

Mössbauer and magnetic studies of nanocrystalline zinc ferrites synthesized by microwave combustion method

Mohamed Mahmoud^{1,5} · Azza Mohamed Hassan² ·
Mamdouh Abdel aal Ahmed³ · Kaixin Zhu⁴ ·
Ayyakannu Sundaram Ganeshraja⁴ · Junhu Wang⁴

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Abstract Zinc ferrite nano-crystals were synthesized by a microwave assisted combustion route with varying the urea to metal nitrates (U/N) molar ratio. The process takes only a few minutes to obtain Zinc ferrite powders. The Effect of U/N ratio on the obtained phases, particle size, magnetization and structural properties has been investigated. The specimens were characterized by XRD, Mössbauer and VSM techniques. The sample prepared with urea/metal nitrate ratio of 1/1 was a poorly crystalline phase with very small crystallite size. A second phase is also detected in the sample. The crystallite size increases while the second phase decrease with increasing the urea ratio. The saturation magnetization and coercivity of the as prepared nano-particles changed with the change of the U/N ratio. The powder with the highest U/N ratio showed the presence of an unusually high saturation magnetization of 16 emu/g at room temperature. The crystallinity of the as prepared powder was developed by annealing the samples at 700 °C and 900 °C. Both the saturation magnetization (M_s) and the remnant magnetization (M_r) were found to be highly dependent upon the annealing temperature. Mössbauer studies show magnetic ordering in the powder even at room temperature.

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✉ Mamdouh Abdel aal Ahmed
mamdouh.2000.2000@yahoo.com
Junhu Wang
Wangjh@dicp.ac.cn

¹ Department of Physics, Assiut University, Assiut, Egypt 71516

² Physics Department, Faculty of Sciences, Assiut University, Assiut, Egypt

³ Physics Department, Faculty of Science, Al Azhar University, 11884, Nasr City, Cairo, Egypt

⁴ Mössbauer Effect Data Center & Laboratory of Catalysts and New Materials for Aerospace, Dalian Institute of Chemical Physics, Chinese Academy Sciences, Dalian 116023, China

⁵ Present address: Physics Department, Faculty of Science & Arts, Al Jouf University, Al Guroyat, Saudi Arabia

The Mössbauer and the magnetic parameters of this fraction are different from the standard values for bulk zinc ferrite.

Keywords Spinel · Microwave combustion method · Mossbauer · VSM · XRD · Zinc ferrite

1 Introduction

Nano-sized zinc ferrites have been studied extensively due to their exciting chemical and physical properties. These properties make them important materials in several technological applications such as in gas sensors [1], magnetic materials [2–4], catalyst [5, 6] and photo-catalyst [7]. Bulk zinc ferrite exhibits normal spinel structure it behaves like an antiferromagnet below the Néel temperature of 10 K and as a paramagnet at room temperature [8]. Because Zn^{2+} ions (non magnetic) have strong preference to occupy tetrahedral (A-sites) and all Fe^{3+} ions reside on octahedral (B-sites). As a result the predominant magnetic interaction in the $ZnFe_2O_4$ with normal spinel structure is the negative super exchange interaction among Fe^{3+} ions in the octahedral sites; this interaction is weak, leading to antiferromagnetism with very low Néel temperature [9]. Many studies, however, reported that nano-sized zinc ferrites show ferrimagnetic order even at room temperature [10, 11]. This magnetic ordering is due to the redistribution of cations between both sites (A,B) where some of Zn^{2+} ions are transferred from A to B site and that of Fe^{3+} ions from B-site to A-site producing partially inverted spinel structure. The presence of Fe^{3+} ions in the A-site switched on the strong A–B interaction. As a result all magnetic spins at A-site align in one direction and those at B-site in opposite direction. According to the Neel's two sublattice model of ferrimagnetism, the magnetic moment per formula unit in μ_B is expressed by the difference between the magnetic moments of A and B sublattices [12]. It is well known that the physical and chemical properties of zinc ferrites depend on the synthesis route and the produced crystallite size as reported previously in many studies. Several methods have been used to synthesize nano-sized $ZnFe_2O_4$ such as reverse micelle technique [13], coprecipitation [14–16] and ball milling [17–19]. Recently Microwave assisted combustion method has attracted the interest of synthesis nano ferrites as a simple, fast and low cost process [20–23]. The present work aims to clarify the crystal structure and magnetic properties of nano-crystalline zinc ferrite prepared by microwave combustion method using respectively, X-ray powder diffraction (XRD) and Mössbauer spectroscopy as well as Vibrating Sample Magnetometer (VSM) technique.

