

Ultra-large-scale, fast, one-step and facile Synthesis of Iron, Nickel, Cobalt, Manganese and Zinc Oxide Nanoparticles by a new and one pot Solution Combustion synthesis method

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Research Article

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Abstract

Here, we report one-step, new, low cost, fast and innovative synthesis by solution combustion synthesis (SCS) method, in order to ultra-large-scale synthesis magnetic nanoparticles of Fe₃O₄, NiO, Co₂O₃, ZnO and Mn₃O₄. The synthesis, was performed in the temperature 350 ° C in a nitrogen atmosphere and in the presence of poly maleic acid as dispersant and fuel agent. Ammonium nitrate, was used as a gasser agent in acidic medium of HNO₃. FTIR and UV-Vis spectroscopy were used for study spectroscopic properties of SCS product nanomaterial. Field emission scanning electron microscopy (FESEM), Energy-dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD) were used to study the microstructure and phase of nanomaterial. VSM for magnetic properties and modified Scherer method, used to determine particle size. This study provides a safe, one-step, facile, large-scale and general method for the synthesis of metal oxide nano materials. Due to the possibility of forming a single layer of synthesized nanoparticles, the biggest advantage of this method compared to other combustion synthesis methods is to obtain nanoparticles with relatively the same particle size. This is a unique result, which is not seen in the combustion methods of nanoparticle synthesis.

Introduction

Without a doubt, one of the greatest scientific challenges in the field of nano-technology is synthesis of low-cost, high-quality and high-scale of nano materials. In recent years, many efforts have been made for this purpose.¹⁻⁴ In the past several years, there has been an increasing amount of research on Nanometer-scale materials with the size of 1 to 100 nm due to their unique optical, magnetic, and chemical properties and a variety of applications in diverse fields of industry such as micro technology, biotechnology, energy storage, fuel cells, surface coatings, electronic devices, likewise utilization as catalysts.⁵⁻⁸

Nanoparticles, in particular magnetic nanoparticles are attractive purpose owing to their exclusive characteristics and they have taken into consideration not only for their magnetic and catalytic⁹ properties but also for their possible applications in numerous fields¹⁰. And a lot of effort has been focused on preparing magnetic nanoparticles with high functionality by many researchers. Magnetic nanoparticles modified with organic molecules have been widely used for biotechnological and biomedical applications such as cell separation, drug delivery, and magnetic resonance imaging due to their properties, which can be magnetically controlled by applying an external magnetic field.^{11, 12}

Over the past few years there are many investigating on magnetic cobalt and nickel nanoparticles that, they have potential to be used in biomedical applications and etch other.¹³⁻¹⁶

They display very attractive features and are also less sensitive to oxidation and therefore maintain stable magnetic responses. Because of their unique properties, magnetic metal Oxides of iron, nickel, and cobalt can be used for many applications in heterogeneous catalysts, gas sensing, information storage systems and so, was very attractive research.^{17, 18}

Several techniques have been employed to producing nano materials. ¹⁹⁻²³ Also magnetic nanoparticles have been produced such as microwave irradiation²⁴, mechanochemical-hydrothermal method²⁵, sol-gel process¹⁷, coprecipitation²⁶, freeze drying²⁷, sonochemical procedure²⁸ and so on, which need some special instruments, high temperature in order to calcination, and also spend so long time, to achieve desired composition and structure of slightly nanomaterial.

Combustion methods for example, solution combustion synthesis(SCS) or self-propagating high-temperature synthesis(SHS), volume combustion synthesis (VCS), Chemical looping combustion (CLC) and Carbon combustion synthesis(CCSO), have been utilized as replaced procedures due to their numerous advantages Compared with other methods proverbially low temperature processing, simplicity of process, and short reaction time. ²⁹

It is possible to produce a variety of nano-sized materials by using SCS method. An oxidizer, the right temperature, and a fuel such as urea, glycine, and various hydrocarbon chains including amino, hydroxyl, carboxyl and other reactive groups, are needed. Generally, the solutions of metal nitrates apply as oxidizer in SCS. Decomposition of metal nitrates in solution constructs oxygen-containing species, which react with fuel leading to appropriate conditions for rapid high-temperature interaction. In this process, formation of various gases prevents particle size growth resulting nano sized powders with high specific surface area. ^{30,31}

In present paper, we used a new SCS method in order to synthesis metal oxide nanoparticles of iron, nickel, cobalt, manganese, and zinc (Fe_3O_4 , NiO, Co_2O_3 , Mn_3O_4 and ZnO). The results showed that, innovative proposed new method could be used to produce relatively large scales of all metal oxide nanoparticles.

