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Sonokimyasal Yöntemle Çok İşlevli MnO Kaplı Ag Katkılı γ-Fe_2O_3 Nanopartiküllerin Sentezi ve Karakterizasyonu

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Mangan(II) nitrat
Parçacık boyutu analizi
Amorf

ÖZ

Bu çalışmada, γ-Fe_2O_3 nanoparçacıklarının sentezi, manyetik karıştırma ve sonikasyon yoluyla yaş çökeltme yöntemiyle gerçekleştirilmiştir. Bu çalışma için kullanılan metodoloji, sentez yolunu optimize etmek için iteratif deneysel bir yaklaşıma dayanmaktadır. γ-Fe_2O_3'ün manyetik, elektronik ve katalitik özelliklerini geliştirmek amacıyla çekirdek-kabuk yapısı oluşturmak için MnO2 kaplaması da uygulanmıştır. Katalitik performans açısından çekirdek/kabuk yapısının işlevselliğini artırmak için Ag katkılama yapılmıştır. Morfoloji ve kristal yapı analizi, taramalı elektron mikroskobu ile gerçekleştirilmiştir.

Synthesis & Characterization of Multifunctional MnO Coated Ag-doped γ-Fe_2O_3 Nanoparticles by Sono-Chemical Method

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ABSTRACT

In this research work, the synthesis of multifunctional γ-Fe_2O_3 nanoparticles was carried out by the wet precipitation method through magnetic stirring and sonication. The methodology employed for this work has been based on an iterative experimental approach to optimize the synthesis route. MnO_2 coating was also applied to form a core-shell structure to enhance magnetic, electronic & catalytic properties of γ-Fe_2O_3. Ag doping was carried out to promote the functionality of core/ shell structure in terms of catalytic performance. The morphology & crystal structure analysis was carried out by scanning electron microscope.

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Introduction

In 1959, the promise of nanotechnology was outlined by Nobel Prize laureate Richard Feynman in his famous talk, “There's Plenty of Room at the Bottom”. Since then, the concepts of molecular nanotechnology have extended to such as “molecular engineering” by Eric K. Drexler *1+ and “molecular electronics” by Aviram and Ratner (1974) and Drexler (1981).

Nanotechnology is a truly revolutionary application and materials such as nanotubes, nanobiotics are the future for best properties. It is the grooming area of research all over the world nowadays. In which we can design material for the best properties on a molecular scale by considering its physical aspects. The material which is designed by using this approach is an improved product (Booker and Boysen, 2005; Leguna et al., 2011).

Today scientist realm, the prefix "nano" shows that the size of the particle is less than 10^{-9}m. Nanoscale material, therefore, lie between bulk micro-scale material and molecules and atoms. This regime has previously been unexplored and back on the researcher with the broad vision, where opportunities abound for those willing to pack their wagons and head into this scientific hinterland (Booker and Boysen, 2005).

These materials have already been manipulated at the nano scale for their efficient utilization as semiconductors, plastics, ceramics, and metals. Their properties have been employed by SEM, TGA, VSM, and thermograph. Internet, computer information technology, wireless, telecommunication, satellite telecommunication integrated circuits, wireless telephone, photocopy, audio, and video cameras are designed on the concepts of nanotechnology (Diwan and Bharadwaj, 2006a). The concept of "nano" comes from a scientist Eric Drexler (1981), who founded a center, called the foresight institute, and developed it to explore his ideas. Concepts being discussed include the development of tiny nano robots that can live inside the human body and repair blood tissues, which are damaged by different diseases. These can also be responsible for the healing of cancer, and chronic diseases. The nano solids may appear in three forms as amorphous, polycrystalline, and crystalline. The growth of these materials takes place on the nano scale, which gives them the naval properties (Booker and Boysen, 2005).

By using this approach, we improve the existing components by making smaller products and better performance (Choi et al., 2012). Initially, the following defining features of nanotechnology were hammered out (Booker and Boysen, 2005). The length scale is also important in nanotechnology. Some scientists say anything smaller than (10^{-6}) include in the nanoscale. However, engineers have been building things with such dimensions for a while. For example, Intel's Pentium III computer chips build during the late 1990s and transistor size is 200nm in width (Kuno, 2012).

