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ENVIRONMENTAL IMPACT OF RADIONUCLIDES AND INORGANIC CHEMICALS FROM AL-QAIM FERTILIZERS COMPLEX, IRAQ

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ABSTRACT

Unfortunately phosphate rock mining and fertilizer industry manufacturing operations are not environment friendly. It releases huge amount of toxic contaminants into air, water and soil, which led this industry to cause serious environmental problems, among which, fluorides and radionuclides the primary toxic pollutants. This brief review highlights many issues of environmental concern; among which are the impact of natural radionuclides present in phosphate ores, and along the processes, waste products, and radon emanation problems. Phosphate fertilizers contaminate agricultural soils with radionuclides and heavy metals through regular application, in such a way that it necessitates their removal from the end products. The cost of extraction of uranium from phosphoric acid as byproduct compensates much of the cost of purifying the acid. The environmental impact and the improved waste management requirements include harmful gas rendering, controlling effluent waste below harmful levels, as well as the proper dumping of the huge amount of phosphogypsum waste. Recent waste management procedure and general guidelines to mitigate or reduce the environmentally harmful impacts of the contaminant are given in this review.

الأثر البيئي للنويدات المشعة والمواد الكيميائية اللاعضوية من مجمع القائم للأسمدة الفوسفاتية، العراق

سعدي محمد ظاهر النزال

المستخلص

إن تعدين صخور الفوسفات وعمليات تصنيع الأسمدة منها ليست صديقة للبيئة لسوء الحظ. وتنجم عنها إطلاق كميات هائلة من الملوثات السامة الى الهواء والماء والتربة، مما جعل هذه الصناعة السبب وراء مشاكل بيئية خطيرة، من بينها الفلوريدات والنويدات المشعة كملوثات سامة أولية. إن هذا الاستعراض الموجز يسلط الضوء على العديد من القضايا ذات الاهتمام البيئي، ومن بينها تأثير النويدات المشعة الطبيعية الموجودة في خامات الفوسفات، وعلى طول العمليات الإنتاجية، وفي النفايات المنتجة منها، ومشاكل انبعاث الرادون. تسمد الأسمدة الفوسفاتية التربة الزراعية مع النويدات المشعة والمعادن الثقيلة من خلال التطبيق المنظم، بطريقة تستلزم إزالتها من المنتجات النهائية. إن تكلفة استخراج اليورانيوم من حامض الفوسفوريك كمنتج ثانوي يعوض الكثير من تكلفة تنقية الحامض. ويشمل هذا الاستعراض الأثر البيئي وتحسين متطلبات إدارة النفايات ومنها الغازات الضارة، والسيطرة على النفايات السائلة دون المستويات الضارة، فضلا عن الطمر السليم لكميات هائلة من نفايات الفوسفوجبسم. وتطرق هذا الاستعراض على الإجراءات الحديثة لإدارة النفايات والمبادئ التوجيهية العامة للتخفيف من الآثار الضارة بيئيا للملوثات أو الحد منها.

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INTRODUCTION

Phosphate fertilizers industry is not environment friendly operations. The production technology releases huge toxic contaminants into air, water and soil, and cause serious environmental problems, among which fluorides and radionuclides as primary toxic pollutants (Dittmar *et al*., 2009; Toama *et al*., 2015).

Iraq is among the world's leading countries in phosphate rock resources estimated to be about 10 billion metric tons (Al-Bassam, this issue), which put Iraq the second after Morocco in that respect. The Iraqi phosphate reserves were estimated in some deposits in the Western Desert to be 5,750 MT, which represents 9.0% of the world's total phosphate rock reserves (Taib, 2013). Akashat phosphate open pit mine is an integrated mining unit having two operating quarries with a designed capacity of 3.4 MT/year. The main task at the mine is limited for extraction of the phosphate rocks, crushing, loading and conveying the raw materials by rail wagons to Al-Qaim Complex Plant, (State Company for Phosphate, 2017), (Fig.1). The beneficiation process is carried out at Al-Qaim Plant to increase the P_2O_5 content of the raw phosphate rock from 21 to 30 % to be processed to phosphoric acid production and then through phosphate fertilizers industry. The processing units in this plant can produce phosphoric acid, TSP, monoammonium phosphate $NH_4H_2PO_4$ (MAP), compound fertilizer NP, NPK, cryolite $NaAlF_6$, ammonia, and sulfuric acid, (Mishra *et al.*, 2010).

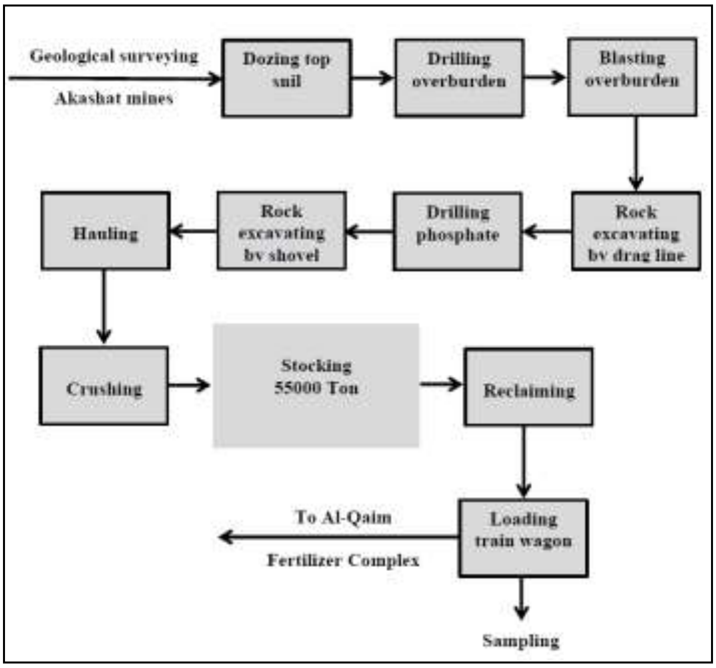


Fig.1: The sequence of mining operations in Akashat phosphate mine with a main task limited for extraction of the phosphate rocks, crushing, loading and conveying the raw materials by rail wagons to Al-Qaim Complex Plant.

AL-QAIM FERTILIZERS PLANT

The Chemical complex at Al-Qaim was established in 1976 and started production in 1985 (Al-Mohammadi, 2015) (Fig.2). The Complex consists of a set of integrated and interrelated plants to produce chemical fertilizers as a final product (Fig.3). These plants include ore beneficiation plant, sulfuric acid plant, phosphoric acid plant, utilities plant, fertilizers plant, ammonia plant, fluorine-salts plant, and materials handling plant.

