



# Plus Laser deposition to preparation Co-Ninanoparticles ferrite and hyperthermia application

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## Abstract

Nickel-doped cobalt nanoparticles (NPs) were with the chemical formula  $\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$  (was,  $x = 0.0, 0.2, \text{ and } 0.4$ ). Manufactured at low temperature ( $70\text{ }^\circ\text{C}$ ) using plus laser deposition technology. Sodium hydroxide is used as a chelating agent with a mixture of nickel nitrate and ferric nitrate solutions in a ratio (3: 1) to balance the oxidizing agent ratio. The obtained ferrite NPs were calcined at temperatures ( $300\text{ }^\circ\text{C}$ ) for 3 h in Air to remove water content and unwanted impurities and get a better single-phase spinel structure. Then the obtained powder is compressed into a disc shape with a diameter of (1 cm) and then we use laser deposition technology to get a ferrite membrane where the structural properties of the calcined ferrite sheets are confirmed by analyzing X ray Diffraction (XRD) technique. XRD analysis shows the structure of a single-phase nanoscale spinel. The crystal size calculated from the FWHM of the strongest peak (311) lies in the range (9.04-15 nm) for  $\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$  Ferrite NPs. The images of the spherical-shaped and monolithic FE-SEM NPs show the Particle size morphology in the range (12-15 nm), reflecting the highly crystalline nature of these nanoparticles. Particle size and grain size of  $\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$  ferrite NPs calculated using XRD and SEM technologies are compatible with each other in the same range

**Keywords:** Co-Ni- $\text{Fe}_2\text{O}_4$  ferrites; magnetic nanocomposite; plus laser deposition technology; antibacterial activity.

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magnetic materials. The combination of magnetic and electrical properties makes ferrite useful in many technological applications. The spinel ferrite is having the chemical formula  $\text{MFe}_2\text{O}_4$  (where M- is a divalent metal ions such as Co, Ni, Mn etc). and possess two sub-lattice namely tetrahedral A and octahedral B sites [1,2]. As an important member in the family of spinel ferrites cobalt ferrite ( $\text{CoFe}_2\text{O}_4$ ) with inverse spinel structure are promising magnetic materials because of

## 1. Introduction

Ferrites are ferrimagnetic oxides consisting of ferric oxide and metal oxides. On the basis of crystal structure ferrites are grouped into three classes namely hexagonal ferrite, garnet and spinel ferrite. The magnetic properties arise from interactions between metallic ions occupying particular positions relative to the oxygen ions in the crystal structure of the oxide. Ferrites with spinel structure represent the important class of



energy and with repetition rates of 1 to 100 Hz. The target is usually scanned by the laser beam while being rotated to ensure uniform ablation from the target surface. [4] Pulsed laser deposition is a physical vapor deposition process, which is performed in a vacuum system. The deposition process is very complex, which includes the features of molecular beam epitaxy and sputter deposition. Pulsed laser with high energy is focused onto the target material to be deposited on the substrate. Each laser pulse only vaporizes or ablates a small amount of the target material, and transfers each ablation into a plasma plume. The ablated target material is ejected from the target surface in a highly forward-directed plume toward the substrate and then deposited on the surface

of the substrate. [5] their moderate saturation magnetization, high electrical properties, high magneto-crystalline anisotropy, good mechanical properties and chemical stability [2]. Ferrites or Ferrimagnetics are oxides, dark grey or black in appearance, possess the properties of ceramic materials, such as being hard, brittle and chemically inert, can be changing the electromagnetic properties of ferrites by controlling the manner of preparation and type of materials used, amount of material added and sintering temperature [3]. In this paper, (Co-Ni) Ferrite was prepared by laser deposition (pld) method. Pulsed laser deposition (PLD) technique has been investigated since late 1980s. This technique has been applied successfully to prepare chemically and structurally complex materials. PLD uses a high-power laser which operates at high

e of the substrate. [5]

## 2. Experimental work

### 2.1 Materials and Methods

Table No. (2.1.1) shows the raw materials and their density.

**Table (3-1-1) Chemicals Used**

Compounds	Chemical formula	Mol. Mass (g.mol <sup>-1</sup> )
Iron nitrate	Fe (NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O	403.8
Cobalt nitrate	Co (NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O	291.031
nickel nitrate	Ni(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O	290.79
Sodium hydroxide	NaOH	39.9971

the samples and allowing them to cool slowly naturally to check their dielectric properties.

