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Elemental analysis of some Egyptian ores and industrial iron samples by neutron activation analysis

A. Srror[1], N. Abdel-Basset[1], A. S. Abdel-Haleem[2], A. M. Hassan[3]

(1. Nuclear Physics Lab., Physics Department, Faculty of Girls, Ain Shams University, Cairo, Egypt; 2. Environmental Radioative Pollution Department, Hot Laboratories Centre, AEA, Cairo, Egypt; 3. Reactor and Neutron Physics Department, Reactors Division Nuclear Research Centre, AEA, Cairo, Egypt)

Abstract: Elemental analysis of iron ore samples and first industrial iron production prepared by the Egyptian Iron and Steel Company of Helwan near Cairo were determined by instrumental neutron activation analysis technique. Five samples from each kind were irradiated for a 48 hours at a thermal neutron flux of 4×10^{12} n/(cm^2. s) in the first Egyptian research reactor ET-RR-1. Also the Pneumatic irradiation Rabbit system (PIRS) attached to the reactor in Inshass, was used to measure the elements of short-life time. The gamma-ray spectra were recorded by means of the hyper pure germanium detection system. The concentration percentage values of major, minor and trace elements are presented. The long and short lived isotopes were considered. A comparative study and a discussion on the elemental concentration values are given.

Key words: elemental analysis; Egyptian iron samples; neutron activation analysis

Introduction

Nowadays, more interest has grown in the determination of the elemental constituents of most industrial materials such as iron where several different methods are used(Hassan, 1982; Abdul, 1990; Failey, 1979; Anderson, 1981). Recently, there were many investigations on elemental analysis of iron ores using neutron activation analysis technique. Suzuki *et al*. (Suzuki, 1992; 1995) determined the trace elements in Japanese iron and steel reference materials by instrumental neutron activation analysis, the determined values of the detected elements were in good agreement with the certified and reference values. Kondo *et al*. (Kondo, 1996) used 14 MeV neutron generator to determine the elemental concentration in iron ore samples for short half-lives. In their study, the cyclic activation method was utilized.

In the present study, the neutron activation analysis was to make elemental analysis of the Egyptian iron ore used in the Egyptian Iron and Steel Company. The company collect the iron ore from two sites, in which the chemical analysis is the approved methods to determine the constituents of the ore. This study aimed to obtain the correct concentrations of elemental constituents of the iron-ore. Also, to determine the suitable production ore fields from economic point of view.

1 Experimental

1.1 Samples preparation and irradiation

Two iron-ore samples were collected from two sites in Egypt, Baharia Oasis at the west desert of Egypt, and from Aswan area south of Egypt. The third sample was supplied by Egyptian Company of Iron and Steel as the first industrial product. In the present study five copies from each sample were irradiated and measured to confirm the accuracy of the detecting system. The samples were crushed by a crusher machine for small granules $\approx$200 mesh. Each sample with a known mass was placed into a suitable container of aluminum foil in order to expose to the thermal neutrons.

For the samples of Baharia Iron Ore BIO and Aswan Iron Ore AIO, a mass of 0.03g of each ore sample and 0.01g of the first industrial iron product, were individually wrapped in an aluminum sheet. While an empty aluminum foil of the same weight was included in the irradiation can with the studied iron samples, for identifying and subtracting the background gamma-ray lines due to aluminum envelopes. A gold foil of a 0.004g was included as well and rolled in separated aluminum sheet just for flux monitoring. Also, 0.03 g of a standard reference material soil-7 10 mg

of was included at the same condition of irradiation to measure and certified the accuracy of the analysis data. The irradiation time was about 48h at 2 MW power of the ET-RR-1 with an average thermal nutron flux of 4×10^{12} n/(cm^2.s). The gamma-ray spectrum for each sample was collected for 2 hours after 24 hours cooling time. In case of using the rapid irradiation facility PIRS, 10 mg of each sample was used. The time of irradiation was 60 and data collected by the same detection system for one hour time.

1.2 Instrumentation

The hyper pure germanium detection system including a detector of 25% efficiency and energy resolution of 1.9 keV at the 1332.5 keV is used. The detector was connected to a low noise preamplifier, a spectroscopy linear amplifier and a multichannel analyzer with a personal computer system of a 8192 channel. The multi-gamma-ray standard source MGS-4, which obtained from nuclear measurements group, microanalysis group, Oxford Instruments Inc., was used to perform the energy and efficiency calibration of the detection system(Nuclear Measurement Group, 1994). The selected gamma-ray energies of Eu-155, Co-57, Sn-113, Cs-137, Mn-54 and Zn-65 were used for these measurements. The absolute efficiency obtained for this system as a function of energy in keV in the range from 100 keV up to 3000 keV is considered. The sample to detector distances was at 10 cm(Abdel, 1996) and the measurements were repeated for each position three times for each sample, with time interval of one month to follow the spectrum of short-life nuclides.