2 Experimental

2.1 Materials and methods

Zinc ferrite powders were synthesized by microwave combustion method. The detailed process can be described as follows: The analytical grade $Fe(NO_3)_3 \cdot 9H_2O$, $Zn(NO_3)_2 \cdot 4H_2O$ and $(CO(NH_2)_2)$ were used as raw materials. Stoichiometric amounts of metals nitrates and urea as a fuel were dissolved in a minimum quantity of distilled water. The product powders produced at different urea to metal nitrates (U/N) molar ratios, varying from 1.0 to 2.0. These molar ratios of the mixture constituting for various samples are given in Table 1. The solution was introduced into a microwave oven operating at a maximum power of 800 W for 20 min. The solution boils and undergoes dehydration followed by decomposition with the evolution of large amount of gases. After the solution reaches the point of spontaneous

Table 1 Structural parameters of the Zinc Ferrite samples

Sample code	Sample name	(U/N) molar ratios	Apparent crystalline size D_{XRD} (nm)	Lattice parameter a_0 (nm)
(a)	ZnFe1U	(1/1)	Poor crystallite~6 nm	0.845
(b)	ZnFe1.5U	(1.5/ 1)	33	0.8448
(c)	ZnFe1.7U	(1.7/1)	33	0.8443
(d)	ZnFe2U	(2/1)	34	0.8440

combustion, it begins burning and releases lots of heat, vaporizes all the solution instantly and becomes a solid powder. The prepared sample at U/N ratio of 1.7/1 was annealed at 700 °C and 900 °C for 3 h.

2.2 Characterizations and measurements

Phase identification of the samples was performed using X-ray diffractometer equipped with an automatic divergent slit (XRD; Philips PW1700 diffractometer, Netherlands). Diffraction patterns were obtained using $CuK\alpha$ radiation ($\lambda = 0.15418$ nm) and a graphite monochromator in the 2θ range from 15° to 70°. The patterns were obtained using pseudo Lorentzian line shapes to account for symmetry of the peaks and for accurate determination of lattice parameters and apparent crystallite size. The Mössbauer spectra of the samples were recorded with a time mode spectrometer at room temperature. The source was $^{57}Co/Rh$. The Magnetic measurements were carried out at room temperature using vibrating sample magnetometer (VSM) Lakeshore Model 7410 equipped with 2 T magnet.

3 Results and discussions

3.1 XRD

The structural properties of the as prepared samples using different U/N ratios were studied by X-ray diffraction. As shown in Fig. 1, the sample prepared at $U/N = 1$ shows low crystallinity, and contains the main peaks of zinc ferrite and impurity peaks attributed to ZnO and α -Fe₂O₃. As the U/N ratio increases the intensity of the impurity peaks decreases. The XRD peaks of α -Fe₂O₃ impurity phase disappear for the samples at U/N ratios greater than one, while ZnO phase is minimum at $U/N = 1.5$. Figure 2 represents the XRD patterns of samples prepared at $U/N = 1.7$ and then annealed to 700 °C and 900 °C for 3 h. It shows that the crystallinity increases with temperature. Peaks of impurity phase decreases with temperature till disappear totally at 900 °C. The peaks width reveals that all the obtained samples are in the nanoscale level. The crystallite sizes of the prepared and annealed samples have been estimated using Scherrer equation [23]:

$$D = \frac{k \cdot \lambda}{\beta \cos \theta}$$

Where D is the crystallite size, β is the observed angular width at half height of the most intensive peak and calculated from:

$$\beta^2 = \beta_s^2 - \beta_o^2$$

Fig. 1 XRD patterns of as prepared $ZnFe_2O_4$ samples at different U/N ratios

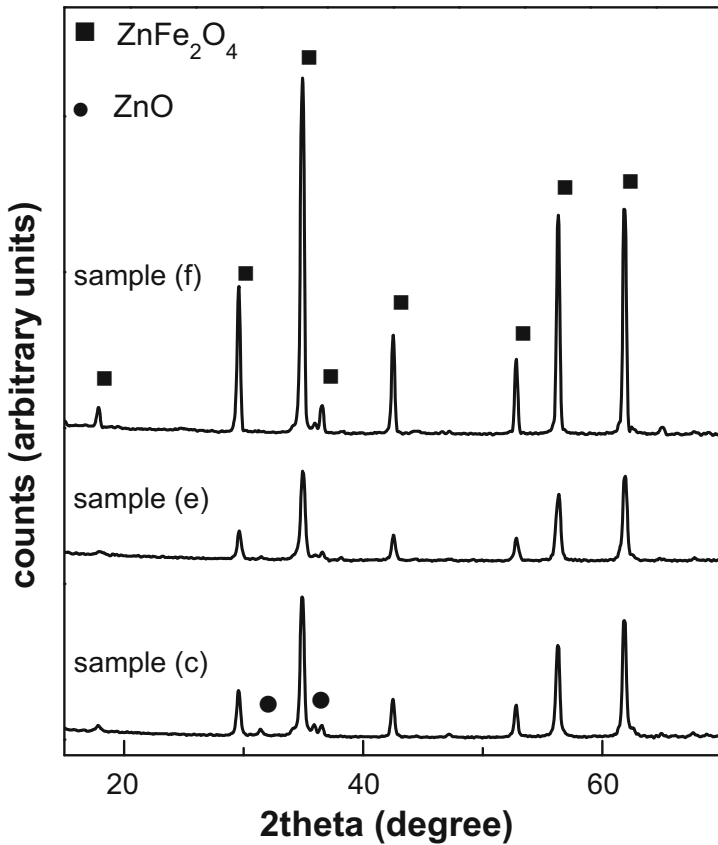
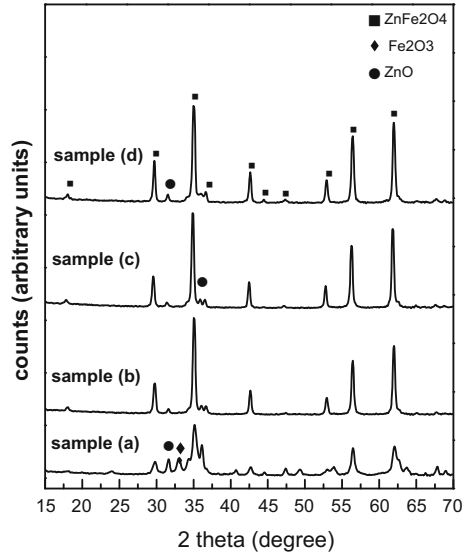


Fig. 2 XRD patterns of as prepared and annealed $ZnFe_2O_4$ samples

Table 2 Structural parameters of ZnFe_{1.7}U samples annealed at 700 °C and 900 °C

Sample code	Annealing temperature °C	Apparent crystalline size D _{XRD} (nm)	Lattice parameter a ₀ (nm)
(c)	ZnFe _{1.7} U	33	0.843
(e)	ZnFe _{1.7} U/700 °C	37	0.845
(f)	ZnFe _{1.7} U/900 °C	41	0.845