The length scale directly affects the energy of the material in which discrete bands of energy are formed, such effects are generally termed quantum confinement. This quantum confinement is in one, two and three dimensions (Kelsall et al., 2005). De Broglie's wave-particle duality also has a concept of nanoscale behind this (Kuno, 2012). In the past, it was

apprehended that growth and size of the structure are very important for the determination of the properties of metal, and fine structure was preferred to obtain better properties. The discovery of Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), & X-ray Photoelectron Spectroscopy (XPS) has revolutionized science and technology to explore or, and modify the structure at the nano-level for better and efficient performance that can be achieved by macro modification. So the impact of mechanical properties on the grain size is also behind the concept of nanotechnology (Booker and Boysen, 2005). Nanotechnology brought prominent changes and innovations in semi-conductive devices which we use in our daily life. Three types of nanostructure are considered "low dimensional systems" such as (Kuno, 2012).

1. Two-dimensional (2D) system
2. One dimensional (1D) system
3. Zero dimensional (0D) system

Nanomaterials known as famous due to their large surface area and their growth on a nano scale decreases the energy of activation and make them reactive as a catalyst which is the key goal and that goal will more closely perform specific chemical conversions with high yield and high energy efficiency (Diwan and Bharadwaj, 2006b). Ferrites as catalysts are the most familiar materials used today (Dong et al., 2000). Different methods are used for its synthesis, in which sol-gel, microemulsion and sonication are most dominant (Dong et al., 2000).

Atomic clusters for nanostructural materials can be synthesized by the vapor condensation route. These materials can be used as a filter and consolidated into the bulk material. This clustering method can be used in the synthesis of ceramic material, mostly in powder form, another method is also used but their use depends on the type of application and available resources. Their production is not an easy task and can be synthesized by (Kelsall et al., 2005).,

1. Top-down process
2. Bottom-up process
3. Method for templating the growth of nanomaterials
4. Ordering of nanosystems.

In the past decade, many efforts to synthesize nanoparticles of basic building blocks of nanostructure and control shape and morphology have been practiced (Rutello, 2006). It is surface-functionalized nanoparticles that are used in bioengineering. The research work includes recent development and various strategies in the preparation of nanostructure for its

catalytic behavior characterization. So, its surface can be modified by organic and inorganic materials (Wu et al., 2008).

This study also is aimed on the surface functionalization of γ-Fe_2O_3 with silver preceded by a coating of MnO_2. Magnetic nanoparticles are of great interest for researchers from a broad range of disciplines including magnetic fluids data storage, and bio application. In Co oxidation process "Fe" functionalize have an improved oxygen exchange ability (Leguna et al., 2011).

Material and Method

This process was performed at the Royal Institute of Technology. In this process microemulsion prepared Fe with varying concentrations was mixed with a series of Ce-Fe mixed oxide. N-octane was a continuous phase, $C_{19}H_{42}BrN$ as the surfactant, 1-$C_4H_{10}O$ as the co-surfactant, and metallic precursor's solution as the aqueous phase. NH_3 was used as a precipitant. Its composition is expressed as weight percent 13% 1-butanol, 53% n-octane, 13% CTAB, and 21% aqueous phase (2M). The amount of both Fe and Ce was different for different solutions. The initial values are 5, 10, 25, and 50%. Initially pure cilium and Fe_2O_3 were prepared.

Fe (at. %) = Fe (mole) × 100 / Fe (mole) + Ce (mole)

For every atomic ratio of Fe/Ce the following molarities were tested 0.4; 1.0; 1.5 and 2M. where M can be calculated as follows.

M = [Ce (mol) +Fe(mol)]/L

An excess of NH_3 (9:1 NH_3/(Ce+Fe) molar ratio) was used as a precipitating agent. The synthesis of these particles was carried out as followed: Two microemulsion systems with the composition shown in Table 1 were prepared separately

Table 1. Two microemulsion systems with the composition

	Ce (at. %)	Fe (at.%)
CeO_2	100	0
$CeFe_5$	94.2	5.8
$CeFe_{10}$	89.2	10.8
$CeFe_{25}$	73.4	26.6
$CeFe_{50}$	45.6	54.4
Fe_2O_3	0	100

ME1 contains an adequate amount of the metal nitrate in the aqueous solution, and ME2 contains NH_3 in the aqueous solution. ME1 was added dropwise in ME2 then we observe that

the precipitated particles appeared in the system, the mixture was stirred continuously for 24 hours, after that it reached a saturation value, and then the particles were centrifuged. These particles were washed with methanol and dried at 60C^{o}, after particles were calcined at 500C^{o} for 2 hours, at the end you obtain the desired product. Both scientists quantify the elementary composition by X-ray diffraction. The surface texture was studied with BET. Similarly, Raman spectra UV-vis spectra, TGA and DSC were also performed on these particles.

