

## Exploiting nickel-organic-frameworks in the temperature-controlled synthesis of carbonized nickel nanoparticles for magnetic purification of acidic water

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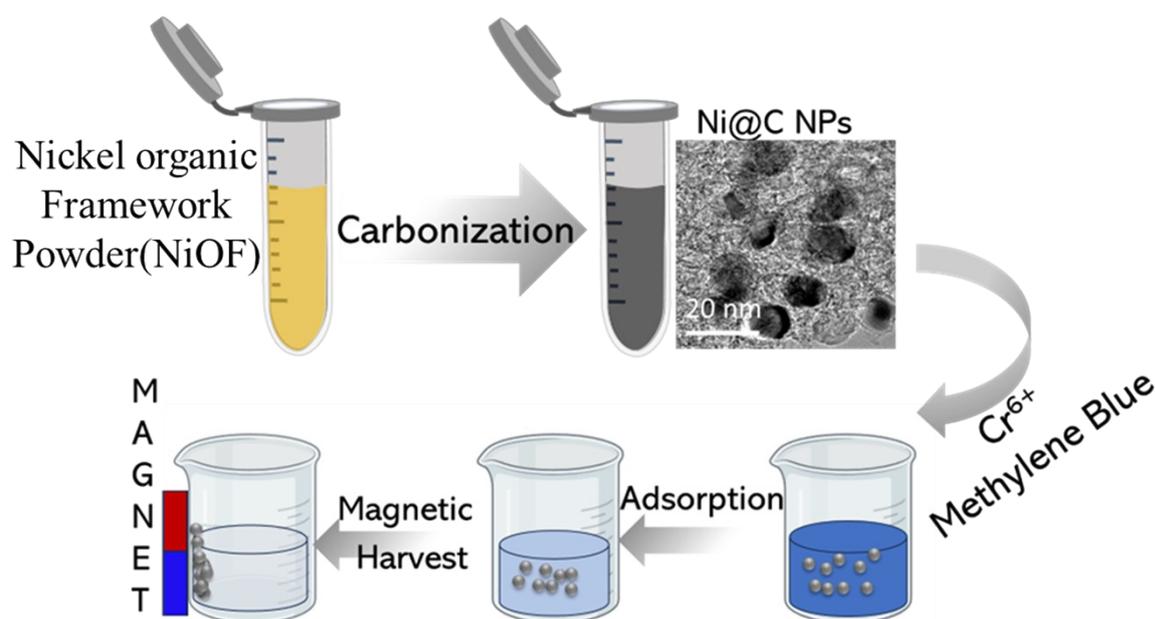
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### Graphical abstract

