



## Synthesis and Characterization of Carbon Xerogel with Nitrogen in a Carbon Matrix

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**Abstract.** The preparation of carbon xerogels using urea as nitrogen components incorporated into a polymer matrix by a sol-gel process is described. The effect of nitrogen groups on the textural and surface chemical properties of the materials was investigated. The samples prepared presented extended porosities, with BET surface areas around 500 m<sup>2</sup>/g and different pore size distributions. The acid/base properties investigated by titration showed that the nitrogen-modification of carbon xerogels increased their surface basicity.

Carbon xerogels are nanostructured carbon materials that have received considerable attention in the scientific literature over the last decade [1]. These materials can be prepared from carbonization of organic xerogels, which can be obtained by resorcinol-formaldehyde aqueous polymerization. These carbon xerogels are highly porous materials possessing a number of exceptional and even unique physical properties that are of a noticeable interest for applications as varied as electric double-layer capacitors, thermal and phonic insulators, chromatographic packing, adsorbents, and catalyst supports. However, a further advancement in the application of these mesoporous carbons in adsorption, catalysis or electrochemistry requires taking advantage of surface chemistry. Recently, the profitable effect of nitrogen functionalities on the performance of porous carbons used as catalyst and adsorbent has been observed by several authors [2]. The main interest in enrichment of porous carbons in nitrogen is to induce basicity to the surface. The present study investigated the carbon xerogels preparation from a new kind of polymer precursor, which employs urea as nitrogen components incorporated into polymer matrix in sol-gel process. The urea can be hydroxymethylolated by the addition of formaldehyde to amino groups. This process consists of a series of reactions that lead to the formation of mono- and dimethylolureas. The hydroxymethylated urea (UF) have functional groups that are theoretically capable of undergo condensation reactions by building methylene or ether bridges, resulting in crosslinked structures. Table-1 shows the textural parameters for the carbon xerogels prepared and a typical carbon xerogel XP350 (nitrogen free) used for comparison. The results of the textural analysis showed that the nitrogen modified carbon xerogels are strongly affected by the catalyst ratio R/C. The initial pH observed without the presence of catalyst was 3.5, which increased to around 7.0 when Na<sub>2</sub>CO<sub>3</sub> was added. However, this value changes very fast during the reaction. Therefore, the pH variation during the polymerization reaction became larger or smaller depending on the proportion of catalyst used. The effect of nitrogen modification in the surface acid-basic properties was studied by Boehm's titration. According to the results summarized in Table-2, more pronounced basic properties are observed for nitrogen-doped carbon xerogels.

Table 1 – Synthesis conditions and textural properties of the carbon xerogel samples.

sample	R/C	V <sub>t</sub> (cm <sup>3</sup> g <sup>-1</sup> )	Área BET (m <sup>2</sup> /g)
XP350	350	0.75	655
XU200	200	0.25	328
XU350	350	0.65	513

R/C= Resorcinol/Catalyst ratio; S<sub>BET</sub>= BET surface area and V<sub>t</sub>= total pore volume

Table 2 – Result of acidity distribution from Boehm's titration

Sample	Acidic sites (mMol g <sup>-1</sup> )			Total basic sites (mMol g <sup>-1</sup> )
	Carboxyl	Lactone	Phenol	
XP350	0.042 ± 0.003	0.067 ± 0.012	0.011 ± 0.008	0.251 ± 0.008
XU200	0.056 ± 0.002	0.067 ± 0.007	0.069 ± 0.006	0.39 ± 0.02
XU350	0.079 ± 0.008	0.007 ± 0.003	0.039 ± 0.002	0.404 ± 0.004

[1] C. Moreno-Castilla, F. J. Maldonado-Hódar. Carbon 43 (2005) 455-65.

[2] M. Pérez-Cadenas, C. Moreno-Castilla, F. Carrasco-Marín, A. F. Pérez-Cadenas. Langmuir 25 (2009) 466-70.