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Hierarchical properties of zirconia ceramic foams prepared by the sol-gel method in presence of different surfactants.

M.A. Alves-Rosa (1), C.V. Santilli (1), S.H. Pulcinelli (1)
(1) Instituto de Química, UNESP/Araraquara, Brazil. e-mail: nalva@iq.unesp.br

Abstract – Zirconia ceramic foams were produced by aeration of the aqueous sol in presence of surfactants by sol-gel process. The results show the presence of high porosity (90%) and pore volume (fig.1) and hierarchical structure of pores size in macro – between 10 and 70μm, and mesoscales – 3,7 to 14,3nm. The hierarchical porous structure and pores walls texturization of ceramic foams produced by this process may certify these materials as heterogeneous catalysts.

Ceramic foams showing multimodal pores size distribution formed by the hierarchical structural organization of pores, which scales ranges from nano to macro size, have attracted great interest. It is due to the possibility of assumpt different functions specific to each structural level [1]. The integration of the sol-gel process, inorganic chemistry, air–liquid foams, biliquid foams, and others, is emerging as a broad area of research and offers the possibility of achieving new architectures at various length scales and with enhanced properties [2,3]. The texturization of pore walls may certify these materials as heterogeneous catalysts. To foams preparation a transparent aqueous sol of sulphated zirconia (3,5 $molL^{-1}$) was aerted under vigorous and constant stirring (2000rpm) in presence of different surfactants (10%w/w): anionic sodium dodecylsulfate (SDS); cationic octadecyltrimethylamonium bromide (OTAB); non-ionic Pluronic F-127 and nonylphenol ethoxy, commercially known as IGEPAL850®, with and without the presence of a co-surfactant (1-pentanol, 5%w/w). Gelation occurred after few minutes at room temperature by adding sulfuric acid. After drying (50ºC, 24 h) and heating (600ºC, 2h) ceramic foams were obtained. The effect of the surfactant (anionic, cationic or non-ionic) and the presence or not of the co-surfactant in the foams microstructures was analyzed by Hg porosimetry, picnometry, nitrogen adsorption-desorption isotherms and electron microscopy. The results show the presence of high pore volume for samples prepared with SDS and IGEPAL850®, i.e., 2.25 and 1.74cm^3g^{-1} (tab. 1, fig.1.a), giving rise to 90 and 87% of porosity, respectively, for the samples without co-surfactant. For these foams the high pore volume is due to the large quantity of macropores (between 12 and 60μm) air bubble. This was evidenced by the low surface area of the foam with SDS (4.9m^2g^{-1}). Mesopores families were observed for foams prepared with and without co-surfactant, the Pluronic F-127 and IGEPAL850® (between 3.7 and 9.2nm) and, in small quantity, OTAB e SDS surfactants (13.4 – 3.9nm). The presence of co-surfactant seems the not stablilize the macroporous structure of the foam, decreasing the pore volume and increasing the bulk density of ceramic foams (tab. 1). The surface area increases when co-surfactant is added in the samples with ionic surfactants (SDS 4.9 to 17.5 and OTAB 52.8 to 63.3m^2g^{-1}). Spherical macropores showing pore wall texturization were observed by scanning electron microscopy for all the foams (fig.2). So that, the aeration process of zirconia sols can be proposed as a promising route to prepare ceramic foams showing high porosity and hierarchical structure.

Table 1: Influence of different surfactants in zirconia ceramic foam properties.

Surfactant	Pore Volume (cm^3g^{-1})		Surface Area (m^2g^{-1})		Bulk Density (gcm^3)	
		Co-surfactant		Co-surfactant		Co-surfactant
SDS	2.25	1.18	4.91	17.48	0.39	0.77
OTAB	1.23	1.22	52.77	63.35	0.61	0.66
IGEPAL850®	1.74	0.49	78.72	69.48	0.50	1.22
PLURONIC F-127	1.04	0.63	104.66	75.76	0.89	1.30

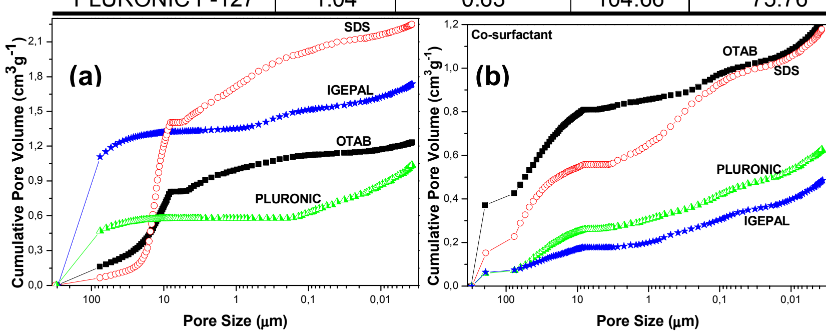

Fig. 1: Hg porosimetry of cermic foam prepared without (a) and with (b) co-surfactant.

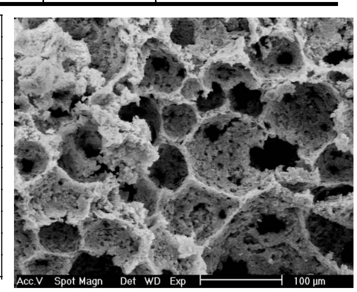

Fig. 2: SEM of foam prepared with SDS.

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