



# Determination of concentrations of Fe, Mg, and Zn in some ferrite samples using neutron activation analysis and X-ray fluorescence techniques



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## ABSTRACT

Mg–Zn ferrite is considered as one of the important materials with potential uses in many applications. In this work, samples of ferrite  $Mg_{(1-x)}Zn_xFe_2O_4$  (where  $x = 0.0, 0.2, 0.4, 0.6, 0.8$  and  $1$ ) were synthesized by the sol–gel method for use in some hyperthermia applications. The composition and purity of the prepared samples hardly affected their properties. Therefore, the elemental concentration of these samples was measured by the X-ray fluorescence technique and thermal neutron activation analysis to check the quality of the prepared samples. The results of both methods were compared with each other and with the molecular ratios of the as-prepared samples. In addition, no existing elemental impurity, with considerable concentration, was measured.

## 1. Introduction

Mg–Zn ferrite is considered as one of the important magnetic oxides with spinel structure because of its high electrical resistivity, relatively high Curie temperature, low cost, high mechanical hardness, and environmental stability (Reslescu et al., 2006; Manjurul et al., 2008). The potential applications of Mg–Zn ferrites in multilayer chip inductors, microwave, and hyperthermia have been studied by many researchers (Jordan et al., 1997; Rosensweig, 2002; Rudolf et al., 1998; Hiromichi et al., 2005; Nikolai et al., 2001). The magnetic and electrical properties of ferrites are very sensitive to the chemical composition, type, and amount of substitution.

The cation concentration study of ferrites provides more valuable information on the behavior of magnetic dipole and charge carriers. A small amount of foreign ions in the ferrite can drastically change its properties. Therefore, the elemental concentrations of the prepared samples were assessed in the present work by X-ray fluorescence (XRF) and neutron activation analysis (NAA) techniques to ensure that all added materials are included in the final samples, and no loss during the preparation process and no impurities were detected. NAA is a technique to determine the composition of the samples based on the thermal neutron activation of the stable isotopes followed by activity measurements of the reaction products. The reaction products to be measured are radiation either released nearly instantaneously upon neutron capture (prompt  $\gamma$ -rays) or after certain decay time for resulting new radioactive nuclei (delayed  $\gamma$ -rays). All stable elements have properties suitable to produce radioactive isotopes at different

reaction rates. Each radionuclide is uniquely characterized by its decay constant (the probability for the nuclear decay in unit time) and the type and energy of the emitted radiation (Robert et al., 2011; Peter et al., 2009).

XRF spectroscopy is an analytical technique for the elemental analysis of a sample. The technique depends on the unique energy levels of the emission of photons (X-ray) ejected from the atoms (Goldstein et al., 2002). When an incident electron or photon, such as X-ray, strikes an unexcited atom, the electron from the inner shell of the atom is ejected leaving a hole or electron vacancy. An electron from the outer shell fills the hole by lowering its energy, and simultaneously, the excess energy is released through X-ray emission. The energy of the X-rays is characteristic of the energy difference between the two shells. Each atom in the periodic table has a unique electronic structure with a defined set of energy levels accounting for a set of peaks in its X-ray spectrum. The number and energy of the X-rays emitted from a specimen are measured; and on the basis of these, a chemical composition of the sample is determined (Gao, 2004).

## 2. Experimental work

Samples of  $Mg_{(1-x)}Zn_xFe_2O_4$  (where  $x = 0.0, 0.2, 0.4, 0.6, 0.8$  and  $1$ ) were synthesized by the sol–gel method. The sol–gel technique has been recently used as a multifunction method to synthesize ferrite. The sol–gel technique provides the following advantages: the temperature needed for the crystallization process is lower, and the particles produced are ultrafine and have a narrow size distribution (Richard B

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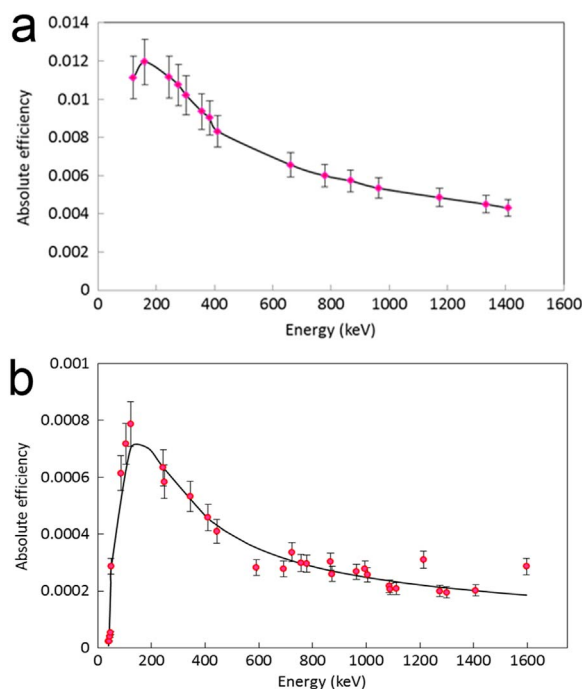


Fig. 1. The absolute detector efficiency calibration curves for the spectrometers, used for short-time (system No. 1) and long-time (system No. 2) radiation, 1.a and 1.b, respectively.

and Virginia S, 1996). Analytical grade  $Mg(NO_3)_2 \cdot 6H_2O$ ,  $Zn(NO_3)_2 \cdot 6H_2O$ ,  $Fe(NO_3)_3 \cdot 3H_2O$  and monohydrate citric acid with 99.98% purity were used without further purification.

