Magnetic and Microwave Absorption Properties in X and Ku-Band Frequencies of $Ni_{0.5}Zn_{0.5}Fe_3O_4$/Ag Core-Shell Micro-nanostructures

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---**ABSTRACT**---
We report on magnetic and microwave absorption properties in X-band (8-12 GHz) and Ku-band (12-18 GHz) of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag core-shell nanostructures. Silver powder was covered with $Ni_{0.5}Zn_{0.5}Fe_2O_4$ spinel ferrites, forming a core-shell nanostructure synthesized by dry mixing at different ferrite/silver ratio (7:1, 5:1 and 3:1 %wt). The spinel ferrite nanoparticles (~25 nm) have been synthesized from iron sand by coprecipitation method and the pure silver powder with a typical particle size of ~3 µm. The core-shell nanostructures morphology has been observed by SEM. Magnetic properties and microwave absorption of these systems were measured using Vibrating Sample Magnetometer and Vector Network Analyzer. It is shown that the increasing silver powder content ratio in core-hell nanostructures considerably reduced both the saturated magnetization (Ms) and remanence magnetization (Mr) from 50.67 to 39.73 emu/gr and 12.82 to 10.36 emu/gr respectively, on the other hand it is not significant for coercive force (Hc). These results suggest that silver powder has reduced magnetic properties.Ni0.5Zn0.5Fe2O4/Ag core-shell micro-nanostructures can absorb microwaves in the X and Ku-band with Reflection Loss < -20 dB (~99% absorbed). The optimum composition for microwave absorption is 5:1 %wt with a value of RLm = -40.27 dB (fm = 10.02 GHz) and -67.54 dB (fm = 12.52 GHz). This shows the effectiveness of the combination of microwave absorption by maganteic dipole moments in ferrite and reflection by silver microparticles so that constructive repetition of microwave absorption occurs in core shell micro-nanostructures.

Keywords: core-shell, ferrites, irond sand, nanostructures, microwave absorption.

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I. INTRODUCTION

Polycrystalline spinel ferrites are widely used in many electronic devices. These are preferred because of their high electrical resistivity, mechanical hardness, chemical stability, and reasonable cost [1]. These systems made up of nanoparticles are intensively studied both theoretically and practically due to their electric, dielectric, and magnetic properties that are sensibly different from those of the bulk materials and their possible applications in various fields [2, 3].

Ni-Zn ferrites are well known technological magnetic materials finding their applications. The physical properties of the Ni-Zn ferrite are very sensitive to the method of preparation, the amount, and the type of substitutions [4]. Nickel and Zinc are known to have very strong preference for the tetrahedral and octahedral sites, respectively, making Ni-ferrite a model inverse ferrite and Zn-ferrite a model normal ferrite. However, nano sized Ni- and Zn-ferrite are known to exist as mixed spinel structures. Various synthesis processes have been used for preparing nano-sized ferrite particles. These include hydrothermal, sol-gel, chemical co-precipitation, sonochemical reactions, mechanical ball milling, etc. Of all these processes, chemical co-precipitation seems to be the most covenient method for the synthesis of nanomagnetic particles as it is very simple and has better control over crystallite size and other properties of the materials [5].

One of Ni-Zn ferrite applications is microwave absorbers. Microwave absorbers from ferrite-related materials used in a high frequency such as X band were particularly noticed. Electronic instrument coatings with microwave absorbers avoid the interferences of electromagnetic waves, while the battle planes coating with microwave absorber evade the detecting by radar. Usually, ferrite composites with a conducting plate as a backing material are used to achieve microwave absorption. The absorbing characteristics of the materials depend on the frequency, layer thickness, complex permittivity, and permeability. The absorbing characteristics could also be changed by modifying the layer thickness of absorbing materials. There were also reports on nanocrystals and core-shell structures of magnetic particles which exhibit unique magnetic properties [6].

However, studies of both nanoparticles and core-shell micro-nanopowders for the applications in microwave absorbers were very limited, specifically for magnetic properties.

