

ELECTRONIC SUPPLEMENTARY INFORMATION

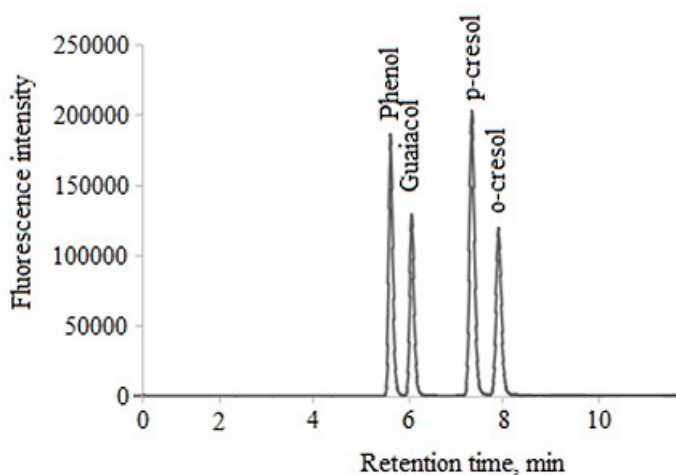
to the paper

**Magnetic headspace adsorptive microextraction using  $\text{Fe}_3\text{O}_4@\text{Cr}(\text{OH})_3$  nanoparticles for effective determination of volatile phenols**

