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# Synthesis, structural and magnetic properties of Ni<sup>2+</sup> and In<sup>3+</sup> doped cobalt ferrite and application as catalyst for synthesis of Bis-(Indolyl) methane derivatives

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## Highlights

- The Ni<sup>2+</sup> and In<sup>3+</sup> substituted cobalt ferrites were successfully synthesized by sol–gel auto combustion method.
- The precursors were calcinated at temperature 500°C.
- The EDAX pattern confirmed stoichiometric composition of elements of synthesized ferrite nano particles.
- The XRD pattern confirmed single phase cubic spinel structure.
- Infrared spectroscopy of samples shows major absorption bands around 400cm<sup>-1</sup> and 600cm<sup>-1</sup>.
- The saturation magnetization (M<sub>S</sub>) increases with increase in Ni<sup>2+</sup> and In<sup>3+</sup> composition.

## Abstract

The Ni<sup>2+</sup> and In<sup>3+</sup> substituted cobalt ferrites were prepared having the chemical composition Ni<sub>x</sub>Co<sub>1-x</sub>In<sub>y</sub>Fe<sub>2-y</sub>O<sub>4</sub> (x=0.0, 0.25, 0.5, 0.75, 1.0 and y=0.0, 0.03, 0.06, 0.08, 0.1) by sol–gel auto-combustion method. The precursors were calcinated at temperature 500°C. The EDAX pattern confirmed stoichiometric composition of elements of synthesized ferrite nano particles. The XRD pattern confirms single phase cubic spinel structure. Lattice constants increase as increase in In<sup>3+</sup> ions content. X–ray density increases whereas bulk density decreases. Infrared spectroscopy of synthesized samples shows absorption bands 'ν<sub>2</sub>' around 400cm<sup>-1</sup> and 'ν<sub>1</sub>' at 600cm<sup>-1</sup> are allocated to the intrinsic stretching vibrations of octahedral and tetrahedral complexes respectively. Scanning electron microscopy was applied to study the surface characteristics and uniform distribution of particle size and also prepared samples are porous in nature. Transmission electron microscopy carried out to know the particle size of synthesized ferrite samples and selected area electron diffraction pattern clearly shows that particles were well crystalline in nature. Magnetic measurements of ferrite nanoparticles were done by vibrating sample magnetometer (VSM). Bis-(indolyl) methane derivatives efficiently prepared from Indole and aromatic aldehydes using Ni<sub>x</sub>Co<sub>1-x</sub>In<sub>y</sub>Fe<sub>2-y</sub>O<sub>4</sub> nano particles as catalyst.

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## Introduction

Spinel ferrites materials with a chemical formula, AB<sub>2</sub>O<sub>4</sub> has a commercial significant due to their outstanding electrical and magnetic characteristics [1]. These characteristics make spinel ferrite appropriate for various device applications viz; magneto-optic sensors, anode for batteries, sensors and catalysts, lasers, phosphorescent sources, microwave absorber, pigments etc. These properties of ferrites are highly subtle to the dopant composition and the processing conditions. Processing conditions are highly accountable for the structure crystallinity, crystal size and shape, phase purity etc. [2]. Various synthesis routes can be used to synthesize the spinel ferrite materials including; ceramic [3], co-precipitation [4], citrate precursor [5], sol-gel auto-combustion [6] etc. Herein, the ferrite samples were prepared by the sol-gel method since it is simple and cost-effective method.

A one-pot reaction, MCRs usually produces superior yields and is dissimilar from the two-component reactions in numerous features [7], [8]. These reactions used for synthesis of complex molecules lead structure identification and optimization in chemical biology and drug discovery [9], [10]. Moreover, the application of several conversions in a single operation is highly attuned with the objectives of sustainable and green chemistry [11]. Indole and its derivatives have paying attention a lot interest in recent decades due to their wide biological activities [12], [13]. 3-substituted indole derivatives are general components of drugs and are commonly found to be of pharmaceutical attention in a diversity of therapeutic areas [14]. Among the 3-substituted Indole derivatives, bis(indolyl) alkanes can be believed as a significant class of organic compounds due to their extensive occurrence in numerous natural products having biological activity [15]. Bis(indolyl) methanes are most active compounds for endorsing valuable estrogen metabolism [16], [17]. Bis(indolyl) methanes have shown pharmaceutical activities such as anticancer [18], [19], antiviral and antimicrobial characteristics [20] and are recognized as a promoter of estrogen metabolism [14].