In the present study, the effectof $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag with a core-shell micro-nanostructure on magnetic and microwave absorption properties behavior is reported. $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocrystalline has been synthesized from irond sand by co-precipitation method and core-shell micro-nanostructure prepared by dry mixing method at different ferrite/micro silver ratio (7:1), (5:1) and (3:1) %wt. To construct core-shell micro-nanostructures, we choose silver and ferrite because (a). Ag and ferrite have unique properties (surface plasmon resonance and superparamagnetism, respectively), (b). both Ag and ferrite nanoparticles are biocompatible and have low cytotoxicity, (c). The surface chemistry properties are different but complementary for Ag and ferrite.

II. EXPERIMENT METHOD

$Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticle have been synthesized by a chemical co-precipitation route method. The starting materials used were pure powder Ni (PA = 99.9%), Fe_3O_4 (pure powder and based iron sand) and Zn (PA = 99.9%), its powder dissolved used HCl (M = 12.063 M, PA = 99.9%) to make $NiCl_26H_2O$, $FeCl_36H_2O$, $FeCl_24H_2O$ and $ZnCl_2$ solutions. Specifically, Fe_3O_4 based iron sand formed by extracted used permanent magnet. For synthesis, equimolar solutions of $NiCl_26H_2O$, $FeCl_36H_2O$, $FeCl_24H_2O$ and ZnCl2 were mixed in their stoichiometric ratio and homogenized at 70 ℃. To this, 25% ammonia solution was added dropwise with contant stirring. All the the chemicals were of analythical grade. The pH of the solution was maintained at 8.5. The mixture was then heated at 80 ℃ for about 1 hour. To prevent agglomeration a surfactant coating of oleic acid was made to the individual particles in the beginning itself. The precipitated particles were then washed several times with double distilled water to remove the salt residues, chloride ions and other impurities. It was further dried at 100 ℃ to obtain the powder. The samples were cooled by slow cooling to room temperature. The dried powder was mixed homogeneously in a cleaned agate mortar and pestle. Silver powders were used pure analythical grade with partcle size ~2-3.5 μm. So, $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag core-shell nanostructures samples synthesized by dry mixing method at different ratios (7:1, 5:1 and 3:1 %wt). The samples were then characterized through various characterization techniques.

The physical characterization was performed by X-ray diffractometer (type: JEOL-3530, with Cu Kα1 λ = 54 Å, 40 kV, dan 30 mA) for analysized particles size and phase, Transmission Electron Microscope (TEM, model: JEM 1200 EX, JEOL) for analysized nanoparticle morphology, Scanning Electron Microscopy (SEM, merk FEI, Type INSPECT S50) for analysized powder particle morphology, while the magnetic characterization was done by vibrating sample magnetometer (VSM, model: Oxford VSM 1.2 H).The microwave absorption in 8 – 18 GHz frequencies has been measured using Vector Network Analyzer (VNA), Advantest R-3770 model.
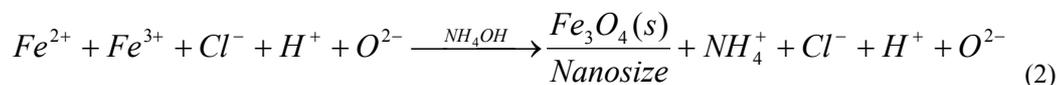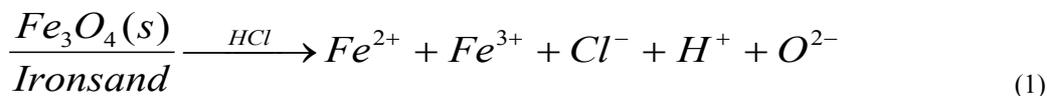
III. RESULTS AND DISCUSSION

3.1. Synthesis of Nano Particles Fe_3O_4 from Iron Sand

Fe_3O_4 powders as raw materials manufacturing magnetic nano particles obtained by the extraction of iron sand powder. Extraction was performed to obtain Fe_3O_4 powders with a strong magnet and separated from other waste using a strong permanent magnet. To ensure powder containing iron sand dominant strong magnetic phase (Fe_3O_4) is carried out phase identification using XRD, as shown in Figure 1a.

