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Effect of pH and RF Plasma Treatment on the Structural and Magnetic Properties of (Fe,Cu)Fe₂O₄ Spinel Ferrites

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ABSTRACT: This work used the sol-gel process to synthesise Fe_{1-x}Cu_xFe₂O₄ ((Fe, Cu) ferrite) magnetic nanoparticles having a radius of 10 nm at two pH values, 8 and 9, and in the ratios $x = 1$ and 0.9. Using field emission scanning electron microscopy (FESEM), the surface morphology and homogeneity of the samples were examined. The electron microscope pictures showed certain groups that were almost dense and had fewer pores. A soft iron material with a tight hysteresis loop was developed after the magnetic characteristics were further examined using the Vibration Magnetic Microscope (VSM). The compound was then subjected to the same amount of radiofrequency (RF) plasma. According to the FESEM study results, the compound's form and size have changed, and it has become heterogeneous since it is no longer spherical as it was before exposure. Following exposure to plasma, VSM measurements of (Fe,Cu)Fe₂O₄ reveal a rise in saturation magnetisation and a change in the hysteresis loop's structure at the concentration $x=0.9$ and at the value of pH=9. The Fourier transform infrared spectroscopy (FTIR) measurements show two main bands for the positions of the atoms in the tetrahedral sites (A) and the octahedral sites (B) in the crystal structure. The produced materials can be utilised to create excellent magnets or to minimise eddy currents in electrical transformer cores.

1. INTRODUCTION

In modern days, from a fundamental science point of view, owing to spinel ferrite nanomaterials, they have gained huge attention for their unique properties and promising applications such as high-density data storage, gas sensors, information storage systems, magnetic bulk cores, microwave absorbers, medical diagnostics and therapy, etc. [1-2]. On their crystal shapes and magnetic-based traits, Ferrites are categorised as spinel (MFe₂O₄), garnet (M₃Fe₅O₁₂), hexaferrite (SrFe₁₂O₁₉ and BaFe₁₂O₁₉), and orthoferrite (MFeO₃, M = rare earth cations) [3]. Spinel ferrites have a face-centred cubic (FCC) unit cell, which has eight formula units. The formula can be written as M₈Fe₁₆O₃₂, with anions being the biggest and creating the FCC lattice. Under these lattices, two types of interstitial spaces exist, which are inhabited by mineral cations, as illustrated in Figure 1. [4-5]. The crystal structure is based on a face-centred cubic structure (FCC) of oxygen anions (O²⁻), whose tetrahedral sites (denoted A) and octahedron sites (denoted B) are partially filled by the metal cations M⁺² and F⁺³. Only half of the octahedral locations and one-eighth of the tetrahedral sites are populated [6]. Ferrite has been explored since the mid-20th century for its impact on magnetic applications. The ferrite resistance at ambient temperature varies depending on its chemical composition [7]. In comparison to other magnetic materials, ferrite is great because it has low eddy current losses and high electrical resistance, which is another essential characteristic in ferrites. Because of the growing interest in ferrites, their qualities have improved. [8-9].

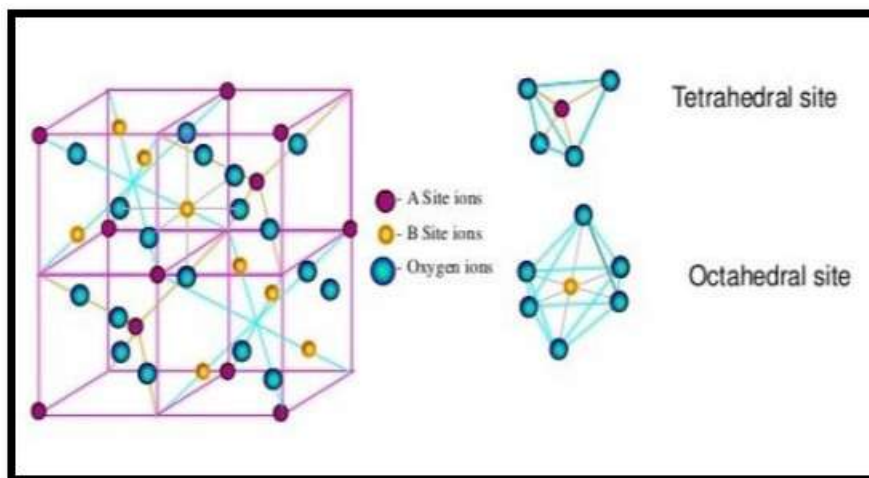


Figure 1: the crystalline makeup of spinel ferrite comprising tetrahedral and octahedral sites [4].

Ferromagnetism exists only in compounds with more complicated crystal structures than pure elements. These compounds, like ferromagnetic materials, spontaneously magnetise at a crucial temperature known as the Curie temperature (T_c). The greatest example of such a substance is iron oxide, which includes the mineral ferrite [10].

Copper ferrite (CuFe_2O_4) is an appealing heterogeneous Fenton-like catalyst because of the synergistic actions of Fe and Cu ions, as studied A. Soufi al.et. Copper-magnesium ferrite nano-particles [11]. The use of the suitable metal activator ion causes intriguing changes in CuFe_2O_4 characteristics, such as particle size and shape control with homogeneous size distribution [12]. Due to their high electric resistance, ferrites are valuable magnetic materials. It is utilised for antenna cores, broadband transformers, ultrasonic generators, and isolators. Soft ferrite is a kind of magnetic material used as a transformer core, primarily for television, telecommunications, computers, medical, and other industrial electronic systems [13-14].

