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INFLUENCE OF SILVER ADDITION ON TEXTURAL PROPERTIES OF NEW SYNTHETIC ACTIVATED CARBON

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Abstract

Starting from macroreticular styrene/divinylbenzene sulfonic acid ion exchange resin the two samples of synthetic activated carbons (SAC), with and without silver, were obtained through carbonization in controlled regime. Detailed sample preparation procedure is described. Characterizations of synthesized samples were conducted using XRD, SEM and N_2 physisorption at –196 °C measurements. The influence of applied silver on textural properties of CAS was discussed.

Introduction

The activity of research and development of materials with controlled porosity and shape has rapidly grown during the last ten years. As for synthetic carbons, the interest was driven by numerous potential application possibilities of these materials such as molecular sieves, adsorbents, catalyst supports, electrodes for electric double-layer capacitors, etc.

In the present work, detailed sample preparation procedure of new SAC is presented. Using controlled thermal decomposition in inert atmosphere [1] two SACs with and without of silver were synthesized. The influence of applied silver on textural properties of SAC was discussed.

Experimental

Sample preparation

Ion exchange resin Amberlite 200® (Rohm & Haas Company) in the Na^+ form was washed with distilled water in a column, converted to the H^+ form by passing through 2 mol/dm^3 hydrochloric acid solution, and rinsed with distilled water in order to achieve neutral pH. This H^+ form of resin was used as the starting material for preparation SAC samples.

First sample denoted as AH800 was obtaining by controlled thermal decomposition of starting material. Heat treatment was performed in a horizontal metal tube (ϕ=3.40 cm) with gas inlet, in a furnace modified for temperature programming. Approximately 20 g of starting material beads, dried at 105 °C for 16 hours, were placed between quartz wool plugs in a tube. AH800 was prepared applying the heat treatment procedure that includes a 1 h heat up to 200 °C and 1 h hold at 200 °C. Then, the temperature was raised by 100 °C increments during 0.5 h and held at the new temperature

for 0.5 h. The temperature rise cycles were repeated until the temperature of 800 °C was reached. After holding 1 h at 800 °C, the samples were allowed to cool down to room temperature during next 18 h. The entire heating and cooling procedure, was performed under the argon flow of 0.4 dm^3/min. Immediately after cooling, the sample was placed in weighing bottle and stored in a vacuum desiccator containing anhydrous $CaSO_4$.

In order to prepare silver containing carbon designated as AAg800, the starting material was soaked in 1.0 mol/dm^3 silver nitrate solution in dark for 12h. The washing with distilled water was conducted until no nitrate ions were detected in the eluate ("Brown Ring" Test). The final sample was obtained after drying at 105 °C in dark followed by same heating-cooling treatment procedure as for AH800.

Sample characterization

The phase analysis of the dried powder was carried out by X-ray diffraction (Philips Diffractometer, Philips PW 1710) using CuKα radiation.

The Scanning Electron Microscopy (SEM) was carried out on a Low Vacuum Scanning Electron Microscope (JEOL JSM-6460LV).

The texture characteristics of the precursors were estimated using N_2 adsorption-desorption at –196 °C (Sorptomatic 1990 Thermo Finnigan).

Results and Discussion

The obtained products are hard spheres with glassy shine, having average diameter 500 μm. According to XRD patterns of AH800 (Fig. 1) the thermal decomposition of starting material leads in formation of glassy carbon like product.

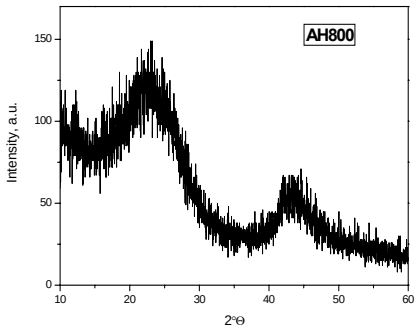

Fig. 1. XRD patern of AH800

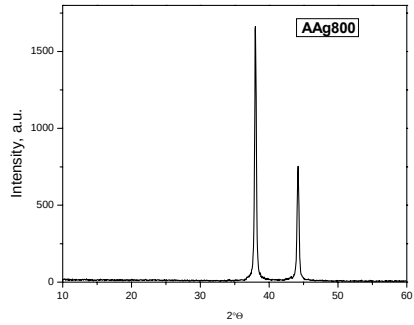

Fig. 2. XRD patern of AAg800

Two broad, low intensity diffraction peaks at 23.5 and 43.5 of 2θ corresponding to 002 plane (parallel graphene sheets) and 01 (two-dimensional reflectance of single graphene sheet) were observed [2].

XRD patterns of AAg800 sample are shown in Fig. 2. Diffractions corresponding to graphene sheets disappear and two intensive peaks originated from metal *fcc* silver phase (planes 111 and 200) exist.

Table 1. Textural data of CAS samples

Sample	S_{BET} m^2/g	V^*_{DR}, cm^3/g	V^{**}_{mes}, cm^3/g
AH800	446	0.220	0.251
AAg800	245	0.107	0.265

V^*_{DR}-Volume of Micopores byDubinin-Radushkevich model

V^{**}_{mes} - Volume of Mesopores

The data from N_2 sorption measurements (Table 1) show that addition of Ag^+ significantly changes the specific surface area (S_{BET}) of SAC. This change is dominantly originated from decrease in micropores region. However, some changes in mesopores region are also observed. Evidently, during thermal decomposition aggregation of silver is causing plugging of pores. This standpoint have stronghold in SEM pictures of synthesized materials (Fig 3, 4). External area of AH800 is porous, with obvious cavity on surface.

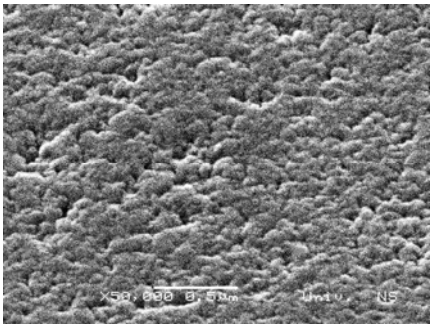

Fig. 3. SEM picture of AH800

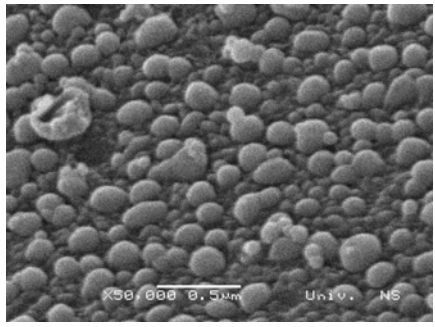

Fig. 4. SEM picture of AAg800

On the contrary AAg800 surface is overlaid by numerous spherical particles. These particles are made of metal silver that is created during thermal treatment. Although silver particle size distribution is not uniform, the most dominant class is around 100 nm.

Conclusions

Starting from H^+ form of resin Amberlite 200® using controlled thermal decomposition up to 800ºC in inert atmosphere, glassy carbon spheres are formed. Embedding of Ag^+ ions in Amberlite 200® before thermal decomposition causes significant change in textural properties of produced SAC. Silver is accumulated on surface of carbon matrix in spherical forms of approx. 100 nm in diameter.

References

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