

NATURAL RADIONUCLIDES ANALYSIS OF PHOSPHATE FERTILIZERS: URANIUM RESOURCES IN TUNISIAN PHOSPHATES

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ABSTRACT

In this paper, we have reported the elemental analysis of phosphate rock and fertilizer located at M'dhila, southern part of Tunisia. This study determined the radioactivity levels of phosphate samples such as phosphate rock, phosphoric acid, triple super phosphate and phosphor gypsum from M'dhila village in the neighborhood of the deposit Gafsa Phosphate Company (CPG). The specific activity (Bq/kg) and concentrations (ppm) of the natural radionuclides ^{238}U and ^{232}Th were determined by Gamma Spectrometry using a High Purity Germanium (HPGe) detector of the National Center for Nuclear Science and Technology (CNSTN) in Tunisia. The average radioactivity concentrations presents in Tunisian phosphates are quantified by natural uranium ^{238}U (60.56 ppm) is also close and sometimes more important to the worldwide average concentration values of the radionuclides reported by The Fertilizer Society Proceedings No.400, 2008 but it can be recovered by a high ppm-values from the Super Triple Phosphate fertilizer (71.065 ppm) and Phosphoric acid (64.742 ppm). Consequently, the extraction of natural uranium can be utilized as an alternative source of energy in Tunisia. The importance of this work is due to his application in many fields specially industrial and electric applications.

KEYWORDS: Natural Radionuclides, Gamma Spectrometry (HPGe), Phosphate fertilizers industry, Uranium, Thorium

I. INTRODUCTION

Nuclear power is expected to be an important part of the worldwide energy mix for the next 50 years. This in turn will make it necessary to develop alternatives and economically feasible technologies for the uranium production from other sources than uranium ore [1].

Nowadays, the global interest is his ancestry to uranium as an alternative energy source compared to others who come year after year to lose its gloss namely oil production in the nuclear field. This comes to the cause of fluctuating oil prices in question: an average 100 \$/lb against 10 \$ in 2001, by cons, uranium is more than 40 \$/lb against 10 \$ in 2003. Therefore, uranium is updated; its extraction becomes profitable and is positioned as an alternative source of energy to oil.

Many countries with limited energy resources, has invested substantial efforts in the study of uranium separation from non-conventional resources such as coal, natural waters, including seawater and phosphate ores. These routes are technically feasible, economically attractive and potentially important because they may allow access to vast uranium resources [2]. Uranium, the gray metal can be extracted as a single ore or as a by-product of mining gold, copper and phosphate. Phosphate rocks have been the most suitable alternative source for uranium recovery. The concentration of uranium in phosphate rocks changes from one deposit to another, ranging from 50 to 200 ppm [3, 4].

Tunisia possesses other large phosphate deposits with reserves matching those of the Gafsa basin. They are located in Sraa-Ouertane region in the north western part of Tunisia. For Tunisia, the most

important source of uranium is the phosphate. The discovery of phosphate deposits in 1896, phosphate production is controlled and operated by Gafsa Phosphate Company (CPG). They are extracted from four main locations in the Gafsa region: Metlaoui, Moulares, Redeyef and Mdhilla. Tunisia's phosphate deposits are found in the North-east of the country, as well as further south. Of these, the most significant are located in the Gafsa basin in the south-central region.

In 2010 [5], CPG company produced nearly 8 million tons of phosphate rock from seven open-pit mines and one underground mine, making Tunisia the world's fifth largest producer. Phosphate rock and phosphate products (phosphoric acid, triple superphosphate (TSP), diammonium phosphate (DAP) and dicalcium phosphate (DCP) together comprise Tunisia's primary export and are sold to 50 countries in five continents. Gafsa Phosphate Company supplies approximately 80% of its phosphate rock production to the Tunisian Chemical Group (GCT), which operates several phosphoric acid plants. The remaining 20% of its production is exported through the port of Sfax. Tunisian Chemical Group has the capacity to produce more than 710^5 tons/year of Phosphoric Acid (AP), 10^6 tons/year of TSP, 610^5 tons/year of DAP, 310^5 tons/year of Nitrogen-Phosphate-Potash (NPP) and 10^5 tons/year of Mono-Ammonium phosphate (MAP). Tunisian Chemical Group owns four industrial production centers located in Sfax and M'dhilla (for TSP), Gabès (for phosphoric acid, DAP and DCP) and Skhira (for phosphoric acid). Approximately 810^5 tons of TSP is produced annually at the Sfax and M'dhilla sites. Because of the huge quantities of processed phosphate ores in Tunisia, the corresponding uranium quantities are large.

