

OPTIMUM FIRING TEMPERATURES FOR FABRICATION OF FERROMAGNETIC $\text{Mn}_{0.85}\text{Zn}_{0.15}\text{Ni}_{0.15}\text{Fe}_2\text{O}_4$ CERAMICS VIA THE SOLID-STATE COMBUSTION TECHNIQUE

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Abstract

In this research, optimum firing temperatures for the fabrication of ferromagnetic $\text{Mn}_{0.85}\text{Zn}_{0.15}\text{Ni}_{0.15}\text{Fe}_2\text{O}_4$ (MZNF) ceramics were investigated. MZNF ceramics were prepared via the solid-state combustion method using glycine as fuel. All samples were calcined in the temperature range of 1,000-1,150°C for 2 h and sintered between 1,200 and 1,275°C for 2 h. Pure MZNF powders were obtained by calcining at 1,150°C for 2 h. Increasing the sintering temperature improved the cubic spinel structure. The average particle size and the average grain size tended to increase from 0.38 to 1.61 μm and 6.38 to 8.15 μm , respectively, with an increase in the firing temperatures. The dielectric constant at room temperature (ϵ_r) of the samples increased with increased sintering temperatures. Density and saturated magnetization (M_s) of the ceramics increased from 4.74 to 4.87 g/cm^3 and 63.40 to 84.67 emu/g , respectively, when the sintering temperature increased from 1,200 to 1,250°C, and then those parameters decreased as the temperature went above 1,250°C. At the optimum sintering temperature (1,250°C), MZNF ceramics exhibited good crystallinity, a packed microstructure, the highest density (4.87 g/cm^3), good dielectric properties and excellent ferromagnetic properties ($M_s = 84.67 \text{ emu/g}$ and $H_c = 1.42 \text{ Oe}$).

Keywords: Ferromagnetic; Microstructure; MZNF; Phase Structure; Solid-State Combustion Technique

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Introduction

Ferrite magnetic materials play a crucial role in the realm of contemporary technological advancements for the development of electronic devices to meet human needs. These materials are integral to various electronic devices, including data storage systems, sensors, actuators, magnetic fluids, microwave absorbers, and medical diagnostic tools (Jeun *et al.*, 2013; Khot *et al.*, 2013; Şentürk *et al.*, 2013; Younas *et al.*, 2011). Ferrite is a ceramic compound made up of iron oxide (Fe_2O_3) combined with metallic elements like nickel, zinc, or manganese (Khan *et al.*, 2024). One significant type of ferrite is spinel ferrites where the unit cell of the spinel contains eight FCC oxygen sub cells arranged in a cubic array. Spinel ferrites are a naturally occurring class of minerals with the chemical formula AB_2O_4 , where A and B are usually metal ions (Hao and Ning, 2021). The most commercially significant ceramics with spinel structures are soft magnetic ferrites, which are utilized in a wide variety of applications (Chiang *et al.*, 1997). Research of spinel ferrites is crucial for both fundamental science and practical applications.

Manganese-Zinc ferrite (Mn-Zn ferrite) is a common type of spinel ferrite known for its notable magnetic properties (high saturation magnetization ($M_s \sim 30$ emu/g) and low coercivity ($H_c \sim 95$ Oe)), excellent chemical stability, high permeability ($\mu_i > 500$), mechanical hardness and small grain size (1-10 μm) (Verma and Chatterjee, 2006; Thakur *et al.*, 2020; Dastjerdi *et al.*, 2023). Due to their exceptional qualities, Mn-Zn ferrites are a topic of great interest and extensive research. Previously, reports have indicated that the magnetic properties of Mn-Zn ferrite can be enhanced by adding metal elements such as Sc, Mg, Gd, Al, and Sm, which can increase the $M_s > 40$ emu/g and reduce the $H_c < 90$ Oe (Thakur *et al.*, 2020). Moreover, Jalaiah and Babu (2017) reported that incorporating nickel (Ni) into Mn-Zn spinel ferrites, using the sol-gel auto combustion method, resulted in the highest magnetic saturation value ($M_s = 137$ emu/g) and a low coercivity value ($H_c = 0.246$ Oe) when 0.15 mol of nickel was added.

