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Research Article

Sustainable, Alternative Conductive Fillers for Flexible Electronics: Investigation of Filler Size on Morphological and Electrical Properties of Styrene-[Ethylene-(Ethylene-Propylene)]-Styrene Block Copolymer (SEEPS) Composites

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Abstract

Sustainability is getting popular for many engineering applications from packaging to textiles, energy to electronics. Since renewable, environmental friendly sources lowers the negative impacts of the end product on ecology, sustainability studies generally start with the raw materials. The sustainability of electronic materials has gained importance because of limited amount of resources and increasing costs as well as environmental restrictions. In this study, pistachio shell waste was used to synthesize conductive fillers for the fabrication of sustainable flexible electronics. Pistachio shell waste was carbonized. After carbonization, two different grounding settings were used to obtain different filler sizes. In order to compare the effects of filler size on electrical and morphological properties of the composites, six different samples were prepared based on filler concentration with styrene-[ethylene-(ethylene-propylene)]-styrene block copolymer. Homogeneous filler distribution and good filler-matrix interface were observed for both composite sets. Filler size was found significant in terms of the electrical conductivity of the composites. For larger fillers, the percolation region was found to shift to lower concentration compared to smaller filler size.

Keywords: Carbonized pistachio shell, conductive filler, flexible electronics, styrene-[ethylene-(ethylene-propylene)]-styrene block copolymer (SEEPS)

Introduction

One of the recent trends in material science is to design and develop sustainable materials. Sustainability of any material is generally evaluated based on type of the raw material, process and their total environmental impacts. However, it should be accepted that complete sustainability is very hard to achieve. So, common approach is to make the materials more sustainable compared to previous state. To make any material more sustainable, starting point is generally raw materials. If renewable, sustainable and environmentally friendly raw materials can be used, the sustainability of the material will increase. From this perspective, if renewable sources are used for fabrication of alternative, sustainable conductive fillers, sustainability of the electronic materials increases. As known, agricultural wastes are good and sustainable source for many materials. Pistacia vera, known as pistachio, is an edible fruit with hard shell. Since it is a good source of nutrients and vitamins, it is generally consumed in food industry in the dried fruit form for ice-creams, candies, snacks, and desserts. Its agriculture is common in countries such as USA, Turkey, Iran, Syria, and China. According to statistics from The Food and Agriculture Organization ('FAO', 2020) world total production of pistachio in 2020 was around 1125000 MT and in Turkey around 300000 MT of pistachio was produced ('FAO', 2020). If we assume that 45-50 wt% of the fruit consists of shell, total shell waste in Turkey was around 135-150000 MT in 2020. If on and off years of pistachio agriculture are considered, waste amount can be given around 70-80000 MT annually.

Pistachio is a renewable agricultural product but evaluation of shell wastes should be considered in terms of its sustainability. Although shell waste is biodegradable, fabrication of pistachio shell based products with added value will offer alternative sustainable products in the industry. The content of pistachio shell can be given as; cellulose (42-47 wt%), hemicellulose (25-27 wt%), lignin (13-14 wt%) wax, ash and volatiles (6 wt%) (Yeganeh et al., 2006). Since a substantial amount of the shell consists of cellulose and hemicellulose, it can be directly used as the bio filler or it can be processed. Polymer composites filled with pistachio shell was studied in the literature and polyethylene (Ghazanfari et al., 2005), polypropylene (Salazar-Cruz et al., 2022), poly(methyl methacrylate) (Kadhim et al., 2020), polyester resin (Alsaadi et al., 2018), epoxy resin (Gairola et al., 2019), and natural rubber/styrene–butadiene rubber (Karaağaç, 2014) blends were used. In the other group, pistachio shell was carbonized in various forms of carbon for energy storage (Xu et al., 2014; Singh et al., 2022), water treatments (Küçük et al., 2019), heavy metal adsorption (Komnitsas

et al., 2015), and electrical conductivity (Çetin et al., 2022).

