Supporting Information (SI) for

Efficient and environmentally friendly microwave-assisted synthesis of magnetic metallic Ni nanoparticles

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Contents:

- Pages: 7 (S1-S7)
- Figures: 2 (Figure S1; Figure S2)
- Tables: 3 (Table S1; Table S2; Table S3)

S0 - Experimental Section

All the reagents were purchased from Sigma-Aldrich Inc, and were used without any further purification.

PXRD patterns were recorded using a Bruker D8 DISCOVER A25 diffractometer with a slit of 0.018° from 2θ=10° to 80° and counting time of 5 s per step using CuKα radiation. Textural properties of the samples were determined by N2 physisorption using a Micromeritics ASAP 2020 automated system with the Brunauer-Emmet-Teller (BET) and the Barret-Joyner-Halenda (BJH) methods. The samples were outgassed for 24 h at 130°C under vacuum (P0 = 10−2 Pa) and subsequently analysed. SEM images were recorded with a JEOL JSM-6300 scanning microscope equipped with Energy-dispersive X-ray spectroscopy (EDS) at 20 kV. TEM images were recorded with JEOL 1200, at the Research Support Service Center (SCAI) from University of Cordoba. Prior to the recording, samples were prepared by suspension in EtOH, assisted by sonication and followed by deposition on a copper grid. The magnetic susceptibility was evaluated at room temperature and at low frequency (470 Hz), employing a Bartington MS-2 instrument. The reaction products were identified using GC (7890A)-MS (5975D inert MSD with Triple-Axis Detector) Agilent equipped with a capillary column HP-5MS (60 m x 0.32 mm), at the Research Support Service Center (SCAI) from University of Cordoba. Helium was used as a carrier gas. Quantitatively analysis were determined using GC (Agilent 7890 A) employing a FID and a PetrolTM DH column (100 m×0.25 mm×0.50 μm). The carrier gas used was N2.

CEM-Discover (CEM Corporation Matthews, NC, USA) microwave reactor was employed to perform the synthesis of the MMN and the hydrogenolysis of BFE. For the synthesis of MMN, the metallic precursor NiCl2, EtOH and EG were added in a 10 mL microwave tube at room temperature. The mixture was irradiated with microwaves at different times between 5-60 mins in the 180-250°C temperature range using a temp. ramp of 1 minute. The precipitate was recovered with a magnet, washed with ethanol (5 mL) and acetone (5 mL) and oven dried (100°C). For the hydrogenolysis of BFE, BFE (25 mg) were added to isopropanol (0,5 mL) and catalysts (7% wt). The reaction was irradiated with microwaves at different times between 5-45 mins at 230°C using a temp. ramp of 1 minute.

Table S1 - EDS analysis of the surface of MWs metallic Nickel

Element	Wt%	Wt% Sigma	Atomic %
C	3.72	0.17	15.69
O	0.45	0.04	1.41
Cl	0.19	0.02	0.27
Ni	95.65	0.17	82.63
Total:	100.00		100.00