Multiple synthesis techniques have been utilized to prepare Mn-Zn ferrites, including the sol-gel method (Tsakaloudi *et al.*, 2019), solid-state reaction (Zapata and Herrera, 2013), microwave-hydrothermal process (Praveena *et al.*, 2015), and combustion method (Fu *et al.*, 2010). The combustion method has been widely used for synthesizing ferrites because of its numerous advantages. This method is a rapid process that can produce ferrite powders in a significantly shorter

time compared to other techniques. Additionally, the combustion method typically results in high-purity products by efficiently eliminating impurities (Thawong *et al.*, 2019; El Heda *et al.*, 2023; Sonchaopri *et al.*, 2023; Vidya *et al.*, 2023).

Moreover, fabrication of $\text{Mn}_{0.85}\text{Zn}_{0.15}\text{Ni}_{0.15}\text{Fe}_2\text{O}_4$ (MZNF) ceramics by the solid-state combustion method has not been reported to date. Therefore, this study investigated the optimum firing temperatures for fabrication of MZNF ceramics by the solid-state combustion technique and the effect of firing temperatures on the phase formation, microstructure, dielectric and magnetic properties were evaluated.

Materials and Method

$\text{Mn}_{0.85}\text{Zn}_{0.15}\text{Ni}_{0.15}\text{Fe}_2\text{O}_4$ (MZNF) ceramics were prepared by the solid-state combustion technique. High-purity chemical compounds of MnO_2 (99.00%; sourced from Sigma-Aldrich, Aust), ZnO (99.00%; obtained from Merck, German), NiO (99.00%; also from Merck, German), and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (101%; provided by Ajax, Aust) were utilized as starting materials. The raw substances were weighed to the correct proportions and milled with zirconium balls in ethanol for 24 h. Subsequently, they were dried at 120°C for 6 h. Crystalline powders were mixed with glycine (99%; obtained from KemAus, Aust) in a ratio of 1:2, and then subjected to calcination at temperatures between 1,000 and $1,150^\circ\text{C}$ for 2 h. The pure calcined powders were combined with polyvinyl butyral (PVB) as a binder in an agate mortar. After the slurry was dried, it was pressed into pellets with a diameter and thickness of ~ 12 mm and ~ 1 mm, respectively, under a pressure of 80 MPa. Finally, the pellets were sintered between 1,200 and $1,275^\circ\text{C}$ for 2 h.

The phase structure of the calcined powders and sintered ceramics were characterized by X-ray diffraction (XRD, Bruker, D8 Discover) and Rietveld refinement techniques. Microstructure morphologies were observed using Field Emission Scanning Electron Microscopy (FE-SEM, Thermo Scientific Apreo S). Average particle size and average grain size were determined by employing the linear intercept method. The density of the ceramics was measured using Archimedes method. To assess the electrical properties, the ceramic samples were polished with sandpaper and coated with silver paste on both faces. Dielectric properties were measured with an LCR meter

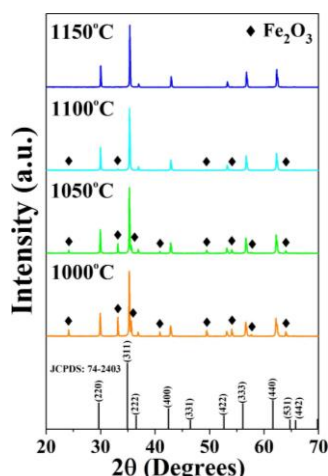


Figure 1. X-ray diffraction pattern of MZNF crystalline powders calcined at temperatures between 1,000°C and 1,150°C for 2 h, where (♦) is Fe₂O₃

(HP4284A, Hewlett-Pachard) over frequencies ranging from 20 Hz to 1 MHz at room temperature. The magnetic properties of the ceramics were determined using a vibrating sample magnetometer (Versa Lab, Quantum Design) at room temperature, and measured under a magnetic field of 15 kOe.

Results and Discussion

Figure 1 shows the phase evolution of MZNF powders using calcination temperatures in the range of 1,000-1,150°C for 2 h. The X-ray diffraction (XRD) results for the MZNF powders, after calcination, revealed a spinel (C) ferrite structure for all samples, matching the Joint Committee on Powder Diffraction Standards (JCPDS) files no.74-2403. A secondary impurity phase of Fe₂O₃ was detected in the powders calcined below 1,150°C for 2 h. The spinel phase purity percentage can be calculated by Equation (1).

$$\% \text{purity phase} = \left(\frac{I_{\text{max}}}{I_{\text{max}} + I_{\text{Fe}_2\text{O}_3}} \right) \times 100 \quad (1)$$

Where, I_{max} and $I_{\text{Fe}_2\text{O}_3}$ represent the intensities of the highest primary peak and the highest Fe₂O₃ impurity peak, respectively. The spinel phase purity percentage increased from ~77.2 to 100% as the calcination temperature increased (Table 1).

