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Electronical Supporting Information

Evaluation of the intrinsic catalytic activity of nanoparticles without prior knowledge of the mass loading

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S1: Characterisation of synthesised Pt-NPs

For complementing the information given in the main manuscript, synthesised NPs have been immobilised on a TEM grid and their size was determined using ImageJ¹ (Figure S1, a and b). The observed diameter of about 18 nm is consistent with the diameter of immobilised NPs after electrochemical treatment as shown in Figure 1.

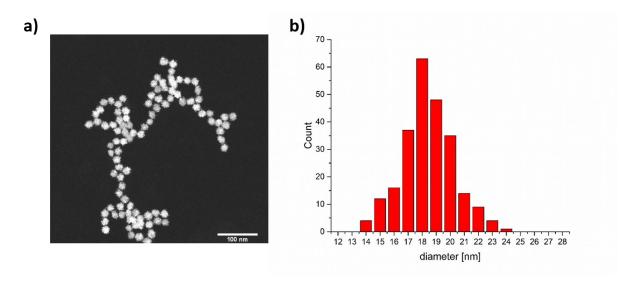


Figure S1: a) TEM image of synthesised Pt-NPs immobilised on carbon grid. b) Related size distribution of Pt-NPs determined with ImageJ.

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EDX of these NPs further confirms that the NPs are indeed made of Pt (Figure S2).

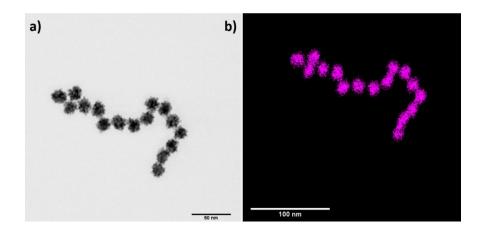


Figure S2: a) TEM image of Pt-NP cluster immobilised on carbon grid and b) EDX of the cluster with Pt being illustrated in pink.

S2: Optimisation of electrode geometry and size

For the electrochemical analysis of immobilised NPs, a trade-off between big electrode size to enhance immobilisation and small electrode size to reduce the background noise level has to be found. For this purpose, carbon nanoelectrodes have been etched for 4 min in buffered HF solution² and the so-obtained electrode surface was illustrated using SEM images (Figure S3 a). The increase in electrochemical surface area (ECSA) was also monitored using the redox mediator 5 mM [Ru(NH₃)₆]Cl₃ in 0.1 M KCl (aq.), confirming the significant increase in ECSA due to exposure of initially covered carbon surface (Figure S3 b).

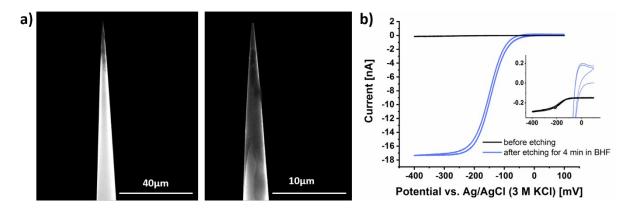


Figure S3: a) Representative SEM images of carbon nanoelectrodes after etching for 4 min in buffered HF (BHF) solution. The white region indicates sealing of insulating glass and the black region shows exposed carbon due to etching of the initial glass coverage. b) Representative CVs in 5 mM [Ru(NH3)6]Cl3 in 0.1 M KCl (aq.) as redox mediator of laser pulled carbon nanoelectrodes before (black) and after (blue) etching in BHF, illustrating the significantly increased electrode surface area after etching.

References

- 1. C. A. Schneider, W. S. Rasband and K. W. Eliceiri, *Nat. Methods*, 2012, **9**, 671.
- 2. J. Clausmeyer, P. Wilde, T. Löffler, E. Ventosa, K. Tschulik and W. Schuhmann, *Electrochem. Commun.*, 2016, **73**, 67.