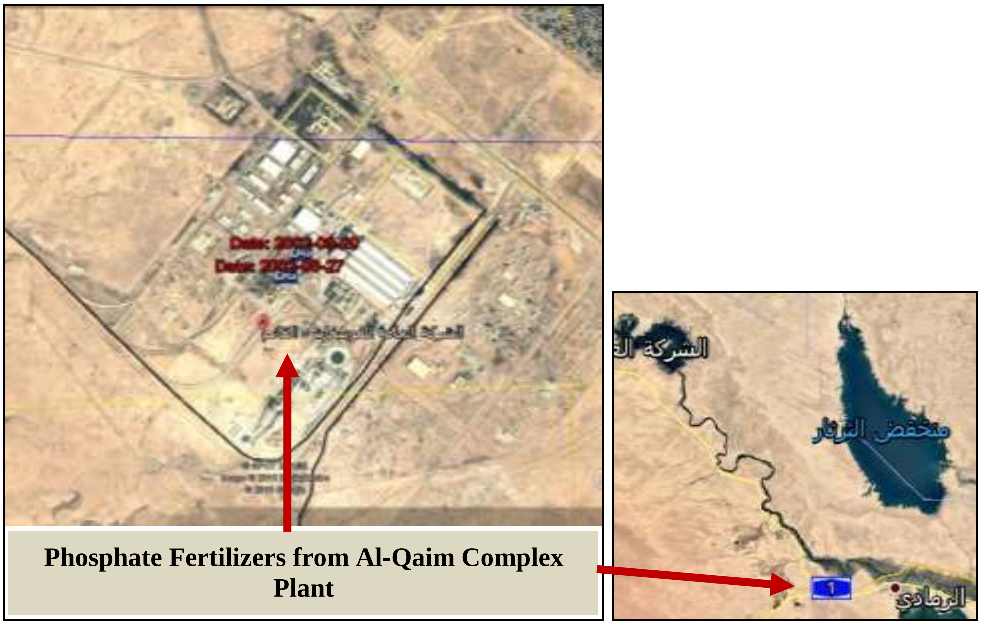


Fig.2: Satellite image of Al-Qaim Fertilizers Plant

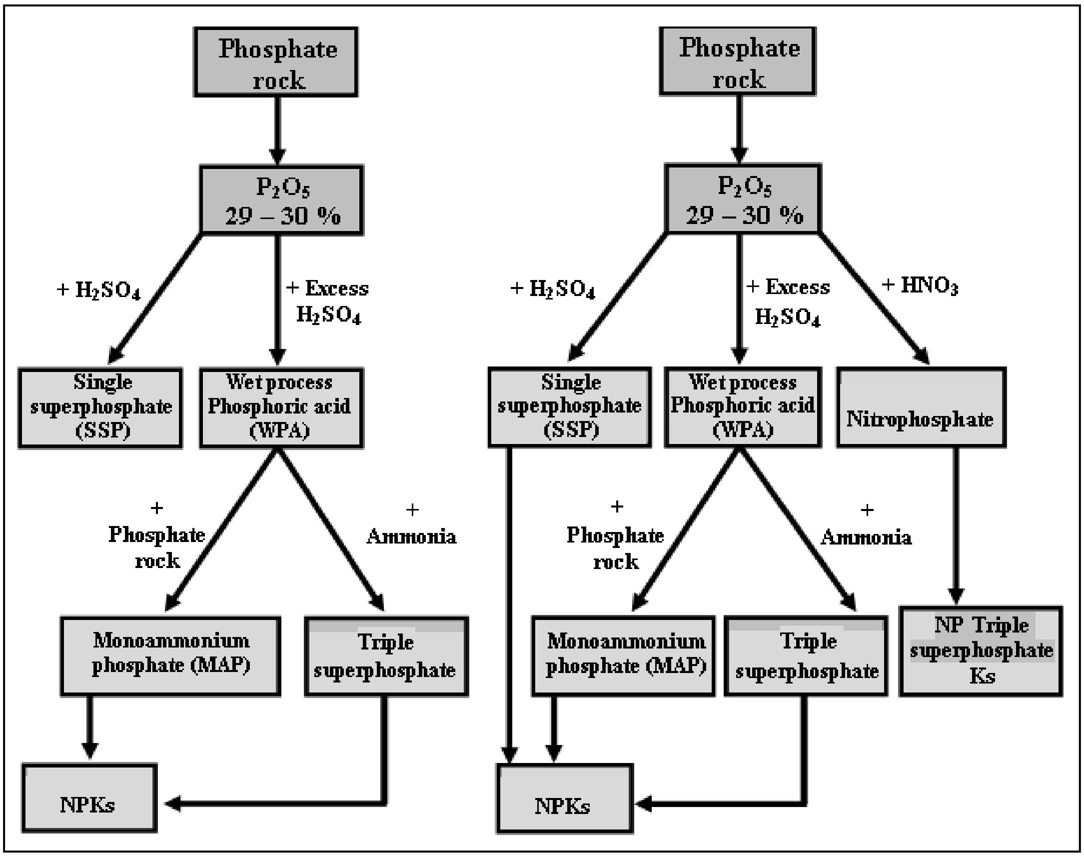


Fig.3: The general production process used in Al-Qaim Complex (a) to convert phosphate rock, P_2O_5 (29 – 30 %), TSP, MAP, Phosphoric acid, and (b) NPK and extended possible plant (Dissanayake and Chandrajith, 2009)

When phosphate rock is treated with sulfuric acid in sub stoichiometric quantity, normal superphosphate is formed. If more sulfuric acid is added, a mixture of phosphoric acid and gypsum (hydrated calcium sulfate) is obtained. Gypsum can be removed by filtration according to the following simplified reaction equation:

$$Ca_3(PO_4)_2 + 3H_2SO_4 + 6H_2O \rightarrow 2H_3PO_4 + 3CaSO_4.2H_2O \quad \ldots\ldots\ldots\ldots \; 1$$

The resultant phosphoric acid can be treated by following different extraction techniques to recover uranium. Half of the fluorides can be removed as gases such as volatile hydrogen fluoride or fluorosilicic acid, and the rest is removed with gypsum. In the process a lot of crude is generated, and this is disposed of with gypsum tailings, despite its low-level radioactivity. The general production process used by Al-Qaim Complex converts phosphate rock, P_2O_5 (29 – 30 %) to TSP, MAP, Phosphoric acid, and NPK, while it is possible to extend the plant for more products (Fig.3) (Dissanayake and Chandrajith, 2009).

