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Synthesis of $MnFe_2O_4$ by co-precipitation method, its characterisation and Photocatalytic study

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Abstract

$MnFe_2O_4$ is prepared by co-precipitation method. In which $MnSO_4.7H_2O$ and $FeSO_4.7H_2O$ are simultaneously precipitated as hydroxides by adding NaOH this on incineration forms $MnFe_2O_4$. This method gives better yield and produces a homogeneous sample. The formula is confirmed by XRD analysis wherein good agreement between observed and standard d values confirms the same. The SEM image confirms particle size to be 0.64 micron. The photocatalytic activity of $MnFe_2O_4$ is studied using Methyl red indicator for degradation. It is found to degrade methyl red to the extent of 50%.

Keywords: Manganese Ferrite, Co-precipitation, XRD, SEM, Methyl red, Photo catalyst.

Introduction

$MnFe_2O_4$ belongs to the spinel class of minerals with formula AB_2O_4, Manganese atoms occupying 8 tetrahedral and 8 octahedral sites, and Ferric atoms occupying 8 octahedral sites[1].

It has a cubic close packed structure. Manganese ferrite is an insulator with a band gap of 0.04-0.06 ev [2].0< i <1 (i= inversion parameter)[2]. $MnFe_2O_4$ is a partially inverse spinel as it has 80% normal and 20% inverse structure. Properties depend on morphology and size, which depend on preparation conditions[1].

It finds applications in various fields including magnetic material, gas sensors, and absorbent material for hot gas. Due to excellent magnetic and electrical properties they are excellent ceramic materials[1].

$MnFe_2O_4$ has many applications ranging from fundamental research to industrial applications. It is found to have excellent chemical stability and mechanical hardness. It has potential scope due to its photo catalytic activity[3]. It can be used in degradation of dyes and therefore in water purification[4].

Synthesis done by co-precipitation results in better homogeneity as compared to other techniques and can be characterised well. It has super paramagnetic property narrow band gap[2]. Thus it has extensive applications ranging from fundamental research to Industries and therefore there is a scope of study for this compound.

Methodology

Synthesis: Co-precipitation method: Methodology: 0.1 M $MnSO_4$, 0.1 M $FeSO_4.7H_2O$, and 0.4 M NaOH was prepared[5]. 100ml $MnSO_4.7H_2O$ was added drop wise using separating funnel into the alkali solution (i.e. NaOH taken in a 500 ml beaker. Immediately 200ml $FeSO_4$ was added to the above solution and magnetically stirred for one hr at room temperature. A precipitate was obtained which was filtered and washed with water and ethanol for 5-10 minutes and oven dried at 100°C. Later it was incinerated wherein the precipitate was heated to 800°C for 2 hour. The dried product was weighed.
Experimental Yield: 1.82 gms.

Theoretical yield was calculated as follows:

Formula	$MnSO_4.7H_2O$, $FeSO_4.7H_2O$	$MnFe_2O_4$
Mol.wt	246.67g+(230.63x2)= 707.93g	230.64g
Amount taken / Expected	2.467g + 5.56g = 8.02g	? x g

x/ Theoretical yield= 2.613g

Percentage yield = Experimental yield/ Theoretical yield x 100
= 1.82/2.613 x100
= 69.65%

X ray Diffraction: The XRD analysis was done at NIO Donapaula. The graph was obtained by plotting Intensity v/s 2θ values.

The d values i.e. interplanar spacing was obtained by using Bragg's law[6].

$n\lambda = 2d\sin\theta$

The d values of peaks from Figure-1 were calculated and Tabulated in Table-1.

Table-1: d values.

2θ	Calculated d values	Std d values
30.06	2.93	3.00
35.42	2.53	2.56
43.04	2.10	2.12
62.54	1.48	1.43

The calculated d values were compared with the std ones as given in JCPDS files[7]. The closeness in values confirmed the sample to be as $MnFe_2O_4$.

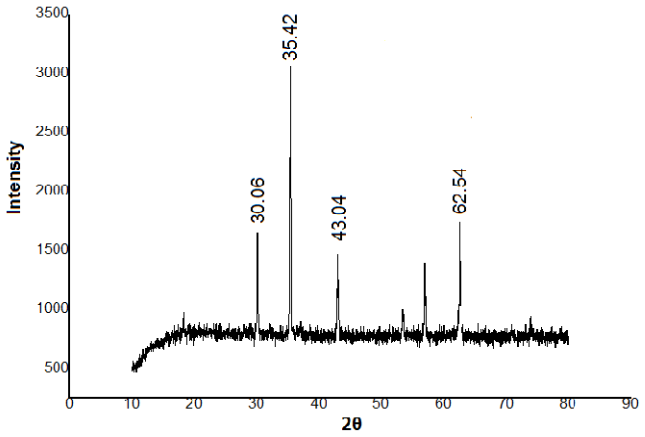

Figure-1: Graph of Intensity v/s 2θ.

