Characterization and Synthesis of CR-Rubber Reinforced by Cadmium-Zinc Ferrite for Microwaves Absorption

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Abstract: The first phase of the process was carried out in two phases, the first process was to prepare material from nano-crystalline structure of ZnFe\textsubscript{2}O\textsubscript{4} and CdFe\textsubscript{2}O\textsubscript{4} depending on formula of Cd\textsubscript{x}Zn\textsubscript{1-x}Fe\textsubscript{2}O\textsubscript{4} combination by ratio (x = 0.9) and mixing the compound of ZnFe\textsubscript{2}O\textsubscript{4} with CdFe\textsubscript{2}O\textsubscript{4} and they were prepared by using co-precipitation method because they give a great homogeneity to the powder particles. The second process used to blend the fractions by ratios (x) to the compound Cd\textsubscript{0.9}Zn\textsubscript{0.1}Fe\textsubscript{2}O\textsubscript{4} with neoprene (Chloroprene Rubber, CR) by adding 3, 6 g. The XRD and Scanning Electron Microscopy (SEM) were investigated the structural properties for prepared samples and to determine length of unit cell, the results showed that ferrites had a cubic spinel structure and when increasing concentration (x) for the compound CdxZn1-xFe2O4 led to an increase the crystalline size and the distance between the crystalline surfaces, the ionic radius of the cadmium is greater than the ionic diameter of the zinc (0.83 Å). FESEM showed the increasing size of nanoparticles with increased concentration of Cadmium Ferrite (CdFe2O4) in relation to compound Cd0.9Zn0.1Fe2O4, the elements of the ferrite structures showed by (EDS) and mixed compound of ferrite with the neoprene (Chloroprene Rubber, CR) according to the weight ratios 3, 6 g. VNA was investigated within the X-band of the microwave 8-12 GHz, the best result was obtained by adding 6 g of neoprene (Chloroprene Rubber, CR) for attenuation microwaves. The real part of the dielectric constant and the imaginary part of the dielectric constant were calculated as a frequency function at the X beam within the microwave region. The real part of amplitude refers to the amount of energy from the external electric field stored in the material, the imaginary part refers to the scattered energy or loss of energy.

Key words: Cadmium-zinc ferrites, microwaves absorption, CR-rubber, co-precipitation, homogeneity, SEM

INTRODUCTION

Electrical industries were mainly based on iron and ferro-alloys to meet their magnetic requirements and the need to operate at high frequencies and standard techniques to reduce eddy currents losses and it was necessary to resort to what is new and special characteristics that leading to discover previously unknown materials (Spaldin, 2010). These materials are the materials of magnetic ceramics such as (ferrite) which has been developing to intervene in the construction of electrical circuits working at low frequencies and high (ferrite) is generally a set of iron oxides which are solid and fragile materials such as ceramics (Kombaiah et al., 2017) With different chemical structures and crystalline structure, all of which are semi-conductive magnetic materials ferrite has a high electrical resistivity of up to 10 Ω and is accompanied by high magnetic permeability and constant isolation ranging from 10-15 as these quantities change by electric and magnetic sfields (Ismail et al., 2007). The increasing need for materials absorbs the energy of electromagnetic waves falling on them there are several applications of electromagnetic pads manufactured from the material of the material (Zhao et al., 2016), for example, avoid unwanted electromagnetic waves and generated internally in the devices and the prevention of electromagnetic noise as well as improve the performance of antennas and the construction of audio rooms and camouflage military places and aircraft and other military applications (Shtarkova and Dishovsky, 2009). The absorbent materials are classified, according to their technical and design specifications to several types, each with its uses and specials including narrow-band pipettes. These materials absorb a certain frequency band from the electromagnetic waves (Mahafza, 2013) and the material
is used to absorb microwave waves within the band 8-12 GHz. After blending the ferrite with the rubber it is the types used neoprene (Chloroprene Rubber, CR) where the characteristics of this kind of rubber (Sindhu et al., 2006; et al., 2006; Mutar, 2010) and the thermal resistance for CR rubber is approximately 121°C. The cold elasticity is approximately -40°C (Brydson, 1978). It contains high polar, paraffin and naphthenic oils but it is partially increased in aromatic solvents and decomposes during connection with engine fuel (Winspear, 1968). It has very low flammability. Winspear (1968) hydrochloric acid can be eliminated at high temperatures and therefore, metal oxides are added and have less insulating properties than non-polar rubber. When blending rubber with ferrites, it is composed of a surfactant that absorbs microwaves and improves the properties of the material (Koledintseva et al., 2006; Mutar, 2010).

