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Synthesization and Characterization of Dual Properties of Nickel Zinc Ferrite for Magnetorheological Materials

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ABSTRACT

The main objective of this study was to synthesize nickel zinc ferrite, $NiZnFe_2O_4$ particles which are believed can exhibit great dual properties of magnetic and electrical conductivity. Magnetorheological (MR) materials are one of the smart materials which respond toward the external magnetic field. Carbonyl iron particles (CIP) are commonly used as magnetic particles in MR materials as they can actively react to the applied magnetic fields. However, CIP is passive towards the electrical properties. Therefore, the synthesization of $NiZnFe_2O_4$ particles *via* sol-gel method was introduced to improve this limitation at sintering temperature of 1000°C. The particle characteristic analysis of $NiZnFe_2O_4$ were performed and determined *via* X-Ray Diffraction (XRD) for its elemental composition, Vibrating Sample Magnetometer (VSM) for magnetic properties, conductivity test using multimeter and Field Emission Scanning Microscope (FESEM) for morphological characterization and structure.

1. Introduction

Magnetic materials comprise a large range of materials that are used in a number of applications. It can be categorized into a few classes and can be produced in different shapes. The permanent magnet is the most accepted magnetic medium in which its magnetic properties can be preserved even without the presence of magnetic fields and may be shaped into different forms such as circular and rectangular bars according to their applications [1]. The intrinsic feature of a magnet is the presence of a spontaneous magnetization. In most magnetic materials, the temperature is decreased due to the increase of magnetization below their magnetic ordering temperature, Tc (Curie temperature) [2]. Higher permeability, greater saturation magnetization and lower remnant magnetization are highly desirable for magnetic particles to achieve stronger magnetic field-sensitive results [3]. This also provides high inter-particle attraction and thus results in a high magnetorheological effect [4].

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Smart materials are types of designed materials whose properties are controllable with the application of external stimuli such as the magnetic field, electric field, stress and heat. Smart materials whose rheological properties are controlled and influenced by externally applied magnetic fields are known as magneto-sensitive smart materials [5]. Magneto-sensitive smart materials are a class of smart composites prepared by dispersing nanometer or micrometer-sized ferromagnetic fillers into various carrier matrixes, often referred to as magnetorheological (MR) materials [3,6]. Any MR material is constituted by at least three elements that are composed of the particles, carriers and additives [7]. Dispersion of magnetic particles in a carrier medium of fluid, elastomer, grease, plastomer and foam is significantly important in MR materials as it has made the materials actively respond to the applied magnetic fields [8]. Upon application of a magnetic field, the rheological properties of these materials are altered rapidly and reversibly [4,9]. There are various applications of MR materials that comprise MR fluid, MR foam, MR plastomer, MR elastomer and MR grease. Novel applications include engine mounts, brakes and clutch systems in the automotive industry, shock absorbing safety devices for cockpit seats in aerospace and shock absorption from movement in semi-active human prosthetic legs [10]. Because the state of MR materials can be controlled by the strength of an applied magnetic field, it is useful in various applications where variable performance is desired. Microprocessors, damper, sensor technologies and increasing electronic content and processing speeds have created real-time control possibilities of smart systems that are applying MR devices [7,11].

Carbonyl iron particles (CIP) consist of highly pure irons which have been widely used in MR materials as magnetic particles because they have high magnetic saturation, low remnant and low coercivity. In contrast to other alloy and oxide powders, carbonyl iron particles with relatively high saturation magnetization (2.1 T/212.7 emu/g) and low cost are widely used [12]. In fact, in many of the MR materials and its applications, prior studies have documented the use of CIP [13-16]. These are because of changeable properties and quickly react to external magnetic fields that will result in excellent magnetic properties [17]. In addition, among various MR materials, CIP has been widely used as a magnetizable particle for MR fluids because of its high magnetic permeability, soft magnetic properties and common availability [18]. For example, there is a study on MR suspensions, which are structures whose rheological properties can be reversibly modified by a magnetic field and include one of the applications where CIP was used due to its favourable magnetic properties [19].

