Sociedad Mexicana de Materiales A.C.

Mexico DF, May 5th, 2015

Juliana M. Juárez
NANOTEC UTN FRC

I am pleased to inform you that your contribution **"PREPARATION AND CHARACTERIZATION OF ACTIVATED CMK-3 MODIFIED WITH VANADIUM APPLIED IN HYDROGEN STORAGE"** by *Juliana M. Juárez, Marcos B. Gómez Costa, Jorgelina Cussa, Oscar Alfredo Anunziata,* has been **accepted** to be presented in symposium **1C, Nano/Meso-Structured Carbon Bulk Materials - Synthesis, Functionalization, Interfacial Properties and Applications** at the XXIV International Materials Research Congress, to be held in Cancun in August 16 - 20, 2015.

The presentation has been accepted in the **Poster Presentation** modality. Remember that in order to include your abstract in the program book you must register before June 7th, 2015.

Organizer of the Simposium
"Nano/Meso-Structured Carbon Bulk Materials - Synthesis, Functionalization, Interfacial Properties and Applications"

PREPARATION AND CHARACTERIZATION OF ACTIVATED CMK-3 MODIFIED WITH VANADIUM APPLIED IN HYDROGEN STORAGE

Juliana M. Juárez, Marcos B. Gómez Costa, Jorgelina Cussa and Oscar A. Anunziata.
NANOTEC (Centro de Investigación en Nanociencia y Nanotecnologia), Facultad Regional Córdoba, Universidad Tecnológica Nacional, Maestro López esq. Cruz Roja Argentina, Córdoba, Argentina.
E-mail: jjuarez@scdt.frc.utn.edu.ar

The aim of this work is to synthesize a nanostructured Carbon CMK-3 modified with V in order to increase its capacity in hydrogen storage. The approach that we have followed includes synthesis of nanostructures with the experimental study of its adsorption capacity and storage properties.
Ordered nanoporous carbon CMK-3 was synthesized via a two-step impregnation of the SBA-15 mesopores with a solution of sucrose using an incipient wetness method. The sucrose–silica composite was heated at 1173 K for 4 h under nitrogen flow. The silica template was dissolved with 5 wt% hydrofluoric acid in order to remove the silica. The template-free carbon product thus obtained was filtered, washed with deionized water and ethanol, and dried. [1]
V-CMK-3 was prepared by wetness impregnation using VCl_3 as source of Vanadium in order to increase the amount of hydrogen adsorbed. The sample of V-CMK-3 was treated under H_2 flow two times at 1173 K.
Porous carbon CMK-3 and the sample modified with V were characterized by XRD, FTIR, XPS, BET, TEM and SEM. These studies indicate that it was possible to obtain a CMK-3 replica successfully from SBA-15, using sucrose as a carbon precursor. [2]
The surface areas are 1320 m^2/g and 1050 m^2/g for CMK-3 and V-CMK-3, respectively. While the nanomaterial area is significantly smaller with the incorporation of the metal, CMK-3`s characteristic structure is maintained after the metal is within the host, in agreement with the XRD studies.
Measurements of hydrogen adsorption at cryogenic temperatures and low pressures were performed. The nanoparticles of V incorporated onto the nanostructured carbon CMK-3 showed higher hydrogen uptake at low and high pressures than CMK-3. (3.4 wt% and 2.2 wt% respectively of H_2 sorption at 10 bar and 77 K).

Keywords: CMK-3, V-CMK-3, hydrogen

[1] K. Xia, Q. Gao, C. Wu, S. Song, M. Ruan, Carbon 45 (2007) 1989-1996.
[2] H. Yang, D. Zhao, J. Mater. Chem., 15 (2005) 1217-1231.