**MATERIALS AND METHODS**

**Preparation of CdxZn1-xFe2O4:** The main material in this research is Cadmium-Zinc Ferrite (CdxZn1-xFe2O4) and Chloroprene Rubber (CR) (Neoprene-type GRT). It was prepared by chemical co-precipitation method were it’s a common to the addition of alkaline bases such as sodium hydroxide or ammonia to solutions containing metal ions in the composition of ferrite as the hydroxides of these metals or chlorides, the process was by mixing these materials with special weight ratios and then adding a distilled water to the mixing solution and combine them until we get a solution placed on a temperature 50°C for a period of time we added alkaline bases such as ammonia to the solution until pH will be between 12-8, the second process was burning the substances with temperature between 400-600°C for a period of time 6 h and leave it to cool until it reaches at room temperature and then grind it and burn it for 6 h at a temperature 800-1100°C.

Tests for the resulting ferrite material X-Ray Diffraction XRD, FE SEM and EDS. Thus, we prepared a rubber paste of CR-rubber and mixing method using (Comerio Ercole Busto Avsizo Company) instrument, it contains two rolls with diameter 150 and 300 mm length. The materials were added according to Table 1 the rubber was inserted between the rollers several times with decreasing the distance between the two rollers material was added, respectively with continuous mixing. This process was carried out at laboratory temperature, the rubber paste was then ready to prepare a layer of GRT-ferrite as L = 20 cm, W = 20 cm, h = 2 mm) and the attenuation was measured with frequencies 8-12 Ghz using (Anritsu-S4642A 10 MHz-20 GHz vector-network analyzer.

| Materials | Parts Per Hundred of Rubber (PPHR) | Weight of embryos (100 g) |
|-----------|-----------------------------------|--------------------------|
| Rubber GRT | 100                               | 63.37                    |
| 6PPD      | 1                                 | 0.62                     |
| Stearic acid | 1                                | 0.62                     |
| Zinc oxide | 5                                 | 3.12                     |
| MBTS      | 0.5                               | 0.31                     |
| Aromatic oil | 10                             | 6.37                     |
| HAF326    | 40                                | 25.37                    |
| CdxZn1-xFe2O4 | 3, 6                           | 3.6                      |

**RESULTS AND DISCUSSION**

**Structural and morphological properties XRD:** It was found that the ferrite compounds ZnFe2O4 and CdFe2O4 were tested at X-ray diffraction after initial combustion at a temperature 600°C and the process of final combustion at 900°C, found that the composite of the ferrite ZnFe2O4 is identical to the standard card (00-022-1012 ICDD) and ferrite of CdFe2O4 also identical with the standard card (ICDD 00-022-1063) were showing that the diffraction peaks at the crystalline levels (111, 220, 311, 222, 400, 331, 422, 511, 440, 531, 620, 533, 622, 444) which confirms to be as spinel ferrite based on FCC lattice for all samples. This indicates that the preparation method ensures the incorporation of positive ions in the spinel structure and after mixing the two compounds to form the compound Cd0.0Zn1.0Fe2O4, XRD was tested for each compound as shown in Fig. 1 and 2: we calculate parameters of (dhkl), lattice constant, crystalline volume, stress and the density of dissociation at the highest peak (311), the calculations were found from X-ray diffraction showing that the lattice splicing constant, ferrite compound of Cd0.0Zn1.0Fe2O4 equal to (8.425887647) and the lattice splicing constant (a) ferrite compound of Cd0.0Zn1.0Fe2O4 equal to 8.916407702. After blending the composite compounds and get the composite Cd0.0Zn1.0Fe2O4. It becomes a fixed lattice 8.916407702 to the highest point as shown in Fig. 3 and 4. In addition, the average size of crystal and crystalline surfaces is increased by increasing the concentration of cadmium ferrite Cd1.0Zn0.0Fe2O4 due to the difference in the ionic radius of both cadmium and zinc. The ionic radius of zinc 0.83 A and cadmium 0.99 A and Table 2 shows the calculations and information derived from the X-Ray Diffraction test (XRD).