Plasma can be described as a collection of ionised particles that interact via long-range electromagnetic forces and are characterised by their charges and mobility [15]. The interaction of plasma and treated surfaces can result in chemical reactions, abrasive patterns, or thick interlocking layers, as studied A. Hefdhhi Abbas al.et showed the effect of plasma on spinel ferrite, which was prepared by the sol-gel method[16]. RF plasma is often used for surface treatment and precipitation. It might include molecule interactions with the surface, which could result in an increase or reduction in surface charge or the number of absorbed monolayers. It also has the ability to change the physicochemical states of the material's superficial monolayers [17]. For surface etching and thin-film semiconductor production, it also contains reactive species and high-energy ions. [18].

2. MATERIALS AND METHODS

2.1 Sample Preparation

Spinel ferrite with the formula $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ weight ratios ($x = 1, 0.9$) as shown in Table (1) were prepared from high purity sources to obtain accurate results by internal combustion method. After dissolving the metal nitrate and citric acid in deionised water and weighing them on a sensitive scale, the sol-gel method's procedures were followed to begin heating the mixture in phases. The sol-gel method is often favoured over other material synthesis techniques due to its ability to produce materials with high purity, homogeneity, and controlled particle size and shape, especially at the nanoscale. It allows for processing at relatively low temperatures, making it suitable for a wider range of materials, and offers flexibility in creating various material forms like thin films, fibres, and powders. Which included the metal nitrate solution's pH was increased to 8 by adding ammonia.. Then it was heated in a water bath regulated to a temperature of 60 degrees Celsius, then the temperature was gradually increased for three hours to form a dry gel. The gel was subjected to a drying process for a duration of two hours at an environment with a temperature of 120 C°, at which point it underwent spontaneous combustion, resulting in the formation of a dispersed powder that ignited upon exposure to air. In the second stage of the second concentration $x=0.9$, we increase the ammonia until we reach a degree $\text{pH} = 9$, then we continue to increase the

temperature also until the sample reaches the gel and then combusts and forms a brittle powder. The powdered particles were crushed and melted well in a thermal oven at a temperature of 1100 degrees Fahrenheit. the first for concentration $x=1$ for 6 hours, and the second at 0.9 for 4 hosurs. The next step is to grind it for a few hours until it becomes a uniform material with nanoscopic dimensions. After that, the final samples are combined with 6% glycerin and compressed using a hydraulic press that applies a pressure force. A disc-shaped mould with a diameter of (10) mm is utilised for this purpose, as illustrated in Figure(2).

2.2 Plasma Exposure

The second step by placing the samples in the RF magnetic spray system, which is a sequence of three major stages stated as follows: After cleaning the copper target, we place the sample located in the chamber's middle, seal the lid, turn on the spinning vacuum and raise the temperature to 523 K. When the proper pressure is applied, the tool is triggered. To increase plasma formation, the interior space must be supplied with argon gas. Next, switch on the RF source and lower the refractive power until it reaches zero. At this point, the deposition procedure begins and takes two hours to complete. The Figure(3) shows a schematic diagram of a plasma generation chamber.

Table 1: Shows the weights utilised in the preparation work of the composite (Fe,Cu) Fe_2O_4 .

No. of mole	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$	$\text{C}_6\text{H}_8\text{O}_7$	X	1-X	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$
1	404.0	241.60	192.12			40.4
0.1	40.4	24.160	192.12	1	0	0
		21.74		0.9	0.1	4.04

3. RESULTS AND DISCUSSION

3.1 Field Emission Scanning Electron Microscope Test(FE-SEM).

It is used to investigate surface analysis techniques, producing high-resolution photos that show surface topography and the appearance of the sample surface by determining the location of the scanning primary electron beam in relation to the secondary electron intensity. Focusing the primary electron beam to a small point (<10 nm) allows for high spatial resolution [19]. Figures (4a-b) show FE-SEM pictures at various magnifications, as well as an analysis of nanoparticle diameters. The photos (4-5-6-7) depict samples of concentrations ($x= 1,0.9$) at two phases before and after plasma exposure.



Figure 2: Samples disc

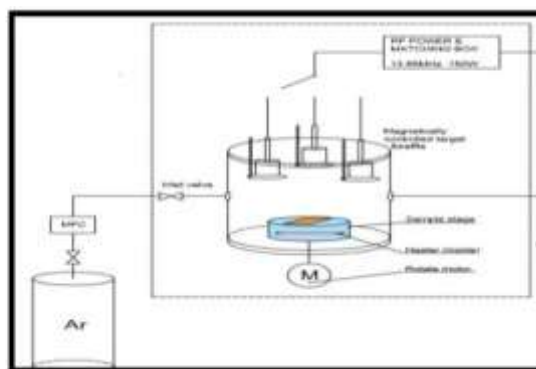


Figure 3: Schematic diagram of sputtering system.

Without plasma exposure and at pH =8, we can see A visual representation of both samples may be found in Figure(4) It was difficult to define the morphology since samples were formed of a mixture of clearly differentiable small and large clusters and are less than 100 nm in size, It was measured by software draw Digmazer. After increasing the pH = 9, we noted that the combustion process was faster to reach the powder, and as shown in Figure (5a-b), there was a change in the form and size of the particles, with measurements taken in nanoscale dimensions. This alteration is due to the effect of doping with the copper ion Cu^{+2} , the results of the examination by C.Lokhande, et al.[20]. Figure (6a-b) depicts the size and form of the sample following exposure to plasma. The structure changed dramatically at a pH concentration = 8 and pH=9 , Figure (7a-b), nanoparticles experienced a minor oxidation process due to heating and exposure to plasma reactive species, which changed their appearance and structure. The Plasma exposure also cleared the sample from the burnt gel. carbon and other unwanted products. [21]. After replacing the copper ion, as shown in the figures, The plasma-exposed surface showed a shift in the crystallographic positions and cohesiveness after the plasma brought them to greatest concentrations, which may be the consequence of increased magnetic influence and lattice defects in the ferrite nanocrystals[22].