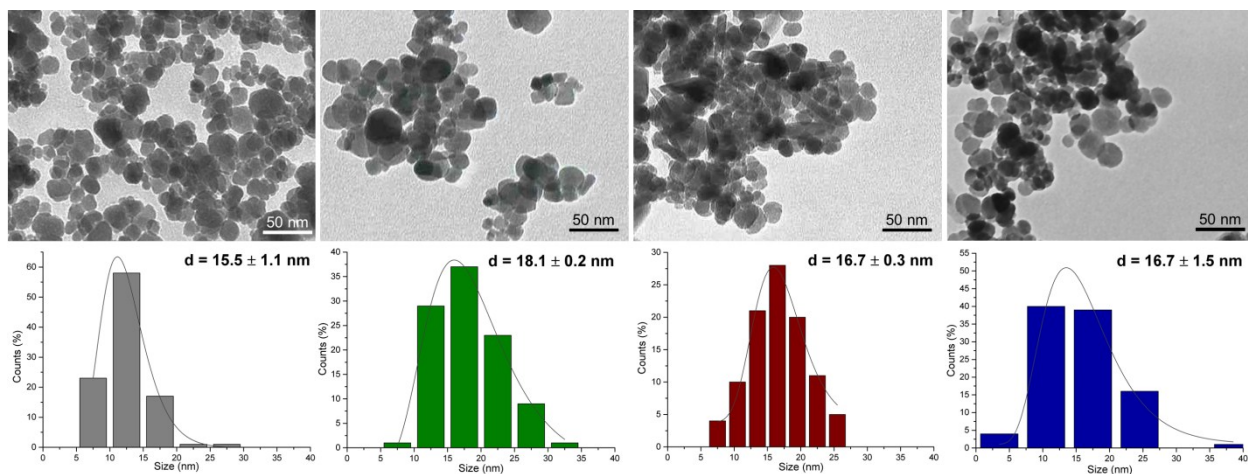
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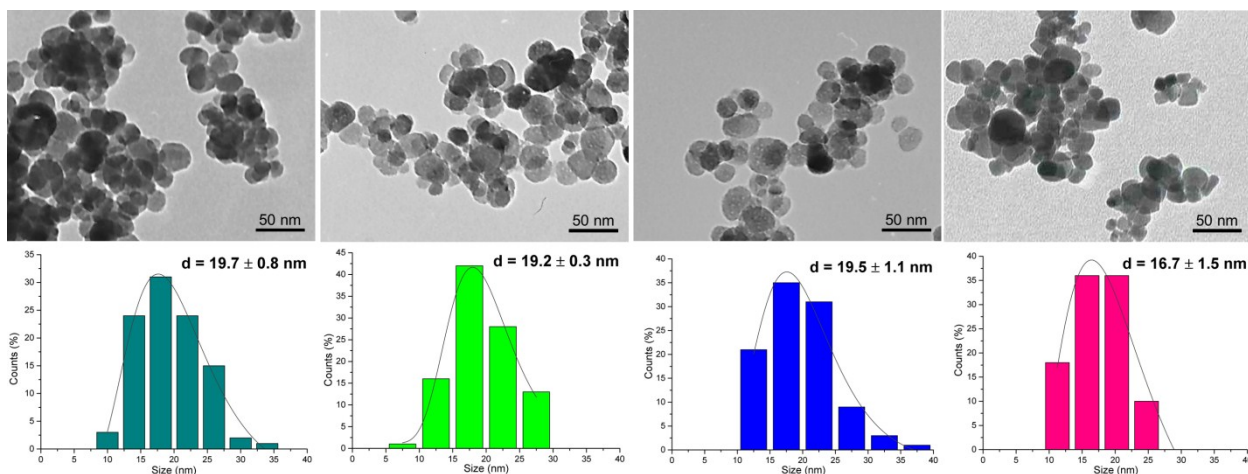
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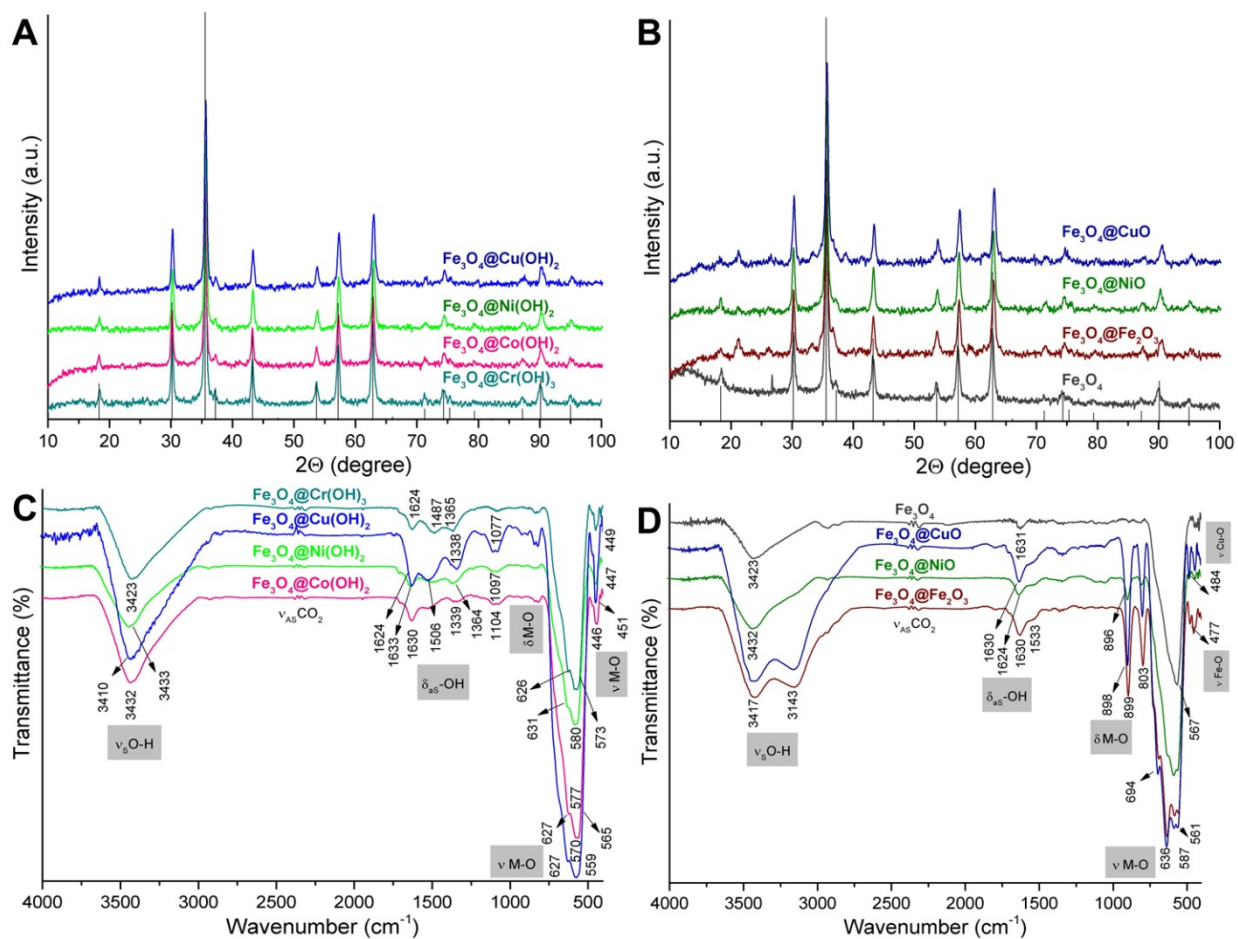
**ESI Fig. 1.** Chromatogram of phenols' solution with concentration of each analyte equal to 1 mg L<sup>-1</sup> (mobile phase: acetonitrile and 0.1 % HCOOH (40:60, v/v), flow-rate of 1 mL min<sup>-1</sup>, an excitation wavelength of 270 nm and emission wavelength of 310 nm).