Figures 2(a–d) shows the microstructure and particle size distribution of MZNF powders calcined at temperatures ranging from 1,000 to 1,150°C for 2 h. The crystalline powders of all samples exhibited a polyhedral shape and were

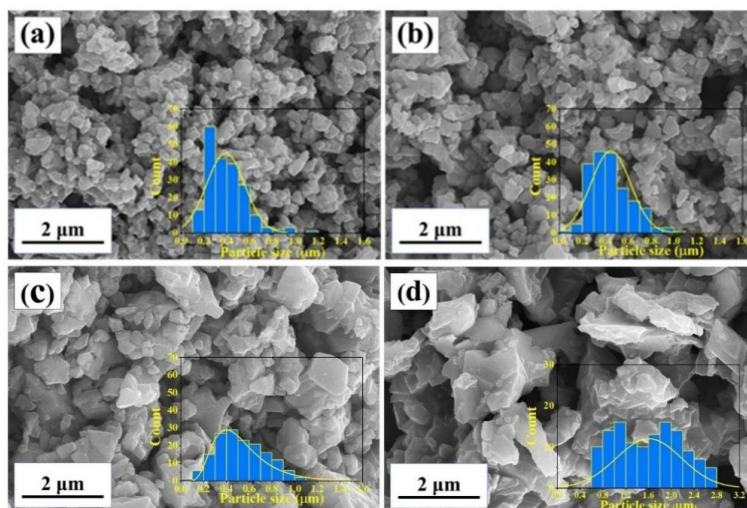


Figure 2. Microstructural images and particle size distribution of MZNF crystalline powder calcined at (a) 1,000°C, (b) 1,050°C, (c) 1,100°C, and (d) 1,150°C for 2 h

Table 1. Percent cubic spinel phase and average particle size of MZNF calcined powders

Calcination Temperature (°C)	Percent cubic spinel phase	Average particle size (μm)
1,000	77.2	0.38
1,050	86.9	0.44
1,100	92.4	0.91
1,150	100	1.61

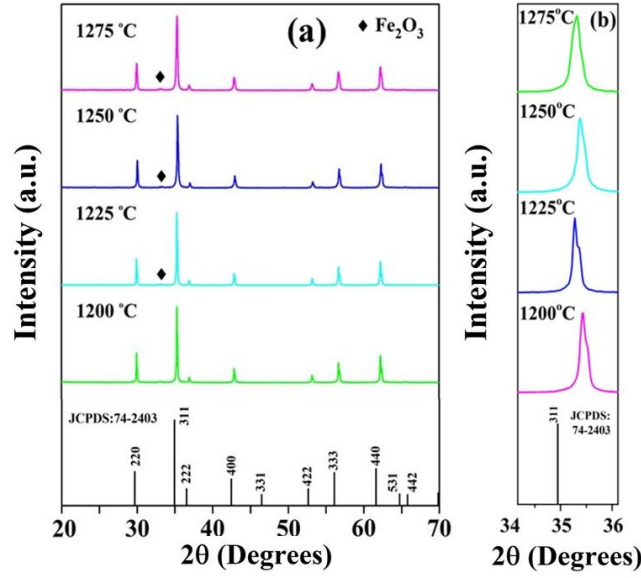
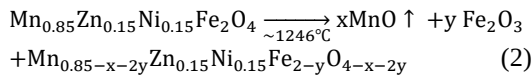


Figure 3. (a) X-ray diffraction pattern of MNZF ceramics, sintered at temperatures between 1,200°C and 1,275°C for 2 h, where (♦) is Fe₂O₃, (b) 2θ magnification around 35°

agglomerated. As the calcination temperature increased, the average particle size tended to increase from 0.38 to 1.61 μm, as shown in Table 1. Each sample displayed crystalline powders of similar size, as evidenced by the image inset showing a narrow particle size distribution curve.

