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Synthesis and Characterization of Copper Ferrite Nano-Particles

Krishna Kumar Thakur[1] and Dr. Atikur Rahman[2]

[1]Research Scholar, MMED NIT Srinagar
[2]Assistant Professor, MMED NIT Srinagar
E-mail: [1]krishnakumarthakur@gmail.com, [2]atikurrhmn@gmail.com

Abstract—*Copper ferrite nanoparticles are synthesized using a laboratory spray pyrolysis method. The samples are annealed at a temperature of 800°C for 2 hr. The structural and morphological properties of the nanoparticles are characterized by SEM analysis, EDS analysis is used to evaluate the chemical composition of samples. Optical properties of the Copper Ferrite nanoparticles are determined by UV-VIS-Spectroscopy analysis.The pH effect on the grain size, the lattice parameter, and photocatalytic properties are studied in the present work.*

1. INTRODUCTION

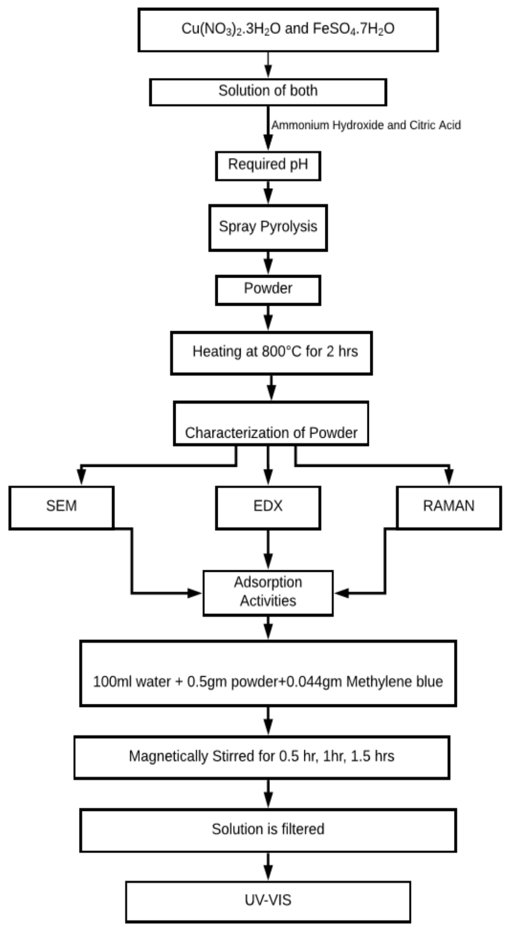
The copper ferrite nanoparticles are being studied because of its excellence magnetic properties and world wide applications in several areas which include water and wastewater treatment, biomedical, catalyst and electronic instrument's printed circuit boards. Therefore the study of copper ferrite is useful. Based on their crystal structures and magnetic properties, ferrites are classified as spinal ($CuFe_2O_4$, where Cu = Mn, Fe, Co, Ni, Co, Zn, etc.), garnet ($M_3Fe_5O_{12}$, where M = rare earth cautions), hexa ferrite ($SrFe_{12}O_{19}$ and $BaFe_{12}O_{19}$) and ortho-ferrite($MFeO_3$, M = rare earth cautions). Among them, especial attention has been given to spinal ferrite nanoparticles (SFNPs).[1-4] $CuFe_2O_4$ is known to exist in tetragonal and cubic structures. Under slow cooling Cu-ferrite crystallizes in a tetragonal structure with lattice parameter ratio c/a of about 1.06. Tetragonal phase of Cu-ferrite has inverse spinel structure with almost all Cu^{2+} ions occupying octahedral sublattice, whereas Fe^{3+} ions divide equally between the tetrahedral and octahedral sublattices[5]. The tetragonal structure is stable at room temperature and transforms to cubic phase only at a temperature of 360 ^{O}C and above. The cubic structure possesses a larger magnetic moment than that of the tetragonal one, because there are more cupric ions ($Cu^{2+)}$) at tetrahedral sites in cubic structure as compared to that in the case of tetragonal structure [6]. Nanosized copper spinel ferrites show unusual properties in comparison with their bulk analogs and receive enormous attention during last decade because of their potential applications. They can be obtained by variety of methods such as solid state reaction [7], mechanochemical, sol–gel, co-precipitation, and combustion synthesis , polyol route and microemulsion hydrothermal route . Most of the previous route led to the formation of tetragonal copper ferrite. From the best of our knowledge, a rarely reported mentioned the synthesis of cubic copper ferrite powders via the hydrothermal route. The correlation between the magnetic and catalytic properties and the microstructure of the produced cubic copper ferrite via the hydrothermal technique need intensive work.

