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PHOTOCATALYTIC ACTIVITY OF ZN-CO-FERRITE NANOPARTICLE SYNTHESIZED USING LEMON AS GREEN BINDING AGENT BY SOL-GEL METHOD

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Abstract:

Spinel ferrite nanoparticles exhibits significant magnetic, optical and oxidation properties compared to the bulk dimensions. Nanoparticles of pure ferrites, mixed ferrites and inverse spinels are popular in the field of photocatalysis. Zn-Co-ferrite nanoparticles have been synthesized by sol-gel auto combustion method from nitrate salts of respective metal ions using lemon as a chelating agent. X-ray diffraction pattern confirmed the formation of single-phase nanoparticles of Zn-Co-ferrite. Fourier transform infrared study was performed to ascertain the structure of the nanoparticles. The objective of this study is to find a synthesized compound such as Zn-Co-ferrite ferrite nanoparticles as photocatalyst for degradation of organic dyes. Absorbance versus time measurements are made at λ max values ***430 nm*** *for* ***Tartrazine dye****. It was found that dyes undergo fast degradation with UV light in presence of doped compound. UV-visible spectrophotometer was used to estimate the rate of degradation of dyes from residual concentration in dyes.*

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INTRODUCTION:

The synthesis of nanocrystal is currently an intensive research area due to the verified size dependence of their functional properties & wide range of application. Polycrystalline spinel ferrites are widely used in many electronic devices. They are preferred because of their high permeability in the radio-frequency (RF) region, high electrical resistivity, mechanical hardness and chemical stability. These types of ferrites are subjects of intense theoretical and experimental investigation due to their remarkable magnetic and electric properties (El-Shabasy M. Et al, 1997, Rosales M. I., Et al, 1997, Mahmud S.T., Et al, 2006). Cobalt ferrite ($CoFe_2O_4$) is a well-known hard magnetic material with high coercivity and moderate magnetization. These properties, along with their great physical and chemical stability, make $CoFe_2O_4$ nanoparticles suitable for magnetic recording applications (Skomski R., Et al, 2003). Many efforts have been made to improve the basic properties of these ferrites by substituting or adding various cations of different valence states depending on the applications of interest. Among spinel ferrites, Zn^{2+} substituted $CoFe_2O_4$ nanoparticles exhibit improved properties such as excellent chemical stability, high corrosion resistivity, magnetocrystalline anisotropy, magnetostriction, and magneto-optical properties (Vaidyanathan.G and Sendhilnathan.S et al, 2008, AktherHossain. A. K. M., et al, 2008). Various preparation techniques, such as sol–gel pyrolysis method (Lee.J.G., et al, 1998, SonalSinghal, 2010), the microwave hydrothermal method (Kim .C. K., 2001), template-assisted hydrothermal method (He.H.Y, 2011), and sol–gel auto combustion technique are used to prepare ferrites nanoparticles. The physical properties of nanoparticles are of current interest due to the size dependent behavior observed in the nano scale and high crystallinity.

In the current study, Zn-Co-ferrite nanoparticles have been synthesized by sol-gel auto combustion method from nitrate salts of respective metal ions using lemon as a chelating agent. The focus was to assure a synthesized compound such as Zn-Co-ferrite ferrite nanoparticles as photocatalyst for degradation of organic dyes.

2. EXPERIMENTAL

2.1. Materials

All the chemicals used in this study are commercial samples and belong to analytical grade. They were used as received without further purification. Iron nitrate nonahydrate$Fe(NO_3)_3.9H_2O$, Zinc nitrate hexahydrate $Zn(NO_3)_2.6H_2O$, Cobalt nitrate hexahydrate $Co(NO_3)_2.6H_2O$, were used as a precursors and lemon as a binding agent. **Tartrazine dye** was also used for this study.