Conductivity is used to indicate the electron flow capacity of the material. When evaluated in the general concept, most of the industrial polymers are insulator and in order to increase their conductivity additives are required. Conductive filler can be metal, polymer or carbon-based material. They can be found in various forms such as plate (Sengupta et al., 2011; Li et al., 2019), fiber (Tibbetts et al., 2007; Al-Saleh et al., 2009; Li et al., 2019), particle (Huang, 2002; Li et al., 2019), tube (Bauhofer et al., 2009; Li et al., 2019) in micro or nanoscale. In all fillers, carbon-based conductive fillers have some advantages including lightness, inertness and no corrosion risk (Nurazzi et al., 2021). In addition to these, they can be fabricated in various forms and particle size. In order to tune the properties of the composites these advantages are of importance. As mentioned above pistachio shell can be carbonized. In most of the studies cited above, pistachio shell was carbonized, activated or functionalized in order to tune its properties and enhance the performance of in terms of energy storage, adsorption, and water treatments. Only in one study carbonized pistachio shell was used as a conductive filler for conductive polymer composites. In that study (Çetin et al., 2022), composites were prepared with poly[styrene-b-(ethylene-co-butylene)-b-styrene] (SEBS) and carbonized pistachio shell. In the study average filler size was around 12.7 µm. Percolation region was determined between 10-30 wt% and positive contribution of carbonized pistachio shell on electrical conductivity of SEBS matrix was shown.

Styrene-[ethylene-(ethylene-propylene)]-styrene block copolymer (SEEPS) is a styrenic thermoplastic elastomer with high flexibility, high resilience, good mechanical properties, low elastic modulus, soft touch and good resistance to atmospheric conditions. It is a triblock copolymer and can be processes as thermoplastics. SEEPS was generally used as a viscosity modifier (Wang et al., 2012), compatibilizer (Xanthos et al., 2002; Lu et al., 2017), or adhesive (Rapra Technology, 2003; Kato, 2018) in most of the studies. There is only one study that focused on the electrical properties and pressure sensing performance SEEPS composites. In that study graphite with the average particle size of 40 µm was used as the conductive filler. Percolation region was reported between 25-35 wt% and 40 wt% graphite filled SEEPS composite was reported to show negative piezoresistance (Karahan Toprakci et al., 2021).

In the present study, SEEPS was used as the elastomeric matrix with the aim of flexible conductive composite fabrication with carbonized pistachio shells for the first time in the literature. Based on our previous findings (Çetin et al., 2022), carbonization process was modified to enhance the conductivity of the filler and two different filler type was used for analyzing the size effect on electrical conductivity and polymer composite morphology.

Materials and Methods

Materials

SEEPS block copolymer with 30% styrene content was used (SEPTON 4033, Kuraray Co Ltd., Japan) as the flexible polymeric matrix. In order to dissolve polymer and disperse the fillers, chloroform was used as a solvent (Merck). All chemicals were used as received. In this study, pistachio shell wastes were carbonized and used as the conductive filler.

Carbonization and Characterization of Pistachio Shells

Carbonization process was modified based on our study (Çetin et al., 2022). Carbonization conditions can be seen from Fig. 1. Pistachio was bought from a local store. The shell was separated from the fruit and shell was washed with deionized water by using an ultrasonic system (Wisd, WUC-A03H) and dried in a convection oven at 80 ºC for 24 h. As given in Fig.2, dried shells were ground by a grain mill (Lavion, HC-100) for 120 s before the carbonization process.

The process was performed by using a tube furnace (OTF-1200X, MTI). In the first step, at 230 °C stabilization was performed under air atmosphere for 5 hours. From 230 to 900 °C, sample was heated under argon atmosphere with an increment rate of 2 °C min^{-1} and carbonization took place for 120 min. (Toprakci et al., 2021). After this process, insulating pistachio shell wastes turned into electrically conducting fillers as given in Fig. 2. The yield of carbonization process was determined as 26.6 %. In order to investigate the influence of filler size, sample was ground by a grain mill (Lavion, HC-100). Depending on the process time 2 different fillers were obtained. Carbonized pistachio shell was donated as CPS. CPS-1 was ground for 240 sec, CPS-2 was ground for 480 sec. Digital images of the precursor and end products can be seen from Fig. 2. Optical microscope images of the fillers were captured (AmScope 40X-2500X LED Digital Binocular Compound Microscope) and size distribution of the samples were determined by using Image J software. Average particle size of CPS-1 and CPS-2 were determined as 64.2 and 13.7 µm respectively. The optical microscope images and size distribution histograms of CPS-1 and CPS-2 can be seen from Fig. 3a and b, respectively.