### 2.3 Pulsed Laser deposition (PLD) Method

Figure 1 shows the experimental setup for laser deposition of solid metal target by pulsed laser deposition. The pulsed laser deposition experiment was carried out inside a vacuum chamber generally in (10-3 mbar) vacuum conditions. The focused Nd:YAG laser beam at 600 mJ with a frequency second radiation at 1064nm (pulse width 9 ns) repetition frequency (3

### 2.2 calcifications and granule formation

The ferrite powder obtained by CCD is placed in a ceramic vessel and then roasted at 300°C for 3 hours to remove reaction residues such as water or carbon dioxide particles from combustion, resulting in the necessary ferrite powder. Then 0.5 g of burnt samples and samples with concentration (x = 0.0, 0.2, and 0.4) round granules 1 cm in diameter and 3 mm thick. This is achieved by pressing 500 tons for five minutes with a hydraulic press using the dry pressing method, then sintering at 300 °C for 3 hours to thicken



the laser was set to (10 cm), and between the target and the substrate was (1.5cm).

Hz), for 650 laser pulses incident on the target surface makes an angle of 45° with it. The distance between the target and

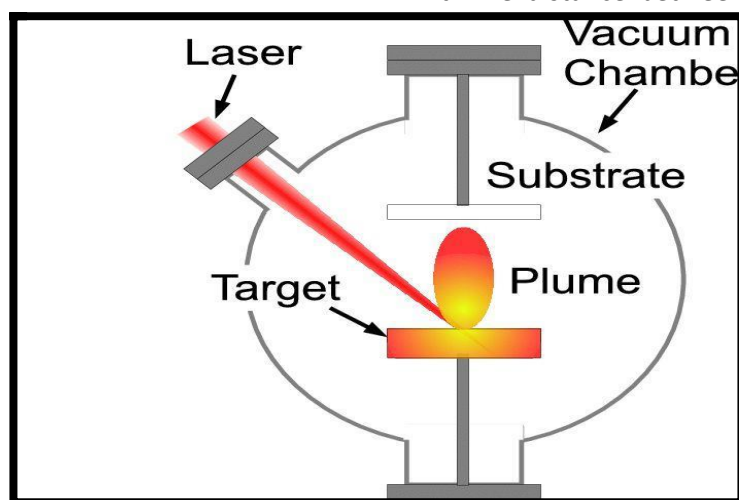


Figure 1: experimental setup for laser deposition

## 2.4 Antibacterial

cell wall, the shape of the cell membrane, and the ability of the outermost membrane to inhibit the creation of reactive oxygen species (ROS) caused by the metal ions are all taken into consideration. Cell membrane damage, the generation of reactive oxygen species (ROS), DNA damage caused by aerobic respiration, and cell division are all examples of NPs' antimicrobial activity [6-7]

Because of their ability to penetrate cells' walls, Co-Ni-Fe<sub>2</sub>O<sub>4</sub> NCs may cause cells to leak and exocytose, as well as inhibit the activity of essential enzymes. Using the following equation [8, 9]