The pure MNZF powder calcined at 1,150°C, was chosen to be sintered at different temperatures. Figure 3(a) shows the XRD patterns for diffraction angles (2θ) from 20° to 70° of the MNZF ceramics sintered between 1,200 and 1,275°C for 2 h. All sintered ceramics exhibited a typical spinel ferrites structure which corresponded to data standard JCPDS no.74-2403. A secondary phase of Fe₂O₃ appeared when the sintering temperature was higher than 1,200°C, and the secondary phase increased as the sintering temperature increased. The impurity phase can be attributed to the evaporation of Mn²⁺ at high sintering temperatures (as shown in Equation 2), as its melting point is 1,246°C (Coursey and Dragoset, 2000). This process promotes iron oxidation, resulting in the formation of Fe₂O₃ (Ghasemi and Mousavinia, 2014).



Where, x represents the amount of MnO evaporated from the system, y represents the amount of Fe₂O₃ produced by the oxidation reaction.

Figure 3(b) is a zoomed in section of the XRD patterns around a 2θ angle of 35°. Figure 3(b) demonstrates that the X-ray diffraction peaks resemble a pseudo-cubic spinel phase at sintering temperatures between 1,200 and 1,225°C, and then transforms into a more distinct single peak at temperatures above 1,250°C, indicating a more cubic spinel ferrite phase was obtained at higher sintering temperatures.

To evaluate the lattice parameters and unit cell volumes of all ceramics, Rietveld refinement was performed by fitting the XRD data with the FullProf program. The initial values for the cell parameters, space group, lattice parameters and atomic positions were obtained from the Crystallography Open Database (COD) structural model of MNZF with a cubic spinel phase and space group *Fd3m*. The peak shape was fitted using a Pseudo-Voigt function, while the background was fitted with a Chebyshev polynomial function, which is a linear combination of the Lorentzian and Gaussian functions (Reetu *et al.*, 2012). The weight-profile R factor (R_{wp}), profile reliability (R_p), anticipated residual (R_{exp}), and goodness of fit (χ^2) were determined using the following formulas (Bhuiyan *et al.*, 2010).

$$R_{wp} = \left[\frac{\sum_i w_i |y_{oi} - y_{ci}|^2}{\sum_i w_i y_{oi}^2} \right]^{1/2} \quad (3)$$

$$R_p = \frac{\sum_i |y_{oi} - y_{ci}|}{\sum_i y_{oi}} \quad (4)$$

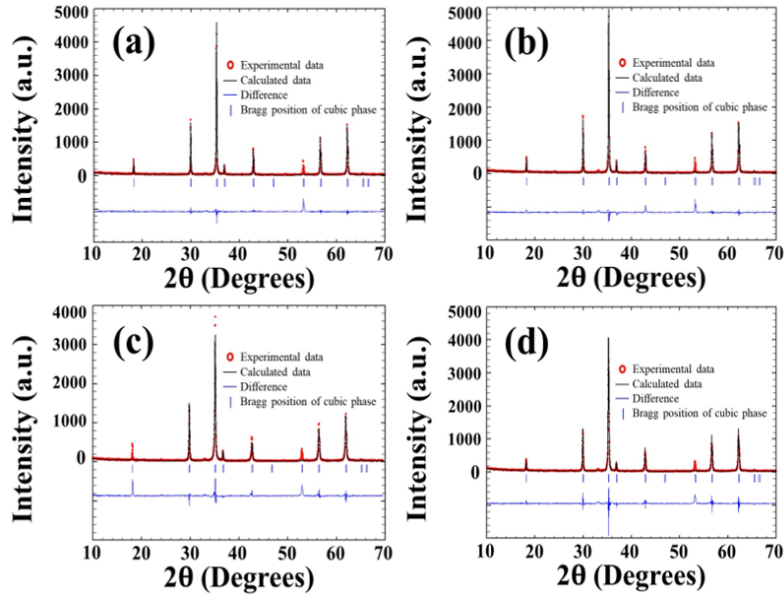


Figure 4. X-ray diffraction pattern of MZNF ceramics, sintered at (a) 1,200°C, (b) 1,225°C, (c) 1,250°C, and (d) 1,275°C for 2 h, analyzed with the FullProf program

Table 2. Results of structural analysis of sintered MZNF ceramics produced with various sintering temperatures, using the FullProf program

Sintering temperature (°C)	Lattice parameters (Å) a=b=c	Unit cell Volume (Å ³)	χ^2	Quality values		
				R _p	R _{wp}	R _{exp}
1,200	8.4331	599.746	2.42	12.1	18.5	11.9
1,225	8.4329	599.693	2.81	13.6	19.6	11.7
1,250	8.4743	608.571	3.53	15.0	21.8	11.6
1,275	8.4367	600.508	3.29	15.5	20.8	11.5

$$R_{exp} = \left[\frac{(n-p)}{\sum_i w_i y_{oi}} \right]^{1/2} \quad (5)$$

$$\chi^2 = \left[\frac{R_{wp}}{R_{exp}} \right]^2 \quad (6)$$

Where y_{oi} are the observed intensities, y_{ci} are the calculated intensities for diffraction angle 2θ and $w_i = (1/y_{oi})$ is an assigned statistical weight. n is the total number of data points; p is the number of adjustable parameters.

