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A Novel Approach to Dispersion and Stabilization of Water Based Ferrofluid Fe3O4

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1.0 INTRODUCTION:

Magnetic fluids, also called ferrofluids, are stable suspensions composed of nano-sized (∼10 nm diameter) magnetic particles coated with surfactants and dispersed into a carrier liquid. As they exhibit both magnetic properties and fluidity,ferrofluids are widely used in industry and clinical applications, such as sealing, filtering, lubricating [1], removal of water pollutants [2], liquid-cooled loudspeakers, high power transformers [3], atherosclerosis detection [4], magnetic resonance imaging (MRI) contrast enhancement, target drug delivery [5], enzyme immobilization, hyperthermia and biosensors [6], and so on.

Fe3O4, CoFe2O4 [7], ε-Fe3N [8], Co [9] and γ -Fe2O3 [10] can be used as magnetic particles. To avoid agglomeration and sedimentation of the nanoparticles, they should be coated with surfactants [11]. A wide range of materials can be used as surfactants, such as oleic acid, chitosan, polyethylene glycol (PEG), lauric acid and so on. There are numerous reports about ferrofluids prepared by nanoparticles coated with monolayer surfactants [12–14] and bilayer surfactants [15–17]. In general, monolayer surfactants are used in organic based ferrofluids while bilayer surfactants are used in water based ferrofluids. Although coated with bilayer surfactants, the stability of water based ferrofluids still requires improvement.

In this paper, we report a highly stabilized ferrofluid prepared by a new method with magnetic particles coated with tetralayer surfactants including oleic acid/PEG/agar/oleic acid. Oleic acid is a commonly used surfactant to stabilize the nanoparticles. The oleic acid layer stably disperses the nanoparticles and prevents them from forming large clusters due to magnetic dipole–dipole interactions [18]. Moreover, a clear shift from a strong electron-donor to a weak electron- donor was confirmed with the bonding of the oleic acid [19]. PEG is another surfactant that has been widely used to coat the nanoparticle [20–23] and it is hydrophilic, water-soluble, biocompatible, nonantigenic. It is usually functionalized by silane [20,21] or used as a secondary surfactant [23] while modifying the nanoparticles. Nevertheless, reports on agar being used as surfactant are scarce. It is also water-soluble and biocompatible and is widely used in food processing as a suspension agent. There are a lot of approaches that have been reported to synthesize the magnetic nanoparticles, such as microemulsion [24], sol–gel synthesis [25], co-precipitation [26], sonochemical reactions [27], hydrolysis and thermolysis of precursors [28], thermal reactions [29], flow injection syntheses [30] and electrospray syntheses [31]. Among all the methods, the co-precipitation technique is probably the simplest and most efficient approach to obtain the magnetic nanoparticles. This method is easy and inexpensive to prepare a large amount of aqueous dispersions of magnetic nanoparticles because the synthesis is conducted in water [32]. We can control the shape, size and composition of nanoparticles by changing the ratio of Fe2+ and Fe3+, pH, and ionic strength of the medium [33]. Here in this paper we use the co-precipitation method to synthesize a highly stabilized ferrofluid. We also characterize the structure, morphology and properties of the magnetic nanoparticles and ferrofluid.

A new method for preparing water-based Fe_3O_4 ferrofluid with high stabilization typically refers to an innovative process designed to improve the properties of magnetic fluids. Ferrofluids are colloidal suspensions of magnetic nanoparticles, like Fe_3O_4 (magnetite), stabilized in a carrier liquid, such as water. These fluids have numerous applications in medicine, electronics, and mechanical systems.

2 MATERIALS AND PREPARATION:

2.1Materials

All the chemicals used were of analytical reagent grade without further purification. Ferric chloride hexahydrate (FeCl3 · 6H2O), ferrous chloride tetrahydrate (FeCl2·4H2O), hydrochloric acid (HCl, 1 mol/L), aqueous ammonia (NH3·H2O, 13 mol/L), PEG-4000, oleic acid, agar, absolute ethyl alcohol .