In the application of MR materials, magnetic particles in powder form are more desirable than a bulk shape since the powder form is quickly dispersed and evenly distributed in the carrier medium of MR materials. This is believed would result in the homogeneity properties of the materials [20]. Materials can be grouped into the following five major classes on the basis of magnetic response which are diamagnetic, paramagnetic, ferromagnetic, ferrimagnetic and antiferromagnetic materials [21,22]. Iron based CIP, which is the filler particles in MR materials and it is classified into the ferromagnetic materials, as well as cobalt, nickel and zinc as the common examples of ferromagnetic substances [23]. In addition, the MR effect is immediately reversible if the magnetic field is reduced or removed, and thus the recorded response time is in the range of 6.5 milliseconds [24].

However, despite the reversible changes of CIP in the presence of magnetic fields, it is passively responded to the electrical fields. Innovative engineering in various fields nowadays has led to development of macro-components and smart sensors that might need both magnetic and electrical properties. As examples, there is a study about MR prosthetic knee joints where previously the system has applied MR fluid [25]. Modification of the device however has changed the material into MR elastomer due to leakage issues. It was found that the magnetic performance toward sensors is brilliant but electrical performance is passive because CIP is commonly not a dual properties material

[15]. In order to invent smart sensors or other advanced applications that would need both excellent properties, these dual magnetic and electrical properties should be considered. Therefore, in the current analysis, the new synthesizing additives will be added into CIP are the major concerns to enhance both properties. There are very limited studies related to the use of $NiZnFe_2O_4$ in MR materials. The ones that have been reported in MR applications are in micron-size particles [26,27]. Besides, most previous researchers have outlined the synthesization of $NiZnFe_2O_4$ particles in micron-size instead of nano-size particles. On the other hand, there is also a study about the synthesization of $NiZnFe_2O_4$ particles that focussed on its magnetic properties only [28]. Based on the recent studies, there is no specific finding on these particles for magnetic and electrical properties. Therefore, the main objective of this study was to synthesize nickel zinc ferrite, $NiZnFe_2O_4$ particles as the combination of nickel and zinc that have high electrical conductivity and ferrite, Fe_2O_4 that has magnetic property. The combination is also known as hybrid particles and it is believed that this powder has great dual properties of magnetic and electrical.

2. Methodology

The synthesis of nickel zinc ferrite particles, $NiZnFe_2O_4$ from three raw chemicals using sol-gel method was established. The sintering process of $NiZnFe_2O_4$ particles took place at 1000°C. The characterizations of the final product of $NiZnFe_2O_4$ particles was done using X-Ray Diffraction (XRD) for the elemental composition, Field Emission Scanning Electron Microscopy (FESEM) was applied for morphological study, Vibrating Sample Magnetometer (VSM) for magnetic properties and digital multimeter for electrical properties analysis. Figure 1 presented the overall flow process of the research work in order to acquire $NiZnFe_2O_4$ particles that were believed can exhibit dual properties of magnetic and electrical conductivity.

2.1 Materials

The chemicals involved were nickel (II) acetate tetrahydrate ($Ni(CH_3COO)_2$), zinc acetate dihydrate ($Zn(CH_3COO)_2$) and iron (II) acetate ($Fe(CH_3COO)_2$) and acetic acid (CH_3COOH). The chemicals were procured by AR Grade from Sigma-Aldrich brand. The chemicals underwent a sol-gel method in order to synthesize and sinter the $NiZnFe_2O_4$ powder. Other apparatuses include hot plate (magnetic plate) with a magnetic stirrer, weighing balance, mortar and pestle, beaker and furnace.

2.2 Sol-gel Method

Sol-gel approach was selected for synthesizing dual properties of $NiZnFe_2O_4$ powder. The method basically consisted of two main steps, beginning with the synthesizing process of the powder and then the product underwent a sintering process at 1000°C. Description for each step will further be described in detail.