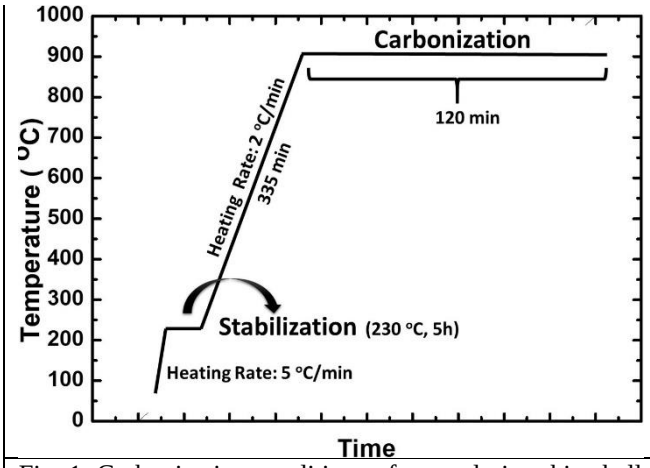

Fig. 1. Carbonization conditions of ground pistachio shell waste

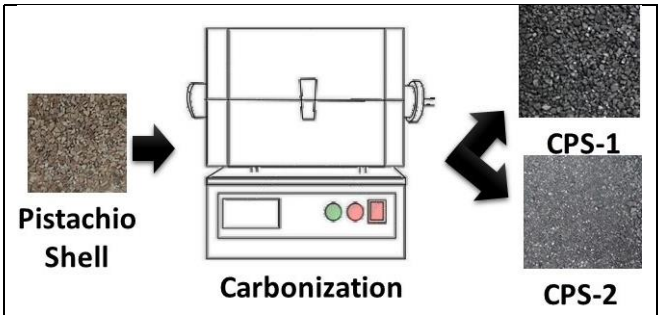

Fig. 2. Pistachio shell waste and conductive filler

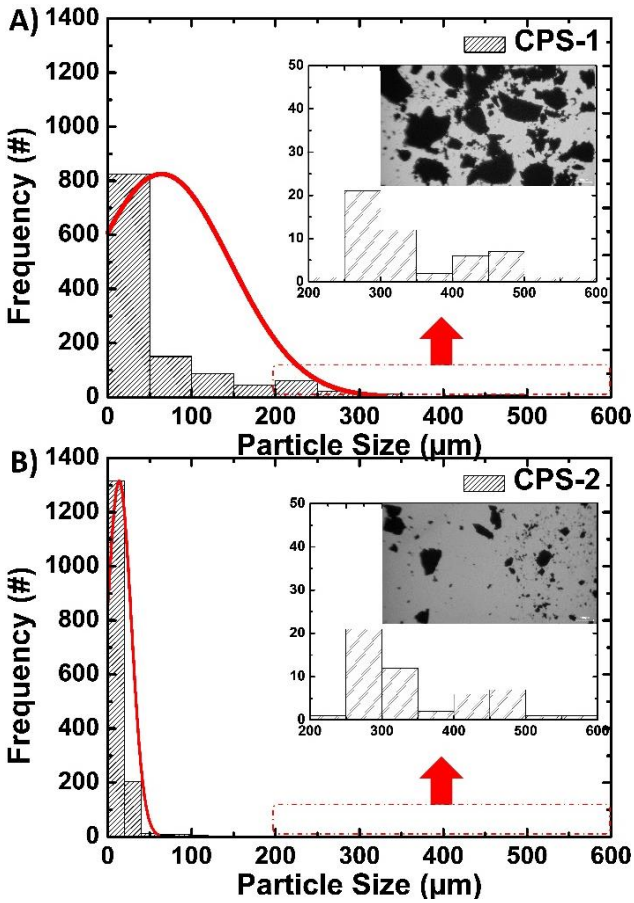

Fig. 3. Optical microscope image and size distribution histogram of a) CPS-1 b) CPS-2 (scale bar: 100 µm)

