

Magnetic Nanoparticles with Silica Coating: Synthesis, Characterization, and Applications

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Abstract

This study examines how the pH of the reaction media affects magnetite nanoparticles' silica coating. Ferrite nanoparticles were created by employing iron chloride as the foundation and an oxidation-precipitation technique. Na₂SO₃ assisted in slightly decreasing the iron chloride to metallic salts, followed by ammonia's alkalization. The particles were coated with water-based or HCl solutions using the sol-gel technique for the base- or acid-catalyzed hydrolysis process. Chemicals and coated magnetic nanoparticles are characterized with Zeta Potential and powder X-ray Fourier transforms infrared. The distinction seen in pHIEP in a hydroxide blend for pure silica, magnetite, and silica-covered magnetite examples shows the viability of the covering methodology regardless of whether the Fourier-change infrared reflections didn't uncover the improvement of Fe-O-Si communications. Surface characteristics resembling natural silica are the result of coated magnetic nanoparticles.

Keywords: magnetite, covering, nanoparticles, silica

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1. Introduction

Silica-coated magnetic nanoparticles are composite elements having a magnetic core and a thin silica (silicon dioxide) covering around the middle. Due to their distinctive characteristics, these particles have attracted considerable interest in many sectors, including medicinal applications, environmental cleanup, catalysis, and data storage [1]. The nanoparticles often have a magnetic substance like iron oxide (Fe₃O₄) or magnetite (FeOFe₂O₃) in their center. These substances' magnetic qualities enable particles to react to an external magnetic field. According to the intended use, the size of the core of these nanoparticles may range from a few nanometers to a few hundred nanometers [2]. A silica coating is put on the magnetic core's substrate to enhance stability, stop accumulation, and increase biological compatibility. Amorphous Polymer silica (SiO₂) is a substance that is simple to produce and may be altered to acquire certain qualities. During the synthesis process, the depth of the coating may be adjusted and generally ranges from a few nanometers to tens of nanometers in diameter [3]. Magnetic nanoparticles with a silica covering are typically made in two steps. The magnetic core nanoparticles are created in the first stage utilizing various processes, including sol-gel procedures, thermal breakdown,

and co-precipitation. The silica layer, known as silica coating, is designed around the magnetic core in the second stage [4]. The silica covering improves the stability and shelf life of the nanoparticles by preventing the oxidation and aggregation of the magnetic core. Silica is a biocompatible substance, which qualifies the nanoparticles for use in various biological processes, including medication transport, imaging, and hyperthermia [5]. Another popular method for covering silica is the sol-gel procedure. Alkoxysilanes are subjected to hydrolysis and polymerization in a solvent before being deposited onto the substrate. This technique may control the coating thickness, and different decants or functioning molecules can be included throughout the manufacturing process [6]. The chemical precipitation approach, the collected nanoparticles may be submitted to magnetic treatment for separation. To improve their stability and stop oxidation, they may also undergo stabilization procedures, including surface modification [7]. Small, generally 1 to 100-nanometer-sized particles with magnetic characteristics are known as magnetic nanoparticles (MNPs). They have attracted much interest in many industries, including medical, electronics, environmental cleanup, and energy storage [8]. The biogenic production of nanoparticles, facilitated by microorganisms,

including bacteria, fungi, and algae, is examined in this study and its uses. For the creation of nanoparticles, microorganisms provide a diverse habitat [9]. The issues to be addressed, however, include achieving the required size and shape with the least amount of effort, increasing the stability of nanoparticles, and tailoring certain microbes for particular uses [10]. The study provides a comprehensive overview of the SPIONS research done so far. At the same time, it began with various physical, chemical, biological, and physical-chemical-biological (using bacteria and plants) methods of SPION synthesis, such as gas-phase deposition, pulsed laser ablation, power ball milling, and micro-emulsions [11]. With an emphasis on Iron Oxide Nanoparticles (IONPs), this study offers an up-to-date summary of the processes for the production and fictionalization of MNPs. First, synthesis methods are described for creating IONPs with various compositions, sizes, forms, and architectures [12]. They discuss the intricate relationship between physicochemical characteristics and magnetic characterization, crucial for creating novel and potent magnetic-driven nanocarriers [13]. The research data on the features and uses of several NPs produced using algae-mediated biosynthesis [14]. Biomedical uses were shown for both dopamine classifications, including increased antibacterial activities, contrast imaging capabilities, and colloidal ZnO NP physiological compatibility and stability in biological factors [15].

