# *INVESTIGATIONS ON CARBON COMPOSITE FOAMS FROM BIOMASS BY FILTER-PRESSING FOR THERMAL INSULATION AND EMI SHIELDING APPLICATIONS*

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**by**


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# ABSTRACT

Carbon foams are new generation materials having an interconnected three-dimensional cellular structure. They exhibit unique properties such as lightweight, tailorable thermal and electrical conductivity, high-temperature tolerance, and good sound and electromagnetic wave absorption. Hence, they have been widely used in various applications such as thermal management, fire-resistant lightweight structures, thermal protection system in aircraft, electromagnetic interference shielding for electronic and telecommunication devices, electrode in energy storage devices, acoustic damping, adsorbent for gases and toxic chemicals and substrate for catalysts. Carbon foams are generally prepared from fossil fuel-based synthetic polymers and pitches. Due to the depletion of fossil fuels and other environmental concerns, the researchers are focused on alternative precursors for carbon foams. Biomass is a good candidate raw material for valuable carbon products because it is available in large quantities at low cost, naturally renewable and environment-friendly. Biomasses such as tannin, lignin and sucrose are extensively researched for the preparation of carbon foams. However, natural cotton, sawdust, rice husk and newspaper waste are not explored for the preparation of carbon foams even though available in large quantities at a very low cost. The development of an environmentally friendly processing route for the production of carbon foam monoliths from these biomasses would not only generate wealth but also help the management of solid wastes. The main objective of this thesis is to develop an eco-friendly processing method for carbon composite foams from agricultural residues such as rice husk, sawdust, cotton and newspaper and their characterization for possible applications in high-temperature thermal insulation and EMI shielding.

A filter-pressing set was fabricated in-house from a PVC pipe, a Buchner funnel and a filter flask for consolidation of the biomasses. Cotton-sucrose composites were prepared by consolidating natural cotton fiber dispersed in aqueous sucrose solution by filter-pressing followed by freeze-drying. The caramelization of the sucrose in the cotton-sucrose composite followed by carbonization produced carbon composite foams. The XRD and Raman spectrum analysis indicated the amorphous (turbostratic graphite) nature of the carbon composite foams. The compressive strength (5 kPa to 1.4 MPa) and thermal conductivity (0.069 to 0.185 W $m^{-1}K^{-1}$) were depended on the foam density (0.06 to 0.31 g $cm^{-3}$) which was modulated by varying the sucrose solution concentration (100 to 700 g $L^{-1}$). The tubular carbon fiber formed from cotton was welded at their contact points by the amorphous carbon produced from sucrose leading to partial flexibility at low sucrose solution concentrations (100 & 200 g $L^{-1}$). The advancement of fiber- to- fiber bonding area at higher sucrose solution concentrations (300 to 700 g $L^{-1}$) resulted in rigid carbon composite foams. The porosity in the inter-fiber space and lumen of the carbonized cotton

fiber contributed to the low thermal conductivity. The carbon composite foams prepared at a sucrose solution concentration at 500 g $L^{-1}$ and above were amenable to machining using conventional machines and tools. The carbon composite foams exhibited absorption dominated EMI shielding. The carbon composite foams showed EMI shielding effectiveness in the range of 21.5 to 38.9 dB.

Sawdust, a waste generated in the wood industry was used for the preparation of carbon composite foams due to its unique cellular structure. Filter-pressing of sawdust powder dispersed in sucrose solution followed by freeze-drying produced sawdust-sucrose composites in which the sawdust particles were cemented together by sucrose. The caramelization of sucrose in the sawdust-sucrose composite followed by carbonization produced amorphous carbon composite foams. The density of the carbon composite foams was modulated in the range of 0.17 to 0.35 g $cm^{-3}$ by using the sucrose solution of concentrations in the range of 200 to 700 g $L^{-1}$. The compressive strength of carbon composites foams in the transverse direction (0.24 to 3.2 MPa) is higher than that in the filter-pressing direction (0.22 to 1.76 MPa) due to the preferential orientation of elongated sawdust particles transverse to the filter-pressing direction. The electrical conductivity increases from 18.2 to 33.8 S $cm^{-1}$ when the foam density increases from 0.15 to 0.35 g $cm^{-3}$. The carbon composite foams exhibited excellent fire resistance with low thermal conductivity in the range of 0.12 to 0.20 W $m^{-1}K^{-1}$. The carbon composite foams showed absorption dominated EMI shielding with total shielding effectiveness in the range of 25 to 53 dB.

