Synthesis of ferromagnetic material nano powder and study of its effect on antibacterial activity

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This work is to synthesize the cobalt ferrites nano powder of with the stoichiometric formula $Co_xFe_2O_4$ (x= 0.8 and 1.4) by using sol-gel auto combustion method , and study structural and magnetic properties, the ferrite powders were calcined at 500, 600, and 700 °C for 3 hrs to remove organic waste and increase homogeneity and, The XRD diffraction analysis for $Co_xFe_2O_4$ nanoferrites with single spinel structure. The crystal size of the formed crystallite of ferrite specimens increases with increasing temperature calcination and cobalt content , (FE-SEM) shows agglomerated with homogenous spherical and polyhedral particles. proportion of each of Fe ,Co, and O in all specimens is confirmed using the Energy Dispersive Spectrum (EDS),the magnetic characteristics are measured with a VSM. The remanence magnetization (Mr) saturation magnetization(Ms), and magnetic moment (n_B) of $Co_xFe_2O_4$ nanoferrites are found to go up with increasing calcination temperature, and *E-coli* bacteria were used to examine the inhibition of each ferrite using the diffusion-drilling agar method, as it is known that these nano powder possess therapeutic efficacy for numerous diseases.

1. Introduction

Recently we have been attracted to nanomaterials. It possesses different physical and chemical properties in the range of those types of bulk materials by using the descriptive large area [1,2]. The properties of nanoferrite are affected by composition and internal structure, which turns out to be sensitive to its preparation method, synthesis, and firing conditions. Ferrite nanoparticles are prepared. Crystalline ferrites are nanomaterials close in their structural, magnetic, electrical, electrical, insulating and optical properties, suitable for modern technological applications such as electric generators, microwaves and transformers [5]. These ferrites consist a series of compounds with the formula MFe_2O_4 in which a mixture of ferrite spinel minerals, which is a kind of magnetic material with (M = divalent metal ion, for example(Ni, Co, Mn, Mg, Zn, Cu , etc.), and it is one of the most characteristic and universal materials with its melting point, magnetic saturation, high specific heating temperature, large expansion coefficient, ferromagnetism, magnetism, etc. [7,8]. It consists of ferric oxide and metal oxides. On the basis of internal crystal structure, ferrites are classified into three groups namely spinel, hexagonal and garnet ferrite. The modern type of spinel has been studied extensively due to its many applications in the fields, the ferrite studies that have begun to catch them have increased exponentially. The works of many research sites on the structural and magnetic characterization of ferrite in the form of very small nanoscales [10, 11]. , ferrite composite is an attractive material due to its large anisotropy, geographic location ,moderate magnetization, malleability, and Curie temperature [12, 13]. The initial formula for the internal composition. It is known that the magnetic properties and composition also depend on the size, morphology and purity of the ferrite powder [14, 15]. The ferromagnetism that the nanoscale generates electromotive force, saturation magnetization, coercivity, etc. compared to raw materials where the particle size to the nanoscale [16]. Magnetostrictive cobalt ferrite nanoparticles have been proposed [17–18]. Genes has already been used in the development of cobalt ferrite nanoparticles including a novel thermo-solvent approach [19]. E .coli bacteria represent a type of intestinal bacteria, which is a negative bacilli of the pigment of kram, which was first isolated from

the feces of children in 1885, is naturally present in the human intestine, but it helps the body to produce vitamin K vitamin K, and prevent the infection of some harmful bacteria, but some strains cause For many diseases, especially urinary tract infections, intestinal influenza and meningitis [20] .The aim of the present work is to study the effect of calcined temperature and cobalt ratio on the internal structural and magnetic properties of cobalt ferrite synthesized by the sol gel method and to study the effect of nano powder on the inhibition the activity of E.coli bacteria.

