Preparation of NanostructuredIron Oxide Particles and Their Surface Functionalization with Oleic Acid, 3-aminopropyltrimethoxysilane and Silver Nanoparticles

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Abstract

In this article, preparation of magnetic nanostructured iron oxide particles (IOs) and their surface functionalization with oleic acid, 3-Aminopropyltrimethoxysilane (APTMS) and Ag nanoparticles are reported. IOs were prepared by co-precipitation method and colloidal silver nanoparticles were synthesized by polyol method. The nanoscale IOs (<100nm) exhibited paramagnetic property and surface functionalization did not alter its magnetism. Colloidal stability of IOs particles is dependent on the nature of the surfactants. Necked IOs, andIOs functionalized with APTMS and Ag nanoparticles are polydispersein size while IOs functionalized with oleic acid are monodisperse. Such functionalized IOscan be employed in medical diagnostics, environmental remediation and energy harvesting technology.

Keywords: Co-precipitation; polyol method; Iron Oxide particles; silver nanoparticles; APTMS; oleic acid.

1. Introduction

Nanoparticles in the nanometer-size range have recently attracted the attention of researchers because of their unique physical and chemical properties[1]. Nano-sized materials, as compared to their bulk counterparts, exhibit new characteristic optical, electrical and magnetic properties due to the enhanced surface to volume ratio and quantum confinement effects emerging in these size ranges[2,3,4]. These new features of nanoparticles offer them the possibility to be used in a wide range of technological (magnetic data storage, sensor, refrigeration), environmental (catalysts,adsorbent, hydrogen storage), energy (lithium-ion batteries, solar cells) and biomedical applications[5].

Nanostructured Metal oxides possess unique properties that include wide bandgaps[6],high dielectric constants[7], reactive electronic transitions [8] and good electrical [9], optical [10] and electrochromic characteristics [11] as well as superconductivity [12]. Therefore, metal-oxides are some of the most fascinating functional materials and have been widely exploited in various technological applications.

Magnetic NPs are of great interest for researchers from a broad range of disciplines, includingcatalysis, magnetic fluids, data storage, and bioapplications, due to their unique magnetic properties such as superparamagnetism, high coercivity, low Curie temperature, high magnetic susceptibility, etc[13].

Iron oxide (IO) have received special attention because of their variety of scientific and technological applications such as catalysis[14] [16], adsorbent [15], antimicrobial activity,magnetic storage media, magnetic refrigeration, magnetic resonance imaging(MRI)[17, 18], hyperthermic cancer treatments, cell sorting and targeted drug delivery [19]. Besides, it has also been widely used in biomedical research because of its biocompatibility and magnetic properties [19]. Plasmonic silver nanoparticles when integrated on magnetic iron oxide surface resulting a multifunctional magnetoplasmonic nanocomposite which produce electron/hole pairs upon excitation of plasmonic nanoparticles by induced electric field[20].

However, it is a technological challenge to control size, shape, stability, and dispersibility of NPs in desired solvents. Magnetic iron oxide NPs have a large surfaceto volume ratio and therefore possess high surface energies [21]. Consequently, they tend to aggregate to minimize the surface energies. Moreover, the naked iron oxide NPs have high chemical activity, and are easily oxidized in air (especially magnetite), generally resulting in loss of magnetism and dispersibility. Therefore, providing proper surface coating and developing some effective protection strategies to keep the stability of magnetic iron oxide NPs is very important. These strategies comprise grafting of or coating with organic molecules, including small organic molecules or surfactants, polymers, and biomolecules, or coating with an inorganic layer,

such as silica, metal or nonmetal elementary substance, metal oxide or metal sulfide. Practically, it is worthy that in many cases the protecting shells not only stabilize the magnetic iron oxide NPs, but can also be used for further functionalization for many applications.

In this research work, we intended to functionalize iron oxide with oleic acid, amino-propyl-tri-methylsilane(APTMS) and with plasmonicmetal silver(Ag) nanoparticles to prepare functional nanomaterials that find application in photocatalysis, photovoltaics and environmental remediation.