Results and Discussion

Some scientists have worked on the modification and surface functionalization of nanoparticles in a variety of ways, Gajovic et al., 2011; Hlawacek et. al. (2013) was two of them. The investigated and described most of the properties were related to the size of nanoparticles. These scientists coated the generated nanoparticles and studied the effect of the coating. Cetyltrimethylammonium-bromide (CTAB) nanorods of ratios 4 and 5 were prepared by seed mediated technique. They removed the excess CTAB by centrifuging at 15000 rpm for 10 min at the rate of 1500rpm/min. In which we obtained the nano-rods at the bottom of the centrifuge. This procedure performs again by suspending the same nano-particles in the same amount of Milli-Q water. In the second time, the rpm and time duration were 5600 and 5 min. In this case, all the suspended nanoparticles are removed from the suspension. UV spectroscopy visualized that this product contains both rods and spheres.

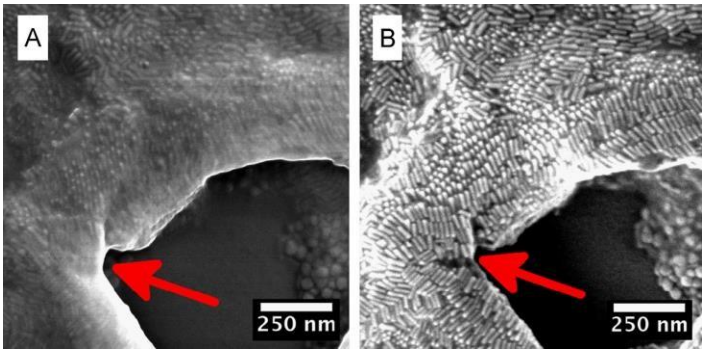


Figure 1. The results of HIM cover several layers of nano-rods.

These are the results of HIM and SEM. Fig 1 showed the results of HIM which cover several layers of nano-rods. Although the alignment of rods is visible, the blanket which has been formed in it makes the rods difficult to visible. Fig 2 showed the results of SEM which show the rods are easily visible, but CTAB is visible only in those locations which are very thick. Akbar et al., (2004) and Lee et al. (2008) did their research at the Korea research

institute of Chemical Technology and they adopted the simple method for their synthesis by using organic solvents via an iron-hydroxy oleate precursor. The material used in their work is Iron nitrate nonahydrate (99.5%), NH_3 (30% v/v), oleic acid, kerosene, hexa decane, and 2-methyl naphthalene. These all chemicals had been purchased from Samchum chemical from Germany.

India is also taking a part in the synthesis of nanoparticles of ferrites in which the National Metallurgical Laboratory, Madras center, and Center for Ceramic Technology are working in this field. Dr. B.R.V Narasimhan is the leader of this group, he used ferrous carbonate as a starting material for the synthesis of γ-Fe_2O_3 nanoparticles. He prepared the ferrous carbonate in his lab by using ferrous sulfate and sodium carbonate and filtered this from the bulk of the system. The precipitate which was obtained in this process was calcined at different rates 2,5, and 10^oC up to 500^oC. the iron oxide found in it is magnetite. This group wanted to investigate the synthesis methods of an allotropic form of Fe_2O_3 and draw the formation deadline between the alpha and gamma iron oxide nanoparticles.