2.2. Synthesis of Zn-Co-ferrite nanoparticle

In the preparation of Zn-Co-ferrite nanoparticles were synthesized by sol-gel auto combustion method, using starting material of high purity AR grade metal nitrate and lemon juice. The metal nitrate solutions were mixed in the required stoichiometric ratios in minimum quantity of distilled water. The solutions will be mixed on a magnetic stirrer at 353K. In order to maintain alkaline condition, ammonia solution was added to the reaction mixture (pH 9 - 9.5). The solution mixture was slowly heated around 373K with constant stirring to obtain a fluppy mass. On further heating, colored powder will obtained. The powder will cooled for some time. The obtained powder nanoparticles were annealed at temperature of 973Kfor 4 hours.

2.3. Size characterization of Zn-Co-ferrite nanoparticle

The ferrite nanoparticle prepared was analyzed by X-ray diffraction technique (XRD) for structure and crystallinity. The formation of mixed metal ferrite nanoparticles was confirmed by Fourier transform infrared (FT-IR) studies. X-ray diffraction (XRD) data was collected at room temperature. Crystallographic properties e.g. phase of the material and crystal structure was determined using the same data.

FT-IR analysis was carried out in the range of **4000-400** cm^{-1}. The samples were pelleted with KBr.

2.4. Photocatalytic studies

The catalytic degradation of the dye was performed in open beakers loaded with 50 cm^3 of dye solution and 10 cm distance separation from the irradiation lamp and the surface of the solution was maintained. At the first, photolysis was also carried for dye solution using UV radiations without photocatalyst. The optimized catalyst feed loading was chosen for dye to be 1mg/1cm^3. The solution was irradiated with UV **lamp of 11 W powers**. About 2-3 ml of sample was taken at 2 hrs intervals of time up to 18 hrs. The course of degradation reaction and concentration of dye in samples was monitored by measuring the absorbance values of the dye at their wavelength maxima at regular intervals of time of irradiation using UV-Visible spectrometer at regular intervals of time.

3. RESULTS AND DISCUSSION:

3.1. X-Ray Diffraction (Xrd) Analysis:-

X-ray diffraction (XRD) patterns for the as prepared and annealed polycrystalline $Zn_{0.5}Co_{0.5}Fe_2O_4$ ferrite powders are presented in Figure 1. The XRD spectra showed all the characteristics peaks corresponding to the characteristic planes (311), (511) and (440) appear at **35°, 57° and 64°** on comparing with the patterns of all the investigated samples with that of standard JCPDS card, a single phase $Zn_{0.5}Co_{0.5}Fe_2O_4$ has formed with no extra peaks. At 973K fully crystallized $Zn_{0.5}Co_{0.5}Fe_2O_4$ has formed with sharp peaks indexed as (220), (311), (400), (422), (511) planes of spinel structure. In the present work, Figure 1 reveals the presence of the spinel structure for the as prepared $Zn_{0.5}Co_{0.5}Fe_2O_4$, and the noticed broadening in the peaks of the as prepared sample could be attributed to the formation of ferrite particles in nano range.