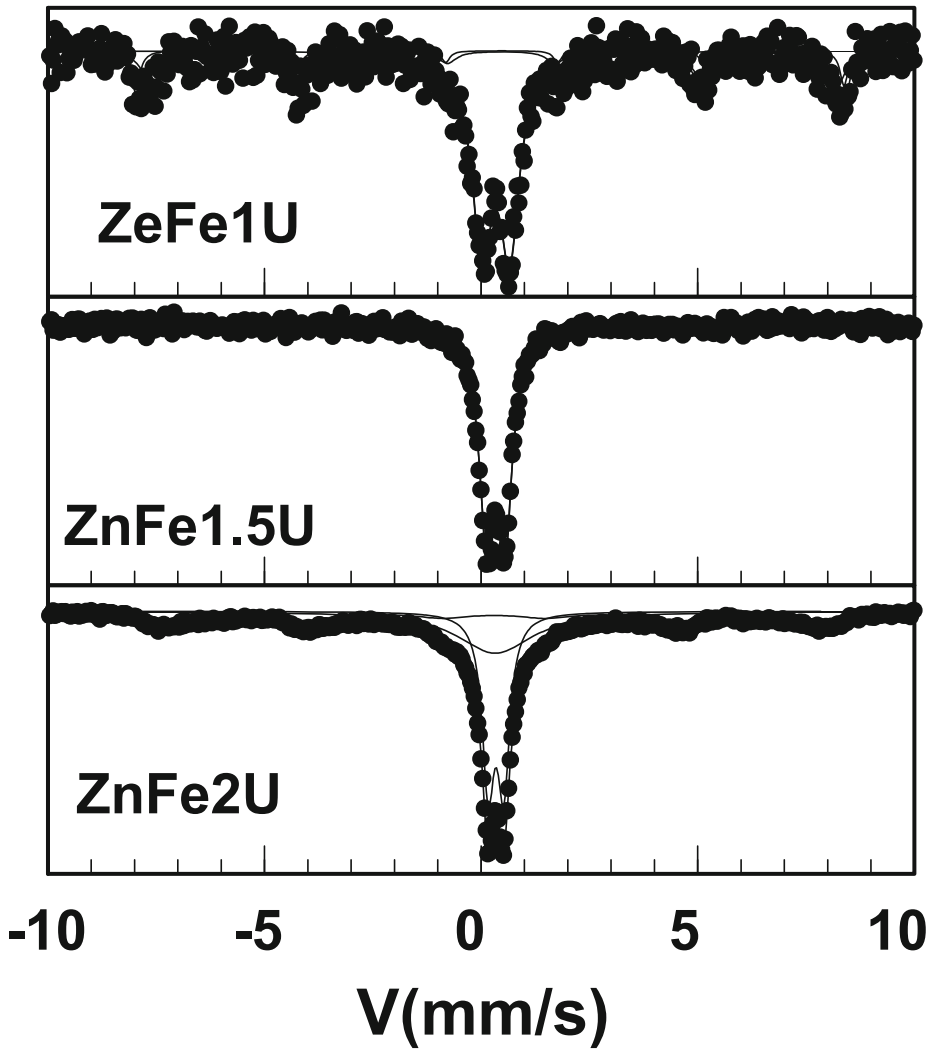
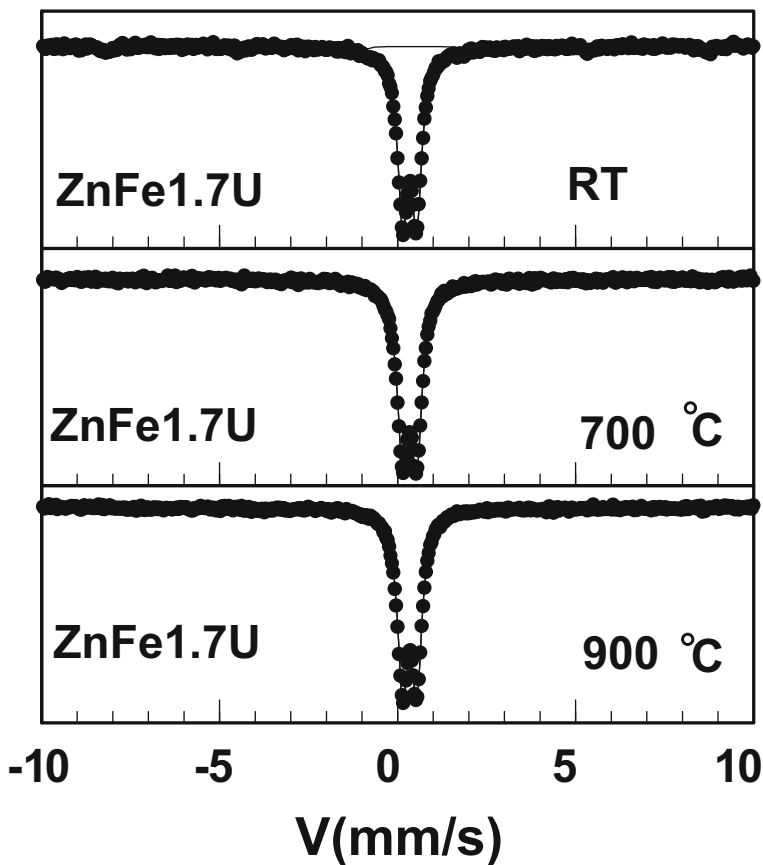


