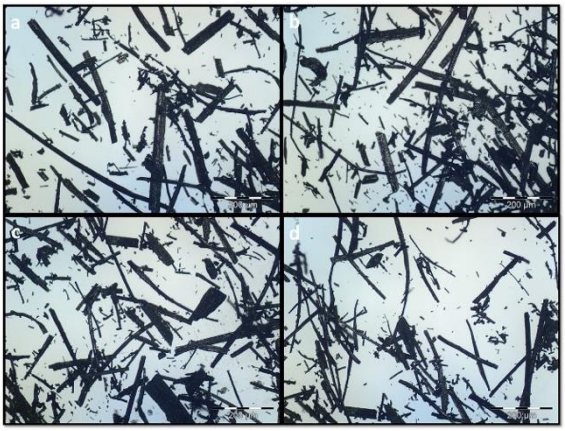

Figure 4. Optical microscope images of HDCFs a) S1, b) S2, c) S3, and d) S4

Since all samples were carbonized at 900 °C, samples showed slight differences in terms of fiber diameter. Generally increased process time led to lower fiber diameter because of higher weight loss and higher shrinkage. Higher level of shrinkage led to higher level of collapse throughout the fiber axis and that resulted in lower fiber diameter [29]. During carbonization shrinkage occurs basically because of the depolymerization of polymer chains that leads to CO, CO_2, tar removal [30, 31]. These results were also parallel with the carbon yield values (Table 2) that were as 28.51, 26.42, 26.17, 24.97 for S1, S2, S3 and S4, respectively. These results were also supporting the resistance values of the carbonized fibers. 4-probe resistance values of S1-S4 were as 6.98 E+1, 1.12 E+1, 1.01E+1, 9.95E+1 respectively. Higher process time led to more shrinkage and lower resistance, higher conductance. As previously given in the literature carbon yield was generally between 10-30 % for cellulose based raw materials [30]. Since in this study fibrous part of the hemp plant was used, values are relatively higher when compared with porous cellulose materials.

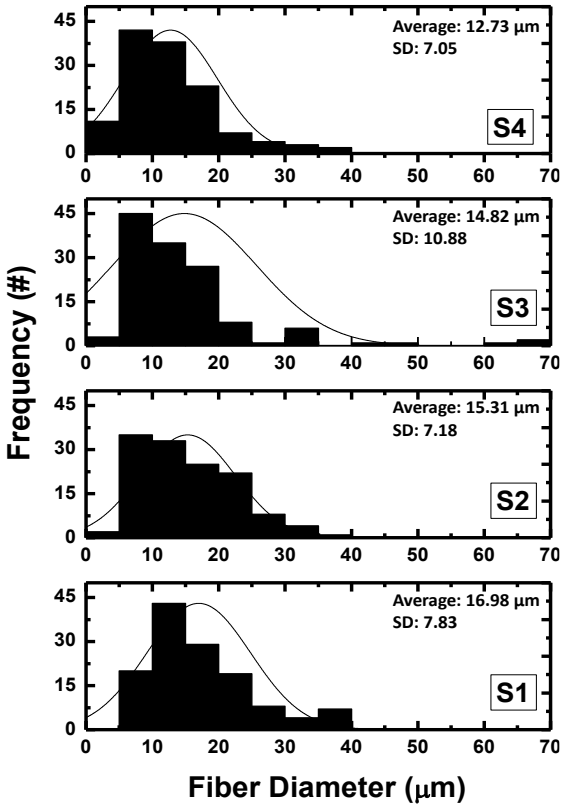

Figure 5. Diameter distributions of HDCFs, SD: Standard Deviation

In addition to fiber morphology analysis, composites were also analyzed by an optical microscope. Fig.6, Fig.7, Fig. 8 and Fig. 9 belong to the cross-sections of the samples from S1/SEBS, S2/SEBS, S3/SEBS, S4/SEBS composites, respectively and in each figure 0, 5, 35, 50 wt% HDCF containing samples were given.

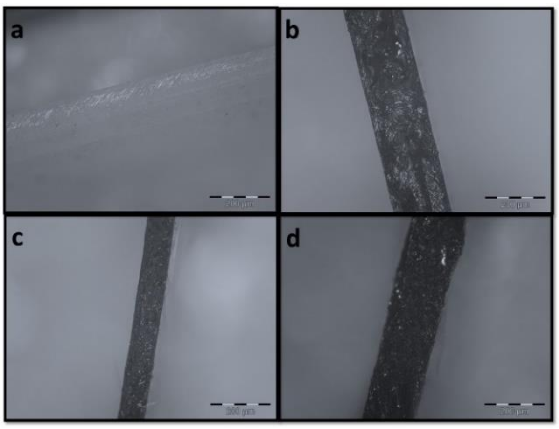

Figure 6. Optical microscope images of S1/SEBS composites at a) 0% b) 5% c) 35%, and d) 50 wt % filler concentration