The production of these nanoparticles, along with their characterization and the uses for them, has all been the subject of substantial research.

The other part of this study is provided as follows: Section 2 presents the materials and methods, Section 3 presents the results and discussion, and Section 4 concludes the study.

2. Materials and methods

$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and Na_2SO_3 of the experimental evaluate the following solvents; the same measured volumes were used for ethanol, a chloride acid solution, and tetraethyl orthosilicate.

2.1. The creation of iron oxide nanoparticles

The technique put out for making nanoparticulate magnetite. The following ingredients were used to make magnetic particles: The components are combined to make 400 mL of solution: 15 mL $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 10 mL Na_2SO_3 reserve solution, and 25.4 mL neutralized nitrogen hydroxide solutions. A protective gas (nitrogen) was bubbled in a 1000 mL³-necked round bottom flask to provide an inert environment for the process. The solution's color quickly transitioned from pale yellow to red and back to yellow when Fe^{3+} and SO_3^{-2} were combined. The answer was promptly put into the diluted ammonia solution while vigorously stirring, resulting in a black residue. Prompting the reaction for an additional 30 minutes completed the process. The excess fluid was eliminated, and distillation water was used to spin the black deposit. This process was repeated five times, after which the resultant product was collected and centrifuged with acetone before being deposited in a desiccant and allowed to dry at a comfortable Manjunath et al., 2023

temperature. Following this, the specimen was decreased in a Pyrex tube with H_2 flowing through it at a rate of 50 mL.min⁻¹ for 2 hours, with a temperature rise of 10 °C.min⁻¹ from 20 to 250 °C. The sample has the name Mt.

2.2. Nanoparticles of iron oxide coated with silica

With the modified described techniques, silica coating was completed. Several alcohols and various ethanol-to-water volume ratios (VE/W) created silica-coated magnetite nanoparticles. TEOS and catalyst feeding amounts were also adjusted, and the synthesis products underwent thorough characterization. We used the ideal experimental setups discovered in previous studies in our research. The nature of the initiator did not affect the composition of the responses. With 160 mL of ethyl liquor, 0.04 g of attractive powder was commonly weakened. Using ultrasound vibration in a water bath for 10 minutes, the dispersion was homogenous. A permanent magnet was used to divide the previously collected magnetic material magnetically, and each time, boiled water was used to clean it thoroughly. The appropriately acidic and basic media, with pH values of 4.1 and 11.4, were called MtSi-(a) and MtSi-(b), respectively.

2.3. Glass synthesis

A control specimen of silica glass was created to conduct the zeta potential research. In this example, a neutralized (HCl - pH 1.7) water solution was used to mix ethanol and TEOS in a 4:1:4 molar ratios. The mixture was agitated magnetically until it gelled. The material was first treated in a dry environment for 24 hours after spending the previous night in a humidified atmosphere.

2.4. Characterization methods

The FTIR spectra of covered and untreated samples were compared to determine silica-coated magnetite nanoparticles' framework, consistency, and production. Materials were compacted with KBr (around 1%) for these examinations, and a Perkin Elmer Range GX spectrophotometer was utilized to lead transmission mode investigation. Using silicon as a source of various light materials and $\text{CuK}\alpha$ power ranging from 20 to 80 at a sweep speed of min⁻¹, estimates of X-beam propagation were made in a Rigaku-type Geigerflex gear. The substrate was homogenized with ultrasonic for 15 minutes before the zeta potential metrics and then trapped in water. Aqueous NaOH 10⁻³ mol will be the solution's pH after it has been determined. L⁻¹ or 10⁻³ mol HNO_3 to alter it, L⁻¹ (ξ) was added. It was calculated using a pH function. An aqueous solution of magnetic powders was ultrasonically agitated for 10 minutes before a 30-minute rest period to determine the particle sizes.

3. Results and discussions

3.1. Magnetite nanoparticles

According to XRD data (Figure. 1, 2, and 3), sample Mt. had two phases that represented magnetite and elemental iron. The nanoparticle size impact is responsible

primarily for the observed line widening. The Scherrer equation's breadths of reflection 311 calculation indicated that the magnetite sample's average particle size was 10 nm. Figure 4 shows the FTIR spectra of sample Mt. The inversion spinel-type arrangement of magnetite results in bands corresponding to the material's MT-O-MO vibrations. Metals that occupy the hexagonal and octagonal configurations are referred to as MT and MO correspondingly. More possibilities are MT-MO and MO-O. Although ' ν^1 '- and ' ν^2 '-type bands were characterized, we could not detect vibrations type ' ν^3 ' due to equipment restrictions. The reported absorbance values for different iron oxides are shown in Table 1.

