Magnetic Behavior at Low Temperature of Carbon Foams Prepared by the Controlled Pyrolysis of Saccharose

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Abstract: Carbon films and foams were obtained by the controlled pyrolysis of saccharose. Their irregular topology was confirmed by SEM. The presence of unpaired electrons was established *via* EPR spectroscopy. Magnetization as a function of both temperature and applied external field was collected using a SQUID Magnetometer and it was confirmed that the samples were paramagnetic at low temperatures (i.e 1.8K-10K) and diamagnetic at temperatures higher than 10K. Results showed that saccharose can be used as a precursor for the synthesis of carbon materials for magnetic applications at low temperature.

Magnetizm is a property conventionally associated with transition metals and transition metal complexes with unpaired electrons. In one allotropic form, carbon exists as graphite, which is structurally highly regular and contains no unpaired electrons. However, some structurally highly irregular carbon materials, with high surface areas have been reported to display magnetic properties [1]. Magnetic properties of carbon materials depend on its allotropic modification, which is important because these solid carbon samples may potentially be employed as optoelectronic and photovoltaic devices [2]. Particularly, in the case of graphite or activated carbon, the magnetic properties are governed by circular currents driven on and between graphene layers. Recent studies have shown the influence of defects and dislocations in graphene layers in carbon nanofoams [3] and physisorption in microporous carbon [4] on magnetic behavior of carbon materials. The main objective of this work is to verify the magnetic behavior of carbon foams. Samples were synthesized by the controlled pyrolysis of saccharose previously dissolved at 80ºC in water [5] or in KOH aqueous solution [6], denoted A_{NW-1} and A_{NW-2}, respectively. Samples were recrystallized at 25ºC, thermally stabilized under vacuum (200mbar) at 110ºC by 1h and carbonized by 1h under N_2 flow at 450ºC. Carbon films and foams were obtained simultaneously and the highly irregular topology of samples was confirmed by Scanning Electron Microscopy (SEM). The presence of unpaired electrons in A_{NW-1} and A_{NW-2} was established by Electron Paramagnetic Resonance (EPR) spectra at ambient temperature in vacuumed-sealed quartz samples in an X-Band EMX Bruker Spectrometer. Magnetization as a function of temperature and applied external field was collected using a Quantum Design MPMS-XL SQUID Magnetometer.

Fig. (**1**) shows the SEM image of A_{NW-1}. The random topological structure of the carbon foam and the lack of structural regularity can be seen. This random organization of carbon foams in the shape of polygonal cells with n-sides have been reported previously [5,6]. It is also important to note the presence of a light hyperbolic curvature in the surface of this carbon foam. This could be associated to the presence of topological and bonding defects in graphene layers as suggested by Rode and co-workers [3]; however, to confirm this, it would be necessary to take high-resolution transmission electron microscopies. The presence of free electrons or radicals is readily observable in both A_{NW-1} and A_{NW-2} by simple observation of the respective EPR spectra in Fig. (**2**). Furthermore, the measured Lande factor or g factor is 2.0075 for A_{NW-1} and 2.0052 for A_{NW-2}, very close to that of a free electron (ge= 2.002319). The line width is also very similar (4.37 against 4.58 Gauss) in both samples and range with values of radicals or free electrons (between 3-15 Gauss). The higher value of line width for A_{NW-2} than for $A_{NW-1,}$ suggest that interaction between dipoles in the solid would suffer a decrease in the magnetic local field due to an increase in the magnetic electrons detected.