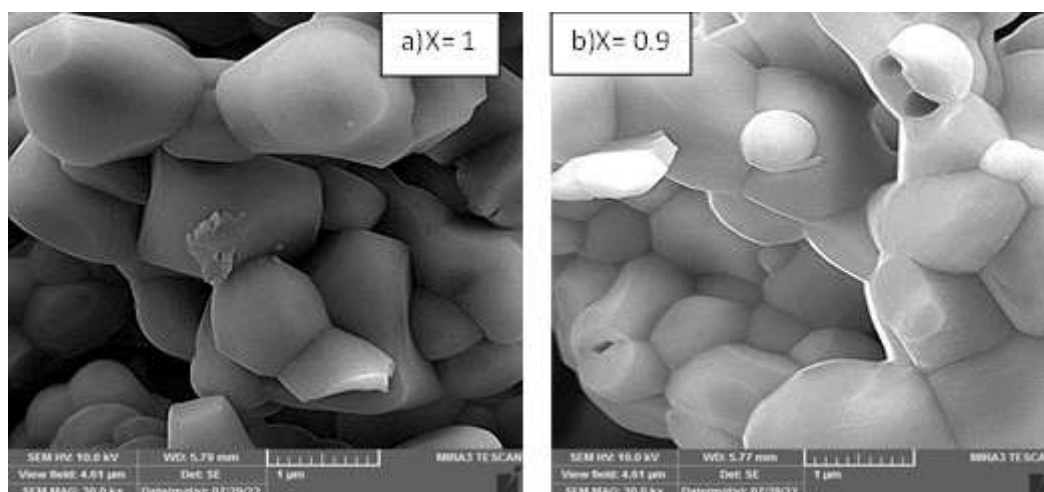


Figure 4: FESEM images of $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ at pH=8 Prior to exposure to plasma

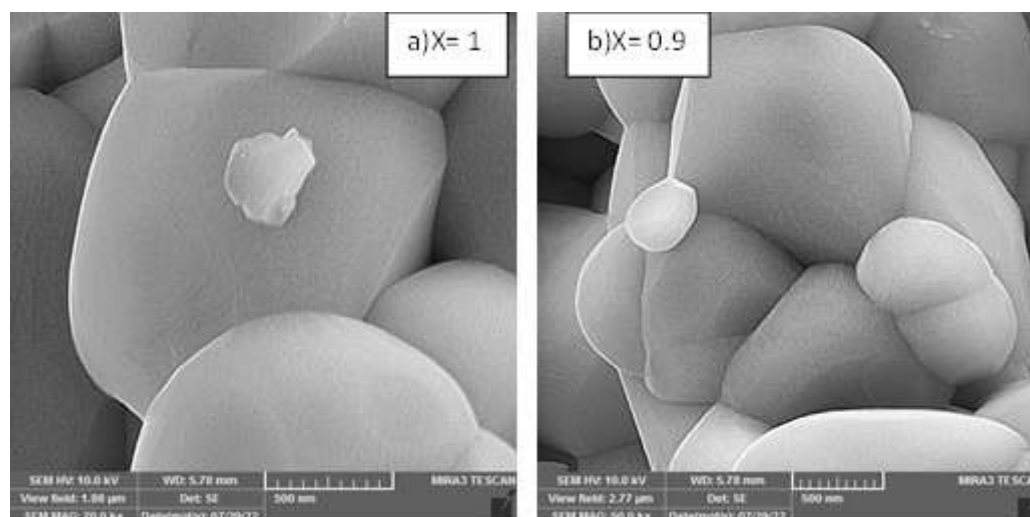


Figure 5: FESEM images of $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ at pH=9 Prior to exposure to plasma

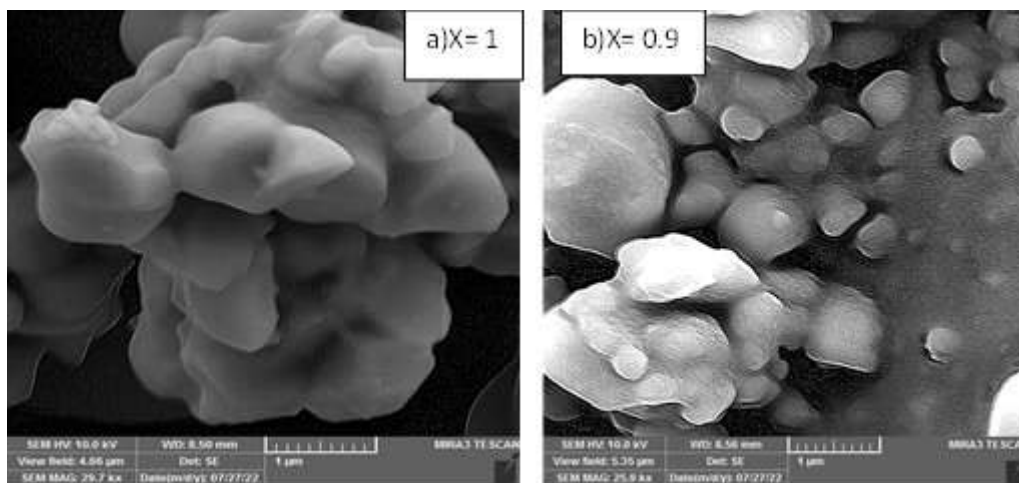


Figure 6: Images from FESEM of $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ at pH=8 after plasma exposure.