Chemical Name	**Chemical Formula**	**Grade**
Ferric chloride hexahydrate	FeCl3 · 6H2O	Analytical reagent grade
Ferrous chloride tetrahydrate	FeCl2·4H2O	Analytical reagent grade
Hydrochloric acid	HCl, 1 mol/L	Analytical reagent grade
Aqueous ammonia	NH3·H2O, 13 mol/L	Analytical reagent grade
PEG-4000	N/A	Analytical reagent grade
Oleic acid	N/A	Analytical reagent grade

TABULATION: 3.1 REQUIRED MATERIAL

2.2 SYNTHESIS OF MAGNETIC NANOPARTICLE AND FERROFLUID

2.2.1Synthesis of magnetic nanoparticles

The magnetic nanoparticles were synthesized following the co-precipitation approach [34]. 5.43 g of $FeCl_3 \cdot 6H_2O$ and 2.02 g of $FeCl_2 \cdot 4H_2O$ were added into 100 ml of distilled water and stirred evenly. The solution was placed in a stir where it could be heated and the temperature could be controlled. 12 ml of $NH_3 \cdot H_2O$ was dropped into the solution when the temperature rose to 50 °C.

The chemical reaction is as follows:

$$\mathbf{Fe^{2+} + 2Fe^{3+} + 8OH^{-} = Fe_3O_4 \downarrow + 4H_2O}$$

The suspension was stirred with an electric stirrer and dispersed with a stir at 50 °C for 40 min and then laid aside for several minutes until the nanoparticles got precipitated. The nanoparticles were washed up with absolute ethyl alcohol for 3–4 times.

Finally we obtained the magnetic nanoparticles. We use oleic acid/PEG 4000, oleic acid/agar, oleic acid/PEG 4000/oleic acid, oleic acid/agar/oleic acid, oleic acid/PEG 4000/agar/oleic acid as surfactants respectively. In the following part we just exhibit the step of coating tetralayer surfactants because this is the best method.

2.2.2 COATING OF THE FIRST LAYER SURFACTANT :

2.34 g of magnetic nanoparticles were dispersed in 100 ml of distilled water. The suspension was then put in a stir heated to 55 °C. $NH_3 \cdot H_2O$ was dropped into the suspension to adjust the pH to 10. Then 7 ml of oleic acid was dropped into the suspension. The suspension was stirred and dispersed at 55 °C for 40 min and then laid under a magnetic field until particles precipitated. The particles were washed up with distilled water. Here we obtained the magnetic nanoparticles coated with monolayer surfactants.

2.2.3 COATING OF THE SECOND LAYER SURFACTANT :

The magnetic nanoparticles coated with oleic acid were dispersed in 100 ml distilled water. HCl was used to adjust the pH to 5 and 0.05 g of PEG-4000 was added into the suspension. The suspension was stirred and dispersed at 50 °C for 40 min and then laid under a magnetic field until particles precipitated. The particles were washed up with distilled water. Here we obtained the magnetic nanoparticles coated with bilayer surfactants.

2.2.4. COATING OF THE THIRD AND FOURTH LAYER SURFACTANTS

The magnetic nanoparticles coated with bilayer surfactants were dispersed in 100 ml distilled water. $NH_3 \cdot H_2O$ was used to adjust the pH to 9 and 0.08 g of agar was added into the suspension. The suspension was stirred and dispersed at 50 °C for 40 min. $NH_3 \cdot H_2O$ was dropped into the suspension one more time to adjust the pH to 10 and 7 ml of oleic acid was added into the suspension. Then the suspension was stirred and dispersed at 55 °C for 40 min. Finally, we got the ferrofluid with magnetic nanoparticles coated with tetralayer surfactants.

