
Characterization of Supermagnetic Cobalt Ferrite Submicrometer Particles Fabricated Under γ -Irradiation

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Abstract: Magnetic cobalt ferrite has wide spread applications, especially as catalyst for the conversion of alkenes to related aldehydes. Despite several studies found in the literature concerning the fabrication of cobalt ferrite, none has reported on gamma irradiation as a tool for the synthesis of submicrometer sized inverse spinel CoFe_2O_4 . The actual investigation reports on the application of γ -irradiation method for the synthesis of superparamagnetic CoFe_2O_4 , using metal salts precursors and organic reagents. The material fabrication occurs in two main steps as sofar described. The obtained powder was isolated after irradiation and was characterized using X-ray diffraction method, transmission electron microscopy, FT-IR spectroscopy, Raman spectroscopy, UV-visible measurements and vibrating sample magnetometer. Furthermore, the X-ray diffraction data revealed the presence of a reverse spinel structure. The magnetic properties of the fabricated powder exhibited the measured lower coercivity and remanence, demonstrating that the spinel powders are made of superparamagnetic particles and finally, to gain information about the photocatalytic properties of the synthesized material, the room temperature recorded optical measurements for different samples proved that these powder materials may probably exhibit new opportunities which could improve their high photocatalytic efficiency under visible light. The prepared materials could be used as potential candidates for the oxidation of organics compounds.

Keywords: Cobalt Ferrite, Supermagnetic, Nanoparticles, γ -irradiation

1. Introduction

Recently nanoscience and technology is exponentially improving owing to their potential applications. Nano-sized materials made of cobalt ferrite (CoFe_2O_4) could be used both from fundamental research and for various physical, chemical and biological applications. They are used for their thermal, mechanical and chemical stability [1], magnetism and electronics properties [2], sensor design [3], catalysis [4–7], and medicine [8–9].

The ferrites spinel has a cubic close-packed structure consisting of oxygen arrangement, in which cations with small radius value less than 1\AA (M^{2+}) occupies tetrahedral and octahedral sites. This structure is represented by M^{2+}O .

$\text{Fe}^{3+}_2\text{O}_3$. A normal spinel structure is obtained in the presence of metal ion ($\text{M}^{2+} = \text{Zn}^{2+}, \text{Cd}^{2+}, \text{Mn}^{2+}$), which occupies a tetrahedral site while occupation of octahedral sites with divalent of metal ion ($\text{M}^{2+} = \text{Fe}^{2+}, \text{Co}^{2+}, \text{Ni}^{2+}, \text{Cu}^{2+}$) results in an inverse spinel structure.

The potential applications of cobalt ferrite needs to gain a fine control of the morphology and the size of the fabricated material. There is a need to find preparative methods, which allow the synthesis of microstructural materials with specific composition and morphology by controlling physico-chemical parameters.

Several methods such as hydrothermal synthesis [10], sol-gel method [11], coprecipitation [12, 13], thermal decomposition [14], reverse micelle synthesis [15], microemulsion [16], have been reported in the synthesis of

CoFe₂O₄ spinel. All these methods are highly energy-consuming and do not allow a control over the reaction pathways often resulting to agglomerated nanoparticles.

As a piece of information, the irradiation of pure water under γ -ray leads to the formation of different species such as: \bullet HO₂, O₂ and \bullet O₂⁻ and eventually the stable products H₂, O₂ and H₂O₂. The concentrations of the radiolysis products depend on radiation energy absorption rate, the solution pH, and temperature, where (\bullet OH, H₂O₂ and O₂) (\bullet H, e⁻_{aq} and \bullet O₂⁻) are respectively the oxidizing and reducing species, which can readily interact with dissolved transition metal ions (Mⁿ⁺) to change their oxidation states. The condensation and the formation of oxide particles are due to the conversion of dissolved metal species to oxidation states with low solubility. However, to avoid the particles agglomeration the use of an appropriated surfactants is necessary [17, 18].

The fabrication at room temperatures in an aqueous environment under gamma irradiation of nanoparticles made of metal oxides offers some benefits over the conventional methods. The gamma irradiation does not required toxic or hard reagents, and does not necessitating complex purification steps and is able of controlling the particle size and structure. The size of nanoparticles depends of the choice of solvents and stabilizer, the pH during synthesis, and absorbed dose.