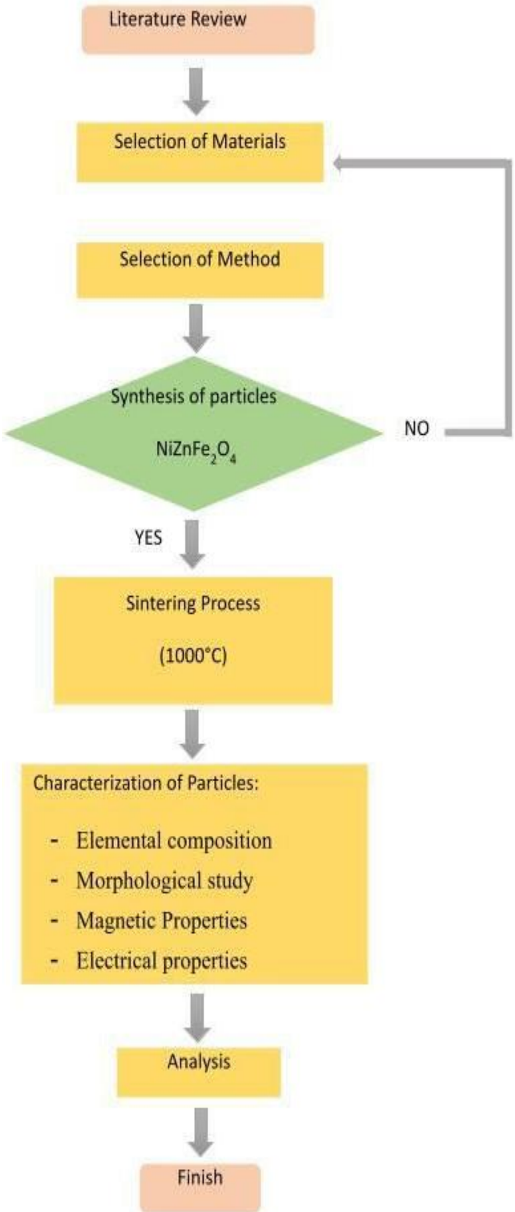


Fig. 1. Flow chart of the experimental process

2.2.1 Synthesize process of nickel zinc ferrite, $NiZnFe_2O_4$ particles

The purpose of the synthesize process was to produce a desired material or product by executing chemical reactions. In this study, $NiZnFe_2O_4$ particles were synthesized *via* sol-gel method. The starting raw materials were nickel acetate ($Ni(CH_3COO)_2$), zinc acetate ($Zn(CH_3COO)_2$) and iron acetate ($Fe(CH_3COO)_2$) of high purity with acetic acid (CH_3COOH) as precursor in this preparation. The experiment began with the weighing of the desired amount of constituents, put separately into three beakers and mixed with 30 ml distilled water for each of the beakers respectively. These three mixtures were stirred gradually for 15 mins until a homogeneous liquid was formed using a magnetic plate with its magnetic stirrer at 600 rpm. Then, these mixtures were mixed together into one beaker and continuously adding 50 ml of 0.1 M acetic acid at a constant temperature of 80°C. This is where the mixture is called 'sol'. The process of stirring and heating took about less than 48 hours to form a dark brownish gel. The gel was continuously heated for another few hours to complete the drying process. To get a finer residue of nano-scale particles, the dried powder was calcined at 400°C for 6 hours.

2.2.2 Sintering process of nickel zinc ferrite, $NiZnFe_2O_4$ particles

The purpose of the sintering process was to produce the pure phase of single-phase spinel structures of the compound and to make the material be more compact [17]. However, before the sintering process started, the $NiZnFe_2O_4$ particles were ground for a while with mortar and pestle to break the agglomerated powder and acquire finer product. In the sintering process, the synthesized powder was heated up at the temperature of 1000°C. It begins with heating up the $NiZnFe_2O_4$ particles at 100°C for 1 hour to warm up the grain of the particles before absorbing energy at higher temperature and then continue the sintering process until it reaches the desired temperature at 1000°C. The brand Carbolite of the furnace was used as it was capable of changing a temperature of 1°/10 seconds. Once the temperature reached 1000°C, the sintering process started for around 8 hours.