Presenting author's email: jjuarez@sdt.frc.utn.edu.ar

PREPARATION AND CHARACTERIZATION OF ACTIVATED CMK-3 MODIFIED WITH VANADIUM APPLIED IN HYDROGEN STORAGE

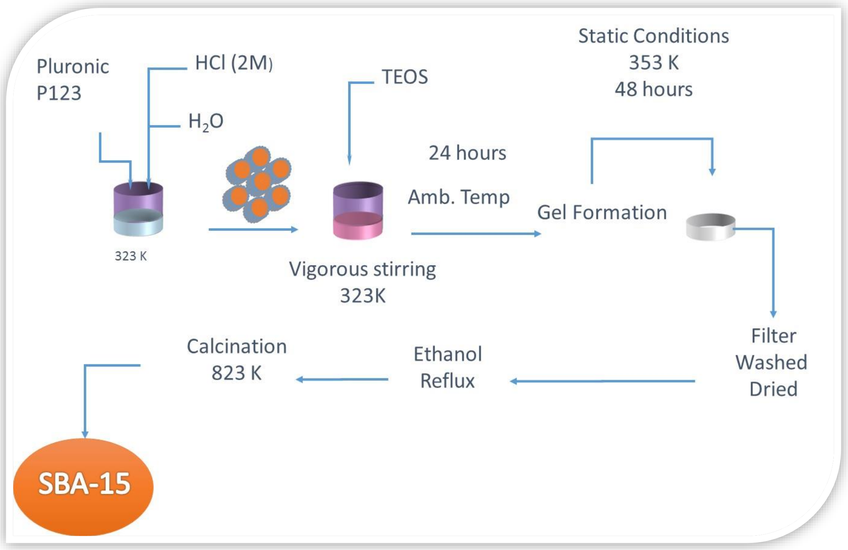
Juliana M. Juárez, Marcos B. Gómez Costa, Jorgelina Cussa and Oscar A. Anunziata.
NANOTEC (Centro de Investigación en Nanociencia y Nanotecnologia), Facultad Regional Córdoba, Universidad Tecnológica Nacional, Maestro López esq. Cruz Roja Argentina, Córdoba, Argentina.