Figure 5 depicts how the pH solution affected the magnitude of the zeta potential variation for specimen Mt. This sample has a pH of roughly 5.0, corresponding to its isoelectric point (IP).

3.2. Silica-coated magnetite particles

As expected, the reach, which incorporates Fe_3O_4 and a couple of Fe diffraction tops, is strikingly like that of the uncoated example. The FTIR spectra of an assigned silica glass specimen (Figure. 4) revealed categories associated with the Si-O-Si connections at 1080, 800, and 460 cm^{-1} , as well as an increase associated with SiOH bond vibrational at 960 cm^{-1} . The sample (MtSi-b 3) had peaks associated to the Si-O-Si bond at 1080 cm^{-1} and 800 cm^{-1} , in addition to a peak at 575 cm^{-1} associated with Fe-O stability. In the FTIR spectra of this specimen, the shoulder pattern at 960 cm^{-1} is caused by Si-O-H stretches and Fe-O vibrations. The spectra of the coated and untreated samples are identical, indicating that Fe-O-Si bond formation was not sped up. The specimen MtSi-a's FTIR spectra (Figure. 4, two broad band's at 570 cm^{-1} and 1200 to 900 cm^{-1} indicate magnetism and Si-O bands. The Mt specimen has a pH equilibrium point (pHIEP) of around 5.0. The pHIEP of unadulterated glass is roughly 2.0, though the pHIEP of tests MtSi-(a) and MtSi-(b) is 2, 3. To establish the impact of external charges on magnetite elements, the zeta value for silica-coated and uncovered nanoparticles was assessed as a measure of the pH of the system (Fig. 5). While all covered nanoparticles

had a dependency on pH similar to that of pure silica, magnetite nanoparticles have a pHIEP of roughly 5.0. Their pHIEP measured at 2.3, compared to pure silica's pHIEP of 2.0. Since the exterior features of appealing nanoparticles are said to be equivalent to those that occur with pure silica, the observed change in pHIEP in the KCl configuration demonstrates the suitability of the most popular method of coverings. Different species must be present in the primary and acid-catalytic media coverings. According to the pHIEP of 5.0 for magnetite particles, the surface of magnetite was opposing when deposited at pH 11.4, but it was positive when coated at pH 4.1. FeO- groups are expected to dominate FeOH+2 groups on the surface of magnetite at pH 11.4 in comparison to pH 4.1. However, according to our research, coating happened in both cases. There have been hypotheses in the study regarding particular encounters between magnetite nanoparticles and silica. Some possible interactions can lead to particle disposability in the silica matrices: covalent, through the formation of Si-O-Fe bonds; electrostatic, between positively charged groups on the particle surface and negatively charged Si-O terminal ligands; then there are hydrogen-bond connections between the surface of the molecule and the hydration layers of silanol groups. Si-O-Fe links in the cracked hydrogen strongly support a relationship between the silica matrix enclosing it and the specific Fe^{3+} ions. NiFe_2O_4 nanocrystals distributed within a silica matrix yielded the same results. In those studies, the IR and EPR spectroscopes were used to track changes. Our investigation utilizing the Fourier-transform infrared wavelength of coated samples did not reveal any additional bands associated with Fe-O-Si linkages. This results in very little, if any, Fe^{+3} -Si exchanges. The Bruni model suggests that the silica matrices or silanol molecules outside of the spaces where the Fe_3O_4 nanoparticles are produced may interact with one another. Due to polycondensation, the vibrational adsorption bands of the silanol groups and H_2O particles were slightly stronger after heating at 300, 500, and 700 °C. The Fe-O-Si band emerges at 700 °C.