2. Experimental

2.1. Sample preparation

Metal nitrate (Cobalt nitrate, Iron nitrate) and citric acid were used. The nitrate solution is mixed with the acid solution in a temperature flask (Pyrex), and the solutions are mixed using a magnetic mixer with the acid function set to a value of (pH = 7) by adding drops of ammonia to the mixture, and Mixing the materials for half an hour at room temperature. The temperature is gradually raised and then fixed at 90°C, with continuous stirring until a gel form is reached. The gel of $CoxFe_2O_4$ is placed in the furnace and heated to 200 °C, and the powders is placed in the porcelain pot and calcined at a temperature of 500,600,700 °C for 3 hours to remove reaction residues such as carbon dioxide from combustion and water molecules.

3. Antibacterial activity

The antibacterial activity by Using the agar well diffusion method, the nanopowder of $Co_xFe_2O_4$ *E. coli* bacteria was investigated. The diffusion by etching approach was used to identify the microbiological activity of the extract as follows: A medium (Muller-Hinton agar) was prepared and poured into dishes and left to solidify [21]. The dishes were inoculated by placing 0.1 ml of activated bacterial inoculum (Inoculum) on the brain-Heart infusion broth medium by a pipette in the center of the dish and spread by the diffuser (Glass spreader) to soften the surface of the agar. Then, little pits were created in each dish using a cork borer with a 6 mm diameter and three evenly spaced holes for each dish. Then, 200 μl of $Co_xFe_2O_4$ solution was added to each hole of each extract in equal amounts. In a hole in the same dish, and be sure not to spill the material on the surface of the dish around the hole. Then, the dishes were left to dry as the fluids diffused through the holes using the diffusion method. The dishes were placed in an incubator at 37 ° C for 24 hours, the inhibition areas were measured, and the next day the inhibition areas were measured. After the incubation period had passed, the results were read as the positive result was the appearance of an aura of inhibition (Transparent area around the holes) and their diameters are measured. As for the negative result, it is the appearance of growth and the absence of an inhibition aura.

4. Results and Discussion

4.1 Structural Properties
4.1.1. X-ray Diffraction Studies

X-ray patterns of $Co_xFe_2O_4$ (x= 0.8 and 1.4). are calcined at different temperature 500, 600 and 700 ℃ ,the pattern of X-ray shows all the peaks corresponding to (111), (311), (222), (220), (422), (400),(511),and (440) planes appears to cubic unit cell. This is identical to the card with code ICSD 00-001-1121. All peaks are narrower and sharper. Specimens with x = 0.8 and 1.4 showed few traces of a second phase of hematite (iron oxide) at 600 and 700 ℃. The points to an increase in crystal as a result of the increased the crystalline volume ratio caused by the nuclei's particle size enlargement[22]. XRD patterns of $Co_xFe_2O_4$ shown in figures (1,2).
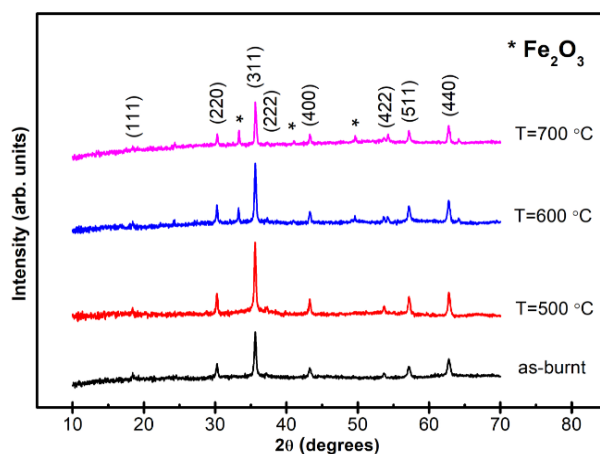
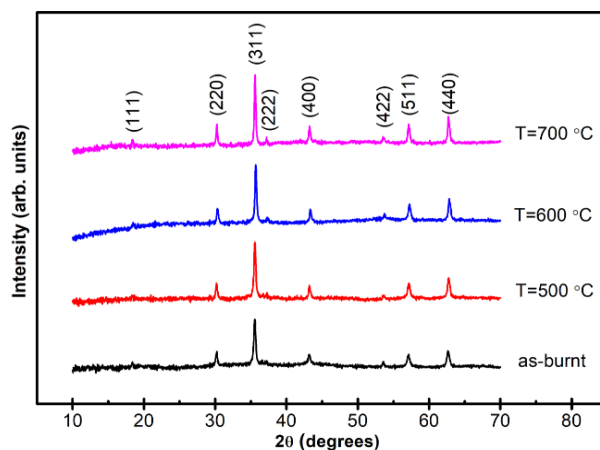*Fig. 1. XRD patterns of Co$_x$Fe$_2$O$_4$ nanoparticles (x=0.8) calcined at 500, 600 and 700 ℃.*