This could be consequence of the formation of the superoxide paramagnetic KO_2 during KOH decomposition in Saccharose carbonization process [7]. Concerning to SQUID analysis, two basic data sets were obtained for A_{NW-1} and A_{NW-2}. The first one described magnetization as a function of applied field at different temperatures, and the second data set described magnetization as a function of temperature at some constant applied field. Reproducibility of magnetic data was made by duplicate. Figs. (**3**, **4**) show the relationship between magnetization and applied magnetic field at various temperatures for A_{NW-1} and A_{NW-2}, respectively, in

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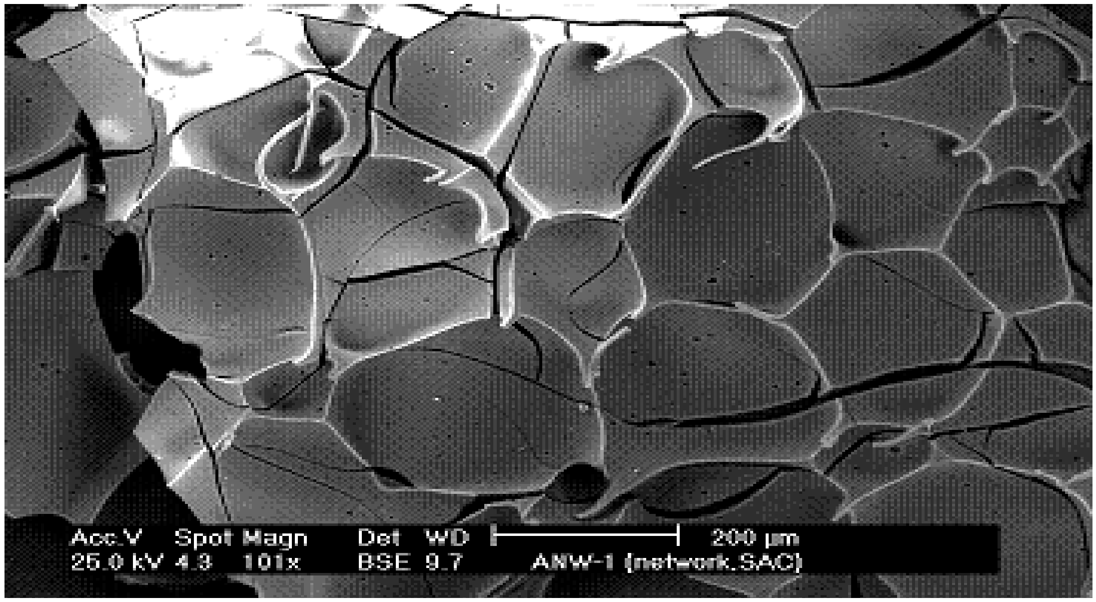

Fig. (1). SEM image of A_{NW-1} (water dissolved ex-Saccharose [5]).