The aim of this paper is to discuss the behavior of Uranium throughout the processes of mining, upgrading and fertilizers industry. An experimental study has been carried out to optimize the main operating parameters affecting the uranium recovery from four samples of Tunisian phosphates: Phosphate rock, Phosphogypsum, phosphoric acid and triple super phosphate.

In this work, we begin with a careful description of the importance of uranium in phosphate. Then, we enumerate different materials used in the prototype of experience. Finally, we illustrate different results and discussions.

II. MATERIALS AND METHOD

In order to obtain radioactivity analysis of uranium from the transformation factory of ores in M'dhilla, recovery process should be investigated in detail. Therefore, experimental work has been conducted on a laboratory scale (figure 1) to select and optimize the operating parameters of each process step. To reach this aim, the experimental design method was used by Gamma Spectrometry high resolution with high purity of Germanium detector doped by the lithium (GeHP) and cooled by the Liquid-Azote to study the effect of each parameter separately and their interactions. The main variables that can affect uranium recovery process were included: temperature, solvent ratio, phosphoric acid concentration, and extracts concentrations.



Fig.1. Gamma Spectrometry Laboratory in the National Center for Nuclear Science and Technology (CNSTN)

Gamma spectrometry is an analytical method that allows the identification and quantification of gamma emitting isotopes in a variety of matrices. In one single measurement and with little sample preparation, gamma ray spectrometry allows you to detect several gamma emitting radionuclide in the sample. The measurement gives a spectrum of lines, the amplitude of which is proportional to the activity of the radionuclide and its position on the horizontal axis gives an idea on its energy. Figure 2 shows a block diagram for a basic gamma spectrometry system.

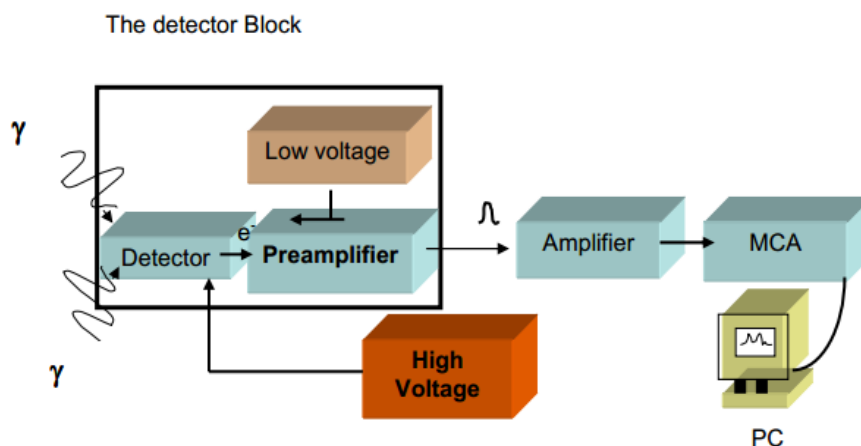


Fig.2. Block diagram of a basic Gamma Spectrometry System

We have taken four Tunisian phosphate samples for radioactivity analysis (Table 1) using Gamma Spectrometry *GeHP*. A normalized sample makes inadequate geometry of measurement in accord with nature and origin. The sample followed in accordance with identical methods with standard sources used. Four samples of grounds hived someone a 1.5 kg of mass, are taken on the industrial site in the temperature of 60°C. So, a length time between 10 to 20 hours and these samples was grinded in fines particles. A plastic box with volume of 600 cm³ (Marinelli Beacker) is partially filled so that lead space for gas liberation (Radon) to taking radioactive stability.