Fig. 2. XRD patterns of Co$_x$Fe$_2$O$_4$ nanoparticles (x=1.4) for calcined at 500, 600 and 700 ℃.

The value of XRD density ($\boldsymbol{\rho_x}$) ,and lattice constant (a) and hopping length (L$_A$) ,(L$_B$) for Co$_x$Fe$_2$O$_4$ nanoparticles and crystallite size (D) are shown in Table 1.

Table 1. Lattice parameter (a) , (D), (ρ_x) (L$_A$) and (L$_B$) for Co$_x$Fe$_2$O$_4$ with (x = 0.8 , 1.4).

x	Composition	Temp.℃	2Theta	FWHM	D(nm)	a(Å)	ρ_x (g/cm^3)	LA (Å)	LB(Å)
0.8	(Co$_{0.8}$Fe$_2$O$_4$)	as-burnt	5.590	0.34	24.530	8.360	5.330	3.6202	2.9559
		500	5.552	0.30	27.798	8.368	5.315	3.6235	2.9585
		600	35.618	0.23	36.265	8.353	5.344	3.6171	2.9533
		700	35.641	0.17	49.067	8.348	5.354	3.6148	2.9515
1.4	(Co$_{1.4}$Fe$_2$O$_4$)\	as-burnt	5.660	0.31	26.909	8.343	5.363	3.6127	2.9497
		500	35.561	0.30	27.798	8.366	5.319	3.6226	2.9578
		600	35.670	0.23	36.270	8.341	5.368	3.6116	2.9489
		700	35.560	0.21	39.712	8.366	5.319	3.6227	2.9579

