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Comparative Study of $MgFe_2O_4$ and $ZnFe_2O_4$ Nanoparticles Synthesized by Different Methods for Use in Magnetic Bioactive Glass for Hyperthermia Treatment

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Abstract. Although magnetic nanoparticles have been widely studied, limited research has compared different ferrite types and synthesis routes for use in hyperthermia-based bioactive glass applications. This study aims to synthesize magnetic materials from two types of ferrites: magnesium ferrite ($MgFe_2O_4$) and zinc ferrite ($ZnFe_2O_4$). These ferrite nanoparticles were synthesized using two distinct methods; the conventional solid-state reaction and the co-precipitation method in order to identify the optimal synthesis route and the most suitable type of magnetic material for hyperthermia treatment. The data demonstrated that $MgFe_2O_4$ powder with synthesis by using the solid-state method consistently presented higher value of magnetic properties compared to those synthesized by co-precipitation method under higher calcination temperature. Moreover, $ZnFe_2O_4$ powder was found to be unsuitable for use as a precursor in hyperthermia treatment because of its structure typically leads to antiferromagnetic or superparamagnetic behavior. The effect of $MgFe_2O_4$ containing in bioactive glass was investigated. The oxide precursors of bioactive glass were mixed with varying amounts of $MgFe_2O_4$ and subsequently melted to form glass at 1400 °C. The phase formation presented SiO_2 was the dominant phase and coexisted with Na_2CO_3, $MgSiO_3$, Fe_3O_4, $Na_2Ca(PO_4)_2SiO_4$, Ca_2SiO_4, and $Na_4Ca_4Si_6O_{18}$. However, the $MgFe_2O_4$ phase was not observed in all of glass-ceramic samples. This may be due to $MgFe_2O_4$ decomposed during the high-temperature melting process at 1400 °C. Nevertheless, these magnetic bioactive glass ceramic samples exhibited magnetic properties, which were attributed primarily to the presence of Fe_3O_4.

Introduction

In recent years, bioglass-ceramics with both bioactive and magnetic properties have gained increasing attention for use as heat-generating materials (thermo-seeds) in hyperthermia treatment of cancer, especially for deep-seated bone tumors. Hyperthermia is considered a promising method for cancer therapy. Currently, several techniques are used in clinical practice, such as regional blood perfusion, implanted heat sources, and energy-based methods including electro-coagulation, ultrasound, microwave, and other electromagnetic sources [1]. However, these techniques often face challenges in providing uniform and localized heating to the tumor area without harming nearby healthy tissues. Magnetic bioactive glass-ceramics are promising materials for this purpose. They are specially developed to generate heat under an alternating magnetic field and also help in repairing bone after tumor removal. Magnetic bioactive glass-ceramic implants can be reheated when necessary to induce localized thermal effects that eliminate malignant cells and help prevent tumor recurrence. Furthermore, the risk of harmful metal ion leaching into body fluids is minimized, as each ferromagnetic particle is encapsulated within the protective glass matrix [2].

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From previously study optimizing heat delivery and biocompatibility, various magnetic materials have been developed, including amorphous magnetic flakes [3], barium ferrite microspheres [4], and ferrite rods [5, 6], each engineered to exhibit controlled heat release under external magnetic excitation. These materials are capable of generating temperatures within the therapeutic range (42–45 °C) when subjected to alternating magnetic fields, primarily via magnetic losses.

Zinc (Zn^{2+}) and magnesium (Mg^{2+}) ions play important roles in the human body, particularly in bone metabolism [7]. Therefore, the incorporation of these ions into magnetic glass-ceramics in the form of $ZnFe_2O_4$ or $MgFe_2O_4$ can contribute significantly to biomedical applications, especially in the field of bone-related health care.