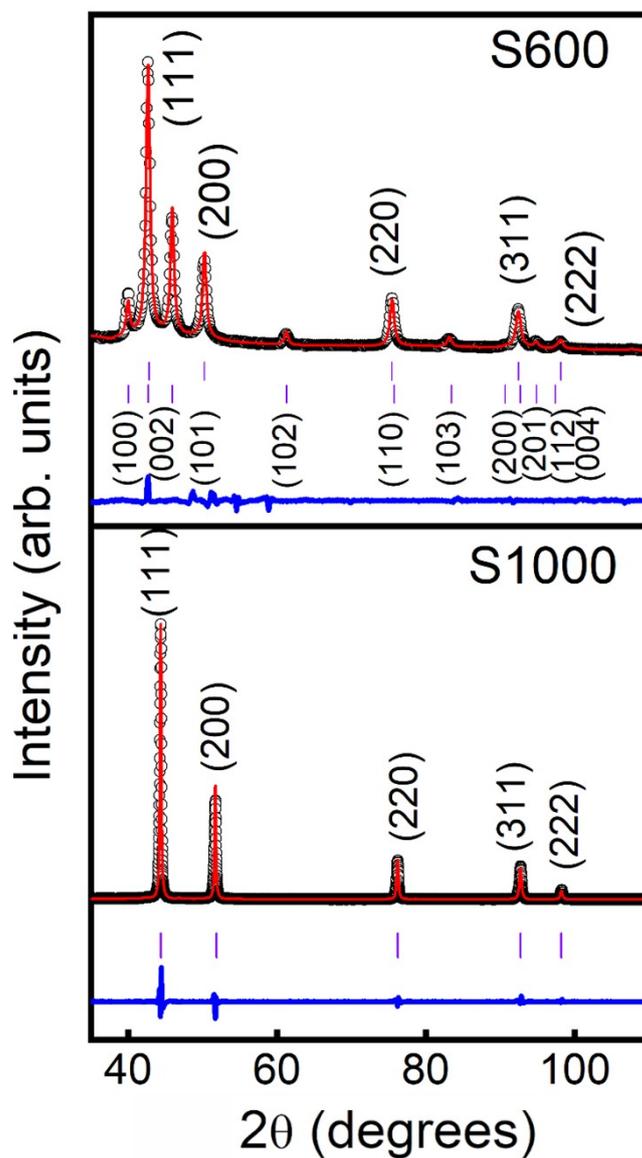


## Supporting Information

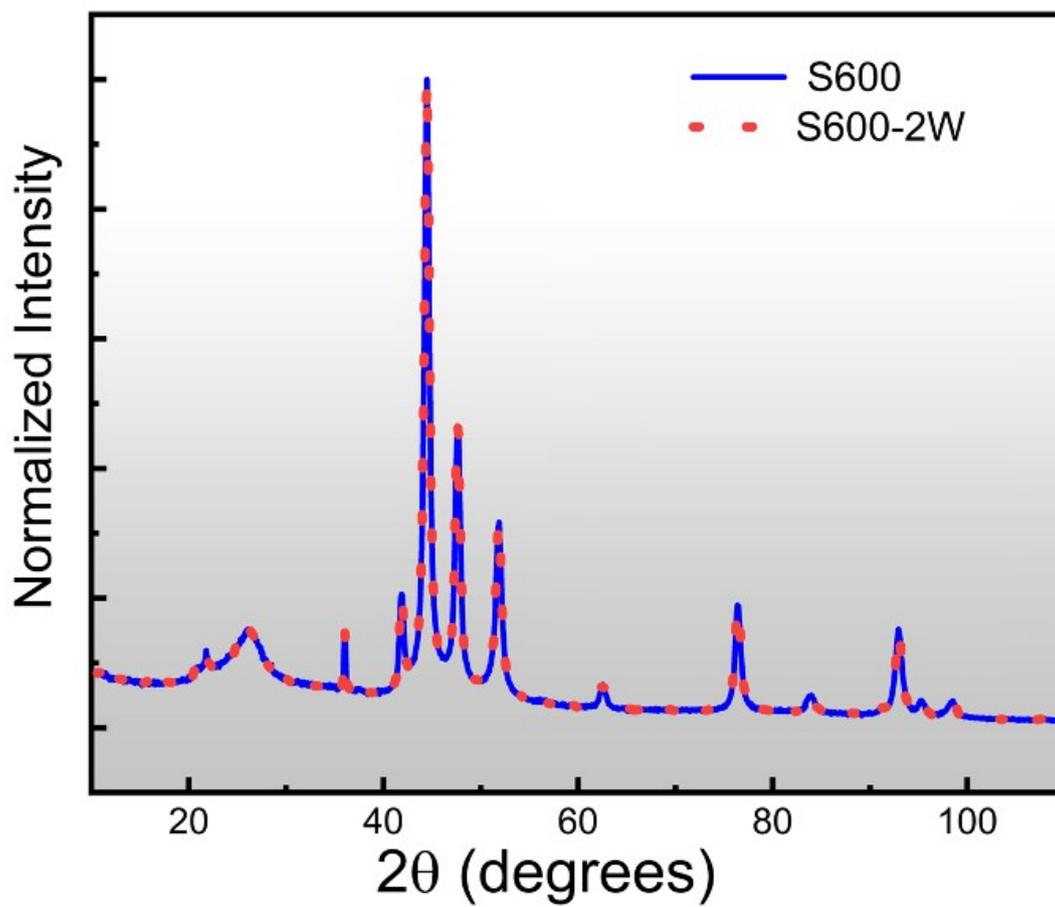
### S.1. Nanoparticle synthesis

In the first step, 20 g of 2-mIm and 10 g of  $\text{Ni}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$  were dissolved separately in 50 mL of distilled water each. The two solutions were preheated to 95 °C while stirring and purged with an argon flow to prevent surface oxidation of the resulting material. The  $\text{Ni}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$  solution was then carefully introduced into the 2-mIm solution, initiating an immediate reaction, which was visually confirmed by a change in color from green to yellow. After vigorous stirring at 95 °C for 30 minutes, the reaction was halted by cooling the solution in an ice bath. The resulting precipitate was collected by centrifugation at a rotation speed of 2500 rpm and washed with distilled water eight times until the supernatant showed no further discoloration. The samples were allowed to settle for two days in water and dried on the third day under vacuum at 200 °C for 6 hours to remove any remaining unreacted 2-mIm molecules that might have blocked the pores of the material. Finally, in the second step, NiOF samples were subjected to a carbonization process by heating in a horizontal tubular furnace under an inert atmosphere at two different final temperatures: 600 °C and 1000 °C, resulting in black powdered samples designated as S600 and S1000, respectively.