In our experimental work, iron power and stoichiometric quantities of sulphuric acid were reacted. An aliquot of stoke was diluted and Na_2CO_3 solution at (70 ^oC) was added. The precipitated $FeCO_3$ was filtered and washed with warm distilled water. The wet precipitate was calcined in the electrical crucible at a heating rate mentioned above up to 500^oC. An experiment is also conducted for 500^oC. The air-dried precipitate was also calcined in the same conditions. The Iron oxide powder was washed at 110^oC for 6 hr. The light green stable precipitate was formed. With the addition of more alkaline, the precipitate was complete at a Ph of 9.5. All the cases mentioned in the table have a constant soaking temperature of 500^oC and the results of X- rays diffraction showed that a faster heating rate promotes the formation of γ-Fe_2O_3, from the table it is clear that decreasing the cooling rate form the mixture of **α** as well as **γ**-Fe_2O_3 and increasing the time of soaking promote the formation of alpha ferric oxide. The samples have different heat treatments and different heating rates. The decomposition of ferrous carbonate produces wustite that will oxidize to magnetite and undergoes further oxidation to form γ- ferrous oxide (Cullity, 1978).

The coating of iron oxide nanoparticles with silica was also took place at the National Institute of Technology, Rourkela. A student Basa (2009) was working on it for the fulfillment of the Master of Science in Chemistry. These silica-coated nanoparticles are used in "Green" packaging with nanoparticles, in food safety, cleaning up the environment, pharmaceutical application, Tissue engineering and catalysis. Basa (2009) used Ferric

chloride, Tetraethylorthosilicaate, conc. NH_4OH, ethanol solution, 25% ammonia, and conc. HCL.

Synthesis of iron oxide nanoparticles

For the synthesis of α-Fe_2O_3, $FeCl_3$ and HCL were mixed at 1:3(v/v), and the deionized water was added of Fe^{+3} is 0.01 mol dm^{-3} and was preserved at 96C^o for 24h, and then quench at room temperature. The resulting Iron oxide nanoparticles were the α-Fe_2O_3, which was orange-red. Similarly, "Merra" synthesis the silica nanoparticles by using tetraethylorthosilicate (TEOS) as a starting material in ethanol.

For the characterization, we used SEM EDAZ and measured the particle size. The particle size which is come in her research was less than 100nm (Basa, 2009). The doping of "Bi" in the iron oxide nanoparticles was also carried out under the supervision of Gajovic et. al. (2011). These researchers studied the effect of doping on the surface structure and morphology of iron oxide nanoparticles which can be used as a catalysis for the degradation of actual pesticides for the photo-Fenton like process. Iron oxide nanoparticles were prepared by hydrothermal heat treatment of the solution in which both iron oxide and bismuth salts were precipitated at high PH. These can be synthesized by two different methods co-precipitation and separate precipitation. It was seen that the crystallization of Iron oxide was obtained at 20% of bismuth.

In our work, the catalysis was prepared under hydrothermal reaction. The precipitation in two series was carried out which contain both Bi, and iron oxide. Their molar percentage varies from 20 moles%. The total mass of the salt in the whole solution is 3g. In the first procedure, the Bi $(NO_3)_3$ was dissolved in 10 mL of distilled HNO_3 by vigorous stirring before adding the $Fe(NO_3)_3.9H_2O$ and 35 mL of distilled water in it. At PH of 13.4, the sodium started to precipitate with the addition of tetramethyl ammonium hydroxide (TMAH). This (TMAH) was used for quality control.

Iron oxide and bismuth salts were precipitated and the Fe $(NO_3)_3.9H_2O$ dissolved in distilled water before precipitation with TMAH, while the $Bi(NO_3)_3$ was dissolved in HNO_3 before the addition of water and precipitation with TMAH. The phase composition can be calculated by using X-ray diffraction of CuK_α radiation in the 2θ from 10^0 to 70^0. The step size was 0.04^0. The morphologies and structure were visualized by using a high-resolution transmission microscope (TEM). Pakistan is also working on the synthesis of nanoparticles of iron oxide. Akbar et al., (2004) synthesized the iron oxide nanoparticles with the sol-gel method. They found the parameters which can control the size of the particles. Iron nitrate

was used as a precursor in 200 mL in quantity and it was gelated in 800 mL of mono hydrated citric acid solution as a ligand molecule and slightly distilled water as a solvent. The iron solution was dropwise added to the citric acid. The solution was heated to a temperature of 70^oC and stirred continuously until the gel was annealed at a temperature ranging from 180-$400C^o$, typically yielding 1.6g of Fe_2O_3 ranging in size from 22-56nm. X-ray diffraction and VSM were used for their characterization.

