Research Journal of Chemical Sciences _____ ISSN 2231-606X
Vol. **3(8),** 18-28, August **(2013)** *Res. J. Chem. Sci.*

A Simple and Effective method of the Synthesis of Nanosized $Cu_{0.5}Zn_{0.5}Fe_2O_4$ Particles

Avnish Kumar Arora and Ritu
Department of Chemistry, Maharishi Markendeshwer University, Mullana -133203, Haryana, INDIA

Available online at: www.isca.in
Received 24th June 2013, revised 1st August 2013, accepted 14th August 2013

Abstract

Nanosized Copper-zinc ferrites $Cu_{0.5}Zn_{0.5}Fe_2O_4$ have been synthesized by aqueous precipitation method and characterized by using XRD (X-ray diffraction), TGA/DTA (Thermo gravimetric analysis), SEM (Scanning Electron Microscopy)/ TEM (Transmission Electron Microscopy) and magnetic measurements by using VSM (vibrating sample magnetometer). XRD studies confirm the formation of cubic spinnel structure. SEM (scanning electron microscopy)/ TEM (transmission electron microscopy) was used to characterize the microstructure of the ferrite samples. A homogeneous and fine grain microstructure was found. Magnetic measurements shows that $Ni_{05}Cu_{0.5}Fe_2O_4$ is super paramagnetic in nature at room temperature and hence used in magnetic device. The particle size of synthesized $Cu_{05}Zn_{0.5}Fe_2O_4$ varied from 18nm to 68nm which is good agreement of the theoretically predicted size of nanomaterials. The method is easier more effective and convenient in comparison to the known methods of the synthesis $Cu_{05}Zn_{0.5}Fe_2O_4$ nano materials like combustion synthesis, thermal cracking and conventional ceramic methods.

Keywords: Nanosized, Spinel, ferrites, Super paramagnetism, copper – zinc Ferrite ($Cu_{05}Zn_{0.5}Fe_2O_4$)

Introduction

$Cu_{05}Zn_{0.5}Fe_2O_4$ is a spinel ferrite. The general formula of the spinel ferrite is $MeFe_2O_4$ where Me usually represents one or, in mixed Ferrites more than one of the divalent transition metals Mn, Fe, Cu, Ni, Zn, Ca or Mg and Cd. Other combinations of equivalent valency are possible and it is also possible to replace some or all the trivalent iron ions with other trivalent metal ions[1-6]. Ferrites are the fundamental functional materials of electronic industry. Ferrites can be cast into complex shapes and can be ground and will take fine finish. Ferrites are ceramic materials. Ferrites may be defined as magnetic materials composed of oxide containing ferric ions as the main constituent. This term is often restricted to materials which have cubic crystal structure of spinel but now days it is also applied to magnetic oxides. Ferrous ferrites are an example of naturally occurring ferrite. Magnetic properties in ferrites arise from interactions between metallic ions occupying particular positions relative to oxygen ion in the crystal structure of the oxide.In majority of the present day magnetically soft ferrite the crystal structure is cubic and has the form of mineral spinel. $Cu_{05}Zn_{0.5}Fe_2O_4$ is developed for a wider range of applications where high permeability is the main requirement. The ferrite core memory was the basis of the IBM 360 computer which is standard for industry[6-15]. The application started in the field of telephony transmission operating in large frequency range from about 40 KHz. Most important use of ferrite is for video recorder[15-21]. Today ferrites are used as noise filters in power lines of electronic equipments[22]. Ferrites are easy to manufacture, low costs, small volume, high efficiency and with greater uniformity have applications in ceramic magnet as medical treatment, filter inductors, magnetic amplifiers, transformers, antenna cores, magnetic memories and flyback transformers.

Material and Methods

Chemicals: All chemicals used in the experiment are analytic reagent grade. Ferric nitrate Fe $(NO_3)3$, Copper NitrateCu $(NO_3)_2$, Zn $(NO_3)_2$ and liquor ammonia were purchased from Merck, India. Deionized water was used throughout the experiment.

Synthesis of Cu 0.5Zn0.5Fe_2O_4: 200 ml of 0.5 M solution of $Fe(NO_3)_2$ was mixed with 200 ml of 0.5M solution of $Zn(NO_3)_2$ and 200 ml of 0.5M solution of $Cu(NO_3)_2$ then aqueous ammonia was added drop wise with constant stirring until the pH of the solution reached to 10. The precipitate thus obtained were filtered on buckner funnel and washed several times with distilled water. The precipitate was dried in oven at 70^0C for 24 hrs and was calcined at 600^0C in a muffle furnance for 5 hours. Obtained material was ground and sieved through 100 mesh size sieve.

Equipments: An X – ray measurement was carried out using X-ray diffractometer system Philips PW 11/90, with nickel filtered CuKα (l = 1.5405 Å).

The crystalline size of Zinc ferrite was calculated using Scherrer equation.

$t = K\lambda / B \cos\theta$

Where t is the average crystallite size of the phase under investigation, K is the Scherrer constant (0.89), λ is the wave length of X – ray beam used, B is the full-with half maximum (FWHM) of diffraction (in radians) and θ is the Bragg's angle.

Transmission electron micrograph (TEM) were recorded on Hitachi H7500. The samples were dispersed in ethanol and then treated ultrasonically in order dispersed individual particles over a gold grid. The surface morphology of $Ni_{05}Cu_{0.5}Fe_2O_4$ prepared by precipitation method was investigated by using scanning Electron Microscope Quanta 200 FEG (FEI Netherlands).

The magnetic properties of the solid were measured at room temperature using a Vibrating sample Magnetometer Model 155.

Results and Discussion

X-ray studies: The X-ray diffraction pattern of synthesized $Cu_{05}Zn_{0.5}Fe_2O_4$ nano particlesis depicted in figure 1. X-ray diffraction pattern of $Cu_{05}Zn_{0.5}Fe_2O_4$ pure indicated that Cu-Zn ferrite in the form of $Cu_{05}Zn_{0.5}Fe_2O_4$ (figure 1). In X-ray diffraction, some prominent peaks were considered and corresponding d-values were compared with the standard i.e. JCPDS (Joint Committee on Powder Diffraction Standards) (File card 86.) Table 1. X-ray diffraction shows that metal oxide is pure $Cu_{05}Zn_{0.5}Fe_2O_4$ having cubic spinal structure . Table1 gave a verification of well defined crystalline phase of spinel structure of Cu-Zn ferrite. The average crystallite size was determined from the broadening of the most intense peaks using Debye Scherer equation (Cullity 2001) and values shown in table 1 and it also supports the SEM/TEM observations. The crystallite size was found within 18-68 nm. The powder x-ray patterns confirm the single phase spinel structure for synthesized material. Thickness of the crystal has been calculated using Debye Scherrer's formula and it support the TEM observations.