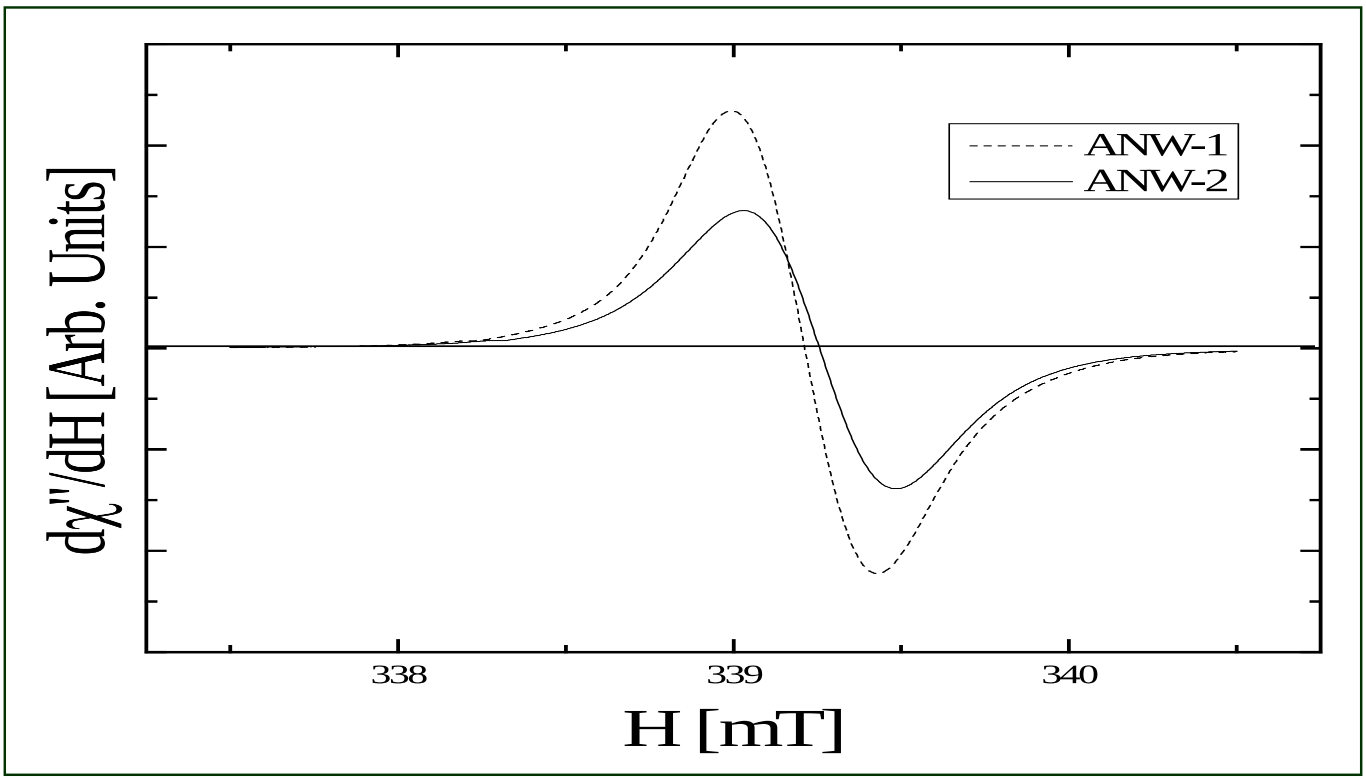

Fig. (2). EPR spectra of A_{NW-1} and A_{NW-2} (KOH aqueous solution dissolved ex-Saccharose [6]).

the temperature ranges from 1.8K to 300K and the field ranges from 0T to 5T. As is readily observable from these plots, the magnetization of A_{NW-1} and A_{NW-2} is the result of paramagnetic and a diamagnetic component. At low temperatures, 1.8K-10K, the paramagnetic component is significant in relation to the diamagnetic component, and thus both A_{NW-1} and A_{NW-2} are standard paramagnets at low temperature. As the temperature is varied from 10K to 300K, the diamagnetic component in both A_{NW-1} and A_{NW-2} outweighs the paramagnetic component, and what we observe is a standard diamagnetic compound.

Figs. (**5**, **6**) illustrate the relationship between magnetism and temperature, plotted as χ per g as a function of T in 1 kOe for A_{NW-1} and in 2kOe for A_{NW-2}. The inverse relation-