From the results of X-rays, it was seen that the samples which were annealed give a different pattern than that of other samples. Particle size was measured by using peak width broadening as a function of the size particle with the help of Scherrer's formula (Equation 1),

$$D = \frac{K\ \lambda}{\beta\cos(\theta)} \quad (1)$$

The chemicals used in this study are 30% HCl, 65% HNO_3, 1020 steel slab, 33% ammonia, distilled water (zero conductivity), $MnNO_3.2H_2O$(Manganese nitrate dihydrate), PVP, Urea, ethanol, and $AgNO_3$(Silver nitrate). All chemicals are used without further purification and belong to in analytical grade. All these chemicals are added according to stylometric quantity. Some chemicals were purchased from "PCSIR" and some from "Central Chemicals Lahore".

Our experiment was conducted in two different schemes one from the sonochemical method and the other by magnetic stirring and at the end, we compared the results of both.

Synthesis of "γ-Fe_2O_3" Nanoparticles by Sonochemical & Magnetic Stirring

The iron turnings were prepared by drilling holes in AISI 1020 steel (BECO PD 20) having a barma diameter of 6mm. We have dissolved these turnings in 30% HCl solution on a magnetic stirrer and solute it with 2-3 drops of HNO_3 were added to accelerate the reaction when all the turnings were dissolved in acid, the solution turned into blue color, this solution containing some undissolved carbon and some other impurities came from the steel alloy additions. Dilute by three times with distilled water having zero conductivity, followed by vacuum filter it. Store this $FeCl_3$ solution in air tight bottle to prevent it from vaporization. Prepared another solution of ammonia in zero conductivity water mix 33 mL of 33% ammonia in 67ml of water. Added $FeCl_3$ in this solution at a rate of 5 mL/min during sonication. From these black precipitates of γ-Fe_2O_3 have started to appear in it. Centrifuge the particles at the rate of 3000 rpm for 25 min in three stages and saved iron oxide in ethanol. A similar process in the same condition and environment was repeated, but in this case, using a magnetic stirrer instead of a sonicator.

Synthesis of" MnO" Nanoparticles by Sonochemical & Magnetic Stirring

Dissolved 10 mg of manganese nitrate dehydrate ($MnO.2H_2O$) in 200 mL of zero conductivity water and sonicate this at 20 kHz,100W/cm^2 for 2hr. Add 0.2gm of PVP (Polyvinylpyrrolidone)and 6 mg of urea of analytical grade and sonicate it for 5hr. The solution turned into light brown color. The solution in this position was a metastable state further sonicate the precipitation of $Mn(OH)_2$ was started. Before precipitation (mean in a metastable state) stored this in an airtight bottle.

Coating of "MnO" on Iron Oxide Nanoparticles

Coating of MnO on iron oxide was taken place by dropping formal solution with constant flow rate on iron oxide ethanol suspension. The basic quantity of iron oxide is 10 mg. Calculation shows how much quantity of suspension we take for 10 mg of iron oxide.

i. Weight of 25 mL iron oxide ethanol suspension = 22.897 mg
ii. Weight of particles = 22.897-20.582 = 2.315 mg
iii. Vol of particles = m/ρ = 0.4469
iv. 2.315 mg of particles are present = 25 mLof solution
v. And for the value of 5 mg the volume of solution is = 53.995 mL.

From the calculation, we observed that 107.99 mLof iron oxide suspension contains 10 mg of iron oxide nanoparticles, placed this in the sonicator at 80C^o and 20kHz frequency and add a formal solution (MnO) with biuret at 5 mL/min drop-wise and sonicate it for 4hr. The same process was repeated but in this condition we used a magnetic stirrer instead of a sonicator. Magnetic stirring was also performed for 2hr.

Synthesis and Doping of "Ag" Nanoparticles by Sonochemical & Magnetic Stirring

The synthesis of Ag nanoparticles was similar to MnO, but its storage was taken place in PVP solution because they become started to agglomerate in ethanol solution. For their synthesis dissolved $AgNO_3$ in 10 mL of deionized water which has zero conductivity and sonicate it for 4hr. Add this solution with the help of buret at 5 mL/min in sonicated MnO coated γ-Fe_2O_3 particles at the temperature of 50C^o and sonicate it for 3hr. A similar process was conducted for magnetic stirring.

Prepared the separate solution of 10 mL of PVP 5wt% in ethanol, and diluted it with 100 mL of ethanol. Saved these doped particles prepared by sonication and magnetic stirring.