Figures 4(a-d) shows the Rietveld refinement of the XRD patterns of all the MZNF ceramics in the range from 10° to 70°. Table 2 presents χ^2 , R_p , R_{wp} , R_{exp} , lattice parameters, unit cell volume, and atomic information for all ceramics produced with different sintering temperatures. The fitting results confirmed that all samples had a spinel structure. The quality of the fits was calculated as $\chi^2 \leq 3.53$, $R_p \leq 15.5$, $R_{wp} \leq 21.8$ and $R_{exp} \leq 11.9$,

exhibiting good agreement between the observed and calculated intensity profiles for all ceramics. As indicated in Table 2, the lattice parameters and unit cell volume values demonstrated an upward trend until reaching a temperature of 1,250°C, followed by a subsequent decrease at 1,275°C.

FE-SEM images of the MZNF ceramics sintered between 1,200 and 1,275°C for 2 h are presented in Figures 5(a-d) to show the microstructure of the ceramics. All ceramics exhibited grains with an irregular polyhedral shape, rough surfaces, and anisotropic growth patterns. As the sintering temperature increased to 1,250°C, the number of pores decreased. Beyond this temperature, they became more numerous and larger. The grain boundaries become sharper as the sintering temperature increased. The average grain size of the ceramic samples sintered with different temperatures were calculated by the linear intercept method and is shown in Table 3. When the sintering

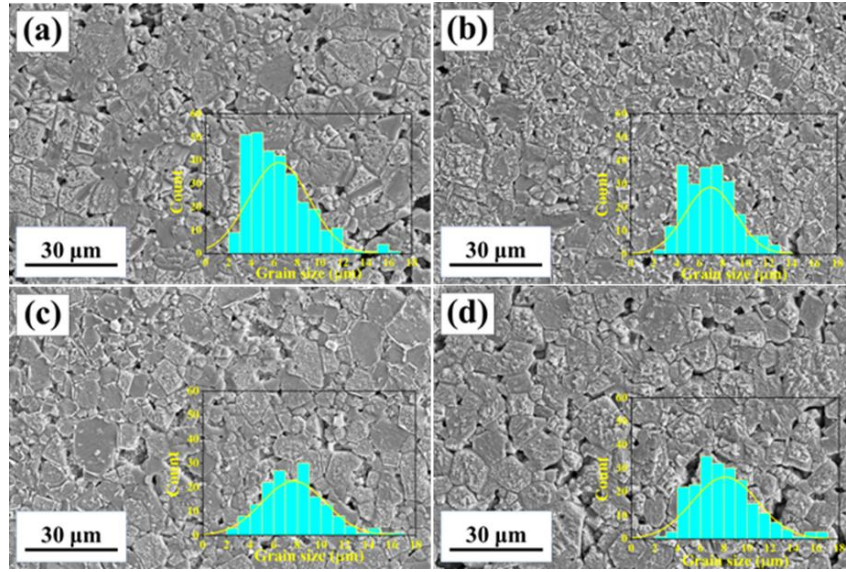


Figure 5. FE-SEM images and grain size distribution of MNZF pellets sintered at (a) 1,200°C, (b) 1,225°C, (c) 1,250°C, and (d) 1,275°C for 2 h

Table 3. Average grain size, measured density (d_{exp}), theoretical density (d_{th}), dielectric properties, and magnetic properties of MNZF ceramics

Sintering temperature (°C)	Average grain size (µm)	Density		Dielectric properties		Magnetic properties		
		d_{exp} (g/cm ³)	d_{th} (g/cm ³)	ϵ_r at frequency 1 kHz	$\tan\delta$ at frequency 1 kHz	M_s (emu/g)	M_r (emu/g)	H_c (Oe)
1200	6.38	4.74	5.34	73.9	12.1	63.40	3.44	2.13
1225	6.92	4.75	5.34	75.1	11.7	65.69	0.25	2.49
1250	8.03	4.87	5.26	78.3	17.7	84.67	2.50	1.42
1275	8.15	4.38	5.33	91.4	14.8	65.62	0.91	2.13

temperature increased from 1,200 to 1,275°C, the average grain sizes increased from 6.38 to 8.15 µm. The distribution is seen to be narrow in Figure 5(a) for the ceramic produced at 1,200°C and can be seen to progressively widen as the sintering temperatures rose in Figures 5(b), 5(c), and 5(d).

