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Dry Plasma Treatment of Organometallic Precursors for the Synthesis of Fuel Cells Catalyst Materials

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INTRODUCTION

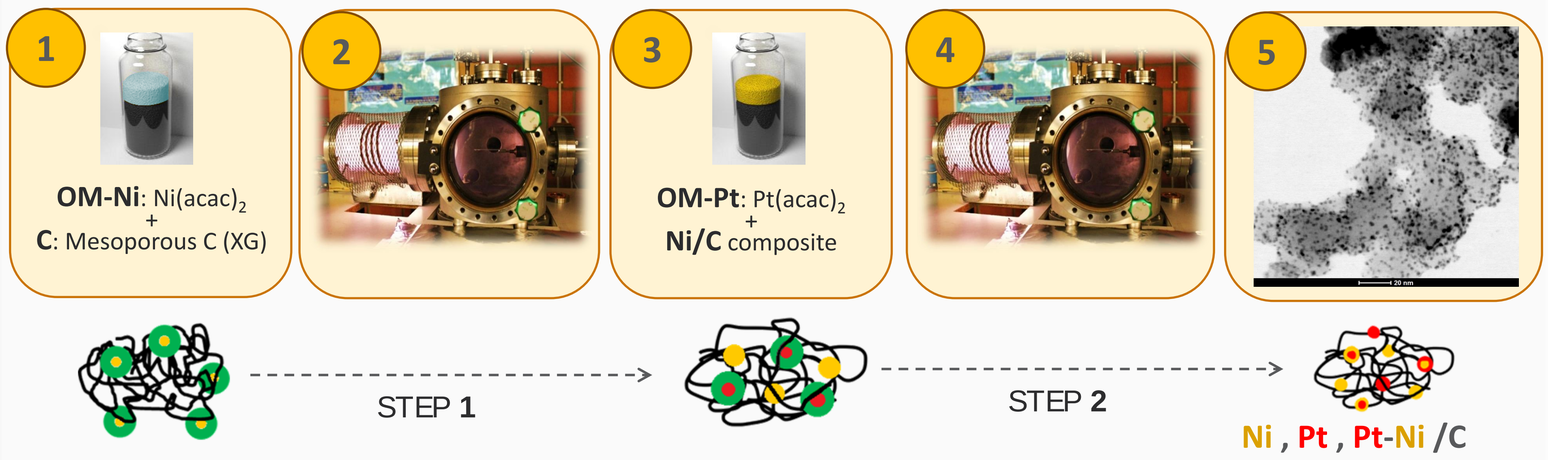
A strong limitation to the commercialization of Proton Exchange Membrane Fuel Cells (PEMFC) is the cost and durability of the nanocomposite catalyst material which relies mainly on rare noble metals, such as Platinum. By depositing bimetallic Pt-Ni nanoparticles (NPs) on a high surface area carbon substrate, we could increase the activity of Pt-Ni/C respect to conventional Pt/C [1].

Moreover, this work explores the synthesis of Pt-Ni/C composites by a novel methodology based on low-pressure plasma treatments [2-3].

Ni-NPs and Pt-Ni NPs with controlled chemical composition, structure and morphology were obtained by plasma processing and characterized by X-Ray photoelectron spectroscopy (XPS), X-Ray diffraction (XRD) and transmission electron microscopy (TEM).

The simultaneous C surface functionalization (addition of N-groups) was obtained with N-containing plasma treatments.

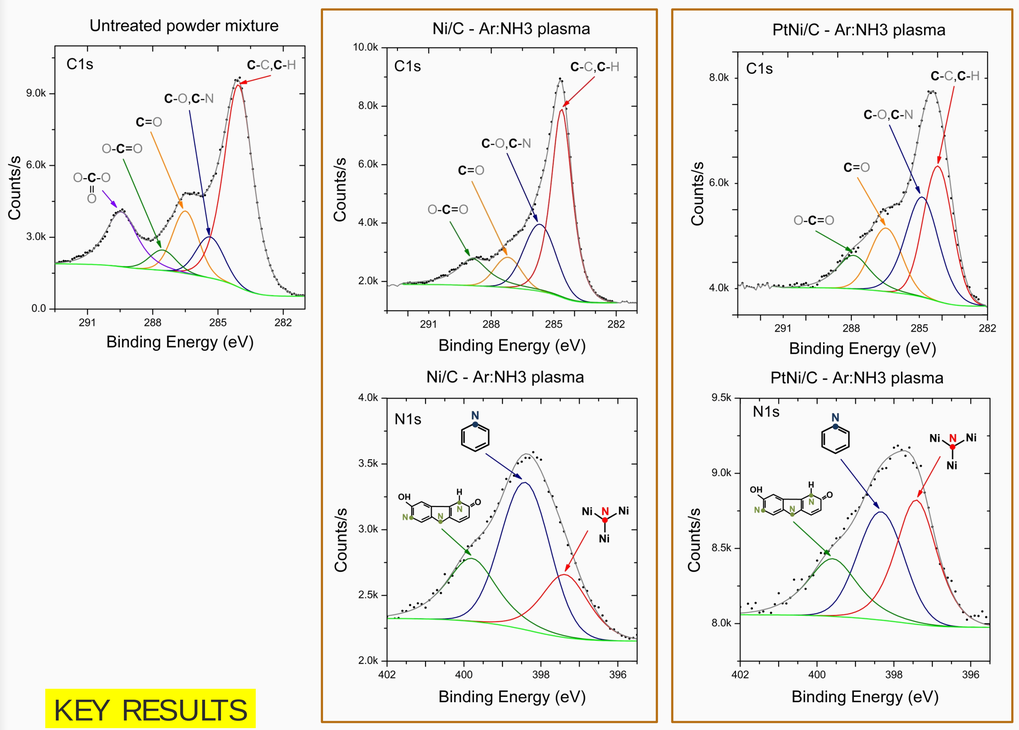
How to synthesize the NPs : plasma methodology



