



## Article

# Effect of Oleylamine on the Surface Chemistry, Morphology, Electronic Structure, and Magnetic Properties of Cobalt Ferrite Nanoparticles

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## 1. EDS

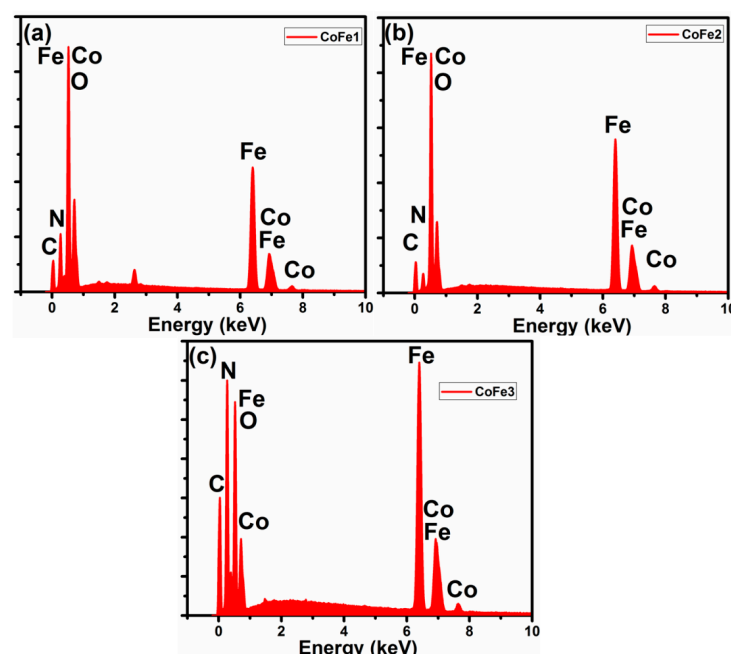
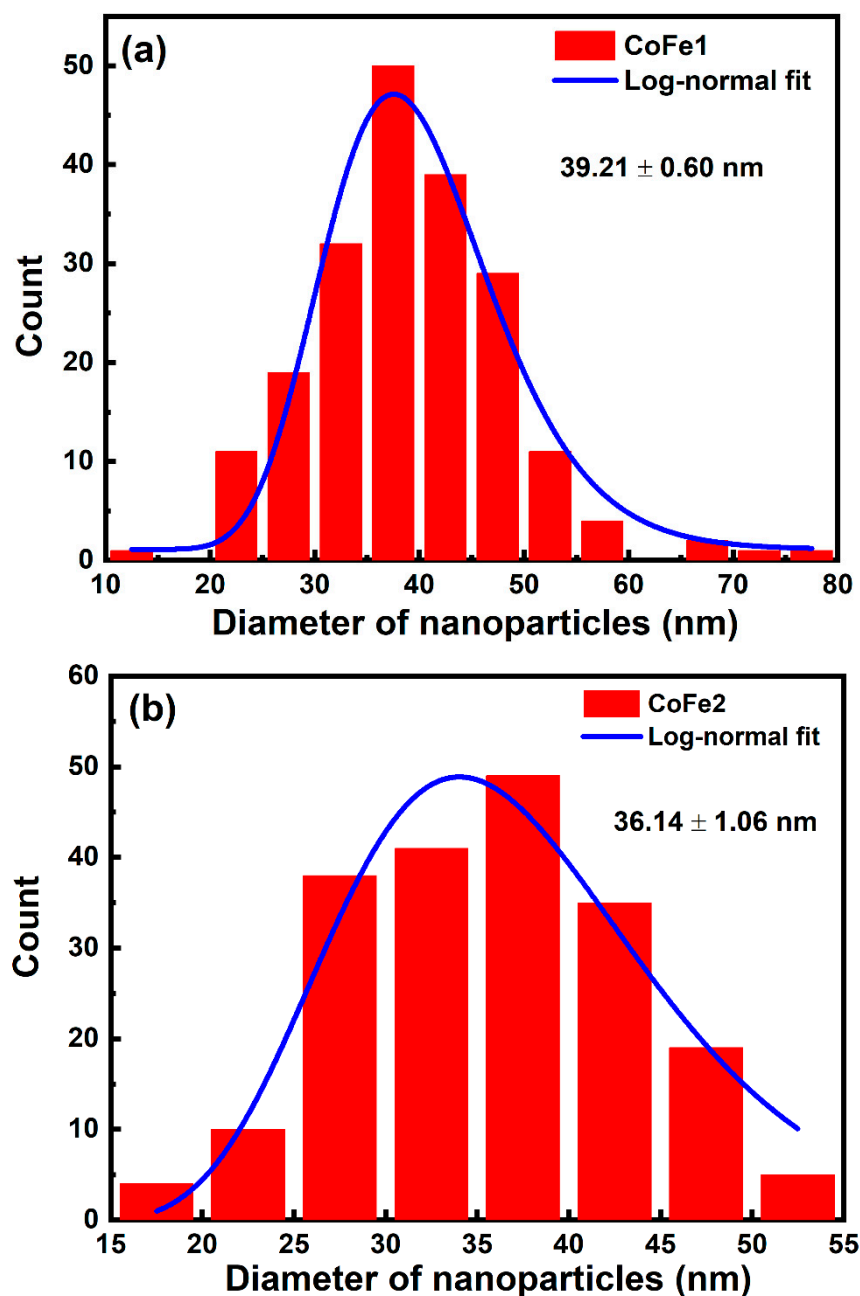


