IMPACT OF CARBONIZATION TEMPERATURE ON ACTIVATED CARBON WEB FOR EMI SHIELDING AND OHMIC HEATING

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Abstract: *In this work porous and electrically conductive activated carbon webs were produced by using acrylic fibrous waste followed by carding and needle punching. The physical activation of acrylic webs was performed by using high temperature muffle furnace at different temperatures (800, 1000 and 1200°C). These activated carbon webs were characterized by using energy dispersive X-rays, scanning electron microscopy and X-ray diffraction analysis. Further the utility of prepared activated carbon webs was checked for electromagnetic shielding ability. Activated carbon webs prepared at 800, 1000 and 1200°C showed the electromagnetic shielding effectiveness of 3.34, 26.06 and 28.29 dB at a frequency of 2.45 GHz. This behavior was attributed because of increased multiple internal reflections and stronger absorption of electromagnetic radiations in activated carbon webs. At the end these activated webs were checked for ohmic heating application. The web prepared at 800°C showed higher generation of heat (185°C) after 60 seconds at 1.5 volt.*

Keywords: *Activated carbon, acrylic fibrous wastes, electromagnetic shielding, textile recycling.*

1 INTRODUCTION

Nowadays electrically conductive textiles are gaining interest because of their possible applications in different fields like military, electromagnetic shielding as well as in sensors and actuators. The electrically conducting textiles can be obtained through different methods like metallization, electroless deposition, chemical coating, insertion of metallic yarns and deposition of thin layers having electrically conductive fillers such as carbon black particles [1]. For electromagnetic shielding (EM) application the surface electrical resistivity should be lower than 10^2 Ω/cm^2, however most of the synthetic fibres used in textiles are insulating materials having resistivity around 10^{14}-10^{15} Ω/cm^2 [2]. Therefore, in this age of telecommunication the widespread usage of electrical devices attracts attention towards electromagnetic interference shielding. The radiant electromagnetic signals emitting from electrical instruments not only can cause interference with the proper working of other instruments [3, 4]. In this context the idea of using conductive fabric to counter electromagnetic shielding is not a preferred choice because electromagnetic shielding inference is controlled by absorption, reflection and multiple internal reflections of EM radiations [5, 6]. However like metals, conductive fabrics are also accompanied with the lack of absorption of EM shielding and reflection is the only mechanism of EM shielding [7]. But for eco-friendly advancement in the field of EM shielding it is preferred to use light weight shielding materials having more absorption and weak secondary reflection. This is possible by a material having porous structure, high bulk and above all higher values of electrical conductivity. Although the much work was done for the development of carbon based porous structures having effective EM shielding ability [8, 9], however, the construction of light weight carbon structures with good EM shielding application is still a problem. In this paper the simple and affordable solution for the development of electrically conductive and porous carbon structures from acrylic fibrous waste is presented and basic properties together with functionality (electromagnetic shielding, ohmic heating) are evaluated.

2 MATERIALS AND METHODS

2.1 Materials

The acrylic fibrous waste used in this study was provided by Grund Industries, Czech Republic. The acrylic fibres were in the form of bath mats. These fibres have acrylonitrile copolymer 85-89%. The physical characteristics of acrylic fibres are shown in Table1.

Table 1 Physical properties of acrylic fibres

Fineness [tex]	117
Tenacity [cN/tex]	23.8
Elongation [%]	45
Wet shrinkage [%]	2.5

2.2 Preparation of activated carbon nonwoven web

The acrylic fibers were separated from bath mats by using mechanical cutting method. The fibers were further opened on laboratory roller card (Befama, Poland) and converted into compact structure of non-woven web by using needle punching machine. Carding is a mechanical process which cleans, disentangles and intermixes fibers to produce a continuous web for further processing and is achieved by passing fibers through differentially moving surfaces covered with card clothing. After carding, the web was transferred to needle punching machine to form a compact structure of non-woven web. The speed of feeding the carded web to needle punching machine was at the rate of 0.4 m/sec. The frequency of strokes was maintained at 200 (strokes per minute) with the depth of needle penetration is 5 mm produced the web having thickness of 11.6 mm and density 2.78 g/cm^3. The acrylic fibrous web was then cut into 30 cm (length) and 20 cm (width) and was stabilized by heating at a rate of 50°C.hr^{-1} under the application of heat. Later these stabilized webs were carbonized and physically activated in single stage at 800, 1000 and 1200°C at a heating rate of 300°C.hr^{-1}.

