

## Supplementary information

### Electrocatalytic Activity of Well-Defined and Homogeneous Cubic-Shaped Pd Nanoparticles

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Due to the difficulty to remove the polyvinylpyrrolidone (PVP) in the chemical synthesis in aqueous media, we used an electrochemical cleaning which is carried out *in situ* (the electrocatalyst is already deposited on glassy carbon plates). The advantages of employs an electrochemical cleaning is due to its simplicity, quickness, and its low cost method. The electrochemical cleaning for the PVP removal consisted in the use of cyclic voltammetry in 0.5 M H<sub>2</sub>SO<sub>4</sub> as acidic media and 0.3 M KOH as basic media.

The procedure was accomplished as follows: First, 15 cycles are performed in acidic media between 0 to 1.45 V vs. NHE at 50 mV s<sup>-1</sup> (Fig. S1A). After that, 15 cycles in basic media in a range of potential of -0.6 to 0.7 V vs. NHE at the same scan rate (Fig. S1B). Further, 15 cycles are newly carried out in acidic media (Fig. S1C).

In Fig. S1A the hydrogen adsorption/desorption region is not observed due to the presence of PVP in the material. In basic media (Fig. S1B), a more defined voltammogram is obtained. Finally, in Fig. S1C the hydrogen adsorption/desorption region is observed, which could be an indicative of the PVP removal. The main change is evident in the peak intensity (marked with \*) related to the hydrogen adsorption and (hydrogen-) sulfate desorption<sup>1</sup>. The high current density of this peak is characteristic of Pd (100) terraces, which could be an indicator that the system preserves its preferential plane<sup>2</sup>; also, is an indicator that the PVP removal was carried out successfully.

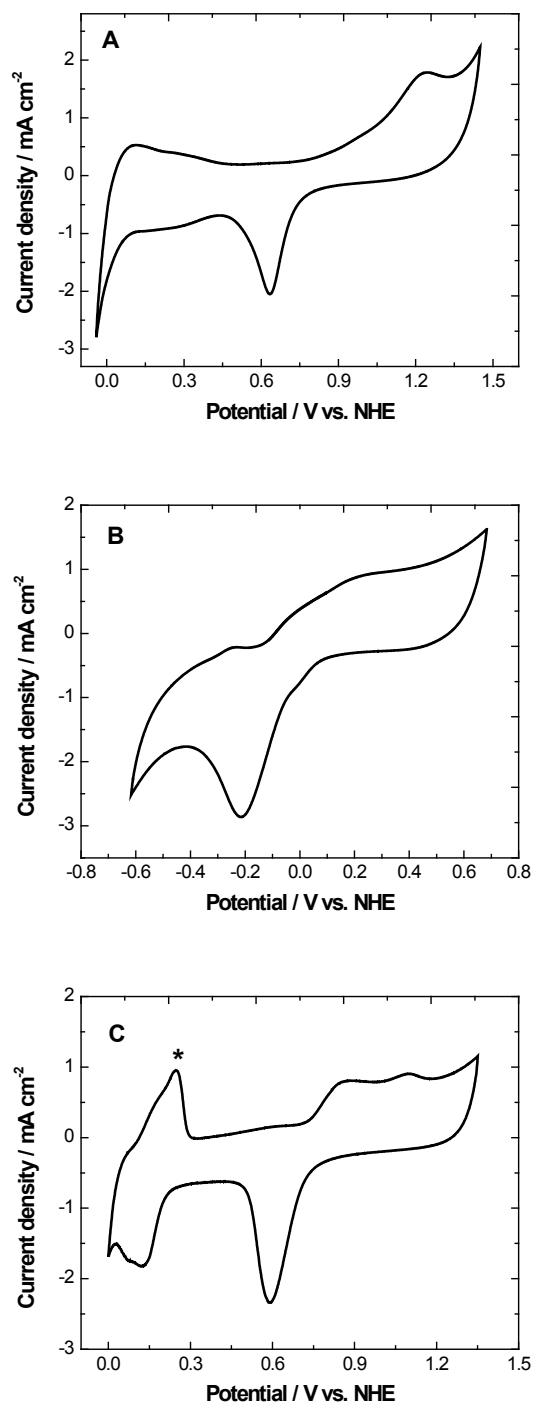


Fig. S1 Cyclic voltammograms for the electrochemical cleaning after 15 cycles in A) acidic media B) basic media C) in 0.5 M H<sub>2</sub>SO<sub>4</sub>, after of basic and acidic media treatment.

On the other hand, SEM images of the based-Pd nanocubes electrocatalyst after the electrochemical measurements including the electrochemical cleaning were performed (Figs. S2A and B). According to Figs. S2A and B, is important to note that the material supported on Vulcan maintains the shape and the approximately size previously obtained by XRD and TEM measurements.

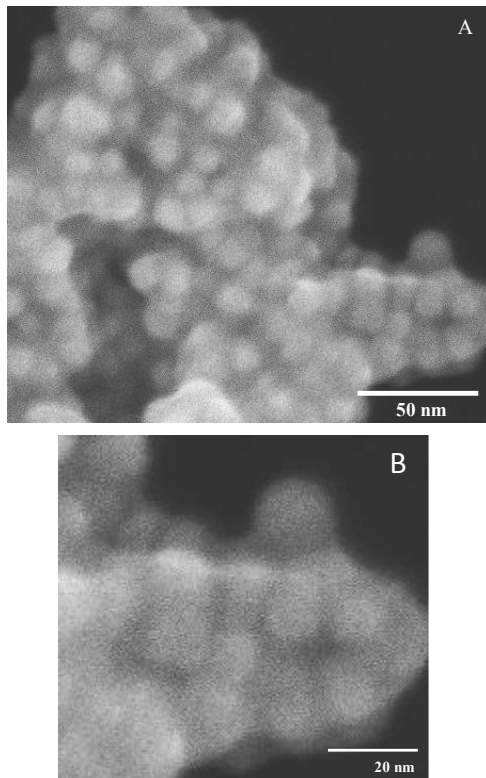


Fig. S2 A) SEM images of Pd nanocubes supported on Vulcan XC-72 after the electrochemical measurements. B) Magnification.

## References

1. N. Hoshi, M. Kuroda, O. Ogawa, O. Koga and Y. Hori, *Langmuir*, 2004, **20**, 5066-5070.
2. H. Erikson, A. Sarapuu, K. Tammeveski, J. Solla-Gulón and J. M. Feliu, *Electrochem. Commun.*, 2011, **13**, 734-737.