The search match results of the analysis Figure 1a, according with reference to PDF number 24-0734 for Fe_3O_4, show that in Fe_3O_4 hematite phase contains a significant (> 75%). These results are in accordance with the test content element composition using XRF in which the Fe of 76.92% so that the iron sand powder can be used as nano particles synthesis of magnetic Fe_3O_4. Peaks diffraction analysis showed that Fe_3O_4particles contained in the iron sand and there was another phase of micrometer size.Synthesized of magnetic nano particles was carried out by changing the particle size (crystal size) Fe_3O_4 from iron sand micron-sized particles into nanometer-sized Fe_3O_4 and simultaneously trying to minimize the waste that may still exist through co-precipitaion methods. The dominant iron sand contains the first phase of Fe_3O_4 dissolved in a solution of HCl. So, in the solution there are ions Fe^{2+} and Fe^{3+} causes Fe_3O_4 phase formation. The reaction of this solution is simple, shown in the following equation (1). Then performed co-precipitation process by adding NH_4OH into the solution and thick black mud will form quickly in the process of this rainfall. This may cause the formation of Fe_3O_4 particles nanometer size. The reaction can be written schematically as following equation (2).
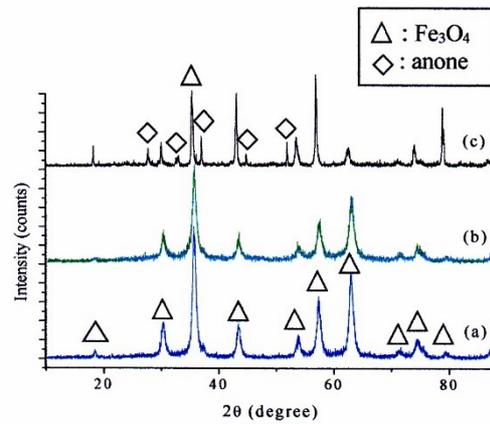
Fig. 1. X-ray diffractogram of (a). nano Fe₃O₄ based FeCl₃ 6H₂O and FeCl₂ 4H₂O, (b). nano Fe₃O₄ based iron sand, (c). powder iron sand

$$\frac{Fe_3O_4(s)}{Ironsand} \xrightarrow{HCl} Fe^{2+} + Fe^{3+} + Cl^- + H^+ + O^{2-} \tag{1}$$

$$Fe^{2+} + Fe^{3+} + Cl^- + H^+ + O^{2-} \xrightarrow{NH_4OH} \frac{Fe_3O_4(s)}{Nanosize} + NH_4^+ + Cl^- + H^+ + O^{2-} \tag{2}$$

As comparisons of the success making nano particles are made of powdered raw materials Fe₃O₄ magnetic iron sand production is also carried out magnetic Fe₃O₄ nano particles are made of feroic ferrous solution by co-precipitation method. Phase identification and estimates of crystal size of Fe₃O₄ magnetic nano particles that make up the tests conducted using XRD. X-ray patterns of Fe₃O₄ nano particles shown in Figure 1b and 1c. The results of phase identification showed that the peaks detected in a sample of hematite is the culmination of phase Fe₃O₄ (11.68 ± 0.35 nm) and undetectable impuritas phase pollutants originating from iron sand. This is in accordance with the characteristics of nano particles Fe₃O₄ pattern (17.64 ± 0.58 nm) made of ferroic ferrous solution, Figure 1c.

3.2. Phase identification core-shell micro-nanostructures.

Figure 2 shows the crystalline phases of core-shell micro-nanostructures, in which the peaks of silver and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ are evident. It confirms the co-existance of ferrite spinel phase and silver phase. For the core-shell micro-nanostructures, the increasing of the silver powder content (low ferrite/silver ratio) increases the intensity of silver's peaks and the phase of spinel becomes moderate. For ferrite/Ag weight ratio 3:1 %wt (Fig.2, curve b), the peaks of silver are very sharp and much stronger than those of spinel peaks. The peak intensity of spinel becomes small as its content in the $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag core-shell particle decreases. The broad peaks of spinel also imply that the particle size of spinel phases was very fine, which was like our previous report.