We recently reported on γ -irradiation technique as a tool in preparing Fe₂O₃ nano-particles [19–21], zinc sulfide nanocrystalline in a non-aqueous system [22], spherically nanocrystalline sized Co₃O₄ in alkaline medium [23] and on the preparation of non- stoichiometric NiO nanocrystalline [24] et silver nanodispesed metal [25].

The objective of the present investigation is to report on γ -irradiation as a facile route to prepare the submicrometer sized particles made of cobalt ferrite, which have been prepared at room temperature and ambient pressure. The fabricated material was characterized to find out about its chemical composition, structure, optical and magnetic properties.

2. Experimental Procedure

2.1. Sample Preparation

The reagents used in the present investigation for the preparation of the spinel made of CoFe₂O₄ were of high-purity analytical grade and were used as received: cobalt (II)

chloride hexahydrate, [CoCl₂.6 H₂O] as Co²⁺ precursor, iron (III) sulphate [Fe₂(SO₄)₃] as Fe³⁺ source, distilled water, isopropyl alcohol, [(CH₃)₂CHOH], anhydrous sodium hydroxide, [NaOH], polyvinyl alcohol [PVA] and ammonium buffers.

The starting solutions made of inorganic precursor containing both iron (III) and cobalt (II) were prepared by mixing an appropriated amount distilled water and the all were stirred till to obtain a lucid solution.

The procedure for preparing cobalt ferrite composite includes two steps (refer to Table 1):

- (i) The preparation of the sols constituted of a mixture of coprecipitated Fe³⁺ and Co²⁺ ions. The sols of different composition were made by slowly dropping an appropriate amount of high purity pellets of sodium hydroxide into the solutions. Mixed solutions were stirred continuously for 20 min at room temperature until sols were formed. A measured volume of isopropyl alcohol (with a concentration of about 3.0 mol /L) was employed in order to scavenge the oxidative radicals ($HO\bullet$) produced during water radiolysis. A specific amount of organic surfactant, Polyvinyl alcohol (PVA 0.1 mol /L) was added in the solution in order to stabilize growth of particles during the spinel preparation. Anhydrous NaOH pellets were added in to the final solutions in order to obtain a mixture of metallic hydroxides in alkaline medium of pH 10.5 under continuous stirring. Finally, to avoid the irreversible precipitation of parasitic cobalt salts, ammoniacal buffer was added into the final solutions.
- (ii) the transformation of the sols, made of different compositions of FeOOH and Co(OH)₂ under γ - irradiation in the field of ⁶⁰Co γ -source of 325,000 Ci with variable absorbed doses (0.25 kGy h⁻¹).

The particles were precipitated and isolated by centrifugation after irradiation. To remove the by-products, these particles were washed several times with distilled water, absolute alcohol, and acetone, and finally they were dried in electric oven at 60°C for six hours. The black product constituted of pure cobalt ferrite was obtained and characterized. The preparation of the solutions of Fe₂CoO₄ is detailed in Table 1.

Table 1. Preparation of cobalt iron oxide, CoFe₂O₄.

Sample number	[Co ²⁺] mol L ⁻¹	[Fe ³⁺] mol L ⁻¹	[IPA] mol L ⁻¹	[PVA] mol L ⁻¹	[NaOH] mol L ⁻¹	[NH ₃ .H ₂ O] mol L ⁻¹	[(NH ₄) ₂ .SO ₄] mol L ⁻¹	Irrad. dose (kGy)
1	0.04	0.08	3.0	0.1	1.0	0.4	0.1	43
2	0.04	0.08	3.0	0.1	1.0	0.4	0.1	11
3	0.08	0.08	3.0	0.1	1.0	0.4	0.1	30
4	0.08	0.08	3.0	0.1	1.0	0.4	0.1	11
5	0.04	0.08	3.0	0.1	1.0	0.4	0.1	30

2.2. Techniques of Characterization

The prepared powders products (before and after γ -

irradiation) were identified by X-ray powder diffraction (XRD) patterns, employing a D/MAX-2550 X-ray diffractometer with Cu-K α radiation ($\lambda=1.54056$ Å) with a

nickel filter (Rigaku Co., Japan). The identification of the characteristic functional groups in the synthesized material was made using a FT-IR spectrophotometer (SHIMADZU model). A field emission scanning electron microscopy (FE-SEM; JEOL JSM-6700F) well equipped with an energy dispersive X-ray (EDAX) spectrophotometer was used to find out about the surface morphology, the size of particles and the elemental compositions of prepared powder. The morphology and the particles size were determined by transmission electron microscopy with an accelerating voltage of 200 kV. Magnetic measurements were made at room temperature using a vibrating sample magnetometer. The sample material was performed through Raman measurements, which were carried out at room temperature to find out about the compositional and the structure of the fabricated material.

