# SYNTHESIS OF POLYETHYLENE GLYCOL-COATED MANGANESE ZINC FERRITE AND POLYVINYL ALCOHOL-COATED MANGANESE ZINC FERRITE NANOPARTICLES VIA CO-PRECIPITATION METHOD

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*To my parents, with love and gratitude.*

*To beloved*

*Father (Hassan Kareem AL-daraji)*

*Mother (B-SH-M*

*Brothers and Sisters (Kareem, Haider, sajjad, all my sisters)*

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## ABSTRACT

Ferrites are important nanomagnetic materials in chemical industry due to their high applicability in pharmaceutical and electronic devices fabrication. Amongst, manganese and zinc modified ferrite (Mn/Zn ferrite) nanocomposites have attracted intensive interest of researchers because of the relatively high magnetic properties. However, the magnetic properties of the Mn/Zn ferrite materials reduce significantly due to agglomeration especially in aqueous solution. In this research, an attempt was carried out to synthesize polymer coated Mn/Zn ferrite via chemical co-precipitation in order to prevent agglomeration among the nanoparticles. A series of $Mn_{1-x}Zn_xFe_2O_4$ (x = 0, 0.2, 0.4, 0.6, 0.8 and 1) were prepared at 75 $^oC$ and calcined at different temperatures (500, 600, 700 ˚C). X-ray diffraction (XRD) result indicated all the prepared $Mn_{1-x}Zn_xFe_2O_4$ crystallized in spinel cubic structure with crystallite size ranging 4.58 – 11.01 nm. Besides, the increase of Zn amount in the ferrite structure resulted in reduction of both degree of crystallinity and crystallite size. Among the synthesized materials, $Mn_{0.8}Zn_{0.2}Fe_2O_4$ had the highest magnetic properties. Two types of polymers: polyethylene glycol (PEG) and polyvinyl alcohol (PVA) were used to coat $Mn_{1-x}Zn_xFe_2O_4$. The spinel structure remained intact after the polymer coating. The presence of polymer on the surface of $Mn_{1-x}Zn_xFe_2O_4$ ferrite was confirmed by Fourier transform infrared spectroscopy (FTIR) and transmission electron microscopy (TEM). Reduction in the agglomeration was observed in the polymer coated Mn/Zn ferrites as evidenced by the field emission scanning electron microscopy (FESEM) analysis. Different precipitation temperatures (25 – 100 $^oC$) were applied to prepare 10 wt% PEG coated $Mn_{0.8}Zn_{0.2}Fe_2O_4$. The XRD and FESEM results showed that both the degree of crystallinity and particle size of the materials increased with increasing of the precipitation temperature. The synthesis temperature of 75$^oC$ appeared to be the optimum temperature to produce PEG coated $Mn_{0.8}$ $Zn_{0.2}Fe_2O_4$ with the highest magnetic properties. The thermal stability of $Mn_{0.8}$ $Zn_{0.2}Fe_2O_4$ increased remarkably after coating onto 10 wt% PEG and 10 wt% PVA. Besides, it was observed that the magnetic properties of the materials increased with increasing of polymer concentration. It has been demonstrated that the magnetic properties of $Mn_{0.8}Zn_{0.2}Fe_2O_4$ (1.92 emu/g) increased more than 10 time after the polymer coating, where 10 wt% PVA coated $Mn_{0.8}Zn_{0.2}Fe_2O_4$ and 10 wt% PEG coated $Mn_{0.8}Zn_{0.2}Fe_2O_4$ recorded 28.99 and 20.67 emu/g, respectively. This research has shown that the magnetic property of Mn/Zn ferrite could be enhanced remarkably via polymer coating using PEG and PVA, leading to its increased magnetic susceptibility, in the medical and pharmaceutical applications.