## 3. Result and Discussion

### 3.1 XRD Analyses

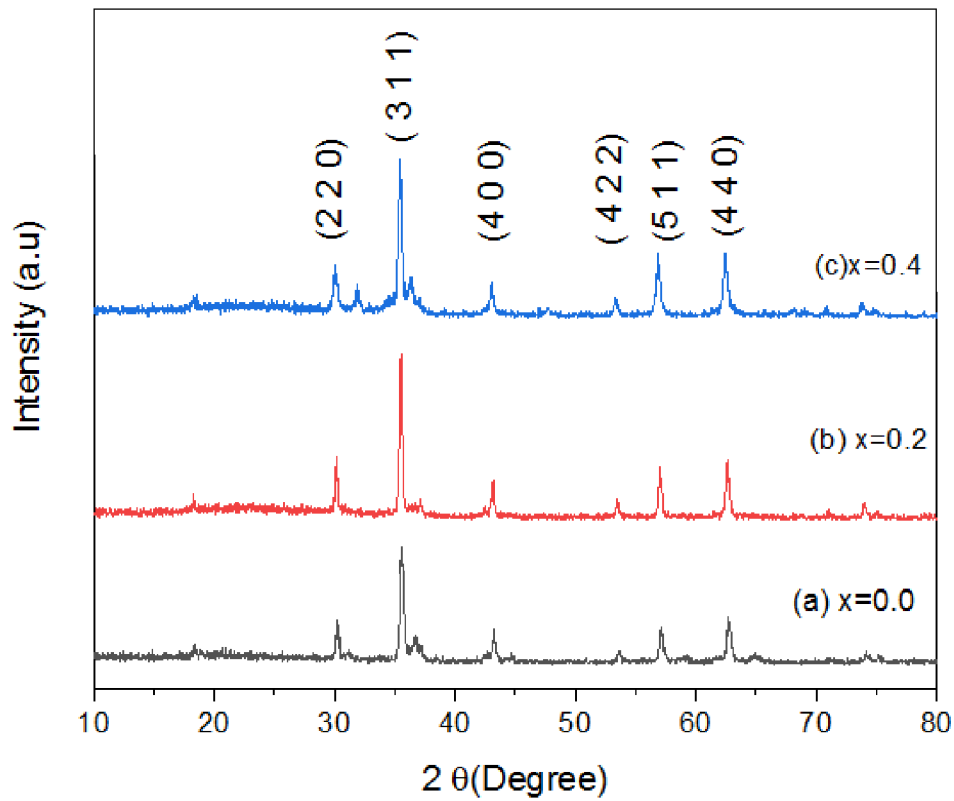
The XRD parameters of the Co<sub>1-x</sub>Ni<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> ferrite nanoparticles calcined at (300°C). Figure (1) show that the crystallite size (D) of the (Co- Ni) ferrites are decreases in the range of (9.04-15.42nm) . Are due to the different ionic radii, Ni<sup>2+</sup> (tetrahedral 0.58; octahedral 0.69 Å) and Co<sup>2+</sup> (tetrahedral 0.58; octahedral 0.74Å) [10].

## activity of Co-Ni-Fe<sub>2</sub>O<sub>4</sub> NCs ferrite nanocomposites

To determine whether or not Co-Ni-Fe<sub>2</sub>O<sub>4</sub> ferrite nanocomposites had the ability to create a zone of inhibition in bacteria in a dose-dependent manner, bacterial cultures were established. In order to demonstrate bactericidal action, the Co-Ni-Fe<sub>2</sub>O<sub>4</sub> NCs ferrite nanocomposites must exhibit a wide zone of inhibition for gram-negative bacteria (*E. coli*) and a smaller zone of inhibition for gram-positive bacteria (*S. aureus*). Gram-negative bacteria have a distinct cell wall structure that distinguishes them from their Gram-positive counterparts. Bacterial cells from the Gram-positive group are covered with a thick layer of peptidoglycan that contains lipopolysaccharide as well as phospholipids and proteins [6-7]

This test allows for the clear observation of the bactericidal action of the Co-Ni-Fe<sub>2</sub>O<sub>4</sub> NCs that have been generated in the laboratory. In order to have a bactericidal effect, the thickness of the