Table 1. Sample contents, conductivity and standard deviation values

Filler Type	CPS (wt%)	SEEPS (wt%)	Conductivity (S cm^{-1})	Standard Deviation
-	-	100	2.04E-17	1.80E-17
CPS-1	5	95	2.95E-07	1.26E-08
CPS-1	10	90	3.31E-07	9.45E-08
CPS-1	20	80	4.57E-07	1.85E-07
CPS-1	30	70	1.02E-06	5.04E-08
CPS-1	40	60	1.48E-06	4.86E-08
CPS-2	5	95	3.04E-14	6.82E-15
CPS-2	10	90	4.37E-07	3.54E-07
CPS-2	20	80	9.35E-07	2.91E-07
CPS-2	30	70	1.25E-06	2.83E-06
CPS-2	40	60	1.33E-06	1.24E-08

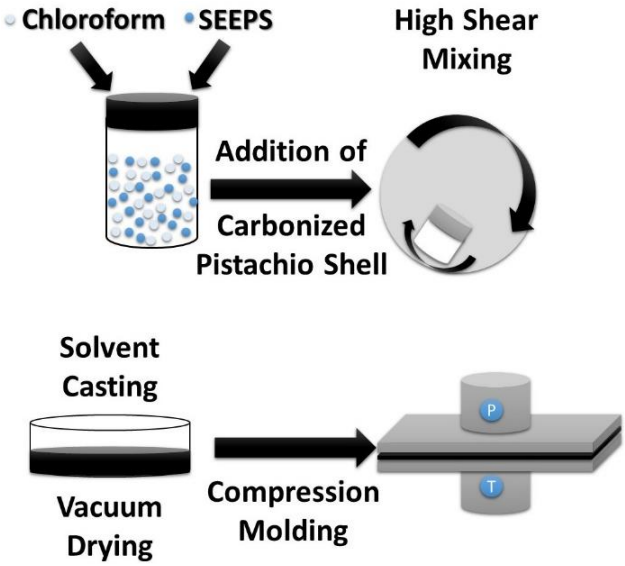

Fig. 4. Schematic representation of the composite production

Preparation of the Composites

The preparation and content of composites can be seen from Fig. 4 and Table 1, respectively. SEEPS and chloroform were mixed with the ratio of 1:4 for 24 hours. Polymer solutions were mixed with CPS-1 and CPS-2 with the ratio of 5, 10, 20, 30, 40 wt% by using a high shear mixer (Kurabo, Mazerustar-KK250) for 90 s. Solvent casted films were dried in a vacuum oven (Wisd, WOV-20). Following that compression molding between Teflon coated plates was performed at 205 °C under 3 MPa for 180 s.

Morphological Analysis

The cross-sectional morphology of the composites was analyzed by a field emission scanning electron microscope (FESEM, JEOL JSM-6400) at 30 kV at 100 x and 500x. Before the analysis samples were cut by a blade and sputter coated Au-Pd alloy. The coating thickness was around 3-6 nm.

Conductivity

The conductivity of the composite films was determined according to on ASTM D257 standard by a resistivity chamber (8009 Keithley) and an electrometer (6517B Keithley). 5 measurements were performed and average values were calculated.

Results

Morphological Analysis

The cross-sectional morphology of CPS1/SEEPS and CPS2/SEEPS can be seen from Fig. 5 and 6, respectively. Composite morphology was analyzed at different magnifications as 100x (scale bar: 1 mm) and 500x (scale bar: 200 µm) respectively. At 100x, filler distribution and orientation can be noticed better esp. for larger fillers. In order to analyze filler-matrix interface, magnified images taken at 500x is useful. Samples with CPS/SEEPS ratio of 5/95, 20/80, and 40/60 were analyzed. In the SEM analysis polymeric matrix behavior, filler morphology, dispersion of fillers, filler orientation, filler-polymer interaction, and conductive network formation were investigated. SEEPS matrix is the dark grey continuous phase in the SEM images. As can be seen Fig. 5 and 6, SEEPS matrix was completely molten under compression molding conditions and homogeneous film thickness was obtained for all samples regardless of the filler concentration or filler type. Film thickness was around 400-700 µm for the composites. CPS-1 and CPS-2 can be noticed from their light grey and/or shinny hue. Parallel with particle size distribution histograms and optical microscope images CPS1 showed larger particle size compared to CPS2. Since CPS is a hard carbon, high shear mixing seemed not to effect the particle size.

