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Structural characteristics of Mg substituted Sol-Gel synthesized Li-Zn magnetic nanomaterial

[1*]Birajdar DD

[1]B.S.S. Arts,Science and Commerce college Makani, Osmanabad (M.S.), India
Email: damodar_birajdar@yahoo.co.in | Phone no.: +9102475250913 | FAX: +9102475252020

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ABSTRACT

Magnetic nanoparticles of $Li_{0.1}Zn_{0.7}Mg_xFe_{2.2-x}O_4$ prepared through sol-gel synthesis method. LiNO3,Zn(NO3)2.6H2O and Fe(NO3)3.9H2O were used to incorporate metal ions needed. Magnesium nitrate was used as a source of Mg^{2+}ions. Thermal decomposition process carried out with TGA/DTA.XRD confirms formation of single phase Li-Zn ferrites. The size of particles increases gradually with increase in Mg concentration.

Key words: Sol-gel synthesis, TGA/DTA, XRD

INTRODUCTION

Synthesis and characterization of magnetic nanoparticles have attracted increasing attention in the past decade. Nanoparticles exhibit unusual physical and chemical properties very different to bulk materials, due to their extremely small size or large surface area [1–3]. Li-Zn and substituted Li-Zn ferrites have been found to be excellent materials in permanent magnets, high-density recording media, absorbers and microwave devices due to their low costs, high resistivity and low eddy current loss[4–6].There are different synthesis methods to prepare Li-Zn ferrites[7] There are many synthesis methods to prepare ferrites as reported in the literature including aqueous colloidal precipitation [8], sol–gel method [9], high-temperature solid-state method [10] and hydrothermal method [11]. Sol–gel approaches have attracted much attention recently [12]. In the present work, we focused on the synthesis of magnesium-substituted Li-Zn ferrites

powder by a sol–gel method at different temperature. The formation of the substituted Li-Zn ferrites phase has been analysed using X-ray diffraction (XRD).

METHODOLOGY

In order to achieve a complete reaction within short time and at the lowest possible temperatures, composition of component cations at an atomic scale is necessary. Sol-gel auto-combustion method is a novel method of preparing nano-materials [13]. The precursor is not calcined at high temperature; thus, it can save energy and avoid agglomeration during the calcination at high temperature. So, sol-gel is an ideal method and has a great potential for commercial process. Nanocrystalline powder of $Li_{0.1}Zn_{0.7}MgxFe_{2.2-x}O_4$ were prepared by sol–gel auto-combustion method. A.R. grade citric acid (C6H8O7) • H2O, LiNO3, Zn (NO3)2.6H2O and Fe(NO3)3.9H2O were used to incorporate metal ions needed. Magnesium nitrate was used as a source of Mg^{2+}ions were used as starting materials. Reaction procedure was carried out in air atmosphere without protection of inert gases. The molar ratio of metal nitrates to citric acid was taken as 1:3. The metal nitrates were dissolved in a minimum amount of double distilled water to get a clear solution and ammonia solution was slowly added to maintain the pH. The mixed solution was kept on to a hot plate with continuous stirring. During evaporation, the solution became viscous and finally formed a very viscous brown gel. When finally, all water molecules were removed from the mixture, the viscous gel began frothing. After few minutes, the gel automatically ignited and burnt with glowing flints. The decomposition reaction would not stop before the whole citrate complex was consumed. The auto-combustion was completed within a minute, yielding the brown-colored ashes termed as a precursor. The as prepared powder then annealed at 600 ^{0}C for 4 h.

RESULTS AND DISCUSSIONS

Structural properties:

The experimental observation showed that the nitrate–citrate gels with all three molar ratios of metal nitrates to citric acid exhibited self-propagating combustion behavior. When the dried gels were ignited at any point, the combustion rapidly propagated forward until all gels were completely burnt out to form powders. It was also observed that the combustion rate is associated with the ratio of nitrates of citric acid. The autocatalytic nature of the combustion process of gels has been studied by thermal analysis (DTA and TGA) of the dried gels. The TGA of the mid sample x = 0.3 decomposing in air atmosphere in the temperature range 20 to 800C with heating rate of 10 ^{0}C/min is shown in Fig. 1.