Figure S1 - Image of the magnetic metallic Nickel precipitate

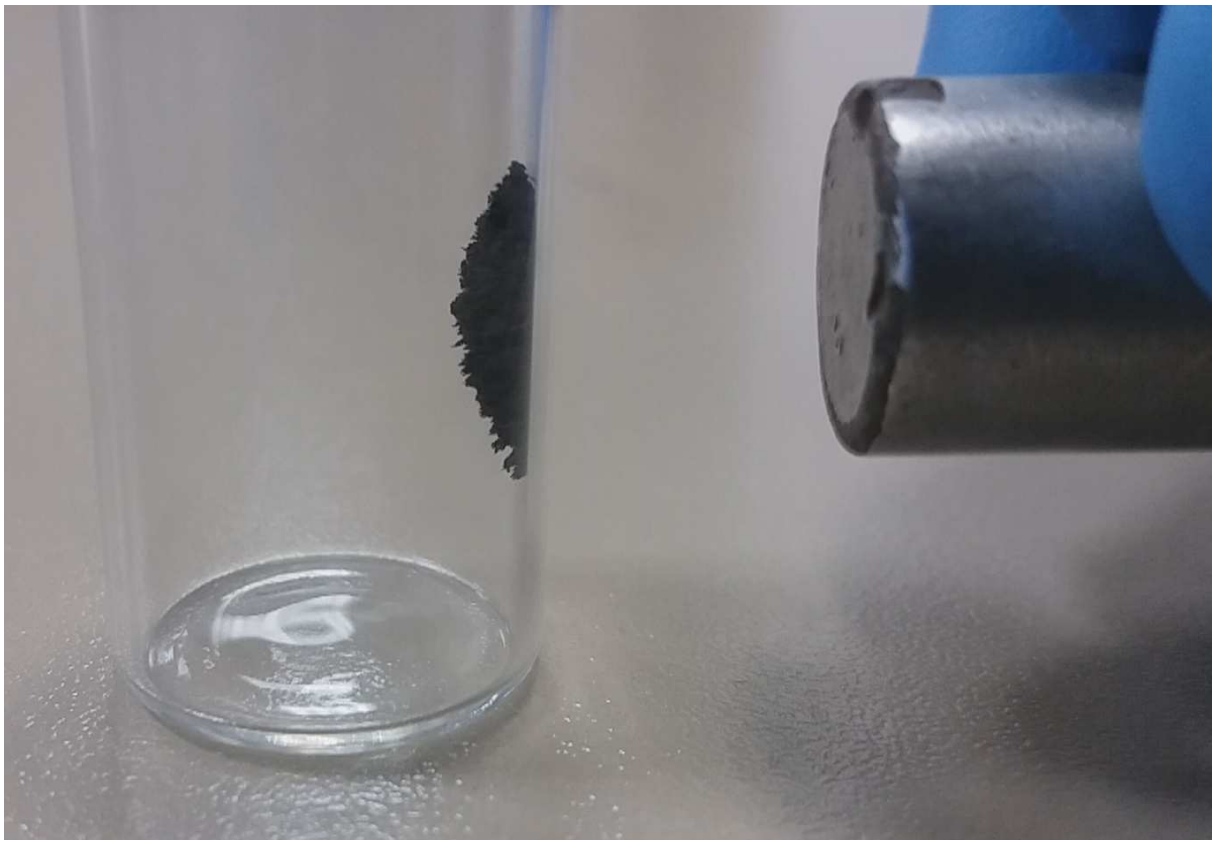

Table S2 - Most relevant trials

[Ni2+] (mol L-1)	Reaction time (min)	Temperature (°C)	Mixture composition (EG Xi)	Yield (%)
Increasing reaction time				
0,031	**1**	200	0,904	0
0,031	**3**	200	0,904	0
0,031	**5**	200	0,904	undetectable*
0,031	**10**	200	0,904	undetectable*
0,031	**30**	200	0,904	22
0,031	**20**	200	0,904	24
0,031	**60**	200	0,904	30
Varying mixture composition				
0,031	5	150	**0,000**	0
0,031	5	150	**0,610**	0
0,031	5	200	**0,807**	0
0,031	5	200	**0,846**	20
0,031	5	200	**0,865**	9
0,031	5	200	**0,884**	undetectable*
0,031	5	200	**0,904**	undetectable*
0,309	5	200	**0,923**	undetectable*
0,031	5	200	**0,962**	undetectable*
0,031	5	200	**0,970**	undetectable*
0,031	5	200	**0,981**	0
0,031	5	200	**1,000**	0
0,031	5	230	**1,000**	0
Increasing reaction temperature				
0,031	5	**150**	0,904	0
0,310	5	**170**	0,904	0
0,031	5	**180**	0,904	undetectable*
0,031	5	**200**	0,904	undetectable*
0,031	5	**220**	0,904	26
0,031	5	**230**	0,904	34
0,031	5	**240**	0,904	44
0,031	5	**250**	0,904	71
Increasing metal precursor concentration				
0,008	20	210	0,904	undetectable*
0,017	20	210	0,904	undetectable*
0,023	20	210	0,904	undetectable*
0,031	20	210	0,904	undetectable*
0,072	20	210	0,904	8
0,110	20	210	0,904	6
0,182	20	210	0,904	4
0,312	20	210	0,904	0
0,471	20	210	0,904	0
0,772	20	230	0,904	0