Thermal Analysis: Thermal analysis includes a group of techniques in which a physical property of a substance is measured as a function of temperature or time while the substance is subjected to a controlled temperature programme. The analysis involves thermogravity (TG), differential thermal analysis (DTA) and derivative Thermogravimetry (DTG). Thermal Gravimetric studies of the calcined oxides prepared were done between a temperature range of 10-1000^{0}C under N_2 atmosphere. The TGA/DTA curves of the oxides are shown in figure 2. The maximum total weight loss observed for Nickel oxide and their corresponding temperature is summarized in table 2 . Results showed that in the synthesized oxides shows some weight loss and ferrite undergoing decomposition, dehydration or any physical change. In DTA curve also, there is exothermic peak which shows phase transition, solid state reach on any chemical reaction occurred during heating treatment.

SEM/TEM studies: SEM studies were carried out to study the morphology of the sample figure (3a,b) shows the SEM micrographs of $Cu_{05}Zn_{0.5}Fe_2O_4$. The micrographs of $Cu_{05}Zn_{0.5}Fe_2O_4$ (figure 3a,b) displayed spherical particles with high agglomeration. TEM studies were carried out to find the exact size of the synthesized $Cu_{05}Zn_{0.5}Fe_2O_4$ nanoparticles fig. (4a,b,c,d,e,f,g) shows the TEM images of the synthesized $Cu_{05}Zn_{0.5}Fe_2O_4$ nanoparticles. It shows that the size of the obtained nano particles is in the range 18-68 nm. Most of the particles are in the range 21-50 nm. TEM images indicate that $Cu_{05}Zn_{0.5}Fe_2O_4$ samples were all spherical particles with uniform grain size distribution.

Magnetic measurements: Magnetic properties of nanometer sized particles have attracted considerable attention in recent years because of their unique properties. The size of the magnetic particles decreases below a critical length. Domain formation was no longer energetically favoured and the particle existed as a single domain. Magnetic nanoparticle has aroused increasing interest among researchers of various fields due to their extensive applications such as in information storage system, medical diagnostics, ferrofluid technology etc. This is mainly because of the properties of nanoparticles differ from those of the corresponding bulk material. The magnetic measurements of $Cu_{05}Zn_{0.5}Fe_2O_4$ was carried out at room temperature and it has been shown that the magnetic measurements shows that the prepared $Cu_{05}Zn_{0.5}Fe_2O_4$ nanoparticles posses good super paramagnetic behavior at room temperature (300K) with saturation magnetization MS value 51 (figure 5). Previously reported values of MS For $Cu_{05}Zn_{0.5}Fe_2O_4$ Nanoparticles prepared by various methods has been reported in table (2). The value of MS ranging from 15-90 emu/g shows that Ms strongly depends on the methods of synthesis.

This MS value at room temperature is good and comparable with methods of synthesis as thermal decomposition method (MS value 43 at 300 K and MS value 68.5 emug-1 at 10k) ball milling (MS value 20.7 in at 4.2k) and other co-precipitation routes which shows a maximum MS 47.8 at 4.2 k (figure 5). Magnetic Hysteresis curve clearly indicate the soft nature of the prepared sample saturation magnetization ms value increase with time.

Conclusion

$Cu_{05}Zn_{0.5}Fe_2O_4$ nanoparticle with cubic spinel structure is synthesized successfully by aqueous precipitation method. From SEM/TEM studies it is found that particles have average size 18-68nm. Magnetic measurements shows that $Cu_{05}Zn_{0.5}Fe_2O_4$ super paramagnetic in nature having saturation magnetization (MS) value 51 emu/g. This method is beneficial over the existing methods of synthesis of nano particles because other methods require expensive materials, highly skilled labour and specialized instrumentation. Therefore, the proposed precipitation method is cheaper, easier, very promising and may have extensive applications.

Table-1
X-RAY Diffraction Data for $Cu_{05}Zn_{0.5}Fe_2O_4$

S. No.	d=λ / 2sinθ (Observed)	d=λ / 2sinθ (Reported)	$I/I_0X100\%$ (Observed)	$I/I_0X100\%$ (Reported)
1.	2.749624311	2.749624302	55.75575576	55.75575570
2.	2.814146374	2.814146360	39.33933933	39.33933930
3.	3.674620393	3.674620378	99.99999999	99.99999999
4.	2.311895273	2.311895271	19.7197197	19.7197187
5.	1.91106274	1.911062777	28.5285285	28.5285283
6.	1.837310919	1.837310900	25.3253253	25.3253245
7.	1.580591523	1.580591521	3.6036036	3.60360366
8.	1.4772508	1.477250000	1.5015015	1.50150166

Table-2
Observations of Weight Loss for $Cu_{05}zn_{0.5}fe_2o$ at Corresponding Temperature Range

Sr.No.	Maximum % loss in weight	Temperature range
1	1.696%	26.68-907.73

Table-3
Practical Size of Synthesized $Cu_{05}Zn_{0.5}Fe_2O_4$ at Different Scales

S.No.	Scale (20nm)	Scale (20nm)	Scale (50nm)	Scale (50nm)	Scale (50nm)	Scale (100nm)	Scale (100nm)
1	28.42105263	18	41.6667	41.891891	29.166666	25	25.641025
2	50.52631579	21.7931	47.9166	21.621621	41.666666	21.4285	25.641025
3	60	36.2413	56.25	68	54.166666	22.8571	30.769230
4	31.57894737	50.3793	64.5833	36.486486	54.166666	37.2857	33.333333
5	44.21052632	24.1379	50	33.783783	66.666666	40.1428	33.333333
6	36.84210526	25.5517	52.0833	18.918918	43.75	28.5714	33.333333
7	57.89473684	18	39.5833	31.081081	41.6666	35.7142	38.461538
8	28.42105263	32.7931	41.6667	36.486486	29.166666	28.5714	41.02564
9	50.52631579	36.2413	47.9166	33.783783	41.666666	28.5714	18
10	60	20.3793	56.25	18.918918	54.166666	25	20.512820
Range	28.42105263 nm to 60 nm	18nm to50.3793nm	41.6667nm to 64.5833nm	18.918918nm to 68nm	29.166666nm to 66.666666nm	21.4285nm to 40.1428nm	17 nm to 41.02564 nm