2.2.3 Characterizations of $NiZnFe_2O_4$ particles

The $NiZnFe_2O_4$ particles that sinter at 1000°C underwent various characterizations, in terms of morphological structure, chemical composition, magnetic and electrical properties. The morphological characteristics were characterized *via* FESEM from the brand JEOL with a model of JSM-7800F as shown in Figure 2 (a). These particles were believed to exhibit a spherical shape. The FESEM equipment was also equipped with Energy Dispersive X-Ray (EDX) that functions to check the chemical composition individually [29]. The composition of $NiZnFe_2O_4$ particles were acquired through XRD from the type of X-Ray Diffractometer Empyrean as shown in Figure 2 (b) [30]. Meanwhile, the magnetic properties of the $NiZnFe_2O_4$ particles were examined *via* VSM from the brand MicroSense USA as shown in Figure 2 (c) [31]. Also, the electrical properties of $NiZnFe_2O_4$ particles were then examined using a digital multimeter with two probes as shown in Figure 2 (d).

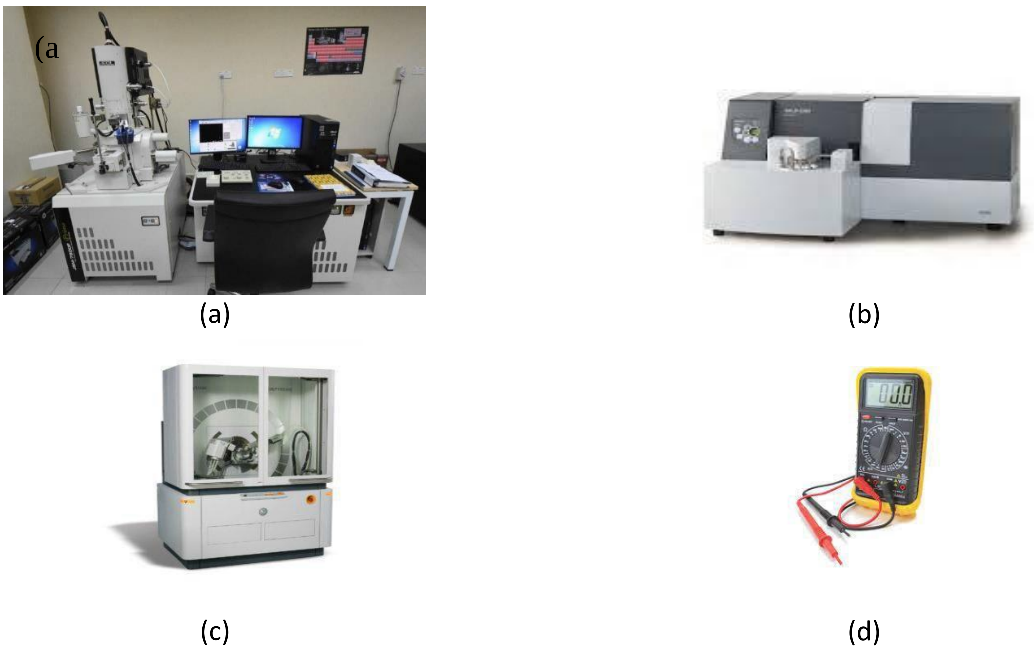


Fig. 2. Equipment used for characterization of $NiZnFe_2O_4$ particles (a) FESEM, (b) XRD, (c) VSM and (d) Digital multimeter

3. Results and Discussion

3.1 Elemental Composition

Figure 3 shows the XRD pattern of $NiZnFe_2O_4$ particles sintered at 1000°C. All of the peaks were compared to the nickel zinc ferrite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$) XRD pattern and matched to the standard ICDD database of 98-018-8043. From the analysis, there were ten major spinel ferrite peaks identified in the selected ICDD database. The peaks were (022), (113), (222), (004), (224), (115), (044), (026), (335) and (226) planes of a cubic unit cell related to the spinel structures. The sample was successfully identified as pure $Ni_{0.5}Zn_{0.5}Fe_2O_4$ crystalline phase since there was no unidentified peak discovered in the graph pattern [32]. The most narrow and highest line on the intense peak (113) exhibited the purity yielded *via* sol-gel method. It showed that the synthesization process of $NiZnFe_2O_4$ particles *via* sol-gel method was achieved.