The purpose of the present study is to synthesize magnetic materials for incorporation into bioactive glass, with the objective of evaluating their potential application in cancer treatment via magnetic hyperthermia. Two types of ferrites; $MgFe_2O_4$ and $ZnFe_2O_4$ were selected as heat-generating agents. These ferrite nanoparticles were synthesized using two distinct methods: the conventional solid-state reaction and the co-precipitation method, in order to identify the optimal synthesis route and the most suitable type of magnetic material for this application. Moreover, the influence of the optimal ferrite addition on the structural properties of the resulting glass-ceramics was investigated

Experiment Procedure

Preparation of magnetic materials. In this study, two types of magnetic materials; $MgFe_2O_4$) and $ZnFe_2O_4$ were synthesized using two different methods: the solid-state reaction method and the Co-precipitation method.

Preparation of materials Solid-State Reaction Method. In the solid-state reaction method, magnesium ferrite ($MgFe_2O_4$) was synthesized using MgO and Fe_2O_3 as precursor materials, while zinc ferrite ($ZnFe_2O_4$) was synthesized from ZnO and Fe_2O_3. The raw powders were subjected to high-energy ball milling for 9 hours using zirconia balls as the grinding media to achieve homogenous mixing and particle size reduction. After milling, the powder mixture was separated from the grinding media and subsequently stirred for 30 minutes at 80 °C to enhance homogeneity. The resulting slurry was then dried and calcined at temperatures of 900 °C and 950 °C for 4 hours.

Preparation of materials Co-precipitation Method. In the co-precipitation method, magnesium ferrite ($MgFe_2O_4$) was synthesized using magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) and iron (II) chloride hexahydrate ($FeCl_2 \cdot 6H_2O$) as precursors. For zinc ferrite ($ZnFe_2O_4$), zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) and iron(II) chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$) were used. The precursor salts were dissolved in deionized water under continuous stirring with a magnetic stirrer and heated to 80 °C to ensure complete dissolution. A hydroxide solution was then added dropwise until a precipitate formed and the pH of the mixture reached between 12 and 14. The reaction mixture was allowed to cool to room temperature, after which the precipitate was filtered and thoroughly washed multiple times with deionized water until a neutral pH (~7) was achieved. The obtained precipitate was then dried and subsequently calcined at 900 °C and 950 °C for 4 hours in air.

Preparation of magnetic bioactive glass. The glass was prepared by conventional melting appropriate combination of SiO_2 45 wt.%, Na_2O 24.5 wt.%, CaO 24.5 wt.% and P_2O_5 6 wt.% and mixed with the calcined $ZnFe_2O_4$ or $MgFe_2O_4$ at 0, 3, 6, and 9 wt.%. The mixture was melted at 1400°C for 2h on brass mold [9].

Materials characterization. The synthesized ferrite materials were characterized to evaluate their structural and magnetic properties. Phase identification was carried out using X-ray diffraction (XRD), while magnetic behavior was examined using a vibrating sample magnetometer (VSM). The glass transition temperature (Tg) and crystallization temperature (Tc) of the crushed and milled glass powders for each composition were determined using differential thermal analysis (DTA; Model 1600 DTA, Du Pont Instruments).