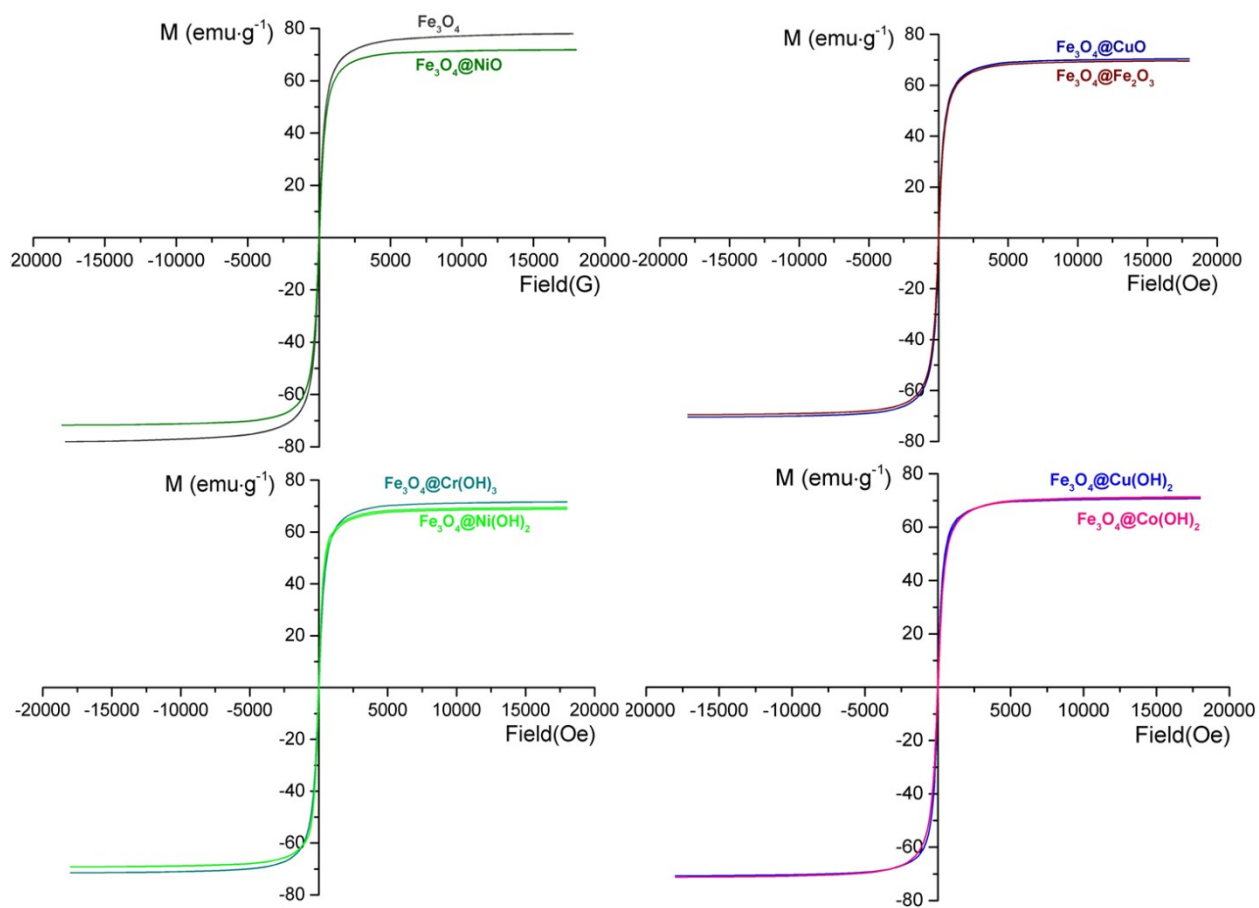
**ESI Fig. 2.A.** TEM images and size distribution of as-prepared  $\text{Fe}_3\text{O}_4$ ,  $\text{Fe}_3\text{O}_4@NiO$ ,  $\text{Fe}_3\text{O}_4@Fe_2O_3$  and  $\text{Fe}_3\text{O}_4@CuO$  (from left to right).