4.1.2. Field Emission Scanning Electron Microscopy (FE-SEM) Results

The findings of FE-SEM investigations for the compound $Co_xFe_2O_4$ showed that the inter particles have a Spheroidal structure have a narrow distribution of particle sizes, the particles were formed and porosity of the nanoparticles, homogeneous agglomeration of the fine spherical particles, as nanoparticles of vary smaller size have permanent magnetization, therefore all particles have the permanent magnetization, Figures (3,4) shows the tests FE-SEM in (x= 0.8, and 1.4) at a temperatures (500, 600, and 700 ℃) respectively, and indicates an increase in crystalline size increases with increase in temperature. Table 2 displays approximate the diameters of $Co_xFe_2O_4$ nanoparticles (x =0.8 and 1.4) for as-burnt and calcined specimens at 500, 600, and 700 ℃ determined by Image J Software. All the data correspond relatively well with the XRD results, by FE-SEM is found the particle size to be larger than that estimated by using XRD, could be responsible for the disruption of lattice strain and molecular structural by different ionic radii and/or nanoparticle collection. As a result, it has a more stringent requirement, which results in smaller sizes[23].The particle size increased with increased the calcination temperature. Figures (3,4) shows the Field emission scanning electron microscopy of $Co_xFe_2O_4$ nanopowder calcined at 500, 600, and 700 ℃.
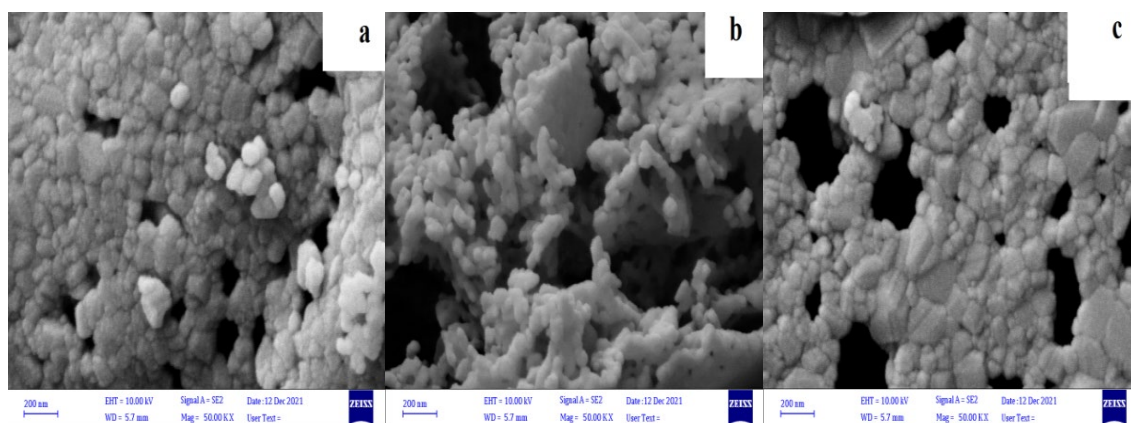
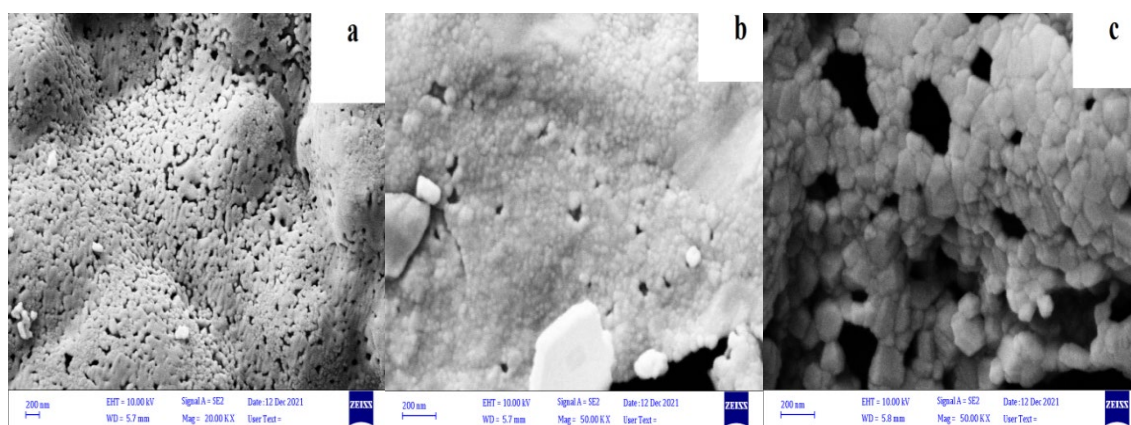

Fig. 3. FE-SEM of $Co_xFe_2O_4$ (x=0.8) for calcined at a=500, b=600 and c=700 ℃.

Fig. 4. FE-SEM of $Co_xFe_2O_4$ (x=1.4) for calcined at a=500, b=600 and c=700 ℃.

Table 2 shows that, except for x=0.8 and 1.4, the particle size (D) increases as the concentration of (X) increases at 500, 600 and 700 ℃. Because of its permanent magnetic moment, the particle is magnetized in all of the time and prefers to clump together. Co-substituted nanoparticles have a greater magnetic moment, which causes more clustering [24,25].