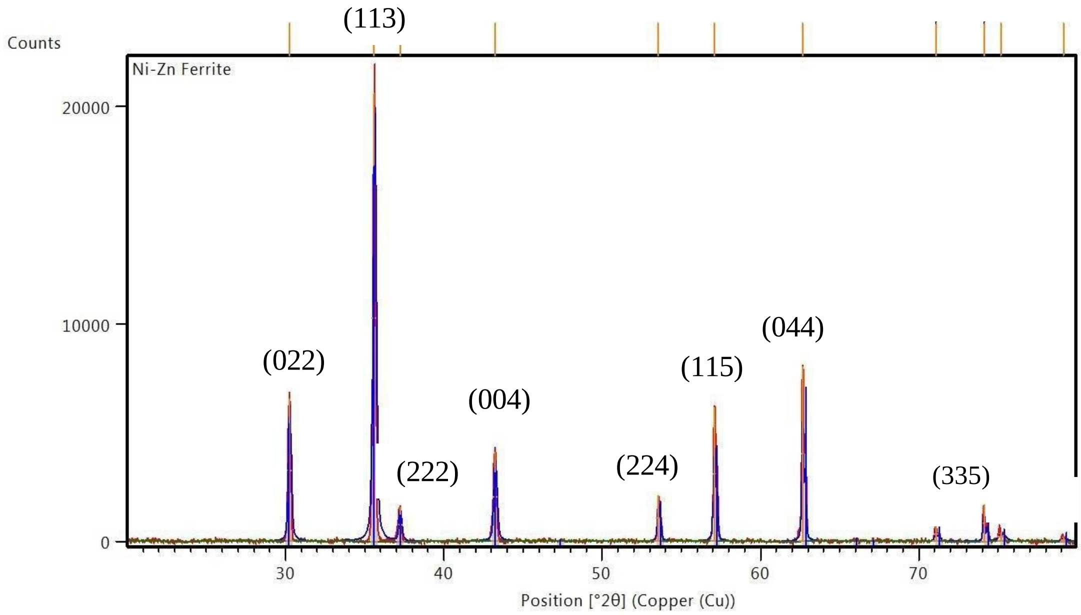
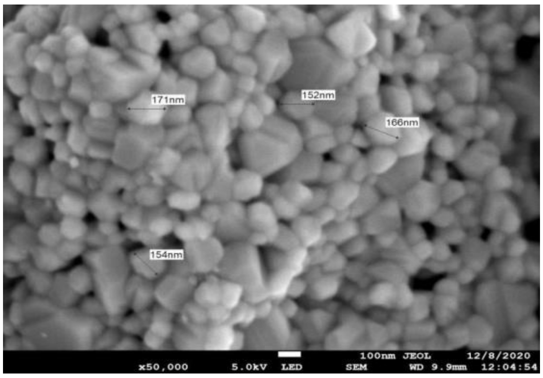


Fig. 3. XRD pattern of $NiZnFe_2O_4$ particles, particularly sintered at 1000°C

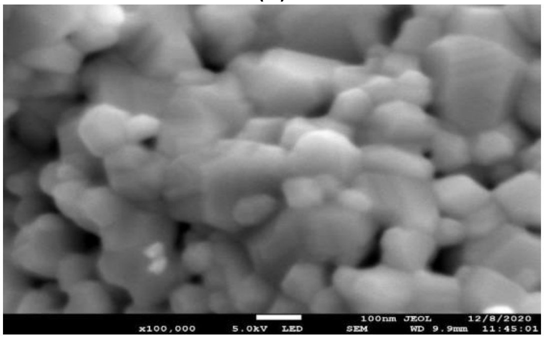
3.2 Morphological Characterizations

Figure 4 illustrates the morphology of $NiZnFe_2O_4$ particles obtained from FESEM under a few magnification values, which were x50000, x100000 and x150000.