Rice husk, an agro-waste produced in large quantity during rice processing was used for the preparation of carbon-silica (**$C/SiO_2$**) and carbon-silicon carbide (**C/SiC**) composite foams due to its inherent pore structure and presence of silica. Sucrose-rice husk composites were prepared by filter-pressing rice husk powder dispersions in sucrose solution followed by freeze-drying. The caramelization of sucrose in sucrose-rice husk composites followed by carbonization produced **$C/SiO_2$** composite foams. Carbothermal reduction of silica in the **$C/SiO_2$** composite for 1600 °C produced **C/SiC** composite foams. The formation of β-SiC and micro-graphite during carbothermal reduction was confirmed by XRD, TEM and Raman analysis. The density of **$C/SiO_2$** and **C/SiC** composite foams were modulated in the ranges of 0.26 to 0.37 and 0.18 to 0.29 g $cm^{-3}$ using sucrose solution concentrations in the range of 300 to 600 g $L^{-1}$. The compressive strength, thermal conductivity and EMI shielding effectiveness of the **$C/SiO_2$** composite foams were in the ranges of 0.32 to 1.67 MPa, 0.150 to 0.205 W $m^{-1}K^{-1}$ and 18 to 38.5 dB, respectively. The compressive strength was decreased to the range of 0.19 to 1.19 MPa and thermal conductivity and EMI shielding effectiveness were increased to the ranges of 0.165-0.431 W $m^{-1}K^{-1}$ and 20 to 43.7 dB, respectively, by carbothermal reduction of silica in the **$C/SiO_2$** composite foam to SiC. The electrical conductivity of **$C/SiO_2$** composite foams increased from 19 to 94 S $cm^{-1}$ when sucrose solution concentration increased from

300 to 600 g $L^{-1}$. The **C/SiC** composite foams showed higher electrical conductivity in the range of 102 to 371 S $cm^{-1}$ due to the graphitization of carbon during heat-treatment at 1600 °C for carbothermal reduction. The electrically conducting carbon, dielectric $SiO_2$ and SiC and the inherent pore channels in rice husk contributed to the EMI shielding.

The waste newspaper was successfully used for the preparation of mechanically robust carbon-gehlenite composite foams. The pulp prepared by mixing newspaper and sucrose solution was subjected to filter-pressing followed by drying produced newspaper-sucrose composites. The caramelization of sucrose in the newspaper-sucrose composite followed by carbonization produced carbon-gehlenite composite foams. The amorphous nature of carbon and the presence of gehlenite in the carbon composite foams were confirmed by XRD analysis. The gehlenite content estimated using thermogravimetric analysis decreased from 41.8 to 13.6 wt.% when the sucrose solution concentration increased from 0 to 700 g $L^{-1}$. The carbon composite foam density increased from 0.18 to 0.39 g $cm^{-3}$ when the sucrose solution concentration increased from 0 to 700 g $L^{-1}$. The porosity of the carbon composite foams, calculated from their bulk and skeletal densities, decreased from 92.8 to 82.7 % when the sucrose solution concentration increased from 0 to 700 g $L^{-1}$. The carbon-gehlenite composite foams showed compressive strength and Young's modulus in the ranges of 0.2 to 1.7 MPa and 3.4 to 69.9 MPa, respectively. The carbon-gehlenite composite foams exhibited excellent fire resistance. The carbon-gehlenite composite foams of density in the range of 0.18 to 0.39 g $cm^{-3}$ showed a room temperature thermal conductivity in the range of 0.1 to 0.2 $Wm^{-1}$ $K^{-1}$. The carbon-gehlenite composite foams exhibited absorption dominated EMI shielding. The total EMI shielding effectiveness increased from 15.9 to 44.9 dB when sucrose solution concentration increased from 0 to 700 g $L^{-1}$.

The prepared carbon composite foams from various biomasses could be good candidates for high-temperature thermal protection in aerospace, lightweight fire-resistant structure for building compartments in ships and absorption dominated EMI shielding in the electronic and telecommunication industry.