2. EXPERIMENTAL DETAILS

Copper Ferrite nanoparticles are synthesized via laboratory spray pyrolysis method. The high purity powders (99.5%) reagent grade such as Copper (ll) nitrate trihydrate {Cu(No3)2.3H2O} and Iron (ll) Sulfate Crystal purified {FeSO4.7H2O} are used as starting materials. The material powders as detailed above are then mixed with distilled water separately. The 20 ml solutions of prepared solution as per stoichiometry are then taken for experiment which is then mixed with 70 ml distilled water & 5 ml Ammonium Hydroxide & 5 ml Citric Acid (which in total is 100 ml.)Then the PH values of the mixture are measured by pH meter. It has been observed that the pH values were 8.5, 9.5 and 10.5.The prepared solution as per stoichiometry is then sprayed on glass substrate. Coated glass substrate is insert into muffle furnace for drying at 80^{o}C for 30 minutes. Warm coated glass is withdrawn from the muffle furnace after 30 minute and allowed to cool to room temperature. Cooled coated glass is scratched with the help of spatula and deposited powder is collected & put into ceramic boat for further experiment. The ceramic boat filled with synthesized powder is inserting into muffle furnace and temperature of the muffle furnace was maintained at 800^{o}C for 2 hours for annealing. Finally, sintered powder is grounded. In the laboratory adsorption is investigated by UV-VIS-Spectroscopy. Photocatalytic study was done by as: The solution is prepared by 0.5gm/0.1 L of copper ferrite nanoparticles + 0.045 gm/0.1L of methylene blue dye + 100 ml of water. The suspension is magnetically stirred for 0.5 hour, 1 hour & 1.5 hour to disperse the powder & establish adsorption equilibrium. After that, the solution is filtered by Whitman filter paper. The change in concentration of methylene blue dye is monitored by a set of measurement.

The maximum absorbance of methylene blue dye is measured at λ_{max}= 650 nm in an UV-VIS-Spectroscopy.

Flow sheet of experimental details:-



3. RESULTS AND DISCUSSION

The SEM micrographs of copper ferrite nanoparticles with different pH of solution (pH=8.5, 9.5, 10.5) are shown in **Fig. 1 (a-c).** It consists of dendrite particles. According to the micrographs, the microstructure shows a dependence on pH values of solution. **Fig. 1(a-c)** represents decreasing particle sizes as pH is increased. It was observed from SEM analysis that copper ferrite spinal nanoparticles with pH value of 10.5 has smallest particle size (**Fig. 1 (c))** as compared to copper ferrite spinal nanoparticles with different pH values 8.5 & 9.5.