**SEM and EDS:** FESEM was investigated for the composite ferrite samples CdxZn1-xFe2O4 which can give clear information on particle size and crystalline form of nanostructures. Figure 4 and 5 show the compound structures with ratio X = 0.0 for the compound Cd0.0Zn1.0Fe2O4 and x = 1.0 for CdxZn1-xFe2O4 as we seen it gives a clear picture for prepared samples. The crystal size for cadmium ferrite is between 210-108.8 nm and crystal size
Fig. 1: X-ray diffraction for the compound ZnFe$_2$O$_4$ at 900°C

Fig. 2: X-ray diffraction for the compound CdFe$_2$O$_4$ at 900°C

Table 2: The information extracted from the XRD examination

| X   | Cd$_x$Zn$_{1-x}$Fe$_2$O$_4$ | d = nλ/2sinθ | a = d$^2$sin$^2$θ | D = Kλ/Bcosθ | Σ = Bcosθ/4 | S = 1/D$^2$ (Å$^2$) |
|-----|-----------------------------|---------------|-------------------|--------------|-------------|-------------------|
| 0.0 | Cd$_{0.0}$Zn$_{1.0}$Fe$_2$O$_4$ | 2.540501319   | 8.425887647       | 364.2269352  | 0.000951676 | 7.538E-06        |
| 1.0 | Cd$_{1.0}$Zn$_{0.0}$Fe$_2$O$_4$ | 2.688398716   | 8.916407702       | 453.8272308  | 0.000763784 | 4.85533E-06      |
| 0.9 | Cd$_{0.9}$Zn$_{0.1}$Fe$_2$O$_4$ | 2.688398716   | 8.916407702       | 981.5383080  | 0.000353146 | 1.03797E-06      |
Fig. 3: The effect of the increase of ferrite cadmium for the ferrite compound Cd$_{0.9}$Zn$_{0.1}$Fe$_2$O$_4$.

Fig. 4: The effect of the increase of ferrite cadmium for the ferrite compounds for zinc ferrite between 79.2-47.14 nm the results shows that crystal size of cadmium ferrite is greater than zinc ferrite because the ionic radius of cadmium is greater than the ionic radius of zinc which is 0.83 Å as shown in Fig. 5 and 6 when ratio X = 0.9 for compound Cd$_{0.9}$Zn$_{0.1}$Fe$_2$O$_4$ note that crystal size increase significantly as shown in Fig. 7 and 8 was noted for having both compounds nanoparticles prepared as described as different particles from each other in size clearly. The crystal size increase and ranging between 52.12-463.6 nm.
to rate size of nanoparticles and increases whenever replace the Zinc ions (Zn²⁺) Cadmium ions (Cd²⁺) in the crystal lattice. The results of X-ray tests are consistent with those obtained from the FESEM test. The composition and chemical elements of the ferrite compounds were determined using the X-ray Dispersion Energy (EDS). The first ratio of X = 0.0 of the compound CdₓZn₁₋ₓFe₂O₄ was obtained and the EDS spectra revealed the presence of the following elements only (iron, zinc, carbon and oxygen) as shown in Fig. 9 and 10 also, EDS spectra for prepared samples with ratio X = 0.9 Cd₀.₉Zn₀.₁Fe₂O₄ as shown in Fig. 11. The EDS
spectra revealed that only the following elements (iron, cadmium, carbon, oxygen and zinc) were found as shown in Table 3, since, the atomic ratio of oxygen ranges from 16.4-19.9, iron ranges from 56.5-62.9 and carbon 4.8-5.4 while zinc decreases and cadmium increases by replacing cadmium with zinc depending on chemical method of co-precipitation.