The Archimedes method was used to estimate the density of the sintered samples, with distilled water serving as the medium. The measured density increased from 4.74 to 4.87 g/cm³ and the theoretical density increased from 88.76 to 92.59% when the sintering temperature was raised from 1,200 to 1,250°C (Table 3). This correlated with a decrease in porosity. Following that (sintering temperature of 1,275°C), the density decreased as the porosity increased. The density of the samples is dependent on the sintering temperature, rising as the temperatures increased up to 1,250°C. This is from a decrease in pores due to diffusion kinetics (Bhuiyan *et al.*, 2010), as shown in the FE-SEM

micrographs of the prepared samples seen in Figures 5(a-c).

Figure 6 shows the variations in the dielectric constant (ϵ_r) and dielectric loss ($\tan\delta$) with frequency in the range of 20 Hz to 1 MHz, measured at room temperature. The ϵ_r and $\tan\delta$ reduced significantly with increased frequency. This is a normal dielectric behavior for spinel ferrites (Belavi *et al.*, 2012; Choudhury *et al.*, 2012). The dielectric behavior of ferrites can be understood through the dielectric polarization mechanism, which is akin to the conduction process. The extent of this exchange is determined by the concentration of Fe²⁺/Fe³⁺ ion pairs at the B sites in the ferrites. The observed decrease in the dielectric constant with increasing frequency occurs because the electronic exchange between Fe²⁺ and Fe³⁺ ions cannot keep pace with a high frequency applied AC field. This behavior can be explained using Koop's phenomenological theory (Koops, 1951),

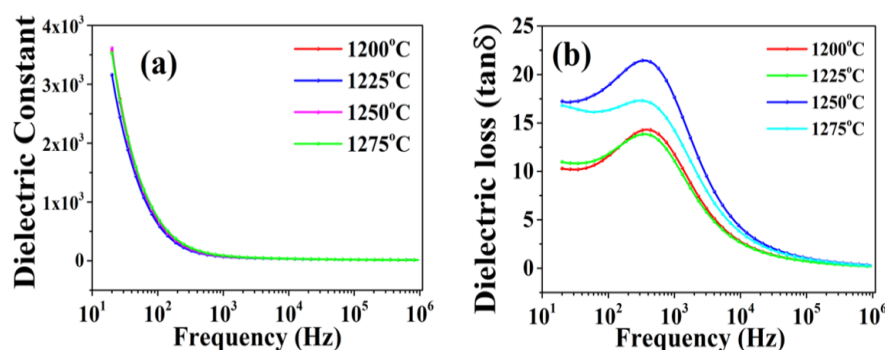


Figure 6. (a) Dielectric constant and (b) dielectric loss ($\tan\delta$) of MZNF ceramics sintered at 1,200-1,275°C for 2 h, measured at room temperature

which is based on the Maxwell-Wagner model for an inhomogeneous double-layer dielectric structure, which is produced due to the inhomogeneous dielectric structure of the material (Aliuzzaman *et al.*, 2014; Maxwell, 1873). The ϵ_r of the MZNF samples measured at a frequency of 1 kHz showed a trend to increase from 73.9 to 91.4 as the sintering temperature increased from 1,200 to 1,275°C. The $\tan\delta$ value increased from 12.1 to 17.7 when the sintering temperature was increased from 1,200 to 1,250°C, as seen in Table 3. The large grain size resulted from a reduction of the grain boundaries. This increase in grain size increased the mobility of the charge carriers, enhancing the efficiency of charge buildup, which in turn boosted the dielectric constant (Padurariu *et al.*, 2020).