**Figure 2 XRD Patterns of synthesized  $\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$  Ferrite**

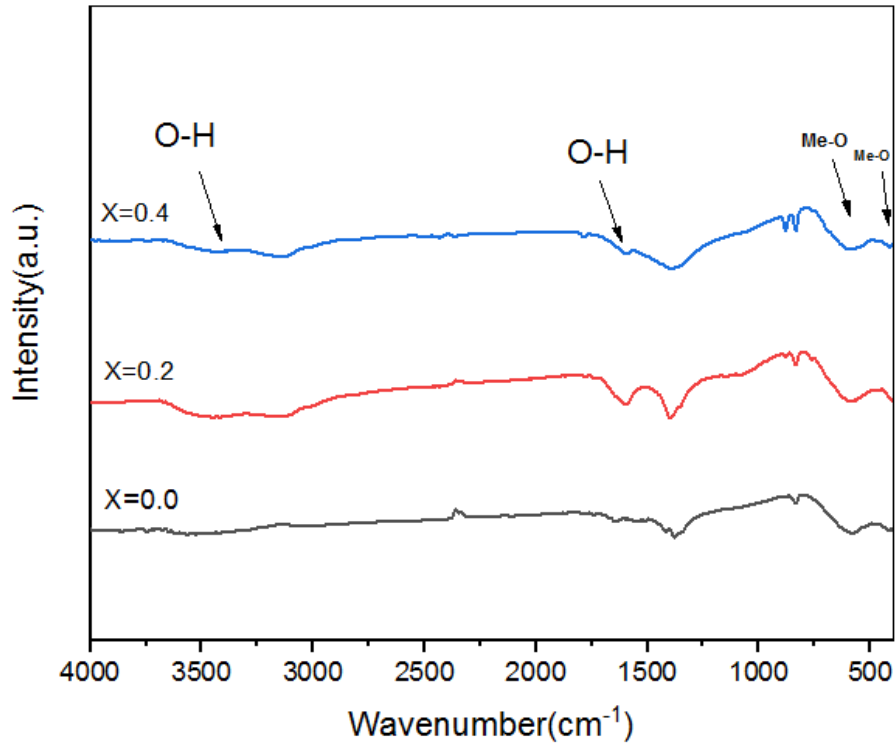
Table 2 : XRD parameters of synthesized  $\text{Ni}_x\text{Co}_{1-x}\text{Fe}_2\text{O}_4$

Material	Ratio	$2\theta$ (deg) Practical	$2\theta$ (deg) Standard	FWHM (deg)	Crystalline size (nm)	D (nm) FE-SEMI	(hkl)
$\text{CoFe}_2\text{O}_4$	0	35.58	35.42	0.485	15.42	15.470	(113)
$\text{Ni}_{0.2}\text{Co}_{0.8}\text{Fe}_2\text{O}_4$	0.2	35.51	35.42	0.784	10.07	12.729	(113)
$\text{Ni}_{0.4}\text{Co}_{0.6}\text{Fe}_2\text{O}_4$	0.4	35.70	35.42	0.873	9.04	12.223	(113)

### 3.2 FTIR Spectroscopy Analysis

Infrared spectrum for spinel ferrites involve two main absorption bands arises due to stretching vibration of both tetrahedral (A-O) and octahedral (BO) sites. Furthermore, it was observed the presence of OH-groups in the spectrum which has been revealed by IR spectrum (bands at 3440 and 1630)[11]. The IR spectrum measurements of samples are given in figure (2). It showed two distinctive absorption bands lie in the region  $\sim 564$  and  $\sim 374 \text{ cm}^{-1}$ . The wave number is attributed to stretching vibration of (A-O) site and  $\nu_2$  is due to stretching vibration of (B-O) site.





Figur 3:FTIR Spectroscopy Analysis of  $Co_{1-x}Ni_xFe_2O_4$

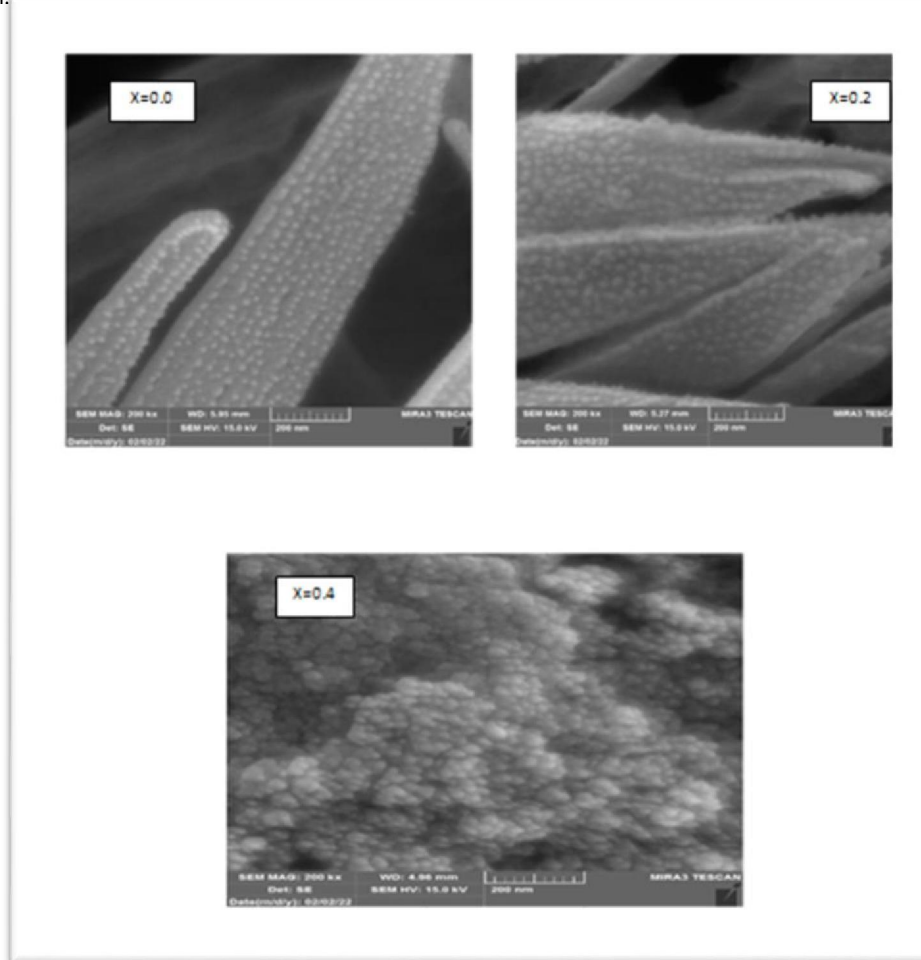
Table 2: IR absorption bands of the samples