Fig. 3 RT Mössbauer spectra of as prepared ZnFe₂O₄ samples at different U/N ratios

Table 3 Mössbauer parameters isomer shift (δ_{Fe}), hyperfine field (H_{hf}) and quadrupole splitting (ΔE_{Q}) and FWHM (Γ) of Zn-ferrite samples annealed at different temperatures

Sample	A (%)	* δ_{Fe} (mm/s)	H_{hf} (kOe)	ΔE_{Q} (mm/s)	Γ (mm/s)
ZnFe1U	10	0.37	506.3	-0.26	0.22
	24	0.38	489.5	-0.16	0.50
	66	0.34	0.00	0.64	0.54
ZnFe1.5U	100	0.34	0.00	0.42	0.41
ZnFe2U	33	0.29	461	-0.01	1.5
	30	0.33	0.00	0.03	1.21
	37	0.34	0.00	0.36	0.33
ZnFe1.7U	7	0.37	523	-0.21	0.27
	93	0.34	0.00	0.39	0.34
ZnFe1.7U/700 °C	100	0.34	0.00	0.39	0.34
ZnFe1.7U/900 °C	100	0.34	0.00	0.39	0.36

* δ_{Fe} is relative to α Fe foil at RT

**Fig. 4** RT Mössbauer spectra of ZnFe1.7U samples annealed at different temperatures

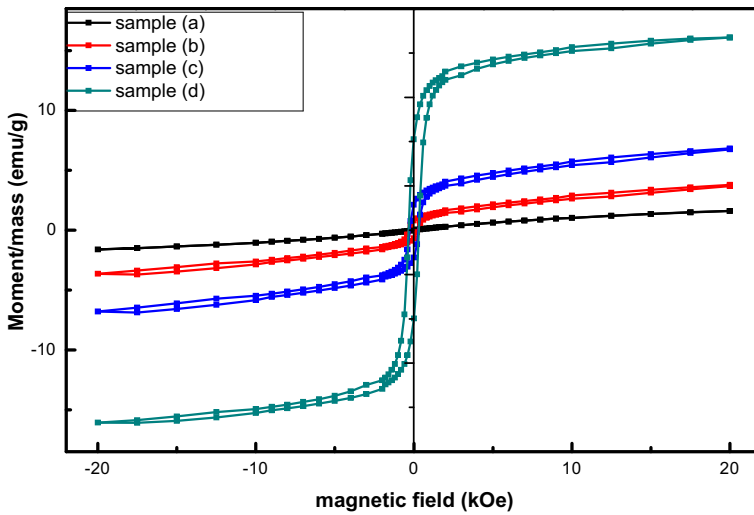


Fig. 5 RT M-H plots of as prepared ZnFe₂O₄ samples at different U/N ratios

Where β_s is the measured line width at half maximum and β_o is the instrumental broadening ($\beta_o = 0.15$) with the apparatus used. K is the Scherrer constant, which is equal to 0.9, λ is the X-ray wavelength (1.548 Å for Cu K α 1) and θ is the diffraction angle. The samples codes, average crystallite sizes and the lattice constants of the prepared and annealed samples are given in Tables 1 and 2 respectively. It is observed that the crystalline sizes and the crystallinity of the prepared and annealed samples increase slightly with increasing U/N ratio and annealing temperature. The lattice constant for each peak for each sample was calculated using the data from the observed diffraction planes. Using a least square fit method [24] exact lattice constants a_0 were calculated and given in Tables 1 and 2. The lattice constant of the samples slightly decreases with U/N ratio. This was explained as an increase in cation inversion parameter as reported by Nachbaur et al. [18]. The lattice constant for ZnFe1.7U sample increases after annealing.