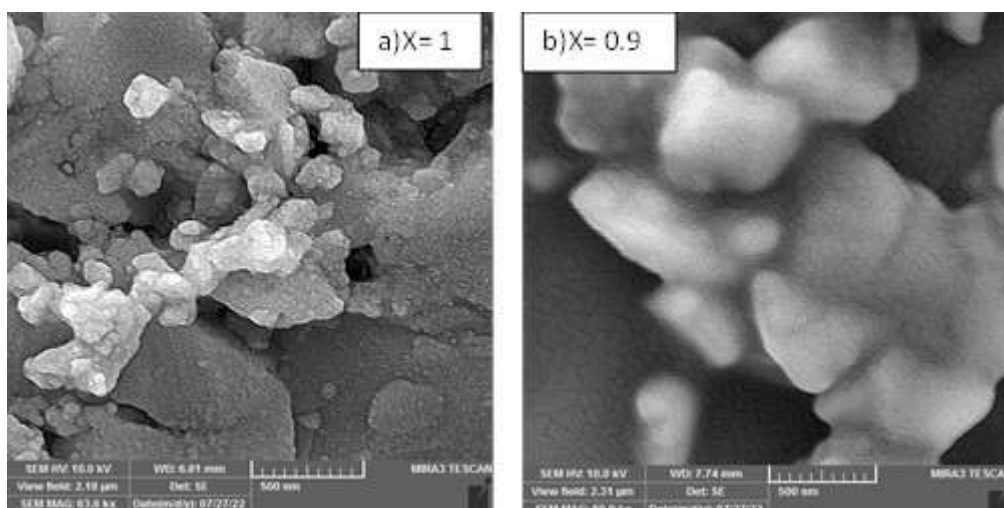


Figure 7: FESEM images of $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ at pH=9 after plasma exposure.

3.2 MAGNETIC DATA

The VSM gadget (type 7400 VSM USA) has an applied field strength of around At room temperature, the O_e value is 15000. Measure the magnetic hysteresis loop of the sample and ascertain its magnetic characteristics[23].

The hysteresis loop of the produced ferrite $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ in Fig. (8a-b) before plasma exposure and at $H = 8$ reveals the samples' magnetic behaviour. The thin curves depict the magnetic characteristics and behaviour of ferrite spinel. This is the attribute of soft ferrite, a material that loses magnetic. The figure depicts the magnetic behaviour of the compound at $x = (1,0.9)$. Magnetic parameters include coercive force H_c (Coercive force (H_c) is a magnetic factor that describes a ferromagnetic material's capacity to resist demagnetisation when exposed to an exterior magnetic field), remanent magnetism M_r relates to a material's residual magnetic strength following the removal of an external magnetic field, and saturated magnetism M_s is the most magnetic moment per unit volume that a completely magnetised magnetic substance can produce, These magnetic characteristics are displayed in the Tables (2-3), along with a comparison of the values before and after exposure to plasma and after varying the concentration. Since concentrating of Cu^{+2} ions increases, the area of the hysteresis loop decreases. The Figure (9 a-b) represents the samples at $x = 1$ and $x = 0$ at pH value = 9. We notice a slight change in the hysteresis loop, but also it remained of the soft type. The Figures (9a-b) depict at $x = 1$ and pH = 9. At $x = 0$, there is a slight variation in the shape of the hysteresis loop boucle due to the replacement of copper, but it remains of the same type. This might be attributed to the production of Cu^{+2} ions in cubic structures and an increase in residual magnification in the ratio

(x), these findings are in line with those of M. Srivastava, who has previously studied the effects of pH on the magnetic properties of nanoparticles and found that these properties vary depending on the pH, likely as a result of variations in nanoparticle size and surface morphology[24].

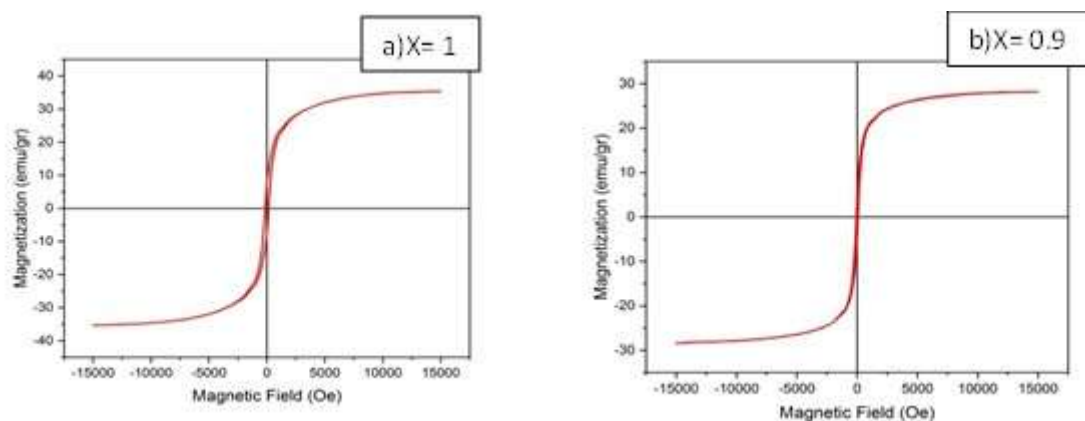


Figure 8: Magnetic hysteresis curves of (Fe,Cu)Fe₂O₄ at pH=8 Prior to exposure to plasma.