**Reflection coefficient and loss of reflection and attenuation and complex permittivity:** The reflection coefficient for prepared samples as a function of frequency (f) was investigated as shown in Fig. 12 the variation of reflection coefficient for compounds when adding 3 g of ferrite to neoprene (Chloroprene Rubber, CR) to mixed compound Cd$_x$Zn$_{1-x}$Fe$_2$O$_4$ shows a lower reflectivity at value 0.046 at 9.82 Ghz and when adding 6 g of ferrite to (CR) rubber as shown in Fig.12 the mean value of the reflection coefficient 0.019 for x = 0.9 at frequency 8.87-9.233 GHz as we seen the difference of addition 3 and 6 g from ferrite compound to (CR) rubber. We observe a greater reduction in the reflection coefficient by increasing the ferrite to rubber within the

| X    | Cd$_x$Zn$_{1-x}$Fe$_2$O$_4$ | O   | σ     | Fe   | σ     | Zn   | σ     | Cd   | σ     | C    | σ     | Total |
|------|---------------------------|-----|-------|------|-------|------|-------|------|-------|------|-------|-------|
| 0.0  | Cd$_{0.0}$Zn$_{1.0}$Fe$_2$O$_4$ | 16.4| 0.1   | 62.9 | 0.3   | 15.8 | 0.2   | -    | -     | -    | 4.8   | 0.2   | 100   |
| 1.0  | Cd$_{1.0}$Zn$_{0.0}$Fe$_2$O$_4$ | 17.1| 0.1   | 74.6 | 0.2   | -    | -     | 4.6  | 0.1   | 2.6  | 0.2   | 100   |
| 0.9  | Cd$_{0.9}$Zn$_{0.1}$Fe$_2$O$_4$ | 19.9| 0.2   | 56.5 | 0.3   | 17.9 | 0.2   | 0.4  | 0.1   | 5.4  | 0.2   | 100   |
Fig. 12: Reflection coefficient

Fig. 13: Loss of reflection

Fig. 14: Attenuation for 3, 6 g
frequency 8.871-9.307 GHz which achieved the lowest value of the reflection coefficient. The lower reflection coefficient met a greater absorption value of the material and loss of reflection was investigated as shown in Fig. 13 where the lower value of reflection coefficient also met higher absorbent value for materials, the greatest value for loss of reflectivity at the ratio $x = 0.9$ is -36.480 dB at the frequency 8.620 GHz. When adding 6 g of ferrite to CR rubber at $x = 0.9$ will be a largest value of loss of reflectivity 27.550 dB at 8.620 GHz as in Fig. 13. This change in the reflection loss coefficient is due to microwave absorption. It was observed that the highest attenuation value for all bands at 18.8-40.8 Ghz is 19.7-19.3 dB. Figure 14 indicates attenuation values by adding 6 g of ferrite to (CR) rubber, note that the highest attenuation value for all bands within the range
Fig. 17: The imaginary dielectric constant

