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o trabalho SILICA - ZIRCONIA MIXED OXIDE WITH SURFACE BONDED ANTIMONY: SYNTHESIS AND SOME PROPERTIES

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SILICA – ZIRCONIUM MIXED OXIDE WITH SURFACE BONDED ANTIMONY: CHARACTERIZATION OF THE MICROPOROSITY BY NITROGEN ADSORPTION

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Characterization of the microporosity of silica mixed oxides is an important stage in their application as catalysts or adsorbents. Several methods have been developed to analyze of nitrogen gas adsorption isotherm for silica-zirconium mixed oxides with surface bonded antimony (V). Specific surface area (S_a) was calculated by the BET method and the external surface area (S_{ext}) - by the t-plot. Cumulative surface area of pore (S_p) was obtained from MP-method data. The internal surface area (S_{int}) was calculated by the subtracting S_{ext} from S_a. Micropore volume (V_{mp}) was estimated by the MP, t-plot and Horvath-Kawasoe (H-K) methods. The average micropore size (radius R_{mp} or diameter D_{mp}) and the pore size distribution were evaluated by the H-K and MP methods. The results are presented in Table.

Table. Chemical composition of mixed oxides and their porous parameters

Sample ID	Zr, %	Sb, %	S_a, m^2/g BET	S_{int}, m^2/g	MP			t-plot		H-K	
					S_p, m^2/g	V_{mp}, m^3/g	R_{mp}, nm	V_{mp}, m^3/g	S_{ext}, m^2/g	V_{mp}, m^3/g	D_{mp}, nm
Si,Zr1	8.4	-	649	422	732	0.29	0.39	0.17	226	0.28	0.76
Si,Zr2	11.6	-	327	275	422	0.14	0.34	0.11	51	0.14	0.66
Si,Zr3	15.4	-	276	241	378	0.12	0.32	0.10	35	0.12	0.64
(Si,Zr1)Sb	8.1	6.3	590	458	691	0.26	0.37	0.18	132	0.25	0.73
(Si,Zr2)Sb	10.3	13.1	344	299	461	0.15	0.32	0.12	44	0.15	0.64
(Si,Zr3)Sb	14.9	11.4	440	365	519	0.18	0.34	0.15	75	0.18	0.69

All materials showed a Langmuir Type I isotherms indicating that the mixed oxides obtained are microporous. The S_a and S_{ext} values for (Si,Zr1)Sb decreased compared to Si,Zr1. The change in V_{mp} for these oxides was low. On the contrary, the S_a and S_{ext} values for (Si,Zr2)Sb and (Si,Zr3)Sb increased compared to Si,Zr2 and Si,Zr3. Particularly, this increase was sizeable for (Si,Zr3)Sb. The change in V_{mp} and S_a for (Si,Zr2)Sb and (Si,Zr3)Sb showed a similar trend. The ratio S_{int}/S_a was constant for (Si,Zr2)Sb and (Si,Zr3)Sb compared to Si,Zr2 and Si,Zr3 and increased in case of (Si,Zr1)Sb. The pore size distribution calculated by H-K and MP- methods showed peaks at 0.8; 0.7; 0.6 nm for Si,Zr1, Si,Zr2, Si,Zr3 and at 0.7; 0.6; 0.7 nm for (Si,Zr1)Sb, (Si,Zr2)Sb and (Si,Zr3)Sb, correspondingly indicating ultramicroporosity of the material obtained.

FAPESP