3. Results and Discussion

3.1. XRD Studies

The X-ray diffraction technique (diffractograms recorded in the 2θ range from 15 to 80°) was used to identify the phase composition of the fabricated power and the patterns from samples 1 and 5 were similar (refer respectively to Figure 1(a) and 1(b)). The crystalline nature of the prepared products is proven via the sharpness of diffractogram peaks. For sample 3 (The XRD pattern not shown here), a little amount of CoO could be identified as a secondary phase and at low irradiation dose (about 11 kGy), the Xray spectrum (not shown) mainly indicated the presence of cobalt ferrite and certain unidentified phases peaks.

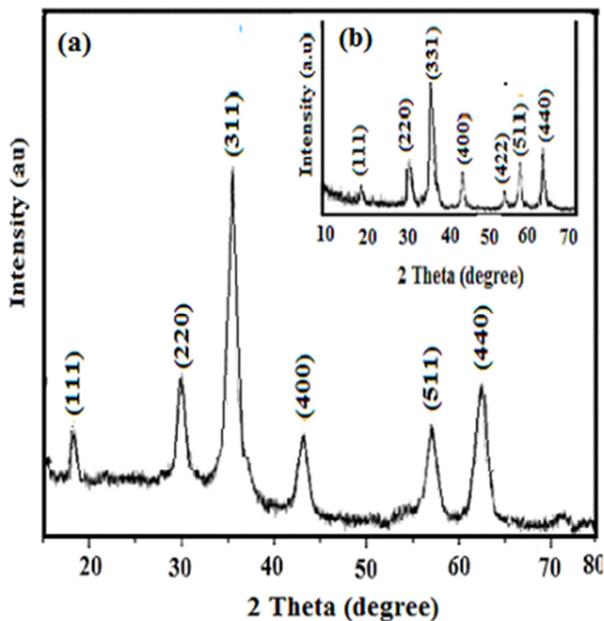


Figure 1. X-ray diffraction patterns of CoFe_2O_4 (sample 1), and the insert shows the sample 5 pattern.

All observed peaks were indexed to the pure face centered cubic CoFe_2O_4 with inverse spinel structure (JCPDS N° 22-

1086) [26-28]. No other phases (or impurities) were observed, indicating the purity of the fabricated product. The location of these peaks matched well with the structure of an inverse spinel (TR_2O_4), where T is the tetrahedral site and R the octahedral site. In the spinel, the full Co^{2+} ions and one half of Fe^{3+} ions are located in the octahedral site, while the other half of Fe^{3+} ions are located in the octahedral sites.

3.2. TEM Images

The TEM images of the fabricated spinel made of CoFe_2O_4 are exhibited in Figure 2. One can observe that the irradiation dose plays an important role in the morphology of the prepared spinels. From Figure 2 (b, c), one can observed that the sample is mainly consisted of snowball-spindle micrometer particles morphology. Some mesoporous structures were observed and could enhance the photocatalytic performance of the fabricated material. The particle get bigger when the absorbed dose increases.

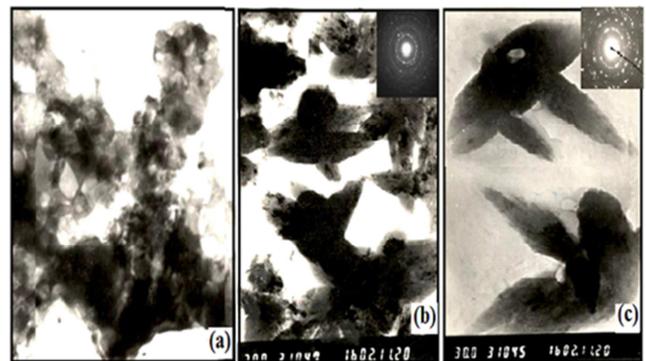


Figure 2. TEM micrographs (with insert, the selected area electron diffraction) of: (a) sols made of coprecipitated Fe^{3+} and Co^{2+} ions (pH=12) before γ -irradiation; (b) Sample 5 of CoFe_2O_4 (30 kGy); (c) Sample 1 of CoFe_2O_4 (43 kGy).