Table 2. Crystallite and particle size of $Co_xFe_2O_4$ (x = 0.8 and 1.4) calcined at 500 ,600 and 700 ℃ from XRD and FE-SEM test.

x	Composition	Temp.℃	D(nm)XRD	D(nm)FE-SEM
0.8	$(Co_{0.8}Fe_2O_4)$	500	27.798	29.964
		600	36.265	39.062
		700	49.067	53.652
1.4	$(Co_{1.4}Fe_2O_4)$	500	27.798	36.350
		600	36.270	38.895
		700	39.712	43.126

4.1.3. Energy Dispersive Spectroscopy (EDS)

(EDS) is a method or Technique for determining the chemical or elemental makeup of a material. It is based on the interaction of an XRD irritation source with a specimen. EDS ability to describe and characterize elements is largely due to the basic premise that each chemical element has a unique atomic structure that allows for a different set of peaks on its electromagnetic spectrum. In the absence of any impurities, The ratio of the chemical elements Co, Fe, and O present in the surface layers of the compounds of $Co_xFe_2O_4$ (x= 0.8, and 1.4) is shown in figures (5,6).

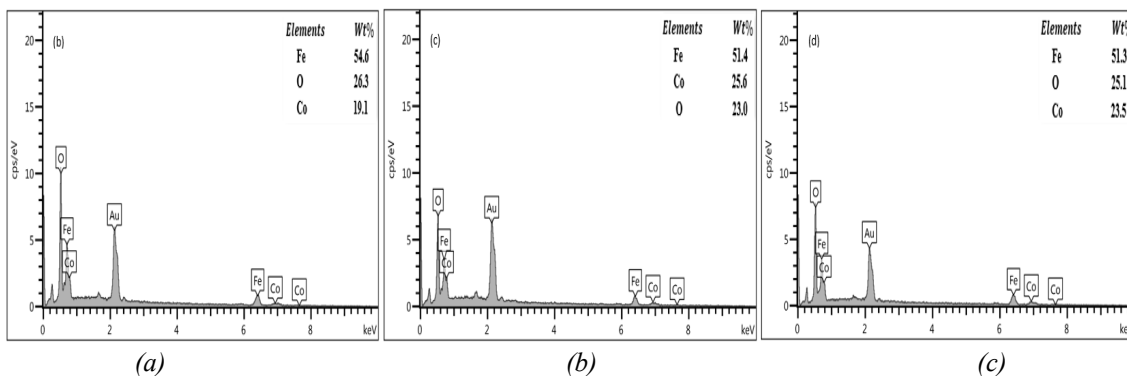*Fig 5. EDS of $Co_xFe_2O_4$ (x=0.8) for calcined at a=500, b=600 and c=700 ℃.*

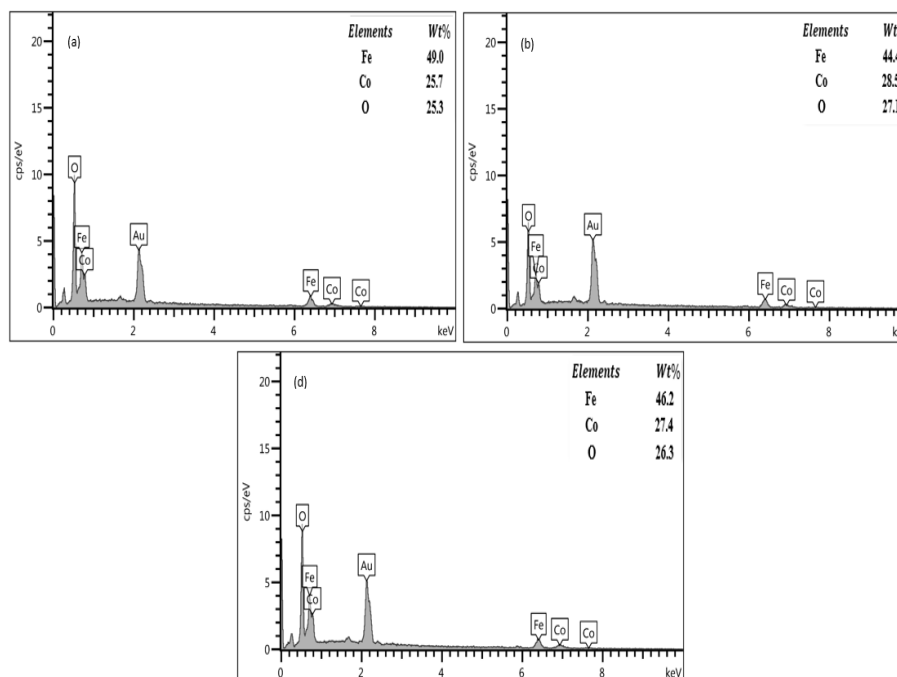*Fig. 6. EDS of $Co_xFe_2O_4$ (x=1.4) for calcined at a=500, b=600 and c=700 ℃.*