Table 1. Radioactivity analysis of four phosphate samples using Gamma spectrometry *GeHP*

	Phosphate	Phosphogypsum	Triple super phosphate	Phosphoric Acid
Mass(g) or volume (ml)	789.09 ± 0.01	473.99 ± 0.01	562.84 ± 0.01	500
Time of measurement (s)	810 ⁴	10 ⁵	810 ⁴	10 ⁵

The samples were put in 0.6 L polypropylene Marinelli beakers for their measurements at the *CNSTN*, and sealed with wax to allow the equilibrium between ²²²Rn and its descendants to be reached for determination of ²³⁸U. In order to monitor the state of the secular equilibrium, all samples were measured for 15 hours about once a week for a period of five weeks. In addition, the sample was monitored for the period of 2 months to ascertain whether no ²²²Rn is lost by penetration through the walls of Marinelli Beakers. Measurements were performed using the spectrometer, parameters of whose are given in Table 2. The detector efficiency was determined using several calibration sources in the 0.6L Marinelli beakers:

²⁴¹Am, ¹⁰⁹Cd, ⁵⁷Co, ⁶⁰Co, ¹³⁷Cs, ⁵⁴Mn, ²²Na, ⁸⁵Sr, ⁸⁸Y and ²²⁶Ra

Each source (from the Czech Metrological Institute: *CMI*) was formed by a silicon resin (density = 0.98 gcm⁻³) containing the source.

The experimental points were fitted on a log-log scale by a third degree polynomial below the knee (200 keV) and by a linear function above the knee to obtain the calibration curve. Self-shielding

effects were assumed to contribute less than 2% error in the activity values measured for these types of samples[6].

For the determination of the individual radionuclides, the following gamma-rays were employed: ^{238}U

• $^{234\text{m}}\text{Pa} - 1001.03 \text{ keV} (0.837 \%)$ or $^{214}\text{Bi} - 609.31 \text{ keV} (44.84 \%)$; $^{232}\text{Th} - ^{228}\text{Ac} - 911.205 \text{ keV} (26.6 \%)$; ^{137}Cs

• $^{137\text{m}}\text{Ba} - 661.66 \text{ keV} (85.1 \%)$; $^{40}\text{K} - 1460.83 \text{ keV} (10.67 \%)$ [7].

The relative expanded uncertainty (*coverage factor* = 2) resulting mainly from relative standard uncertainties of peak area calculations (0.55 – 69 %), calibration curve (0.04 – 1.1 %) and self-shielding effects (2 %) amounted up to 69.5 % [8].

Table 2. Characteristics of the Gamma spectrometers used

Spectrometer	1	2	3	4
Coaxial detector	EG&G Ortec GEM 35190	PGT IGC 20	EG&G Ortec GEM – 10175 – Plus	EG&G Ortec GEM – 50195 – Plus
FWHM resolution ^a (keV)	1.69	1.75	1.8	1.75
Rel. efficiency %	34.1	20.8	11.5	52.9
Peak/Compton ratio	60.6 / 1	52.6 / 1	48.2 / 1	73.1 / 1
Shielding	ORTEC HPLBS1 ^b	6.5 cm steel	5 cm Pb ^c	5 cm Pb ^c
(inner space)	0.025 m ³	0.5 m ³	0.1 m ³	0.1 m ³
Background ^d	1.776	2.164	1.053	3.096
MCA	TRUMP 8K	ND 570	ND 581	Canberra 8713

a: at 1332.5 keV; b: lead with 9.5 mm steel casing + 0.5 mm Sn + 1.6 mm Cu; c: inner lining with 1 mm Cd + 0.5 mm Cu; d: (counts s⁻¹ in the 50 – 2000 keV region)

III. RESULTS AND DISCUSSION

The activity concentration (*in Bq/kg*), A_{Ei} of a radionuclide i and for a peak of energy E , in samples was calculated using the following formula [9]:

$$A_{Ei} = \frac{N_{Ei}}{\gamma_d \varepsilon_E m_s T} \quad (1)$$

Where N_{Ei} is the net peak area at energy E , ε_E is the detection efficiency at the energy E , T is the counting live time in seconds, γ_d is the gamma ray yield per disintegration of the specific radionuclide for a transition at energy E , and m_s is the mass of the dry weight in kg of the measured sample.

The phosphate rock used as ore for the production phosphoric acid is naturally radioactive, like many minerals. It's therefore natural to find them in products of this manufacturing sector. It was found that ^{238}U and ^{232}Th , radioactive, remained preferably in the acid, while ^{226}Rn found almost entirely in the phosphogypsum. Measures high resolution gamma spectrometry performed at the National Center for Nuclear Science and Technology of Tunisia gave the following results in terms of average activity expressed in $809.28 \pm 0.03 \text{ Bq/Kg}$.