8.38-8.87 GHz is 20.7 -19.4 dB. The composite permittivity of the prepared samples was calculated as a function of the frequency for adding 3 g of the ferrite to (CR) rubber. The permittivity value at $x = 0.9$ is $12.02$ at 8.075 GHz and for adding 6 g of the ferrite to (CR) rubber the permittivity is $11.23$ at 8.075 GHz as in Fig. 15. The actual part of the dielectric constant and the imaginary part of the dielectric constant as a function of the frequency at the X band within the microwave region indicate the real part of the amplitude to the amount of energy from the external electric field stored in the material where dielectric constant at 3 g was $8.53 \times 10^{-11}$ and at 6 g was $8.02 \times 10^{-11}$, we notice an increase in value of the dielectric. The real part which refers to the amount of energy stored as shown in Fig. 16. The imaginary part refers to the scattered energy or loss of energy which is a measure of the loss of material for the true external electric field. The value of the imaginary insulation at the addition of 3 g of the ferrite to the (CR) rubber with the highest value at $6.47 \times 10^{-11} - 2.38 \times 10^{-11}$ and when adding 6 g of ferrite the material became $6.001 \times 10^{-11} - 2.33 \times 10^{-11}$ which refers to the loss of energy as in Fig. 17.

CONCLUSION

The process of preparation of cadmium and zinc ferrite is a very important process, found the composition of compound $\text{Cd}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ when adding $x = 0.9$ of cadmium ferrite to zinc will increase the crystal size of compound $\text{Cd}_0\text{Zn}_0\text{Fe}_2\text{O}_4$ and when the compound $\text{Cd}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ mixed with (CR) rubber by adding 3 and 6 g from ferrite we noticed the reflection coefficient was decreases as the amount of ferrite increases, so, we get a substance with high absorption of microwaves and with high attenuation. The higher the amount of ferrite to the (CR) rubber increases the absorption of the material. The addition of the ferrite to the rubber improves the properties of the material and the ability to absorb the microwave waves.

REFERENCES

Brydson, J.A., 1978. Rubber Chemistry. Elsevier Applied Science Publishers, London, ISBN:9780853347798, Pages: 462.
Ismail, H., S.T. Sam, M.A.F. Noor and A.A. Bakar, 2007. Properties of ferrite-filled natural rubber composites. Polym. Plast. Technol. Eng., 46: 641-650.
Koledintseva, M., P.C. Ravva, J. Drewniak, A.A. Kitaitsev and A.A. Shinko, 2006. Engineering of ferrite-graphite composite media for microwave shields. Proceedings of the 2006 IEEE International Symposium on Electromagnetic Compatibility (EMC 2006), August 14-18, 2006, IEEE, Portland, USA., pp: 6-10.
Kombaiah, K., J.J. Vijaya, L.J. Kennedy, M. Bououdina and K. Kaviyarasu et al., 2017. Effect of Cd$^+$ concentration on ZnFe$_2$O$_4$ nanoparticles on the structural, optical and magnetic properties. Opt., 135: 190-199.
Mahafza, B.R., 2013. Radar Systems Analysis and Design Using MATLAB. 3rd Edn., CRC Press Publishing Company, Boca Raton, Florida, USA., ISBN-13:9781439884959, Pages: 751.
Mutar, M.A., 2010. A study in vulcanization of neoprene rubber (WRT) by polymethylol resin (RESOL). J. Al Nahrain Univ. Sci., 13: 1-6.
Shtarkova, R. and N. Dishovsky, 2009. Elastomer-based microwave absorbing materials. J. Elastomers Plast., 41: 163-174.
Sindhu, S., M.R. Anantharaman, B.P. Thampi, K.A. Malini and P. Kurian, 2002. Evaluation of a.c. conductivity of rubber ferrite composites from dielectric measurements. Bull. Mater. Sci., 25: 599-607.
Spaldin, N.A., 2010. Magnetic Materials: Fundamentals and Applications. 2nd Edn., Cambridge University Press, Cambridge, UK., ISBN-13:978-0521886697, Pages: 290.
Winspear, G.G., 1968. The Vanderbilt Rubber Handbook. RT Vanderbilt Co. Inc., USA.
Zhao, C., W. Huang, X. Liu, S.W. Or and C. Cui, 2016. Microwave absorbing properties of NiFe2O4 nanosheets synthesized via a simple surfactant-assisted solution route. Mater. Res., 19: 1149-1154.