3.3. Morphology and Magnetic Properties

Figure 3a shows TEM nanograph of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanopowders. It's showed that the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ particles (dark area) were deposited with numerous nanometer-sized spherical, which were estimated to be in the range of ~25 nm. The silver particles (contrasts area) as shown micrograph SEM, Figure 3b. It's showed that the silver particles were numerous micrometer-sized spherical particle silver, which were estimated to be in the range of ~3 μm. Figure 4c, shows SEM micrograph of the core-shell nanopowders for different $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag ratio (7:1 %wt). For these ratios, the nanocrystals of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ agglomerate and provide a shell around silver particles. The thickness of shell varies depending on the $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag ratio, which ranges as a negligible thickness (uncovered) for 3:1, 5:1 and 7:1 %wt. High $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag ratio results in a covered surface or thick full shell as shown in Figure 3c. From the observation of SEM, dry mixing method is

applicable for synthesis of core-shell nanostructures but may not be versatile to be able to control the uniformity of the shell thickness. This image morphology of nanoparticle and core-shell micro-nanostructure agrees very well with the XRD measurement [7].
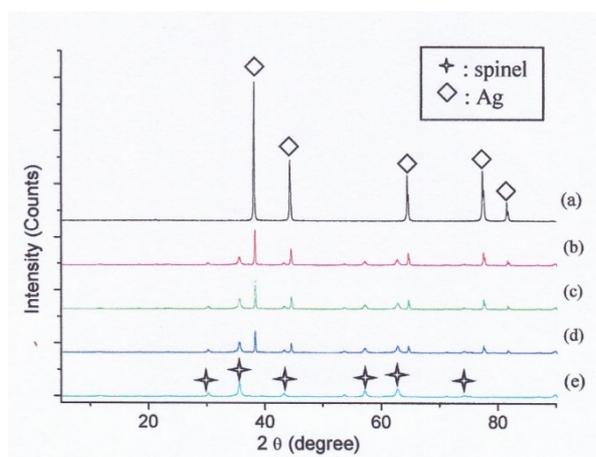

Figure 2. X-ray difractogram of (a). Ag powder, core-shell micro-nanostructures $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (b). 3:1 %wt, (c). 5:1 %wt, (d). 7:1 %wt, (e). $Ni_{0.5}Zn_{0.5}Fe_2O_4$
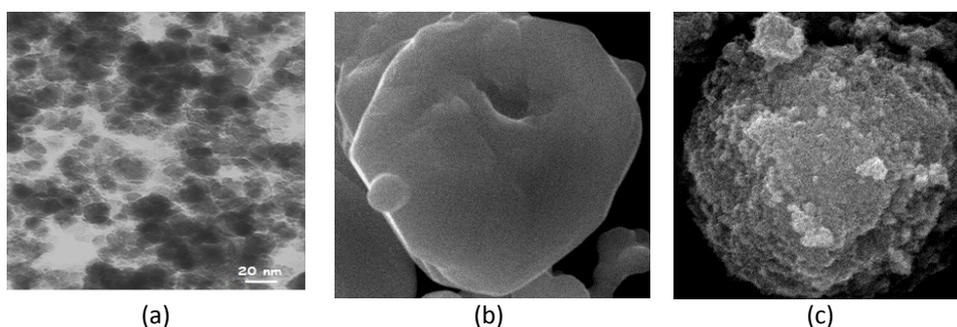
Figure 3. (a). TEM images of $Ni_{0.5}Zn_{0.5}Fe_2O_4$, (b). SEM image of silver powder, (c). SEM image of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (7:1 %wt)core-shell micro-nanostructures