Centrifuge these doped particles at 3000 rpm for 25min in 4 stages and stored these in air-tight bottles.

Sample Preparation

Prepared a total of six samples three from each scheme, one sample belonged to simple γ-Fe_2O_3, one was from "MnO" coated and the other from "Ag" doped on "MnO" coated sample. Similarly, three samples from magnetic stirring. Weight these samples for SEM. Sample weights were shown in Table 2.

Table 2. Sample weights

Sr#	Sample destination	Wt of Bottle	Wt of sample + Bottle	Wt of sample
1	Sa1	13.860	14.444	0.584
2	Sa2	14.114	14.315	0.201
3	Sa3	14.452	14.681	0.229
4	Sa4	14.526	15.156	0.63
5	Sa5	14.557	14.922	0.365
6	Sa6	14.296	14.674	0.378

Characterization Techniques

There are several techniques used for the characterization of different nanoparticles. In which BET, Particle size analysis, SEM (Scanning electron microscope). But in our research we do SEM. These tests are an experimental part of our project. And some tests we performed in the future are TGA, VSM, DSC, and TMA.

SEM (Scanning Electron Microscope)

SEM bombards a high beam of electrons on a surface that is under investigation (Kiss-Eross, 1976; Smith, 1997). In which the surface is scanned by using a beam of electrons (Kassman et al., 1997), and a reflected beam is collected which is come from the surface is scanned at the same rate on CRO. The image on the screen, which may be a photograph, represents the surface feature of the specimen. In which scanning coils allow the beam to scan a small area of the surface of the sample. Low angle backscattered electrons interact with the protuberances of the surface and generate secondary backscattered electrons to produce the electronic signal. This electronic signal is responsible for the development of the image. The resolution of many SEM is 5nm which is a very high magnification. This process is very useful for fractography of an intergranular corrosion fracture (Kiss-Eröss, 1976).

Scanning Process

A stream of the electron is generated on the surface of the electrode and is accelerated toward the metal surface by applying a potential gradient.

This stream of electrons is focused on the surface of an investigation utilizing a magnetic field (Kiss-Eröss, 1976).

SEM Analysis

The surface morphology of the iron oxide, coated iron oxide with MnO and Ag-doped MnO coated have been studied by scanning electron microscope. These are shown in these Figure 2 (a,b).

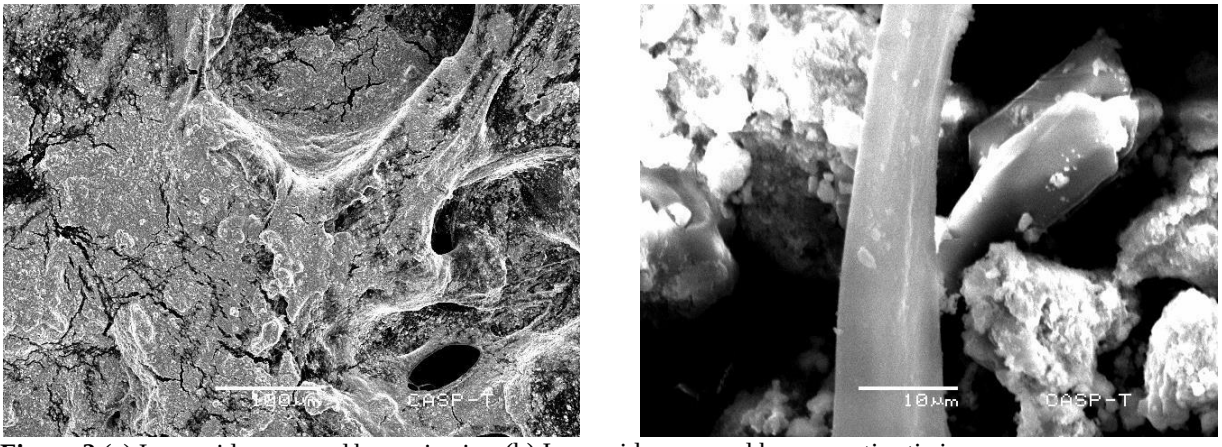
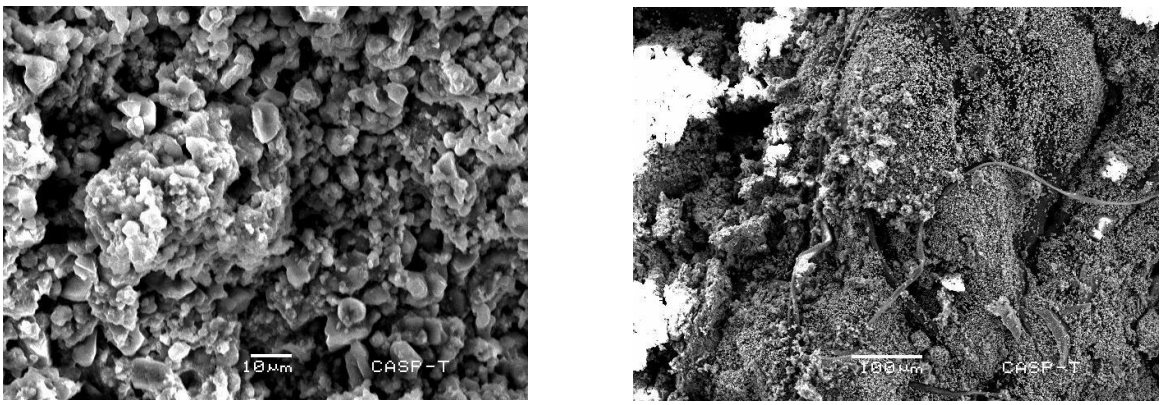