1.3 Results and discussion

Table 1 Comparison between present work and certified values for the standard material soil-7

Elements	Certified, μg/g	Present work, μg/g	Error
La	28	27.11	−3.17
Mn	631	625.15	−0.93
Sc	8.3	8.5	+2.40
Sm	5.1	5.04	−1.17
Th	8.2	8.65	+5.4
U	2.6	2.71	+4.2
V	66	64.3	−2.5
Zn	104	100	−3.8

Note: the error within ±2.94%

The elemental constituents of the samples under investigation in this work were estimated by means of the activities induced by n, γ reaction(Nicholas, 1983). The gamma-rays emitted were identified according to the energies of the well resolved gamma-ray lines taking into consideration that some of the product isotopes could exhibit more than one gamma-ray line. Two elements ^{52}V, and ^{56}Mn which are a short life isotopes were measured by the Pneumatic irradiation Rabbit system, in addition to the other 7 elements ^{24}Na, ^{76}Sc, ^{59}Fe, ^{76}Ga, ^{82}Br, and ^{140}La which are a long life isotopes were measured by the delayed technique after 24 hours cooling time. To satisfy the accuracy of the detection system, the results of the present study of the reference material soil-7 of some elements which appear clear and well resolved are compared with the certified values of this standard material as shown in Table 1. The nuclear data mentioned in Table 2 were very useful for evaluating the concentration for each element (Practical Aspects of Operating A Neutron Analysis Laboratory, 1990) according to absolute method. Also, the relative method of calculation for some detected isotopes was used to obtained the accuracy and standard deviation of the measured data.

A brief discussion on the presence of some elements is given as follows:

Iron (Fe) is presented in both samples with high percentage values, at 57.06% for BIO sample, at 55.33% for AIO sample and 90.22% for the first product which are in good agreement with the chemical analysis results obtained from the producer company(Egyptian Iron and Steel Co., 1998).

The concentration percentage value of Na was at 0.167% for BIO sample, at 0.209% for AIO sample and 0.066% for the first product, this was estimated by using the Na-24 isotope.

Table 2 Nuclear parameter of the measured nuclide

Nuclear data(Egyptian Iron and Steel Co., 1998)					
Elements	Nuclear reaction	Half-life	Energy keV, (intensity, %)	Cross section σ barns	Abundance, %
Sodium	$^{23}Na,(n,\gamma)\ ^{24}Na$	15.03h	1369(100%) 2754(100%)	0.513	100
Scandium	$^{45}Sc(n,\gamma)\ ^{46}Sc$	83.9d	889.2(100%) 1121(100%)	26.3	100
Vanadium*	$^{51}V(n,\gamma)\ ^{52}V$	3.75m	1343.2(100%)	4.89	99.75
Manganese*	$^{55}Mn\ (n,\gamma)\ ^{56}Mn$	2.58h	846.6(99%) 1811.2(30%)	13.3	100
Iron	$^{58}Fe(n,\gamma)\ ^{59}Fe$	45d	1099(56.5%) 1292(43.2%)	1.31	0.29
Galium	$^{71}Ga(n,\gamma)\ ^{72}Ga$	14.1h	629.9(39.9%) 833.9(96%)	4.61	39.9
Arsenic	$^{75}As(n,\gamma)\ ^{76}As$	26.44h	559.1(44.6%) 657.03(6.4%)	3.86	100
Bromine	$^{81}Br(n,\gamma)\ ^{82}Br$	35.3h	619.1 (43.1%) 776.5(83.4%)	2.58	49.3
Lanthanum	$^{139}La(n,\gamma)\ ^{140}La$	40.2h	487(43%) 1597(95.5%)	9.34	99.91

* Calculated by pneumatic irradiation facility

Scandium-46 isotope is used for estimating the concentration value of scandium as 0.031×10^{-2}% and 0.135×10^{-2}% for BIO and AIO samples, respectively.

Arsenic-76 is used for the present determination of As-concentration values. The concentration were found to be 0.484×10^{-2}%, 4.23×10^{-2}% and 0.0022% for BIO, AIO and for the first product samples, respectively.

^{140}La isotope is used for estimating the concentration values in the range of 0.0164% and 0.021% for BIO and AIO samples respectively.

The ^{82}Br isotope is used for estimating the concentration values of Br in the range of 0.173%, 0.197% and 0.0758% for BIO, AIO and the first product samples, respectively.

The $^{71}Ga(n,\gamma)\ ^{72}Ga$ nuclear reaction of half-life time of 14.1h due to the thermal neutron cross-section of 4.71×10^{-28} m^2 gives two gamma-ray lines at 629.9 keV(39.9%) and 833.9 keV (96%) which were clear in the spectra obtained in this work. The concentration values of Gallium were calculated using the ^{72}Ga γ-ray lines, as 2.81×10^{-2}%, 2.70×10^{-2}% and 0.056% for BIO, AIO and the first product, respectively.

^{52}V isotope is used to estimating the concentration value of vanadium. The value was in the range of 0.024% for BIO and 0.002% for first product.