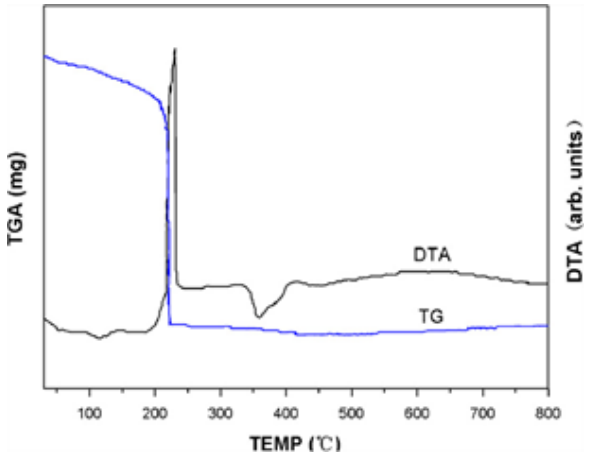

Fig. 1: TG-DTA curves of sample

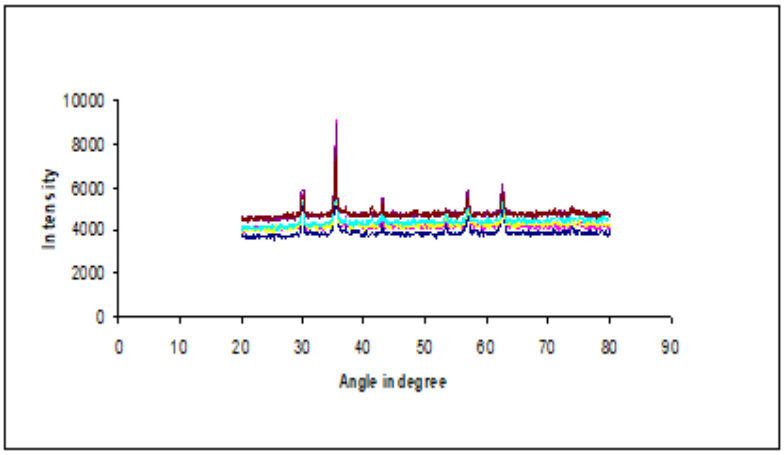

Fig. 2: X-ray diffraction patterns of sample annealed at different temperature

The observed weight loss below 350^{0}C of x = 0.3 sample for Mg is attributed to the loss of physically or chemically absorbed OH groups. The weight loss around 350 ^{0}C, which is ascribed to the vaporization of absorbed water. The third wet loss around 350 to 600^{0}C, which is associated with the residual organic matter including citric acid.

The weight loss below 600 ^{0}C is due to loss of absorbed water and the decomposition of organic derivatives. Final weight loss above 600 ^{0}C, due to weight loss of as received powder. It indicates that the nonreacted metal nitrate is oxidized in this step. Using experimental data of ferritization temperatures, the oxalate complexes were decomposed at their respective ferritization temperatures. The end-products after decomposition were identified as single spinel phase Li–Mg–Zn ferrite, from the analysis of their recorded XRD patterns (Fig. 2). This proves the simultaneous completion of decomposition process of oxalate complex and ferritization.

The samples were powdered for X-ray investigations. Part of the powder was X-ray examined by Phillips X-ray diffractometer (Model 3710) using Cu-K$_\alpha$ radiation (λ=1.5405Å). The scanning step was 2^0/min and scanning range was 20-80°. The X-ray generator was operated at 40 kV and 30 mA. A specially processed Si powder sample was used for instrumental standard. The (1 1 1) reflection of Si at around 28.25^0 indicates that the instrumental broadening is very small (0.25Å).Phase identification of the as-prepared powder was carried out using XRD patterns (Fig. 2). All peaks could be indexed to the standard patterns reported inASTM cardsfor single-phase cubic spinel $Li_{0.1}Zn_{0.7}Mg_xFe_{2.2-x}O_4$. No characteristic peaks of impurities are detected in the patterns. The main diffraction planes are (220), (311), (222), (400), (422), (511) and (440),with maximum diffraction intensity from (311) plane. For nanocrystalline materials, the size of primary nanoparticles can be estimated by the amount by which the X-ray line is broadened.The average diameter (D) of the ferrite crystal was calculated from the XRD line broadening of the (311) XRD-peaks by using Scherrer'sequation ,$D_{hkl} = 0.9\lambda/B\cos\theta$[14]where D_{hkl}, λ, B, and θ are the volume-averaged particle diameter, X-ray wavelength, full width at half maximum (FWHM), and diffraction angle, respectively.

CONCLUSION

In summary, we had successfully prepared magnesium-substituted Li-Zn ferrites by a sol–gel method and annealed at different temperature. XRD analysis reveals that $Li_{0.1}Zn_{0.7}Mg_xFe_{2.2-x}O_4$ crystallizes in a spinel-type cubic structure and their average crystallite size is about 50–90nm. The calcination temperature effected the crystal-lite sizes. Thus the fine control of crystal growth by varying the calcination temperature can be exploited for the production of fine magnetic powder of various sizes for a variety of practical applications.

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