## S.2. Crystal structure

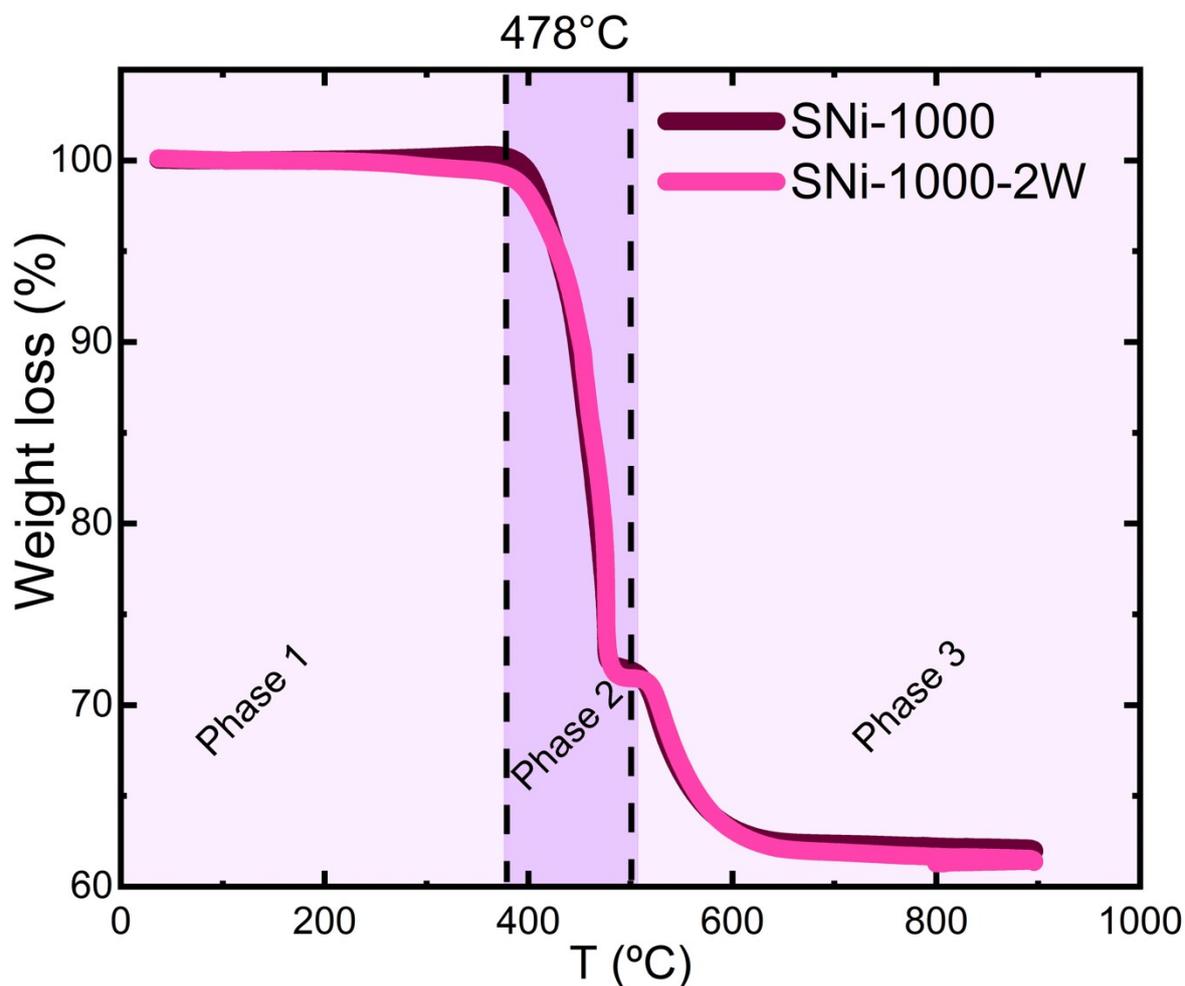


**Figure S1.** X-ray powder diffraction patterns of the investigated samples S600 and S1000 were measured at room temperature. Black open circles and red solid lines represent the experimental data and the corresponding fit, respectively. A solid blue line at the bottom represents the difference between the experimental data and the theoretical models. The first series of vertical bars corresponds to the positions of the Bragg reflections associated with the crystal structure of Ni-FCC, and the second one with that of Ni-HCP.



**Figure S2.** X-ray powder diffraction patterns of the investigated samples SNi-600 and SNi-600-2W measured at room temperature, normalized to the maximum intensity of the Ni (111) peak at 44.5°.