NATURAL RADIONUCLIDES IN MARINE PHOSPHORITES

Phosphate rocks, or phosphorites, originated in a marine environment, are characterized by active concentrations of uranium much higher than those of volcanic and biogenic rocks. Deposits of volcanic phosphate rocks in Russia, show activity concentrations of ^{238}U equal to $Bq.kg^{-1}$, while some phosphate deposits of Tanzania exceed 4000 $Bq.kg^{-1}$. The concentration of uranium in sedimentary phosphate rocks, originated in a marine environment is much higher than those of volcanic and biological rocks (Righia *et al*., 2005; Dissanayake and Chandrajith, 2009; Al-Eshaikh *et al*., 2016). The UNSCEAR indicates 1500 $Bq.kg^{-1}$ as the average concentration of ^{238}U in phosphate deposits of sedimentary origin (UNSCEAR, 1988; 1993; 2000; 2013).

Phosphate rocks contain significant amount of uranium concentration in different phosphate ores around the world. Usually they contain naturally occurring radionuclides such as ^{238}U often 70 to 200 ppm and ^{232}Th together with their decay progeny, among which fourteen alpha and eleven beta emitting nuclides (Dittmar *et al*., 2009) (Fig.4). The presence of these naturally occurring radionuclides can be related to a large ability of phosphates compounds to bound and retain these metals as compounds and/ or complexes, provided that these products possess limited solubility in water (Annunzizta, 2003; IAEA, 2013).

The Iraqi phosphorites are relatively poor in uranium; rarely exceeding 50 ppm, generally ranging from 20 – 45 ppm, corresponding to P_2O_5 content ranging from 11.44 to 21.52 % (Al-Bassam, 2007). A general positive relation exists between uranium and P_2O_5, but the U/P_2O_5 ratio varies within a wide range from about 1.52 to 2.9 (Table 1). The uranium extraction facility at the Al-Qaim Complex never reached maximum production. Through January 17, 1991, when Coalition forces bombed the facility, total production at the site was 170 tons of uranium pentoxide, containing 110 tons of uranium (Albright, and Hinderstein, 2002). The plant that removes these radioactive radionuclides from phosphoric acid was out of work and the potential supply is tied to the economy of phosphate production, in spite of the environmental benefits of removing uranium from the waste stream and/ or products.

Note: *One Becquerel (Bq) is the SI derived unit of radioactivity equivalent to one disintegration incidence per second, 1 Becquerel = 2.7027027 × 10^{-11} Curies. Becquerel is a small activity unit compared to Curie and is more convenient for use in this aspect.*

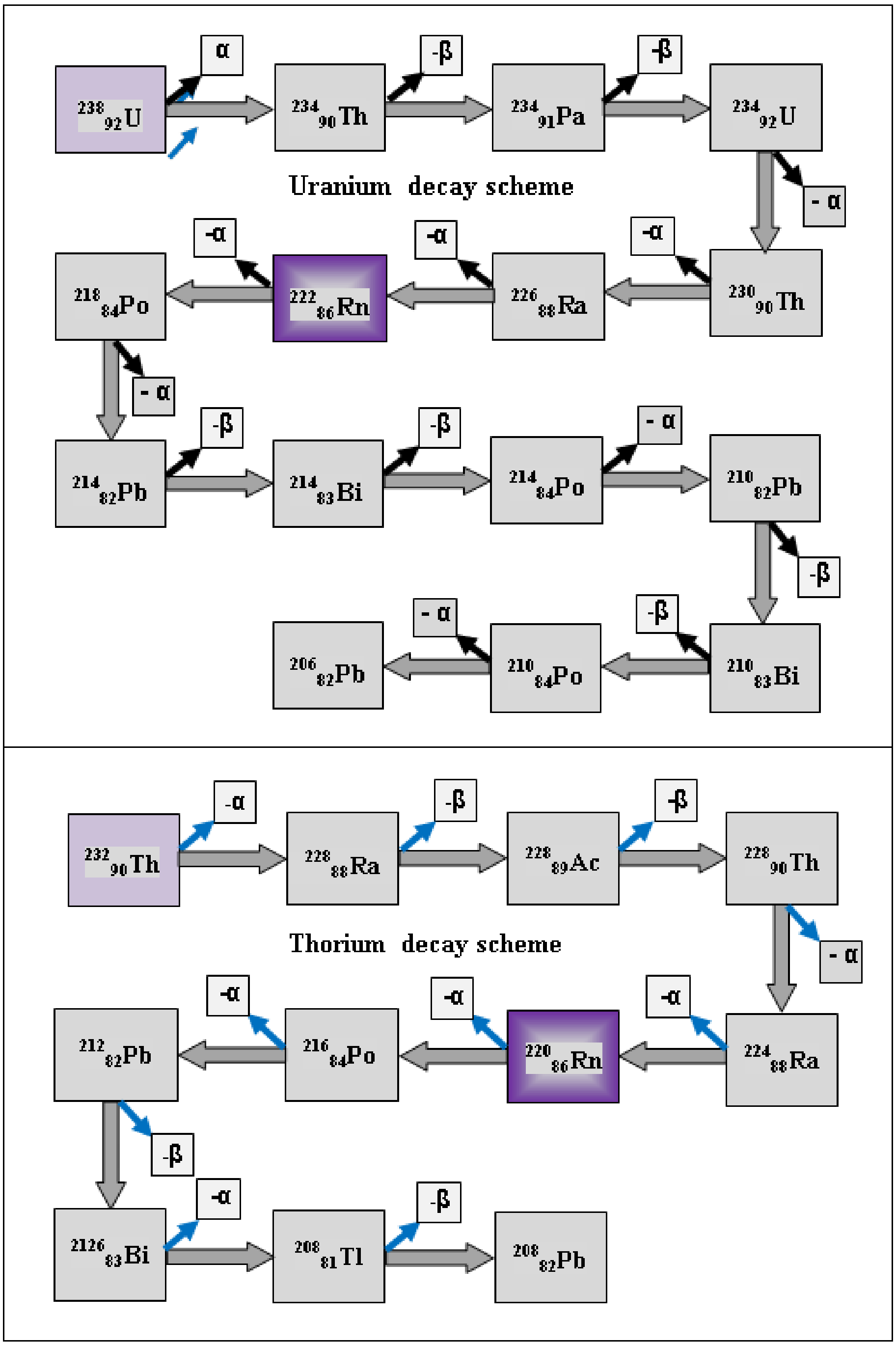


Fig.4: The decay schemes of uranium $^{238}_{92}$U, which give the daughter nuclide as a radioactive radon gas $^{222}_{88}$Rn, and the decay schemes of thorium $^{232}_{90}$Th, which gives the very short-lived thoron nuclide $^{220}_{86}$Rn

Table 1: Uranium and P_2O_5 contents of some Iraqi phosphorite-bearing rock units (Al-Bassam, 2007)