X	$\nu_{Tcm}^{-1}$	$\nu_{Bcm}^{-1}$
0.0	574.22	364.81
0.2	567.55	364.48
0.4	566.22	363.48

### 3.3 FE-SEM Image Analysis

Investigation of nanostructure is vital to comprehend the superparamagnetic properties of polycrystalline ferrites. The particle size and surface morphology of  $Co_{1-x}Ni_xFe_2O_4$  had been investigated by a set of FE-SEM images as shown in figure (4). All the samples show a spherical shape with narrow size distribution.





FE-SEM of  $\text{Co}_{1-x}\text{Ni}_x\text{Fe}_2\text{O}_4$  Figure 4:

when  $x=0.2$  but fast decreases with Ni concentration at ( $x=0.4$ ).

The magnetic order in the cubic spinels is due to the super-exchange interaction mechanism occurring between the metal ions in the tetrahedral A-sites and octahedral B-site sublattices. Substitutional ions ( $\text{Ni}^{2+}$ ) with zero magnetic moment cause the variation in the magnetic properties of the spinel ferrites. The results obtained are in well accordance with the earlier reported values by [12].

### 3.4 Magnetic Properties

The magnetic properties of the prepared samples are analyzed using a magnetometer (VSM) at room temperature applied field  $\pm 15$  KOe. Figure. (5) shows the M-H curves of the prepared samples by normal Co-precipitation method. Two prepared samples of Co-Ni ferrite ( $x = 0.0, 0.2$ ) at room temperature exhibit ferrimagnetic coupling but one of them it superparamagnetic  $x = (0.4)$ . saturation magnetization and coercivity increasing



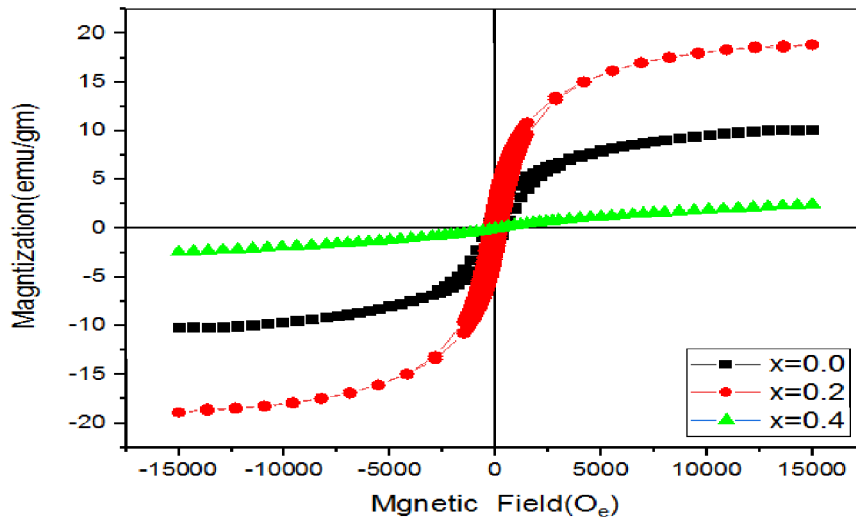


Figure (5): Magnetic hysteresis loops of  $Co_{1-x}Ni_xFe_2O_4$  ( $x=0.0, 0.2, 0.4$ )