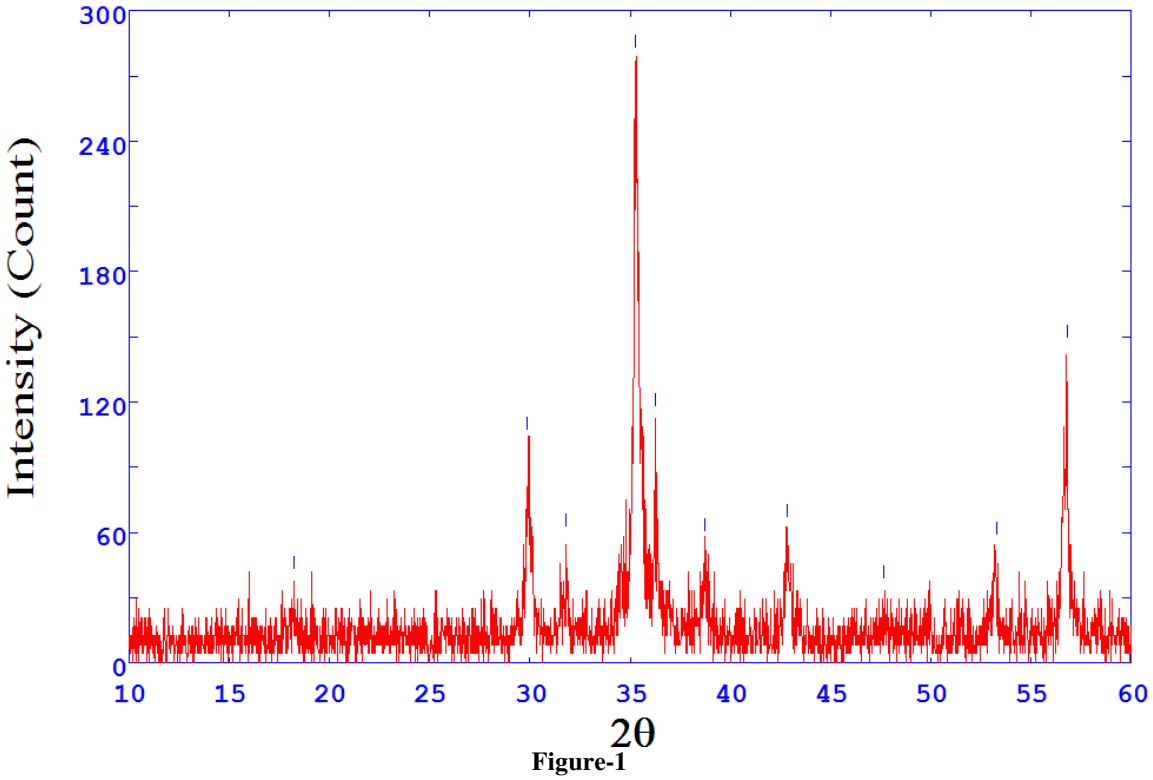


Figure-1
X-Ray diffraction spectra of $Cu_{05}Zn_{0.5}Fe_2O_4$ Particles

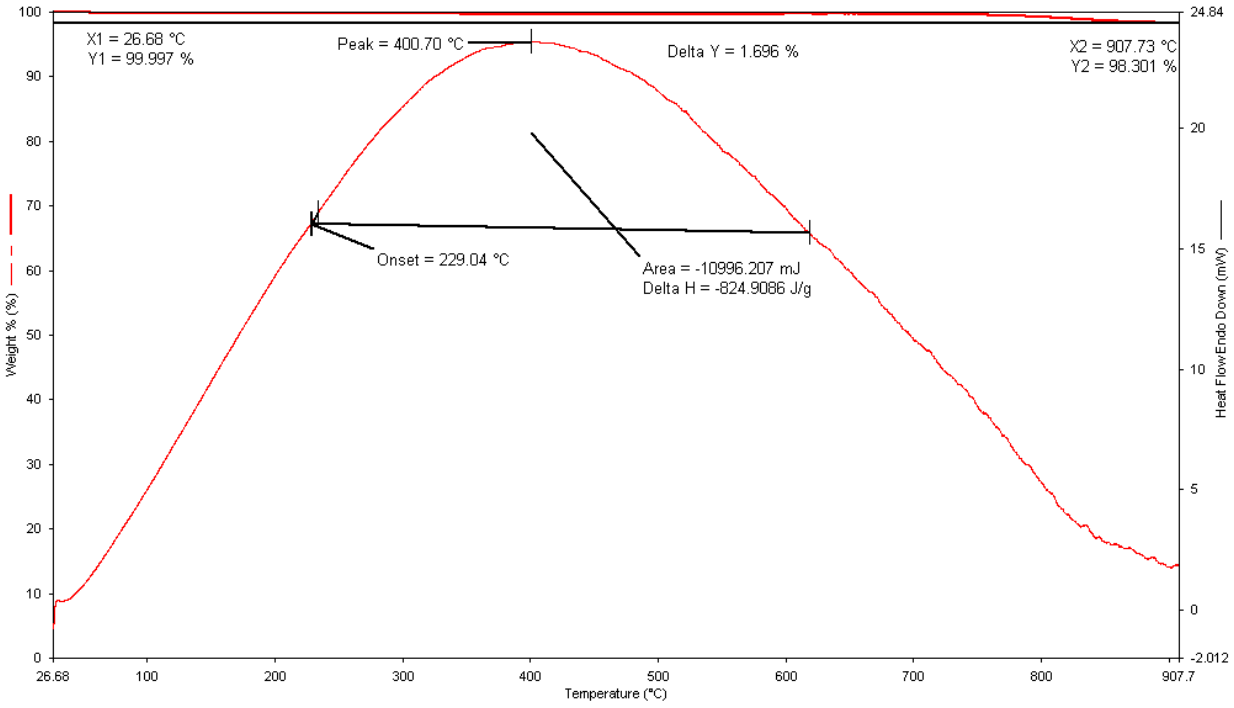


Figure-2