3.1 Gamma-rays spectrum of phosphoric acid before and after stability

We cannot directly identify ^{238}U in the histogram (figure 3 and figure 4) because it's not detectable by the (GeHP) detector and he has weak gamma energy. For this party, we can determine the activity and the concentrate phosphoric ion of ^{238}U in our samples.

If a radionuclide of a natural parents was announced in the spectrum results of environment and one of these radionuclide (the very detectable) in his filiations' series cannot be detectable in the spectrum, so his validity presence will be putting in cause.

The spectral analysis by gamma-rays of phosphoric acid (figure 3-4) show a big difference of measurement before and after 21 days. This difference is mainly due to the release of radon gas in the vacuum party higher than Marinelli Beaker of the radioactive stability. The peak of the Protactinium ^{234}Pa is equivalent a canal 3800 present an important variation for the four samples: we have a totally weak intensity of the phosphogypsum, than higher for the phosphate and important intensity for the phosphoric acid and the super triple phosphate. The spectrums presents the different peaks of the absorbents isotopes having gamma-rays between 0 and 8001 (0 – 2MeV) of intensity and having position depending nature of isotope and his position in the series (parents' chain):

Uranium: ^{238}U ; Thorium: ^{232}Th ; Uranium: ^{235}U ; Neptunium: ^{237}Np

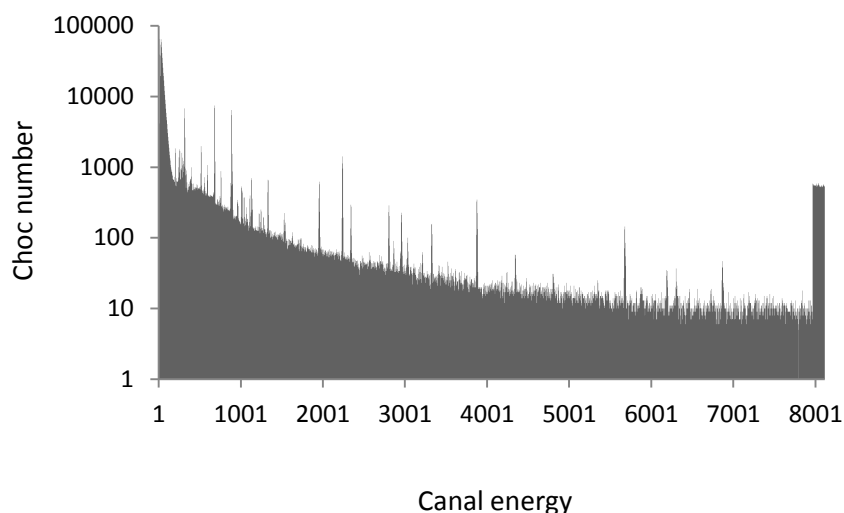


Fig.3. Gamma-rays spectrum of phosphoric acid before stability

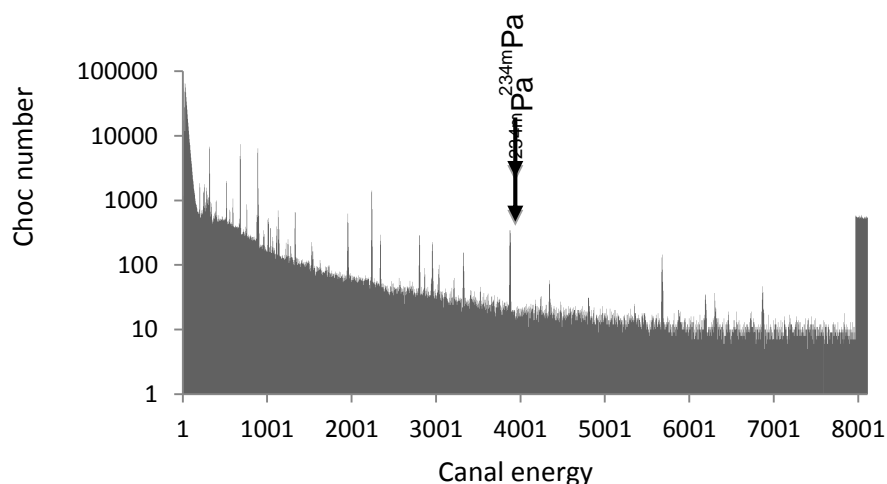


Fig.4. Gamma-rays spectrum of phosphoric acid after stability

3.2 Activity(Bq/kg)for radionuclide's of phosphate and derives products

We choose two radioactive parents chain to analyze the presence of isotope-uranium elements in four examples of phosphate such as the uranium and thorium chains. Measures taken (Table 3-6) gives an