Results And Discussion

Combustion synthesis or fire synthesis is also known as self-propagating high temperature synthesis (SHS). To generate fire, an oxidizer, a fuel, and the right temperature are needed. All self-propagating high-temperature synthesis reactions are redox reactions; however, all redox reactions need not be SHS reactions. For any reaction to end up being self-propagating, the temperature evolved, must to be a lot more than the temperature, necessary for starting the combustion. Since mentioned above, the particular majority of combustion systems usually are exothermic enough to make high reaction temperature ranges which are enough for propagation of combustion. ^{32,33}

The SHS process has been used to prepare a large number of technologically useful oxide materials (refractories³⁴, magnetic³⁵, semiconductors³⁶, dielectric³⁷, catalysts³⁸, sensors³⁹, phosphors⁴⁰, etc.) and nonoxide materials (carbides, nitrides, borides, silicide's) by the solid-state reaction between the corresponding metals and nonmetals. The process requires high-purity fine precursors, which ignite at high temperatures. The process is highly exothermic and self-propagating resulting in coarse products.

In addition, SCS has been used to prepare a wide variety of oxide materials of desired composition and structure. The equivalence ratio of an oxidizer and fuel mixture is expressed in terms of the elemental stoichiometric coefficient. A mixture of metal nitrate and fuel solution, when rapidly heated around 500°C in a muffle furnace, foamed and ignited to burn with a voluminous product, which was identified by characterizations methods such as: XRD, EDX, FESEM, FTIR and VSM.

In this work we used, SCS method for our nanomaterial synthesis. Because on combustion form have some advances such as: simple gaseous molecules of CO₂ and H₂O, relatively simple equipment, compatibility with the all metal ions, facilitating homogenous mixing, components in turn decompose to produce combustible gases like H₂CO, NH₃ which ignite with NO_x. Also in the work we used a new polymer compound of poly maleic acid that, introduced as potential dispersant and fuel in their moieties and are particularly found to assist the combustion better and effective. Polymaleic acid (CAS Reg. No. 26099-09-2) and its sodium salt (CAS Reg. No. 70247-90-4) may be safely used in food industry.

Mechanism of solution combustion synthesis

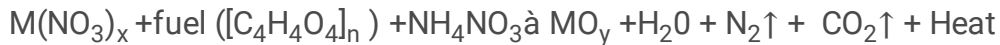
The following equation approximately can be introduced combustion temperature, which is a crucial parameter in the synthesis of materials.

$$T_c = T_0 + (\Delta H_r^0 - \Delta H_p^0)/C_p$$

Where T₀ denotes the room temperature, ΔH_r⁰ – ΔH_p⁰ are the enthalpies of formation of the reactants and products, respectively, and C_p is the heat capacity of the products at a constant pressure. Factors affecting the synthesis of nanoparticles by combustion method discussed and examined in detail.³¹

Fig. 1 shows the coordination structure for metal ion poly maleic acid. It is suggested that poly maleic acid molecules are situated in the second coordination sphere near the carbon atom. This structure in the presence of ammonium nitrate as gasser and amine group such as urea, facilities the dispersion and stabilization of reactive nitrates at an nitrogen atmosphere.

This new fuel have some important criteria quality as an ideal and nontoxic fuel. These ideal quality of this new fuel are water soluble, low ignition temperature (<160°C), be compatible with metal nitrates and the combustion reaction should be controlled and smooth and not lead to explosion. In addition, this new SCS system evolve large amounts of gases that are of low molecular weight and harmless during combustion with using ammonium nitrate as gasser. The most important result of this study: it is possible to produce large amounts of nano-materials with readily available and easy to prepare. Depending upon the fuel used and the type of metal ion produced metal oxide of iron, cobalt, Zinc, manganese and nickel oxide. The choice of fuel is critical in deciding the exothermicity of the redox reaction between the metal nitrate and the fuel. The general reaction mechanism for the preparation of metal oxide is:³³



Among various metal salts, hydrated nitrates have efficient oxidizing power of NO_3^- -groups, lower decomposition temperature and good solubility in water. Therefore, they are preferred as metal precursors for this synthesis method. Metal nitrates on combustion synthesis yield corresponding metal oxides. The decomposition temperature of the metal nitrates is lowered by the addition of this new polymer fuel that have a lot of carbonyl group. So instead compounds such as urea or glycine, this polymer should be introduced as a convenient alternative and attractive fuel in SCS synthesis method. In addition, polymaleic acid, by acting as a dispersant in the solution, prevented the accumulation of particles, and as a result, the synthesis product contained uniform and suitable nanoparticles in terms of size.