TGA-DTA of $Cu_{05}Zn_{0.5}Fe_2O_4$Particles

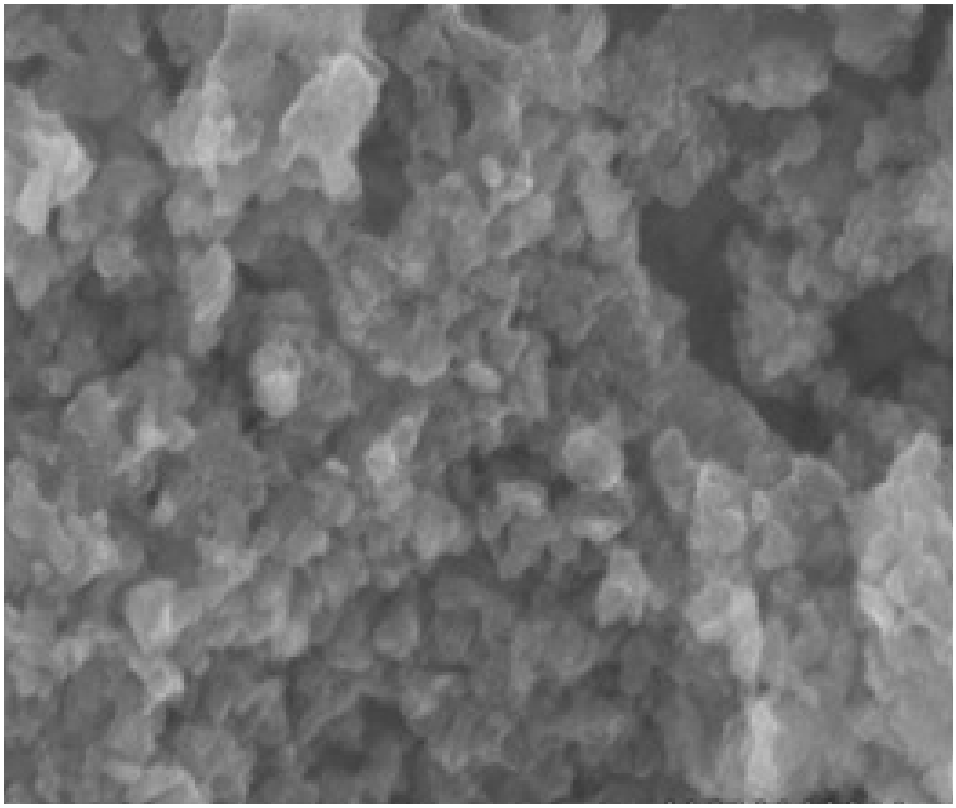


Figure-3(a)
SEM micrographs of $Cu_{05}Zn_{0.5}Fe_2O_4$ Particles

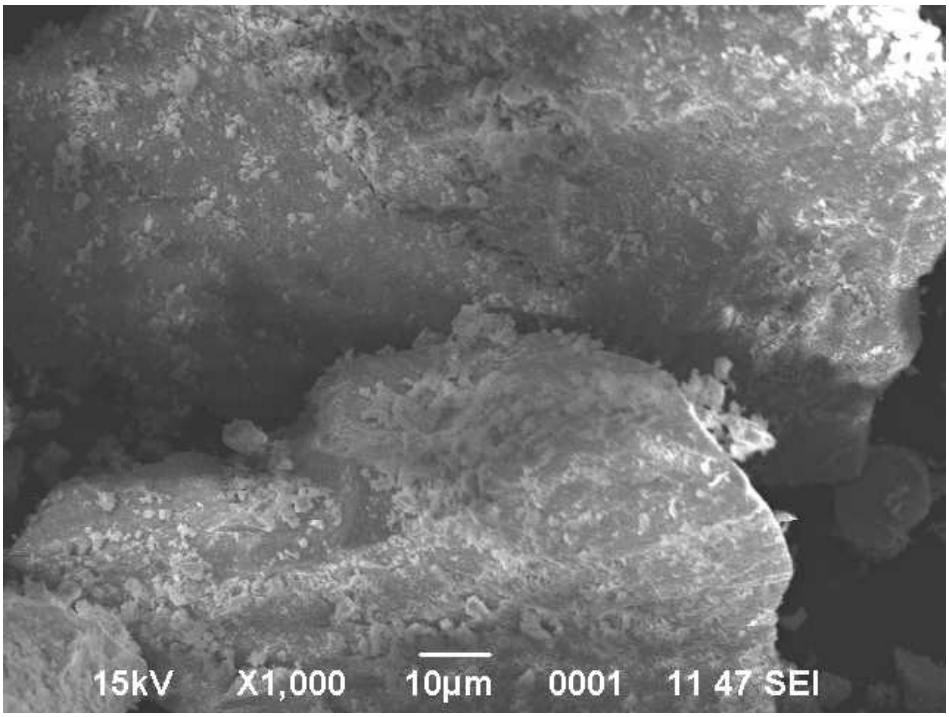