Figure S1. Energy dispersive X-ray spectroscopy (EDS) patterns for CoFe1 (a), CoFe2 (b) and CoFe3 (c) samples.

## 2. SEM

The particle size distribution is calculated from a FESEM micrograph by plotting the histogram and fitted with a log-normal function.



**Figure S2.** Histogram for CoFe1 (a), and CoFe2 (b) samples along with log-normal function showing the size distribution of nanoparticles.

### 3. SAXS Analysis

Small-angle X-ray scattering (SAXS) measurements were carried out using a Rigaku small-angle goniometer mounted on a rotating anode X-ray generator. Scattered X-ray intensity  $I(q)$  was recorded using a scintillation counter with a pulse height analyzer by varying the scattering angle  $2q$  where  $q$  is the scattering vector given by  $4\pi \sin(q)/\lambda$  and  $\lambda$  is the wavelength of incident (Cu  $K\alpha$ ) X-rays. The intensities were corrected for sample absorption and smearing effects of collimating slits [1].

**Table S1.** Structural parameters obtained from SAXS.

Parameter	Samples	CoFe1	CoFe2	CoFe3
Outer radius ( $R_1$ , nm)		5.48	4.00	3.05
Inner radius ( $R_2$ , nm)		3.5	1.5	1.0
The polydispersity index ( $\sigma$ , nm)		0.36	0.31	0.29

The radius of the monomer ( $r_0$ , nm)	5.61	3.49	2.17
Size of aggregate ( $x_i$ , nm)	11.77	13.52	27.69
Fractal dimension ( $D$ )	3.00	3.00	3.00

#### 4. FTIR

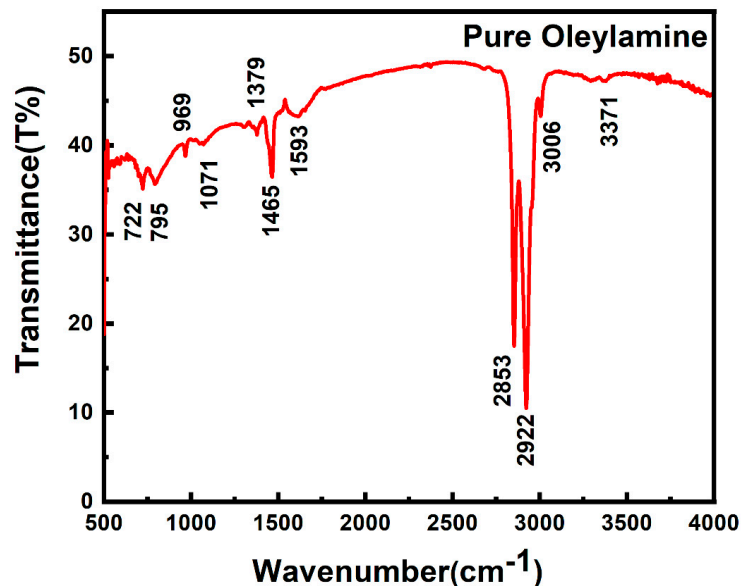


Figure S3. FTIR spectra of pure oleylamine.