2.3 Characterization of activated carbon web

The physical properties of acrylic web and activated carbon web were characterized in terms of flexibility, shrinkage, yield and dusting tendency. The shrinkage was evaluated from change in length of web before and after carbonization. Similarly like shrinkage, yield of activated carbon before carbonization and after carbonization at different temperatures was calculated by using the eq. 1:

$$Yield = \frac{\text{Final weight of activated carbon web}}{\text{initial weight of acrylic web}} \times 100 \qquad (1)$$

The taber fabric stiffness tester (model 112) was used for measuring flexibility or stiffness by employing the principle of cantilever bending of the web under its own weight as per ASTM D 1388 standard. The dusting tendency was evaluated from amount of generated dust particles after rubbing the surface of web on Taber wear and abrasion tester as per ASTM D 3884 standard.

<u>*X-ray diffraction (XRD) analysis*</u>

It was carried out on a PANalytical X_0 Pert PRO MPD diffraction system. The development of crystalline and amorphous regions in prepared activated carbon web was investigated with respect to change in carbonization temperature. X-ray diffraction (XRD) is a technique used for the identification of crystalline material and analysis of unit cell dimensions. Degree of crystallinity can be calculated by using equation 2:

$$I_c = 1 - \frac{I_1}{I_2} \qquad (2)$$

where I_1 is intensity at minimum peak and I_2 is intensity at maximum peak [10].

<u>*Energy dispersive x-ray (EDX) analysis*</u>

EDX analysis was performed on Oxford Instruments, LZ 5 EDX detector, UK, to know the change in relative proportion of different elements with respect to change in carbonization temperature. EDX analysis is an analytical technique used for the chemical characterization or elemental analysis of a sample.

<u>*Scanning electron microscopy (SEM)*</u>

The field emission scanning electron microscope Sigma, Zeiss, Germany, was employed to investigate the morphology of prepared activated carbon web of 800, 1000 and 1200°C carbonization temperature. As the carbon was electrically conductive so no need to be metalized before conducting the test. The micrographs were taken at 2 kV accelerated voltage and 1000 magnification.

<u>*Electrical resistivity of activated carbon webs*</u>

The surface resistivity of AC webs prepared at 800, 1000 and 1200°C was measured at relative humidity 40% and at temperature 22°C according to ASTM D 257-14 with the help of Prostat PRF-911 concentric ring set. The specific voltage of 1 V using direct current was applied across opposite ends of activated carbon web and resultant current flowing across the sample was measured after 15±1 s. Surface electrical resistivity of activated carbon webs was measured by concentric electrode and parallel electrode method.

<u>*Electromagnetic shielding effectiveness of activated carbon web*</u>

The electromagnetic shielding effectiveness of activated carbon webs was determined by using waveguide method [11]. By using this method, the shielding effectiveness was calculated in high frequency range (i.e. at 2.45 GHz). This device comprises on a hollow cylinder in which receiving antenna was placed, however the sample was placed at the entrance of waveguide. From equation (3), the electromagnetic shielding effectiveness *SE* [dB] was calculated:

$$SE = 10 \log \frac{P_t}{P_i} \qquad (3)$$

where P_t and P_i is power density [W/m^2] measured in presence of sample (transmitted), and without the sample (incident) respectively.

<u>*Ohmic heating of activated carbon webs*</u>

For calculating the evolution of heat by activated carbon webs prepared at different temperatures, 0.5, 1.0 and 1.5 V, DC electromotive forces by DC voltage supplier NZ-2229.2 from Statron, Czech Republic, was applied via stainless steel clamp type electrodes. The rise in temperature was determined by the help of infrared thermal camera by Fluke.