### S.3. Thermogravimetric analysis (TGA)



**Figure S3.** Thermogravimetric curve of the samples S1000 and S1000-2W using air-drying methods.

TGA was used to examine the thermal degradation, desorption, and evaporation of organic compounds present on the NP surface, revealing three distinct stages (see Figure S3). The initial stage involves gradual weight loss due to the decomposition of organic components or the removal of surface-bound carbonaceous residues. This process is typically endothermic, involving the breakdown of carbon structures with minimal oxidation. In the second stage, an exothermic reaction occurs, most likely due to the combustion of the carbon coating of the Ni NPs, releasing energy as carbon oxidizes to  $\text{CO}_2$ . Finally, the third stage corresponds to the oxidation of the exposed Ni NPs, resulting in the formation of NiO and a subsequent weight gain. The molar mass of Ni and O was used to determine the nickel proportion in NiO.

### Mass of Nickel:

The weight gain in Phase 2 corresponds to the formation of NiO. Ni and O's molecular weights were used to find the nickel proportion in NiO. The molecular weight of NiO is approximately 74.69 g/mol (Ni: 58.69 g/mol, O: 16 g/mol). The mass percentage of Ni in NiO is given by:

$$\text{Ni wt.}\% = \left( \frac{\text{Residue wt.}\% \times \text{Molar mass of Ni}}{\text{Molar mass of NiO}} \right)$$

### S.4. Chromium and Methylene Blue Adsorption Experiments.

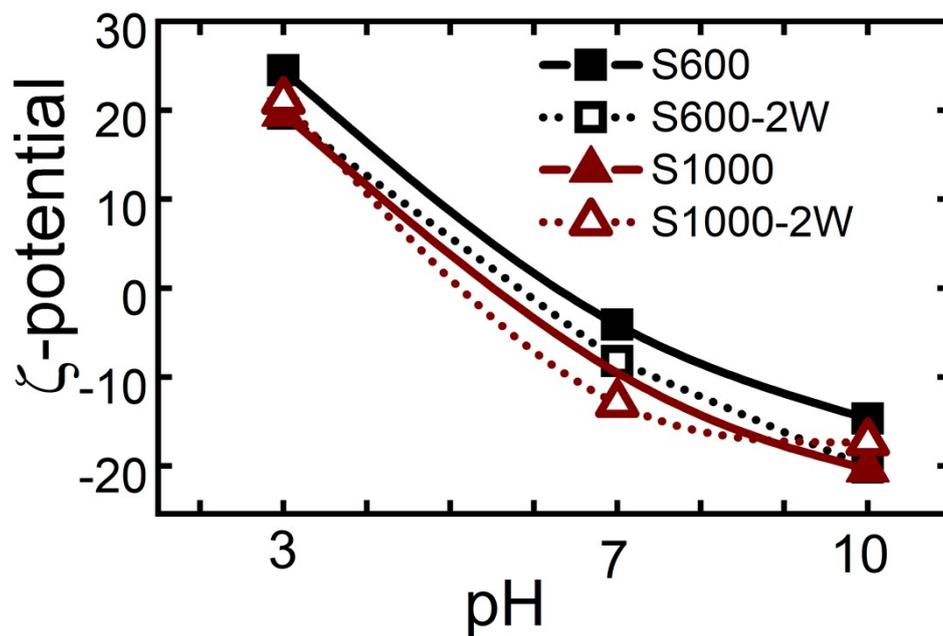


Figure S4. ζ-potential for samples at different pH levels.

## S.5. Comparison with previous works.

**Table S1.** Maximum adsorption capacities of Cr(VI) and MB on various adsorbents.

Adsorbent	Pollutant	BET (m <sup>2</sup> /g)	pH	q <sub>max</sub> (mg/g)	Reference
Active Carbon (AC)	MB	870	4	330	1
Fe (AC)	MB	940	4	357	1
Fe <sub>3</sub> O <sub>4</sub> -AHSO	MB	---	3	7.8	2
Discarded Post Ro-carbon (RO900)	MB	---		223	3
Peanut husk (MPN-cite)	MB	0.443	5.4	245	4
S600-2W	MB	328	3	54	THIS WORK
S1000-2W	MB	213	3	95	THIS WORK
Fe <sub>3</sub> O <sub>4</sub> -SiO <sub>2</sub>	Cr(VI)	16	2	13	5
Fe <sub>3</sub> O <sub>4</sub> @AC	Cr(VI)	51	2	5.5	6
MnFe <sub>2</sub> O <sub>4</sub> /chitosan	Cr(VI)	---	5	35	7
Carbon-encapsulated MnFe <sub>2</sub> O <sub>4</sub>	Cr(VI)	---	2	73	8
S600-2W	Cr(VI)	328	3	36	THIS WORK
S1000-2W	Cr(VI)	213	3	29	THIS WORK

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