# ABSTRAK

Ferit adalah bahan nanomagnet penting dalam industri bahan kimia kerana kebolehgunaannya yang tinggi dalam farmaseutikal dan pembuatan peranti elektronik. Antaranya, nanokomposit ferit terubahsuai mangan dan zink (Mn/Zn ferit) telah menarik minat intensif para penyelidik kerana sifat magnet yang secara relatif kuat. Walau bagaimanapun, sifat magnet bahan Mn/Zn ferit berkurangan secara ketara disebabkan oleh pembentukan aglomerat terutamanya dalam larutan akueus. Dalam penyelidikan ini, usaha telah dijalankan untuk mensintesiskan polimer bersalut Mn/Zn ferit melalui ko-pemendakan secara kimia untuk mengelakkan pengaglomeratan antara nanopartikel. Satu siri $Mn_{1-x}Zn_xFe_2O_4$ (x = 0, 0.2, 0.4, 0.6, 0.8 dan 1) telah disediakankan pada 75˚C dan dikalsin pada suhu yang berbeza (500, 600, 700 ˚C). Analisis pembelauan sinar-X (XRD) menunjukkan bahawa kesemua $Mn_{1-x}Zn_xFe_2O_4$ yang disediakan menghablur dalam struktur spinel dengan julatsaiz kristalit antara 4.58 – 11.01 nm. Selain itu, peningkatan amaun Zn dalam struktur ferit menyebabkan pengurangan kedua-dua darjah kehabluran dan saiz kristalit. Antara bahan yang disintesis, $Mn_{0.8}Zn_{0.2}Fe_2O_4$ mempunyai sifat magnet yang tertinggi. Dua jenis polimer: polietilena glikol (PEG) dan polivinil alkohol (PVA) telah diguna untuk menyalut $Mn_{1-x}Zn_xFe_2O_4$. Struktur spinel kekal utuh selepas penyalutan polimer. Kehadiran polimer di permukaan $Mn_{1-x}Zn_xFe_2O_4$ ferit telah disahkan oleh analisis spektroskopi inframerah transformasi Fourier (FTIR) dan mikroskopi penghantaran elektron (TEM). Pengurangan pengaglomeratan telah diperhatikan dalam Mn/Zn ferit bersalut polimer seperti yang dibuktikan oleh analisis mikroskopi pengimbasan elektron pancaran medan (FESEM). Suhu pemendakan yang berbeza (25-100 $^{o}$C) telah diguna untuk menyediakan 10 wt% $Mn_{0.8}Zn_{0.2}Fe_2O_4$ bersalut PEG.keputusan XRD dan FESEM menunjukkan bahawa kedua-dua darjah penghabluran dan saiz zarah bahan meningkat dengan peningkatan suhu pemendakan. Suhu sintesis 75$^{o}$C merupakan suhu optimum untuk menghasilkan $Mn_{0.8}Zn_{0.2}Fe_2O_4$ bersalut PEG dengan sifat magnet yang tertinggi. Kestabilan terma $Mn_{0.8}Zn_{0.2}Fe_2O_4$ meningkat secara luar biasa selepas penyalutan ke atas 10 wt% PEG dan 10 wt% PVA. Selain itu, diperhatikan bahawa sifat magnet bahan tersebut meningkat dengan peningkatan kepekatan polimer. Adalah ditunjukkan bahawa sifat magnet $Mn_{0.8}Zn_{0.2}Fe_2O_4$ (1.92 emu/g) meningkat lebih daripada 10 kali ganda selepas penyalutan polimer, dengan 10 wt% PVA bersalut $Mn_{0.8}Zn_{0.2}Fe_2O_4$ dan 10 wt% PEG bersalut $Mn_{0.8}Zn_{0.2}Fe_2O_4$ merekodkan 28.99 dan 20.67 emu/g, masing-masing. Penyelidikan ini telah menunjukkan bahawa sifat magnet Mn/Zn ferit dapat dipertingkatkan dengan ketara melalui penyalutan polimer menggunakan PEG dan PVA, seterusnya membawa kepada peningkatan kerentanan magnetnya, dalam aplikasi perubatan dan farmaseutikal.