Two gamma spectrometers based on high-purity germanium detectors, with necessary electronics, were used to measure the delayed gamma activity after short-time (system No. 1) and long-time (system No. 2) irradiation. Both systems were calibrated for energy and efficiency to increase the accuracy of the results. Hence, a set of standard point gamma sources were used, such as  $^{137}Cs$ ,  $^{133}Ba$ ,  $^{60}Co$  and  $^{152,154,155}Eu$ . For system No. 1, HPGe detector model Ortec GEM-100210 with relative efficiency of 100% and energy resolution, full width at half maximum (FWHM), of 2.1 keV for the 1332.5 keV gamma line of  $^{60}Co$  was used. For system No. 2, HPGe detector model Canberra GC-6020 with relative efficiency of 60% and energy resolution of 2.9 keV for the 1332.5 keV gamma line of  $^{60}Co$  was used. The absolute efficiency calibration curves for the two spectrometers are shown in Fig. 1(a and b).

## 2.1. Sample irradiation and detection

All samples and high-purity titanium foils, used as a neutron flux monitoring, were placed in polyethylene vials and irradiated in the

Table 1  
Nuclear data for the interested isotopes.

Irradiated isotope	Abundance (%)	Reaction	$\sigma$ (barn)	Produced isotope	$T_{1/2}$	$\gamma$ -energies (keV)	$I_\gamma$ (%)
$^{50}Ti$	5.40	$^{50}Ti(n,\gamma)^{51}Ti$	0.179	$^{51}Ti$	5.75 min	320.07	92.9
						608.55	1.17
						928.63	6.87
$^{58}Fe$	0.28	$^{58}Fe(n,\gamma)^{59}Fe$	1.14	$^{59}Fe$	44.50 d	142.65	1.03
						1099.25	56.2
						1291.59	43.2
$^{64}Zn$	48.63	$^{64}Zn(n,\gamma)^{65}Zn$	0.82	$^{65}Zn$	244.3 d	1115.56	50.6
						843.74	71.8
$^{26}Mg$	10.01	$^{26}Mg(n,\gamma)^{27}Mg$	0.038	$^{27}Mg$	9.46 min	170.68	0.8
						1014.42	28

thermal irradiation sites at the second Egyptian research reactor for short- and long-time irradiation. After irradiation, the samples were analyzed qualitatively and quantitatively. Qualitative analysis is used for identifying the present elements in the sample regardless of their concentrations. It can be viewed as a “probing” process for the quick assay of the materials.

The quantitative analysis was used for the exact determination of the element weight (g) in the samples. The weight of an element was calculated according to Eq. (1) (Alfassi, 1998):

$$w = \frac{\lambda MA}{\phi a \sigma N_a I_\gamma \zeta (1 - e^{-\lambda t_i}) e^{-\lambda t_d} (1 - e^{-\lambda t_c})} \quad (1)$$

where:

w = Element weight (g).

$\lambda$  = Decay constant of the product radioisotope ( $S^{-1}$ ).

M = Atomic weight of target nuclide.

A = Area under the characteristic photo peak.

$\phi$  = Thermal neutron flux ( $n\text{ cm}^{-2}\text{ S}^{-1}$ ).

a = Isotopic abundance of a given isotope in the natural target element, which is used for characterization.

$\sigma$  = Thermal neutron absorption cross section (b).

$N_a$  = Avogadro's number ( $6.022 \times 10^{23}\text{ mol}^{-1}$ ).

$I_\gamma$  =  $\gamma$ -ray emission probability per decay (absolute intensity).

$\zeta$  = Detector absolute efficiency at the energy of the characteristic photo peak.

$t_i$  = Irradiation time (S).

$t_d$  = Cooling time (S).

$t_c$  = Counting time of  $\gamma$ -spectrum (S).

The thermal neutron flux was estimated by measuring the activity of an irradiated  $^{nat}Ti$  foil of known mass and neutron absorption cross-section, placed at the same position, with the irradiated unknown samples. The gamma spectrum of the irradiated Ti foil was measured, and the characteristic gamma peak of  $^{51}Ti$  isotope was used for the flux value estimation. The estimated value of the thermal neutron flux was  $2.10^{11}\text{ n cm}^{-2}\text{ S}^{-1}$ . The uncertainty of the estimated flux value was about 9%. The characteristic gamma peaks for  $^{59}Fe$ ,  $^{65}Zn$ , and  $^{27}Mg$  isotopes appeared clearly in the spectrum without any interference. The decay characteristics for  $^{51}Ti$ ,  $^{59}Fe$ ,  $^{65}Zn$ , and  $^{27}Mg$  are listed in Table 1 (Richard B and Virginia S, 1996). The gamma peaks (320.07), (1099.25, 1291.60), 1115.56, and 1014.42 keV were used for the characterization of  $^{51}Ti$ ,  $^{59}Fe$ ,  $^{65}Zn$ , and  $^{27}Mg$ , respectively.

To detect  $^{27}Mg$  (9.46 min), the irradiation time  $t_{irr} = 200\text{ S}$  (short irradiation), cooling time  $t_d = 3\text{ min}$ , and counting time  $t_c = 5\text{ min}$  were used. To detect the relatively long-lived produced isotopes ( $^{59}Fe$  (44.5 d) and  $^{65}Zn$  (244.3 d)), the following sequence was used  $t_{irr} = 2\text{ h}$ ,  $t_d = 4.5\text{ d}$ , and  $t_c = 30\text{ min}$ . The dead time for the samples and standards was nearly constant at about 5%.

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