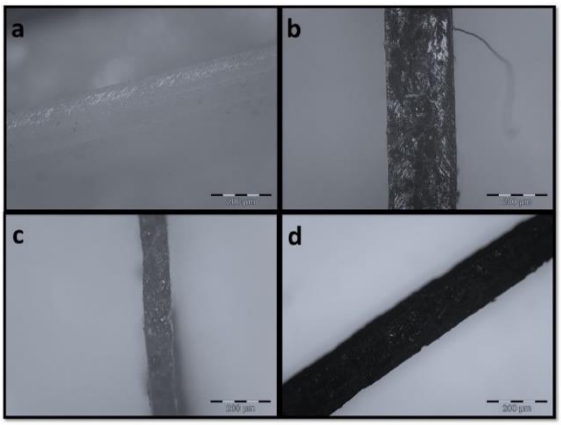

Figure 7. Optical microscope images of S2/SEBS composites at a) 0%, b) 5%, c) 35%, and d) 50 wt % filler concentration

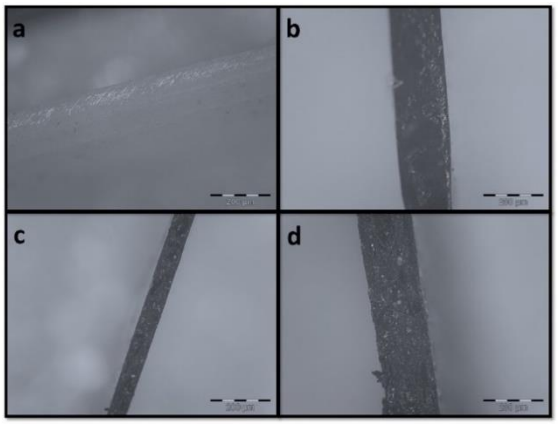

Figure 8. Optical microscope images of S3/SEBS composites at a) 0%, b) 5%, c) 35%, and d) 50 wt % filler concentration

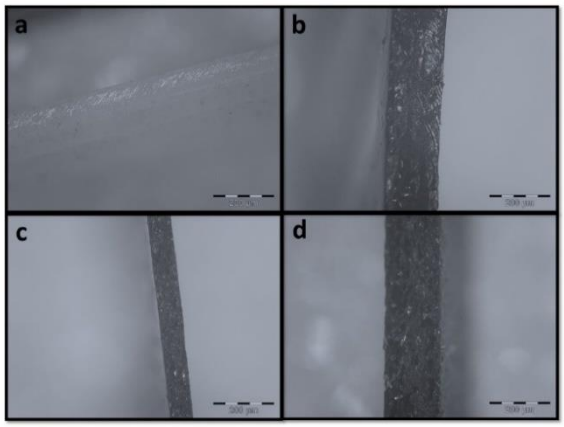

Figure 9. Optical microscope images of S4/SEBS composites at a) 0 wt%, b) 5%, c) 35%, and d) 50 wt % filler concentration

As obvious from the images at low filler concentration such as 5 wt%, fillers were homogeneously dispersed throughout the matrix. This was probably caused by good adhesion of SEBS on the porous surface of HDCFs. In addition to that samples were prepared in two steps. In the first step polymer solution wetted the fiber surface and in the second step samples were compression molded at 180 °C for 120 s. On the other hand, HDCFs tend to form agglomerates at higher filler concentrations. At 50 wt% matrix was not found sufficient enough to wet and cover the surface of the fillers. This behavior was also observed by naked eyes. The film formation of 40-50 wt % filler containing samples was difficult and surface of the samples were not as smooth as the others.

The electrical properties of the composites were also investigated. Percolation graphs of S1/SEBS, S2/SEBS, S3/SEBS, S4/SEBS composites can be seen from Fig. 10. Resistivity values are given in Table 3.

Table 3. Resistivity values of HDCFs/SEBS composites

Filler Ratio (wt %)	S1/SEBS (Ωm)	S2/SEBS (Ωm)	S3/SEBS (Ωm)	S4/SEBS (Ωm)
0	4.25E+17	4.25E+17	4.25E+17	4.25E+17
5	3.47E+16	1.31E+16	1.22E+16	4.52E+15
10	6.13E+13	2.50E+13	1.50E+13	2.07E+12
20	5.24E+09	6.93E+09	5.08E+09	2.93E+09
30	7.09E+08	5.46E+08	3.98E+08	2.45E+08
35	6.41E+08	3.25E+08	2.92E+08	9.18E+07
40	1.98E+08	9.74E+07	9.20E+07	3.28E+07
45	1.74E+08	9.21E+07	8.58E+07	3.47E+06
50	6.71E+07	4.01E+06	2.30E+06	2.21E+06

As can be seen from Fig. 10 and Table 3, all samples showed a percolation threshold around 5-20 wt%. As obvious from outcomes, carbonization time was found significant for the electrical properties of the carbon fibers and composites.