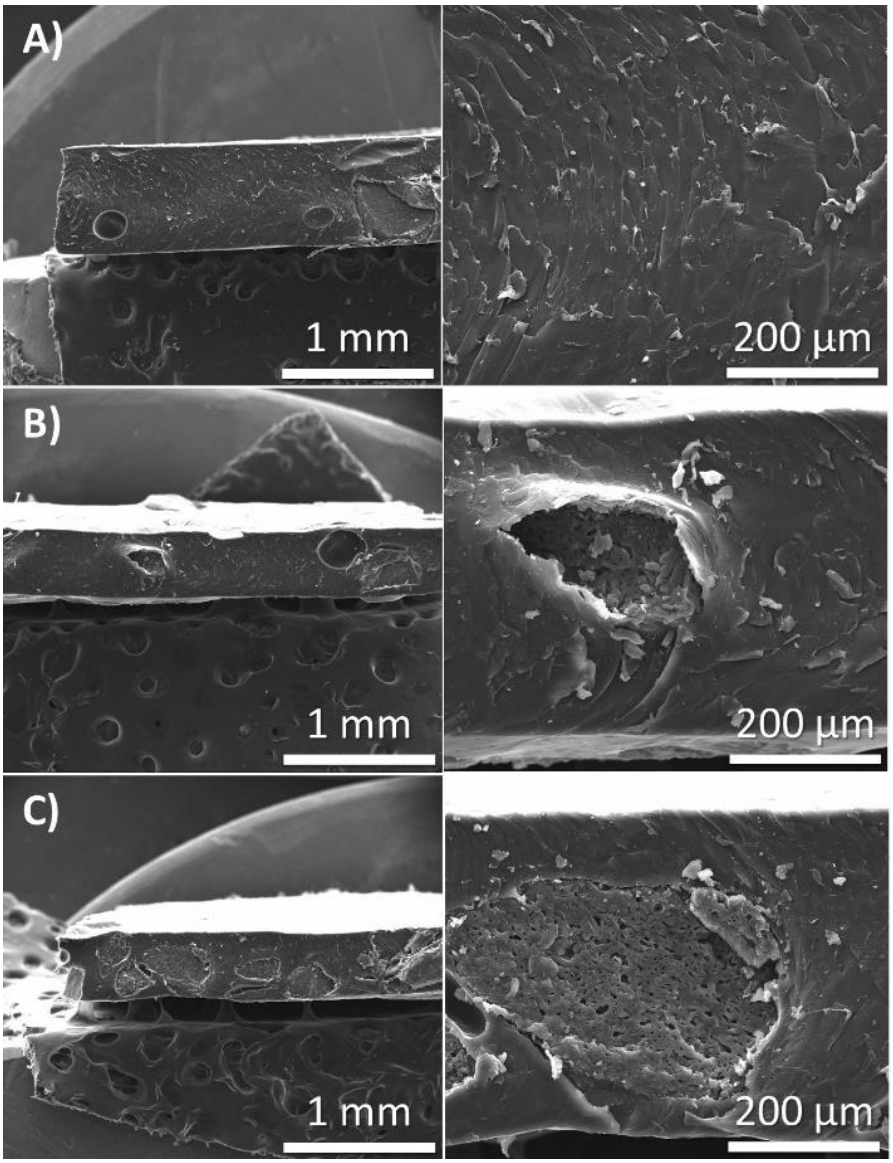

Fig. 5. FESEM images of a) 5/95, b)20/80, c) 40/60 CPS1/SEEPS composites at 100x and 500x