4.1.4. Magnetic Properties

Use the magnetic field of ±15 KOe, magnetic characteristics of produced $Co_xFe_2O_4$ nanoparticles (x= 0.8 and 1.4) were measured at room temperature for as-burnt and calcined specimens at varied temperatures 500, 600, and 700 °C. The remanent magnetization (M_r), the saturation magnetization (M_s), the coercive field (H_c) are calculated by using hysteresis loops are showing in figure(7.8).

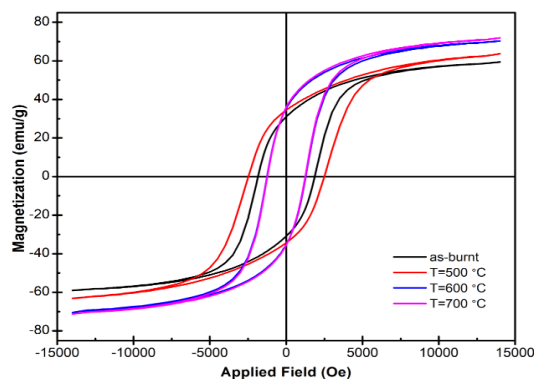Fig. 7. Hysteresis curves of $Co_xFe_2O_4$ nanoparticles (x=0.8) for calcined at 500, 600 and 700 °C.
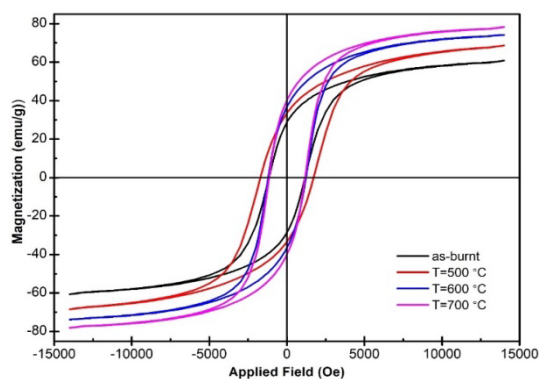

Fig. 8. Hysteresis curves of $Co_xFe_2O_4$ (x=1.4) for calcined at 500, 600 and 700 °C.

Use VSM to obtain magnetic parameters such as the remanence ratio (Mr/Ms), magnetic moment(n_B), magnetic anisotropy(K) are showing in table (3,4).

Table 3. Magnetic parameters 0f $Co_{0.8}Fe_2O_4$ calcined at 500, 600 and 700 °C.

Temp.°C	M_s (emu. g^{-1})	M_r (emu. g^{-1})	H_c (O_e)	n_B (μ_B)	M_r/M_s	$K \times 10^3$ (emu .O_e g^{-1})
As-burnt	59.37	31.04	1854.6	2.49	0.52	114.70
500	63.71	34.41	2474.5	2.68	0.54	164.22
600	70.31	35.41	1239.7	2.95	0.50	90.80
700	71.93	36.02	1228.4	3.02	0.50	92.03

Table 4. Magnetic parameters of Co$_{1.4}$Fe$_2$O$_4$ calcined at 500, 600 and 700 ℃.