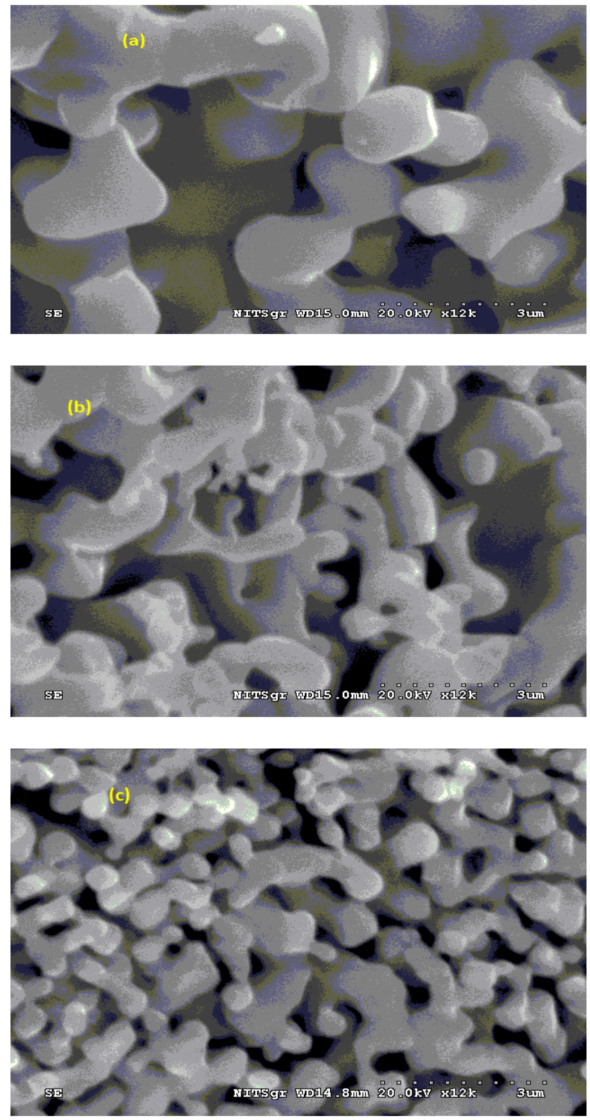
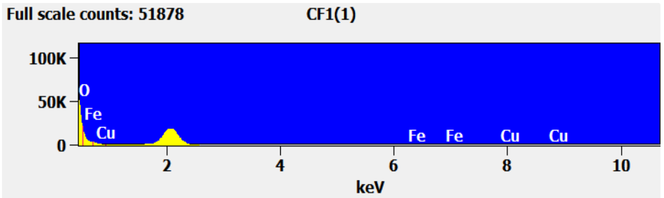


Fig. 1: SEM surface micrographs of copper ferrite spinel nanoparticles with different pH values (a) at pH=8.5, (b) at pH=9.5, (c) at pH=10.5

The chemical composition of the sample was determined by EDS analysis and their results is shown in **Fig.1(d).**

Elements line	**Weight %**	**Weight Error**
O K	27.36S	---
Fe K*	59.93	± 7.53
Fe L*	---	---
Cu K*	12.71	± 5.04
Cu L*	---	---
Total	100.00	
*** -- Standard**		



Adsorption Activities Measurement

In the laboratory adsorption is investigated in glass beaker (250ml) over the magnetic stirrer. The solution for adsorption measurement is prepared by 0.5 gm/0.1 L of copper ferrite nanoparticles + 0.045 gm/0.1 L of methylene blue dye + 100ml of water. The suspension is magnetically stirred for 0.5 hour, 1 hour & 1.5 hour to disperse the powder and establish adsorption equilibrium. After that, the solution is filtered by Whitman filter paper. The change in concentration of methylene blue dye is monitored by a set measurement. The maximum absorbance of methylene blue dye is measured at λ_{max}=664 nm in an ultraviolet (UV-Vis) spectrometer.

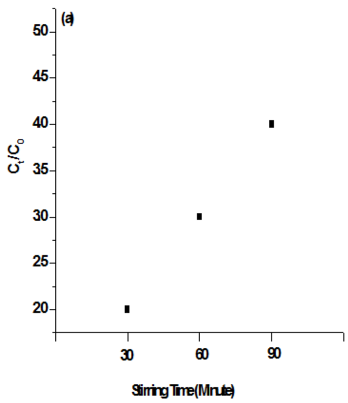


Figure 3.(a) Ratio of remaining concentration to initial concentration of methylene blue in the solution

The fig.3.(a) shows the degree of adsorption of methylene blue dye in the presence of copper ferrite nanoparticles as a function of time under UV light. The relative concentration of the dye increases with the time. This indicate that if stirring time is increased then desorption may take place.