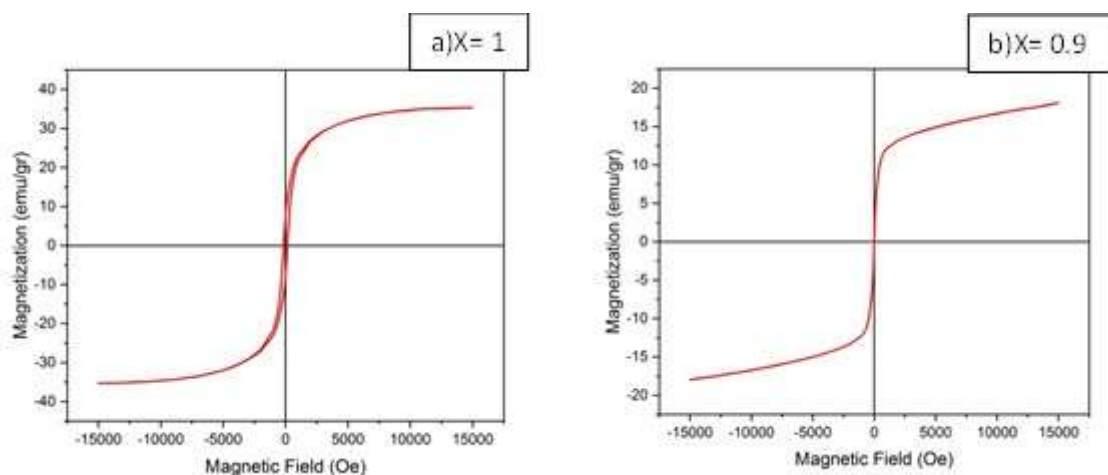


Figure 9: Magnetic hysteresis curves of (Fe,Cu)Fe₂O₄ at pH=9 Prior to exposure to plasma.

Hind pH=8 and Figure (10 a,b) depict all of the rings on the soft magnetism of copper-iron ferrite after exposure to plasma, as seen in Figure(11 a,b). The narrow hysteretic ring shows magnetic loss. A variety of characteristics influence such as the manufacturing method and the value of pH the magnetic properties of spinel ferrite, Which enhances combustion moldability upon combustion , as well as the arrangement of cations at tetrahedral and octahedral locations with change value x. Ms levels rise in plasma-treated ferrite instances, Alyaa Hefdh Abbas Aziz et al. conducted morphological tests on ferrite before and after treatment, which revealed uneven grain distribution and dominant aggregation and the researchers reached almost the same results[25]. This rise in saturation magnetisation is due to the direct interaction between ferrite samples and radiofrequency plasma, which causes a change in coercive value during plasma exposure, where Ms can be impacted by plasma treatment through changes in the material's composition or the density of magnetic moments. By altering the alignment and stability of magnetic domains, remanence (Mr) can also have an impact on Mr [26].

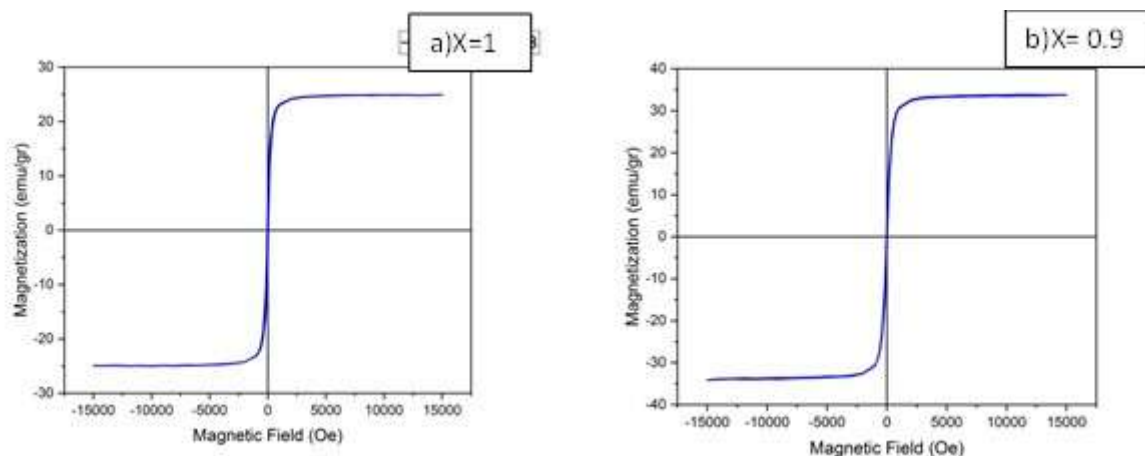


Figure 10: Magnetic hysteresis curves of (Fe,Cu)Fe₂O₄ at pH=8 after plasma exposure.

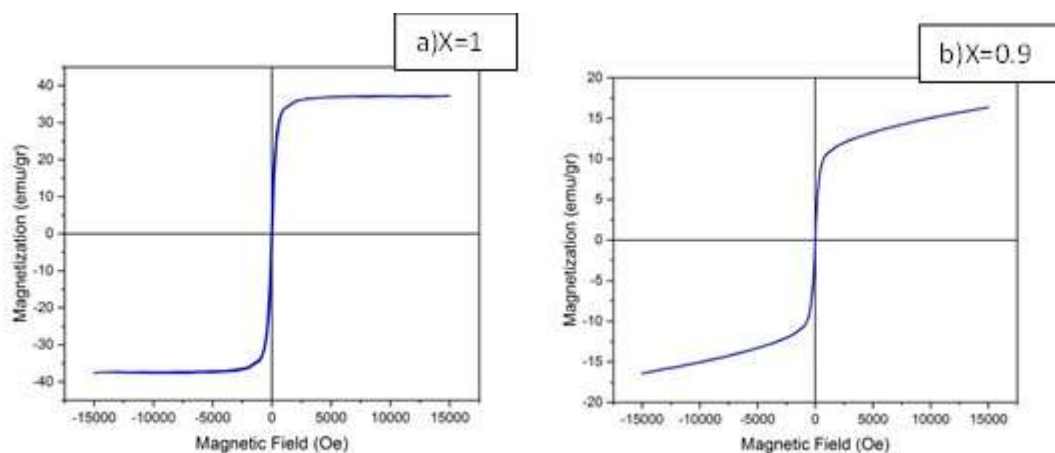


Figure 11: Magnetic hysteresis curves of (Fe,Cu)Fe₂O₄ at pH=9 after plasma exposure.