Figure-3(b)
SEM micrographs of $Cu_{05}Zn_{0.5}Fe_2O_4$ Particles

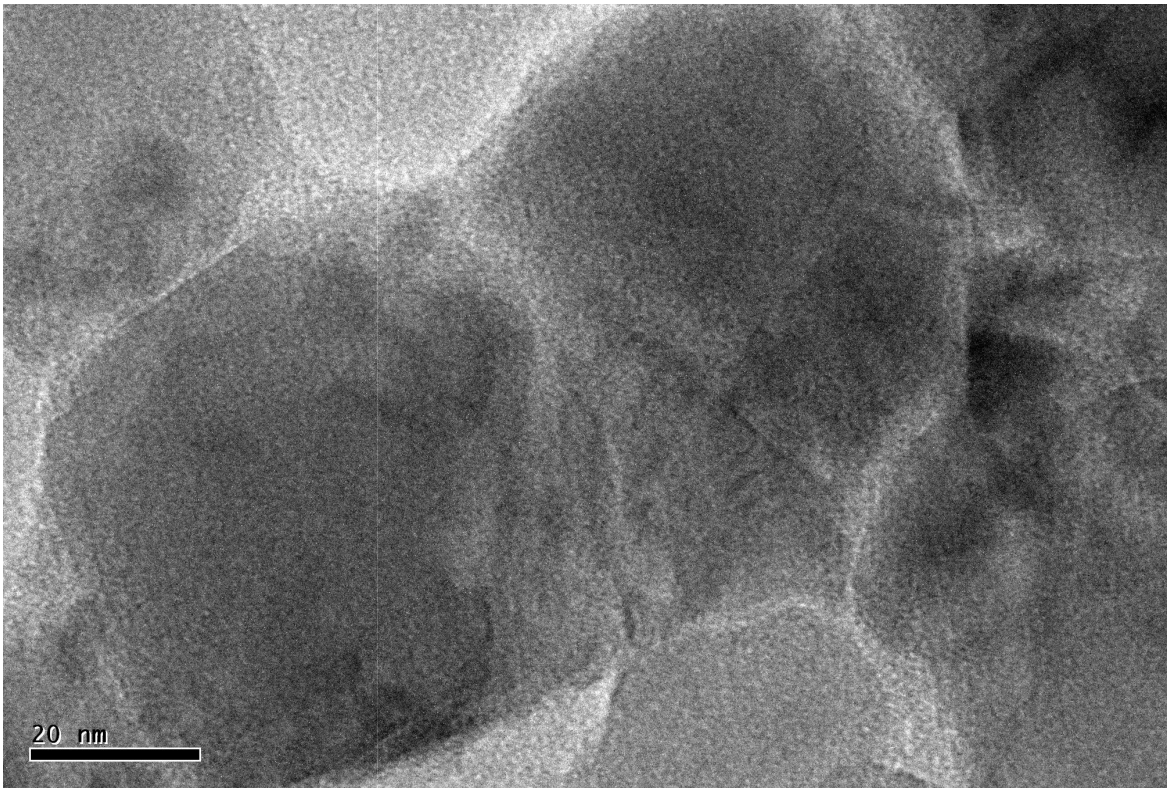


Figure-4 (a)

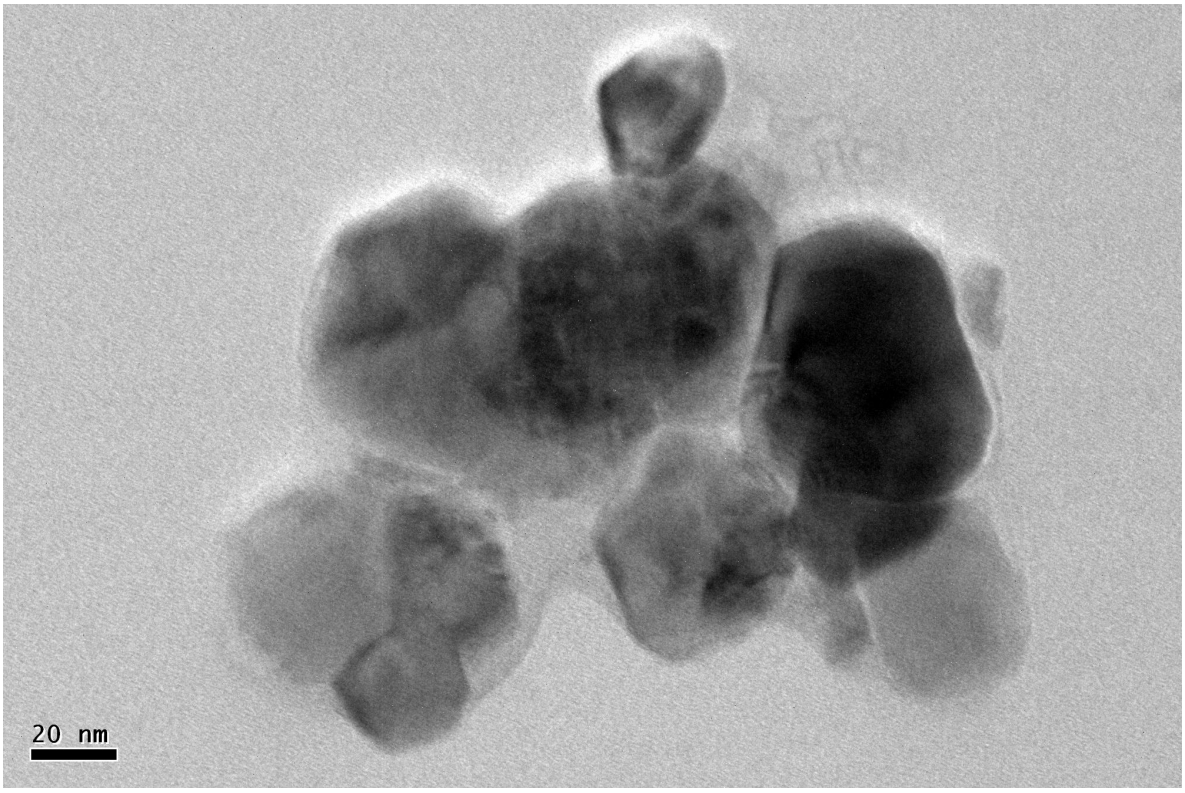


Figure-4 (b)