Figure 2 (a) Iron oxide prepared by sonication (b) Iron oxide prepared by magnetic stirring

The iron oxide nanoparticles which are prepared by sonication and magnetic stirring can be used as a core by developing a coating of MnO on it, by magnetic stirring and sonication separately.

Figure 3 (a) Iron oxide MnO coated prepared by sonication (b) Iron oxide MnO coated prepared by magnetic stirring



After coating MnO on Iron oxide nanoparticles, the doping was done with “Ag” and this doping was taking place by using silver nitrate having water of crystallization of 2. The microstructure obtained after doping with sonication and magnetic stirring can be shown in the Figures 5 and 6 from which we observed that “Ag” attaches to the surface.

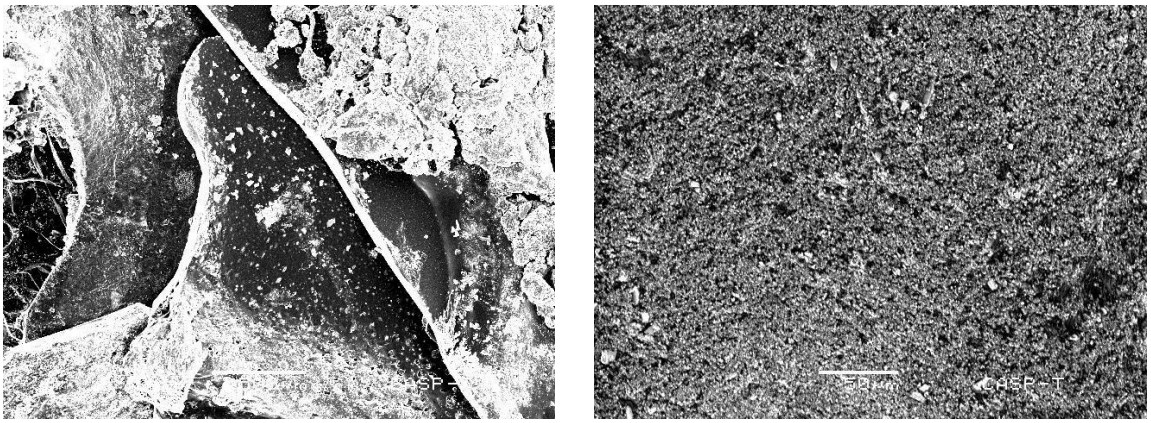


Figure 4 MnO coat Ag-doped iron oxide nanoparticles prepared by Magnetic stirring process