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## LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| MNPs | - | Magnetic nanoparticle |
| PEG | - | polyethylene glycol |
| PVA | - | polyvinyl alcohol |
| PVP | - | polyvinylpyrrolidone |
| PLGA | - | poly(lactic-*co*-glycolic acid |
| XRD | - | X-ray Diffraction |
| FTIT | - | Fourier transform Infrared |
| FWHM | - | Full with at half maximum |
| FESEM | - | Field emission scanning electron microscopy |
| EDX | - | Energy dispersive X-ray |
| TEM | - | Magneto electric Measurements |
| TGA | - | Polarization electric felid measurement |
| VSM | - | Vibrating sample magnetometer |
| Ms | - | Saturation Magnetization |
| Mr | - | Remanence Magnetization |
| Hc | - | Coercivity |
| wt% | - | Weight percentage |

## LIST OF SYMBOLS

| | | |
|---|---|---|
| $\lambda$ | - | wavelength of X-ray |
| $\Delta E$ | - | Activation energy |
| A | - | Area |
| $\mu_0$ | - | Permeability |
| *B* | - | magnetic induction |
| $\chi$ | - | Magnetic susceptibility |
| Å | - | Angstrom |
| a | - | Lattice parameter |
| $\theta$ | - | Bragg's angle |
| Cu $K_\alpha$ | - | Copper K-alpha line |
| $d_{hkl}$ | - | inter-planar spacing |
| *hkl* | - | Miller indices |
| KBr | - | Potassium bromide |
| $\beta$ | - | FWHM |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

Since the ancient, iron and its alloys have been widely used as magnetic materials for various applications in the electrical industry. The first known magnetic material is lodestone, which is $Fe_3O_4$ (i.e. $FeO.Fe_2O_3$) in the form of double oxide of iron (Sharifi *et al.*, 2012). Magnetic materials are always referred to iron oxides which are in small particles and behave as a magnet. The complex oxide, which contains trivalent iron ion as the main ingredient is generally called ferrite. In fact, ferrites are considered as very well-established group of magnetic ceramic materials. These materials have general formula $MFe_2O_4$ where M is the divalent ion like $Zn^{2+}$and $Mn^{2+}$. Iron oxides exist as the basic component in ferrites.

Ferrites can be classified into two groups: hard ferrites and soft ferrites. Hard ferrites are permanent magnetic materials which are generally written as $MO\text{-}6Fe_2O_3$, where M = Ba, Sr, Pb. Materials including $BaFe_{12}O_{19}$, $SrFe_{12}O_{19}$ and $PbFe_{12}O_{19}$ are hard ferrites. These materials exhibit hexagonal crystal structure beside relatively high value of the permanent magnetic (Pullar, 2012; Veverka *et al.,* 2007,). Meanwhile, soft ferrites are ferrites those coercive field is small with the chemical formula $MFe_2O_3$ (M = transition elements such as Fe, Ni, Mn and Zn) (Ahmed *et al.*, 2004). Soft-ferrites are crystallized in spinel structure, in which the cations can be found in tetrahedral (A) and octahedral (B) sites. The spinel structure can be described as a cubic close-pack arrangement of oxygen ions in which tetrahedral A and octahedral B interstitial lattice sites are occupied by cations. In the normal spinel, the tetrahedral sites are occupied by divalent cations while trivalent cations occupy octahedral sites. In contrast, divalent cations occupy octahedral sites in inverse spinel, whereas trivalent cations are distributed equally among A and B-sites. In ferrites, $Fe^{3+}$ serves as the trivalent cation (Shinoda *et al.*, 2011). The unit cell of spinel ferrite belongs to the cubic structure. The crystal structure of cubic spinel ferrite consists of 8 $MOFe_2O_3$ molecules and 32 of $O^{2-}$ anions. The oxygen anions form the close face-centered cube (FCC) packing consisting 64 tetrahedral (A) and 32 octahedral (B) empty spaces which are partly populated by $Fe^{3+}$ and $M^{2+}$cations (Naseri *et al.*, 2013).