Formation	Member	Location	Uranium (ppm)	P_2O_5 %	U/ P_2O_5
Tayarat	-	Wadi Swab	35	12.88	2.72
Digma	-	Wadi Swab	20	11.44	1.75
Akashat	Traifawi	Marbat	32	21.00	1.52
Akashat	Hirri	Swab	37	21.17	1.70
Akashat	Dwaima	Akashat	42	21.52	1.95
Akashat	Dwaima	Area-1	45	21.94	2.05
Ratga	Damluk (A)	Nhaidain	45	20.84	2.16
Ratga	Damluk (A)	Wadi Akash	35	12.05	2.90

Specific radioactivity of ^{238}U, ^{226}Ra, ^{232}Th, and ^{40}K was measured for selected samples of Iraqi phosphate rocks, fertilizers, and phosphogypsum. Gamma spectroscopy system based on HPGe detector with efficiency of 30% was used for the analysis. The activity: ^{238}U, 53.1 – 682.7; ^{226}Ra, 15.1 – 409.7 Bq/kg; and ^{232}Th, was below detection level and 2.3 Bq/kg, while ^{40}K activity was below detection level in all the samples (Ali and Awad, 2015). The result shows that about 13% of the initial ^{238}U is transferred to the phosphogypsum while its activity in triple superphosphate samples is increased to about 166% of initial ^{238}U in the phosphate rock and about 75% of ^{226}Ra in phosphate rock migrates to the phosphogypsum. The use of monoammonium phosphate fertilizer causes a less effective dose because it contains low concentration of radium. The resulting radiation dose using phosphogypsum as plaster material is 0.27 µSv per year, which may be considered negligible.

NATURAL RADIONUCLIDES IN THE RAW MATERIAL AND THE PRODUCTS

Phosphate rocks emanate radon in significantly higher rates, consequently, they may result in increased radon levels in dwellings and excessive radiation exposure to workers residing on the stack (Lyssandroua *et al.*, 2007; Máduar *et al.*, 2011). Exposure to radon originating from phosphate industry materials through the process is one of the main health concerns associated with their use and disposal. During the chemical treatment of the phosphate rock, the chemical equilibria of uranium and thorium are disrupted and the radionuclides migrate to intermediate, final products, by-products and waste, according to their solubility and chemical properties (Barišić *et al.*, 1992; Sam and Holm, 1995; Ioannides *et al.*, 1997; Saueia *et al.*, 2009).

The natural radioactivity level of phosphate rock, P_2O_5 (29 – 30 %), TSP, MAP, phosphoric acid, NPK, cryolite, zeolite samples obtained from Al-Qaim Complex Plant in Iraq, as well as three organic bitmuse fertilizers were measured by using solid state nuclear track detector, SSNTD (CR-39). Three positions were selected to position these detectors; the upper position which can record radon originated from uranium; the middle detector at the sample surface, records the thoron as well as other alpha emission daughters at the sample surface, and the inside detector records the total alpha emitting daughters. It was found that the lowest radioactivity level values belong to Cold (an organic bitmuse fertilizers) and MAP-3 (Monoammonium phosphate) samples, respectively. The later was obtained from the treatment process of ~ 60 % phosphoric acid (WPA) with phosphate rock in the Wet Process.

Generally, the value of thoron radioactivity (radon emitted by thorium), Ca, indicated by the surface detectors, throughout the chemical process was generally twice that related to uranium. Unusually, zeolite sample showed higher radioactivity level.

The storage of phosphate fertilizer in warehouses results in levels of ^{222}Rn in the air that are over ten times higher than background levels in the same area (Sammet and Eradze, 2000; ANL, 2005; NNDC, 2005; Ochmann, 2005; Sarma, 2013). Radon exhalation from phosphogypsum may pose a health risk to workers on a stack or people living in houses equipped with phosphogypsum panels or build on an area used to be agricultural land treated with phosphogypsum as soil amendment (Lyssandroua *et al*., 2007).The calculated values of the alpha radioactivity level of radionuclides in nine phosphate fertilizers and cryolite and zeolite produced in Al-Qaim Complex Plant for Phosphate Fertilizers and three bitmuse fertilizers available in the Iraqi market were measured and presented in Fig.5 (Al-Nuzal *et al*., 2016).

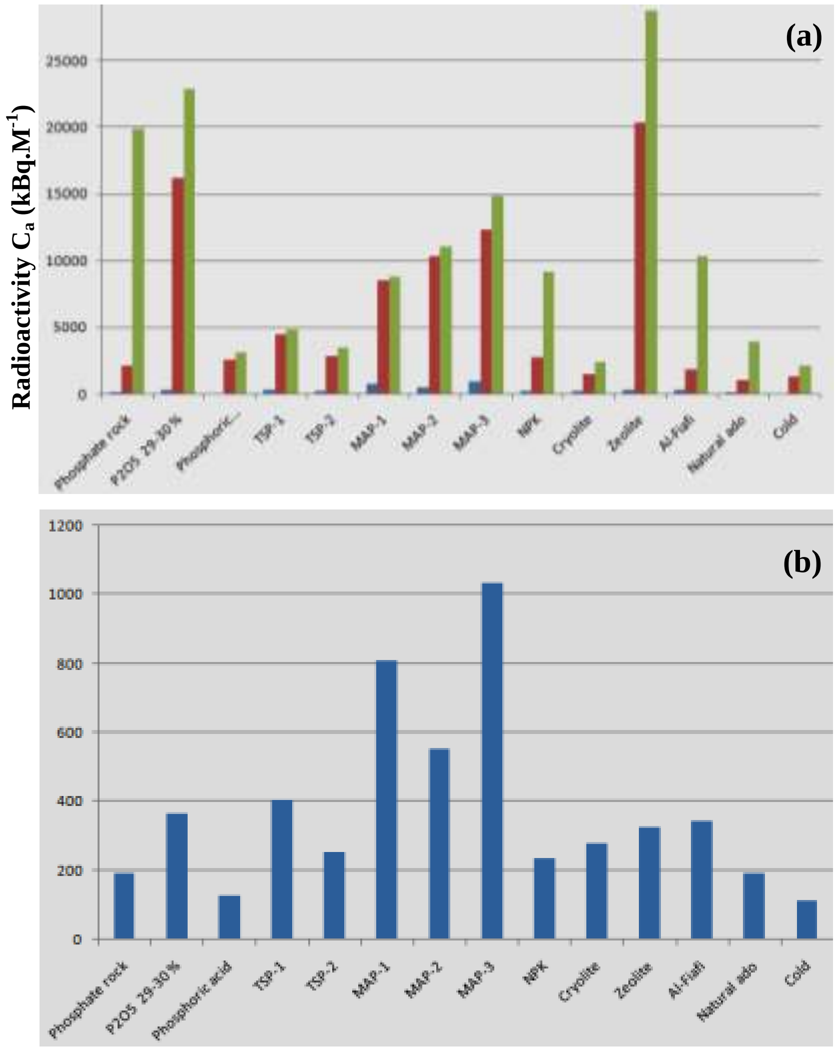


Fig.5: The calculated values of the alpha radioactivity level of radionuclides in nine phosphate fertilizers and cryolite and zeolite produced in Al-Qaim Complex Plant for Phosphate Fertilizers and three bitmuse fertilizers available in the Iraqi market.
a) Relative radioactivity's measured for radon, thoron and all decay daughters in C$_a$ kBq.M^{-3},
b) radon radioactivity originated from uranium only in C$_s$ in kBq.M^{-3}