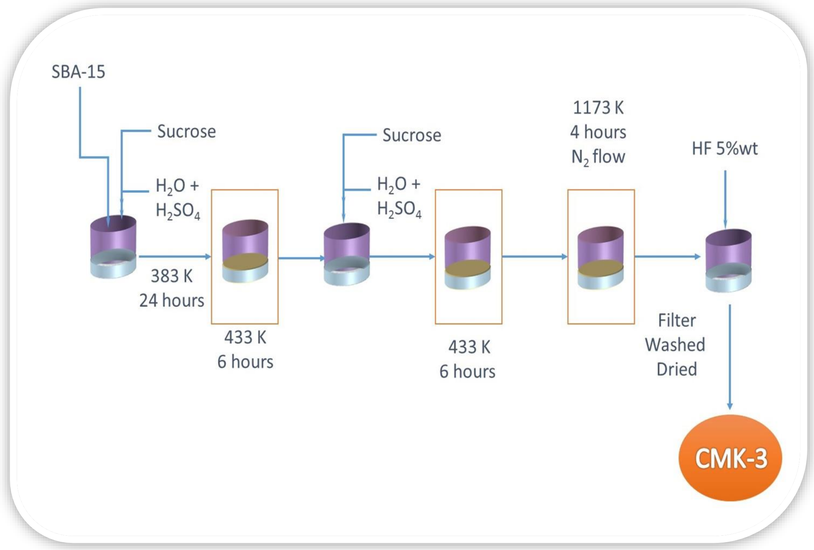
XXIII INTERNATIONAL MATERIALS RESEARCH CONGRESS

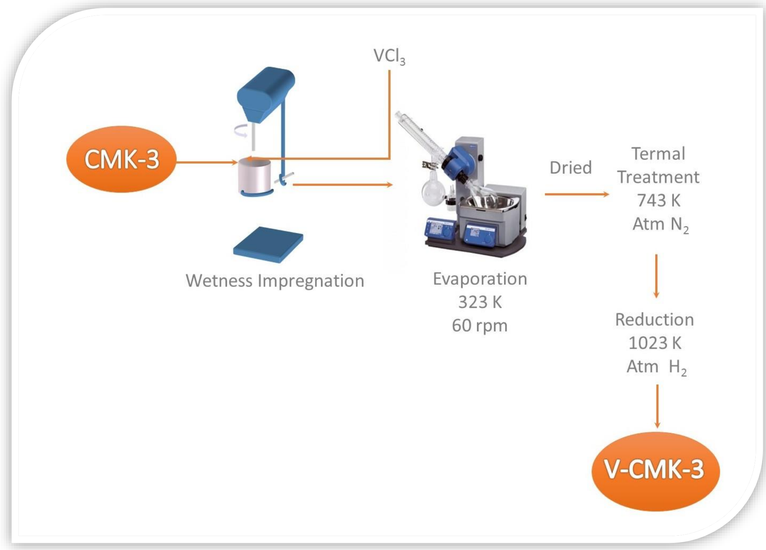
ABSTRACT

Ordered nanoporous carbon CMK-3 was synthesized via a two-step impregnation of the SBA-15 mesopores with a solution of sucrose using an incipient wetness method. V-CMK-3 was prepared by wetness impregnation using VCl_3 as source of Vanadium in order to increase the amount of hydrogen adsorbed. Porous carbon CMK-3 and the sample modified with V were characterized by XRD, FTIR, XPS, BET, TEM and SEM. Measurements of hydrogen adsorption at cryogenic temperatures and low pressures were performed. The nanoparticles of V incorporated onto the nanostructured carbon CMK-3 showed higher hydrogen uptake at low and high pressures than CMK-3 (3.4 wt% and 2.2 wt% respectively of H_2 sorption at 10 bar and 77 K).