Results and Discussion

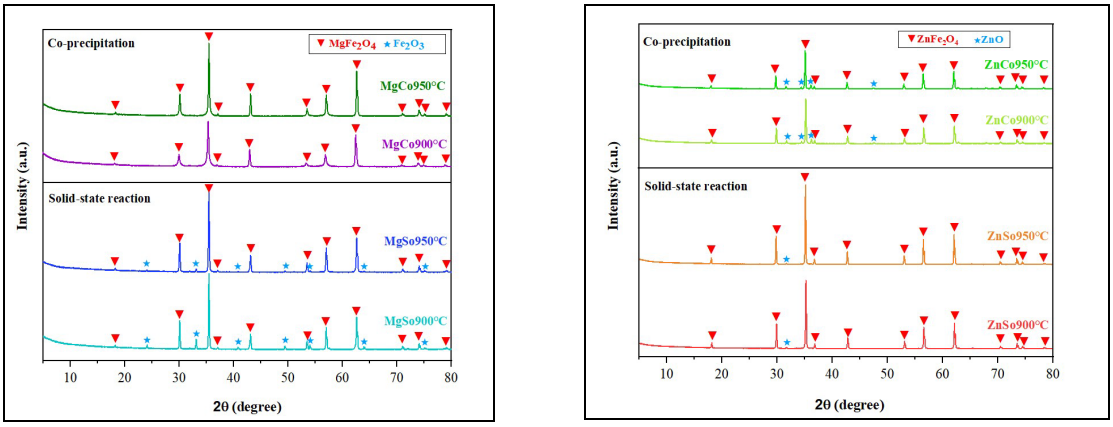


Fig. 1. The phase formation of (a) $MgFe_2O_4$ powders (b) $ZnFe_2O_4$ powders synthesized by two different methods-solid-state reaction (So) and co-precipitation (Co)

The phase composition of magnesium ferrite and Zinc ferrite magnetic powders synthesized via the solid-state reaction and co-precipitation methods and subsequently calcined at 900 °C and 950 °C was investigated using X-ray diffraction (XRD), as illustrated in Fig1. In Fig 1(a) the samples synthesized by the solid-state reaction method, the primary crystalline phase identified was $MgFe_2O_4$, accompanied by a secondary phase of iron oxide (Fe_2O_3). In contrast, the samples prepared via the co-precipitation method exhibited a single-phase composition corresponding $MgFe_2O_4$ only. This indicates that the co-precipitation method is more effective in achieving phase-pure magnesium ferrite, minimizing the presence of undesired secondary phases.

Figure 1(b) presents the XRD patterns of zinc ferrite powders synthesized via co-precipitation and solid-state reaction methods. In the co-precipitation method, two major phase, $ZnFe_2O_4$ and ZnO were identified in all samples. Moreover, the intensity of the $ZnFe_2O_4$ peaks increased with increasing calcination temperature, indicating enhanced crystallinity and improved phase development.

In contrast, the samples synthesized via solid-state reaction exhibited only a single phase of $ZnFe_2O_4$, with no detectable secondary phases. This result implies a more complete reaction and better phase purity compared to the co-precipitation method.

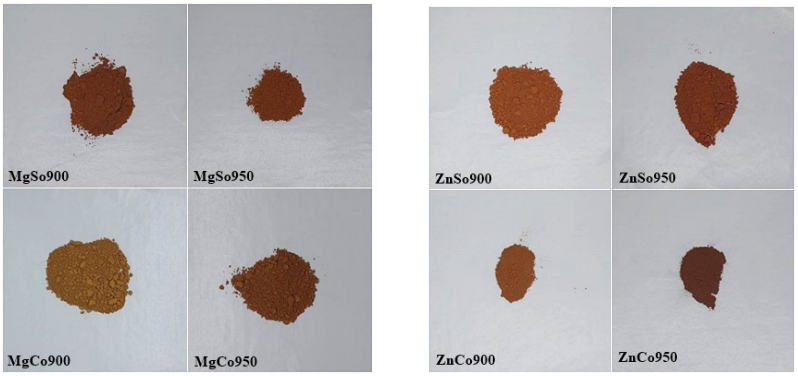
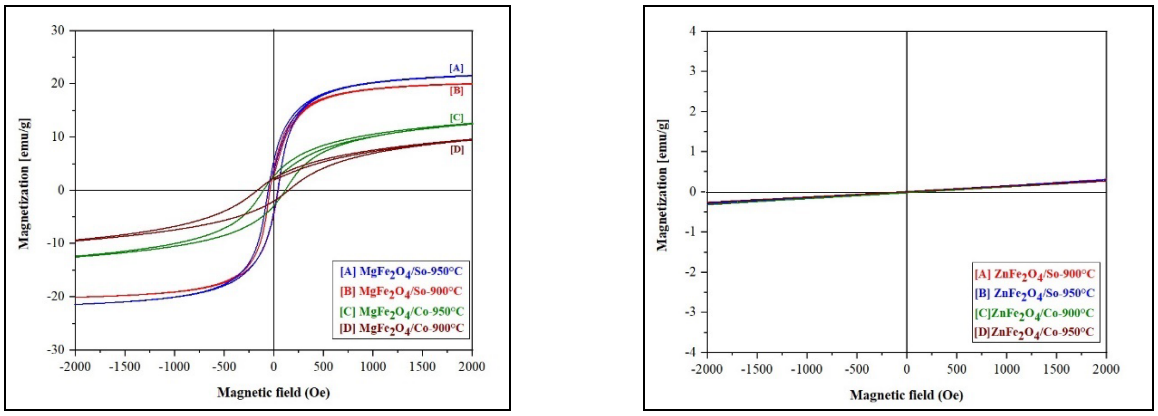