In the world market, demand for ferrites, especially the soft ferrites is increasing every year with annual average growth rate of 10%. In China, the soft ferrite market demand is being higher than 300,000 tons annually and this is about 20% of the annual average growth rate of development in the world. Rising demand for miniaturization is coupled with advancement in technology that leads to the availability of better quality ferrite magnets and it will drive market to spend about to $18.8 billion by 2018 for ferrite manufacture (Cushen *et al.*, 2012; Kulikowski,1984).

In recent years, a lot of work have been done on the synthesis of nanocrystalline materials because of their unusual properties compared to the properties of bulk materials (Abe *et al.*, 2009). Due to their high surface area, magnetic nanoparticles have been widely used in the synthesis of magnetic ferrofluid, electronic applications such as transformers, choke coils, noise filters, and recording heads as well as biologically active molecules (Pankhurst *et al.*, 2003). The physical and chemical properties of magnetic ferrite nanoparticles greatly depend on the synthesis method, preparation conditions, action distribution and magnetic interactions etc. (Caltun *et al.*, 2002).

Various synthesis methods for ferrites have been reported, including sol–gel synthesis, ball milling, chemical precipitation, hydrothermal reactions and co-precipitation. Among, co-precipitation method could be the most promising method due to its simplicity and high productivity. This method is also able to synthesize products in narrow size distribution, small particle sizes and controllable shape. In fact, this method has been widely used for biomedical applications due to ease of implementation (Amiri and Shokrollahi, 2013; Shokrollahi, 2013).

For their applications in pharmaceutical and biomedical areas, the nanoparticles (NPs) must possess high magnetic susceptibility for an optimum magnetic enrichment and loss of magnetization after removal of the magnetic field. However, the magnetic nanoparticles (MNPs) always behave differently in a liquid phase. These MNPs tend to agglomerate in aqueous, resulting in significant decrease in their magnetic properties.

To solve the problem, surface coating on MNPs appears as one of the most efficient approaches for high physically and chemically stability of MNPs in aqueous. It has been reported that the water-dispensability of MNPs enhanced after coating using polyethylene glycol (PEG) dextrin (Zhang *et al.,* 2008). Formation of layers of inorganic metals like gold, nonmetals (e.g. graphite) and oxide surfaces ($SiO_2$) could also improve the water-dispensability of MNPs (Reddy *et al.*, 2012).

Manganese/zinc ferrite (Mn/Zn ferrite), a soft ferrite, is one of the most important ferrites due to its high magnetic properties. It has been used extensively as ferrite cores in electronics, magnetic transformer and other electrical applications (Maspol, 2001). In this study, nanostructured magnetic materials of manganese and zinc doped ferrite were prepared via chemical co-precipitation method. These materials were characterized in order to understand the effect of Zn/Mn ratio and calcinations temperature towards properties of the resulted materials. Besides, novel Mn/Zn ferrite materials coated with biocompatible polymer including polyethylene glycol (PEG) and polyvinyl alcohol (PVA) were synthesized in order to further improve their thermal stability and magnetic behavior.

PEG is a neutral, hydrophilic, linear synthetic, biocompatible polymer that can be prepared with a wide range of terminal functional groups. By varying these functional groups, PEG can be bound to different surfaces. PEG permits limited grafting of further macromolecules since it has only one site available for ligand coupling. Modification of MNPs using PEG is believed to promote better internalization of particles; remain stable at high ionic strengths of solutions with varying PH values (Nabiyouni *et al.*, 2011).

PVA is a hydrophilic, synthetic polymer. Coating of PVA onto MNPs surface would enhance the colloidal stability of. PVA irreversibly binds on MNPs surface due to interconnected network with interface, which means a fraction of PVA remains associated with the nanoparticles despite repeated washing. For this reason PVA more effect in the magnetic properties (Umut, 2013; Nabiyouni *et al.*, 2011).