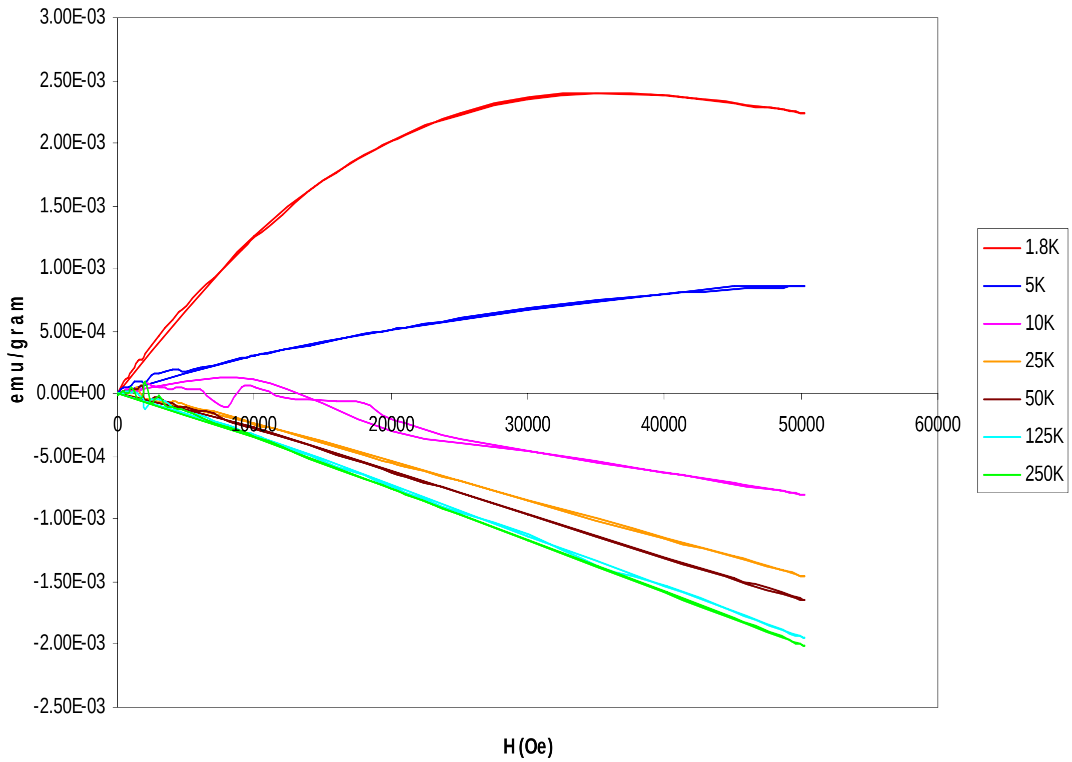

Fig. (3). Magnetization (M) as a function of H at various temperatures for A_{NW-1}