3 RESULTS AND DISCUSSION

3.1 *Effect of temperature on physical characteristics of AC web*

When the temperature was increased from 800 to 1000°C and finally to 1200°C, the reaction of forming carbon with oxygen increased which resulted in decreasing yield of carbon and consequently shrinkage was increased and more rigid structure of activated carbon was achieved. Because of this reason, activated carbon web at high temperature showed poor flexibility and dusting behavior as can be seen from Table 2. However as far as thickness is concerned it decreased from 11.6 mm (thickness of acrylic web) to 6.87 mm, 6.11 mm and 5.23 mm when the carbonization temperature was increased from 800 to 1000°C and finally to 1200°C.

Table 2 Effect of carbonization temperature on physical properties of activated carbon webs

Temperature [°C]	Yield [%]	Shrinkage	Flexibility	Dusting
800	61.27	Good	Good	Good
1000	57.12	Good	Average	Average
1200	45.11	Average	Poor	Poor

3.2 *Energy dispersive x-ray (EDX) analysis*

The EDX analysis helped to determine the relative proportion of different elements in activated carbon web prepared at different temperatures. From Table 3, the increase in carbon content and reduction in oxygen content was found with increase in carbonization temperature from 800 to 1200°C. The activated carbon web produced at 1200°C exhibited 92.49% carbon content and 6.61% oxygen content. This behavior was attributed to removal of hydrogen, sulphur, nitrogen and other elements due to decomposition at higher temperature [8].

Table 3 Effect of carbonization temperature on elemental composition of activated carbon web

Element	App conc.	Intensity	Weight [%]	Atomic [%]
800°C				
C K	0.26	2.12	0.13	91.76
O K	0.01	0.761	0.01	8.24
1000°C				
C K	0.37	2.12	0.18	91.87
O K	0.02	0.760	0.02	8.13
1200°C				
C K	0.18	2.10	0.09	92.49
O K	0.01	0.744	0.01	6.61
Ca K	0.00	0.902	0.00	0.90

3.3 *X-ray diffraction (XRD) analysis*

In order to know the development of crystallinity with increase in carbonization temperature, the XRD analysis was carried out. Figure 1 shows the XRD pattern of different activated carbon samples prepared at 800, 1000 and 1200°C temperature. The degree of crystallinity is increased from 82.21% to 86.7 and then finally reached to 92.41% by increasing temperature from 800 to 1000°C and then finally to 1200°C. The higher degree of crystallinity at higher temperature indicates more parallel orientation of chains.

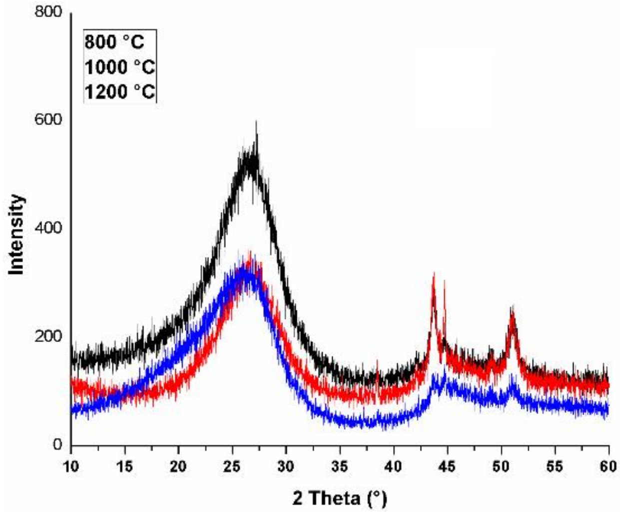

Figure 1 Effect of carbonization temperature on crystallinity of activated carbon web

3.4 *SEM morphology*

The surface morphology of acrylic web before and after carbonization was studied from SEM images. Figure 2(a-d) show the SEM images of acrylic fibrous web and activated carbon web produced at temperature of 800, 1000 and 1200°C respectively.