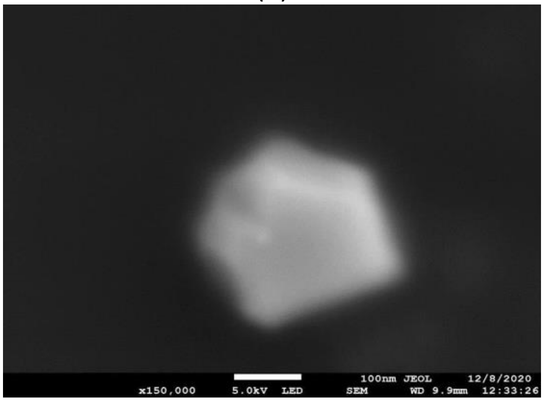
Under the magnification value of x50000 as in Figure 4 (a), the size particles of $NiZnFe_2O_4$ can be obtained in nano-size which were 152 nm, 154 nm, 166 nm and 171 nm. The smaller the particles, the easier it can attach in the CIP matrix, so that will result in good performance for applications that need magnetic and electrical properties. In addition, it can be seen that the particles exhibit an irregular polygonal shape and there was also an octahedron shape of particles. However, the $NiZnFe_2O_4$ particles were shown to be agglomerated as in Figure 4 (b). In Figure 4 (c), it is shown that the FESEM image captured a single particle of $NiZnFe_2O_4$ under the magnification value of x150000. From my view, perhaps it is illustrated as the cuboid shape of the particle.



(a)



(b)



(c)

Fig. 4. (a) Size particles of $NiZnFe_2O_4$, (b) Shape of $NiZnFe_2O_4$ and (c) Single particle of $NiZnFe_2O_4$

EDX analysis was carried out to determine the chemical composition of the observed powder. Figure 5 presents the image under EDX of the $NiZnFe_2O_4$ particles for five spots at 200 nm. From the analysis, the $NiZnFe_2O_4$ particles were composed of Ni, Zn, Fe and O as listed in Table 1. The atomic and weight percentage of oxygen were the highest composition element in $NiZnFe_2O_4$ particles for all the spots captured. Meanwhile, zinc was considered as the lowest composition element contained in the particles for both atomic and weight percentage, respectively.

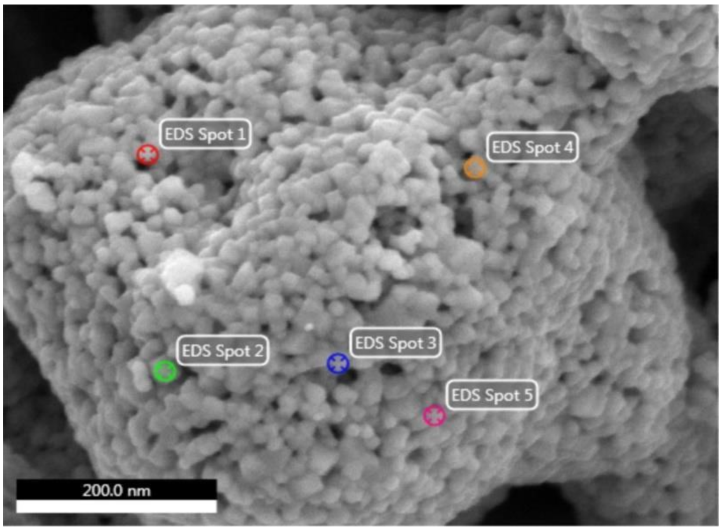


Fig. 5. Image under EDX of $NiZnFe_2O_4$ particles

Table 1
Atomic and weight percentage of elements in $NiZnFe_2O_4$ particles

Location	Percentage (%)	O	Fe	Ni	Zn
EDS Spot 1	Weight	36.43	33.76	15.77	14.03
	Atomic	67.67	17.97	7.98	6.38
EDS Spot 2	Weight	39.95	32.87	13.83	13.35
	Atomic	70.83	16.70	6.68	5.79
EDS Spot 3	Weight	41.20	32.68	13.48	12.64
	Atomic	71.87	16.33	6.41	5.40
EDS Spot 4	Weight	36.49	34.18	15.85	13.49
	Atomic	67.70	18.17	8.01	6.12
EDS Spot 5	Weight	47.32	31.36	9.86	11.46
	Atomic	76.58	14.54	4.35	4.54