EXPERIMENTAL







RESULTS and DISCUSSIONS

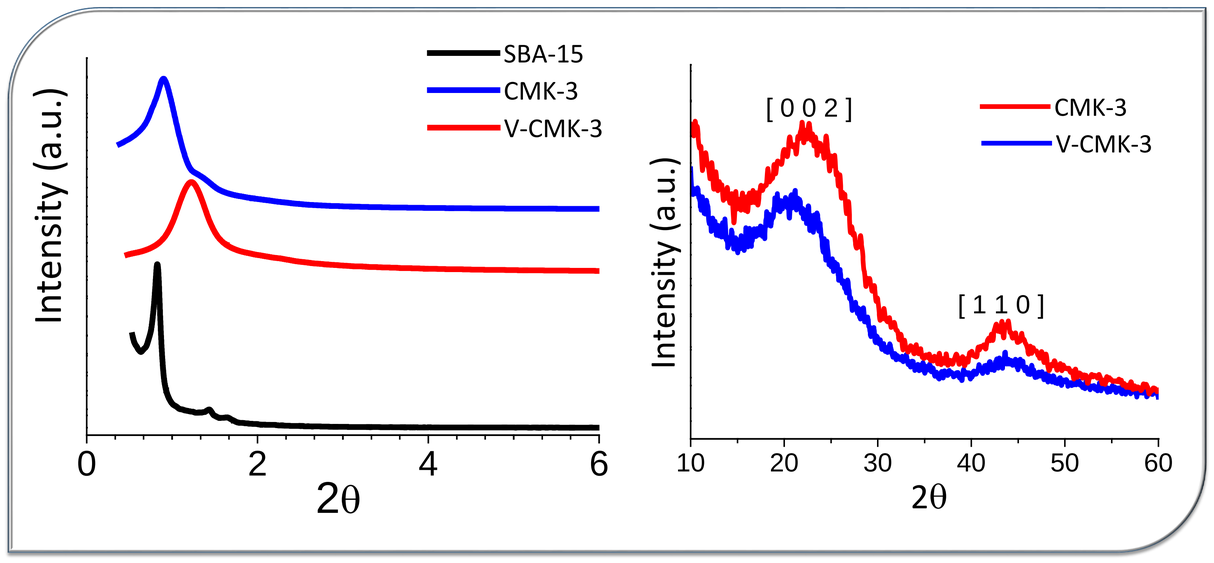


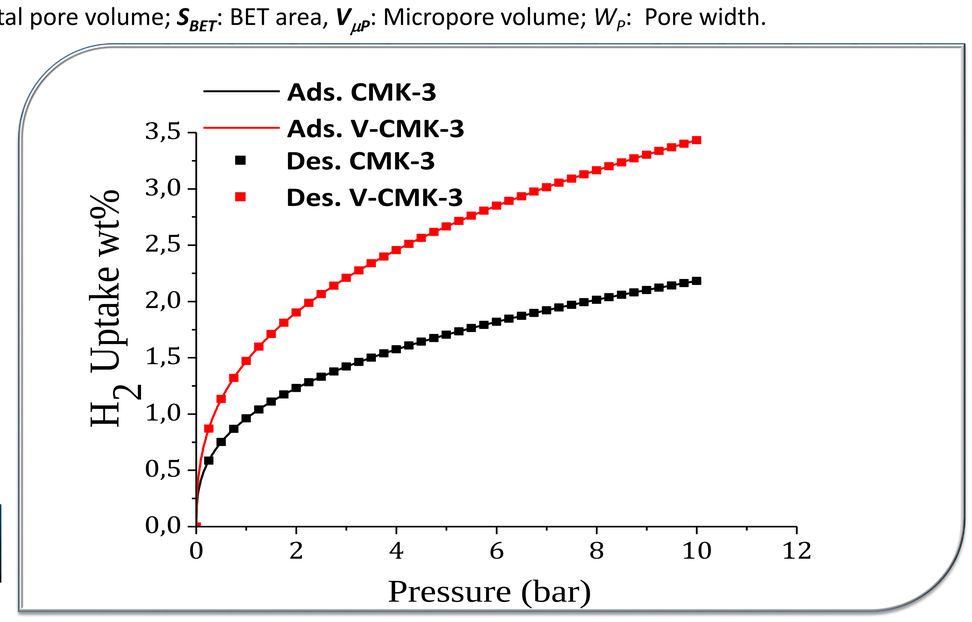
Figure 1: XRD patterns of CMK-3 and V-CMK-3.

According to XRD studies, the hard template SBA-15 shows excellent structural order for the hexagonal P6mm crystallographic space group. In the case of the mesopouros carbon CMK-3, obtained by hard templating of SBA-15, the pattern indicates that the carbon CMK-3 is an exact replica of the template [1]. CMK-3`s characteristic structure is maintained after the metal is within the host, in agreement with the XRD studies.

The wide-angle X-ray diffraction patterns shows in both cases two broad diffraction peaks which can be indexed as [0 0 2] and [1 0 0] diffraction for typical graphite carbons [2]. In the case of the sample modified no reflections typical of vanadium have been found, which is an evidence that the clusters have nanometric size and high dispersion [3].

Material	S_{BET} ($m^2\ g^{-1}$)	$V_{\mu P}$ ($cm^3\ g^{-1}$)	V_{TP} ($cm^3\ g^{-1}$)	W_p (nm)
V-CMK-3	1054	0.16	0.95	4.5
CMK-3	1323	0.23	1.01	4.2

V_{TP}: Total pore volume; S_{BET}: BET area, $V_{\mu P}$: Micropore volume; W_P: Pore width.



At low and high pressures, the amount of hydrogen uptake is higher in V-CMK-3 sample than CMK-3 sample

CONCLUSIONS

The nanometric carbon CMK-3 modified with V was synthesized and applied as a reservoir for hydrogen uptake. We have shown that CMK-3 ordered porous carbon modified with V nanoclusters is a promising material for hydrogen uptake. The samples were characterized by XRD, N_2 isotherms, XPS and TEM. The nanometric carbon modified with V showed higher hydrogen uptake at low and high pressures (3.4 wt% of H_2 sorption at 10 bar and 77 K) than CMK-3.

REFERENCES
[1] Yang H, Zhao D. Synthesis of replica mesostructures by nanocasting strategy. Journal of Material Chemistry 2005; 15:1217–1231,
[2] Suryavanshi U, Iijima T, Hayashia A, Hayashi Y, Tanemura M. Fabrication of ZnO nanoparticles confined in the channels of mesoporous carbon. Chemical Engeneering Journal 2012; 179:388-393.
[3] Gómez Costa MB, Juárez JM, Martínez ML, Beltramone AR, Cussa J, Anunziata OA. Synthesis and characterization of conducting polypyrrole/SBA-3 and polypyrrole/Na-AlSBA-3 composites. Material Reseach Bulletin 2013; 48:661-667.

ACKNOWLEDGMENT
O.A.A. Inv. PRI. CONICET, M.B.G.C. Inv. Adj. CONICET, J.C Inv. Asist. CONICET, J.M.J. Pos-Doctoral Fellowship CONICET. CONICET GRANT PIP Nº 11220120100218CO (2013-2015).

S1C-P001