The morphological details of the CPS are very obvious from Fig.5c. The interconnected channel structure leads to lower filler density and light composites. By increasing the filler concentration from 5 to 20 and 20 to 40 wt%, density of filler in the cross-section increased. As a positive outcome of that, decreased filler-filler distance led to increase in conductivity. Although CPS-1 easily noticed in SEEPS matrix even at low magnification, CPS-2 was more obvious at higher magnification and filler size seemed not to be affected

by high shear mixing as CPS-2. Since CPS-2 has lower size, filler-matrix interaction surface was higher and at high concentrations some agglomerates were observed. For both CPSs, the filler-matrix interface was good because of effective high shear mixing and film formation under compressive force. Although composite production was a solution based process, since chloroform evaporates quickly and vacuum drying was carried out, no pores or foam-like morphology was observed. Since CPS-2 had lower filler size, and fillers were generally in the form of particles, no dominant filler orientation was observed. On the other hand for CPS-1, plate-like and particle-like fillers were observed and depending on the particle geometry, orientation of the fillers changed. As shown in Fig. 8a, in the case of particle-like CPS-2, aspect ratio of the fillers were around 1 and orientation could not be observed. On the other hand, plate-like fillers tend to orientate along the x direction that was probably stemmed from compression molding process. Based on these observations it can be concluded that not only size but also geometry of the fillers is of importance for the morphology and other properties of the composites.

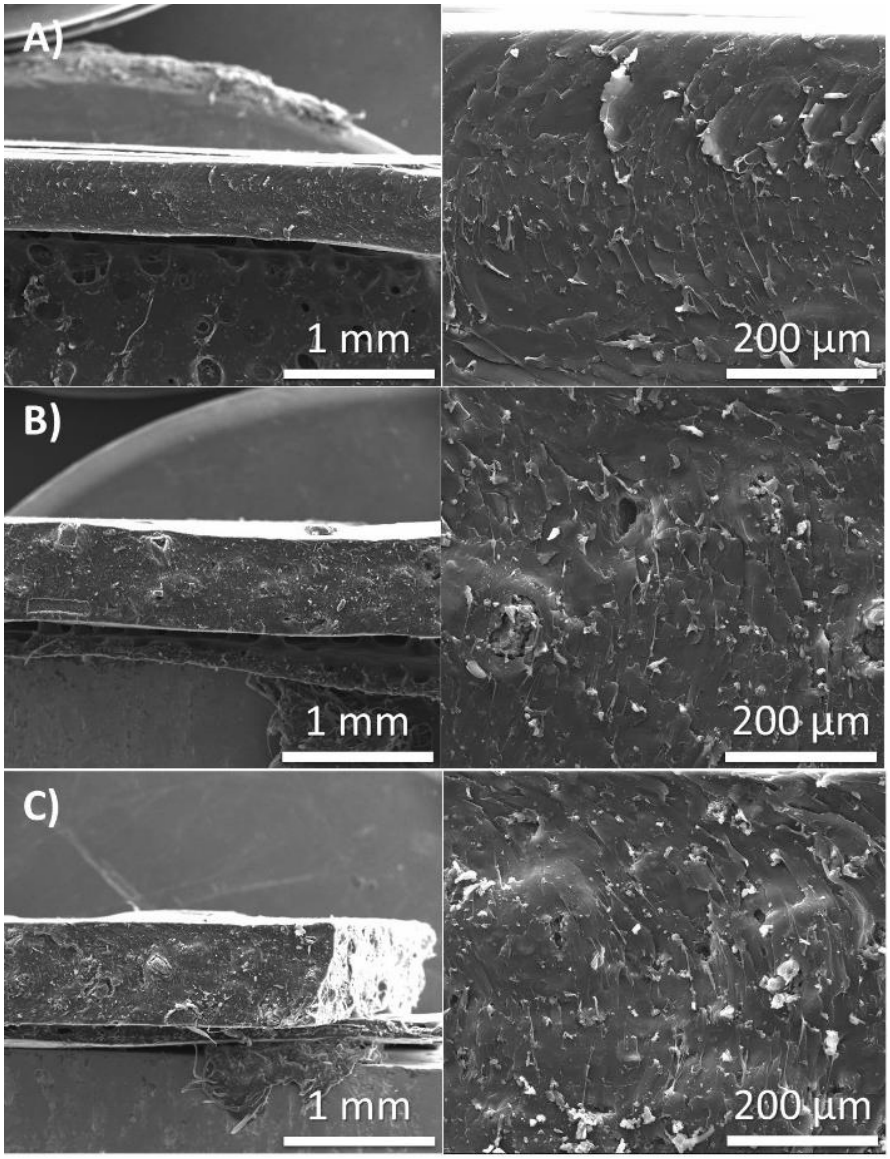

Fig. 6. FESEM images of a) 5/95, b)20/80, c) 40/60 CPS2/SEEPS composites at 100x and 500x

