Supplementary Information

Relevant parameters for the mechanochemical synthesis of bimetallic supported catalysts

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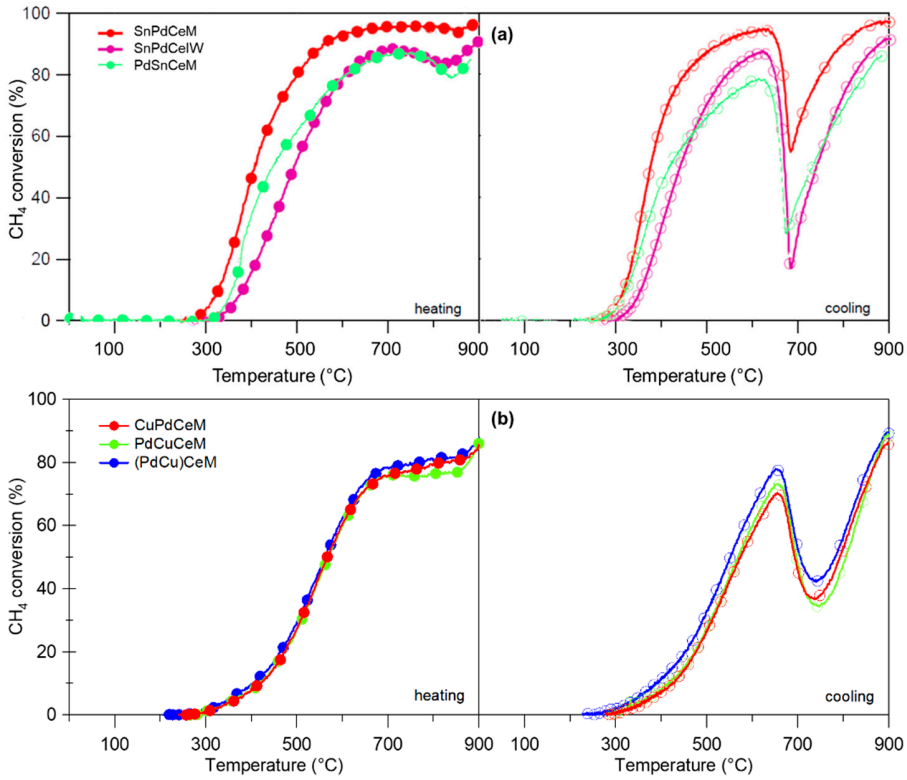

Figure S1. Methane oxidation activity of (a) PdSn/CeO_2 and (b) PdCu/CeO_2 catalysts. Gas feed: 0.5%CH_4, 2%O_2 in He, GHSV = 180'000 h^{-1}, heating and cooling ramps 10 °C min^{-1}. M: Milled samples, IW: prepared by Incipient Wetness Impregnation.

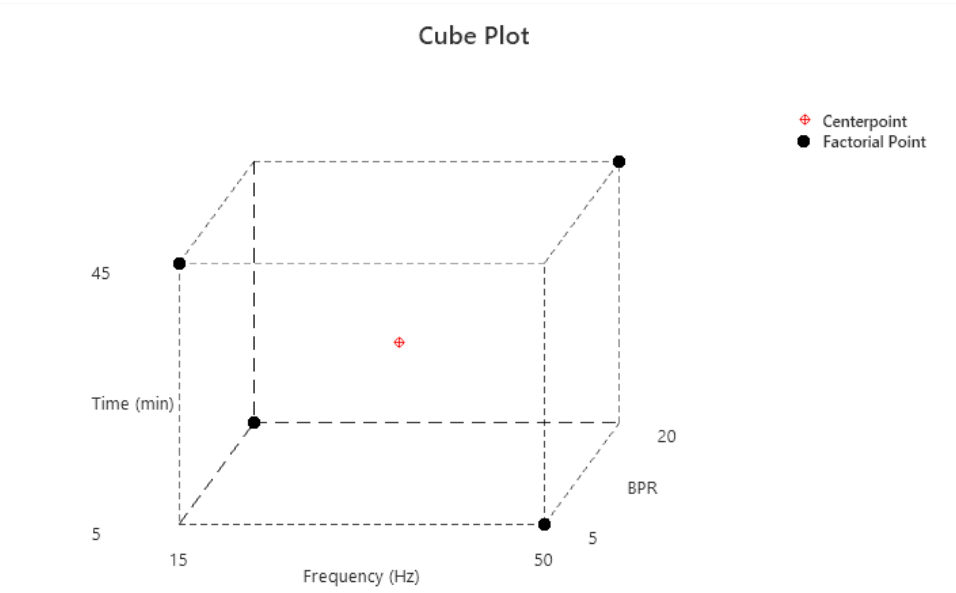

Figure S2. Graphical representation of the 3D space generated by the three variables, including the central point (in red). For comparison, the OVAT method would start from one point and then move first in one direction, following one axis, and then in the other directions starting from the optimum of each axis, progressively. This approach usually requires a higher and unplanned number of experiments [1], without necessarily reaching the optimum and hindering extrapolation of the effect of each parameter on the final result.

Table S1. Methane conversion at 700 °C (steam-to-carbon = 2, F/W = 202 500 mL/g_{cat} h), NiO crystallite size inferred by XRD measurements, and normalized Raman intensity of the signal at 570 cm^{-1} (typical of Ni-O-Ce bonds and oxygen vacancies [2]) for the samples prepared with the Fractional Factorial design.

Sample	X_{CH4} at 700 °C	NiO size (nm)	Raman intensity (a.u.)
1-PtNiCe(--+)	21.8%	19.8	2.0
2-PtNiCe(-+-)	44.8%	22.9	2.5
3-PtNiCe(+--)	60.2%	14.6	4.4
4-PtNiCe(000)	59.9%	13.6	7.3
5-PtNiCe(+++)	83.5%	12.0	7.1

References

1. Leardi, R. Experimental Design in Chemistry: A Tutorial. *Analytica Chimica Acta* **2009**, *652*, 161–172, doi:10.1016/j.aca.2009.06.015.
2. Liu, Y.-M.; Wang, L.-C.; Chen, M.; Xu, J.; Cao, Y.; He, H.-Y.; Fan, K.-N. Highly Selective Ce–Ni–O Catalysts for Efficient Low Temperature Oxidative Dehydrogenation of Propane. *Catal Lett* **2009**, *130*, 350–354, doi:10.1007/s10562-009-9977-z.