Fig. 2. Appearance of the powders of (left) magnesium ferrite (right) Zinc ferrite powders synthesized by two different methods-solid-state reaction (So) and co-precipitation (Co)

The experimental results (Fig 2) revealed that $MgFe_2O_4$ and $ZnFe_2O_4$ powders synthesized via both the solid-state reaction and co-precipitation methods, and calcined at two different temperatures 900 °C and 950 °C-exhibited color variations ranging from yellowish-brown to reddish-brown. An increase in calcination temperature to 950 °C resulted in a visibly darker color in both types of ferrite powders compared to those calcined at 900 °C.



(a) (b)

Fig. 3. Room temperature hysteresis loops up to 2000 Oe of (a) $MgFe_2O_4$ powders (b) of $ZnFe_2O_4$ powders synthesized via different methods

The magnetic behavior of $MgFe_2O_4$ powders calcined at 900 °C and 950 °C was investigated using a vibrating sample magnetometer (VSM). The obtained hysteresis curves, as shown in Fig 3(a) exhibit typical ferromagnetic behavior, characterized by the presence of hysteresis loops. The loops were observed to be narrow and well-defined, indicating that the materials possess soft magnetic properties. Moreover, it can be seen that the magnetization (Ms) increases with increasing calcination temperature. This suggests that higher thermal energy during calcination promotes improved crystallinity and magnetic domain alignment, thereby enhancing the overall magnetic response of the material [8, 9]. All of samples, $MgFe_2O_4$ synthesized via the solid-state reaction method with calcined at 950 °C exhibited the highest saturation magnetization. Moreover, for the XRD result, the appearance of secondary phase of Fe_2O_3 also has a positive effect on the magnetic behavior of the sample. This result implies that the synthesis method and calcination temperature significantly influence the magnetic properties of $MgFe_2O_4$, with the combination of solid-state synthesis and higher calcination temperature yielding superior magnetic performance.

Table 1. Magnetic properties evaluated from hysteresis loops of $MgFe_2O_4$

Magnetic and structural Parameters	Condition ($MgFe_2O_4$)			
	MgCo900˚C	MgCo950˚C	MgSo900˚C	MgSo950˚C
Saturation magnetization Ms (emu/g)	8.90	11.12	18.74	20.81
Remanence magnetization Mr (emu/g)	2.14	2.80	4.33	4.82
Coercive force Hc (Oe)	23.52	24.47	24.80	27.63

The magnetic properties of $MgFe_2O_4$ samples prepared by co-precipitation and solid-state synthesis at two different calcination temperatures are summarized in Table 1. The saturation magnetization values range from 8.90 to 20.81 emu/g, remanent magnetization (M_r) ranges from 2.14 to 4.82 emu/g, and coercive force (Hc) values vary between 23.52 and 27.63 Oe. It can be confirmed that the samples prepared by solid-state synthesis and calcined at 950 °C exhibited the highest M_s (20.81 emu/g), M_r (4.82 emu/g), and Hc (27.63 Oe), indicating a significant improvement in magnetic characteristics. These results highlight that both synthesis method and calcination temperature play critical roles in tailoring the magnetic performance of $MgFe_2O_4$-based materials. The solid-state route appears to enhance magnetic ordering, especially when combined with a high-temperature calcination process.

