Study of the stability of highly oxidized metals (Ir, Ti, Ta, Sn) in ethanol-water and isopropanol-water dispersions previous to EPD

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Preliminary to EPD, a study of suspension stability is a paramount step. Zeta potential (ζ) is commonly used to indicate if a colloidal suspension will become unstable and coagulate. [1-2] In general, for mineral aqueous suspensions, the zeta potential value is positive at pH < 6, and is negative at pH > 6. To obtain a stable suspension the absolute zeta potential values typically are greater than 25 mV. [1-2]

IrO_2, Ta_2O_5, TiO_2 and SnO_2 powders were used in this research, which would be considered the active catalysts for a dimensional stable anodes, DSAs. [3-11] To ensure a large catalytic surface area, the active particles should be dispersed on an inert support. The support of a DSA should be a porous, inert body that holds its shape, dimensions and chemical integrity during the catalytic process, such as titanium. [3-11]

Dispersions were prepared with powders and reagents as received (from Alfa Aesar). The powders were suspended in the molar ratios previously reported [9], in 100 mL solutions of ethanol (EtOH) or isopropanol (IPA) with water (10:90 % by vol.). Typically 0.018 g, 0.640 g, 0.916 g and 0.916 g of IrO_2, Ta_2O_5, SnO_2 and TiO_2 powders, respectively, were dispersed in 100 mL of the solution with 0.4 g/L of hexadecyl-trimethyl-ammonium bromide (CTAB). Dispersions were mixed with a magnetic stirrer and ultrasonic bath (Branson 2210) alternately for 45 min before EPD.

Table 1 shows initial measurements of pH, effective particle size (PS) and zeta potential (ζ), of metallic oxide single dispersions with and without CTAB. The zeta potential results indicated that the presence of CTAB in the metallic oxide single dispersions was increased as we expected, being a positively charged agent. These behavior were showed in all metallic oxides studies, such as IrO_2, Ta_2O_5, TiO_2, SnO_2 in both mediums EtOH:H_2O and IPA:H_2O, indicating that the application of CTAB in the metallic oxide single dispersions should be able to consider cathodic EPD as needed.

In Table 2, metallic oxides mixtures were prepared and characterized. Initially, zeta potentials of the mixture dispersions of IrO_2-Ta_2O_5 in EtOH:H_2O and IrO_2-Ta_2O_5 in IPA:H_2O at their natural pH of 4.31 and 5.31 were measured to be -5.24 mV and -12.15 mV respectively. This indicated that a positively charged agent or surfactant to charge the particles positively would be needed to consider cathodic EPD, as we studied in Table 1 with single dispersions, therefore CTAB should be added to the mixture dispersions to obtain the expected results.

EPD-DSAs were characterized by SEM-EDS and compared with those obtained with traditional Pechini´s method.

Table 1.

	0 g/L CTAB				0.4 g/L CTAB			
	(1)	(2)	(3)	(4)	(1')	(2')	(3')	(4')
EtOH:H_2O (10:90)	IrO_2	Ta_2O_5	TiO_2	SnO_2	IrO_2	Ta_2O_5	TiO_2	SnO_2
pH	6.1	5.6	6.5	5.9	5.7	5.5	6.5	5.9
PS (nm)	397	312	120	248	628	213	802	205
ζ (mV)	-9.5	-14.7	-30.7	-18	4.7	30.8	-10.7	32
IPA:H_2O (10:90)								
pH	5.4	5.6	6.4	5.6	6.1	5.6	6.5	6.0
PS (nm)	569	363	170	348	3,359	302	358	95
ζ (mV)	-5.6	-12.5	-19.2	-11.5	9.4	35	-18	31.7

Table 2.

	0.4 g/L CTAB			
	(A)	(B)	(C)	(D)
EtOH:H_2O (10:90)	IrO_2 Ta_2O_5	IrO_2 TiO_2	IrO_2 Ta_2O_5 TiO_2	IrO_2 Ta_2O_5 SnO_2
pH	4.3	5.9	6.2	7.9
PS (nm)	308	2	2	1,770
ζ (mV)	54.6	37.7	35.4	19.2
IPA:H_2O (10:90)				
pH	4.6	6.3	5.9	7.6
PS (nm)	33	1	3	1,710
ζ (mV)	25	27.5	34.7	1.9

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