Table 2: Magnetic characteristics of RF Prior plasma-treated and (Fe,Cu) Fe₂O₄ nanoparticles

Sample	pH	x	Ms (emu/g)	Mr (emu/g)	H (Oe)
Prior to Treatment	8	1	38	17	44
		0.9	26	11	45
	9	1	36	15	44
		0.9	16	8	45

Table 3: Magnetic characteristics of RF following plasma-treated and (Fe,Cu) Fe₂O₄ nanoparticles

Sample	pH	x	Ms (emu/g)	Mr (emu/g)	H (Oe)
After the Treatment	8	1	24	17	44
		0.9	32	15	43
	9	1	38	24	45
		0.9	18	13	44

3.3 INFRARED FOURIER TRANSFORM SPECTROSCOPY (FTIR):

A sample fingerprint with an absorbance peak that corresponds to the frequency of atom bond vibrations is captured using FTIR spectroscopy[27]. The structure of samples and unknown materials may also be determined using the vibrational mode frequencies[28].

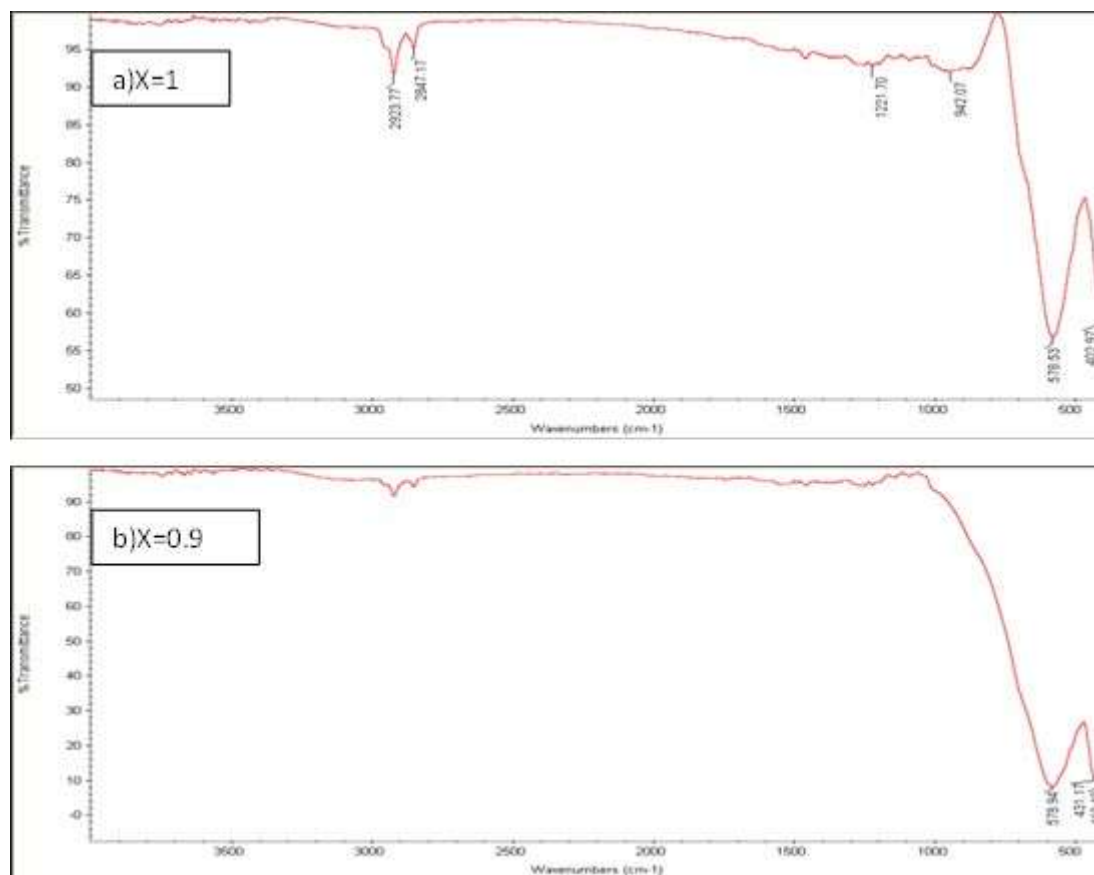


Figure 12: FTIR for the ferrite component $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ with at pH=8 before plasma exposure.

The infrared spectrum of ferrite $\text{Fe}_{1-x}\text{Cu}_x\text{Fe}_2\text{O}_4$ (Fe,Cu) at ambient temperature, within the $400\text{--}4000\text{ cm}^{-1}$ frequency range, is displayed in Figure 12 and 13 at $x=1, 0.9$ at pH=8,9 before plasma exposure. The range of the FTIR spectrum between $(586\text{--}437)\text{ cm}^{-1}$ is displayed in the figure. The main vibrations occur led to a small displacement of red in the peak with the value (x) when increasing the **(Fe,Cu)** ion due to the increased contribution of this component to the iron-oxygen electronic density [29].

