RESEARCH PAPER

The Stage Dependent Effect of Capping Agent Introduction in the Synthesis of Magnetite Nanoparticles

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ABSTRACT

In this paper, three techniques to obtain capped magnetite nanoparticles were compared. In the formation of magnetite nanoparticles via the co-precipitation route, capping agents were introduced pre-, simultaneously with, or post-addition of the precipitating agent, ammonia. The amino acids L-glutamine and L-glutamic acid were used as the capping agents. Characterization via TEM, pXRD, EDX, and magnetic analysis displayed that the stage of introduction affected the properties of the nanoparticles obtained. Confirmation of capping was performed by FTIR and X-ray photoelectron spectroscopy. TEM displayed that the post-addition method yielded nanoparticles with the narrowest size distributions, having attractive dispersity values. The pre- and simultaneously-introduced methods produced smaller nanoparticles but had relatively higher size distributions. Crystallite size determined from pXRD showed that the post-addition method had the highest crystallite size, even compared to the uncapped nanoparticles, while the pre-introduced were much less crystalline. From the magnetic studies, the post-introduction method was shown to yield the highest magnetic saturation values, even when taking magnetically dead layers into account. It was also shown that the simultaneous and pre-introduction methods yielded similar magnetic saturation values despite size differences.

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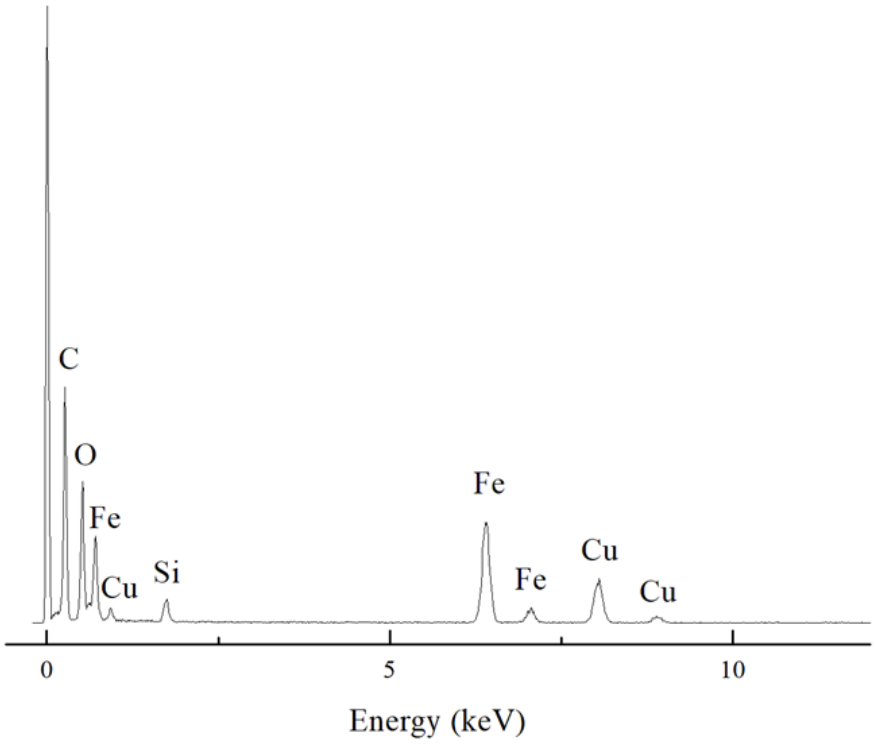

Fig. S1: EDX spectrum of the bare nanoparticles.