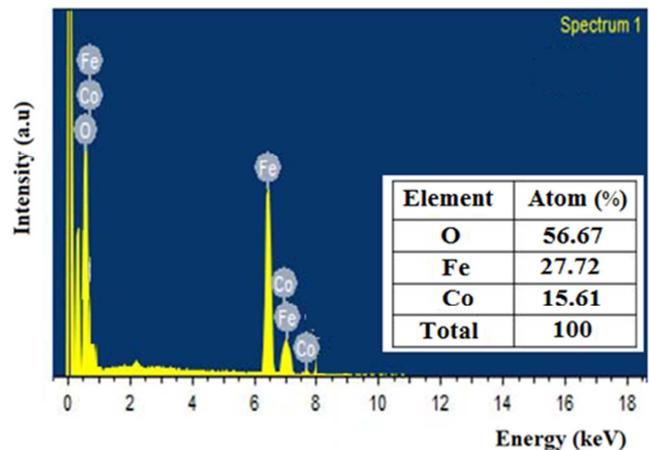


Figure 3. EDS spectrum of prepared CoFe_2O_4 (sample 5).

3.3. Energy Dispersive Studies (EDS)

The EDS analysis of the prepared sample 1 CoFe_2O_4 sample as presented in the spectrum of Figure 3. Based on the energies appeared in the presented spectrum, it can be concluded that the mainly chemical composition of the

prepared powder was very close to the planned one. The spectrum shows the presence of Co, Fe, and O elements. The experimental atomic percentages of Co, O and Fe are found to be 15.61%, 56.67%, and 27.72% respectively. The EDS spectrum showed strong signals of cobalt and iron in fabricated sample. No additional peaks were observed, which confirmed that the cobalt ferrite synthesized by γ -ray irradiation was pure. The EDS spectrum supports others characterization techniques. The calculated atomic ratio of Co: Fe: O is consistent with the real composition of cobalt ferrite.

3.4. Analysis by FT- IR Spectroscopy

In order to ascertain the purity and nature of metal the prepared spinels, the FT- IR spectroscopy investigation was carried out in the region from 4000 to 400 cm^{-1} . The recorded information is shown in Figure 4.

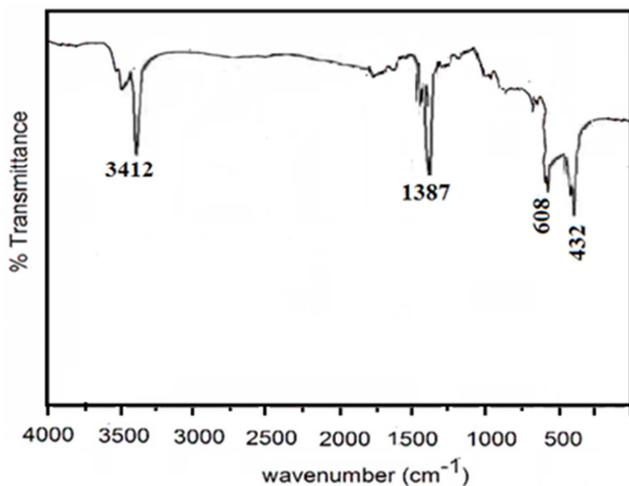


Figure 4. The FT-infra red spectra of the fabricated CoFe_2O_4 (sample 5).

The band located at 3412cm^{-1} is attributed to OH stretching of the capping agent, polyvinyl alcohol used for the preparation of CoFe_2O_4 spinel. The positioned band at 1387cm^{-1} can be assigned to the binding vibrations of water molecules absorbed on the synthesized CoFe_2O_4 surface. The peaks positioned at 608 and 432cm^{-1} are due to stretching vibration of Metal-Oxygen bond in octahedral and tetrahedral sites [29, 30]. The observed absorption band located at 432cm^{-1} could be associated to the tetrahedral cation ($\text{Fe}^{3+} - \text{O}^{2-}$, in $\alpha\text{-Fe}_2\text{O}_3$) vibration mode of prepared submicrometer spinel particles [31, 32].