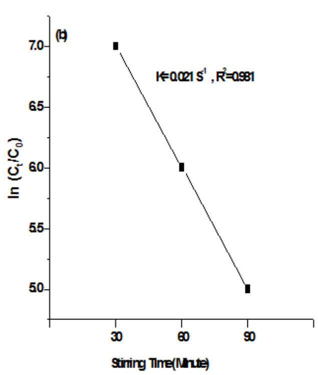


Figure 3.(b) $\ln(C_0/C_t)$ Vs Time

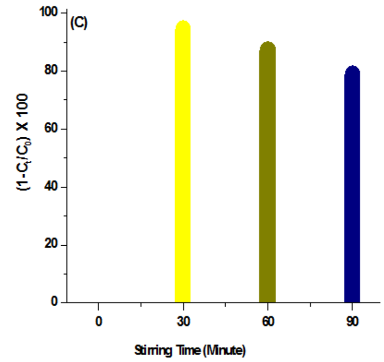


Figure 3. (c) percentage of methylene blue adsorbed Vs time

The methylene blue dye degradation over the copper ferrite spinal nanoparticles with pH value of 10.5 is studied to investigate the adsorption activity. However, it is worth to mention here that the copper ferrite nanoparticles with pH value of 10.5 has been specifically chosen, in order to exploit high surface area of this product because as we increase pH value, it might be nucleation sites increase which result in small particles sizes as compared to powders of lower pH values. The UV-Vis spectra of the adsorbed methylene blue dye at different stirring times is shown in **Fig 3.(a-c)** It shows that the dye exhibits an adsorption peak at 650 nm and the adsorption intensity of the dye solution gradually decreases as the stirring time is increased , indicating a decrease in the methylene blue dye concentration. This study revealed that almost 96% of the methylene blue dye was adsorbed in 30 minutes.

The kinetic behavior of the adsorption of methylene blue is further investigated. It is clear that there is linear relationship between $\ln(C_t/C_0)$ value and irradiation time intervals, where C_t is the concentration after time t and C_0 is the initial concentration before adsorption of dye.

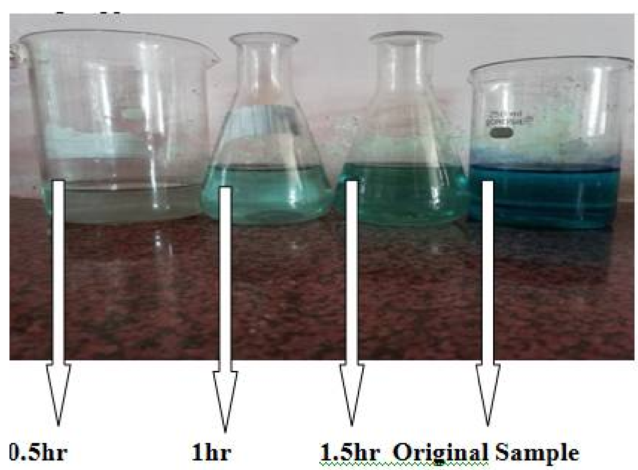


Figure 4: Shows the degradation color of methylene blue dye after different interval of time.

The linear relationship, $\ln(C_t/C_0)=Kt$, of the curves are presented in figure **Fig. 3.(b)**, where K is the pseudo first order rate constant. Generally, as shown in **Fig. 3(c),** good correlations are obtained indicating that the reaction kinetics follows a pseudo first order rate law for adsorption of the dye using copper ferrite spinel nanoparticles. However the rate constants are estimated to be $0..21s^{-1}$ for adsorption of dye using copper ferrite.

4. CONCLUSIONS

Copper ferrite spinal nanoparticles with different pH values (8.5, 9.5, 10.5) have been synthesized by spray pyrolysis method using a mixture of $FeSO_4$.&H_2O, $Cu(NO_3)_2$, ammonium hydroxide, citric acid. The effect of ammonium hydroxide on particle size, microstructure are studied. From the result of the experiment it was concluded that with the increase in stirring time of copper ferrite and methylene blue solution adsorption decreased in significant amount which indicates that desorption takes place with the increase in stirring time.

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