The magnetic properties of these core-shell nanostructures are analyzed using a vibrating sample magnetometer (VSM). The VSM analyses of the silver-ferite core-shell micro-nanostructures are shown in Figure 4 and the magnetic properties of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag core-shell micro-nanostructures depend on $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag ratio (7:1, 5:1, 3:1 %wt) are given in Table 1. The increasing Ag particles content ratio in core-shell micro-nanostructures considerably reduced both the saturated magnetization (M_s) and remanence magnetization (M_r) from 50.67 to 39.73 emu/gr and 12.82 to 10.36 emu/gr respectively, on the other hand it is not significant for coercive force (H_c). These phenomenon magnetic properties of core-shell micro-nanostructurescause the greater fraction of surface at interface, covered thickness and dispersivity of core-shell structure [8]. It was observed for samples showing a super paramagnetic behavior. So, the magnetic $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag core-shell nanostructures are attributed the superparamagnetic although Ag powder is not magnetic.
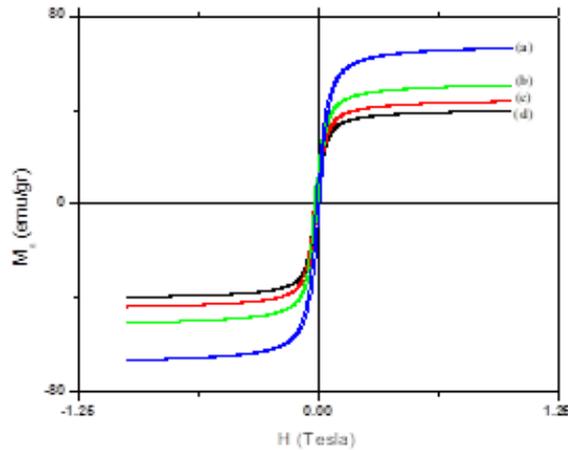
Figure 4. Dependence of magnetization on the applied magnetic field at room temperature (a). $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticle, and core-shell micro-nanostructures $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (b). 7:1 %wt, (c). 5:1 %wt, (d). 3:1 %wt

Table. 1. Magnetics of $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag micro-nanostructures.

Samples	M_s (emu/gr)	M_r (emu/gr)	H_c (Tesla)
$Ni_{0.5}Zn_{0.5}Fe_2O_4$	66.44	11.82	0.010
$Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (7:1 %wt), core-shell	50.67	12.82	0.019
$Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (5:1 %wt), core-shell	43.78	11.09	0.019
$Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (3:1 %wt), core-shell	39.73	10.36	0.018

III.4.   Microwave Absorption in X and Ku-band.

To determine the absorption properties of microwaves of the as-synthesized samples, reflection loss of samples was measured by using VNA advantest R-3770 in 8-18 GHz frequency at room temperature. The microwave absorbing properties of the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles and $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag micro-nano structures defined by maximum reflection loss (RL_m) are given by theory of the absorbing wall and based on the transmit-line theory [9], the maximum reflection loss is calculated by equation:
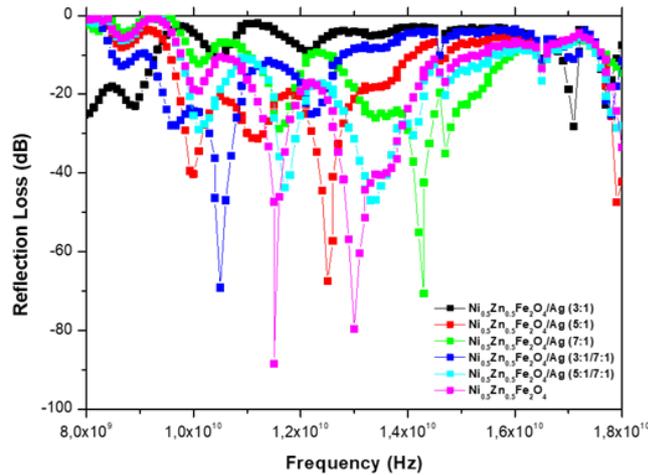

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where is the input impedance of the absorber, is the velocity of electromagnetic waves in free space, is the microwave frequency, and is the absorber thickness. There are two ways to increase the absorption of radar waves. First, the material impedance value must be the same as the free space impedance so that microwaves can be absorbed maximally. Another way to increase is by producing a zero reflect state and incoming waves must be absorbed with maximum intensity when the waves reach the absorbent material.