3.3 Magnetic Properties

Figure 6 presents the hysteresis loop of $NiZnFe_2O_4$ particles at a sintering temperature of 1000°C. From the experiment, the value of magnetic saturation, M_S, the remanence, M_r and the coercivity, H_C obtained were 59.1 emu/g, 2.30 emu/g and 38.97 Oe respectively. The magnetic saturation, M_S can be achieved when all domains are oriented with the applied magnetic field. The lower the remanence, M_r of $NiZnFe_2O_4$ particles, the lower the chances to lose their magnetic energy after release of applied field. $NiZnFe_2O_4$ particles experienced small coercive force as the shape of the hysteresis loop shown in the graph was very small and narrow. Correspondingly, low coercivity, H_C in the $NiZnFe_2O_4$ particles, proved that it is a group of soft magnetic materials and hence, it can be easily magnetized and demagnetized. Low coercivity was important to minimize the hysteresis losses. As the outcome, it was proven that $NiZnFe_2O_4$ particles have magnetic properties at the temperature of 1000°C.

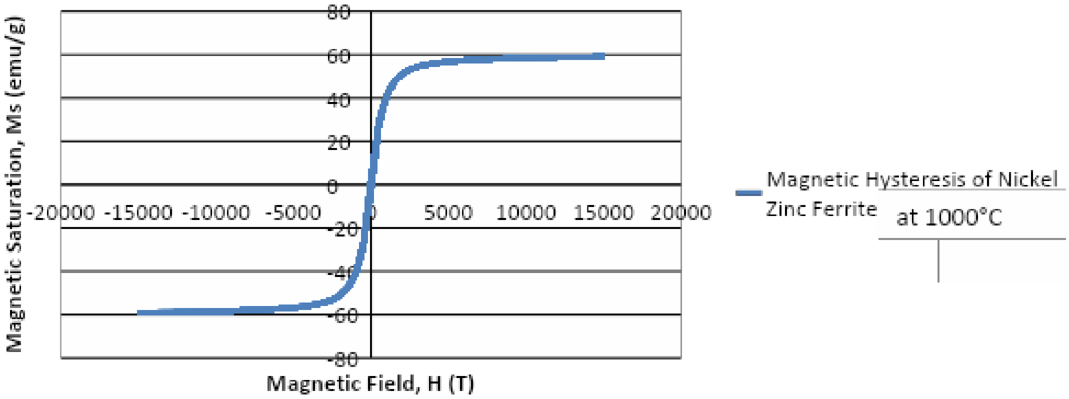


Fig. 6. Magnetic hysteresis of $NiZnFe_2O_4$ particles at sintering temperature of 1000°C

Table 2
Significant data from magnetic hysteresis of $NiZnFe_2O_4$ particles

Temperature	1000°C
Ms (emu/g)	4.28
Mr (emu/g)	2.30
Hc (Oe)	38.968

3.4 Conductivity Test

Conductivity of $NiZnFe_2O_4$ particles were obtained using a digital multimeter. The electrical conductivity of $NiZnFe_2O_4$ particles were tested using two probes after the powders were pressed into a pellet shape. Several readings from the multimeter were taken and the average value was calculated. The resistivity of the nickel zinc ferrite particles were noted to be around $8.1828x10^9$ Ω.m showing that it exhibited electrical properties at 1000°C. The resistivity was reciprocal of conductivity. High resistivity means low conductivity because they are inversely proportional to each other.

4. Conclusions

The synthesization of $NiZnFe_2O_4$ particles through the sol-gel method and being sintered at 1000°C were achieved. The characterization of $NiZnFe_2O_4$ particles shows that the magnetic saturation, M_S is 4.28 emu/g. Meanwhile, the conductivity test conducted using a digital multimeter shows the resistivity value of the resistivity obtained through conductivity testing is $8.1828x10^9$ Ω.m. From these testing, it can be proven that $NiZnFe_2O_4$ particles exhibit both magnetic and electrical properties at 1000°C.

In addition, the shape of $NiZnFe_2O_4$ particles were mostly in polygonal shape, with the average size of 152 nm, 154 nm, 166 nm and 171 nm at sintering temperature of 1000°C, respectively. With these dual properties of magnetic and electrical, it is believed that the synthesized powder could be further applied in MR materials and corresponding rheological effect could be further analyzed. Perhaps, the $NiZnFe_2O_4$ particles would be a great replacement for CIP in MR materials especially for devices that require response to the applied magnetic and electrical field.

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