3.5. Raman Phonon Modes Analysis

To gain information about the vibrational energy states within the synthesized sample, we used Raman spectroscopy. Measurements were made at room temperature (Figure 5). From, this figure, one can clearly see that all the peaks characteristic of cobalt spinel ferrites appear in the spectra, which confirms the formation of cubic cobalt ferrite [33-36].

The prepared sample displays phonon modes ($A_{1g} + E_g + 3T_{2g}$) [36, 37]. The Raman active modes respectively located

at about 292, 460, 600 and 680cm^{-1} are attributed to the phonon vibration modes E_g , $T_{2g}(2)$, $A_{1g}(2)$ and $A_{1g}(1)$, which are well matching with the one of the spinel cobalt ferrite (CoFe_2O_4) particles as reported in the literature [38].

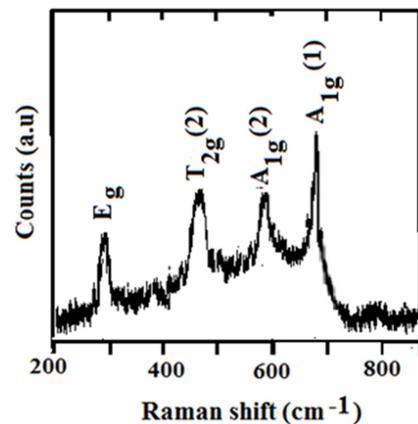


Figure 5. Raman spectra of CoFe_2O_4 (sample 5).

3.6. UV-Visible Measurements

To gain information about the photocatalytic properties of the fabricated material, the optical measurements for different samples were recorded at room temperature using a UV-Visible absorption spectrophotometer as shown in Figure 6.

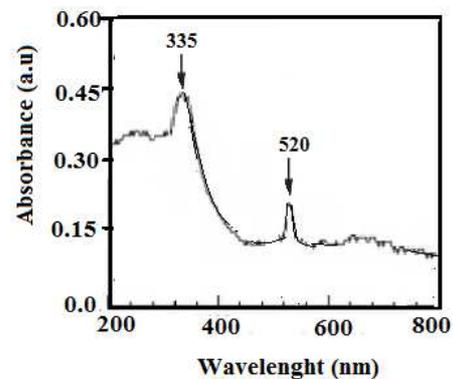


Figure 6. UV-Visible spectrum of CoFe_2O_4 (sample 5).

The spectrum exhibits two absorption bands at 335 and 520 nm, where the first one could be assigned to the O^{2-} to Co^{2+} charge transfer process and the second band could be related to the O^{2-} to Fe^{3+} charge transfer [39]. These two characteristic bands are expected for the spinel made of CoFe_2O_4 . From the spectrum, one can see that the fabricated samples exhibited an absorption from UV to visible light region, which demonstrates the possibility of these materials as catalysts under visible light.

3.7. Magnetic Properties

The synthesized spinel magnetic properties (sample of 30kGy gamma irradiated dose) at room temperature (300 K) is shown in figure 7, where the magnetization (M) as a function of the applied field (H) is presented.

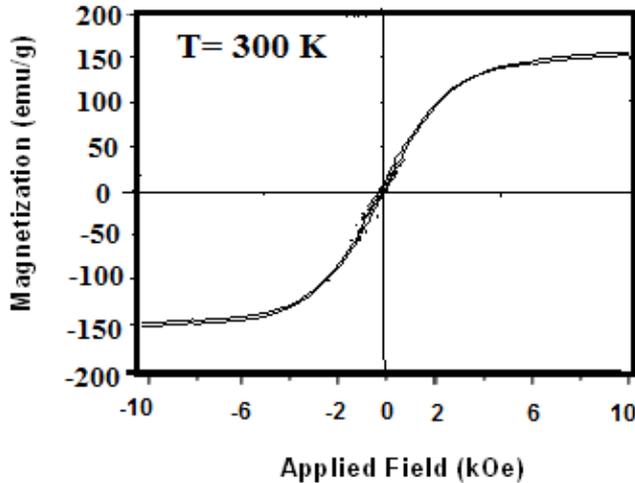


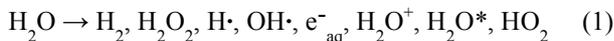
Figure 7. Magnetic properties of magnetite CoFe_2O_4 (sample 5).