The results of Reflection Loss measurements from the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ micro nanostructure using the Vector Network Analyzer in the X and Ku-band are shown in Figure 5 and Table 5. $Ni_{0.5}Zn_{0.5}Fe_2O_4$ (3:1, 5: 1, 7:1 %wt) micro-nanostructure has the property of absorbing microwaves in the X and Ku-band with a reflection loss value of < - 20 dB (~99% absorbed). Microwave absorption is determined by the magnetic dipole moment in ferrite nanoparticles, while silver microparticles as a metal material have an electric dipole which is conductive and makes it easier to reflect microwaves to ferrites nanoparticles, making it easier for repeated absorption to occur by the magnetic dipole moment in ferrite [10, 11]. The presence of silver microparticles as the core in the micro-nano structure causes the reflection speed to reduce the absorption of microwaves by ferrite nanoparticles. Increasing the nano ferrite composition in the micro-nano structure can increase microwave absorption as indicated by an increase in reflection loss values. The optimum composition for microwave absorption in the X

and Ku-band is 5:1%wt with maximum Reflection Loss (RL_m) = -40.27 dB at matching frequency (f_m) = 10.02 GHz and -67.54 dB at f_m = 12.52 GHz. This indicates that the coupling of microwaves by magnetic dipole moments in ferrite and accelerated reflection by silver microparticles is constructive. Microwave absorption is determined by the magnetic dipole moment in ferrite nanoparticles, while silver microparticles as a metallic material have an electric dipole which is conductive and makes it easier to reflect microwaves. The presence of silver microparticles as the core in the micro-nano structure causes the reflection speed to reduce the absorption of microwaves by ferrite nanoparticles. This is also shown in the composition of (7:1/5:1 %wt) $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag micro-nanostructures with a wider absorbed frequency band[12].

Figure 5. Reflection **Loss of** $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag micro-nanostructures

Table 2. Reflection Loss **of** $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag micro-nanostructures in X and **Ku-band**.

Samples	Reflection Loss, RL_m (dB)	Frequency, f_m (GHz)	Bandwith (GHz)
$Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (3:1 %wt)	-22.94	8.91	8.40 – 9.16
	-28.16	17.12	15.52 – 17.23
$Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (5:1 %wt)	-40.27	10.02	8.99 – 10.40
	-67.54	12.52	11.64 – 13.15
$Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (7:1 %wt)	-28.79	11.62	10.60 – 12.14
	-70.67	14.33	12.14 – 14.60
$Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (7:1/3:1 %wt)	-69.12	10.51	9.08 – 11.12
	-25.18	17.82	17.23 – 18.00
$Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag (7:1/5:1 %wt)	-43.68	11.72	10.81 – 12.22
	-46.97	13.32	12.22 – 14.41

IV. CONCLUSSION

To involve magnetic properties of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ ferrite has been successful core-shell micro-nanostructures synthesized with silver micropowder ratio (7:1, 5:1, 3:1 %wt) by dry mixing method. The increasing silver powder content ratio in core-hell nanostructures considerably reduced both the saturated magnetization (M_s) and remanence magnetization (M_r) from 50.67 to 39.73 emu/gr and 12.82 to 10.36 emu/gr respectively, on the other hand it is not significant for coercive force (H_c). These results suggest that silver powder has reduced magnetic properties but the magnetic $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag core-shell nanostructures are attributed the superparamagnetic. $Ni_{0.5}Zn_{0.5}Fe_2O_4$/Ag core-shell micro-nanostructures can absorb microwaves in the X and Ku-band with Reflection Loss < -20 dB (~99% absorbed). The optimum composition for microwave absorption is 5:1 %wt with a value of RL_m = -40.27 dB (f_m = 10.02 GHz) and -67.54 dB (f_m = 12.52 GHz). This shows the effectiveness of the combination of microwave absorption by maganteic dipole moments in ferrite and reflection by silver microparticles so that constructive repetition of microwave absorption occurs.

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