After exposure of the compound to plasma in Figure 14 and 15 at $x=1, 0.9$ and pH=8,9 after plasma exposure, FTIR spectroscopy indicates in Figure (4.10) show two essential components peaks around $(557.38\text{--}434.34)\text{ cm}^{-1}$ as well as at $(2922.31\text{--}2583.56)\text{ cm}^{-1}$ for most samples, which correspond to crystal vibrations of bonds in the iron ring. It is very likely that clusters that already possess a significant dipole momentum will start such as C-O, O-H that is an asymmetric or parabolic side with iron oxygen as a deformation of the system leads to an increase in the relaxation of Cu with oxygen and thus its absorption by means of a red displacement [30]. pH variations influence the surface charge of the material, elucidating the reactive groups present and indicating the dominance of acidic or basic characteristics[31]. A negative charge changes, which increases the electrostatic attraction between the metal cations and the surface, increasing cation adsorption. This is somewhat consistent with what researchers M. Syazwan Mustaffahave a.l.et. found where the absorption bands at 1446 cm^{-1} detected in the pure pH sample were attributed to the vibrating bands of Fe-O-Fe owing to the breakdown of metal with oxide band [32].

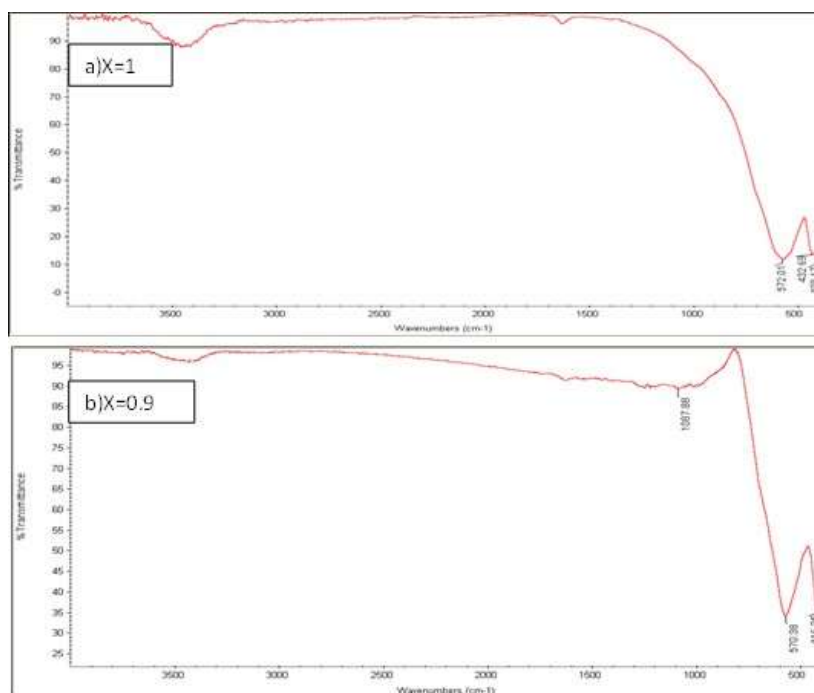


Figure 13: The ferrite component (Fe,Cu)Fe₂O₄ using FTIR with at pH=9 before plasma exposure.

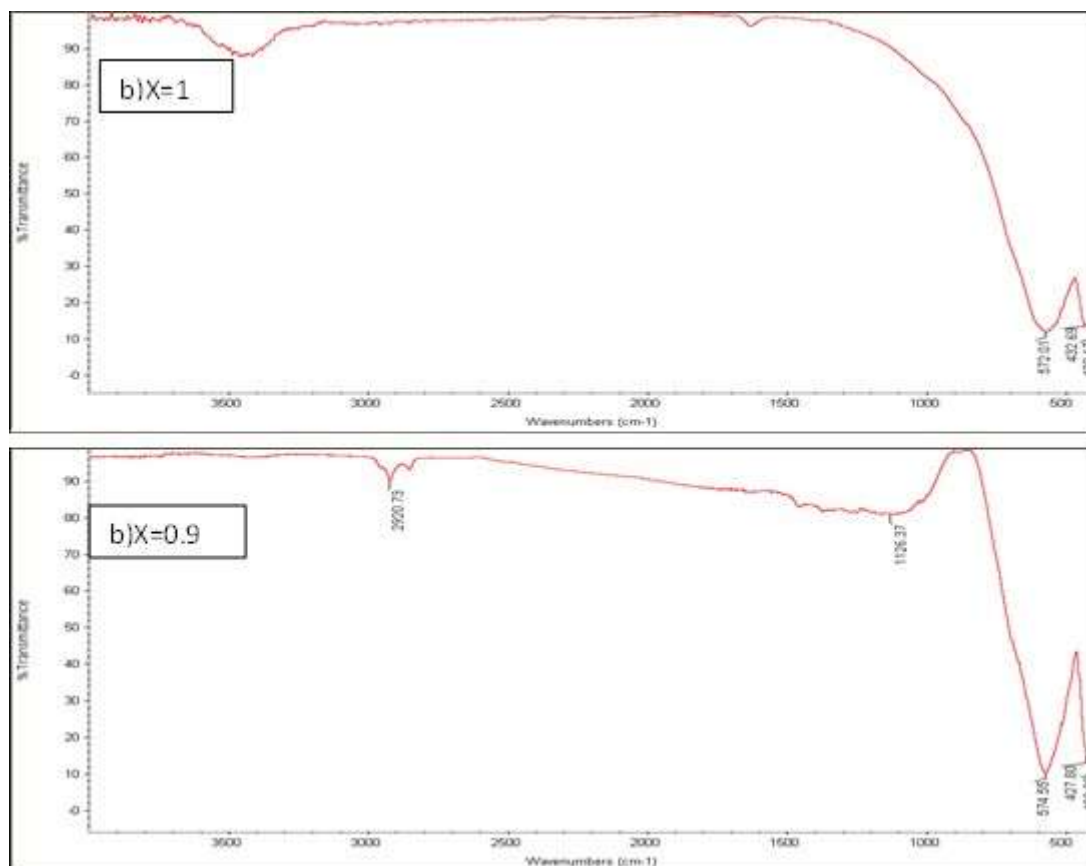


Figure 14: The ferrite component (Fe,Cu)Fe₂O₄ using FTIR with at pH=8 after plasma exposure.