2. Experimental

2.1.Materials

Ferrous sulfate (Merck), Ferric chloride (Merck), Ammonia (25%, Merck), Distilled water, Ethylene glycol (Sigma-Aldrich), Silver nitrate (Merck), Polyvinylpyrrolidone (PVP, Mw 55000, Merck), 3-Aminopropyltrimethoxysilane (APMTS, Sigma-Aldrich), Methanol (Merck), Ethanol (95%, Merck). All chemicals were used without further purification.

2.2.Methods

2.2.1. Preparation of IOs

Iron oxide nanoparticles was synthesized by the reverse co-precipitation method where in iron(II) chloride and iron(III) chloride was used as the iron precursor and ammonium hydroxide was used as the base as reported earlier [22]. Briefly, amixed solution of FeCl₂.4H₂O and FeCl₃.6H₂O was added to ammonium hydroxide solution to get the precipitate. Formation of IOs was characterized by its characteristic FTIR spectrum, response to external magnetic field and SEM-EDS.

2.2.2. Preparation of Colloidal Ag Nanoparticles

A colloidal solution of silver nanoparticles is synthesized by a modified polyol process developed by Xia et al [23,24]. In a typical synthesis, 10 mL of EG is placed in a 100-mL triple-necked round-bottom flask and heated at 140 °C for 1 h under stirring with Teflon-coated magnetic stirring bar using a temperature-controlled heating mantle. While EG is heated, EG solutions containing AgNO₃ and PVP is prepared. 6 ml of both AgNO₃ and PVP solutions are added dropwise simultaneously in the hot reaction mixture using pipettes. The resulting solution was heated for 1 h. The final product is cooled to room temperature and centrifuged by ethanol. The plasmonic signature and the morphology of the Ag were characterized by UV-Visible and SEM-EDS respectively.

2.2.3. Functionalization of IOs with Ag Nanoparticles

Ag functionalized IOs is fabricated by silanisation

process as reported elsewhere[25]. Briefly, 1% APTMS solution is prepared in ethanol. 5 mg of IOs is added in 10 ml APTMS solution. Then 2 ml of as prepared Ag nanoparticles solution is added in the beaker. The solutions mixture is kept for 1hr. After 1 hr., the IOs-Ag materials was washed for 5 mins with ethanol. Washing cycle is repeated 15 times. Then the materials are filtered with filter paper and dried at room temperatures. The FTIR spectra of bare-IOs and Ag/IOs are recorded respectively.

2.2.4. Functionalization of IOs with Oleic Acid

10 ml aqueous solution of IOs (1mg/ml) was mixed with 5×10^{-4} M oleic acid solution in methanol and stirred for 6 hours till all the nanoparticles get precipitated at the bottom. After decanting water-methanol solution, addition of 10 ml toluene and 15 minutes' sonication in the precipitate resulted in clearly dispersed functionalized NPs in toluene [26].

2.2.5.Surface Functionalization of Synthesized Fe3O4 Nanoparticles with APTMS

1% APTMS solution was prepared in methanol from which 10 ml solution was taken in a beaker. 5.0 mg of IOs was added in the beaker containing 10 ml of 1% APTMS. The solution-mixture was stirred for 1h. After 1h of stirring, the solution mixture was centrifuged and precipitate was collected. The precipitate was washed 15 times with ethanol by centrifuge and finally the wet precipitate was filtered and dried at room temperature[25].

3. Result and Discussion

3.1. Magnetic Properties of IOs

When placed an external magnetic bar in the vicinity of the container containing the as synthesized IOs, they are attracted by the magnetic bar and formed spikes, as shown in Figure 1,which revealed the magnetic behaviour of IOs. The spike like structures indicate the formation of $Fe_3O_4[27]$.



Figure 1: Response to external magnetic field of the as synthesized IOs

3.2. Optical Absorption and Emission Properties of Ag Nanoparticles

It is known that colloidal silver nanoparticles exhibit yellowish color (Figure 2.a, inset) in solution due to excitation of surface plasmon in the visible region of spectrum. Formation of silver nanoparticles was confirmed by its characteristic surface plasmon resonance absorption peaks in its UV-Vis spectrum (Figure 2). The absorption spectrum of silver nanoparticles consisted of multiple resonance peakscentering at 360 nm, 410 nm and 450 nm (Fig.2a), which revealed the formation of anisotropic silver nanoparticles [28].