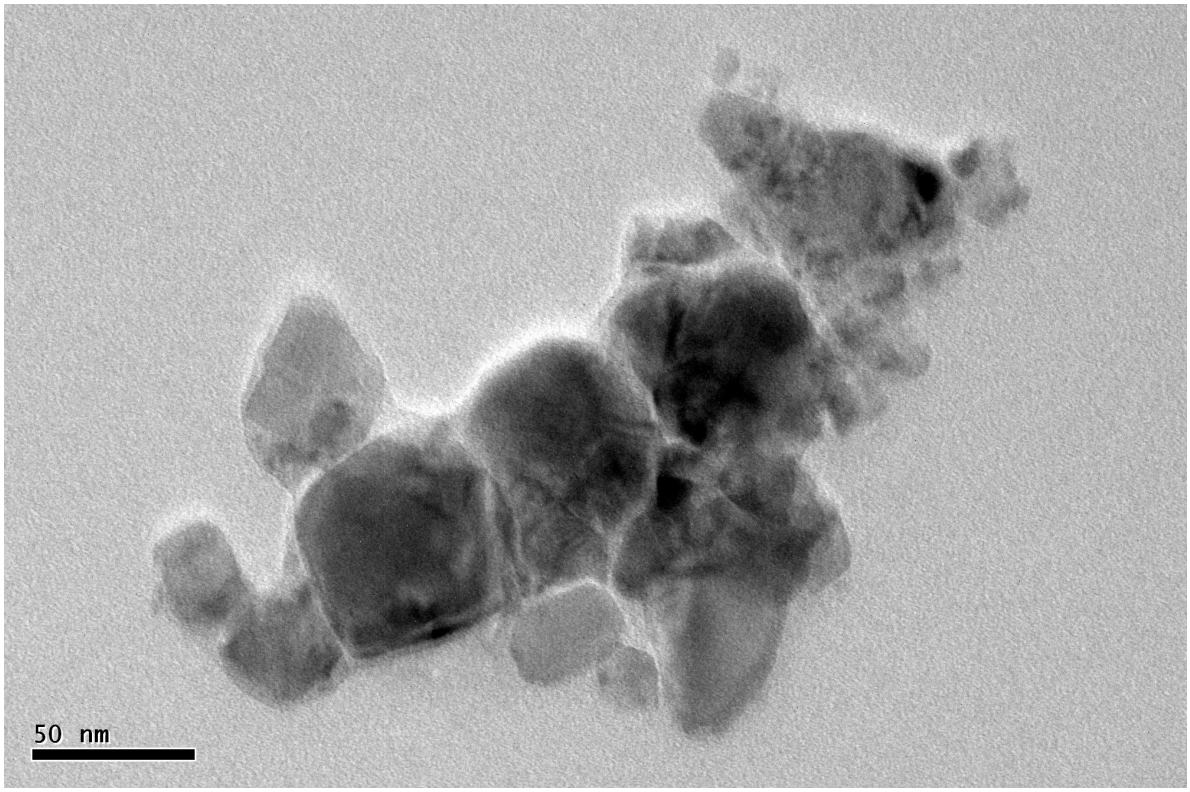


Figure-4 (c)

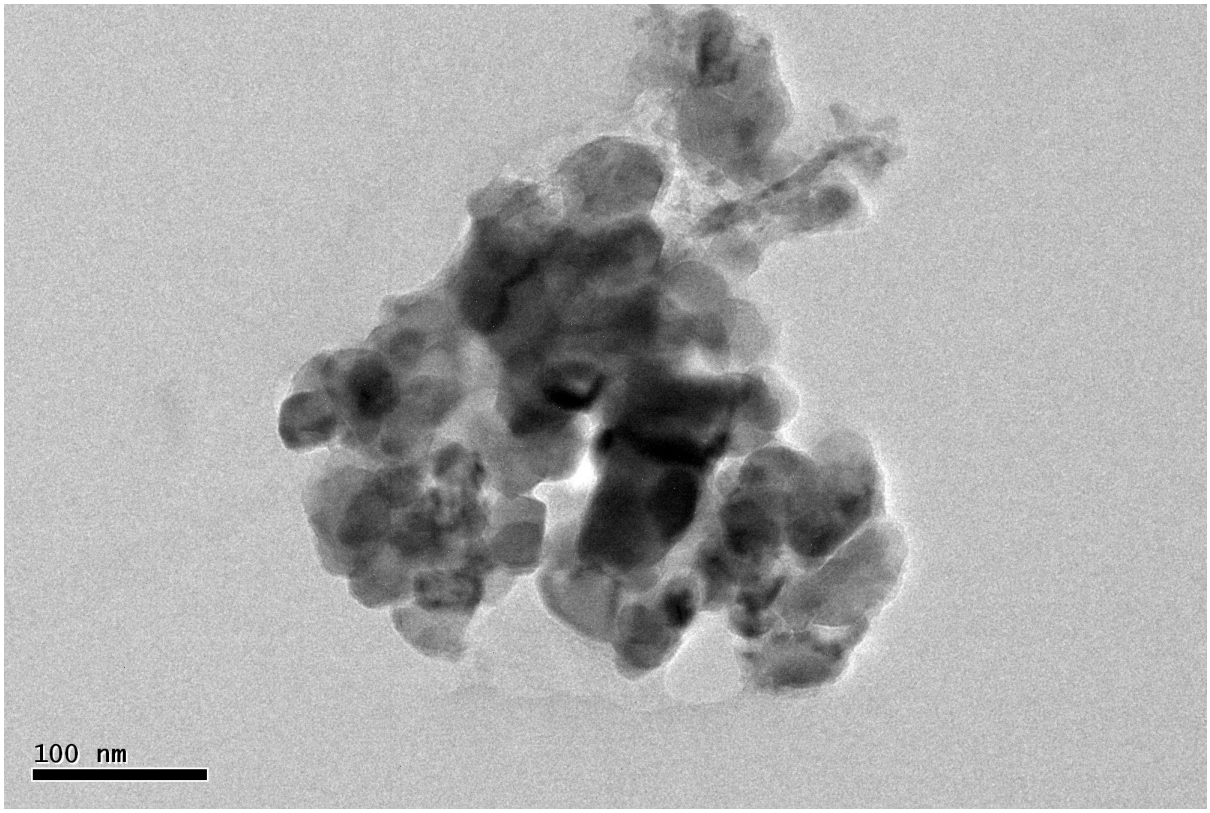


Figure-4 (d)