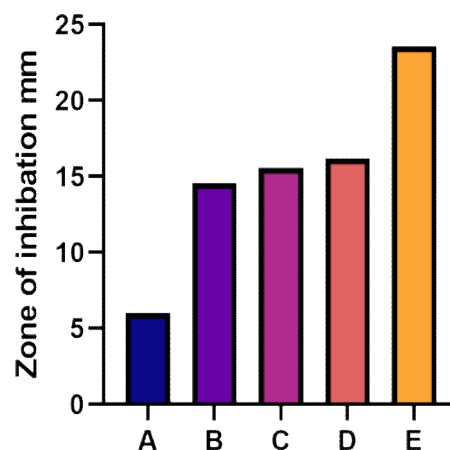
Table(3):The values of saturation magnetization, remanence, coercivity

x	$M_s$	$M_r$	$H_c$
0.0	10.19	1.92	0.012
0.2	18.90	3.23	0.005
0.4	2.49	0.002	0.009

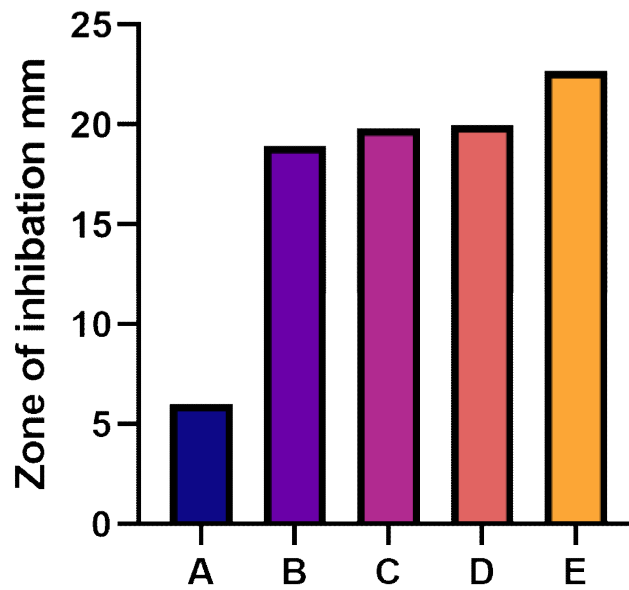
### 3.5 Biology effect of $Co_{1-x}Ni_xFe_2O_4$ ferrite in bacteria :

#### Inhibition zone of E.coli bacteria

In figures (6)-(8) showed inhabitation zone of bacteria E.coli was the inhabitation zone about (15,15.5,16,16.5,16.5,16.9) for Co-Ni ferrite samples Effect of Co-Ni ferrite in E.coli bacteria (Ni=0),it can explain alternating magnetic field that applied in E.coli bacteria lead to rise in temperatures between 40°C and 45°C are generally being referred to as hyperthermia. temperatures up to 42oC can render kill cells more susceptible to the effect of irradiation and cause a certain degree of apoptosis, whereas temperatures >45°C attributed with research. In figures (9) showed inhabitation zone of bacteria E.coli.