Conductivity

To investigate the electrical properties of the composites and observe the difference between two sets, resistivity of the samples was measured and conductivity was calculated from the reciprocal of resistivity. Electrical conductivity of CPS-1/SEEPS and CPS-2/SEEPS composites as a function of filler ratio between 0-40 wt% can be seen from Fig. 7 and Table 1. Conductivity of SEEPS was 2E-17 S cm^{-1}. The conductivity values of 5/95, 10/90, 20/80, 30/70 and 40/60 CPS-1/SEEPS composites were 2.95E-7, 3.31E-7, 4.57E-7, 1.02E-6, 1.48E-6 S cm^{-1}, respectively. The conductivity values of 5/95, 10/90, 20/80, 30/70 and 40/60 CPS-2/SEEPS composites were 3.04E-14, 4.37E-7, 9.35E-7, 1.25E-6, 1.33E-6 S cm^{-1}, respectively. As obvious from outcomes CPS-1 and CPS-2 filled composites showed percolation behavior. Conductivity of the samples increased with the addition of CPS fillers. However, there were some differences between two sets. While 5/95 CPS-1/SEPS composite had conductivity around 2.95E-7 S cm^{-1}, 5/95 CPS-2/SEPS had the value around 3.04E-14 Scm^{-1}.

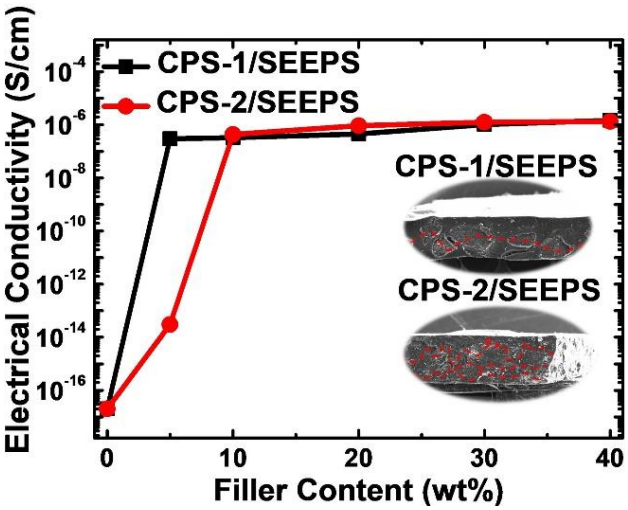

Fig. 7. Electrical conductivity vs filler concentration for CPS1/SEEPS and CPS2/SEEPS composites (Inset images: 40 wt% CPS-1 and CPS-2 filled composites)

Although conductivity of both composites increased compared to SEEPS, CPS-1 incorporation led to higher increase and conductivity values at 5 wt% can be given as CPS-1/SEEPS>CPS-2/SEEPS>SEEPS. At 10 wt%, CPS-1 filled composite did not show significant difference that was an indication of saturation behavior. On the other hand, 10 wt% the conductivity of CPS-2 filled composite increased around seven orders of magnitude. CPS-2 filled composite showed no change in conductivity at 20 wt% filling ratio that is an indication of saturation region.