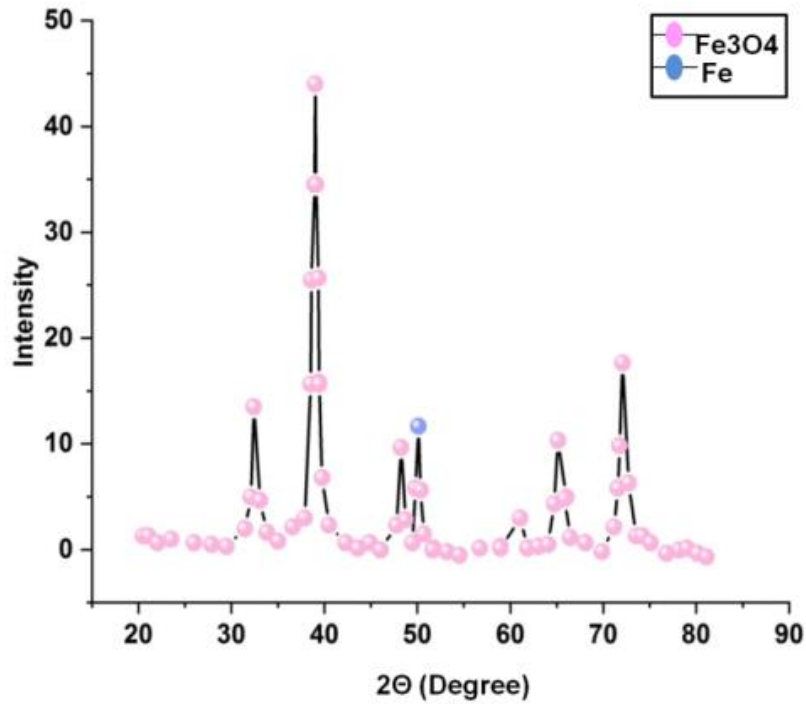


Figure 1: The samples shown in the X-ray diffraction patterns are Mt

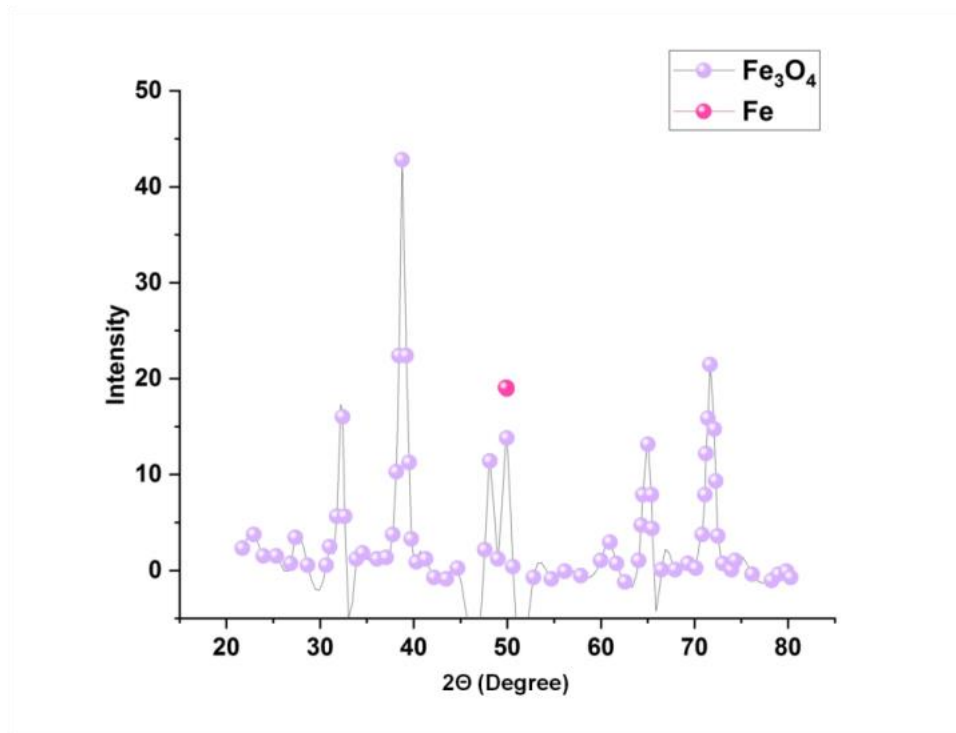


Figure 2: MtSi-b samples are seen in the X-ray diffraction patterns

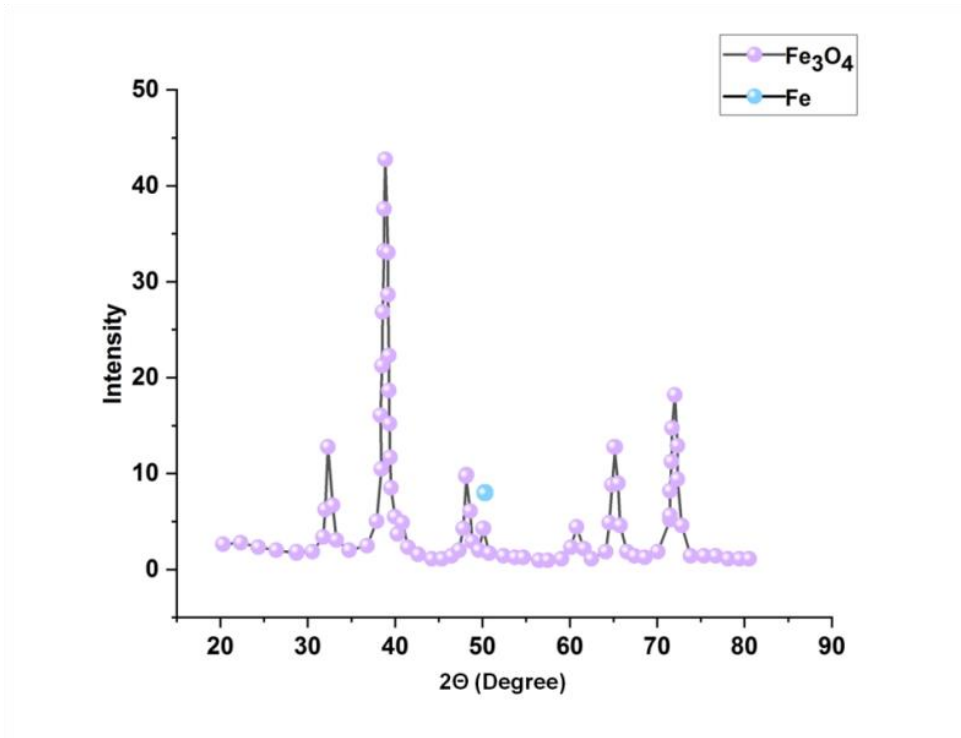


Figure 3: The X-ray diffraction patterns show MtSi-samples