3.2 Mössbauer studies

Room temperature (RT) Mössbauer spectra of the prepared and annealed ZnFe₂O₄ samples are shown in Fig. 3. Dots in Fig. 3 represent the experimental points and the fine lines are the least squares fit of the experimental spectrum. The spectral parameters namely as isomer shift (δ_{Fe}), quadrupole splitting (ΔE_Q), hyperfine magnetic field (H_{hf}) and the half width of the spectral were computed and summarized in Table 3. The spectrum of the ZnFe₂O₄ at U/N = 1 consists of three components, a doublet due to super paramagnetic particles and two sextets: one associated to α -Fe₂O₃ as an impurity phase and the other sextet is due to magnetically ordered spinel zinc ferrite in agreement with that reported in the literature. Several reports have revealed hyperfine magnetic field at RT in nanosized ZnFe₂O₄ [10]. It has been shown that in nanosize ZnFe₂O₄, a fraction of Zn²⁺ ions prefer to occupy the B sites, with a simultaneous occupancy of the A sites by Fe³⁺ ions making the system partially inverted [25]. In contrast, the spectrum of ZnFe1.5U sample prepared at U/N = 1.5 exhibits super paramagnetic doublet spectrum. This can be explained on the basis of transformation from multi domains in bulk samples to single domains in fine powder [26]. In

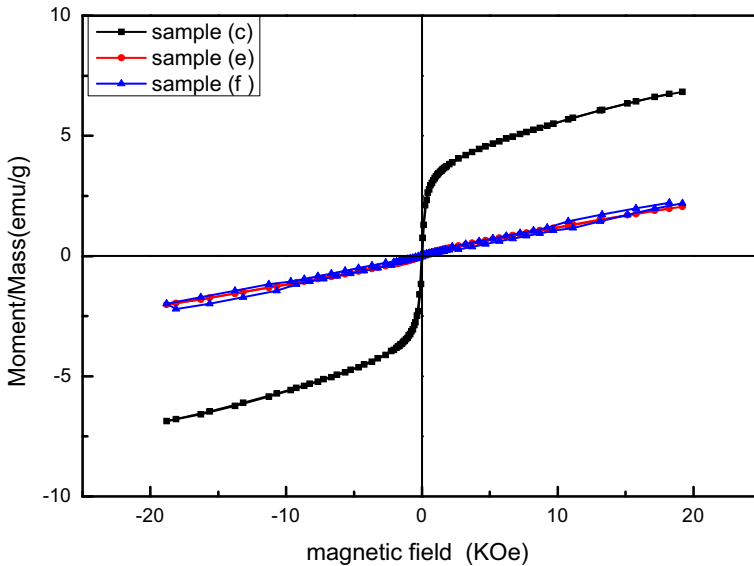


Fig. 6 RT M-H plots of ZnFe_2O_4 samples annealed at different temperatures

other hand the sample prepared at $U/N = 1.7$ consists of one doublet due to super paramagnetic zinc ferrite beside a few amount ($\sim 6\%$) of a sextet with an average hyperfine field 523 kOe which attributed to small fraction of $\alpha\text{-Fe}_2\text{O}_3$ impurity, so it was not shown in the XRD pattern. On the other hand, one can also see that the spectrum of the sample prepared at $U/N = 2$ is composed of a sextet due to $\alpha\text{-Fe}_2\text{O}_3$, a broad doublet and a collapsed sextet. The latter is an indication of relaxation effects which are expected to occur in small-particle systems. A material can be considered to behave magnetically if the fluctuations of the magnetic moment are slow enough in comparison with the observation time-scale. For ^{57}Fe Mossbauer spectroscopy the latter is the precession time τ_L of the nuclear spin in a local magnetic field and amounts 10^{-8} s. For paramagnetic substance the fluctuations are very fast and the time average of hyperfine field over the period τ_L is zero and consequently doublet or singlet spectrum is observed. For a magnetically ordered system such as a ferro- ferri or antiferromagnetic component below its ordering temperature (Curie or Neel temperature) the relaxation is very slow and results in a magnetically split spectrum (Zeeman 6 lines). A particular case, however, is obtained when the relaxation time is of the same order as τ_L . In that region a so called relaxation spectrum is observed as a combination of broad doublets and/ or collapsed sextets with asymmetrically six lines. On the other hand, the Mössbauer spectra of the annealed samples at 700°C and 900°C consist of one doublet which is typically assigned to normal zinc ferrite. The isomer shift (δ_{Fe}) values of all samples are in a good agreement with that of bulk sample ($\delta_{\text{Fe}} = 0.35$ mm/s). The Quadruple shift (ΔE_Q) of the prepared nano-crystalline samples show high value reach to 0.64 mm/s compared to the reported value of bulk sample ($\Delta E_Q = 0.34$ mm/s) [14]. The increase in ΔE_Q for the nano-size ZnFe_2O_4 could be attributed to asymmetric environments around Fe^{3+} ions results from surface effects and cationic inversion takes place [25]. The ΔE_Q value decreases with increasing U/N ratios and annealing temperature due to increase the symmetry around Fe^{3+} ions. The distribution of quadrupole interactions,

