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Research Article

Synthesis and Charactrization of Ni-Zn Ferrite Based Nanoparticles by Sol-Gel Technique

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A b s t r a c t

A series of Ni-Zn ferrite based naoparticles ($Ni_{0.76-x}Zn_{0.04+x}Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$) in which x is varied from 0.01 to 0.04, were prepared by sol-gel technique. Characterization of the samples was Performed by X-Ray Diffraction (XRD), Raman Spectroscopy, and Field Emission Scanning Electron Microscope (FESEM). The XRD results show the major peak (311) of the spinel cubic structure for Ni-Zn ferrites. FESEM results show that single phase Ni-Zn ferrite nanoparticles with average diameters 17-45 nm can be obtained by sol gel technique. Initial permeability and Q factor for all samples were measured and the magnetic measurements of the nano ferrites show that it can be used as a magnetic feeder for the transmitter.

1. Introduction

Ferrites represent an important category of materials, which are largely used, due to their numerous practical applications, as for example magnetic devices in electronic, optical and microwave installations [1-2].Substituted Ni-Zn ferrites have wide applications in radio frequency (RF) electronic devices due to the high initial permeability in combination with giant resistivity [3-5]. Most of the physical properties of these materials, especially the magnetic ones (coercivity, saturation magnetization, losses) dramatically change with the decrease of the particle size, especially at nano-size range [3-4]. Thus, special interests have been paid to the synthesis that leads to formation of nanoferrites. In this context, unconventional synthesis methods [5-8] have advantages over the conventional methods [9-10]. One of the most popular unconventional synthesis methods that recently have been extensively used is the sol-gel method [11-13]. This method was successfully was used in obtaining nanomaterials, especially magnetic nanoparticles that lead to formation of ferrite particles having sizes between 10 to 100 nm [1- 4].

Some new techniques have been tried to reduce the sintering temperature of Ni-Zn based ferrites: such as addition of CuO and some sintering aids [1-8]. Moreover, the reduction of the particle size of the raw materials was also favorable for decreasing sintering temperature [14].

In this paper Ni-Zn ferrite based nanoparticles were prepared using a sintering temperature of 5000C. Due to the CuO addition also another decrease of the sintering temperature was obtained which is due to low melting point of CuO compared to other oxides. So a decrease in sintering temperature from 1200 °C (Conventional method) to 500 °C was obtained.

2. Materials and Method

2.1. Materials and mix design

The samples were synthesized using Ni, Mg, Co ,Cu ,Zn ,Ti and Fe nitrates and citric acid as precursors, by dissolving them in distilled water while stirring at room temperature. The solutions were evaporated by heating at 75oC with continuous stirring until a viscous gel formed. After the gel formed, the powders were dried at 110 ◦C in an oven for 24 hours and dried powders were crushed by using mortar for 1 hour. Then, the samples were annealed at 500 ◦C with holding time of 4 hours [8].

2.2. Experimental test

PVA (Polyvinyl alcohol) was added into them for their granulation and pressed into toroidal shapes. The green bodies were sintered at 700 °C in air for 5 h [7]. The resultant toroidal cores had 10 mm outer diameter, 5mm inner diameter and 2 mm thickness. Initial permeability and Q factor were carried out after each toroid was wound with 25 turns of 0.3mm diameter insulating copper wire. The wire ends were scraped by using a sand paper. The sample was then connected to a Hewlett Packard 4284A Precision LCR meter. A series of inductance, L_S, and Q factor values were recorded from the lowest frequency to resonance frequencies. The initial permeability values were calculated by introducing Ls to the equation below [14]:

$$\mu i = \frac{2\pi L_s}{N^2 \mu_0 t In(\frac{D_0}{D_1})}$$

where L_S is the parallel inductance, N is the number of turns, μ0 is the permeability of free space (4π x10^{-7} m/A), t is the thickness, D_0 is the outer diameter, and D_i is the inner diameter of the toroid, respectively. The magnetic measurements of the component ferrites demonstrated that it can be used as a magnetic feeder for the transmitter [15].