idea of the activity (Bq/kg) and the concentration (ppm) of various radioactive elements. Indeed, we have elements corresponded of an alone gamma energy like

$$^{234m}Pa, ^{226}Ra, ^{212}Pb \text{ and } ^{40}K$$

Others elements corresponded of much gamma energy such as

$$^{214}Pb, ^{214}Bi, ^{235}U, ^{228}Ac, ^{212}Bi \text{ and } ^{208}Ti.$$

It is an isotope with different gamma energy (peak 2 or 3), why we have the highest value. The activity values for the different samples are dissimilar the one to the other. We find for the phosphate the child of ^{238}U chain the values between 278.8 and 309 Bq/kg and a weak value for ^{235}U (17 Bq/kg). The isotope having the most important activity for the phosphogypsum are ^{226}Ra , ^{214}Pb and ^{214}Bi , varied between 202 Bq/kg and 213.4 Bq/kg except for the ^{234m}Pa having 24.34 Bq/kg of activity and zero activity of the ^{235}U , but for the super triple phosphate and the phosphoric acid, the activity and the concentration of the protactinium is 800 Bq/kg . It's the interesting element. For the phosphoric acid, after the phosphate attack by the sulfuric acid, the important quantity of the protactinium passes in the phosphoric acid.

3.2.1 Activity before stability Uranium chain

Table 3. Activity of Isotopes- Uranium chain before stability

$E_\gamma(keV)$	Radionuclide	Activity (Bq/kg) of phosphate	Activity (Bq/kg) of phosphogypsum	Activity (Bq/kg) of super triple phosphate	Activity (Bq/kg) of phosphoric acid
1001.03	^{234}Pa	309.1 ± 0.002	24.34 ± 0.3	524.1 ± 0.003	504.5 ± 0.02
686.21	^{226}Ra	288 ± 0.002	202 ± 0.003	186 ± 0.004	822 ± 0.003
95.22 351.93 242	^{214}Pb	299.1 ± 0.002	192.9 ± 0.003	183 ± 0.003	6.5 ± 0.03
609.31 1120.29 1764.46	^{214}Bi	278.8 ± 0.002	213.4 ± 0.003	189.1 ± 0.06	8.23 ± 0.04
163.36 143.76 205.31	^{235}U	17.1 ± 0.1	0	25.9 ± 0.07	37.31 ± 0.05

Thorium chain

Table 4. Activity of Isotopes-Thorium chain before stability

$E_\gamma(keV)$	Radionuclide	Activity (Bq/kg) of phosphate	Activity (Bq/kg) of phosphogypsum	Activity (Bq/kg) of super triple phosphate	Activity (Bq/kg) of phosphoric acid
911.20 968.67 1588.20	^{228}Ac	25 ± 0.02	3.8 ± 0.09	17.3 ± 0.02	0.4 ± 0.02
238.63	^{212}Pb	17.3 ± 0.01	1.4 ± 0.1	26.3 ± 0.01	42.37 ± 0.001
727.33	^{212}Bi	24.5 ± 0.05	6.1 ± 0.1	35.08 ± 0.04	47.1 ± 0.03
1620.50					
583.19	^{208}Ti	6.4 ± 0.02	0.6 ± 0.14	9.58 ± 0.01	12.8 ± 0.01
860.56					
1460.83	^{40}K	21.2 ± 0.04	24.1 ± 0.03	35.3 ± 0.03	21.82 ± 0.03