Table S2. Infrared vibrational assignments for the pure oleylamine.

Vibrational modes	Frequency ( $\text{cm}^{-1}$ )
$\nu_{\text{as}}(\text{NH}_2)$ and $\nu_{\text{s}}(\text{NH}_2)$	3376, 3300
$\delta(\text{=C-H})$	3006
$\nu_{\text{as}}(\text{C-H})$ and $\nu_{\text{s}}(\text{C-H})$	2922, 2854
$\delta(\text{-C=C})$	1647
$\delta(\text{NH}_2)$	1593, 795
$\delta(\text{CH}_3)$	1465
$\delta(\text{C-N})$	1071
$\delta(\text{C-C})$	722
$\nu_{\text{as}}$ — asymmetric stretching vibration; $\nu_{\text{s}}$ — symmetric stretching vibration; $\delta$ — Bending vibration	

#### 5. XRD

Table S3. Structural parameters obtained from Rietveld refinement of XRD.

Sample	Average crystallite size (nm)	Lattice parameter ( $a$ , Å)	X-ray density ( $d_{\text{X-ray}}$ ) ( $\text{gm}/\text{cm}^3$ )
CoFe1	19.18	8.3769	5.301
CoFe2	19.14	8.3719	5.311
CoFe3	14.88	8.3894	5.278

Table S4. Atomic position, occupancy, and agreement factors obtained for CoFe1, CoFe2, and CoFe3 from Rietveld Refinement.

Atom	Atomic position			Occupancy	Agreement factors
	x	y	z		
Sample CoFe1					

Co1	0.000	0.000	0.000	0.0300	$R_{wp}= 52.10$
Fe1	0.000	0.000	0.000	0.0533	$R_{exp}= 50.97$
Co2	0.625	0.625	0.625	0.0133	$R_B= 1.186$
Fe2	0.625	0.625	0.625	0.0283	$S = 1.0221$
O1	0.247	0.247	0.247	0.1785	$\chi^2= 1.04$
Sample CoFe2					
Co1	0.000	0.000	0.000	0.0300	$R_{wp}= 63.70$
Fe1	0.000	0.000	0.000	0.0533	$R_{exp}= 63.26$
Co2	0.625	0.625	0.625	0.0133	$R_B= 1.142$
Fe2	0.625	0.625	0.625	0.0283	$S = 1.006$
O1	0.240	0.240	0.240	0.1983	$\chi^2= 1.01$
Sample CoFe3					
Co1	0.000	0.000	0.000	0.033	$R_{wp}= 16.70$
Fe1	0.000	0.000	0.000	0.053	$R_{exp}= 15.98$
Co2	0.625	0.625	0.625	0.013	$R_B= 1.243$
Fe2	0.625	0.625	0.625	0.028	$S = 1.045$
O1	0.245	0.245	0.245	0.1769	$\chi^2= 1.10$

$R_{wp}$ : weighted profile factor,  $R_{exp}$ : expected weighted profile factor,  $R_B$ : Bragg factor,.

$S$ : Goodness of fit =  $R_{wp}/R_{exp}$ , reduced chi-square: ( $\chi^2$ ).