Manganese-56 is used for the present determination of Mn-concentration value which was found to be 0.5316% for BIO sample and 0.5% for first product.

Table 3 Elemental concentration values of the sample under investigation

Elements	Baharia ore sample	Rep. values (EISC, 1998)	Aswan ore sample	Rep. values (EISC, 1998)	First product	Rep. values (EISC, 1998)
Sodium	$0.167\pm3.44\times10^{-3}$	0.091	$0.209\pm6.2\times10^{-3}$	0.165	$0.066\pm3.0\times10^{-3}$	
Scandium	$0.031\times10^{-2}\pm3.07\times10^{-5}$	–	$0.135\times10^{-2}\pm6.754\times10^{-5}$	–	–	–
Vanadium*	0.024 ± 0.0016*	–	–	–	$0.002\pm6\times10^{-4}$	0.003
Manganese*	0.5316 ± 0.008*	–	–	–	$0.5\pm1.5\times10^{-2}$	0.59
Iron	57.06 ± 0.878	59.59	55.33 ± 1.146	56.73	90.22 ± 1.43	91.55
Galium	$2.81\times10^{-2}\pm2.65\times10^{-3}$	–	$2.70\times10^{-2}\pm1.747\times10^{-3}$	–	$0.056\pm3.9\times10^{-3}$	–
Arsenic	$0.484\times10^{-2}\pm1.60\times10^{-4}$	–	$4.23\times10^{-2}\pm6.053\times10^{-4}$	–	$0.0022\pm1.3\times10^{-4}$	–
Bromine	$0.173\pm5.30\times10^{-3}$	–	$0.197\pm3.71\times10^{-4}$	–	$0.0758\pm2.9\times10^{-3}$	–
Lanthanum	$0.0164\pm2.33\times10^{-5}$		$0.021\pm6.533\times10^{-5}$	–	–	–

* calculated by pneumatic irradiation facility

Table 3 lists the concentration values with their accuracy for the identified nine elements in the three samples. It is clear that iron is the major constituent of the BIO, AIO and the first product samples. The present results of iron concentration in samples are in fair agreement with the reported chemical analysis (Egyptian Iron and Steel Co., 1998).

A portion of the gamma-ray spectrum obtained for Bahariasample is shown in Fig. 1.

2 Conclusion

In conclusion, the applied technique of NAA has a good success in obtaining an accurate concentration of some elemental constituents of the iron ore samples. The uses of thermal neutron flux of 4×10^{12} n/(cm^3. s) at the reactor core and 1.65×10^{11} n/(cm^3. s) in case of using the PRS facility and the hyper pure germanium detection systems with accurate efficiency and energy calibration, gave a good results for both low and high concentration levels of the presented elements. Also, it can be concluded that Baharia Oasis iron ore fields may be more suitable for iron production than Aswan fields from the economical point of view, due to the higher percentage of iron which is mentioned as well in the chemical analysis report(EISC, 1998). In addition to this higher values of iron concentration, the results of other undesirable elements are less than that in Aswan ore sample. It is noticed that in case of investigating the first product iron, Ga, As, Br and Na were appeared in our analysis and not appear in the chemical analysis of the producer company. This makes us to warn the company to take the present data of these toxic elements into consideration according to the purpose of its uses.

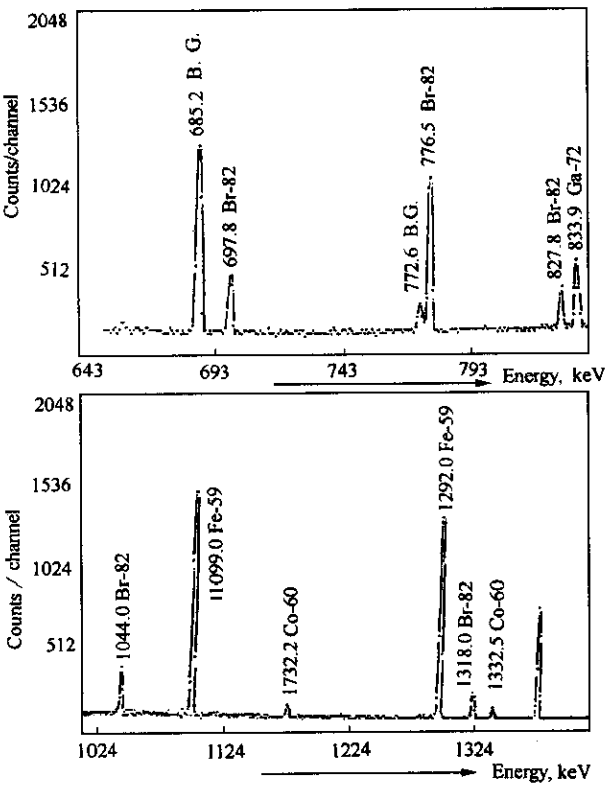

Fig. 1 Portions of the gamma-ray spectra obtained for Baharia sample

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