The magnetic properties of $ZnFe_2O_4$ synthesized via co-precipitation and solid-state reaction methods and subsequently calcined at 900 °C and 950 °C are shown in Fig 3(b). Overall, the results indicate that $ZnFe_2O_4$ exhibits relatively weak magnetic behavior compared to $MgFe_2O_4$, which is consistent with the known characteristics of normal spinel-type $ZnFe_2O_4$. This structure typically results in antiferromagnetic or superparamagnetic behavior at room temperature due to the occupation of Zn^{2+} ions in the tetrahedral (A) sites and Fe^{3+} ions in the octahedral (B) sites, leading to minimal net magnetic moment [10,11].

Table 2. Magnetic properties evaluated from hysteresis loops of $ZnFe_2O_4$

Magnetic and structural Parameters	Condition ($ZnFe_2O_4$)			
	ZnCo900˚C	ZnCo950˚C	ZnSo900˚C	ZnSo950˚C
Saturation magnetization Ms (emu/g)	2.52	2.81	2.78	3.05
Remanence magnetization Mr (emu/g)	0.00032	0.00054	0.0012	0.00059
Coercive force Hc (Oe)	0.0040	0.0057	0.014	0.0077

The magnetic properties of $ZnFe_2O_4$ powders synthesized via the solid-state reaction and co-precipitation methods with various calcination temperature are summarized in Table 2. All of the M_s values for the $ZnFe_2O_4$ samples range from 2.52 to 3.05 emu/g. The highest M_s value (3.05 emu/g) was observed in the sample prepared by the solid-state method and calcined at 950 °C. Moreover, $ZnFe_2O_4$ powder exhibited exceptionally low remanent magnetization (M_r) values, ranging from 0.00032 to 0.0012 emu/g, and low coercive force (Hc) values between 0.0040 and 0.0140 Oe. From these results, it can be concluded that $ZnFe_2O_4$ powder is not suitable for use as a precursor in hyperthermia treatment. It can be indicated that samples synthesized by the solid-state method consistently showed higher values of M_s, M_r, and Hc compared to those prepared via co-precipitation under identical calcination conditions. This suggests that the solid-state method enhances crystallinity and promotes better magnetic domain alignment, especially at higher temperatures [12, 13]. These findings confirm that both the synthesis route and calcination temperature significantly influence the magnetic performance. Based on the results, $MgFe_2O_4$ synthesized via the solid-state reaction method is identified as the most suitable precursor for incorporation into bioactive magnetic glass intended for hyperthermia treatment.

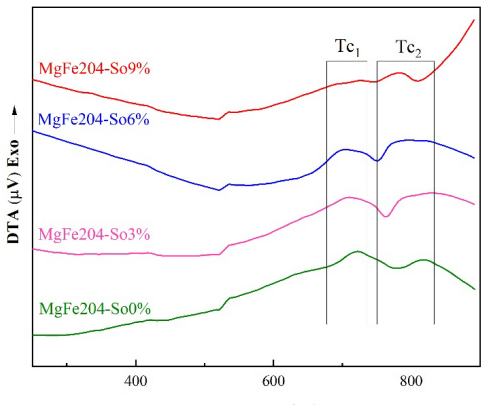


Fig. 4. DTA traces.