In this way, it is possible to produce a large number of nano metal oxide materials at 30-50 nm range by using this suitable fuel. Also this method can be used by dopants with takes place throughout the host material due to the atomic mixing of the reactants in the initial solution that can be used in different fields as catalyst support, biocompatible material, electrical applications, solar cell and any other usages. Formation of nano crystalline oxide materials is confirmed by FTIR, X-ray line broadening, FESEM, EDX and VSM.

UV-visible spectrum of metal oxide nanoparticles

The results obtained from Uv-Vis spectral data are in good agreement with previous reports of synthesized nanoparticles Includes: magnetite⁴¹, zinc oxide⁴², cobalt oxide⁴³, manganese oxide⁴⁴ and nickel oxide⁴⁵. For all nanoparticles, an absorption peak of about 200 nm is observed. Also, for nickel oxide nanoparticle, three absorption peaks at 210, 235 and 290 nm are observed. For zinc oxide nanoparticle, as expected, an absorption band of 372 nm is evident. Fig.2.

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of PMA and SCS of all metal oxide: Fe_3O_4 , NiO, Co_3O_4 , Mn_3O_4 and ZnO are shown in Fig. 3. As shown in the FTIR spectra the peak at 3500 cm^{-1} and 1635 cm^{-1} was due to free O-H and C=O stretch of PMA. In all other cases M-O stretch were shown in the rang $350-800\text{ Cm}^{-1}$. As we expect nano magnetite, Fe_3O_4 showed tow stretch peak at 380 and 550 Cm^{-1} . The same is true in the case of cobalt and manganese oxide at 562 and 663 Cm^{-1} . In the case of zinc oxide and nickel oxide M-O stretch band appears at 400 wave number. Fig.3. All spectral data are in good agreement with previous reports for magnetite⁴⁶, nickel oxide⁴⁷, cobalt oxide⁴⁸, manganese oxide⁴⁹ and zinc oxide^{50, 51} respectively.

FE-SEM and EDX of NPs

The high quality of the synthesized nanoparticles can be related to the presence of polymaleic acid. Figure 4 shows a very suitable particle size of synthesized nanoparticles. The synthesis of several tens of grams per synthesis and with high purity is also evident from the EDX data. The particle size distribution

is excellent. This is due to the high dispersing power of polymaleic acid, which prevents the particles from accumulating and becoming larger. This is one of important advantages of this synthesis method.

Vibrating sample magnetization (VSM)

The magnetic properties of metal nanoparticles are of great importance and application, and various synthesis methods have been proposed by researchers for this purpose⁵²⁻⁵⁴. The applications of these magnetic nanomaterials in various fields show the great importance of this group of nanomaterials⁵⁵⁻⁵⁷. Synthesis conditions such as temperature and structural properties such as shape and morphology of nanoparticles affect the type of application of these nanoparticles^{58, 59}. The quality of synthesized nanomaterials is important in terms of dimensions, particle size distribution, particle size uniformity, and ultimately particle magnetization. Magnetite nanoparticles synthesized in this research have all three advantages and supermagnetic properties. Fig.5.

X-ray diffraction (XRD) analysis

X-ray diffraction is one of the most widely used techniques for characterizing nanoparticles. The lattice parameters, crystal structure, nature and phase, and information about particle size based on the Scherer equation can be obtained from the analysis of X-ray diffraction data. Direct analysis of powder resulting from the synthesis of nanomaterials, diversity and validity of the results has made this method as a desirable method among the various methods of characterization of nanomaterials. X-ray diffraction analysis data for nanomaterials synthesized in this study are shown in Figure 6. Very good compliance of experimental data with standard peaks is quite clear. The broadening of the most intense peak, which indicates the nano-dimensionality of the synthesized material, is also quite evident. The X-ray diffraction patterns of the samples were analyzed with X-Perth software. The crystal structure and phase composition obtained from the X-ray diffraction results for the magnetite nanoparticles were matched to the standard number pattern (ref code. 96-900-5815). As shown in Figure 6, the spinel crystal structure for magnetite is provable according to previous reports⁶⁰.

