Synthesis And Characterization Of Pure Cs Ferrite And Mixed Cs-Li Ferrite And Their Application In Adsorption Of Dyes

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Abstract: Pure Cs ferrite and mixed Cs-Li ferrite have been synthesized by using solution combustion method. For solution combustion method oxalic dihydrazide has been used as a fuel. Solution combustion method leads to the formation of single-phase ferrite materials. By using this method ferrite materials have been synthesized at lower temperature and in shorter time. The identity of synthesized ferrite materials has been established by using Fourier Transform Infrared Spectroscopy (FTIR). As synthesized ferrite materials have been employed for adsorption of dyes from their aqueous solutions. Ferrite materials have proven to be efficient adsorbents due to their nano-sized particles, reusability and easy removal from the solution owing to their magnetic nature.

Introduction

Since last few decades ferrite materials have gained noteworthy attention due to their amazing properties. Owing to these properties, ferrite materials have found potential applications in various fields. Generally, their applications involve usage in transformers, high frequency devices and quality filters which are attributed to their remarkable electrical and magnetic properties. Moreover, these materials are readily available, stable at higher temperatures and have very low price [1-5]. The properties of these materials are mainly dependent upon their size and composition which in turn depends upon the method used for the synthesis of these materials. Researchers have used various methods for the synthesis of alkali metal ferrites [6-10]. Recently, ferrite materials have also been employed for adsorption of dyes from solutions, which is due to their greater surface area, easy removal after usage and reusability [11-17]. In the present study, pure Cs ferrite and mixed Cs-Li ferrite have been synthesized by using solution combustion method and synthesized ferrite powders have been employed for adsorption of dyes from their aqueous solutions.

Experimental

1. Preparation of oxalic dihydrazide (ODH)

ODH, $(CON_2H_3)_2$, was synthesized by adding one mole of diethyl oxalate into two moles of hydrazine hydrate in the temperature range of 0-4 ^{0}C as the reaction was highly exothermic. Oxalic acid white precipitates were obtained and these were repeatedly washed with distilled water. The product was then dried and stored.

2. Synthesis of pure Cs ferrite and mixed Cs-Li ferrite:

For the synthesis of pure Cs ferrite, CsFeO₂, through solution combustion method, following procedure was adopted:

- a) 1 mole of caesium nitrate was dissolved in minimum quantity of distilled water (Solution A).
- b) 1 mole of ferric nitrate was dissolved in minimum quantity of distilled water (Solution B).
- c) Solution A and B were mixed together.

- d) ODH (2 moles) was added in above solution very slowly and with proper stirring as the reaction is very exothermic.
- e) After complete addition of ODH, viscous solution then obtained was concentrated on water bath.
- f) The concentrate was then subjected to heating in a muffle furnace by gradually increasing the temperature (step by step) up to 600⁰C. Brown coloured product was obtained and this was grinded further by using mortar and pestle to obtain fine powder.

Following reaction was involved in the synthesis of caesium ferrite.

 $Fe(NO_3)_3.9H_2O + CsNO_3 + 2C_2H_6N_4O_2 \rightarrow CsFeO_2 + 4CO_2(g) + 6N_2(g) + 15H_2O$

Mixed Cesium-Lithium ferrite $(Cs_{0.5}Li_{0.5}FeO_2)$ and $(Cs_{0.3}Li_{0.7}FeO_2)$ were synthesized by solution combustion method by following the same steps.

$$\frac{1}{2}$$
 LiNO₃ + Fe(NO₃)₃.9H₂O + 2C₂H₆N₄O₂ + $\frac{1}{2}$ CsNO₃ \rightarrow Cs_{0.5}Li_{0.5}FeO₂ + 4CO₂ (g) + 6N₂ (g) + 15H₂O

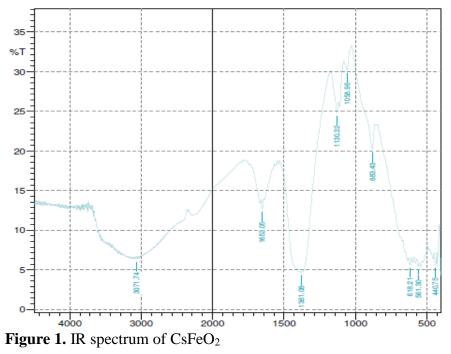
$$0.7LiNO_3 + Fe(NO_3)_3.9H_2O + 2C_2H_6N_4O_2 + 0.3C_8NO_3 \rightarrow Cs_{0.3}Li_{0.7}FeO_2 + 4CO_2 (g) + 6N_2 (g) + 15H_2O$$

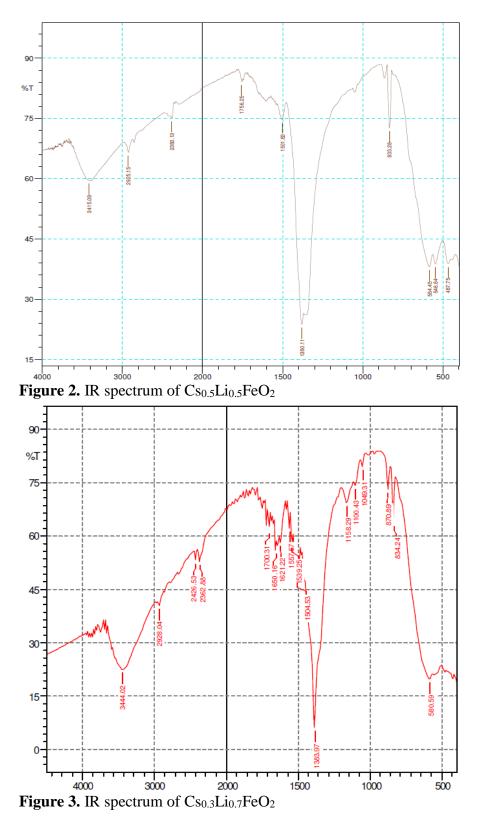
Identity of synthesized ferrite materials was established by recording infrared spectrum with FTIR-8400S spectrometer (Shimadzu), in the range of 4000- 400 cm⁻¹ using KBr pallets as reference.