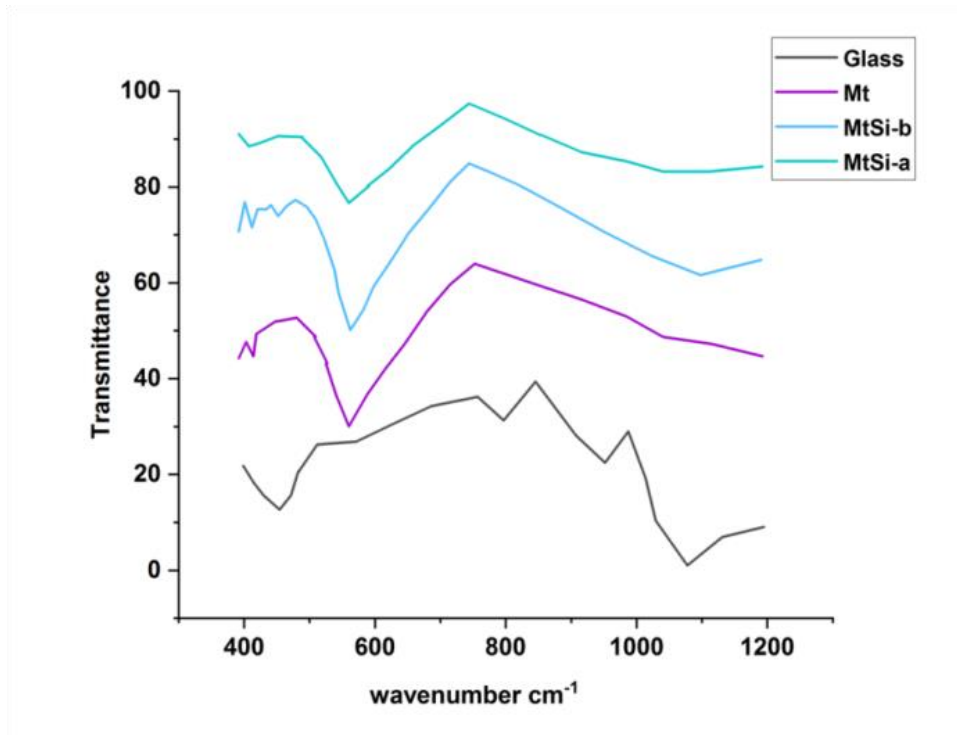


Figure 4: FTIR spectrum data for glass, MT, MtSi-b, and MtSi-a

Table 1: Different iron oxides' IR bands

Iron oxide	IR bands (cm ⁻¹)
Akaganeite	850, 650
Feroxyhyte	1120, 930, 780, 660
Goethite	880, 787
Lepidocrocite	1036, 1151, 743
Magnetite	580
Hematite	550, 480
Maghemite	620, 580, 560, 440