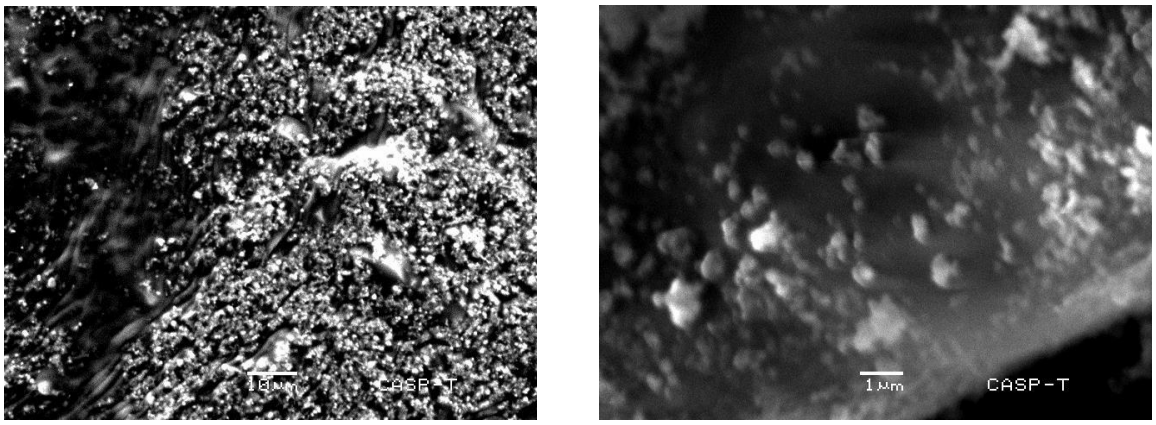


Figure 5. MnO coat Ag-doped iron oxide nanoparticles prepared by sonication process**.**

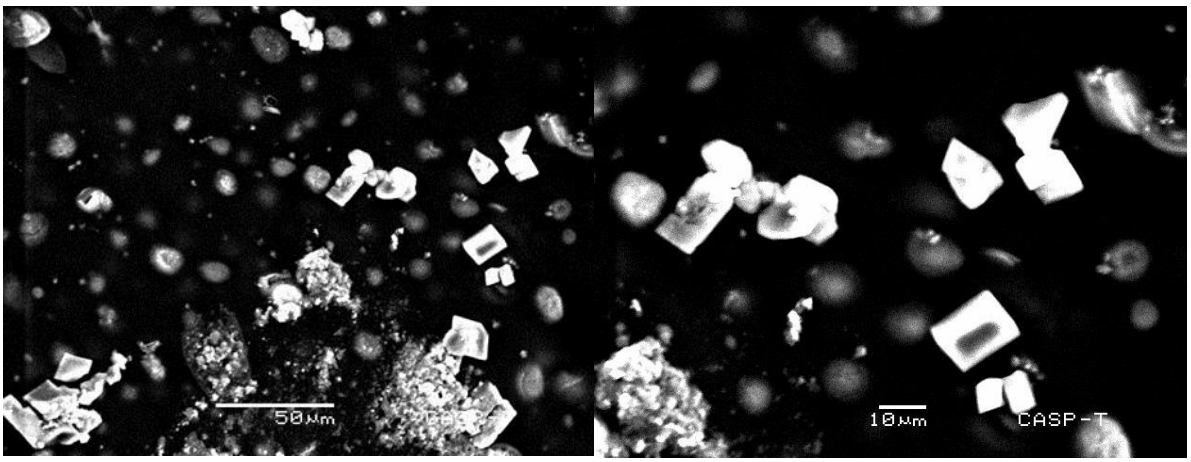


Figure 6. MnO coat Ag-doped iron oxide nanoparticles Single-crystal obtain in sonication process.

Conclusion

The main conclusion of this work is summarized below:

1. We have successfully synthesized γ-Fe_2O_3 particles by sono chemical and magnetic stirring method.
2. MnO-coated iron oxide nanoparticles can be synthesized by using manganese nitrate.
3. We easily doped "Ag" with silver nitrate.
4. The core-shell particles can easily be characterized by SEM.
5. From the particle size analyzer it was proved that the particle size was below 100 nm, which fulfilled the definition of nanoparticles.
6. From SEM we observed that the particles were amorphous.

Future work

1. Find and validate the application in the medical field.
2. VSM, TGA, DSC, TEM, and EDX are the tests that are planned to be included in future work.
3. Use as a radar absorbing material.
4. Apply the coating of the transformer core.
5. Used in Hair transplant.

Statement of Conflict of Interest

Authors have declared no conflict of interest.

Author's Contributions

The contribution of the authors is equal.

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