In all other cases references code number are shown in the above of X-ray diffraction spectrum. Also in each case, standard peak indicates the purity of the synthesized crystal structure. From the analysis of the results in X-Perth software, based on Scherer relationship, the size of crystals and nanoparticles were calculated. The calculated particle sizes for samples Fe₃O₄, NiO, Co₂O₃, Mn₃O₄ and ZnO were 20.61, 16.91, 20.01, 23.50 and 17.83 nm, respectively. These calculations are in good agreement with the electron microscope data.

Experimental

First, an appropriate amount of polymaleic acid was poured into a beaker. Then, in another beaker, dissolve a suitable amount of iron (III) nitrate in distilled water, and then the equivalent of half a mole of iron(III), hydroquinone was added. The solution was stirred well to complete the dissolution and reduction

process. The two solutions were mixed together and placed on the heater after complete stirring. The heater temperature was adjusted to 350 ° C and the synthesis reaction was continued until drying and complete combustion. The synthesized sample was used for characterization analysis after three washing steps with distilled water and ethanol. Hydroquinone was not used for the synthesis of other nanoparticles and the rest of the synthesis steps were repeated.

Conclusion

Nowadays, there is a great interest in the synthesis of nanoparticles by combustion method. This is due to the simplicity and significant impact of this method in reducing process costs, along with the relatively favorable properties of nanomaterials in synthetic products. In addition to the many important successes obtained from this method of nanomaterial synthesis, extensive research has been reported in modeling and explaining the common mechanism of this type of nanoparticle synthesis method. Despite these studies, there are still shortcomings in modeling and accurate expression. There is a mechanism for this type of synthesis method. One of the most important advantages of this technique for the general synthesis of metal oxide nanoparticles is its very simple mechanism, in which polymaleic acid is used as a dispersing agent and fuel simultaneously.

Also, special attention should be paid to obtain more desirable nanoparticles with pre-designed morphology. In recent years, the combustion method of nanoparticle synthesis has not only created a new perspective for the preparation of various new nanoparticle oxides and composites, but also in the methods of stable nanopowder synthesis and the development of various catalysts and nanocoatings. It has gained a lot of brilliance and prominence. The authors of this paper have developed this method for the synthesis of multi-element nanocomposites for use as catalysts.

In this research work, the main focus has been on introducing a simple, easy, fast and one-step method. The results obtained from the characterization of metal nanoparticles have well added to the suitability of this synthesis method. The capability of this method for the synthesis of multicomponent composites has also been performed and confirmed by the researchers of this research. This can easily enable a variety of syntheses with this technique. As a final conclusion, the full conditions for progress in these areas over the next few years show a very clear horizon for this method. In addition to all these advantages, it is possible to commercialize this combustion method of nanoparticle synthesis, based on the capabilities of this method and the simple, accurate and better model of this method.

Complex nanoparticles monolayers in a Fe_3O_4 hydrosol sub-phase Nanoparticles have been investigated on solid substrates⁶¹. In this work, in order to synthesize magnetite nanoparticles, this property has been used to prevent the accumulation of synthesized magnetite nanoparticles. Therefore, the biggest advantage of this method compared to other combustion synthesis methods is to obtain nanoparticles with relatively the same particle size. This is a unique result, which is not seen in the combustion methods of nanoparticle synthesis.