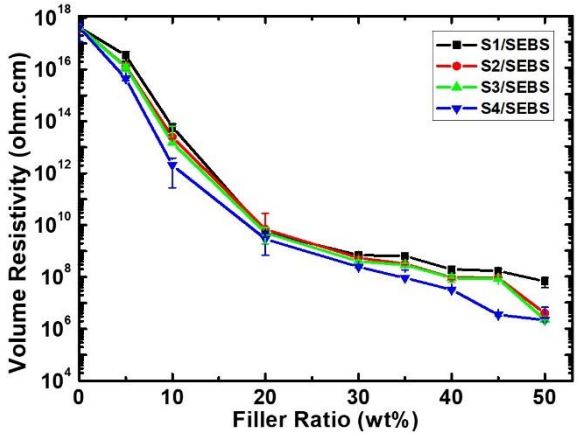

Figure 10. Percolation graph of HDCFs/SEBS composites as a function of filler type and filler ratio

In all samples, S4/SEBS composite set showed the lowest volume resistivity and S1/SEBS composite set showed the highest volume resistivity at all filler concentrations. S3 and S4/SEBS composite sets were found to be almost same. As can be understood from the results, longer carbonization time led to more conductive carbon fibers and that resulted in lower volume resistivity.

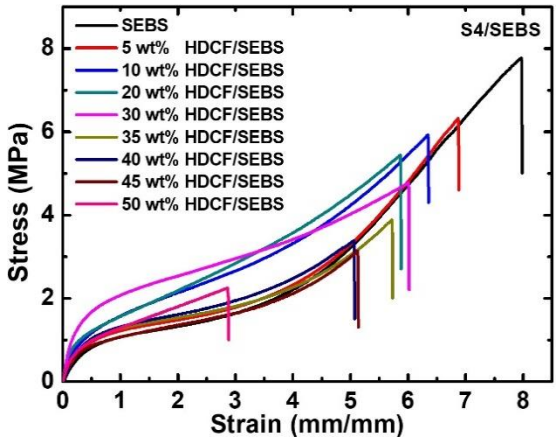

Figure 11. Stress strain graphs of S4/SEBS composites

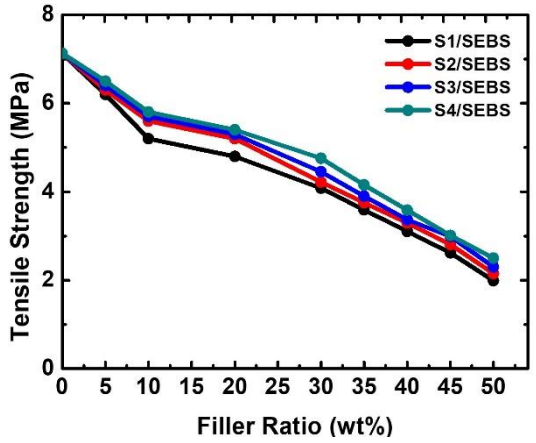

Figure 12. Tensile strength of composites as a function of filler ratio

Stress-strain graphs of S4/SEBS composites can be seen from Fig. 11. The summarized data for all samples can be seen from Fig. 12. As can be seen from the graph, all samples showed elastomeric behavior. By increasing the filler ratio, stress and strain values generally decreased. This was probably caused by the lower level of interaction between fillers and matrix. Generally speaking load bearing capacity of the composites decreased by the addition of fillers. Similar trend was observed for all sets. At higher level of filler loading the decrease was higher. This was probably caused by the aggregation of fillers and inadequate wetting of fillers by the matrix. This behavior is desired for electrical properties but not for mechanical properties. Even though, tensile strength values decreased, tensile strain values were between 300-700 % that is pretty good when compared with thermoplastic composites. They generally fail at lower strain values. With these mechanical properties all sets can be used for various applications including sensors, conductive coatings and electromagnetic shielding applications.

4. CONCLUSIONS

Conductive flexible polymer composites were prepared by using HDCFs and SEBS. In the first step, HDCFs were prepared at 4 different conditions. By using these fibers, 4-sets of composite films were prepared with the filler ratio between 5 – 50 wt %. Morphology of the HDCFs and composite films were analyzed by an optical microscope. At high filler ratios, filler agglomerations were observed regardless of the filler type. Electrical resistivity and percolation behavior of 4-sets were determined. The set that was fabricated by using S4 (900 °C, 120 min) fibers showed the lowest percolation concentration. Both tensile strength and strain values of composites showed decrease with the addition of the HDCFs. This study is important for showing the applicability of hemp as a natural and sustainable source for conductive carbon fibers. Future work will focus on applications of HDCFs filled conductive flexible composites in secondary batteries, piezoresistive sensors and electromagnetic shielding applications.

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