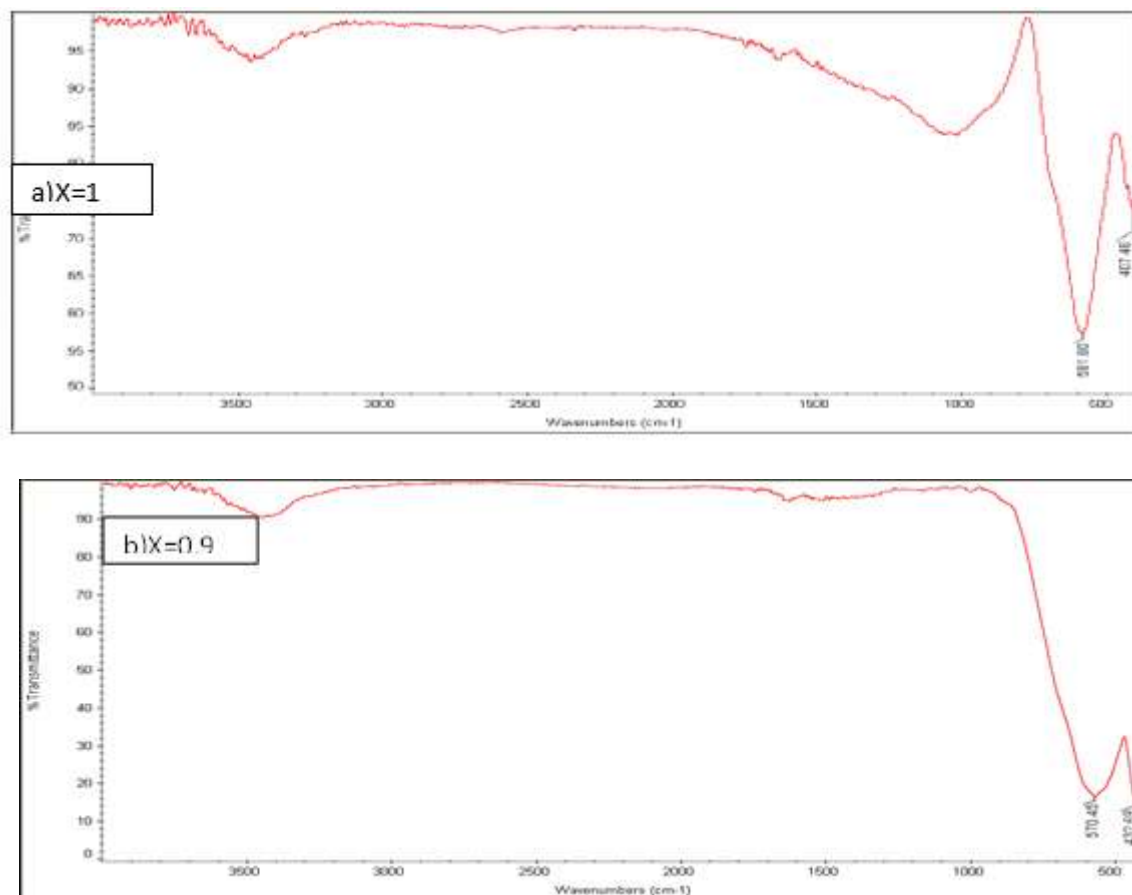


Figure 15: The ferrite component $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ using FTIR with at pH=9 after plasma exposure.

4. CONCLUSIONS

The sol-gel has benefits since it is easy to purify liquids (which are the beginning supplies needed for the procedure), and it produces high purity materials for creating ferrites. Expose the prepared samples to plasma utilising the RF system and employing the target component CuO. FESEM data indicate polymorphic particles of $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ with concentrations of $x=1, 0.9$ and pH values of 8,9 for all generated samples. FESEM micrographs demonstrate uneven grain size distribution. The particles have a relatively spherical sharpness. It is apparent that the crystal's size and form significantly alter after exposure to plasma and after adjusting the pH and concentration levels; it is no longer spherical and becomes heterogeneous.

The soft ferrite materials contained a little residual magnetic ring, according to VSM tests. Because of the size and form of the particles, the produced $(\text{Fe,Cu})\text{Fe}_2\text{O}_4$ complex changed the shape of the Cu^{+2} hysteresis ring with changes in Mg ion and pH values both before and after plasma exposure. M_s , M_r , and H_c for the unexposed samples really increased after plasma exposure. The microwave plasma flattens the ferrite specimens as a result of the significant temperature increase. It is evident from the work's conclusion that the produced chemical has several industrial uses, including gas sensors, information storage systems, bulk magnetic cores, microwave absorption, high-density data storage, and medical diagnosis and treatment. Its characteristics enhance after being exposed to plasma. Prior to exposure to plasma, FTIR studies show two prominent absorption bands in both materials' spectra. The sample's FTIR spectrum shows two concentric absorption bands, representing tetrahedral vibrations and octahedral complexes (metal-oxygen bonds). After plasma exposure, both strips expand and retain O-H and C-O.

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