Colloidal Ag nanoparticles also exhibitedphotoluminescence property room temperature. Emission spectrum of colloidal Ag nanoparticles is shown in Figure 2.b. The visible luminescence of silver is due to the excitation of electrons from occupied d bands into states above the Fermi level and then, electron-phonon and hole-phonon scattering process take place results in energy loss and subsequently, radiative recombination of an electron from an occupied sp band with the holetakes place. Emission spectrum of colloidal Ag nanoparticles is shown in Figure 2.b. Excitation of the sample with 370 nm wavelength produced emission spectrum with maximum intensity 453 nm at showed photoluminescence property in the visible region[29].

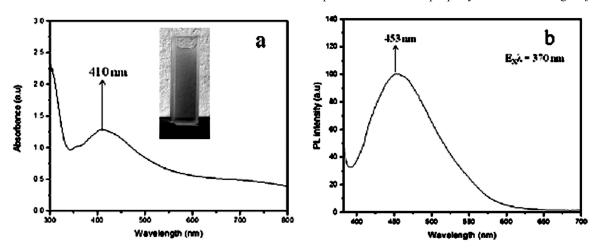


Figure 2: (a) UV-Visible absorption and (b) emission spectra of colloidal Ag nanoparticles (in set picture, a)

3.3. Fourier Transform Infrared(FTIR) Spectral Analysis 3.3.1. FTIR spectrum of the IOs

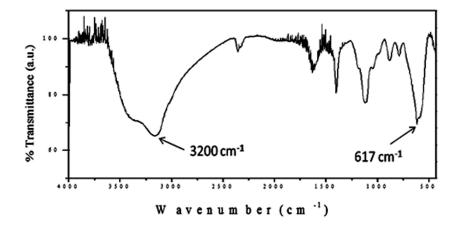


Fig.3. FTIR spectrum of IOs.

The characteristic FTIR peaks are shown in Figure 3. The peak at 3200cm⁻¹ is attributed to the stretching vibrations of -OH, the range of frequency of the -OH group is 3650-3200 cm⁻¹, which is assigned to -OH absorbed by IOs and the peak centering at 617 cm⁻¹ is attributed to the Fe-O bond vibration of Fe₃O₄[30].

3.3.2. FTIR Spectrum of IOs Functionalized with Oleic Acid

To confirm the functionalization of IOs with Oleic acid, FTIR spectrum of pure oleic acid was recorded which is shown in Figure 4.

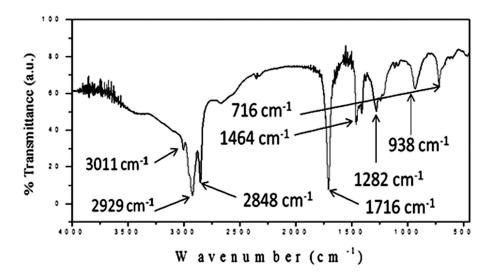


Fig. 4: The FTIR spectrum of oleic acid(OA)

Two sharp peaks at 2929, 2848 cm⁻¹ were attributed to the asymmetric CH_2 stretch and the symmetric stretch, respectively. The intense peak at 1716 cm⁻¹ was derived from the C = O stretch, and the peak at 1282 cm⁻¹

reflected the presence of C-O stretch. The O-H in plane and out of plane bands appeared at 1464 and 938 cm⁻¹ respectively which is consistent with literature[30]. The characteristic peaks of IOs functionalized with oleic acid is shown in Figure 5.