EXPERIMENTAL PARAMETERS

RF Power, Treatment time, Plasma chemistry (Ar, N_2, NH_3, etc.), Organometallic (OM) precursors

Functionalization of the mesoporous C substrate – HR-XPS Spectra of C1s and N1s



Plasma conditions

OM-Ni + C
- STEP **1**:
  - Ar:NH_3 / 100W / 60 min
  - +
  - Ar:NH_3 / 140W / 90 min
- STEP **2**:

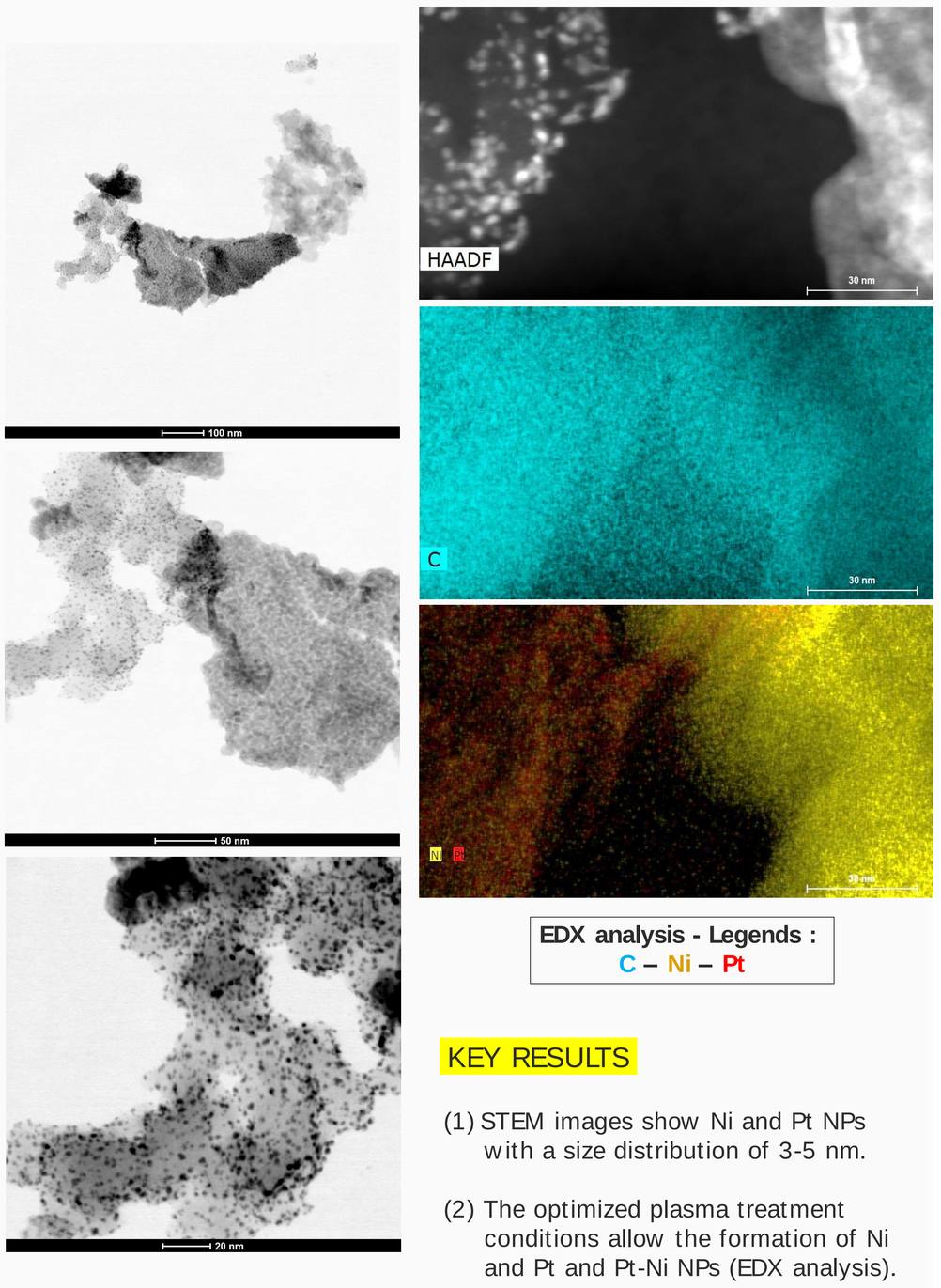
OM-Pt + Ni/C
- Ar:NH_3 / 120W pulsed / 120 min
- +
- Ar:NH_3 / 100W / 75 min
- +
- Ar:NH_3 / 140W / 5 min