Declarations

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Figures

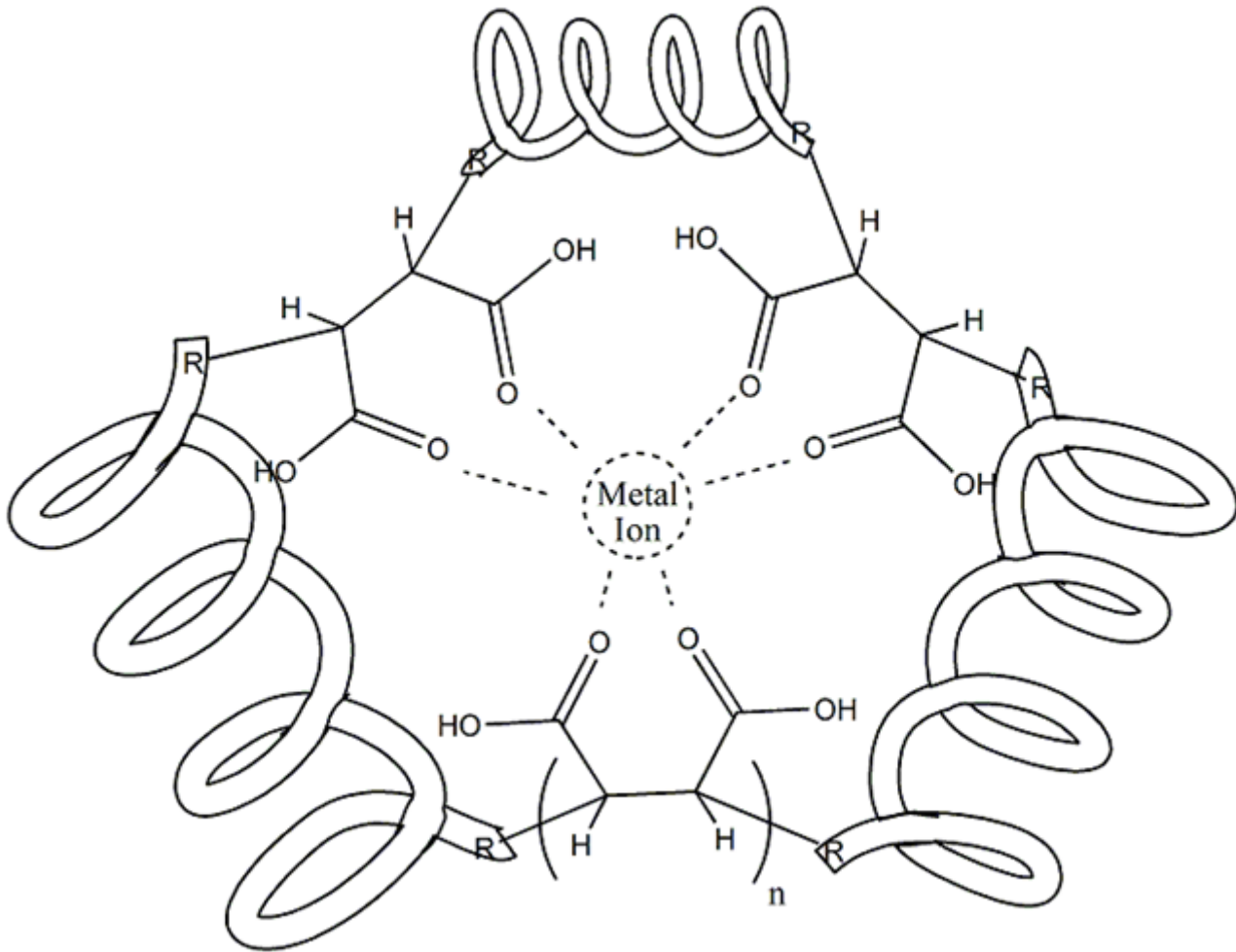


Figure 1

coordination structure for