The phase identification of the calcined powders was performed by X-ray diffraction(XRD) on a Philips PW-1730 X-ray diffractometer using Cu Kα radiation (λ=1.54 A^0).The average crystallite sizes of the synthesized powders were determined by using the XRD patterns, via the well-known Scherrer equation [16]. The morphology of the powders was investigated by Field Emission Scanning Electron Microscope (FESEM).

3. Results and discussion

3.1 X-Ray Diffraction (XRD)

Figure 1 shows X-ray diffraction pattern of the samples prepared by sol-gel method. The X-ray diffraction patterns show well developed diffraction lines assigned to pure spinel phase, with all major peaks matching with the standard pattern of Ni-Zn ferrite, JCPDS 8-0234. The average crystallite sizes of the ferrites calculated from the broadening of the (311) X-ray diffraction peak by the Scherrer equation [16] were about 15-20 nm.

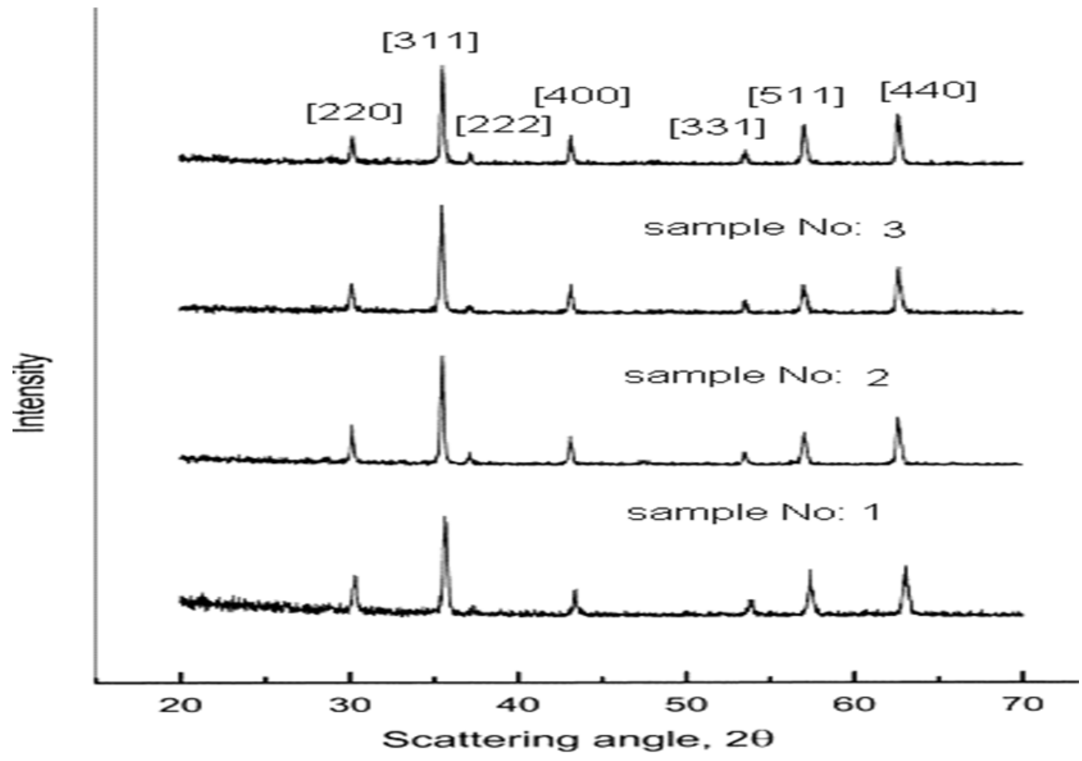

Fig.1. X -ray diffraction Pattern of $Ni_{0.76-x}$ $Zn_{0.04+x}$ $Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$ after annealing at 500ºC for 4 hours which shows the major peaks of all samples is in [311].