*the precipitate consisted of a fine magnetic powder, which was impossible to properly weight

Table S2 Most relevant trials for the outlining of the parameters combination for the reaction occurrence

Figure S2 - GC-MS analysis of 1,3-Dioxolane-2-methanol

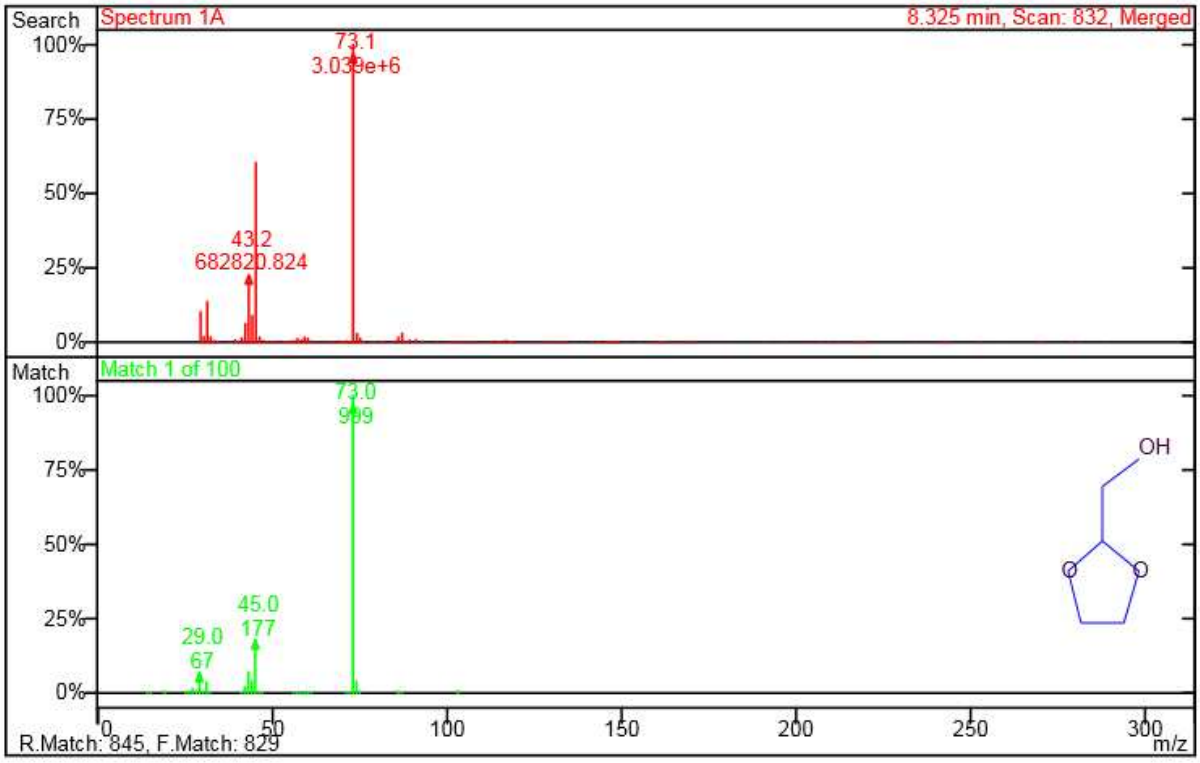

Table S3 - BPE hydrogenolysis with MWs Metallic Ni and commercially available 5% Pd/C

Catalyst	Time (min)	BFE conversion (%)	Toluene selectivity (%)	Phenol selectivity (%)
MMN	5	2	12	40
MMN	15	12	18	46
MMN	30	17	19	44
MMN	**45**	**24**	**15**	**44**
5%Pd/C	**45**	**43**	**24**	**56**
MMN*	**45**	**21**	**22**	**43**

* reused 5 cycles