metal ion poly maleic acid

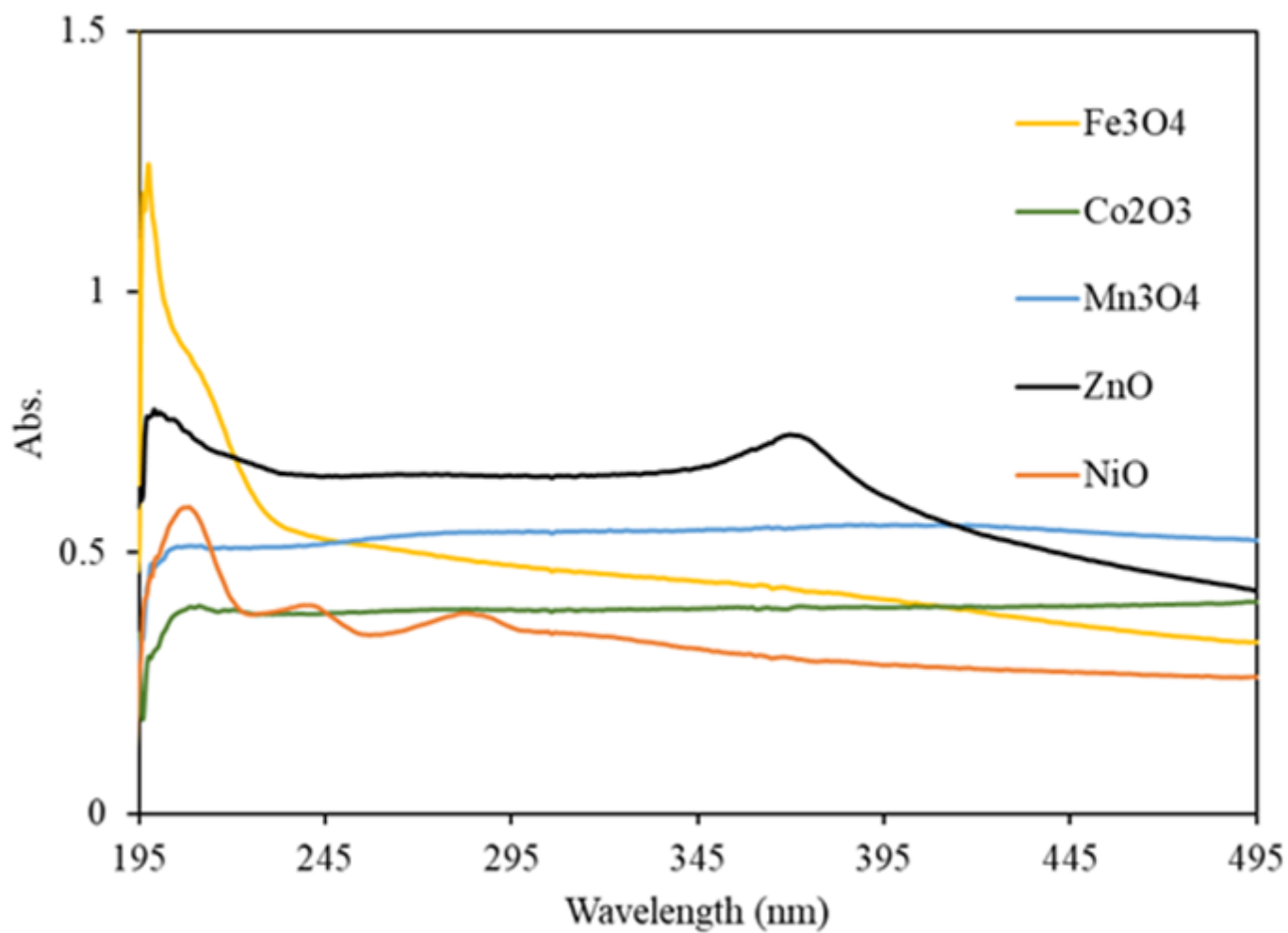


Figure 2

Uv-Vis Spectrum of nanoparticles

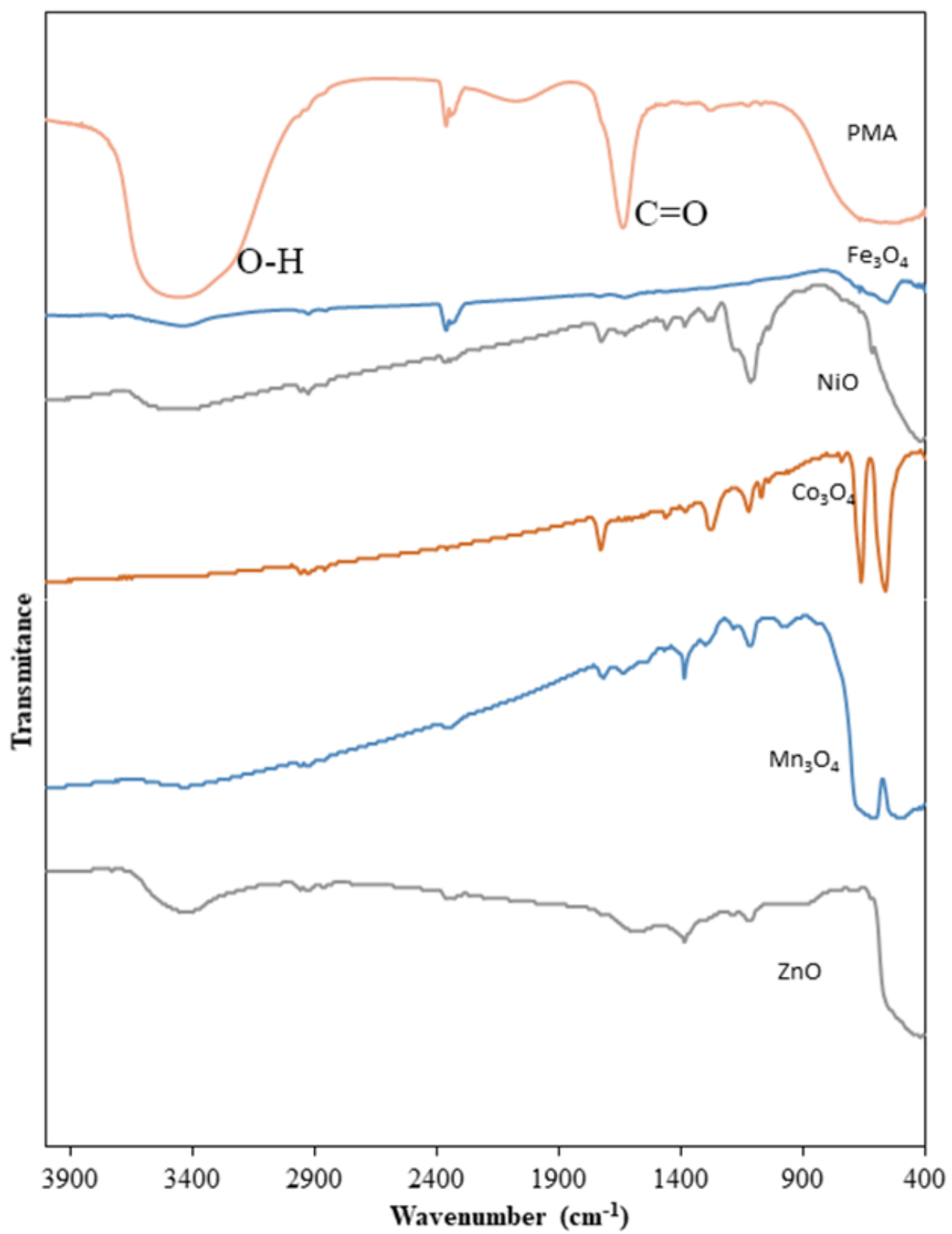


Figure 3

FTIR Spectrum of PMA and nanoparticles

Figure 4

Scanning electron microscopy (SEM) and The energy-dispersive X-ray spectroscopy (EDX) of NPs