3.2 Raman Spectra

The purpose of Raman Spectroscopy is to see the vibrations of the atoms in $Ni_{0.76-x}Zn_{0.04+x}$ $Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$ samples after annealing at 500◦C for 4 hours. Figure 2 shows the major peaks of five samples is in range 486 to 698.392 cm−1. These ranges for major Peaks of Raman shift were confirmed as Raman shift for Ni-Zn ferrite [3].

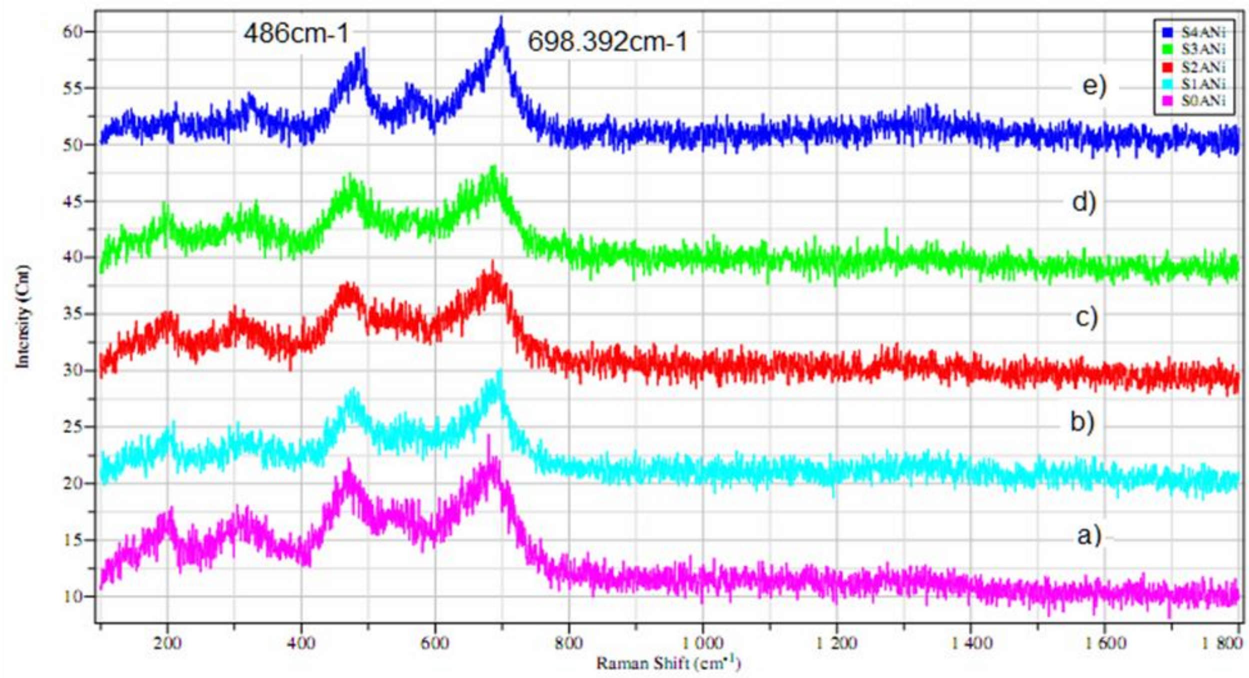

Fig.2. Raman pattern of $Ni_{0.76-x}Zn_{0.04+x}Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$ after annealing at 500°C for 4 hours which shows the major peaks of five samples is in range 486 to 698.392 cm^{-1}.

3.3 FESEM results

(FESEM) is used to identify the morphology, dimension for grain size of the bulk and nano-sized particles [11].Figure 3a-3d (for samples prepared by sol gel methods) show nearly uniform spherical ferrite particles having a distribution size of 17-45nm. The ED pattern (inset) reflects that these nanoparticles are well crystallized, the *d* values calculated from the ring diameters correspond well to the spinel structure and are consistent with the XRD pattern. The EDX spectrum revealed that the darker particles contain Zn, Ti, Mg, Cu, Ni and Fe.