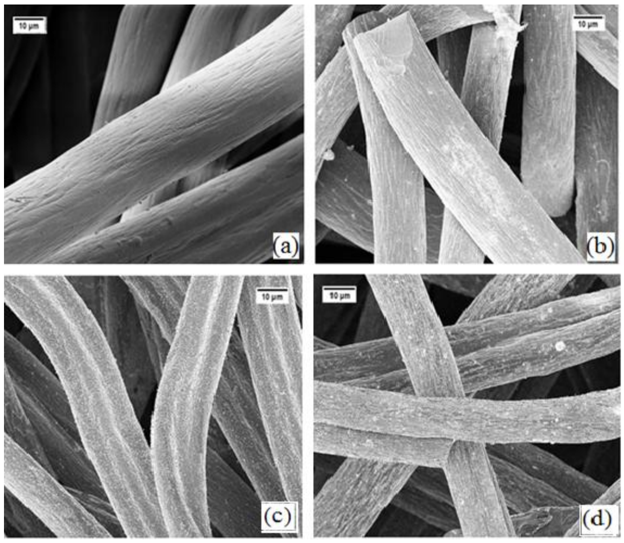

Figure 1 SEM images of (a) acrylic fibrous web (b) 800°C activated carbon web (c) 1000°C activated carbon web and (d) 1200°C activated carbon web

The activated carbon web showed noticeable rough surface as compared to acrylic fibrous web. The surface roughness was found to increase with increase in carbonization temperature, which indicated the development of more porous structure after physical activation of acrylic fibrous wastes. Further at high temperature carbonization due to more elimination of gases from acrylic fibers the diameters of fibers keep on decreasing as can be seen from Figure 2(a-d).

3.5 Results of electrical resistivity of activated carbon webs

The knowledge of electrical resistivity is very important for the selection of material to be used for electromagnetic shielding applications. The results of surface resistivity of activated carbon webs are shown in Table 4. The electrical resistivity was found to decrease with increase in carbonization temperature. The lower resistivity in activated carbon web at 1200°C was attributed due to more graphitization, which was confirmed from presence of sharp diffraction peak observed in XRD spectra and higher degree of crystallinity. As the temperature during carbonization was increased, the degree of crystallinity also increased. The degree of crystallinity was found to be 82.21%, 86.7% and 92.1% at carbonization temperature 800, 1000 and 1200°C. Further at higher temperature the content of carbon also increased which is also another cause for decrease of electrical resistivity by increasing carbonization temperature as can be seen from Table 4.

Table 41 Surface resistivity of activated carbon webs

800°C	1000°C	1200°C
Parallel electrode (surface resistivity Ω)		
3.33×10^2	1.28	0.27×10^{-1}
Concentric electrode (surface resistivity Ω)		
10	2.85	0.09

3.6 EMI shielding effectiveness by wave guide method

Figure 3 shows the electromagnetic shielding effectiveness of activated carbon web measured at 2.45 GHz frequency. The shielding effectiveness was found to increase with increasing carbonization temperature. The electromagnetic shielding effectiveness of 28.29 dB, 26.06 dB and 3.34 dB was exhibited by activated carbon web produced at 1200, 1000 and 800°C, respectively. At very low carbonization temperature, the shielding effectiveness remained similar to that of non-carbonized polyacrylonitrile substrate (i.e. zero). Then, the shielding effectiveness was found to increase dramatically over a very narrow range of carbonization temperature, which was connected to the amount of carbon/graphite phase present in the structure. This point is called the percolation threshold [12], which showed minimum carbonization temperature required for maximum increase in conductivity for higher shielding effectiveness. In present study, the percolation threshold was found between the range of 800 and 900°C carbonization temperature. The maximum shielding effectiveness in this range was attributed not only due to absorbance but also due to increased multiple internal reflections due to higher electrical conductivity, higher porosity and higher surface area. The dramatic increase of shielding ability could not be expected with further increase of carbonization temperature (T>1100°C). Therefore, the usage of 1000°C carbonization temperature was considered optimal with regard to its relatively high electromagnetic shielding ability and satisfactory mechanical properties [6].