NATURAL RADIONUCLIDES OF THE WASTE MATERIALS

Slime

The slime is alkaline waste results from the beneficiation of calcined phosphate rock, and is composed mainly of lime in ~ 80% CaO, as well as P_2O_5, 0.0 – 8.0 gm/L and Al_2O_3, 10% (Table 2). The presence of aluminum oxide will hold water and give jelly consistency, with 10 million ton stock. Many main applications of phosphate slime were examined through hydrologic modeling: mixing the surface soil with the phosphate slime and irrigating with polluted (phosphate) water or irrigating with only phosphate water. The results show that there is no significant risk of groundwater pollution with Cd, Cr, Ni, and Sr elements if the phosphate slime or the phosphate water is used for agricultural purposes. The addition of slime in the upper soil layer retards the mobility of the pollutants. The results also show that the mobility of these elements is highly dependent on the applied irrigation dose (Diamantopoulos *et al*., 2011). Due to its rheological properties, phosphate slime can be used as substitution for bentonite in geothermal well drilling fluids (Goode and Sadler, 1975).

Table 2: Some properties of phosphate slime

	Properties/ contents	**Unit or gm/l**
1	pH	12.5 – 12.7
2	Solid	80 – 160
3	CaO	70 – 100
4	P_2O_5	0.0 – 8.0
5	F	1.3 – 2.0

Phosphogypsum

Phosphogypsum (mainly calcium sulphate dihydrate), a waste by-product from Al-Qaim phosphate fertilizers industry is derived from the wet process production of phosphoric acid, according to the following equation:

$$Ca_{10}(PO_4)_6F_2 + 10H_2SO_4 + 20H_2O \rightarrow 6H_3PO_4 + 10CaSO_4.2H_2O + 2HF\uparrow \quad \ldots\ldots\ 2$$

Phosphogypsum, as an unwanted by-product, as well as its low pH of (< 3.5), with a 12 million ton stock, represents a serious problem facing the phosphate industry (Table 3). The wet process is economic but produces large amount of phosphogypsum estimated to be 5 Tons per Ton of produced phosphoric acid. Phosphogypsum is mainly $CaSO_4{\cdot}2H_2O$, but also contains impurities such as H_3PO_4, $Ca(H_2PO_4)_2.H_2O$, $CaHPO_4.2H_2O$ and $Ca_3(PO_4)_2$, residual acids, fluorides (NaF,Na_2SiF_6, Na_3AlF_6, Na_3FeF_6 and CaF_2), sulphate ions, trace metals (e.g. Cr, Cu, Zn and Cd), and organic matter as aliphatic compounds of carbonic acids, amines and ketones, adhered to the surface of the gypsum crystals (Máduar *et al*., 2011; USEPA, 2002).

Table 3: Some chemical properties of phosphogypsum

Components		**(wt. %)**	**Component**		**(wt. %)**
1	CaO	32.94	**8**	MgO	0.45
2	SO_3	44.94	**9**	H_2O	19.19
3	P_2O_5	0.27	**10**	$CaCO_3$	4.29
4	F	0.5	**11**	pH	< 3.0
5	SiO_2	0.45	**12**	Gypsum	80.0
6	Fe_2O_3	0.4	**13**	EC (ds/m)	2.4
7	Al_2O_3	1.05	**14**	Moisture (105 °C)	25

Only 15% of the world's phosphogypsum production is recycled as building material, agricultural fertilizers or soil stabilization amendments and asset controller in the manufacture of Portland cement and the remaining 85% is disposed of without any treatment. This byproduct is usually dumped in large stockpiles exposed to weathering processes, occupying considerable land areas and causing serious environmental damage (chemical and radioactive contamination), particularly in coastal regions.

The physical properties of phosphogypsum are dependent upon the nature of the phosphate ore, the type of wet process employed, the plant operation efficiency, the disposal method, and the age, location and depth of the landfill or stack where the rest is dumped. It is a powdery material that has little or no plasticity and is composed mainly of calcium sulphate dihydrate (> 90% gypsum) and sodium fluorosilicate, Na_2SiF_6 (Rutherford *et al.*, 1994). Due to the residual phosphoric, sulphuric and hydrofluoric acids contained within the porous phosphogypsum, it is considered an acidic by-product ($pH < 3$) (Fig.3). It from filter cake usually has a free moisture content of 25 – 30 %. The free water content may vary greatly, depending on how long it has been allowed to drain after stacking and on local weather conditions. The solubility of phosphogypsum is dependent upon its pH, and it is highly soluble in salt water ≈ 4.1 g/L (Guo *et al.*, 2001). Its particle density ranges between 2.27 and 2.40 g/cm^3 (SENES, 1987) and its bulk density between 0.9 and 1.7 g/cm^3 (Vick, 1977; Keren and Shainberg, 1981; May and Sweeney, 1982).

Phosphogypsum contains relatively high levels of uranium daughters radionuclides naturally present in the phosphate rock. Depending on the quality of the rock source, it may contain as much as 60 times the levels normally found prior to processing, and the most important source of phosphogypsum radioactivity is reported to be ^{226}Ra (Sahu *et al.*, 2014). Wide variation of ^{226}Ra activity concentrations has been observed, and related to the nature of the phosphate rock, the depth of sampling, and some ^{226}Ra migration (Dueñas *et al.*, 2007). Radium, ^{226}Ra emanate radon gas (^{222}Rn), with half-life of 3.8 days, can cause significant damage to internal organs (Lee *et al.*, 2012). USEPA classified phosphogypsum as a technologically enhanced naturally occurring radioactive material, TENORM, and it has been banned from all uses when exceeding a radioactivity 370 Bq.kg^{-1} by the EPA since 1992 (USEPA, 2002). The phosphogypsum piles present a potential threat to the surrounding environment and to the individual occupationally exposed. The proposed use of this material as a raw material in cement production will result in elevated human exposures to radionuclides, particularly radon gas.

