Synthesis and Optimization of Co3O4/CoO Nanostructures by Spray Pyrolysis Technology for the Process Oxygen Reduction Reaction (ORR)

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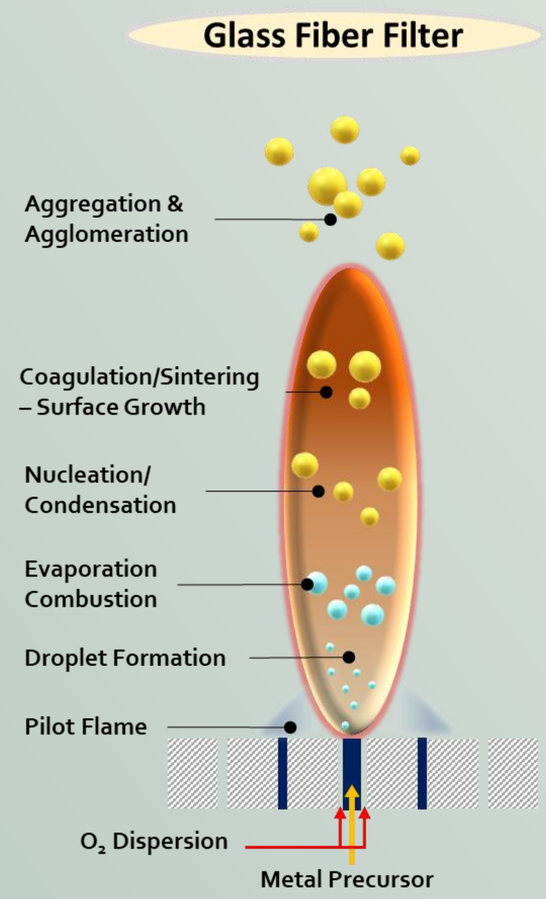
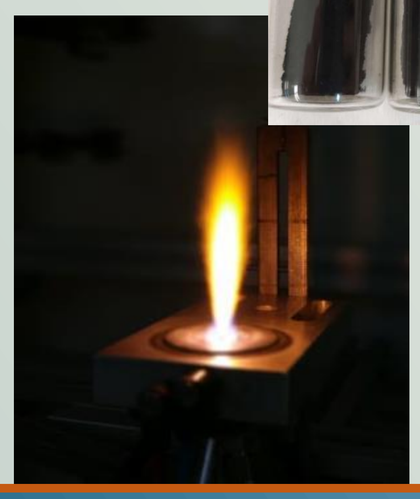
Introduction

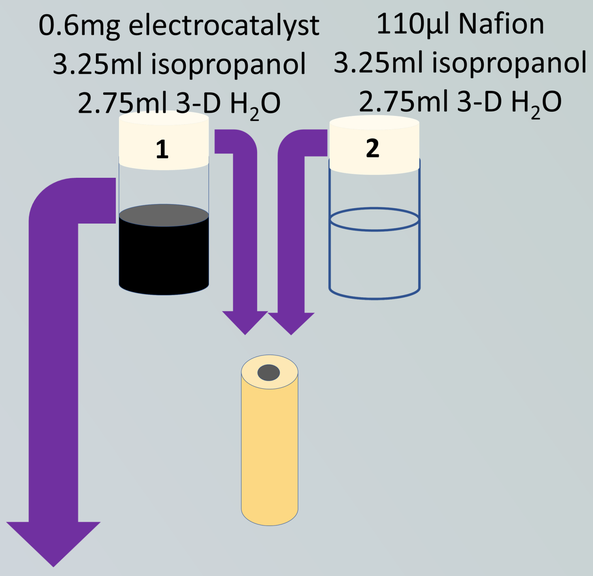
The Flame Spray Pyrolysis (FSP) method is suitable for mass synthesis of metal oxide nanoparticles with controlled properties (phase composition, crystallinity, size, morphology, defects). The Oxygen Reduction Reaction (ORR) is the most important reaction that occurs at the cathode of any fuel cell, because this reaction is not spontaneous so there is a need for some catalyst and some voltage. Usually, this reaction can be done either through process 4e- or through two serial processes 2e- and is affected by pH, temperature and finally by the properties of the catalyst material. Carrying out the electrochemical reduction of oxygen (ORR) in an alkaline instead of an acidic environment offers comparative advantages, the most important of which are a less corrosive environment and faster intrinsic kinetics. Nevertheless, with the FSP method it is possible to synthesize nanocatalysts resistant to acidic environments. The combination of Pt with a second transition metal, while reducing the cost, can lead to an increase in ORR activity due to the change in the electronic properties of the metal phase. In this direction, in this work, a series of FSP nanocatalysts on conductive carbon (Vulcan XC72R) of 10 wt.% (Pt – Co)/C bimetallic catalysts were prepared and studied in terms of ORR and study in both acidic and alkaline environments .