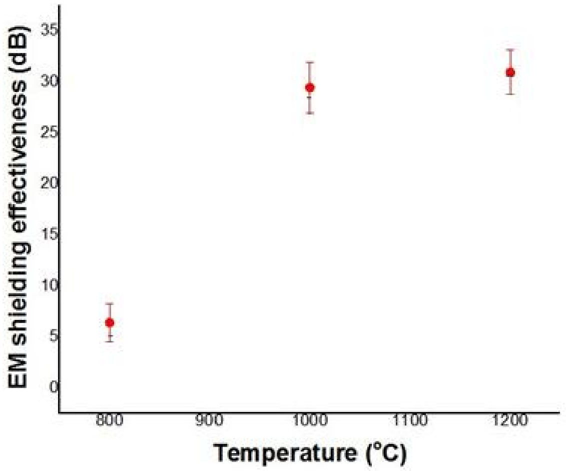

Figure 3 Effect of carbonization temperature on EM shielding effectiveness at 2.45 GHz

3.7 Ohmic heating of activated carbon webs

Joule heating also known as ohmic heating or resistive heating is the process by which the passage of electric current through a conductor produces heat. Joule heating or ohmic heating of activated carbon webs prepared at 800, 1000 and 1200°C were checked at 1.5 voltage.

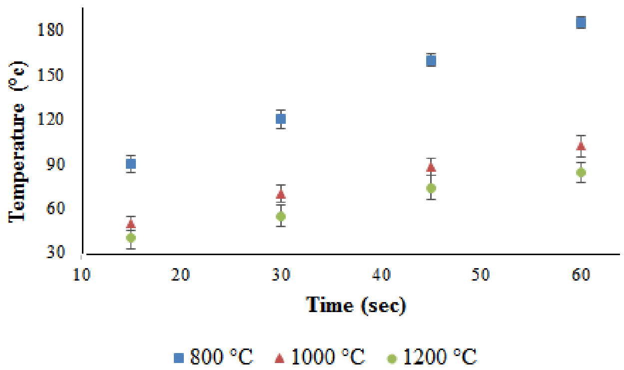

Figure 4 Rise in temperature as a function of time

After every 15 seconds of exposure to DC supply to the webs the generation of heat was observed with the help of thermal camera. From Figure 4 the rise in heat generation with increase in time can be seen. A sharp increase in temperature in all specimens was observed in the initial 30 seconds with the applied voltage than followed by a gradual increase in temperature as shown in Figure 4. It is clear from the figure that the web prepared at 800°C showed maximum increase in temperature due to less parallel orientation of chains hence more resistance offered for the flow of electrons as can be seen from Figure 5.

Since the rate of thermal energy generated within a resistive material is directly proportional to resistance. As the activated carbon web prepared at 800°C has more resistance as compared to AC web at 1000°C and 1200°C, because of more resistance offered by the flow of electrons it shows more rise of temperature. Hence when ohmic heating is measured it shows more rise of temperature in AC web prepared at low temperature after short interval of time at localized point (where electrodes were connected) shown by thermal camera. However, if both sides of AC are connected completely instead of electrode at one point it can be a good idea for measuring ohmic heating over long interval of time.

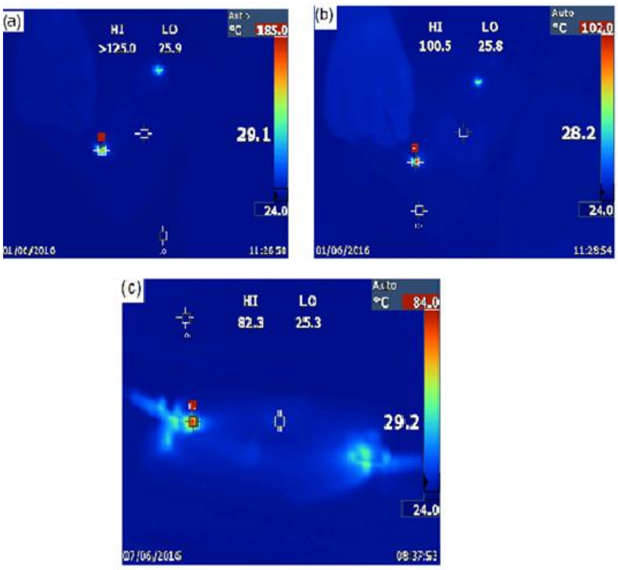

Figure 5 Ohmic heating of activated carbon webs

4 CONCLUSION

The present study aims at the development of a porous and electrically conductive activated carbon structure based electromagnetic shielding materials from acrylic wastes. This simple and new approach use here can introduce reflection and absorption properties of electromagnetic radiations into the shielding materials. This has been achieved by physical activation of needle punched nonwoven web of acrylic fibers. The carbonization is performed under the layer of charcoal at 800, 1000 and 1200°C with the heating rate of 300°C.h^{-1} and without any holding time. Further, the influence of carbonization temperature on physical and morphological properties of activated carbon has been investigated by using X-ray diffraction, EDX and SEM analysis.