3.0 RESULT AND DISCUSSION

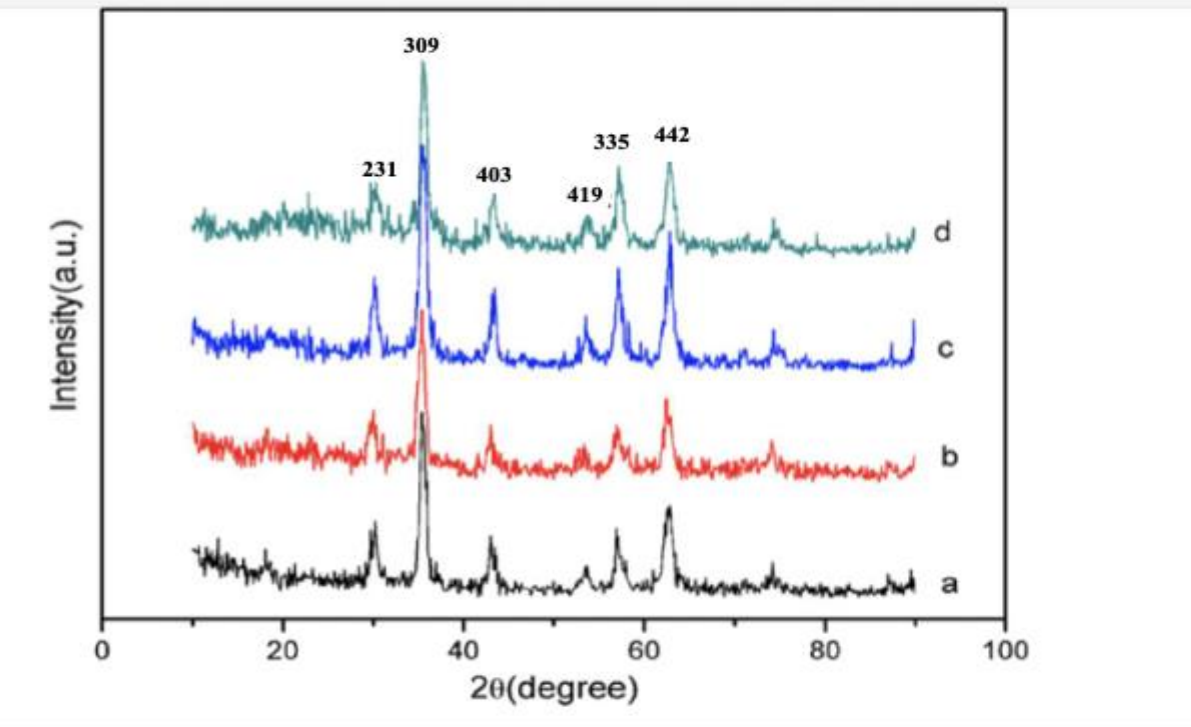
3.1 POWDER X-RAY DIFFRACTION :

The x-axis (2θ in degrees) represents the diffraction angle. The y-axis (Intensity in arbitrary units, a.u.) represents the intensity of the diffracted X-rays.There are four different diffraction patterns (labeled a, b, c, d), suggesting different samples or conditions.Distinct peaks are present at specific 2θ values with corresponding intensity changes.The most intense peak (~309) indicates a dominant crystalline phase in all samples.Other peaks (231, 403, 419, 335, 442, etc.) suggest the presence of additional crystalline structures.

PEAK POSITION (2θ in DEGREES	(hkl) Values
30	231
38	309
45	403
55	419
58	335
65	442

TABULATION : 3.1 PEAK POSITION(hkl) VALUES

FIG . 3.1 POWDER X-RAY DIFFRACTION The intensity of peaks increases from sample (a) to sample (d), suggesting increased crystallinity or phase purity.There may be phase transitions or structural modifications occurring across the samples.If these are nanomaterials or thin films, the variations could be due to changes in synthesis conditions.If these represent composite materials, the differences could



indicate varying compositions.The broadness of some peaks suggests the presence of nanocrystalline phases.

3.2 QUINCKE'S METHOD OF MAGNETIC SUSCEPTIBILITY

The x-axis is labeled as "% (percentage)".The y-axis is labeled as "weight".The graph shows a linear increase in weight as the percentage increases.The relationship between the two variables appears to be directly proportional.The x-axis starts at around 20% and extends beyond 60%.The y-axis ranges from around 2 to 22.The legend indicates that the plotted line represents "weight".

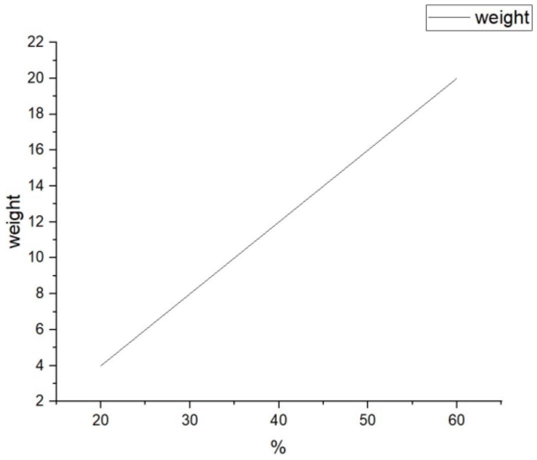


FIG. 3.2 ANALYSIS OF MAGNETIC SUSCEPTIBILITY

The single line suggests a single-variable dependency rather than multiple comparisons.The graph suggests a positive linear relationship between percentage (%) and weight.This could indicate that as the percentage value increases, the weight also increases at a constant rate.If this represents an experiment or a dataset, it may suggest a proportional dependency between weight and percentage.