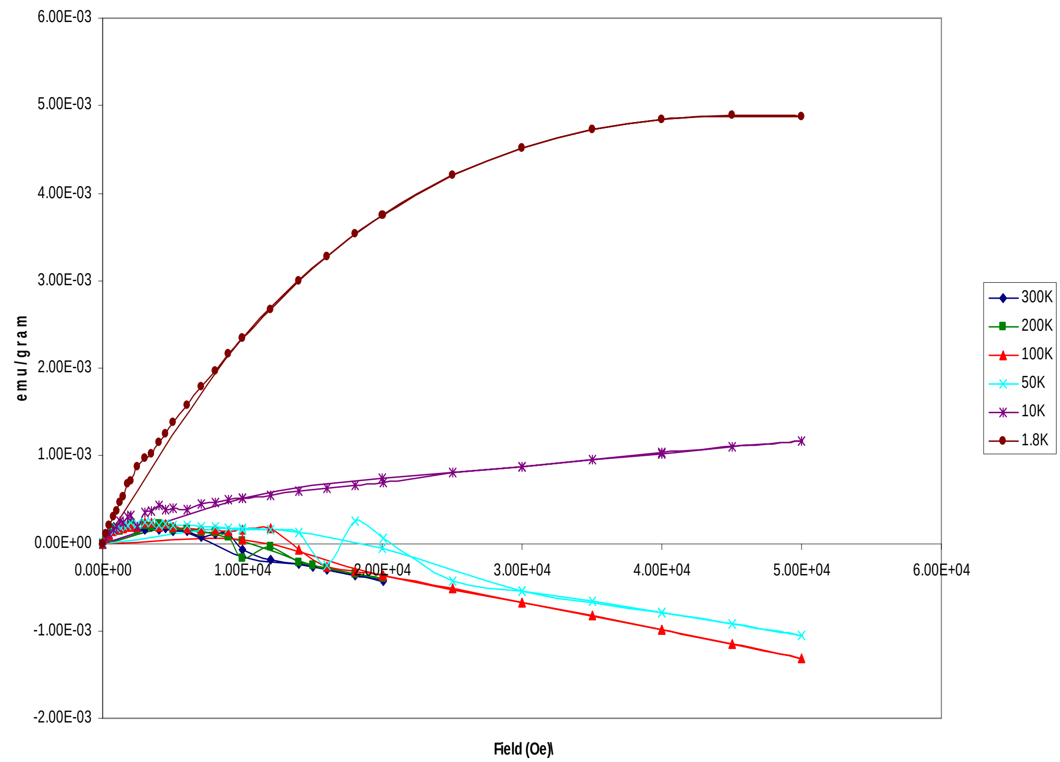

Fig. (4). Magnetization (M) as a function of H at various temperatures for A_{NW-2}

ship between χ and T, which is indicative of a paramagnet, can be readily seen for both A_{NW-1} and A_{NW-2}.

The data in Figs. (**5**, **6**) have been corrected for the intrinsic background of the sample holder, but no corrections have been made for either diamagnetizm, due to the variable nature of the structure and composition of the material. It must also be noted that the magnitude of the magnetization vector varies on the basis of the synthetic methods used to prepare the samples. This clearly reflects the variable number of free electrons or radicals in the samples based on the synthetic

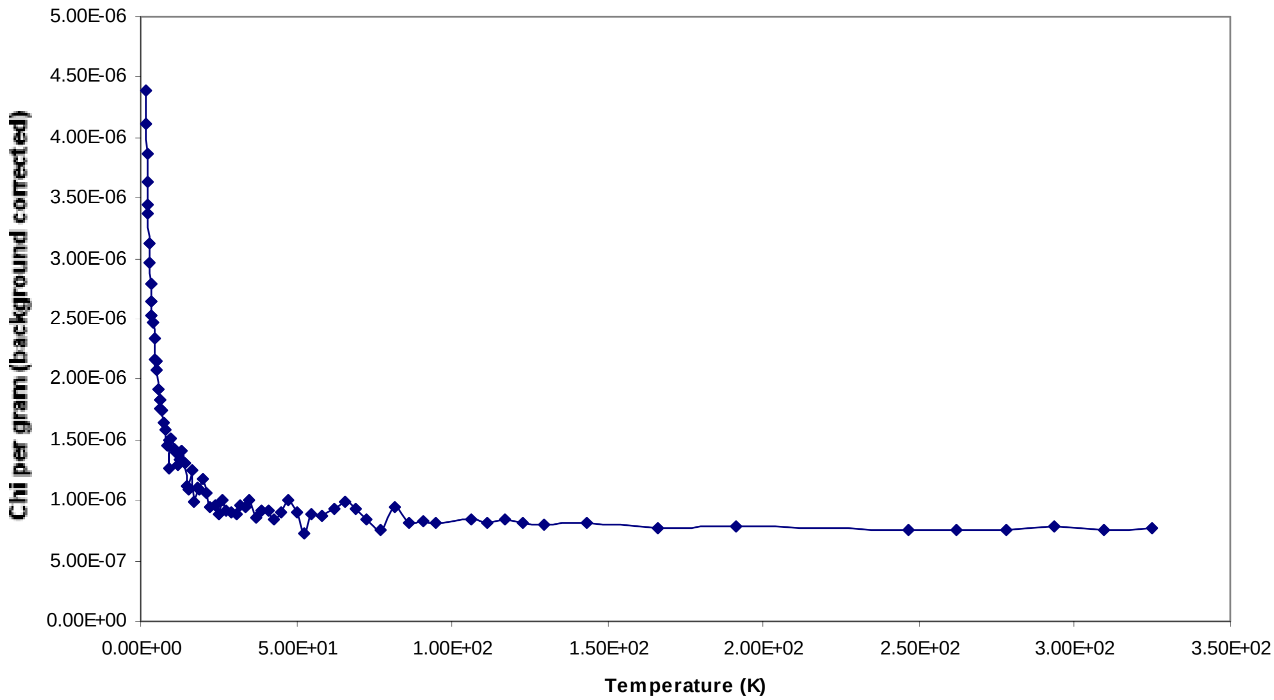

Fig. (5). χ/gm as function of T (1 kOe) for A_{NW-1}.