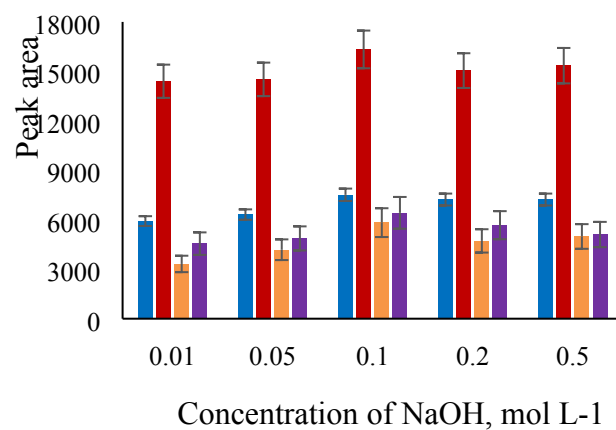
**ESI Fig. 2.B.** TEM images and size distribution of as-prepared  $\text{Fe}_3\text{O}_4@Cr(OH)_3$ ,  $\text{Fe}_3\text{O}_4@Ni(OH)_2$ ,  $\text{Fe}_3\text{O}_4@Cu(OH)_2$ ,  $\text{Fe}_3\text{O}_4@Co(OH)_2$  (from left to right).



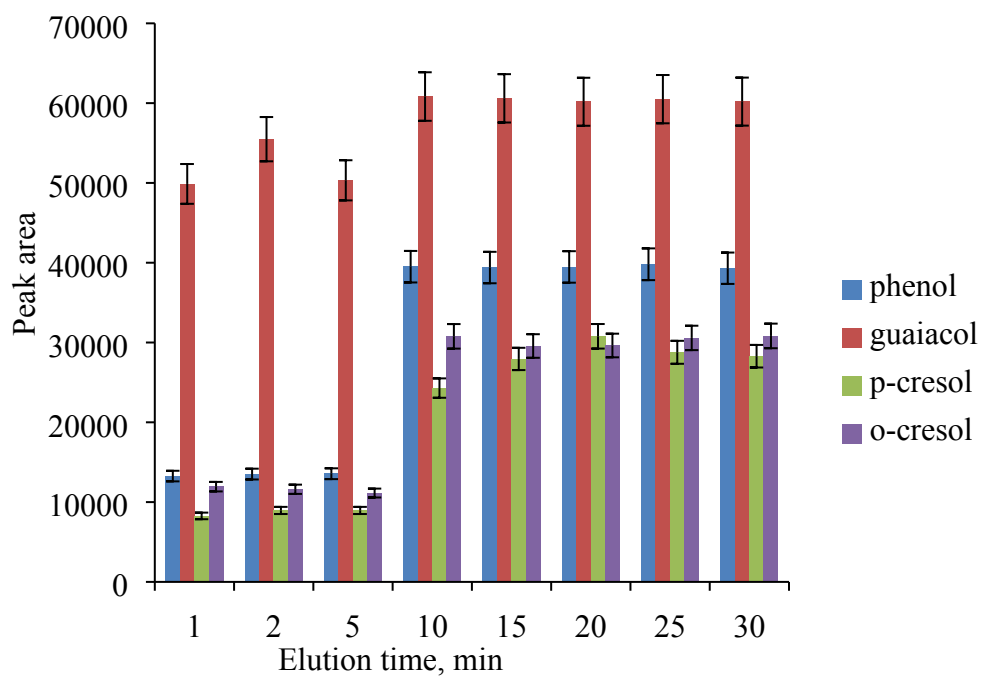
ESI Fig. 3. XRD pattern (A and B) and FTIR data (C and D) of MNPs.