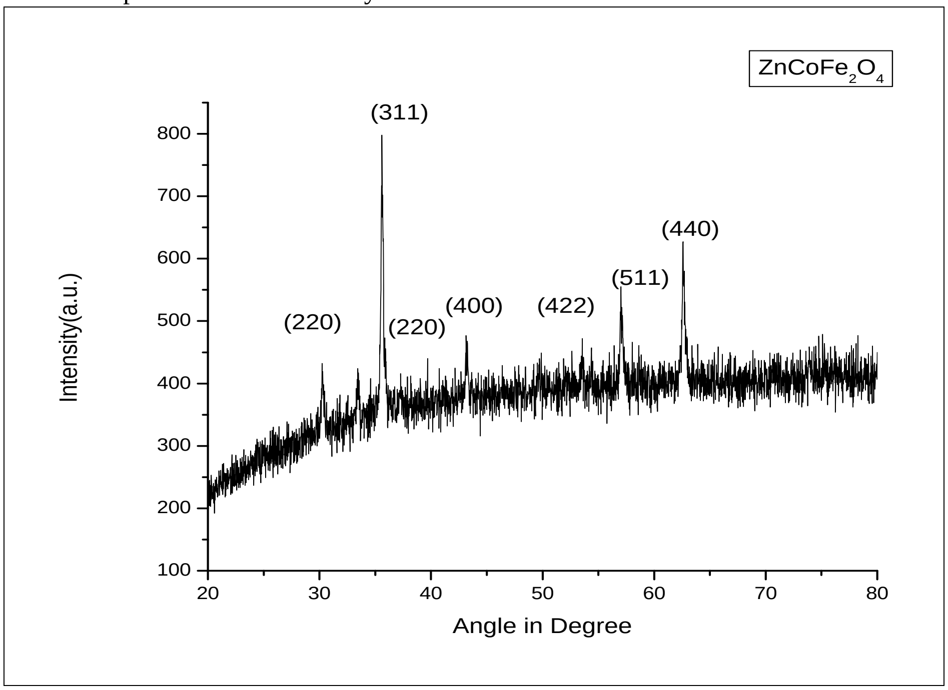

Fig.1: The X-ray diffraction (XRD) patterns for the polycrystalline $Zn_{0.5}Co_{0.5}Fe_2O_4$ ferrites powders as prepared and that was annealed at different temperatures (973K)

3.2. Particle Size Calculation Of $Zn_{0.5}Co_{0.5}Fe_2O_4$ NANOPARTICLES BY SCHERER FORMULA:

The size of the nanoparticle have been determined using Scherer equation from the Full width at Half Maximum (FWHM) value of [311] diffraction peak.

$$D = 0.9\lambda/\beta \cos\theta$$

Where D is the particle size, λ is the X-ray wavelength (1.5418), θ is the Bragg angel and β is the half maximum width.

The size of nanoparticles was obtained as **15.7 nm for 973K** annealing temperature for 4 hours.

3.3. Fourier Transforms Infrared (Ft-Ir) Studies:

IR spectrum is considered an important tool to get information about the structure and the positions of ions in the crystal through the crystal's vibration modes (15).The formation of spinel $Zn_{0.5}Co_{0.5}Fe_2O_4$ structure in the calcined zinc ferrite nanoparticles is further supported by FT-IR spectra shown (Figure 2). The peaks at **551.56 cm^{-1}** correspond to the metal–oxygen (Fe–O) stretching vibrations and it is the characteristic peak of the spinel structure of $Zn_{0.5}Co_{0.5}Fe_2O_4$ nanoparticles. The peak at **3659 cm^{-1}** corresponds to the vibration of O-H and the light band at **1622.7 cm^{-1}** could be attributed to the adsorbed water or humidity. This was further supported by disappearance of these bands at higher temperature. The strong absorption band at **400 cm^{-1}** is described as the stretching modes of Zn–O (16).