3.4 Initial permeability and Q factor

It is obvious that the increase of frequency has led to an increased initial permeability and Q factor up to resonance frequency [7 and 12]. From figure 4, it was observed that a fall of initial permeability with increasing Zn content. A probable explanation is that it was highly difficult to disperse the Zn powder particles which tend to agglomerate, during the mixing stage of the raw materials [17].

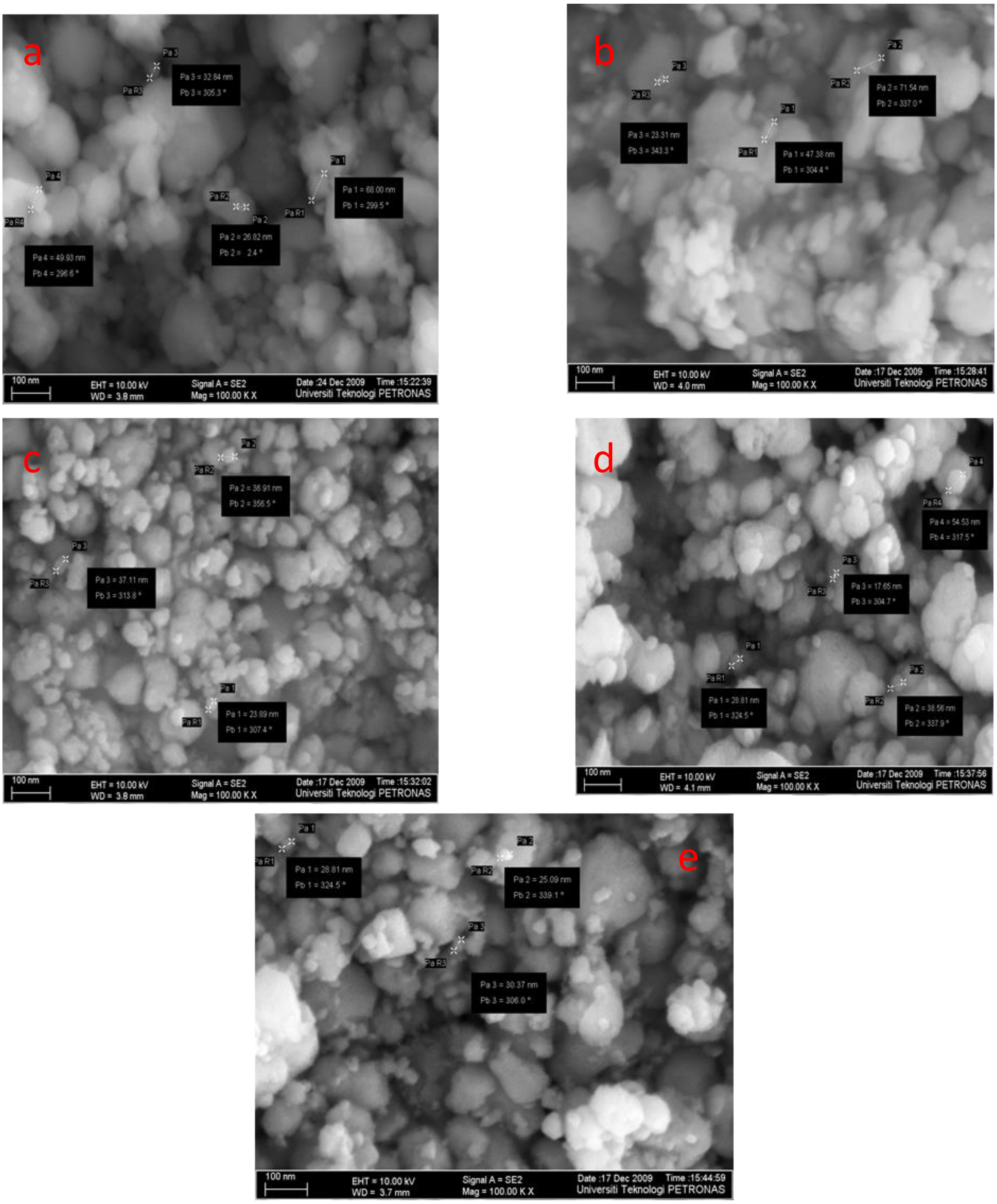

Fig. 3. Show nearly uniform spherical ferrite particles having a distribution size of 17-45 nm:

a) $Ni_{0.75}Zn_{0.05}Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$ (sample No.1)

b) $Ni_{0.74}Zn_{0.06}Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$ (sample No.2)

c) $Ni_{0.73}Zn_{0.07}Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$, (sample No.3)

d) $Ni_{0.72}Zn_{0.08}Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$ (sample No.4)

e) $Ni_{0.71}Zn_{0.09}Ti_{0.0025}Co_{0.1}Cu_{0.075}Mg_{0.04}Fe_{1.96}O_{3.96}$ (sample No.5)

Table 1. EDX result for five samples prepared by sol –gel method

Element	Weight % Sample No: 1	Atomic % Sample No: 1	Weight % Sample No: 2	Atomic % Sample No: 2	Weight % Sample No: 3	Atomic % Sample No: 3	Weight % Sample No: 4	Atomic % Sample No: 4	Weight % Sample No: 5	Atomic % Sample No: 5
Zn K	2.22	7.81	1.29	55.59	5	12.25	4.76	11.50	5.30	23.83
O K	32.06	55.20	26.23	0.83	32.91	56.94	30.20	54.79	21.95	36.66
Mg K	0.44	0.88	0.59	30.65	0.83	0.59	1.53	1.13	2.43	1.42
Fe K	43.59	26.47	50.48	11.02	41.88	0.73	44.09	22.91	52.36	30.83
Ni K	20.09	8.98	19.8	1.00	16.78	20.76	16.73	8.47	16.53	11.02
Cu L	1.40	0.43	2.00	0.67	1.87	7.91	2.06	0.94	2.15	1.11
Ti K	0.2	0.23	0.33	0.24	0.74	0.82	0.94	0.42	1.16	0.53
Totals	100.00		100.00		100.00		100.00		100.00	

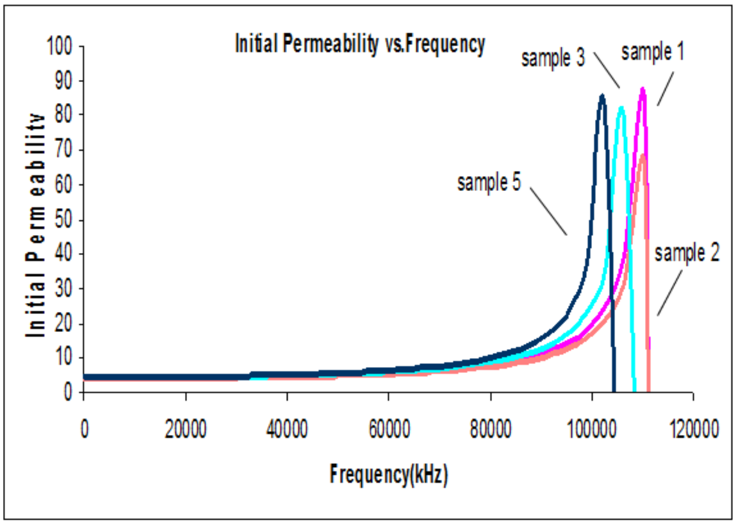

Fig.4. Initial Permeability of the toroids prepared by Ni-Zn ferrite based nanoparticles.

Fig.5. LCR meter and the toroids prepared by Ni-Zn ferrite based nanoparticles.

There is a marked shift (to the left) of resonance frequency due to the increment of Zn. Thus nano-regions of badly non-stoichiometric compositions would be created, give rise, for instance to Fe^{+2} ions which could contribute to eddy current through $Fe^{+2} \rightarrow Fe^{+3} + e^{-}$ reaction [17]. Thus by adding more Zn, the lower the resonance frequency and thus the lower initial permeability and Q factor are obtained (Figure 6).