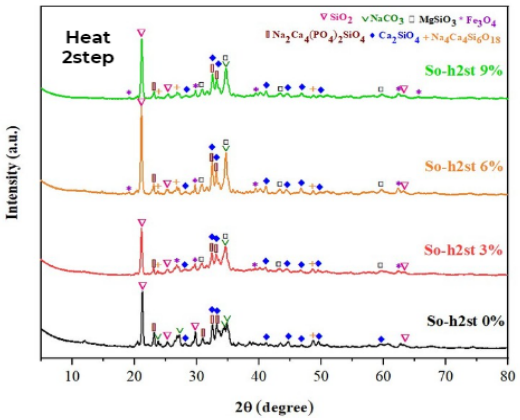


Fig. 5. XRD patterns of the glass ceramic samples with various $MgFe_2O_4$ contents after heat treatment 2 step

Fig 4 show the DTA trace of the investigated glass, both the glass transition temperature (T_g) and crystallization temperature (T_c) decrease when the level of $MgFe_2O_4$ increases.The thermal parameter of all glasses present the same result, with two exothermic peak (715-725°C and 730-800°C). For the DTA result, the glass samples were heated at two step heat treatment at 720°C for 1 h and 800 °C for 1 h and the characterization with XRD pattern as shown in Fig 5. The XRD result present that SiO_2 is the main phase and was found to coexist with the $NaCO_3$, $MgSiO_3$, Fe_3O_4, $Na_2Ca(PO_4)_2SiO_4$, Ca_2SiO_4 and $Na_4Ca_4Si_6O_{18}$. Almost phase formation increased with the addition of $MgFe_2O_4$ up to 9%. However, the desired $MgFe_2O_4$ phase was not observed in any of the glass-ceramic samples. Instead, it was found to decompose during the high-temperature melting process at 1400 °C. Upon cooling, devitrification led to the formation of Fe_3O_4 and $MgSiO_3$ phases.

The magnetization hysteresis loops of the heat-treated glass-ceramic samples are shown in Fig. 6. All samples exhibited comparable magnetic behavior, characterized by narrow hysteresis loops typical of soft magnetic materials. The observed enhancement in magnetic properties can be attributed to the crystallization process. A higher content of $MgFe_2O_4$, along with the presence of the Fe_3O_4 phase, contributes significantly to the improvement in magnetic performance.

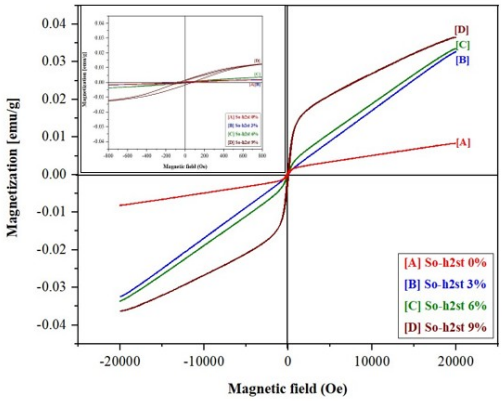


Fig. 6. Hysteresis loops up to 2000 Oe of glass ceramic samples with various $MgFe_2O_4$ contents

Conclusion

In this study, $MgFe_2O_4$ and $ZnFe_2O_4$ nanoparticles were synthesized using different method via the solid-state reaction and co-precipitation methods to identify the most suitable precursor for magnetic bioactive glass for hyperthermia applications. The results demonstrated that $MgFe_2O_4$ synthesized by the solid-state method exhibited a good magnetic property. $ZnFe_2O_4$ exhibits relatively weak magnetic behavior, typically resulting in antiferromagnetic or superparamagnetic characteristics. This makes it unsuitable for hyperthermia applications. Magnetic bioactive glass ceramics of $MgFe_2O_4$-bioglass system were obtained by the incorporation method. Phase formation analysis also confirmed the presence of bioactive phases such as $Na_2Ca(PO_4)_2SiO_4$ and Ca_2SiO_4, which are beneficial for bone regeneration. Moreover, it was found that $MgFe_2O_4$ addition and calcination temperature caused an alteration in magnetic properties. Although the $MgFe_2O_4$ phase decomposed during the high-temperature melting process used to fabricate glass-ceramic composites, the resulting materials still displayed appreciable magnetic behavior. This was primarily attributed to the formation of Fe_3O_4 as a secondary magnetic phase

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