Temp.℃	(emu. g^{-1})M$_s$	(emu. M$_r$ g^{-1})	(O$_e$)H$_c$	(μ$_B$)n$_B$	M$_r$/M$_s$	K × 10^3 (emu .O$_e$ g^{-1})
As-burnt	61.77	28.85	1151.6	2.54	0.46	72.93
500	67.63	34.43	1707.5	2.84	0.59	121.10
600	73.13	36.79	1199.3	3.13	0.49	92.59
700	78.12	40.03	1149.5	3.22	0.52	93.66

4.2. Antibacterial activity application

Through the biological application on bacteria, the extracts prepared by the mentioned method were used. The results showed the Co$_{0.8}$Fe$_2$O$_4$ and Co$_{1.4}$Fe$_2$O$_4$ killing *E. coli* bacteria, this demonstrated its antibacterial action. the results were read as the positive result was the appearance of an aura of inhibition (Transparent area around the holes) shown in figure (9).
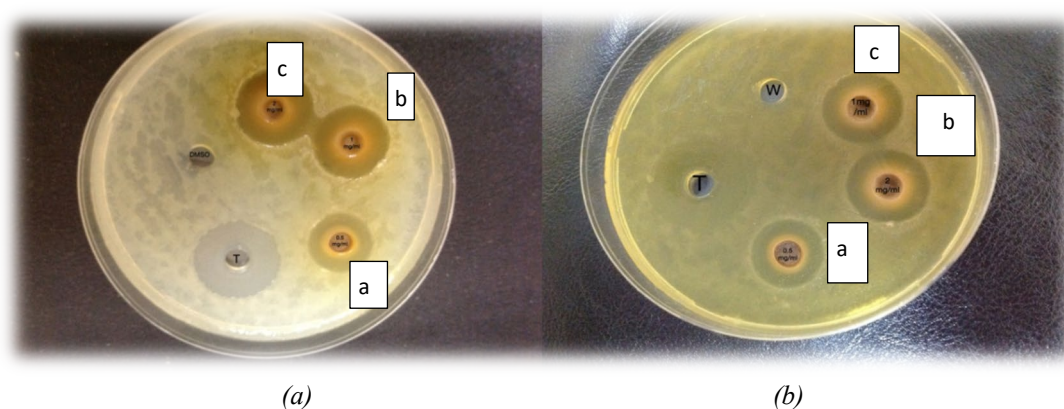

(a)   *(b)*

Fig. 9. Antimicrobial activity of A=(Co$_{0.8}$Fe$_2$O$_4$) and B=(Co$_{1.4}$Fe$_2$O$_4$), at a=500, b=600 and c=700 ℃.against E.coli bactria.

There are many mechanisms that explain the effectiveness of nano powder in killing pathogenic bacteria, where nanoparticles bind with the bacterial cell because the surface of microorganisms is negatively charged, while nano-oxides have a positive charge, which leads to an electromagnetic link between bacteria and nanoparticles, as the nanoparticles release ion It binds with the (-SH) group in bacterial membrane proteins, causing a defect in the bacterial cell membrane functions, which leads to bacterial cell death.

5. Conclusion

Preparation of (Co$_x$Fe$_2$O$_4$) cobalt ferrite by a sol-gel Technique, Obtaining a nano powder. The face-centered cubic phase of Co$_x$Fe$_2$O$_3$ with high purity formed in samples, with average crystallite sizes of around (24 -49 nm) with high magnetic specifications. Ferrite magnetic solution was used to inhibit *E.coli* bacteria by using well-diffusion method. The results appeared that the diameter of inhibition zone was found that it has the ability to inhibit bacteria by measuring the diameter of the hole.

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