Scanning Electron Microscopy: The SEM image of prepared $MnFe_2O_4$ was recorded at Instrumentation centre Goa University on JEOL JSM – 5800LV scanning microscope. The data obtained was used to calculate the particle size.

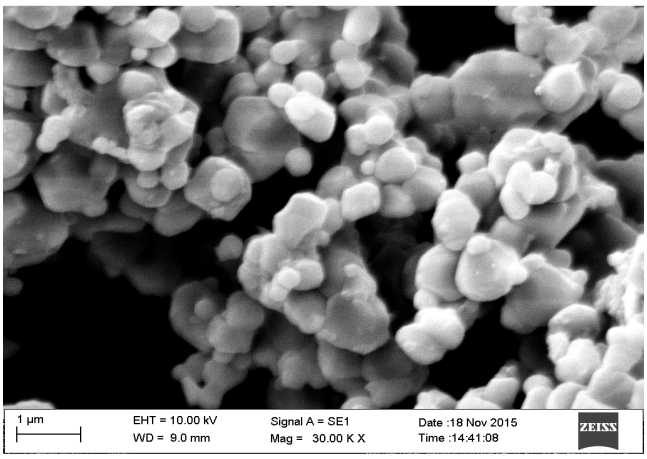

Figure-2: SEM image of $MnFe_2O_4$.

The particle size is found to be 0.64 µm.

Photocatalytic study: Dye degradation is a process in which large dye molecule is broken down chemically into smaller molecules by certain chemical compounds[8]. In Photodegradation this is carried out in presence of light. The rate of this photocatalytic degradation depends on the basic structure of catalyst and the nature of auxiliary group attached to aromatic dye. $MnFe_2O_4$ composites are found to be efficient catalysts for dye degradation, and to understand this Methyl red was selected as dye[4].

Photo degradation using Methyl Red: Procedure: Initially 20 ppm of methyl red was prepared by weighing 0.02 g of methyl red and diluting it to 1000ml with distilled water. 50 ml of this solution was taken in two beakers and 100 ml of distilled water was added. 0.5 g of $MnFe_2O_4$ was added to one beaker and the other was used as blank. The optical density of these solutions were recorded on a uv-visible spectrophotometer at zero time and then they were kept in sunlight and OD was recorded after 30 mins at regular intervals and eight such readings were taken.

Table-2: Observations.

Time in minutes	O.D Blank	O.D Sample	% degradation= C_o-C_t/C_o x100
0	0.12	0.12	0
30	0.12	0.11	83.33
60	0.12	0.10	16.66
90	0.12	0.09	25
120	0.11	0.09	25
150	0.11	0.08	33
180	0.11	0.07	41
210	0.11	0.07	41
240	0.11	0.06	50

Calculations: The percentage degradation was calculated using the formula C_o-C_t/C_o x 100[4].

As time of exposure increases the optical density decreases so we can conclude that $MnFe_2O_4$ shows photocatalytic activity in the degradation of methyl red to 50% as seen in Figure-3.

Results and discussion

$MnFe_2O_4$ synthesized by co-precipitation method was a fine orange powder of appreciable quantity with a satisfactory percentage yield of 69.65%.

The photo catalytic degradation of methyl red when carried out with $MnFe_2O_4$ has a shown a steady decrease in optical density

values when exposed to sunlight and recorded at regular intervals of 30 mins. Therefore we can conclude that $MnFe_2O_4$ is capable of catalysing the degradation of methyl red[8]. The XRD analysis has been carried out, and the experimental d values match well with standard d values as obtained from JCPDS files[7].

The SEM image of $MnFe_2O_4$ shows the formation of well formed crystals with an average particle size of 0.64µm.

Conclusion

The Co-precipitation method is quite suitable for synthesis of $MnFe_2O_4$ producing good yield. The XRD details confirm the Cubic structure which verifies the product to be $MnFe_2O_4$. The SEM image indicates the formation of agglomerates due to magnetic nature. The Photocatalytic study has shown that $MnFe_2O_4$ is able to degrade Methyl red indicator as revealed from the decreasing OD values. Therefore it can used for removal of organic based pollutants likely to be present as contaminants in water.