KEY RESULTS

(1) NH_3 plasma leads to the incorporation of N functional groups in the mesoporous carbon substrate.

(2) The *at*% of nickel nitride component in the N 1s spectrum increases with Ar:NH_3 plasma treatment time.

Is our sample a good catalyst candidate ? STEM with EDX analysis



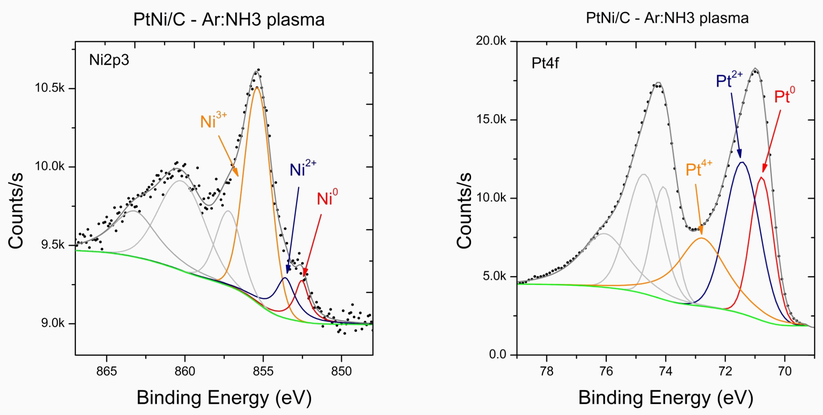
EDX analysis - Legends :
C – Ni – Pt

KEY RESULTS

(1) STEM images show Ni and Pt NPs with a size distribution of 3-5 nm.

(2) The optimized plasma treatment conditions allow the formation of Ni and Pt and Pt-Ni NPs (EDX analysis).

Chemical compositions of Pt-Ni/C nanocomposites : HR-XPS

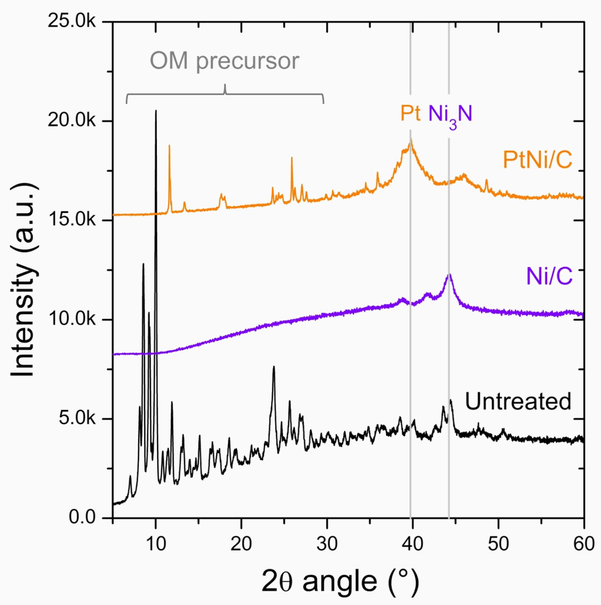


KEY RESULTS

(1) *Composition analysis*
For a starting mixture of 37 wt% of Pt and 20 wt% of Ni:
Pt~9 *at*% and Ni~2 *at*%

(2) *Chemical analysis*
Pt^0~30-35% of Pt
Ni^0~10% of Ni

Is the OM precursor still present ? XRD analysis



KEY RESULTS

(1) The untreated powder mixture shows peaks attributed to the OM precursor.

(2) STEP 1: full degradation of the Ni OM and formation of nickel nitride.

(3) STEP 2: the final catalyst shows strong peaks of Pt-NPs masking the weaker Ni-NPs reflections.

(4) The FWHM of Pt reflections confirms the NPs size ~ 3-5 nm. (Scherrer formula)

CONCLUSION

Results validate our plasma methodology to synthesize Pt-Ni/C composites. NH_3 plasma are efficient to fully degrade the Ni and Pt OM precursors and allow to control the chemical composition of our catalysts.

Catalytic activity tests are ongoing and will allow comparing the catalyst activity with commercial Pt/C catalysts. Further studies will be directed to improve our methodology in order to make Pt-Ni alloys by varying the OM precursors or by developing a one-step process.

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