Thus, the preparation of Bis(indolyl) methanes has acknowledged a lot interest since last few years. A modest process for the preparation of bis(indolyl) methanes is the condensation of two equivalents of Indole with the carbonyl aldehydes. A variety of reagents such as Bronsted acids [60], [21], [22], Lewis acids [19], [23], acetic acid [23], In(OTf) [24], InF<sub>3</sub> [25], silica bonded S-sulfonic acid [26], Zeolites [27], ionic liquids [28], have been used to for this transformation.

Most of these catalysts have one or more disadvantages such as longer reaction periods, low yields, severe reaction situations and use of expensive and or toxic catalysts and solvents. Hence, there is an adequate necessity for a mild, clean and efficient route for the preparation of this worthy moiety.

The application of nanoparticles as catalysts in organic transformation had attracted a substantial

attention in this decade. Although application of a nano-catalyst might attain a considerable improvement of its catalytic activity, the significant task for green chemistry is the invention of novel technologies for catalyst separation and recycling to substitute conventional processes [29]. Therefore, ample consideration is given to apply the nanoparticles of magnetic metal oxides as heterogeneous and easily recycled catalysts for numerous organic reactions [30]. Currently, nanoparticles of functionalized magnetite ferrite can be used as an effective catalyst in various chemical reactions such as CO oxidation [31], catalytic combustion of hydrocarbons [32] or selective oxidation and reduction of several organic molecules [33], synthesis of benzimidazoles [34],  $\alpha$ -amino nitriles [35], 1,1-diacetates from aldehydes [36], 1,4-dihydropyridines [37], etc. Considering the significance of multicomponent reaction [61], [38], [39], [40], [41], we used magnetic nanoparticles as a recoverable and reusable catalyst for the preparation of bis-(indolyl) methanes.

In present work, the effect of Ni<sup>2+</sup> and In<sup>3+</sup> substituents on the structural, magnetic properties of cobalt ferrites with composition Ni<sub>x</sub>Co<sub>1-x</sub>In<sub>y</sub>Fe<sub>2-y</sub>O<sub>4</sub> (x=0.0, 0.25, 0.5, 0.75, 1.0 and y=0.0, 0.03, 0.06, 0.08, 0.1) and its catalytic application for the preparation of Bis-(indolyl) methanes have been investigated.

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## Section snippets

### Methods and materials

Nanocrystalline ferrite powders with compositions Ni<sub>x</sub>Co<sub>1-x</sub>In<sub>y</sub>Fe<sub>2-y</sub>O<sub>4</sub> (x=0.0, 0.25, 0.5, 0.75, 1.0 and y=0.0, 0.03, 0.06, 0.08, 0.1) were synthesized by Sol-gel auto-combustion technique [6]. The Analytical Reagent grade of Pure Nickel nitrate [Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O], Cobalt nitrate [Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O], Indium Nitrate [In(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O], Ferric Nitrate [Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O], and Citric acid [C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>·H<sub>2</sub>O] were used as starting materials.

Reaction procedure was carried out in air atmosphere without protection of...

### Thermal analysis

The typical TGA/DSC plot of the sample x=0.50 and y=0.06 is shown in Fig. 1. The TGA plot shows initial weight loss of 11.55% within 135°C–195°C temperature range related to the loss of adsorbed and residual water. The second weight loss in the range of 225–500°C of 34.32% is observed due to decomposition of organic material and solid state reactions to form final ferrites. No weight loss is in the range of 500–700°C was observed, illustrates the complete decomposition of the...

### Conclusions

The Ni<sup>2+</sup> and In<sup>3+</sup> substituted cobalt ferrites were prepared having the chemical composition Ni<sub>x</sub>Co<sub>1-x</sub>.

$x\text{In}_y\text{Fe}_{2-y}\text{O}_4$  ( $x=0.0, 0.25, 0.5, 0.75, 1.0$  and  $y=0.0, 0.03, 0.06, 0.08, 0.1$ ) successfully synthesized by sol-gel auto combustion method. The precursors were calcinated at temperature 500°C. The EDAX pattern confirmed stoichiometric composition of elements of synthesized ferrite nano particles. The XRD pattern confirmed single phase cubic spinel structure. Lattice constants increased from...

## CRediT authorship contribution statement

**K.A. Ganure:** Conceptualization, Investigation, Writing - original draft. **B.L. Shinde:** Methodology, Investigation, Writing - original draft. **U.M. Mandle:** Formal analysis, Investigation, Writing - original draft. **L.A. Dhale:** Formal analysis, Methodology, Writing - original draft. **R.M. Tigote:** Formal analysis, Writing - review & editing. **K.S. Lohar:** Conceptualization, Funding acquisition, Supervision, Project administration....

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper....

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