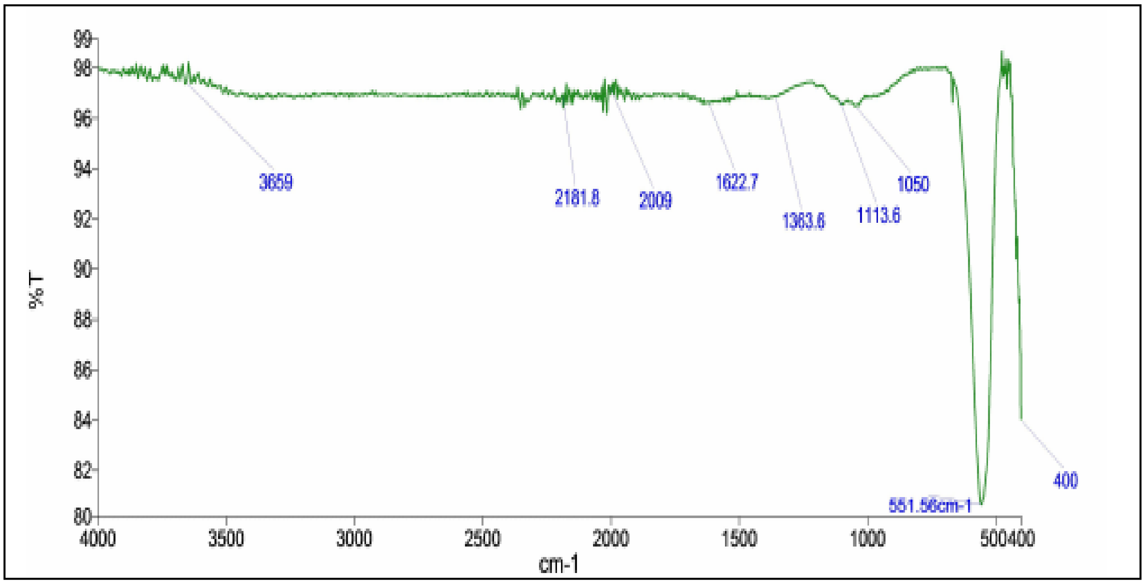

Fig. 2: FTIR spectra of $ZnCoFe_2O_4$ synthesized by using lemon sintered at 973K

Photocatalytic Activity

The photocatalytic activity of synthesized samples under UV light was evaluated by performing experiments on the degradation of tartrazine dye in aqueous solution. Figure 1 illustrates the degradation of this dye by photocatalysis in the presence of $Zn_{0.5}Co_{0.5}Fe_2O_4$ nanoparticles for 18 hour. The results showed that, the concentration of dye solution barely changed after a solution had been directly illuminated. The absorption peaks of dyes became weaker along with the irradiation time and hence azo groups as well as aromatic part of the dyes molecule were destructed under UV light. This indicates the zinc substitution in cobalt-ferrite increase the photocatalytic degradation of dyes. It was found that dye degraded to **100%** by catalyst $Zn_{0.5}Co_{0.5}Fe_2O_4$.

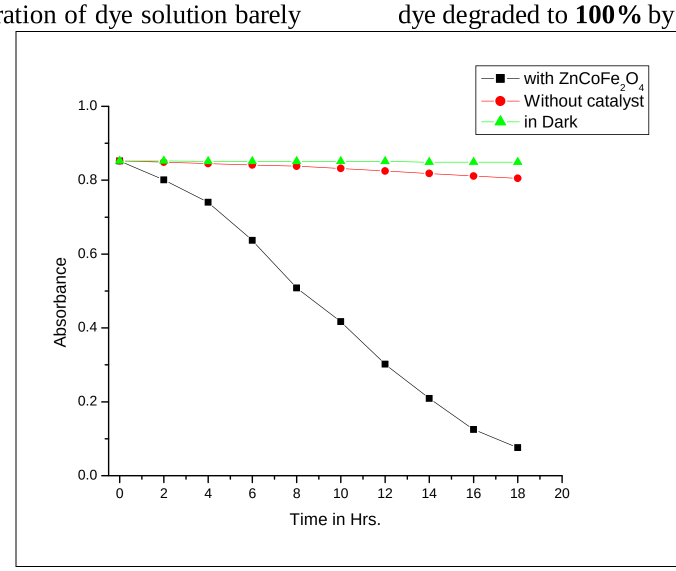

Fig.3:The time varying decrease in absorbance for used photocatalyst is shown.

CONCLUSIONS:

The mixed metal oxide such as $Zn_{0.5}Co_{0.5}Fe_2O_4$ nanoparticle is successfully prepared by using lemon as a binding agent via sol-gel auto combustion method. X-Ray Diffraction (XRD) analysis confirmed that formation of $Zn_{0.5}Co_{0.5}Fe_2O_4$ with cubic spinel structure. The progress of photocatalytic study of dye shows that the facile sol-gel synthesized $Zn_{0.5}Co_{0.5}Fe_2O_4$ is the effective catalysts in decolorisation and degradation of tartrazine dye. This recyclable photocatalyst are reusable as there is no more major change in their structure after photocatalysis.

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