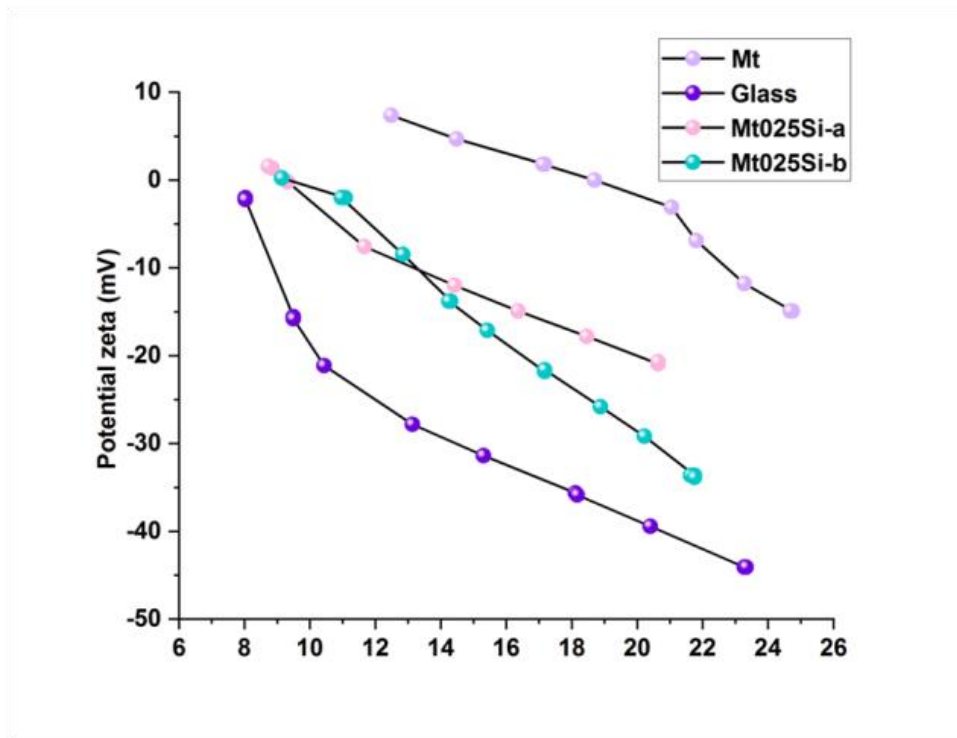


Figure 5: Glass, Mt, MtSi-b, and MtSi-a have different zeta potentials depending on the pH

4. Conclusions

In conclusion, creating, analyzing, and using magnetic nanoparticles coated with silica opened up various intriguing research and technology application areas. Magnetic nanoparticles with specific sizes, shapes, and magnetic behavior may be produced in a controlled manner thanks to the synthesis process. After being coated with a layer of silica, these nanoparticles gain stability, biocompatibility, and fictionalization properties that increase their adaptability. The assessment of the structural, chemical, and magnetic characteristics of the nanoparticles is made *Manjunath et al., 2023*

possible by characterization methods such as transmission electron microscopy, X-ray diffraction, and magnetic measurements. These investigations make it possible to understand the behavior of the nanoparticles and improve their performance for specific applications. Magnetic nanoparticles coated with silica have several uses in a variety of industries. These nanoparticles may be used in biomedicine for biosensing, magnetic resonance imaging (MRI) contrast agents, and targeted medication administration. Under base- and acid-catalyzed synthesis conditions, potential zeta tests demonstrated that magnetite nanoparticle silica coating was effective. Covered magnetite regularly displayed a pHIIEP similar to the pH of the

specimen of pure glasses. Additionally, the nanoparticles maintained their appealing characteristics after covering, and their molecule size decreased by around 30% after covering, likely due to the silica covering's anticipated impact of preventing nanoparticle collecting.

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