## 1.2 Problem Statement

Mn/Zn ferrite materials have been widely reported due to their excellent magnetic properties. These materials are usually prepared via hydrothermal, ball milling and ceramic methods. However, particle size of the products is always not controllable and inhomogeneous. Chemical co-precipitation method is one of the promising approaches to synthesize homogeneous nanoparticles. In fact, it has been recognized as one of the most simple and economic method in preparing nanostructure materials. Recently (Mohapatra and Anand, 2010; Mamalis, 2007)

Mn/Zn ferrite nanoparticles synthesized via co-precipitation was reported. Unfortunately, these nanoparticles tend to agglomerate especially in aqueous solution, leading to the decrease in magnetic properties. Besides, the agglomeration formed has also limited the application of Mn/Zn ferrites such as in drug delivery system. Therefore, Mn/Zn ferrites with high magnetic properties in aqueous is always desired.

Polymer coating appears as a promising approach to evade the formation of agglomeration, hence improving magnetic properties of nanoferrite. However, usage of polymer as coating material for Mn/Zn ferrite prepared via co-precipitation method has not been reported yet. Therefore, the effect of polymer coating on properties of Mn/Zn ferrite is worthy for further exploration.

## 1.3 Objectives

The objectives of the research were

i. To synthesize and characterize the Mn/Zn ferrite and polymer coated Mn/Zn ferrite.
ii. To investigate the physical-chemical properties including crystallinity and particles size of the Mn/Zn ferrites.
iii. To evaluate the magnetic properties of the synthesized Mn/Zn and polymer coated Mn/Zn ferrite.

## 1.4 Scope of the Research

In this study, a series of Mn/Zn ferrite ($Mn_{1-x} Zn_xFe_2O_4$, x = 0.0, 0.2, 0.4, 0.6, 0.8, 1.0) were synthesized via chemical co-precipitation at 75$^{\circ}$C and pH =11. Chemicals including $FeCl_3$, $MnCl_2.4H_2O$, $ZnCl_2$ and NaOH were used as precipitate reagents in aqueous solution. The resulted materials were characterized using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX) analysis and Transmission electron microscopy (TEM). The magnetic and super paramagnetic behaviors of the materials were confirmed using a vibrating sample magnetometer (VSM). Effect of calcination temperatures (500 , 600 and 700 $^{o}$C) towards structural properties of the ferrites was studied.

In order to increase magnetic properties of Mn/Zn ferrites in aqueous solution, two biocompatible water-soluble polymers were used to coat the surface of those magnetic nanoparticles. For this purpose, $Mn_{1-x}Zn_xFe_2O_4$ was further coated with polymers of 10 wt% PEG and PVA. The polymer coated Mn/Zn ferrite of the highest magnetic property was used to further investigate the effect of polymer content (5-25%) and precipitation temperature (25-100°C) on their properties.

## 1.5 Significance of Study

Magnetic materials of polymer coated Mn/Zn ferrite were synthesized via chemical co-precipitation method. For the first time the appeared to be promising approach to synthesize homogeneous with high purity nanoparticles. In this research, an attempt was made to synthesize polymer coated Mn/Zn ferrite at low synthesis temperature to reduce the agglomeration of nanoparticles. The research remits demonstrated that the polymer coating using polyethylene glycol (PEG) and polyvinyl alcohol (PVA) could be effective was to evade the formation of agglomeration among the magnetic nanoparticle. The resulted polymer coated ferrite are reported also to have higher thermal stability and a high magnetic properties in the aqueous condition

The agglomeration formed has also limited the application of ferrites. Polymer coating appeared as a promising approach to evade the formation of agglomeration. Polymer coating would overcome the lack of stability of the nanoparticles in the synthesized system. As a result, both bioactivity and magnetic properties of Mn/Zn ferrite was improved.

The resulted materials could be applied as ferrofluid and be used in medical application such as for drug delivery and hydrothermal treatment. The findings of the study provide fundamental information on relationship between structure and magnetic properties of ferrite-based materials. This will definitely contribute to the development of new magnetic materials which are widely used in pharmaceutic and electronics industries.

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