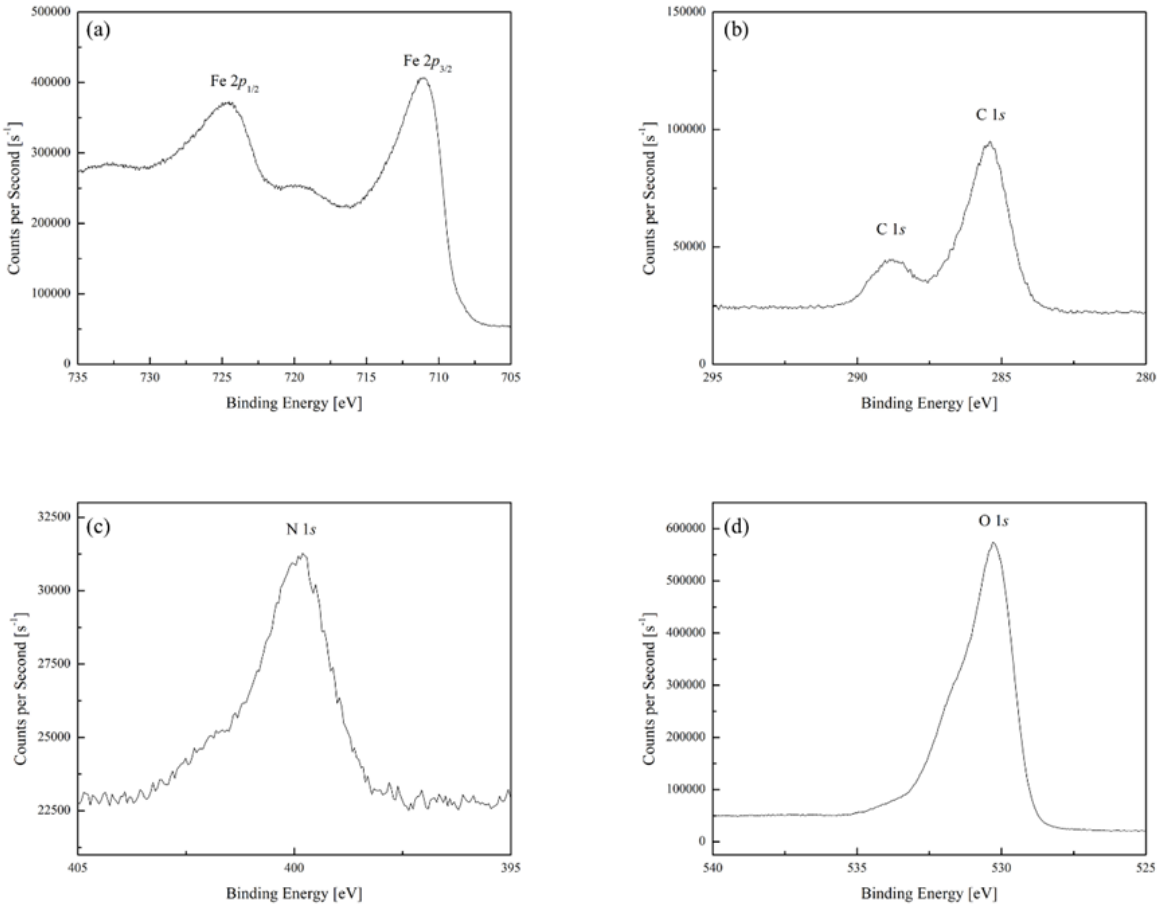

Fig. S2: High resolution XPS spectra of the iron (a), carbon (b), nitrogen (c) and oxygen (d) regions of the PostE nanoparticles

Table S1: Peak positions observed in FTIR spectroscopy for the bare and capped nanoparticles.

Assignment	Peak Position						
	Bare	PreQ	SimQ	PostQ	PreE	SimE	PostE
ν(O-H)	3352	3346	3323	3374	3328	3325	3330
δ(O-H)	1634	-	-	-	-	-	-
ν(-COO)	-	1632	1631	1634	1630	1615	-
δ(N-H)	-	-	-	-	-	-	1575
ν(-COO)$^{s.c.}$	-	-	-	-	-	-	1534
ν_s(-COO)	-	-	1392	1393	1394	1391	1401
ν(Fe-O)	541	544	542	545	543	543	556

s.c.: Amino acid side chain

Table S2: Binding energies of the Fe $2p_{3/2}$ and Fe $2p_{1/2}$ peaks.

Particle Type	Binding Energy (eV)	
	Fe $2p_{3/2}$	Fe $2p_{1/2}$
Bare	711.27	724.75
PreQ	709.23	722.67
SimQ	710.23	723.71
PostQ	710.99	724.23
PreE	709.03	722.79
SimE	710.75	724.11
PostE	710.95	724.51

Table S3: Binding energies of carbon, nitrogen, and oxygen 1 s peaks.

Particle Type	Binding Energy (eV)		
	C 1*s*	N 1*s*	O 1*s*
Bare	285.43; 289.07	-	530.43
PreQ	282.83; 285.67	397.62	527.67
SimQ	285.19; 289.07	399.98	530.03
PostQ	285.39; 289.15	399.97	530.11
PreE	282.67	397.89	527.51
SimE	285.43; 289.23	400.01	530.19
PostE	285.39; 288.87	400.04	530.31