Usually phosphogypsum waste is transported and disposed off as an aqueous slurry; its piles can be affected by many weathering conditions among which dissolution/ leaching of the elements naturally present and tidal variations can occur. Dissolved elements may be deposited in nearby soils or transferred to waters and finally to living beings (Reijnders, 2007). The international limit prescribed by the European Atomic Commission (Sunil, 2002) is 500 Bq.kg^{-1}. Hence, phosphogypsum disposal by piling the waste into open environment subjecting it to natural weathering processes without any treatment may lead to radioactive contamination. The distribution of ^{238}U, ^{232}Th, ^{226}Ra, and ^{40}K in soil in and around a phosphate fertilizer industry which stores and disposes its solid waste in the surrounding environment was carefully studied by many workers (Barišić *et al.*, 1992; Guo *et al.*, 2001; Saueia and Mazzilli, 2006; Saueia *et al.*, 2009).

It is worth mentioning that calcined phosphogypsum followed by leaching with acidified aqueous solutions, 4N HCl or 22.1 N H_3PO_4, no ^{226}Ra was detected. The average ^{226}Ra leaching rate of 26.4%, could be cut to 6% when the leached phosphogypsum had previously been calcined at 800 °C. Analysis of a water sample from an area close to the phosphate mine reveals the presence of 0.2 $Bq.L^{-1}$of ^{226}Ra, which is below the radium chemical safety limit for water is 1.0 $Bq.L^{-1}$ (Azouazi *et al*., 2001).

CONTAMINATION OF SOILS WITH RADIONUCLIDES AND HEAVY METALS

Regular application of phosphate fertilizers containing elevated levels of radionuclides and heavy metals (Table 4) causes many-fold accumulative increases in its levels in soils (Akhtar *et al*., 2005a; Akhtar *et al*., 2005b). Concentrations of natural radionuclides in phosphorus fertilizers can be similar to mean concentrations in soil (25 $Bq.kg^{-1}$), or ten-fold higher (Drichko *et al*., 2008). However, during the past 20 years, numerous regulations were revised and finally the normative rate of radionuclides in phosphorus fertilizers was set to:

$$C(U) + 1.5C(Th) \leq 4.0 \text{ kBq kg}^{-1} \quad \ldots\ldots\ldots\ 3$$

where C(U) and C(Th) are the concentrations of ^{238}U(decay daughter is ^{226}Ra) and ^{232}Th (decay daughter is ^{228}Th) in radioactive equilibrium with the other isotopes of the U and Th decay series.

Table 4: Toxic levels of some heavy metals in soil and their health impacts to plants, and humans

Heavy Metal	**Health Impact**			**Reference**
	Humans	**Plants**	**Limits**	
Cd	Affects kidney, liver and GI tract[1].	Chlorosis, necrosis, purple coloration.	4	(Ayeni *et al*., 2012)
Pb	Mental lapse or even death[1].	Dark-green leaves.	50	(Ayeni *et al*., 2012)
Ni	Various kinds of cancer[1].	Decrease in leaf area, chlorosis, necrosis and stunting.	30	(Kukkola *et al*., 2000)
Cr	Allergic dermatitis[1].	Alterations in the germination process, stunted growth, reduced yield and mutagenesis.	1	(Ayeni *et al*., 2012)
Zn	Zinc shortages can cause birth defects[1].	Stunting and reduction of leaves elongation.	50	(Bonnet *et al*., 2000)
Cu	Anaemia, liver and kidney damage, and stomach/intestinal irritation	Chlorosis in plants, yellow coloration, inhibition of root growth and less branched roots.	100	(Kukkola *et al*., 2000)
Fe	-	Dark green foliage. Thickening of roots, brown spots on leaves.	100	(Ayeni *et al*., 2010)
Mn	-	Marginal chlorosis and necrosis of leaves, crinkled leaves.	300	(Ayeni *et al*., 2010)
As	Skin damage, cancer, affects kidney. and central nervous system	-	-	(Wuana, R.A., and Okieimen, 2011)
Hg	Kidney damage.	-	-	(Wuana, R.A., and Okieimen, 2011)

1. Wuana, R.A., and Okieimen, 2011. **2.** Phytotoxic limits in soils (mg/Kg)[2]

The mobility of radionuclides and heavy metals in the soil and their availability to plants depend on the following soil properties: **a)** composition and texture; **b)** pH; **c)** organic matter content; **d)** cation composition of soil solution; and **e)** Ca^{2+} and K^{+} concentrations (Anon, 1999). The toxic levels of some heavy metals in soil and their health impacts to plants were measured (Kukkola *et al*., 2000; Bonnet *et al*., 2000; Ayeni *et al*., 2012), and humans (Wuana and Okieimen, 2011) (Fig.3). Accumulation of radionuclides in human bodies may be harmful if the maximum dose exceeds a situation which may cause serious health problems to human beings (Kukkola *et al*., 2000; Ayeni *et al*., 2010; Lema *et al*., 2014).

The transfer of radionuclides from farm soils to human body may be harmful if the maximum dose is exceeded, a situation which may cause serious health problems to human beings (ICRP, 1990; UNSCEAR, 1988; 1993; 2000; 2013). Radionuclides present in phosphate fertilizers affect the common people and farmers immensely, while direct inhalation of dust of phosphate fertilizers could affect the farmers on agricultural land (Ghosh *et al*., 2008; Hussain and Hussain, 2011)

REMOVING URANIUM FROM PHOSPHORIC ACID

There were 438 operating nuclear reactors depending on uranium fuel in 2007, and the number expected to expand to 523 by 2030 (Table 5) (Walters *et al*., 2008). Consequently the demand for uranium is expected to grow by 2 to 3.5 % per year over the next 20 – 30 years. This increase is related to two major factors: high fossil fuel prices and climate change. Most radionuclides pass into phosphoric acid during wet processing causing selective separation and concentration of naturally occurring radium, uranium and thorium.

Table 5: Top ten nuclear generating countries - 2006

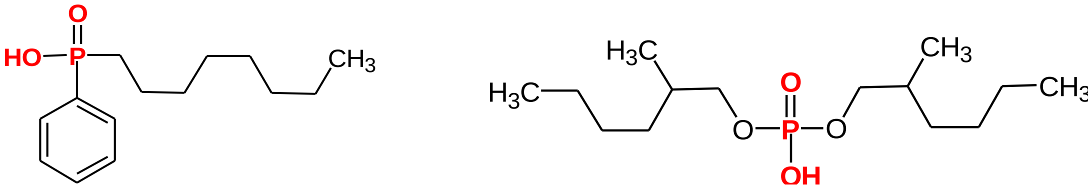
#	Country	Electricity generated (Trillion Wh)
1	United States	787
2	France	429
3	Japan	292
4	Germany	159
5	Russia	144
6	South Korea	141
7	China (+Taiwan)	93
8	Canada	92
9	Ukraine	85
10	United Kingdom	69

About 80% of radium, ^{226}Ra is concentrated in phosphogypsum while nearly 86% of uranium and 70% of thorium end up in phosphoric acid. Uranium in the form of U_3O_8 can be recovered from phosphoric acid which contains 25 – 30 % P_2O_5 produced in wet process. Filter acid is fed to a separate extraction circuit for removal and concentration of the uranium (Sahu *et al*., 2014). After extraction, the uranium-depleted phosphoric acid can then be returned for evaporation and conversion to concentrated phosphate fertilizers (for example DAP, MAP), MGA or technical grade acid. Currently the major producers of mined uranium are Canada, Australia and Kazakhstan. Total production in 2006 was 102 million lbs as U_3O_8 (Fig.5) (Walters *et al*., 2008).