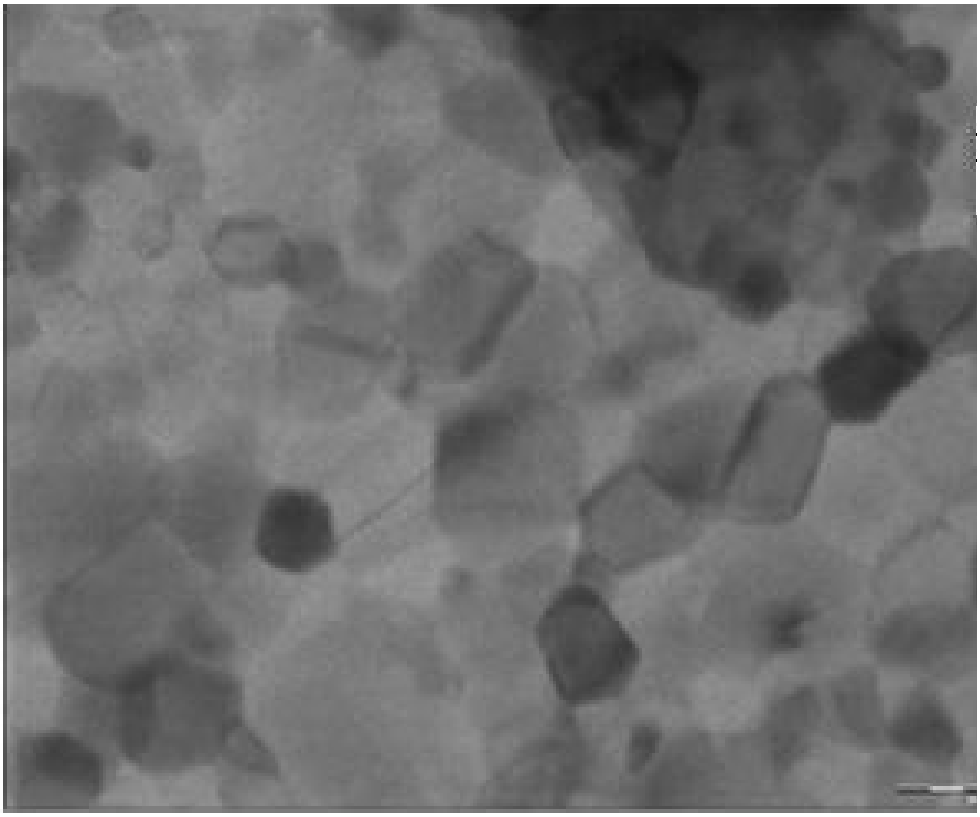


Figure-4 (e)

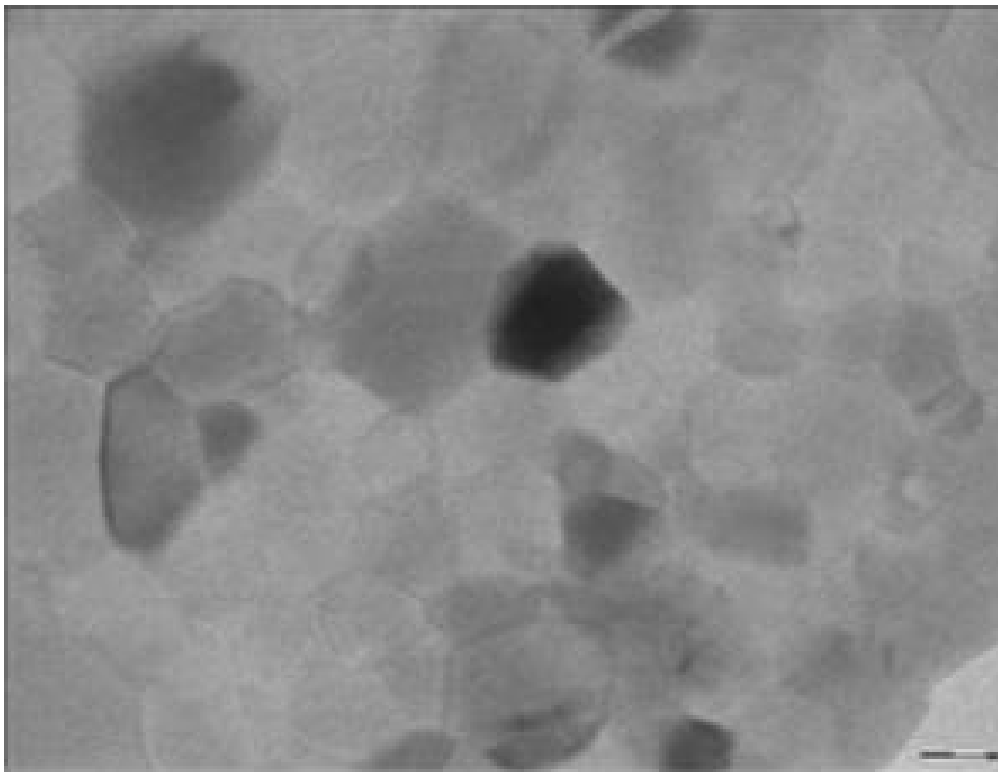


Figure-4 (f)

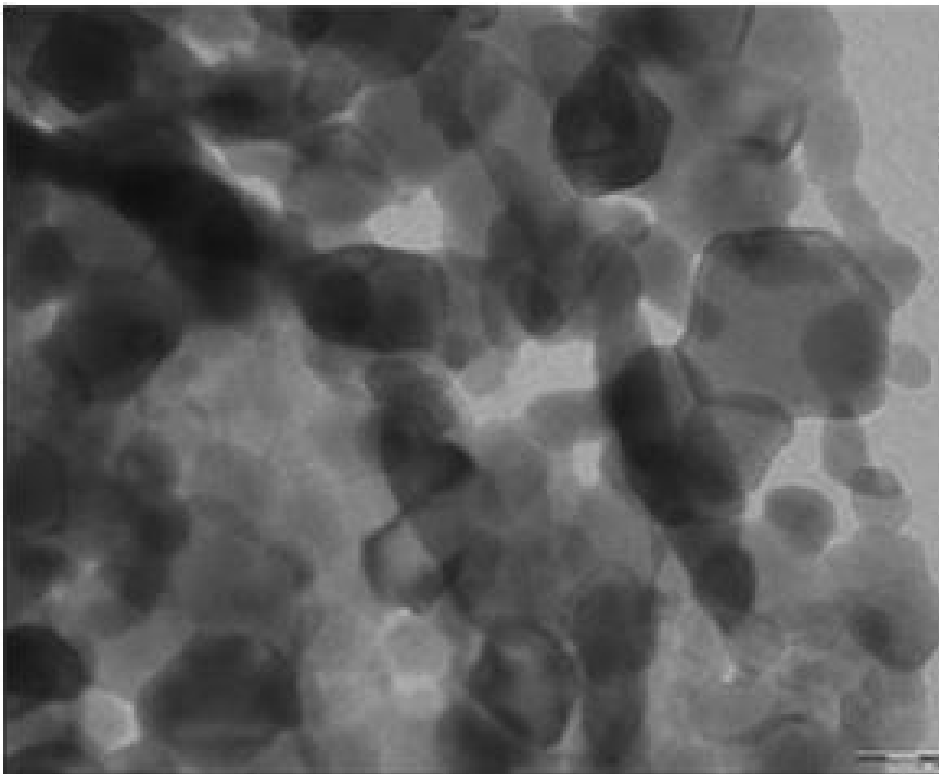


Figure-4 (g)