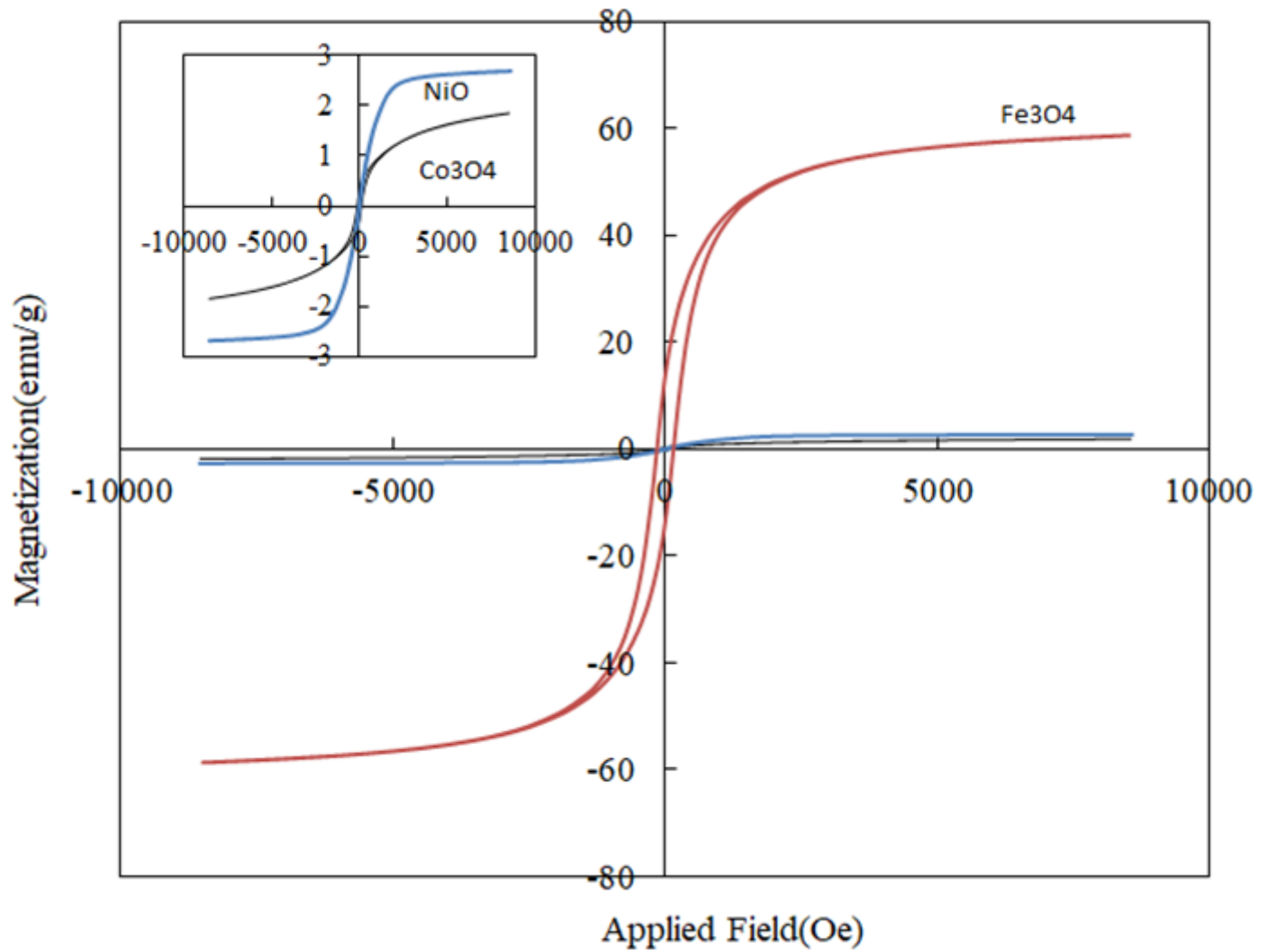


Figure 5

Vibrating sample magnetization (VSM) of nano-Fe₃O₄, Co₃O₄, and NiO.

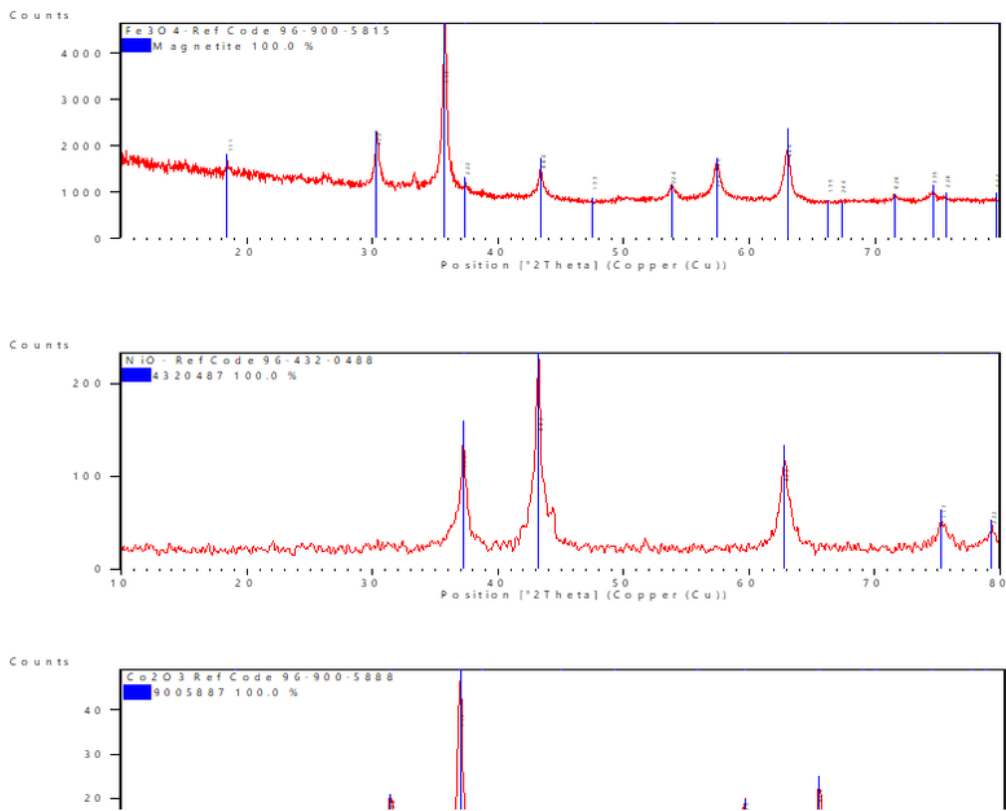


Figure 6

XRD pattern of Fe₃O₄, NiO, Co₂O₃, Mn₃O₄ and ZnO.