After reaching the saturation concentration, conductivity values of composites did not change for both sets. From the outcomes it can be concluded that, CPS-2/SEEPS composite set showed percolation region between 5-10 wt% and CPS-1/SEEPS composite showed percolation region below 5 wt%. This was attributed to the filler size. For smaller fillers, filler-matrix interaction was higher as shown in SEM images. In the case of good mixing and homogeneous filler distribution, the wetting of fillers took place. As a consequence of this, dispersed fillers were separated by the polymeric matrix and electron flow from one filler to another was limited by that polymeric layer. Polymer matrix led to increase in contact resistance between fillers and that was reflected as a lower conductivity value for 5/95 CPS-2/SEEPS. On the other hand, for larger particles total interaction surface with polymer was relatively lower as shown in SEM images in Fig. 8. Since particles are larger, electron flow in the filler is easier. In addition to that it was shown in Fig. 3 that CPS-1 had smaller sized particles and those particles functioned as linkages between larger particles and can be given as one of the reasons for higher conductivity.

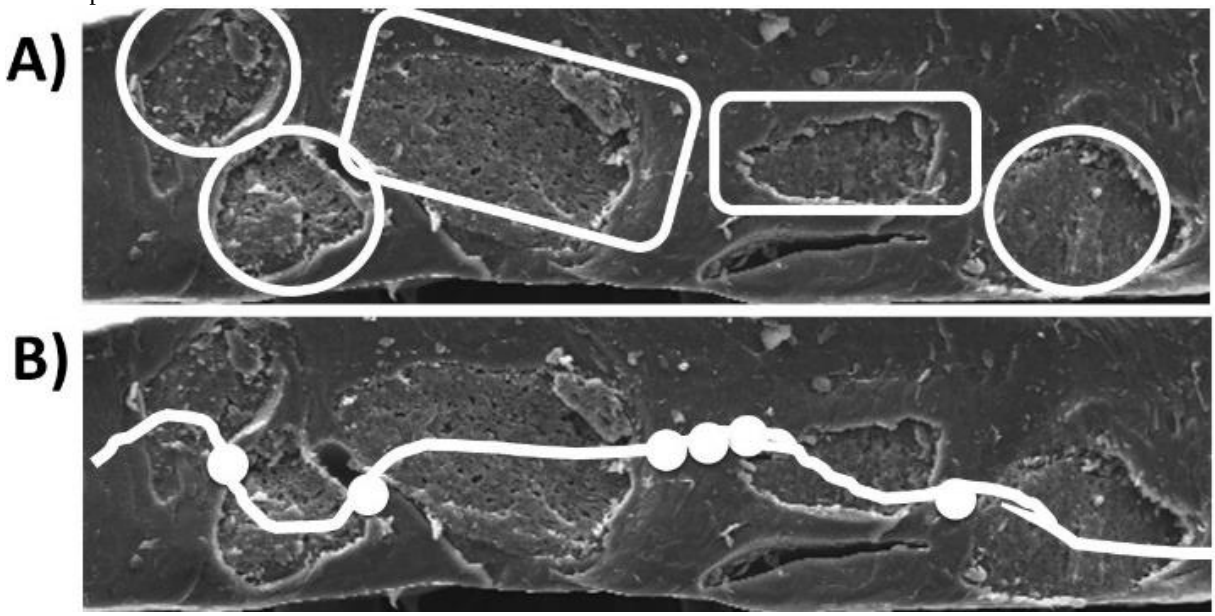

Fig. 8. a) Orientation of CPS-1 fillers in SEEPS matrix b) conductive linkages formed by smaller particles in SEEPS matrix (40/60 CPS-1/PEEK composite)

Discussion and Conclusions

In this study, electrically conductive flexible SEEPS composites filled with carbonized pistachio shell was fabricated for first time in the literature. The effects of filler particle size on morphological and electrical properties have been studied. In order to determine the percolation concentration filler concentration was varied as 5, 10, 20, 30, 40 wt%. CPS-1 showed good dispersion and filler-matrix interface at all concentrations. However, filler orientation was observed to be affected by the filler geometry. CPS-2 showed good dispersion and filler-matrix interface, however some agglomerations were observed at 40 wt% filling ratio. The percolation concentration of CPS-2 based composite set was found to be between 5-10 wt% and CPS-1 based set showed percolation concentration lower than 5 wt%. The lower percolation concentration and higher electrical conductivity can be attributed to the filler size and conductive linkages between large particles. The sustainable engineering approach represented in this study can be expanded to polymer based sustainable sensors and wearable electronics.

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