3.3 THERMOGRAVIMETRIC ANALYSIS

TGA of uncoated magnetic nanoparticle(a),PEG-4000(b),coated magnetic nanoparticle(c), agar(d) and oleic acid(e). If weight loss is minimal, the material has low moisture or volatile content.Higher loss here means it contains absorbed water or low-boiling-point compounds.The temperature range where weight loss occurs shows thermal degradation onset.Lower decomposition temperature → Less thermally stable material.Higher decomposition temperature → More thermally stable material.More residue means he material has more thermally stable components (e.g., ceramics, carbon structures)

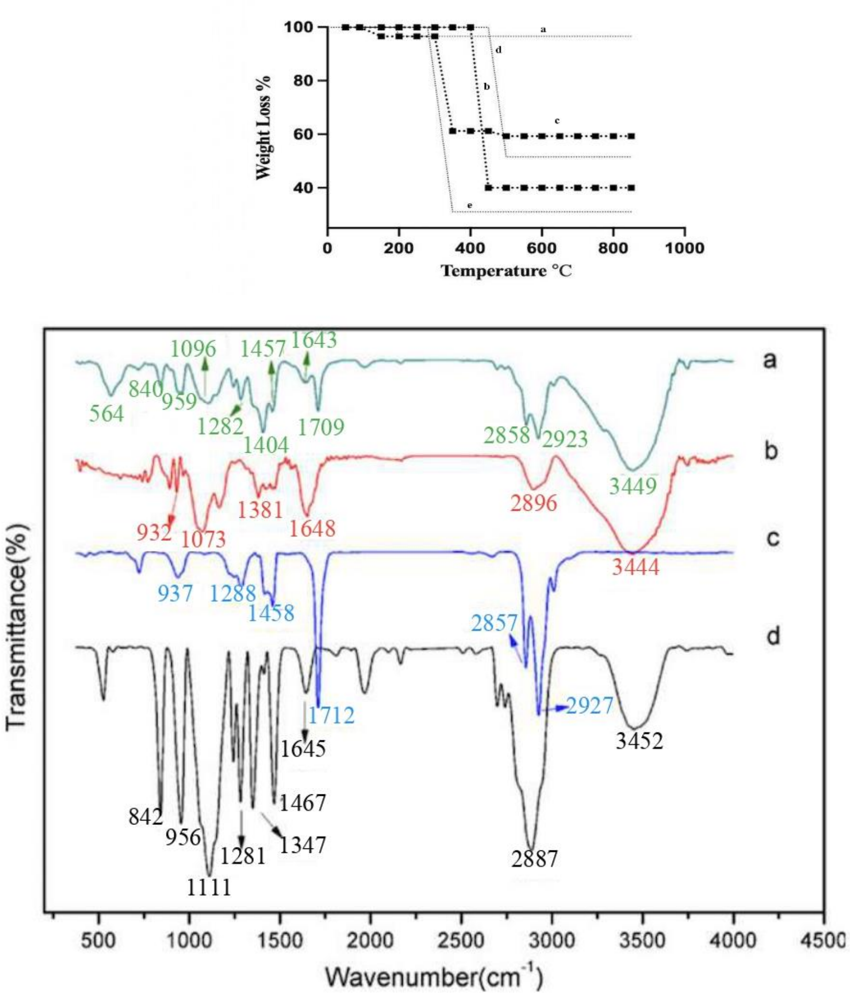


FIG.3.3 ANALYSIS OF THERMOGRAVIMETRIC

Less residue means more organic decomposition, indicating lower thermal stability. The material with the highest plateau (~60% remaining weight) after 600°C is more thermally stable.The material with the lowest plateau (~40% remaining weight) has less stability since more mass is lost. Agar is the most stable, leaving 20–30% residue at high temperatures. PEG 4000 decomposes significantly but leaves 5–10% residue. Oleic Acid is the least thermally stable and fully decomposes with almost no residue.