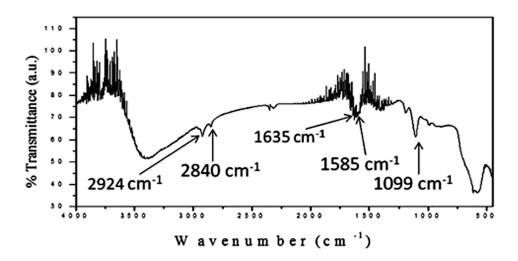


Fig. 5. The FTIR spectrum of IOs functionalized with oleic acid

The asymmetric $\mathrm{CH_2}$ stretch and symmetric $\mathrm{CH_2}$ stretch of oleic acid shifted to 2924 and 2840 cm⁻¹ respectively. The shifting of these peaks to a lower frequency region indicate that the hydrocarbon chains in the monolayer surrounding the nanoparticles were in a closed packed crystalline state. It is noticed that C=O stretching band of the carbonyl group appears at 1716 cm⁻¹ in pure oleic acid, Figure 4, which is absent in the spectrum, Figure 5, of the functionalized IOs. In addition, two new bands appeared at 1585 and 1635 cm⁻¹, which were characteristic of the asymmetric $v_{\rm as}(\mathrm{COO}$ -) and $v_{\rm ns}(\mathrm{COO}$ -) stretch. This result indicates that the bonding pattern of the carboxylic acids on the surface of the nanoparticles

was a combination of molecules bonded symmetrically and molecules bonded at an angle to the surface. A strong adsorption at 1099 cm⁻¹ arose from C-O single bond stretching. These results revealed that oleic acid molecules were chemisorbed onto the IOs as a carboxylate[30].

3.3.3. FTIR Spectrum of IOs Functionalized with APTMS

To confirm the functionalization of IOs with APTMS, FTIR spectrum of pure APTMS was recorded as a reference which is shown in Figure 6.

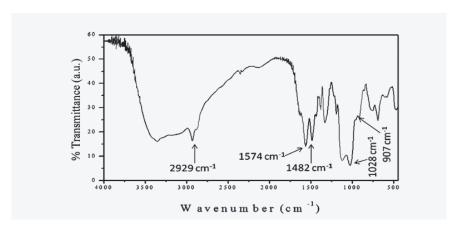


Fig. 6.The Infrared spectrum of APTMS

FTIR spectrum of APTMS clearly shows a sharp band at 2929 cm-1 which is attributed to the C-H stretch. The two intense peaks at 1574 and 1482 cm⁻¹ were derived from the NH₂ and methylene groups respectively. The Si-O-Si

bridge and Si-O bonds appeared at 1028 cm⁻¹ and 907 cm⁻¹ respectively[31].

Figure 7 shows the FTIR spectrum of IOs functionalized with APTMS.

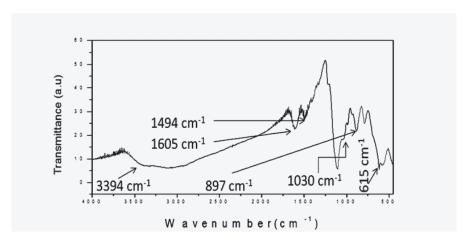


Fig. 7. The Infrared spectrum of IOsfunctionalized with APTMS

In this spectrum, peak around 1605 cm-¹is assigned to the -NH₂ terminal of APTMS. The CH₂ bending was found at 1494 cm-¹, the Si-O-Si bridges were appeared at 1030 cm-¹, and the Si-O bond at 915 cm-¹. The peak at 3394 cm-¹ is attributed to the stretching vibrations of -OH which is assigned to -OH absorbed by IOs and the peak at 615 cm-¹ is attributed to Fe-O bond vibration of IOs[30, 31]. Due to the functionalization of IOs using a

chemical linker APTMS Fe-O bond becomes weaker. In this bond, bond length of Fe-O is increased. So, the band shifts to the lower wavenumber i.e.; longer wavelength.

3.3.4. FTIR Spectrum of Nanocomposite Consisted of IOs and Ag Nanoparticles

The characteristic peaksin the FTIR spectrum of the nanocomposite consisted to IOs.APTMS.Ag is shown in Figure 8.

