“Synthesis, characterization and application of mesostructured silica-carbon composites”

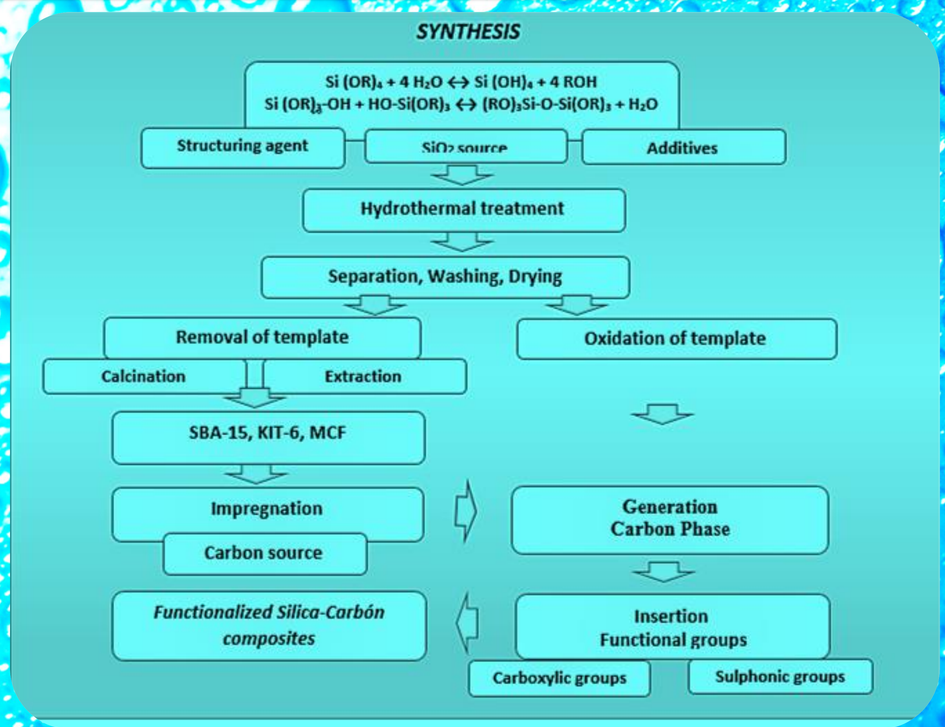
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ABSTRACT

Mesoporous silicas SBA-15, KIT-6 and MCF and corresponding silica-carbon composites have been synthesized, varying the template extraction method, carbon precursors and synthesis conditions. Subsequently carboxylic or sulphonic groups were incorporated to the prepared composites to obtaine mesoporous silica-carbon composites with active surfaces. These materials have been characterized to evaluate their structural, morphological, textural and elemental composition properties. Its functional tests have been carried out on catalytic reactions of esterification (as acid catalyst) and on the remediation of water contaminated by cationic dyes and heavy metals.

INTRODUCTION

The current research is focused on replacing conventional catalysts with solid heterogeneous catalysts, to solve the operational and environmental problems of the chemical industry [1]. Porous solids like mesoporous silica-carbon compounds with specific functional groups incorporated on their surface use as heterogeneous catalysts [2]. The research proposal of this work consists of the synthesis based on the use of only structuring agent as the precursor of the carbon surface layer of functionalized mesostructured silica-carbon materials.



RESULTS AND DISCUSSION

Table 1 summary of textural properties of the materials obtained calculated by methods standard BET and BJH.

Table 1. Textural properties of the materials obtained

Materials	Surface area BET (m^2/g)	Total pore volume (cc/g)	Average por size Dp (Å)
Silica (SBA-15) ST2aH	710.7	1.232	69.34
Composite STDC1-800	573.1	1.098	76.65
Composite STDC1-500	416.1	0.818	78.66
Functionalized Comp. ST1aDC3-5sul	545.8	1.342	98.33
Functionalized Comp. ST2aDC4-8ox	511.4	1.306	105.6
Silica (KIT-6) KT3H	830.5	1.162	55.99
Composite KTDC1-800	539.9	0.967	71.66
Composite KTDC2-500	724.5	1.115	61.59
Functionalized Comp. KT2aDC4-5sul	470.2	1.107	94.2
Functionalized Comp. KTDC1-8ox	551.7	1.007	73.8
Mesocellular silica MT1H	873.6	3.579	163.8
Composite MTDC1-800	521.6	2.761	211.7
Composite MTDC2-500	425.1	1.017	95.7
Functionalized Comp. MT1DC3-5sul	357.8	2.460	275.0
Functionalized Comp. MT1DC5-8ox	357.9	1.678	195.4

Nitrogen adsorption - desorption isotherms and hysteresis cycles (Figure 1) correspond to materials with developed uniform mesoporous systems [3, 4]. The pore sizes are in the range 5-20 nm characteristic for mesoporous materials

a) *b)*

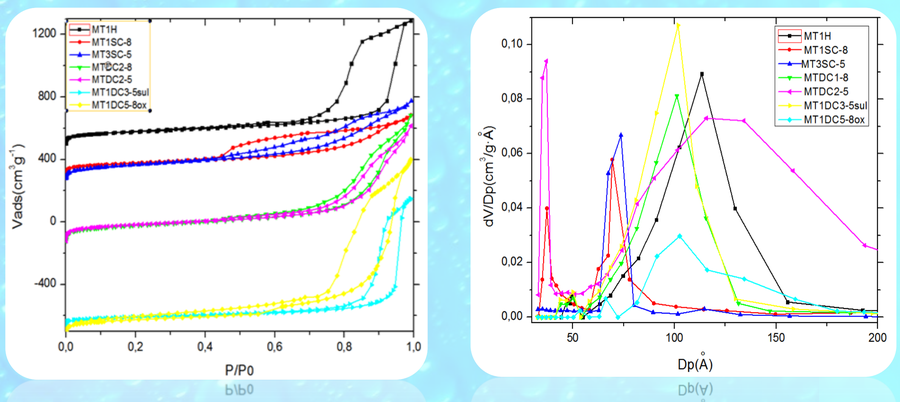


Figure 1. Nitrogen sorption isotherms (a) and pore size distributions (b) of the materials obtained from the mesocellular silica.