3. Adsorption of dyes from aqueous solutions

The synthesized ferrite materials in powdered form have been used for adsorption of dyes (crystal violet and methylene blue) from their aqueous solutions and studies have been reported by using Shimadzu UV-Visible spectrophotometer. Adsorption studies have been carried out for varying concentration (ppm) of dye solutions and for change in pH of solution. **Results and Discussion**

Infrared spectra of ferrite materials are shown in Figure 1-3, which show characteristic absorption bands due to Metal-Oxygen bonding in these materials [18-20].





Adsorption of different dyes using ferrite materials and effect of dye concentration: A) Dye: Crystal Violet

λ_{max}: 590 nm

Table:1 Adsorption of crystal violet dye with ferrite materials.

Sr.	Concentration o	f Absorbance	Absorbance	Absorbance
No.	dye (ppm)	(blank)	(with CsFeO ₂)	(with Cs _{0.5} Li _{0.5} FeO ₂)

1	10	1.683	0.083	0.188
2	20	2.845	0.116	0.455
3	50	3.098	2.138	0.857

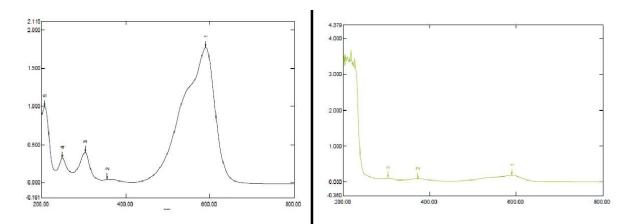


Figure 4. UV-Visible spectrum for adsorption of 10 ppm CV dye with Cs_{0.5}Li_{0.5}FeO₂

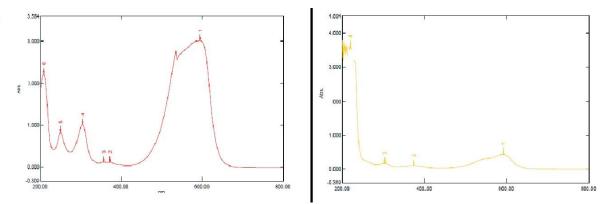


Figure 5. UV-Visible spectrum for adsorption of 20 ppm CV dye with Cs0.5Li0.5FeO2

B) Dye: Methylene Blue

λ_{max}: 665 nm

Table:2 Adsorption of methylene blue dye with ferrite materials.

Sr.	Concentration of	Absorbance	Absorbance	Absorbance
No.	dye (ppm)	(blank)	(with CsFeO ₂)	(with Cs _{0.5} Li _{0.5} FeO ₂)
1	50	3.519	1.497	3.25

Effect of pH on adsorption of crystal violet dye

Table:3 Effect of pH on adsorption of crystal violet dye by ferrite materials

Sr.	pH of	Absorbance	Absorbance	Absorbance
No.	solution	(blank)	(with CsFeO ₂)	(with Cs _{0.5} Li _{0.5} FeO ₂)
1	4	3.059	1.892	1.106
2	5	3.062	1.083	0.852
3	6	3.063	2.511	0.405
4	7	3.088	0.925	0.582
5	8	3.061	0.804	0.326
6	9	3.055	0.378	0.510
7	10	2.591	0.434	0.619

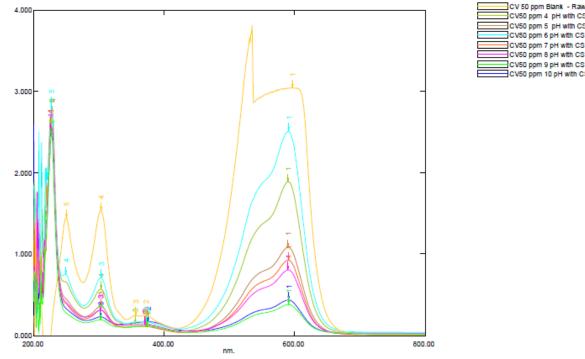


Figure 6. UV-Visible spectrum for adsorption of CV dye at varying pH with CsFeO₂ **Conclusion**

For the synthesis of ferrite materials, solution combustion method has proven to be an efficient technique. In this method, due to stoichiometric mixing and atomic scale mixing of reagents, single-phase nano sized ferrite materials can be synthesized at lower temperature and in shorter time. FT-IR studies have shown the presence of characteristic absorption peaks in ferrite structure due to Metal-Oxygen bonding vibrations. Synthesized ferrite materials have shown the adsorption properties regarding adsorption of crystal violet dye and methylene blue dye from their aqueous solutions. Adsorption properties of these ferrite materials have also shown a change due to change in concentration of dye solutions and pH of the solutions. These ferrite materials are acting as efficient adsorbent materials for dyes, which is attributed to their size, easy removal from solution and reusability.

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