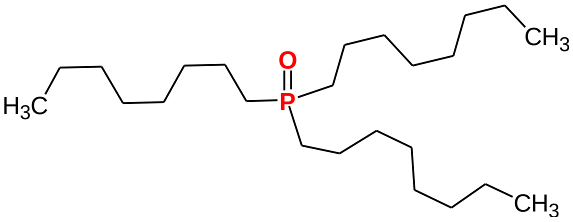
Al-Qaim phosphate fertilizers plant used to have uranium production facility associated with the production and purification of phosphoric acid and was built by a Swiss company (Beltrami *et al*., 2014; and Chauhan, 2003). It had the capacity to extract 28 tons of uranium per year and by the mid-1980`s Iraq had at least 164 tons of yellowcake, obtained and processed in Iraq at Al-Qaim plant. The extraction facility was destroyed during the 1991 Gulf War (Albright and Hinderstein, 2002).

Solvent extraction is the most economic method of extracting uranium from phosphoric acid and the ion exchange method is a promising alternative, but the technology is as yet unproven economically on a commercial scale. Continuing research into uranium recovery from phosphoric acid has resulted in a number of new and more powerful solvents as well as new ion exchange and membrane processes (IAEA, 2013). There are essentially three different solvent extraction processes used, all of which include the use of organic phosphoric acid esters in petroleum compound as solvents, Fig.6. These solvents are:

1. Octyl-phenylphosphoric acid (OPPA) process, which include uranium reduction by ferrous solution, and the extraction with octyl pyrophosphoric acid solution in kerosene. This process was used by IMC and US Phosphoric Products (later Gardinier).
2. Two Stage of di-(2-ethylhexy) phosphoric acid (DEPA) and tri-octylphosphine oxide (TOPO) process, which is the most popular and efficient extraction process, developed by MC/Prayon, Freeport, Wyoming Mineral Corp (WMC).
3. Octyl-phenylphosphoric acid (OPAP) process, in which octyl phenyl acid phosphate (OPAP) is used in the first cycle and DEPA-TOPO in the second cycle. This was used by UNC Recovery Corporation, (URC) and Earth Sciences, Inc. (ESI).



OPPA (Octyl-phenylphosphoricacid) DEPA {Di-(2-ethylhexy) phosphoric acid}



tri-octylphosphineoxide (TOPO)

Fig.6: The most important reagent used to extract uranium (IV) and (VI) oxidation state in petroleum solvents from phosphoric acid

Typically, three to six stages of contact are used to remove 90 – 98 % of the uranium from the acid. Depending on the solvent-to-acid ratio, the concentration of uranium attained in the solvent varies from 0.1 to 0.6 g/L, which is about 1 – 4 times the concentration in the original phosphoric acid. Stripping of the uranium from the solvent is accomplished by contacting the loaded solvent with phosphoric acid (27 – 50 % P_2O_5). The concentration of uranium attained in the strip acid is typically 3 – 20 g/L. The acid uranium solution is reacted with hydrogen peroxide to precipitate a uranyl peroxide salt (UO_2), which is then thickened, washed, dried, and calcined to produce U_3O_8, which is called “yellowcake” (IAEA-TECDOC-533, 1989; Singh *et al*., 2016).

ENVIRONMENTAL IMPACT AND WASTE MANAGEMENT

The radiological hazards for workers and the public due to using and manufacturing processes of the phosphate fertilizers were estimated. The radiation doses due to exposure to the direct gamma rays emitted from a stock of phosphate ore are found to be 0.16 μSv/h while doses caused by using three types of fertilizers are in the range of 0.03 to 2.2 mSv per fertilizer application (Ali and Awad, 2015).

Phosphate fertilizer factories are often shrouded in an acidic haze during the early morning hours when the air is still and moist. The floating minute acid droplets when come in contact with eyes cause burning and watering in eyes. Constant inhalation of the noxious smog causes choking and coughing. Fluoride is a major contaminant in the fumes from phosphate fertilizer plants. When inhaled many fluoride salts react with water and breakdown into hydrofluoric acid and a toxic component. Both of these could cause serious damages to lungs tissues. Environmental issues associated with phosphate fertilizer plants include the following: air emissions, wastewater, hazardous materials (radionuclides, metals, and chemicals), and wastes, which will be highlighted in the following section (Ana and Sridhar 2005).

Al-Qaim Complex Plant was supplied with complete recent technology plant for solid and liquid waste treatment to protect the environment around the complex. It recycles the industrial waste from production units as a side product and mainly the phosphogypsum from phosphoric acid wet process. Future planning should follow the national regulations and regulations of the environmental impact of all units in the entire complex. Among these plans is to build huge gypsum stack with an area of 500000 m^2, with cooperation with the State Company for Industrial Design and Construction (Iraqi Ministry of Industry and Minerals). This landfill will be covered with an efficient plastic insulating material (high density polyethylene, HDPE). The heap capacity is designed to deal with the expected amount of waste after the rehabilitation of the complex units to the designed production capacity, such a way no contaminated effluents can escape to the rivers (Al-Mohammadi, 2015). However the following sections will give the wider vision for the most important waste and the right waste management procedure and some guidelines to mitigate or reduce their impacts on the environment.

▪ Management of Waste from Air Emissions

Dust emissions, containing water-insoluble fluoride, may occur during the unloading, storage, handling and grinding of the phosphate rock, which is transferred to storage and grinding sections by conveyor belts or trucks (Mishra *et al.*, 2010). Sulphuric acid plant usually produces sulphur dioxide and acid mist. Emissions of gaseous hydrofluoric acid, HF, and silicon tetrafluoride, SiF_4 generated from acid treatment, granulation and drying, occur with limited amounts of organic compounds (including mercaptans) having bad odor. Recommended air emissions management measures include the following:

1. Phosphate rock with best P_2O_5/CaO ratio, low fluorine content, and better physical quality to minimize the amount of acid needed in the wet production process, reduce emissions into the environment and increase the possibility of phosphogypsum reuse.
2. Recover dust from phosphate rock grinding through use of properly operated and maintained fabric filters, ceramic filters, and/ or cyclones. Treat gaseous fluoride emissions using scrubbing systems.
3. The use of fabric filters or high efficiency cyclones and/ or fabric filters rather than a wet scrubbing system should be considered to treat exhaust air from neutralization, granulation,

drying, coating and product coolers and equipment vents, in order to avoid creation of additional wastewater. Filtered air should be recycled as dilution air to the dryer combustion system.