## 6. XPS

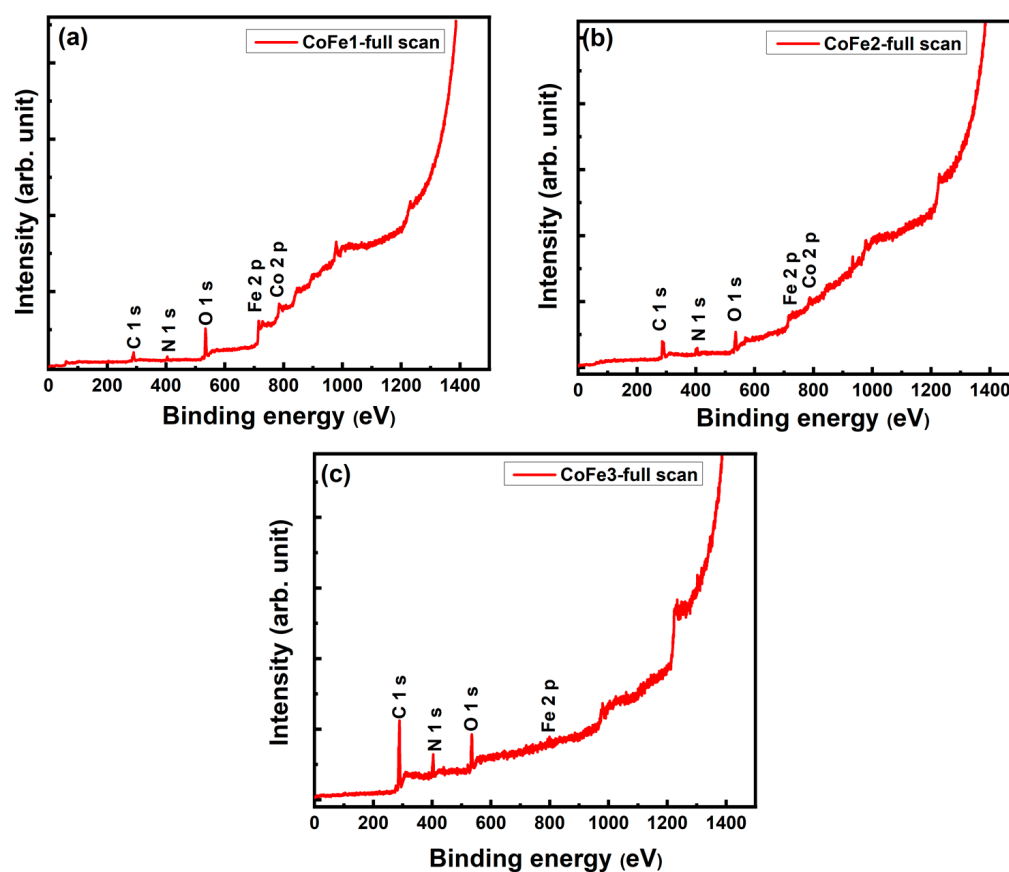


Figure S4. XPS survey spectra of CFO NPs.

## 7. Thermal Behavior - Thermogravimetric (TGA) Analysis

The number of OLA molecules per particle can be determined using the following formula [2] based on the weight loss.

$$N = \frac{W N_0 \rho (4/3) \pi R^3 \times 10^{-23}}{M} \quad (1)$$

Where  $N$  is the number of surfactant molecules (ligands) per particle,  $W$  is the weight loss in percent (%),  $N_0$  is Avogadro's number,  $\rho$  is the density of NPs,  $R$  is the average radius of the CFO NPs (obtained from SAXS analysis), and  $M$  is the molecular weight of OLA (267.49 g/mol).

## References

1. Ahrenstorf, K.; Heller, H.; Kornowski, A.; Broekaert, J.A.C.; Weller, H. Nucleation and Growth Mechanism of  $\text{Ni}_x\text{Pt}_{1-x}$  Nanoparticles. *Adv. Funct. Mater.* **2008**, *18*, 3850–3856, doi:<https://doi.org/10.1002/adfm.200800642>.
2. Ansari, S.M.; Sinha, B.B.; Phase, D.; Sen, D.; Sastry, P.U.; Kolekar, Y.D.; Ramana, C.V. Particle Size, Morphology, and Chemical Composition Controlled  $\text{CoFe}_2\text{O}_4$  Nanoparticles with Tunable Magnetic Properties via Oleic Acid Based Solvothermal Synthesis for Application in Electronic Devices. *ACS Appl. Nano Mater.* **2019**, *2*, 1828–1843, doi:10.1021/acsanm.8b02009.