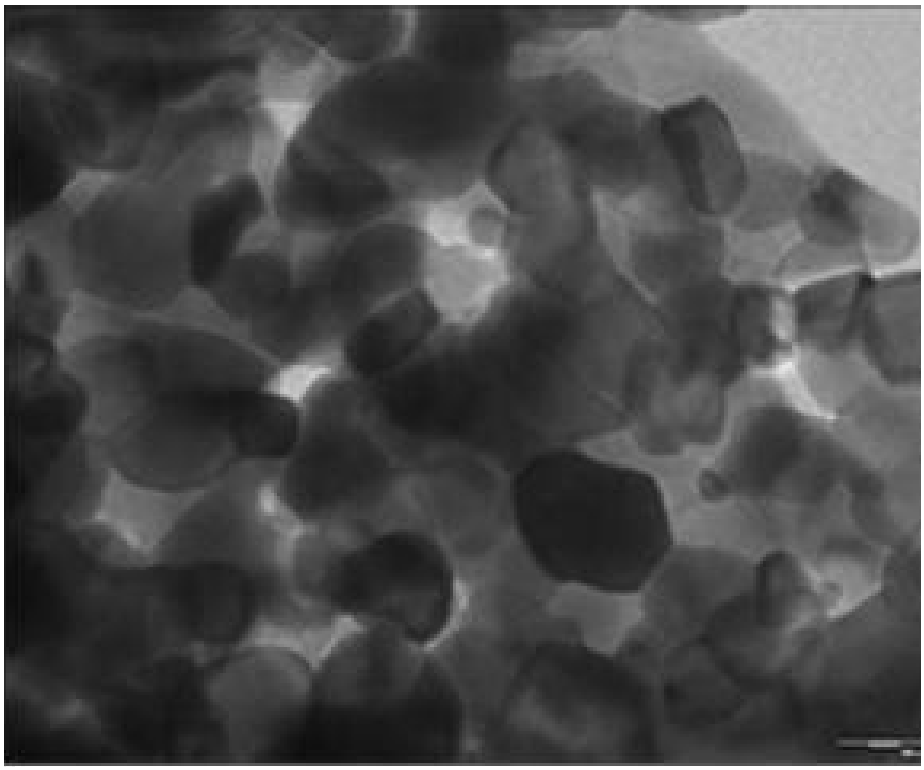
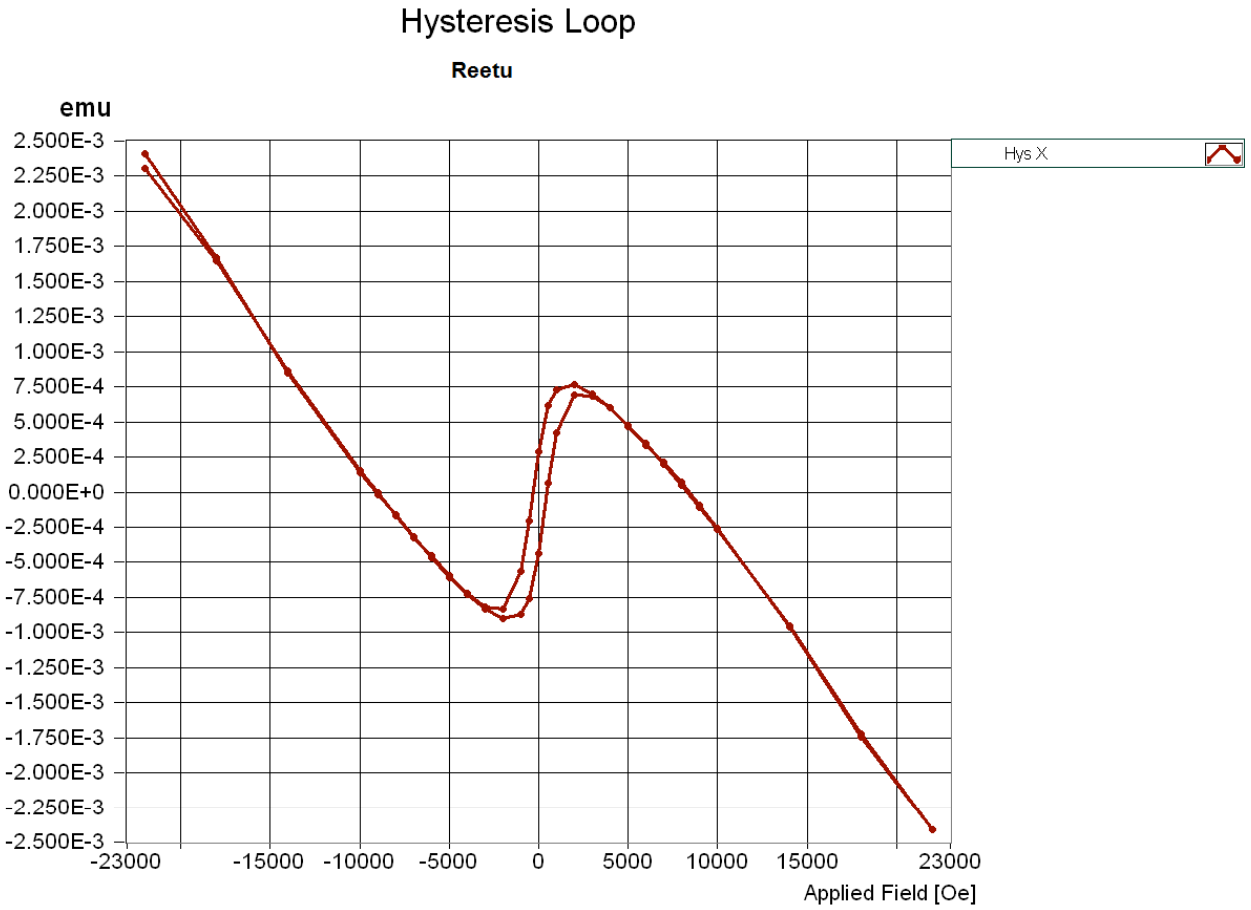


Figure-4 (h)
Figure-4 (a, b, c, d, e, f, g, h)
TEM micrographs of $Cu_{05}Zn_{0.5}Fe_2O_4$ Particles



Sample Name : 10

Field Angle: 0.00 [deg]

Parameters

	Upward Part	Downward part	Average	Parameter 'definition'
Hysteresis Loop				Hysteresis Parameters
Hc Oe	-22004.000	-22000.000	-2.000	Coercive Field: Field at which M//H changes sign
Mr emu	-437.089E-6	282.260E-6	359.675E-6	Remanent Magnetization: M at H=0
S	0.181	0.117	0.149	Squareness: Mr/Ms
S*	1.069	1.121	1.095	1-(Mr/Hc)(1/slope at Hc)

MicroSense EasyVSM

Figure-5
Magnetic measurement of synthesized $Cu_{05}Zn_{0.5}Fe_2O_4$ particles

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