3.2.2 Activity after stability Uranium chain

Table 5. Activity of Isotopes- Uranium chain after stability

E_γ (keV)	Radionuclide	Activity (Bq/kg) of phosphate	Activity (Bq/kg) of phosphogypsum	Activity (Bq/kg) of super triple phosphate	Activity (Bq/ kg) of phosphoric acid
1001.03	^{234}Pa	380 ± 0.04	21.34 ± 0.4	888.31 ± 0.02	809.28 ± 0.03
686.21	^{226}Ra	316.02 ± 0.02	198.19 ± 0.19	241.6 ± 0.03	6.12 ± 0.05
95.22 351.93 242	^{214}Pb	340.59 ± 0.002	207.9 ± 0.03	245.09 ± 0.002	8 ± 0.05
609.31 1120.29 1764.46	^{214}Bi	315.68 ± 0.002	188.48 ± 0.03	238.14 ± 0.003	4.24 ± 0.06
163.36 143.76 205.31	^{235}U	15.31 ± 0.13	0	36.45 ± 0.06	203.3 ± 0.01

Thorium chain

Table 6. Activity of Isotopes-Thorium chain after stability

E_γ (keV)	Radionuclide	Activity (Bq/kg) of phosphate	Activity (Bq/ kg) of phosphogypsum	Activity (Bq/kg) of super triple phosphate	Activity (Bq/kg) of phosphoric acid
911.20 968.67 1588.20	^{228}Ac	32.54 ± 0.01	5.57 ± 0.06	24.85 ± 0.02	2.5 ± 0.16
238.63	^{212}Pb	24.12 ± 0.009	2.37 ± 0.06	32.61 ± 0.01	38.33 ± 0.01
727.33 1620.50	^{212}Bi	26.88 ± 0.05	7.19 ± 0.05	40.95 ± 0.04	24.88 ± 0.03
583.19 860.56	^{208}Tl	7.24 ± 0.018	1.77 ± 0.05	12.65 ± 0.1	14.86 ± 0.02
1460.83	^{40}K	27.77 ± 0.03	14 ± 0.05	45.2 ± 0.02	21.57 ± 0.04

3.3 .Quantification of natural uranium ^{238}U

The most intense γ -rays of the ^{238}U have emission intensity in order of $10^{-2}\%$. The two directly descendants ^{232}Th and $^{234\text{m}}\text{Pa}$ having a very weak periods (24 days and 1.17 minutes) in front of ^{238}U ($4.48 \times 10^9 \text{ ans}$). These three radionuclides are usually in the old stability. ^{238}U is dosed by her descendants ^{232}Th and $^{234\text{m}}\text{Pa}$. The most intense ray of ^{232}Th is in the bass energy positions can be eased. The ^{234}Th dosage will be very hard. So, the ^{238}U is preferentially quantified by the $^{234\text{m}}\text{Pa}$. In his disintegration, ^{238}U produce the radioactive ^{234}Th with 24.1 days of period. In the waterfall spring system, the speed of disintegration of thorium was controlled by the contribution of the uranium. Usually, we have a stability of sale like the conservation of water between basin and faucet, like of the activity corresponding in many stones disappearing via seconds like a sale. We can sad that the activity is in stability: we don't have an acting retention. The ^{234}Th can disintegrate in the

radioactive ^{234m}Pa with a period equal to 1.2 minutes. The protactinium activity will be in stability with the uranium and the thorium [9]:

$$\text{Activity}(^{238}\text{U}) = \text{Activity}(^{234m}\text{Pa}) = \text{Activity}(^{234}\text{Th})$$

A stable 7, illustrates the specific activity and concentration of ^{238}U and ^{235}U in various phosphate products. The average specific parameter of Uranium was determinate by the following relation:

$$\bar{\chi} = \frac{\sum_i \chi_i f_i}{\sum_i f_i} \quad (2)$$

where

$\bar{\chi}$ is the average activity-concentration of Tunisian phosphates (in Bq/g or ppm);

χ_i is the activity-concentration of i -Tunisian phosphate sample (in Bq/g or ppm)

$f_i = \frac{\chi_i}{\sum_i \chi_i}$ is the fraction of activity-concentration of i -Tunisian phosphate sample;

The results show that the average activity ^{238}U is 4.5 times greater than ^{235}U with an average value equal to $760.77 \pm 0.33 \text{ Bq/Kg}$ (deposit of M'dhila). This concentration represents a significant potential source of uranium as large amounts of phosphate rock are processed in [3,4]. Therefore, we must choose natural uranium to determine their content in phosphate products.