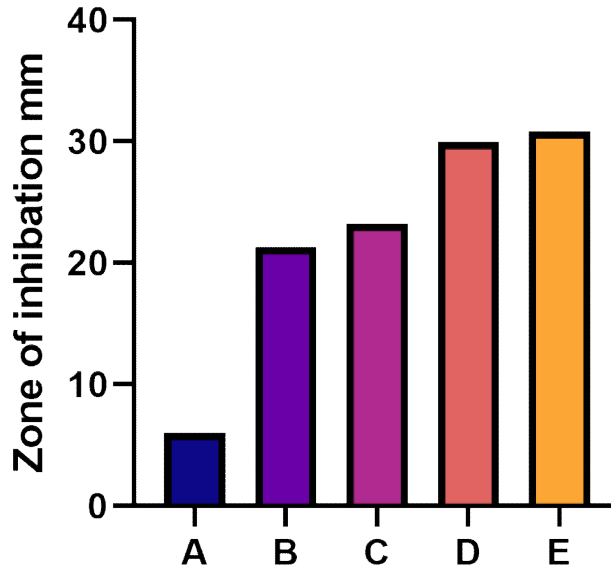


**Figure (6) Inhibition zone of E.coli bacteria via Co-Niferrite enhancement (sample 1)**



**Figure (7) Inhibition zone of E.coli bacteria via Co-Niferrite enhancement (sample 2)**

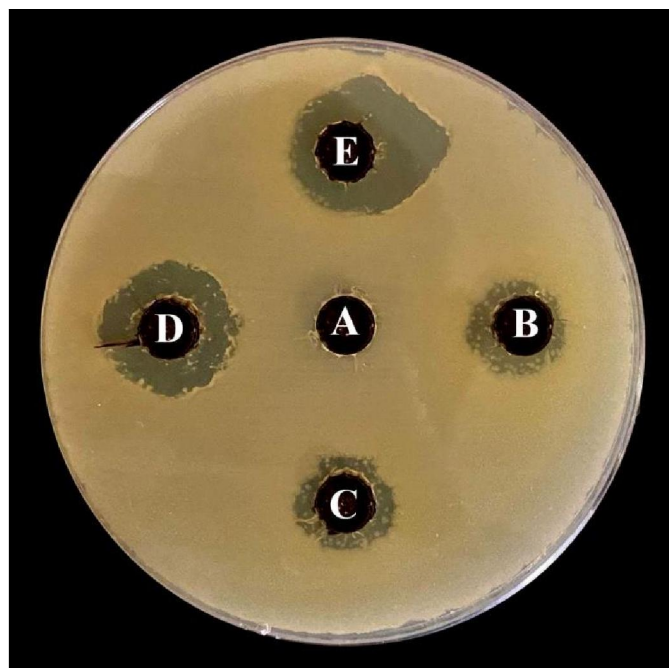
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**Figure (8) Inhibition zone of E.coli bacteria via Co-Niferrite enhancement (sample 3)**







**Figure (9): Inhibition zone of E.coli bacteria via Co-Ni ferrite**

Table (4): Inhibition zone results of Co-Ni-Fe<sub>2</sub>O<sub>4</sub> ferrite by pulsed laser deposition (PLD) techniques, against one type bacteria at 30 (mg/mL) concentrations.

SAMPLES	A	B	C	D	E
1	6	14.573	15.549	16.19	23.581
2	6	18.936	19.824	19.995	22.704
3	6	21.323	23.223	29.994	30.855

are responsible. To test the antibacterial efficacy of Co-Ni-Fe<sub>2</sub>O<sub>4</sub> ferrite, researchers used growth inhibition zones to prevent the development of E. coli and Staphylococcus aureus, both gram-negative bacteria. Co-precipitated Co-Ni-Fe<sub>2</sub>O<sub>4</sub> NCs had growth inhibition zones of 33 and 28.565 mm, respectively, when grown using this method. Using the pulsed laser deposition (PLD) approach, the Co-Ni-Fe<sub>2</sub>O<sub>4</sub> NCs had narrower zones (31 mm and 20 mm, respectively). Using these NCs to treat infections caused by these bacteria is a possibility.

#### 4. Conclusions

Cobalt-Nickel ferrite nanopowders with chemical formula of Co<sub>1-x</sub>Ni<sub>x</sub>Fe<sub>2</sub>O<sub>4</sub> (where, x= 0.0, 0.2, 0.4) were synthesized using pulsed laser deposition method. The cubic spinel phase is seen in the ferrite samples' XRD patterns. As established by X-ray diffraction, the size of a single cubic spinel structure ranging from 9 to 15 nm is the typical crystalline size of ferrite nanoparticles. The nanocrystal lines in the produced products are confirmed by FESEM pictures. Infrared spectroscopy of the ferrite powders reveals two distinct bands in the 500-600 cm<sup>-1</sup> and 385-450 cm<sup>-1</sup> wavelength range, It is possible that the tetrahedral and octahedral complexes



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