The study of physisorption and low angle XRD show that the incorporation of carbon phase doesn’t significantly affect the structure of sililca (Figure 2).

Energy-dispersive X-ray spectroscopy (EDS) study (Table 2) confirms the composition of composite materials (silicon, oxygen, carbon and sulfur).

IR spectroscopy (Figure 4) allowed the identification of carboxylic and sulfonic groups on the surface of silica-carbon composites [5].

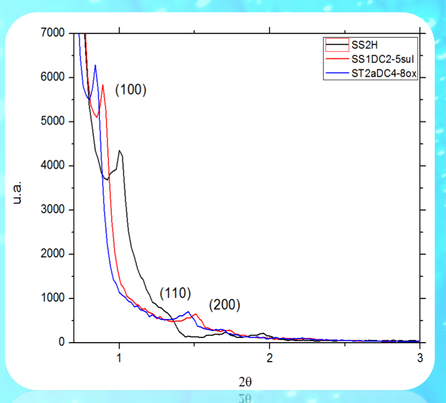


Figure 2. XRD patterns in the low-angle region of the surfactant-based functionalized silica (SBA-15)-carbón composites.

Table 2. Elemental Composition of Composites *obtained*

	SS1DC2-5sul	KT2aDC4-5sul	MT2DC4-5sul
C %	23.12	14.19	16.59
O %	54.04	54.77	54.37
Si %	22.84	30.76	28.67
S %		0.27	0.37

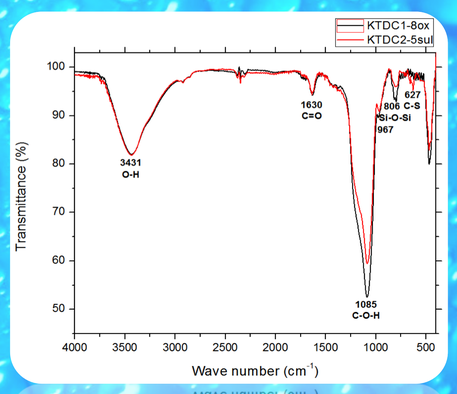


Figure 3. FTIR spectra of the surfactant-based functionalized silica (KIT-6)-carbón composites [5].

The efficiency of the *sulfonated composites* is top that of some commercial catalysts [5] and compares with that of the homogeneous catalysts (Figure 4). The oxidized silica-carbón composites show good ability to act as sorbents of cationic dyes (MB~300-450 mg/g). The amounts of the adsorbed dye were adjusted to linear equations of Langmuir, Freundlich and BET. Langmuir's theory is the one that best fits the experimental data.

a) *b)*

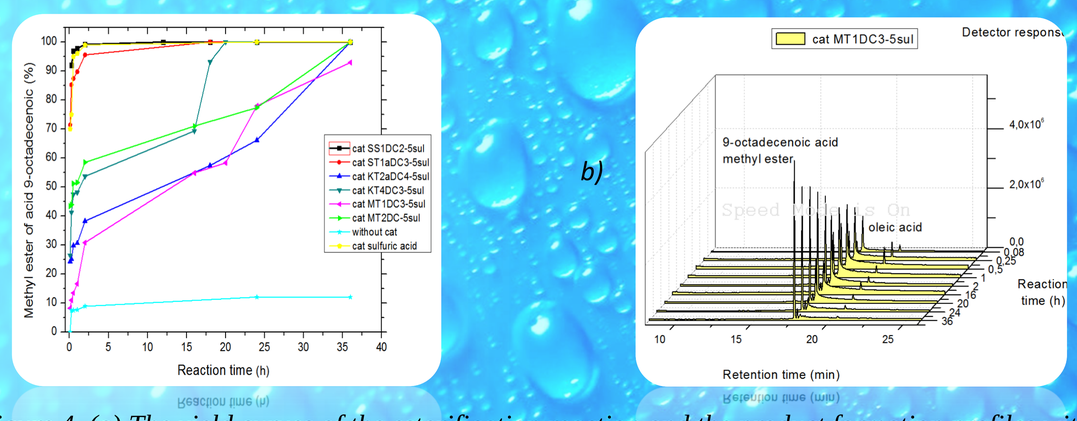


Figure 4. (a) The yield curves of the esterification reaction and the product formation profiles with time (Functionalized silica-carbon composite based on surfactant as catalyst)
(b) Chromatograms of the esterification process

CONCLUSIONS

- It was verified the feasibility and the reproducibility of the synthesis of mesostructured silica-carbon composites from the template as a source of the carbon phase. The resulting materials have structural, textural and chemical properties desirable for applications in adsorption and catalysis.
- The incorporation of carbon and functionalities does not change notably the characteristic porous structure of pure mesoporous Silicas. Pore sizes are suitable to allow the mass flow of reagents and products in catalytic applications.
- Sulfonated composites have good potential as acid catalysts. Oxidized silica-carbon composites have a good adsorption capacity of the cationic dye.

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