In order to measure the magnetic properties, all ceramics were ground into fine powder. The magnetic properties of the MZNF ferrites produced with different sintering temperatures, using a magnetic field of 15,000 Oe at room temperature are depicted in Figure 7, which shows the hysteresis (M-H) loops for all samples. These loops are very narrow, indicating typical soft ferrite behavior. As the sintering temperature increased from 1,200 to 1,250°C, the saturation magnetization (M_s) rose from 63.40 emu/g to 84.67 emu/g and then decreased to 65.62 emu/g at the sintering temperature of 1,275°C. The coercive field (H_c) ranged from 1.42 to 2.49 Oe, which has the lowest value when sintered at 1,250°C, as detailed in Table 3. The highest M_s and the lowest H_c were obtained from the ceramic sintered at 1,250°C due to the reduced grain boundaries, which reduced the impedance to domain wall motion and enhanced the magnetic properties. Additionally, this sintering temperature enhanced the cubic spinel phase which promoted better alignment of the magnetic domains (Islam *et al.*, 2012; Han *et al.*, 2019; Zou *et al.*, 2023). In addition, the formation of Fe_2O_3 impurity phase also enhances

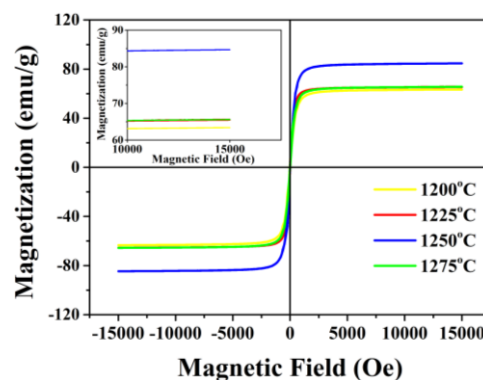


Figure 7. Magnetic hysteresis loops of MZNF ceramics sintered at 1,200-1,275°C for 2 h

the value of M_s because the magnetic properties are greatly influenced by the number of d electrons present in the metal ions. In Fe^{3+} ions, which have a $3d^5$ electron configuration, five unpaired d electrons are present, which makes them stable and gives them high magnetic moments. When Fe_2O_3 is alloyed with MnZn ferrite, these unpaired d electrons participate in superexchange interactions with other metal ions (Mn^{2+} , Zn^{2+}), which further affects the overall magnetic properties of the ferrite (Jayaprakash *et al.*, 2013). Although the ceramic sintered at 1,275°C had larger grain sizes, excellent crystallinity and higher Fe_2O_3 impurity phase, the increase in the number of pores lead to a reduction in the saturation magnetization. The solid-state combustion method, used to prepare the MZNF ceramics in this study, is more convenient, less expensive (Varma *et al.*, 1998; Merzhanov and Mukasyan, 2007), and produced better magnetic properties than previous Mn-Zn ferrite research (Fu *et al.*, 2010; Syue *et al.*, 2011; Mirshekari *et al.*, 2012; Islam *et al.*, 2012, 2013; Gabal *et al.*, 2013; Haralkar *et al.*, 2013; Zapata and Herrera, 2013; Phong *et al.*, 2015; Praveena *et al.*,

2015; Yadav *et al.*, 2015; Tsakaloudi *et al.*, 2019; Thawong *et al.*, 2019; Sonchaopri *et al.*, 2023). From our results, MZNF ceramics with good ferromagnetic behavior may be produced for applications including MRI equipment and electromagnetic interference (EMI) suppression (Hema and Sambhudevan, 2021).

Conclusions

The influence of the firing temperature on the microstructure, dielectric and magnetic properties were investigated on $\text{Mn}_{0.85}\text{Zn}_{0.15}\text{Ni}_{0.15}\text{Fe}_2\text{O}_4$ (MZNF) ceramics produced using the solid-state combustion method with glycine as the fuel source. All samples were calcined in the temperature range of 1,000–1,150°C for 2 h and sintered in the range of 1,200–1,275°C for 2 h. The XRD patterns indicated the formation of a pure-phase cubic spinel ferrite MZNF powder obtained at a calcine temperature of 1,150°C. Increasing the sintering temperature promoted the formation of the cubic spinel phase. Average particle size, average grain size, and ϵ_r exhibited a tendency to increase from 0.38 to 1.61 μm , 6.38 to 8.15 μm , and 73.9 to 91.4, respectively, as the firing temperatures were increased. The MZNF ceramic with the highest density (4.87 g/cm³) and good ferromagnetic behavior ($M_s = 84.67$ emu/g, and $H_c = 1.42$ Oe) was obtained using the sintering temperature of 1,250°C for 2 h which had high crystallinity and good morphology.

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