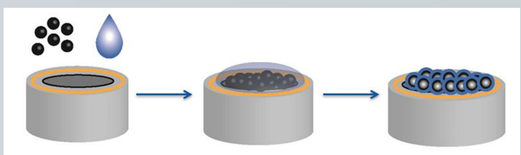
Synthesis of Electrocatalysts



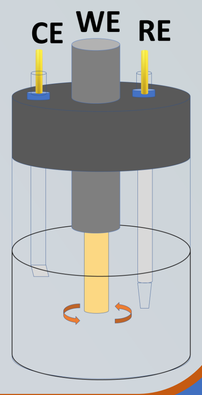


Development of electrodes



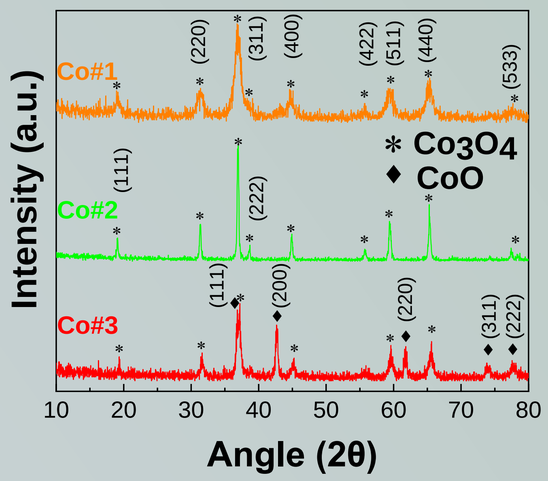


Evaluation FSP catalysts

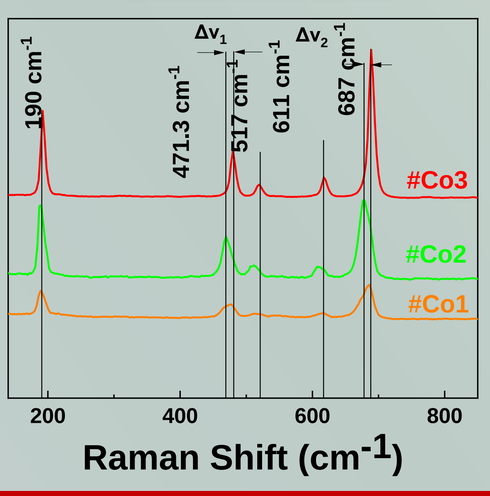


Structural and Electrochemical Analysis

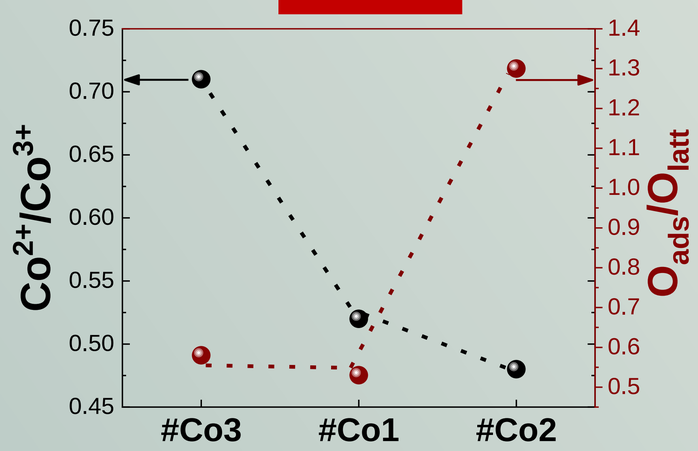
XRD



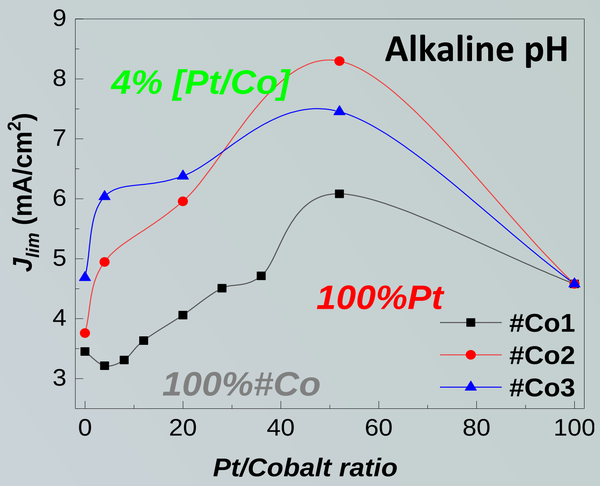
Raman

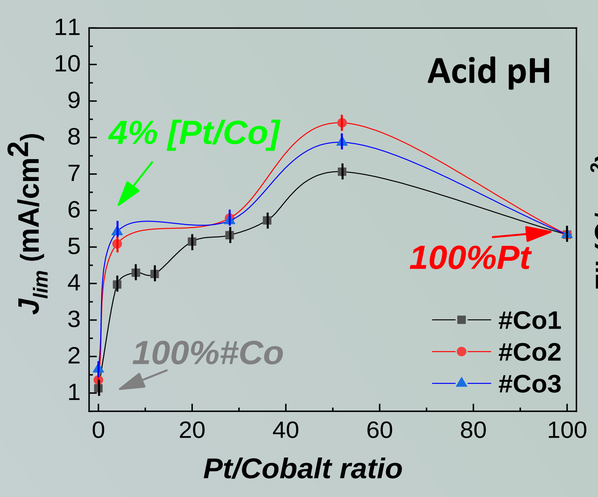


XPS

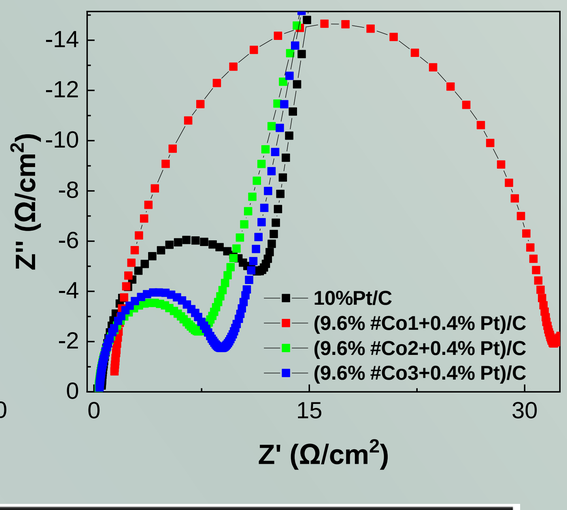


ORR





EIS



Material	d_{BET} (nm ± 0.5)	d_{XRD} (nm ± 0.5)	d_{TEM} (nm ± 2)	SSA ($m^2 g^{-1}$ ± 5)	wt%Co_3O_4 (±5%)	wt%CoO (±5%)	Co^{2+}/Co^{3+} *	O_{ads}/O_{latt} *
Pt/C	3.5	4	-	100	-	-	-	-
Co#1	10	10	11	100	100	0	0.52	0.53
Co#2	32	27	37	30	100	0	0.48	1.3
Co#3	53	11(Co_3O_4) 9 (CoO)	44	18	66	34	0.71	0.58

Conclusions

- Synthesis of Co3O4 and CoO/Co3O4 nanostructures and phase control by FSP.
- 5.2%Pt+4.8%#Co2/C with J_{lim}=8.31 mA/cm² at pH=13.1
- 0.4% Pt + 9.6% #Co3/C with J_{lim}=6mA/cm² at pH=13.1
- 5.2%Pt+4.8%#Co2/C with J_{lim}=8.26 mA/cm² at pH=1.3
- 0.4% Pt + 9.6% #Co3/C with J_{lim}=5.5mA/cm² at pH=1.3

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