Table 7. Activity and Concentration of natural uranium in Tunisian Phosphate products

Products	Activity of ^{235}U (Bq/kg)	Activity of ^{238}U (Bq/kg)	^a Concentration of ^{235}U (ppm)	^a Concentration of ^{238}U (ppm)
Phosphogypsum	0	21.34 ± 0.4	0	1.707
Phosphate	15.31 ± 0.13	380 ± 0.04	1.225	30.40
Phosphoric Acid	203.3 ± 0.01	809.28 ± 0.03	16.264	64.742
Super Triple Phosphate	36.45 ± 0.06	888.31 ± 0.02	2.916	71.065
Average (Eq.2)	168.17 ± 0.08	760.77 ± 0.33	13.453	60.559

a: $\text{Concentration (ppm)} \cong 0.08 * \text{Activity (Bq/Kg)}$

The radioactivity measured in samples of Tunisian phosphate give idea of the important content of uranium in chemical fertilizers such as Super Triple Phosphate and phosphate. So, we can extract this uranium by using chemical purification transactions from these two products. When it's extracted, the uranium will be a new source of energy. During the process, the uranium content of the phosphate ores is preferentially transferred to the phosphoric acid and Super Triple Phosphate.

The average activity of natural radioactivity from Tunisian phosphates (deposit of M'dhila) were compared with the range and average of natural radioactivity concentration levels in phosphate rocks of eight countries give in Table 8. The mean concentration of ^{238}U found in this study may be considered an important results compared to the mean concentrations in phosphate rocks of the countries reported by [10]. Thus, the 60.56 ppm of ^{238}U is courageous for our country in order to extract uranium from phosphate which aims to reduce fuel costs, meet future needs civilian nuclear power and also for export.

Table 8. Comparison of natural radioactivity ^{238}U levels from Tunisian phosphates (deposit of M'dhila) with those in other countries

Country	Deposit	^{238}U (ppm)
Algeria	Djebel Onk	25
	Djebel Kouif	100
Australia	Duchess	80 – 92
China	Undifferentiated	10 – 39
Egypt	Abu Tartur	40 – 120

Jordan	Shidyia	46
Morocco	Bucraa	70 – 80
	Khourigba	80 – 120
Peru	Sechura	47 – 80
Saudia Arabia	Ma'aden	25 – 85
Senegal	Taiba	64 – 70
Syria	Khneifiss	75
Tanzania	Minjingu	390
Togo		77 – 110
USA	North Carolina	41 – 93
	Central Florida	59 – 200
	North Florida	50 – 143
	Idaho	60 – 141
Present study	Md'hila	60.56

Like other developing countries, Tunisia is planning to engage in the near future with the use of nuclear energy for civilian purposes. In 2025, nuclear strategy will be made for the implementation of a civilian position with an average capacity of 900 *megawatts*. Thus, Tunisia could export the ore as most neighboring countries have planned in the medium term, the construction of nuclear power plants to produce electricity. Tunisia's interest to optimize the operation of its multidimensional uranium as well as the image of nuclear power in the world has improved and that security in this area has made great progress.

IV. CONCLUSION

The analyze of the radioactivity in Tunisia phosphate (deposit of M'dhila) can giving idea of the sharing out of specific activity in the radio-isotopes of uranium, radium and radon. In view of the diminution of the energetic sources in the world, we research a new economical renewable source of energy. Example the nuclear energy based of the radioactive elements (Uranium, Plutonium...etc.).For these raisons, we work to extract the uranium of the phosphate. The uranium exists in some continents and in many type of soil. The radioactivity measured in sample of Tunisian phosphate give idea of the higher uranium-concentrations in chemical fertilizers such as the Super Triple Phosphate fertilizer(71.065 *ppm*) and Phosphoric acid(64.742 *ppm*) and sometimes more important values compared to the mean concentrations in phosphate rocks of the countries reported by [10]. The very important producers in the world are usually a big area country. The yield of the extraction operations is 99.5 %. It was respected for economic and political raisons [11].So, the internationals authority controls the proliferation of the nuclear matter for respecting this minimum yield. Especially, in Tunisia, we can use the phosphate like an energetic source of energy because he contains an important content of uranium.

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