Finally the utility of these activated carbon webs is checked for electromagnetic shielding at high frequency region (i.e. at 2.45 GHz) and ohmic heating at 1.5 voltage. The electromagnetic shielding effectiveness of 28.29 dB, 26.06 dB and 3.34 dB is shown by activated carbon web produced at 1200, 1000C and 800°C, respectively. This behavior can be attributed due to stronger absorption and increased multiple internal reflections of electromagnetic radiations. However results of ohmic heating are otherwise due to more resistance offered by the activated carbon webs at low temperature carbonization. As the temperature for carbonization is keep on increasing more parallel orientation of carbon chains makes flow of electrons easily as a result decrease in the generation of heat.

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5 REFERENCES

1. Negru D, Buda C.T., Avram D.: Electrical conductivity of woven fabrics coated with carbon black particles, Fibres and Textiles in Eastern Europe 90(1), 2012, pp. 53-56
2. Chen H.C., Lee K.C., Lin J.H., Koch M.: Comparison of electromagnetic shielding effectiveness properties of diverse conductive textiles via various measurement techniques, J. Mater. Process. Technol 192-193, 2007, pp. 549-554, doi: 10.1016/j.jmatprotec.2007.04.023
3. Safarova V., Tunak M., Militký J.: Prediction of hybrid woven fabric electromagnetic shielding effectiveness, Textile Research Journal 85(7), 2015, pp. 673-686, doi: 10.1177/0040517514555802
4. Safarova V., Militký J.: Electromagnetic shielding properties of woven fabrics made from high performance fibers, Textile Research Journal 84(12), 2014, pp. 1255-1267, doi: 10.1177/0040517514521118
5. Arjmand M., Sundararaj U.: Electromagnetic interference shielding of nitrogen-doped and undoped carbon nanotube/polyvinylidene fluoride nanocomposites: a comparative study, Compos Sci. Technol. 118, 2015, pp. 257-263, doi: 10.1016/j.compscitech.2015.09.012
6. Sano E., Akiba E.: Electromagnetic absorbing materials using nonwoven fabrics coated with multi-walled carbon nanotubes, Carbon 78, 2014, pp. 463-468, doi: 10.1016/j.carbon.2014.07.027
7. Rubeziene V., Baltusnikaite J., Varnaite-Zuravliova S., et al.: Development and investigation of electromagnetic shielding fabrics with different electrically conductive additives, Journal of Electrostatics 75, 2015, pp. 90-98, doi: 10.1016/j.elstat.2015.03.009

8. Farhan S., Wang R., Li K.: Electromagnetic interference shielding effectiveness of carbon foam containing in situ grown silicon carbide nanowires, Ceramics International 42(9), 2016, pp. 1133-1134, doi: 10.1016/j.ceramint.2016.04.054
9. Fletcher A., Gupta M., Dudley K L., Vedeler E.: Elastomer foam nanocomposites for electromagnetic dissipation and shielding applications, Composites Science and Technology 70(6), 2010, pp. 953-958, doi: 10.1016/j.compscitech.2010.02.011
10. Siqueira G., Abdillahi H., Bras J., et. al.: High reinforcing capability cellulose nanocrystals extracted from syngonanthus nitens, Cellulose 17(2), 2010, pp. 289-298, doi: 10.1007/s10570-009-9384-z
11. Safarova V, Tunak M, Truhlar M, et al.: A new method and apparatus for evaluating the electromagnetic shielding effectiveness of textiles, Textile Research Journal 86(1), 2016, pp. 44-56, doi: 10.1177/0040517515581587
12. Militky J., Safarova V.: Numerical and experimental study of the shielding effectiveness of hybrid fabrics, Vlakna a Textil (Fibres and Textiles) 19(1), 2012, pp. 21-27