These dyes are found in industrial waste and are very important from aesthetic point of view and are a visible pollutant and thereby increases aquatic pollution[4]. Methyl red has low biodegradability, hence persists for long time in flowing waters, it affects the rate of photosynthesis inhibits aquatic biota by blocking out sunlight. The Industrial waste which is likely to contain methyl red can be effectively treated with $MnFe_2O_4$ so that it can degrade methyl red and thereby eliminate its toxicity, before releasing waste in water bodies[8].

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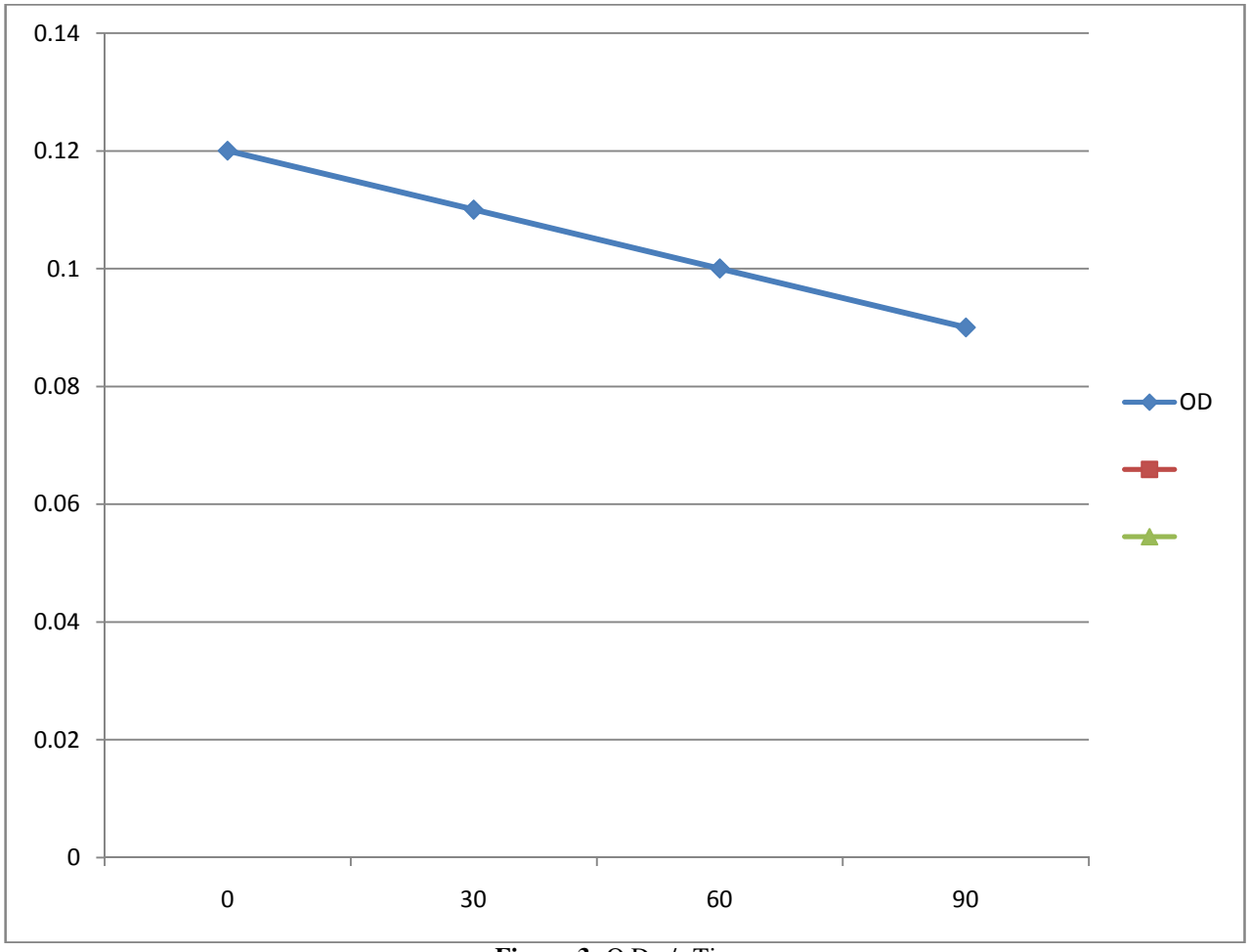

Figure-3: O.D v/s Time.

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