3.4 FTIR

The FTIR spectra labeled a, b, c, and d display transmittance (%) as a function of wavenumber (cm^{-1}). Each spectrum represents different samples, with characteristic peaks corresponding to different functional groups.

FIG. 3.4 ANALYSIS THE FOURIER TRANSFORM INFRARED SPECTROSCOPY

1. Spectrum (a) - Green Peaks

- Broad peaks at 2858 cm^{-1} and 2923 cm^{-1} → Indicate C-H stretching (alkane groups).
- Peak at 1643 cm^{-1} → Associated with C=O stretching (carbonyl groups).
- Peaks at 1457 cm^{-1} and 1404 cm^{-1} → Indicate C-H bending (alkane or aromatic compounds).
- Peaks in the 500–1500 cm^{-1} region → Likely correspond to fingerprint regions of various organic functional groups.

2. Spectrum (b) - Red Peaks

- Broad peak at 3449 cm^{-1} → Represents O-H stretching (hydroxyl group, alcohols, or water).
- Peak at 2896 cm^{-1} → Suggests C-H stretching.
- Peak at 1648 cm^{-1} → Indicates C=C stretching (aromatic or alkene).

3. Spectrum (c) - Blue Peaks

- Broad peak at 3444 cm^{-1} → Indicates O-H stretching (hydrogen-bonded hydroxyl groups).
- Peak at 2857 cm^{-1} → Suggests C-H stretching.
- Peak at 1712 cm^{-1} → Strong C=O stretching (carbonyl group from esters, ketones, or carboxylic acids).

4. Spectrum (d) - Black Peaks

- Broad peaks at 3452 cm^{-1} and 2927 cm^{-1} → Correspond to O-H and C-H stretching, respectively.
- Peak at 1712 cm^{-1} → Strong C=O stretching (carbonyl compound presence).
- Multiple peaks in the 500–1500 cm^{-1} region → Correspond to fingerprint region vibrations, possibly indicating aromatic rings, C-N stretching, or phosphate groups.

Spectrum	**Wavenumber (cm^{-1})**
a (Coated magnetic nanoparticle)	564, 840, 959, 1096, 1282, 1404, 1457, 1643, 1709, 2858, 2923
b (Agar)	932, 1073, 1381, 1648, 2896, 3449

c (Oleic acid)	937, 1288, 1458, 1712, 2857, 3444
d (PEG 4000)	842, 956, 1111, 1281, 1347, 1467, 1645, 1712, 2887, 2927, 3452

TABULATION: 3.2 SPECTRUM AND WAVENUMBER OF FTIR

The presence of O-H stretching (3449–3452 cm^{-1}) in spectra b, c, and d suggests hydroxyl-containing compounds (e.g., alcohols, phenols, or water absorption). The C-H stretching (2858–2927 cm^{-1}) in all spectra confirms alkane groups. The C=O stretching (1643–1712 cm^{-1}) indicates carbonyl-containing compounds like aldehydes, ketones, or esters. The variations in fingerprint regions (500–1500 cm^{-1}) suggest different structural compositions in the analyzed samples.

4.0 CONCLUSION:

In this paper, we have synthesized a highly magnetized and stabilized water based Fe3O4 ferrofluid by a new method. We use oleic acid/PEG/agar/oleic acid as surfactants and add them into the fluid one after another. The magnetic nanoparticles will be oxidized with the increase of coating time and the best coating time is about 40 min. The result of XRD shows that the magnetic nanoparticles are cubic spinel structures (30.9°) Fe3O4. The result of FT-IR spectra shows that all three surfactants are included in the coated magnetic nanoparticles (1000–500)and from TGA data(Above 800) we can tell the content of oleic acid, PEG-4000 and agar is 4.06%, 27.55% and 6.04% respectively. The magnetic weight measured by Quincke's method, confirm that the Fe3O4 nanoparticles are superparamagnetic and the saturation magnetization of the magnetic nanoparticles of our own is larger than that of nanoparticles coated with bi-surfactant with same weight. The measurement of change of magnetic weight shows that the ferrofluid has high stabilization under applied magnetic field. Compared with ferrofluid

using agar/PEG as surfactants, the change of magnetic weight of the novel ferrofluid is much smaller.

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