Table 4 RT magnetic parameters of as prepared ZnFe₂O₄ samples annealed at different temperatures

Sample code	Sample name/temperature (°C)	H _c (Oe)	M _s (emu/g)	M _r (emu/g) *10 ⁻²
Sample (a)	ZnFe1u/RT	29.86	1.6	0.54
Sample (b)	ZnFe1.5u/RT	21.98	3.7	8.9
Sample (c)	ZnFe1.7u/RT	30.66	6.8	30
Sample (d)	ZnFe2u/RT	38.40	16.1	123
Sample (e)	ZnFe1.7u/700 °C	22.50	2.0	0.69
Sample (f)	ZnFe1.7u/900 °C	6.03	2.2	0.13

in the presence of strong magnetic interaction produces an appreciable broadening of the Zeeman lines but does not produce observable quadrupole line shifts [26]. The magnetic hyperfine field values of 489.5 KOe and 461 kOe are observed respectively in ZnFe1U and ZnFe1.5U samples which are higher than those of the bulk samples [16]. Last but not least, it is worth to point out that the Mossbauer profiles are the same in containing traces of hematite in all samples studied except for ZnFe1.5U whereas the phase of ZnFe₂O₄ is only observed. Although, this result ensures the phase purity of the final product in such sample however; it has led us to continue the work to find out the optimal operation conditions like pH, the water molar ratio that may control the formation of single Zinc Ferrite phase (Fig. 4).

3.3 Magnetization properties

The magnetization measurements of the prepared and annealed zinc ferrite nanopowders were carried out using VSM at room temperature with an applied magnetic field in the range of approximately -20 to 20 kOe. The obtained M-H curves of the as prepared ZnFe₂O₄ samples are shown in Fig. 5. The hysteresis loops were characterized to have superparamagnetic feature for samples prepared with small urea ratio with increasing U/N ratio the ferrimagnetic feature of the hysteresis loops increases due to increase the cation inversion parameter. Figure 6 shows the M-H curves of nanocrystalline ZnFe₂O₄ prepared at U/N = 1.7 which annealed at 700 °C and 900 °C for 3 h. The M-H loop shape of the as prepared sample (c) indicates super-paramagnetic behavior. After annealing process the M-H curves exhibit a paramagnetic behavior which is typical of normal spinel structure [25]. The magnetic parameters of zinc ferrite nano-crystals samples are given in Table 4. The prepared samples at different U/N ratios show an enhanced net magnetization with maximum value of 16 emu/g compared with that of bulk. This attributed to change the cation distribution from normal (bulk) to mixed spinel structure. The magnetization increases with increasing U/N ratio which suggests an increment in the cation inversion parameter. The magnetization of sample (c) decreases with annealing temperature, this reflect low value of inversion degree similar observation was reported by Chinnasamy et al. [27] where Zn²⁺ cations prefer to return in tetrahedral site and Fe³⁺ cations migrate to octahedral sites. The Mössbauer and magnetization measurements indicate that the prepared zinc ferrite samples have a mixed spinel structure. The annealed samples show similar behaviour of bulk zinc ferrite. It was observed in many papers that the magnetization of zinc ferrite nanoparticles depends strongly on the synthesis route as shown in Table 4.

4 Conclusion

Microwave assisted combustion method was used successfully to synthesize nanocrystalline ZnFe_2O_4 from nitrate precursors and urea powder as the fuel with different U/N ratios. The prepared samples exhibit a cubic spinel structure with the appearance of small peaks represents secondary phases which totally disappear after annealing to 900 °C. Mössbauer results and Magnetic measurements at room temperature revealed that the as-prepared nanocrystalline ZnFe_2O_4 at small urea ratio show typical superparamagnetism feature. As the urea ratio increases the ferrimagnetic ordering enhanced this suggest mixed spinel structure. The net magnetization value reaches to 16 emu/g and the hyperfine magnetic field 489.5 KOe. The magnetic ordering disappears after annealing.

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