**ESI Fig. 4.** VSM data of MNPs.



**ESI Fig. 5.** Effect of NaOH concentration ( $C(\text{phenols}) = 0.5 \text{ mg kg}^{-1}$ ; 10 mg of  $\text{Fe}_3\text{O}_4@\text{Cr}(\text{OH})_3$ ; 90 °C; extraction time – 10 min; elution time – 30 min; NaOH volume – 0.5 mL).



**ESI Fig. 6.** Effect of elution time (0.1 mL of 0.1 mol L<sup>-1</sup> NaOH; concentration of each phenol in sample – 0.5 mg kg<sup>-1</sup>).

**ESI Table.** Comparison of the developed and previously reported methods for the determination of phenols in food samples.

Sample	Volume/ mass of sample	Analytes	Extraction technique	Method of detection	LOD	Linear range	RSD,%	Recovery, %	Extraction time, min	Ref.
smoked sausage	1 g	guaiacol, 4-methylguaiacol, syringol, eugenol, trans-isoeugenol	SPE	GC-MS	0.1 $\mu\text{g kg}^{-1}$	-	<20	91-113	-	1
smoked herring	2 g	phenol, p-cresol, o-cresol, guaiacol, 4-methyl guaiacol, 4-ethyl guaiacol, syringol, eugenol, 4-propyl guaiacol, isoeugenol, 2- chlorophenol	SPME	GC-FID	1 $\text{mg kg}^{-1}$	-	5.45 - 8.70	-	55 min	2
smoked sausages	0.25 g	phenol, o-cresol, m-cresol, p-cresol, isoeugenol, guaiacol	USLE-GD	FI-CL, HPLC- FLD	0.01 $\text{mg kg}^{-1}$	0.038–40 $\text{mg kg}^{-1}$	4-6	-	25 min	3
smoked sausage, fish	0.2 g	phenol, o-cresols, p-cresols eugenol, isoeugenol, guaiacol	DEM-MME	HPLC- FLD	0.3 – 1.0 $\mu\text{g kg}^{-1}$	1-5000 $\mu\text{g kg}^{-1}$	4.5-8.0	-	17 min	4
milk	5 mL	guaiacol, eugenol, phenol, 2-ethylphenol	Vac- HSSPME	GC-FID	0.14-13 $\mu\text{g L}^{-1}$	1–1000 $\mu\text{g L}^{-1}$	0.3-10	-	20 min	5
smoked sausages	0.5 g	phenol, guaiacol, p-cresol, o-cresol	MHS-AME	HPLC- FLD	0.2 $\mu\text{g kg}^{-1}$	0.5 - 2500 $\mu\text{g kg}^{-1}$	1-8	90-118	20 min	This work

**SPE - solid-phase extraction**  
**SPME - solid-phase microextraction**  
**USLE-GD – ultrasound assisted solid-liquid extraction with GD separation**  
**FI-CL – flow injection chemiluminescence**  
**DEM-MME – deep eutectic mixture membrane-based microextraction**  
**Vac-HSSPME - vacuum headspace solid-phase microextraction**  
**MHS-AME - magnetic headspace adsorptive microextraction**

1 M. Pöhlmann, A. Hitzel, F. Schwägele, K. Speer, W. Jira, *Meat Science*, 2012, **90** (1), 176-184.

2 T. Sérot, C. Lafficher. Optimisation of solid-phase microextraction coupled to gas chromatography for determination of phenolic compounds in smoked herring. *Food Chemistry*, 2003, **82**(4), 513–519.

3 C. Vakh, E. Evdokimova, A. Pochivalov, L. Moskvina, A. Bulatov, *Food Chemistry*, 2017, **237**, 929-935.

4 A. Shishov, S. Gagarionova, A. Bulatov. *Food Chemistry*, 2020, 126097. DOI: 10.1016/j.foodchem.2019.126097

5 M.J. Trujillo-Rodríguez, V. Pino, E. Psillakis, J.L. Anderson, J.H. Ayala, E. Yiantzi, A.M. Afonso, *Analytica Chimica Acta*, 2017, **962**, 41-51.