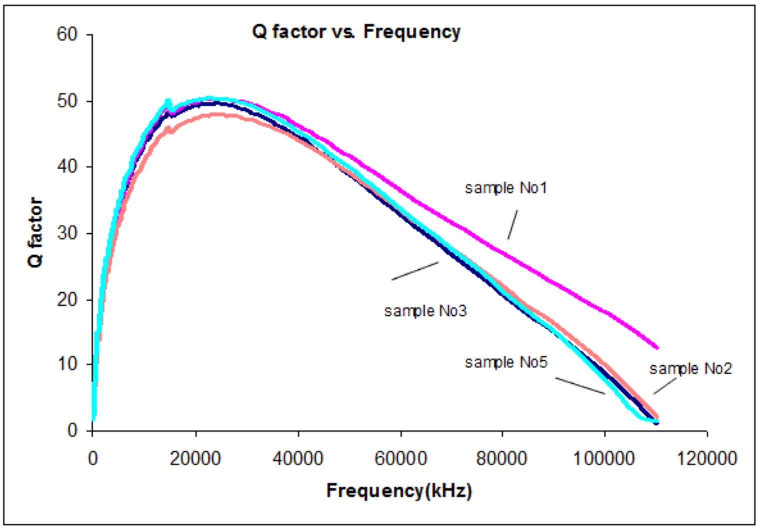

Fig.6. Q factor of the samples prepared by sol- gel method

Initial permeability and Q factor of the samples were measured (Figure 4 and Figure 6) and the sample No 1 has the most resonance frequency and initial permeability and we can used it as a magnetic feeder for the transmitter (Figure 7).

Figure 7 shows comparison of aluminum transmitter with and without ferrites. We can see magnetic field for aluminum transmitter with sample No:1 is 41% higher than the magnetic field obtained without ferrite.

4. Conclusions

Ni-Zn ferrite based nanoparticles ($Ni_{0.76-x}$ $Mg_{0.04+x}$ $Ti_{0.0025}$ $Co_{0.1}$ $Cu_{0.075}$ $Zn_{0.04}Fe_{1.96}O_{3.96}$) prepared by sol-gel method and single phase for this compound obtained at 500^{0}C. Nanocrystalline size for samples prepared by this method is about 15-20 nm. Initial permeability and Q factor measured and investigated. Magnetic field of aluminum transmitter with sample No:1 is 41% higher than aluminum transmitter without ferrite.

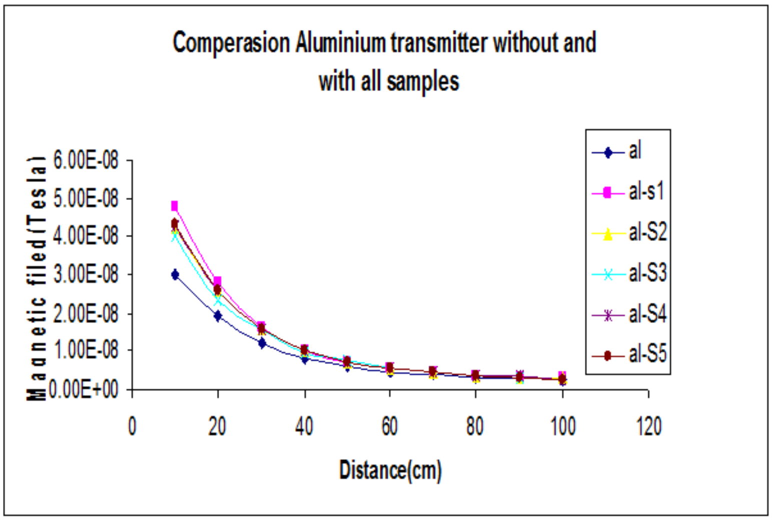

Fig.7. Comparison of aluminum transmitter with and without ferrite.

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