

PREPARATION OF HIGHLY POROUS CARBONACEOUS SORBENTS FROM SEWAGE SLUDGE BY PHYSICAL AND CHEMICAL ACTIVATION

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Among the different porous materials, activated carbon (AC) is the most widely used for environmental applications such as gas-phase pollutant removal, gas separation, solvent recovery and water purification, because of its high specific surface area. Commercial activated carbons are generally produced from coals and lignocellulosic materials [1]. Additionally, in the latest years great attention is being focused on the reuse of wastes such as sewage sludges [2,3]. The objective of this study is to find more effective activating procedures to prepare AC from sewage sludge. Both dried sludges from two different WWTP (SL, SB) and their corresponding chars prepared by pyrolysis (SL-P, SB-P) were activated using physical activation (CO₂) and chemical activation (H₃PO₄, NaOH and KOH).

As shown in Table 1, pyrolysis of the raw sludges slightly increased the specific surface area, up to 50m²/g. Acid-washing of the pyrolyzed samples with 5M HCl removes part of the metal content leading to an increase in the specific surface areas for both SL-PD and SB-PD, with surface areas of 428 m²/g and 188 m²/g, respectively. This simple washing treatment is useful for the development of porous adsorbents, especially in the case of SL derived materials.

Our results show that CO₂ physical activation of the pyrolysed and the HCl-washed samples develops no appreciable porosity in the final materials, under the experimental conditions used. For CO₂ activation, maximum temperatures of 870°C and holding times up to 4 hours in CO₂ (100 ml/min) were used. Chemical activation with H₃PO₄ of the pristine samples and those pyrolysed was performed at 450°C, a temperature typically used for the activation by H₃PO₄ of lignocellulosic precursors, and using two different H₃PO₄ solutions (30 and 50% wt). These conditions were not either suitable for an efficient porosity development.

A detailed study about the influence of several experimental variables in chemical activation with alkaline hydroxides (Na, K) was carried out following three different mixing processes: impregnation (I), physical mixing of the hydroxide lentils and the precursor (PM), and a variation of this physical mixing method, grinding the activated agent prior to the physical mixing (GPM). The heat-treatment was performed in N₂ at 700°C for one hour. Our results show that the mixing method of the precursor and the hydroxide has an enormous influence in the porosity of the AC. Figure 1 compares the total micropore volume, deduced from N₂ data, of AC prepared from either SL-P or SB-P, using the three methods studied (I, PM and GPM) with a NaOH/precursor ratio 1/1. This figure shows that, for both precursors, the porosity of AC prepared by impregnation (I) and physical mixing (PM) is very similar, and much lower than that obtained when ground hydroxide is used (GPM). By GPM, highly activated carbons were obtained, using an activating agent/precursor ratio of 3/1, with an important microporosity contribution and specific surface areas, up to 0.46cm³/g and 1224m²/g respectively for NaOH activation, and up to 0.64cm³/g and 1686m²/g respectively for KOH. In this sense, this work remarks the importance of the mixing step during the sewage sludge activation in terms of porosity development, highlighting the interest of grinding the hydroxide.

Table 1. Porosity characterization of the precursors studied by N₂ and CO₂ adsorption (77 and 273 K, respectively).

Sample	BET (m ² /g)	VDR N ₂ (cm ³ /g)	VDR CO ₂ (cm ³ /g)
SL	< 5	-	-
SL-P	49	0.02	0.02
SL-PD	428	0.16	0.11
SB	< 5	-	-
SB-P	13	0.01	0.03
SB-PD	188	0.09	0.07

[1] M.A. Lillo-Ródenas et al., Carbon **39** (5), 751, 2001.

[2] S. Rio et al., Water Science and Technology **49** (1), 139, 2004.

[3] M. Martín et al., Carbon **42** (7), 1389, 2004.

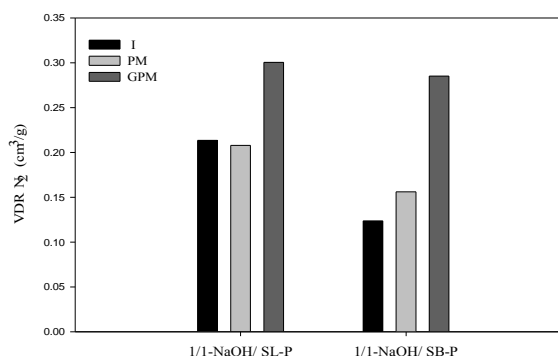


Fig. 1. Micropore volume calculated from N₂ data for AC prepared from SL-P and SB-P by chemical activation with NaOH and the three mixing methods studied (I, PM, GPM).