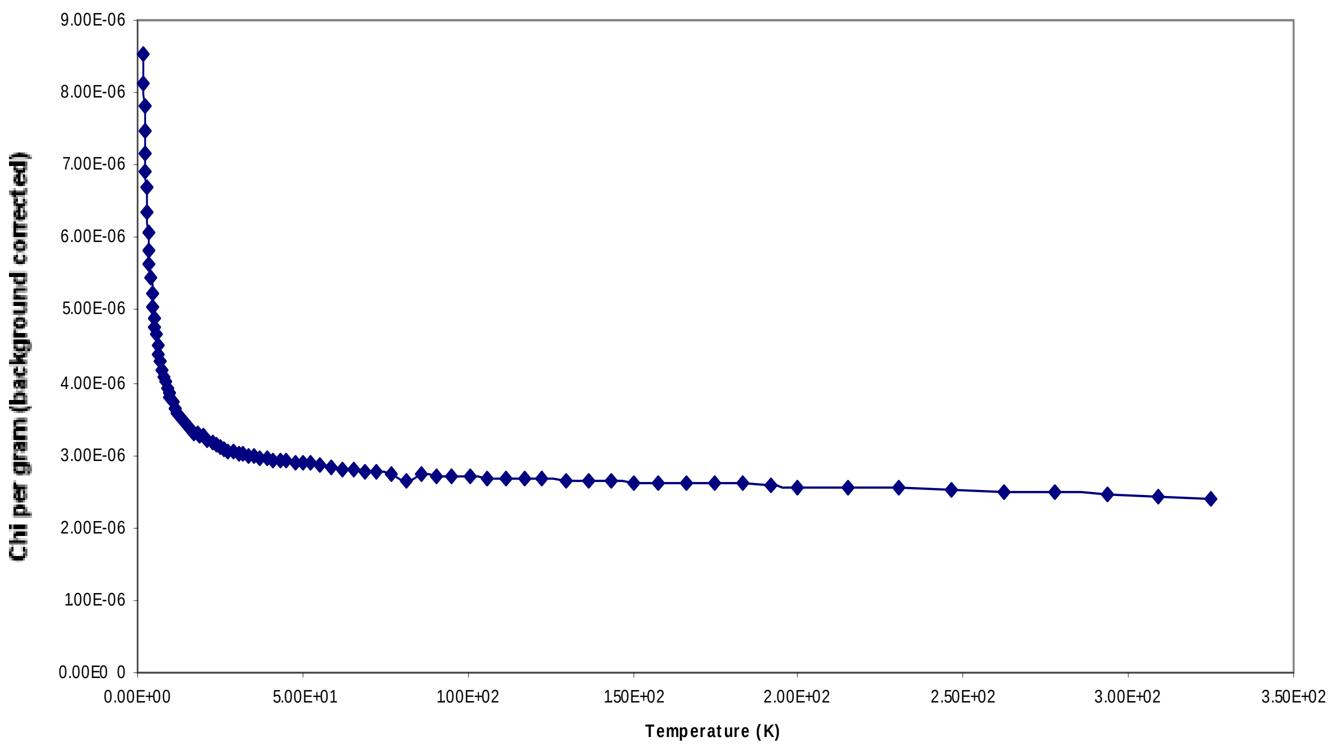

Fig. (6). χ/gm as function of T (2kOe) for A_{NW-2}.

approach utilized and network structure play an important role on the magnetic behavior of carbon foams. In conclusion, we confirmed that our samples were paramagnetic at low temperatures (i.e. 1.8K-10K) and the diamagnetic temperatures are higher than 10K. Results showed that saccharose can be used as a precursor for the synthesis of carbon materials for magnetic applications at low temperature.

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