The remanence of the fabricated spinel could be extrapolated to 150 emu g^{-1} at 10 kOe . The saturation magnetization is decreased, which is due mostly to the existence of surfactants on the surface of the spinel constituted of CoFe_2O_4 . The measured lower coercivity and remanence are demonstrated that the spinel powders are made of superparamagnetic particles [40].

The superparamagnetic properties observed in the present investigation could be due to the special morphology of the prepared CoFe_2O_4 sample [41]. The gamma irradiation could be expected as a new route for preparing other metal oxides semiconductor.

3.8. Radiolytic Formation of CoFe_2O_4 Mechanism

To illustrate the production of CoFe_2O_4 under γ -irradiation process, the following mechanisms could be suggested. The radiolysis of water leads to the production of free radicals such as: e^-_{aq} , $\text{H}\cdot$, $\text{OH}\cdot$ and HO_2 or O_2^- and molecular species such as H_2 and H_2O_2 . It is well known that, the hydrated electron, e^-_{aq} and hydrogen radical $\text{H}\cdot$ are reducing species, and their standard electrode potentials at 25°C are respectively -2.77V and -2.31V while $\text{OH}\cdot$, HO_2 , O_2^- , H_2O_2 are oxidizing species [42, 43].

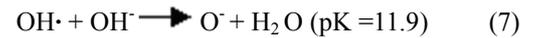
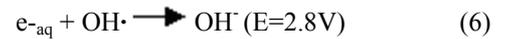
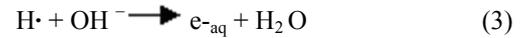


The presence of Co^{2+} species [$\text{Co}(\text{OH})_2$] and Fe^{2+} species [$\text{Fe}(\text{OH})_2$] are required in the production of the spinel (CoFe_2O_4) upon γ -irradiation, the Fe^{2+} adsorbed on [$\text{Co}(\text{OH})_2$] will be rapidly oxidized to FeOOH by the oxidizing species from water radiolysis, such as $\cdot\text{OH}$ and H_2O_2 , through reactions (2) [44].



In alkaline media, the reducing species $\text{H}\cdot$ are converted to e^-_{aq} , which mainly play the major role in the course of reaction. The 2-propanol ($(\text{CH}_3)_2\text{CHOH}$) scavenges both reducing and oxidizing species ($\text{H}\cdot$ and $\text{OH}\cdot$) but under the given condition, this alcohol is inert in reacting with the

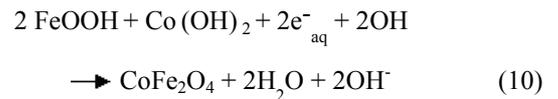
hydrated electron e^-_{aq} [45]. The hydrated electron e^-_{aq} reacts rapidly with the hydroxyl radical $\text{OH}\cdot$, leading to the deprotonation of $\text{OH}\cdot$ radical, which is converted to O^- via the following reactions [46, 47]:



Then the reactions of O^- species with the reducing species such $\text{H}\cdot$ and e^-_{aq} could produce of OH^- according to the reactions:



Under alkaline condition, the formation of the spinel cobalt ferrite, CoFe_2O_4 could be due to ferric oxyhydroxide, $\text{FeO}(\text{OH})$ reduction by hydrated electron (produced upon gamma irradiation), in the presence of stable cobalt ion, Co^{2+} species.



4. Conclusion

In the present investigation, gamma irradiation technology has been applied at room temperature, ambient pressure to prepare submicrometer CoFe_2O_4 particles. This spinel was fabricated without any kind of catalysts in water system. All characterization methods employed to get information about the structure, the composition, the purity and the nature of the prepared powder showed that, the fabricated sample was made of highly pure cobalt ferrite, which is exhibiting photo absorption from ultraviolet domain to visible light region, which implies the possibility for the prepared material to be efficiently used as photocatalyst under visible light.

The gamma irradiation could be expected as a new route for preparing other metal oxides semiconductor.

The degradation of textile dyes and the oxidation of alkenes (using the prepared material as catalyst) are under investigation and the results will be submitted soon.

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