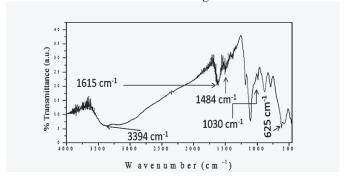


Fig. 8. The Infrared spectrum of nanocomposite IOs.APTMS.Ag

The peak around 1615 cm-1 is ascribed to the -NH₂ terminal of APTMS. The -CH₂ bending was found at 1484 cm-1. The peak at 3394 cm-1 is attributed to the stretching vibrations of -OH, which is assigned to -OH absorbed by IOs. The peak at 625 cm-1 is attributed to the Fe-O bond vibration in nanocomposite [30, 31, 32]. It is hypothesized that due to electron tunneling from Ag nanoparticles (plasmonic particles) to conduction band of IOs, electron density in Fe-O bond is increased.

Because of increased electron density, the bond strength is increased and thereforewavenumber of absorbed radiation is increased compared to that of in Fe-O bond in necked IOs.

3.4. Morphologies of the Nanostructures

To obtain detailed information about the morphology and the particle size of the nanostructures, scanning electron microscopy(SEM) wasemployed.

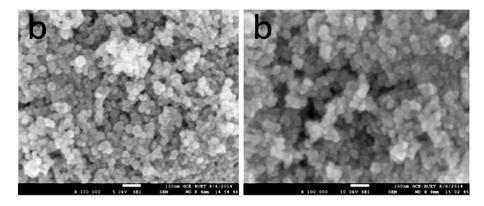


Fig. 9(b). The SEM images of Fe3O4 functionalized with oleic acid.

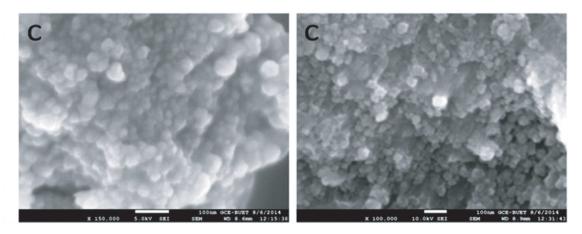


Fig. 9(c). The SEM images of Fe₃O₄ functionalized with APTMS

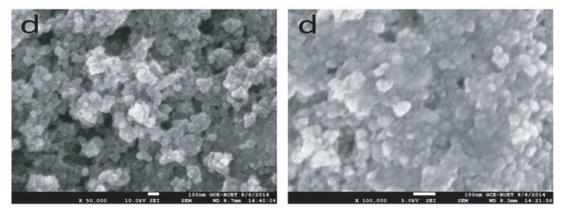


Fig. 9(d). The SEM images of the nanocomposite consisted of Fe₃O₄, APTMS and Ag nanoparticles.

Figure 9 shows the SEM images of pure IOs and its functionalized counterparts. It is evident from the images (from scale bar) that all four types of nanostructures had their dimensions in nanoscale range i.e.; diameter below 100 nm. The nanostructures were quasi-spherical in shape. However, except oleic acid functionalized IOs, which was monodispersed in size and formed stable colloids, the other three types of nanostructures were polydispersed in size and formed less stable colloids. This is because of formation of aggregates either in the

absence of any surfactant (pure IOs) or presence of APTMS molecules which form crossed-linked polymer. However, $\rm Fe_3O_4$ functionalized with oleic acid gives mono dispersed and stable colloidal IOs.

3.5. Energy Dispersive X-Ray Spectroscopic (EDS) Analysis

Energy dispersive X-ray spectroscopic (EDS) analysis of the samples was carried out to get information on elemental compositions of the nanostructured samples.

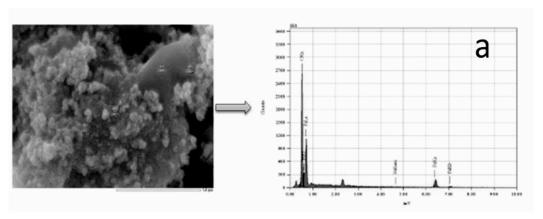


Fig. 10(a). The EDS spectrum of IOs particles

The composition components of IOs is given in the following table.

Elem ent	keV	Mass%	Sigma	Atom %
ок	0.525	40.41	0.28	70.30
Fe K	6.398	59.59	1.49	29.70
Total		100.00		100.00

Table3(a): The composition of IOs nanostructures

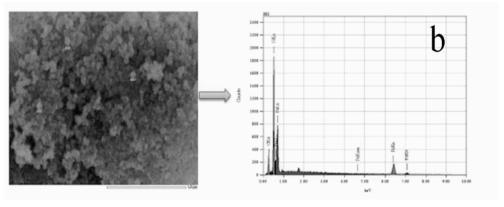
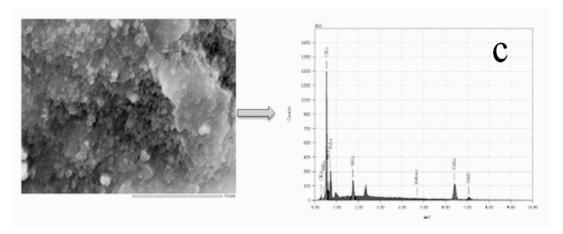


Fig. 10(b). The EDS spectrum of IOs functionalized with oleic acid

Elem ent	keV		Sigma	Atom %
CK	0.277	10.86	0.14	22.75
ок	0.525	33.05	0.28	51.97
FeK	6.398	56.09	1.45	25.27
Total		100.00		100.00

Table 3(b). The composition of oleic acid functionalized IOs.



10(c). The EDS spectrum of IOs functionalized with APTMS

Elem ent	keV	Mass %	Sigma	Atom %
C K	0.277	2.45	0.02	6.51
N K	0.392	0.17	0.04	0.38
ок	0.525	25.23	0.09	50.27
Si K	1.739	2.92	0.08	3.31
Fe K	6.398	69.24	0.76	39.53
Total		100.00		100.00

Table 3(c): The composition of IOs functionalized with APTMS

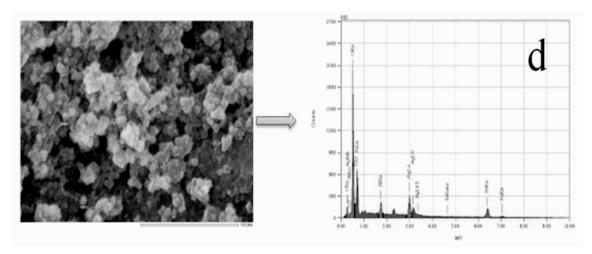


Fig. 10(d). EDS spectrum of nanocomposite consisted of IOs-APTMS-Ag nanoparticles.

Elem ent	keV	Mass %	Sigma	Atom %
CK	0.277	3.60	0.02	8.06
NK	0.392	0.45	0.06	0.87
ок	0.525	39.81	0.13	66.97
SI K	1.739	1.98	0.06	1.89
FeK	6.3983	37.39	0.54	18.02
AgL	2.983	16.77	0.24	4.18
Total		100.00		100.00

Table. 3(d). The composition component of nanocomposite consisted of IOs-APTMS-Ag nanoparticles.

Point and shoot analysis were employed to determine the presence and distribution of elements in the samples. The major components of the spot of the samples in the EDS spectra (the resolution of the spectra are poor as the original file received were image files and could not be reproduced), are shown in Figure 10. The elemental analysis of the samples corroborated with the desired materials and consisted with FTIR results of the corresponding samples.

4. Conclusion

In this research, preparation of magnetic nanostructured iron oxide particles and their surface functionalization with oleic acid, APTMS and Ag nanoparticles were accomplished. Surface functionalization of IOs did not change its magnetic property. However, colloidal property of IOs particles is dependent on the nature of the surfactants.

The particle size of IOs and IOs functionalized with APTMS and Ag nanoparticles are polydisperse while IOs functionalized with oleic acid are monodisperse. Research is ongoing to unveil the phase of the as synthesized IOs particles i.e.; Fe₃O₄ or Fe₂O₃. Such functionalized nanoparticles (IOs-OA, IOs-APTMS) and nanocomposite (IOs-APTMS-Ag nanoparticles) can be employed in biomedicalapplication, environmental remediationand energy harvesting technology.

5. Acknowledgement

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