4. Select proper size of screens and mills (e.g. roller or chain mills), and covered conveyor belts and indoor storage.
5. Use of direct granulation help to reduce the levels of dust emissions compared with curing emissions from indirect granulation. If indirect granulation is selected, the curing section should be an indoor system with vents connected to a scrubbing system or to the granulation section.

- **Management of Wastes from the Process**

During the processing of phosphate rocks, many side products are emitted during handling from grinders and pulverizers, and the resulting dusts are emitted to the air. Most of the evolved fumes contain fluoride compounds such as silicon tetra fluoride and hydrogen fluoride (Ana and Sridhar, 2005). Sulphuric acid plant has two principal air emissions, sulphur dioxide and acid mist, while phosphoric acid plants generate dust and fumes containing hydrofluoric acid and silicon tetra fluoride. The major solid Wastes is phosphogypsum and is often disposed of as slurry to storage/ settling pond or in form of waste heaps (Haridasan *et al*., 2001).

- **Management of Wastewater**

Effluents from phosphoric acid plants usually consist of discharges from the vacuum cooler condensers and the gas scrubbing systems used for condensation and cleaning of vapors from process operations. Condensed acidic vapors may contain fluorine and small amounts of phosphoric acid. Water from the slurry used to transport phosphogypsum, the by-product from wet phosphoric acid production, may be released as effluent if it is not recirculated back into the process. Drainage from material stockpiles may contain heavy metals (such as Cd, Hg, and Pb), fluorides, and phosphoric acid. Specific emissions to water from the thermal process of phosphoric acid production may include phosphorus and fluorine compounds, dust, heavy metals, and radionuclides (e.g., ^{210}Po and ^{210}Pb). Recommended effluents management measures include the following:

1. Selection of phosphate rock with low levels of impurities to produce clean gypsum and reduce potential impacts from disposal of gypsum;
2. Considering dry systems to minimize air pollution to reduce wastewater generation. To reduce fluoride emissions, the installation of scrubbers with suitable scrubber liquids may be necessary.
3. Recovering released fluorine released from the reactor and evaporators as a commercial by-product (fluosilicic acid).
4. Scrubber liquors should be disposed of after neutralization with lime or limestone to precipitate fluorine as solid calcium fluoride, if the fluorine is not to be recovered. Minimize contamination of the scrubber effluent with phosphorus pentoxide P_2O_5 using entrainment separators.
5. Recycle water used for the transport of phosphogypsum back into the process following a settling step.
6. Minimize contamination of the scrubber effluent with phosphorus pentoxide (P_2O_5) by conveying vapors from vacuum flash coolers and vacuum evaporators to a separator to remove phosphoric acid droplets.
7. Additional phosphate removal can be achieved by applying magnesium ammonium phosphate (struvite) or by calcium phosphate precipitation.

8. The extraction of the H_3PO_4 up to 95% by reactive extraction with an organic solvent should be considered.

- **Management of Phosphogypsum**

Phosphogypsum is the most significant by-product in wet phosphoric acid production (Mishra *et al.*, 2010). It contains a wide range of impurities (residual acidity, fluorine compounds, trace elements such as mercury, lead and radioactive components (IFC, 2007). These impurities and considerable amounts of phosphate might be released to the environment (soil, groundwater and surface water). The discharge of phosphogypsum on earth surface deposits is a potential source of enhanced natural radiation and heavy metals, and the resulting environmental impact should be considered carefully to ensure safety and compliance with environmental regulations. Depending on its potential hazardousness (e.g. whether it emits radon) phosphogypsum may be processed to improve its quality and reused (e.g. as building material). Possible options include:

1. Production of cleaner phosphogypsum from raw materials (phosphate rock) with low levels of impurities by the use of repulping is important.
2. If phosphogypsum cannot be recycled due to the unavailability of commercially and technically viable alternatives, it should be managed as a hazardous or non-hazardous industrial waste, depending on its characteristics, according to the guidance in the General EHS guidelines. Alternative management procedures may include back filling in mine pits, dry stacking 15, and wet stacking. Di-hemihydrate recrystallization process use is preferred with double stages filtration.

Many suggestions can be presented to deal with the environmental management of phosphogypsum and slime in Iraq. Among these is the reuse of a mixture of both of the above for the treatment of caves of Al-Mishraq sulfur mines, injected in acid location seepages at Al-Qaim, sinkhole filling at Mosul Dam and in cement production. In the meantime their mixture could be used for the production of block and brick covering 1/4 of a million medium resident units, also as a sub-base for a road of 15000 Km length and 10 m width (Al-Youzbakey and Al-Dabbagh, 2007; Mustafa *et al*., 2016). Phosphogypsum could also be used as sub-base for roads, cement production and/ or as a cementing material, in agriculture, and chemicals production.

- **Noise Management**

Noise is generated from large rotating machines, including compressors and turbines, pumps, electric motors, air coolers, rotating drums, spherodizers, conveyors belts, cranes, fired heaters, and from emergency depressurization. Guidance on noise management is provided in the General EHS Guidelines. Blasting and handling of raw material during mining operations is another source of noise.

- **Guidelines for Future Phosphate Fertilizers Plants**

Adoption of pollution prevention measures can yield both economic and environmental benefits. Emission of SO_2 level into the atmosphere can be minimized by adopting double contact, double absorption process. Guideline values for process emissions and effluents in this sector are indicative of good international industry practice as reflected in relevant standards of countries with recognized regulatory frameworks. The ideal air emission and effluent level should be achieved, as shown in Table 6. These guidelines are assumed to be achievable under normal operating conditions in appropriately designed and operated facilities through the application of pollution prevention and control techniques discussed in the preceding sections of this document (IFC, 2007).

Table 6: Air Emissions and effluent guidelines for phosphate fertilizers plants (IFC, 2007)

Air Emissions		
Pollutant	**Units**	**Guideline Value**
Phosphoric Acid Plants		
Fluorides (gaseous) as HF	mg/Nm^3	5
Particulate Matter	mg/Nm^3	50
Phosphate Fertilizer Plants		
Fluorides (gaseous) as HF	mg/Nm^3	5
Particulate Matter	mg/Nm^3	50
Ammonia	mg/Nm^3	50
HCl	mg/Nm^3	50
NO$_X$	mg/Nm^3	500 nitrophosphate unit
		70 mix acid unit
Effluents Guidelines		
pH	S.I	6-9
Total Phosphorus	mg/L	5
Particulate Matter	mg/Nm^3	5
Fluorides	mg/L	20
TSS	mg/L	50
Cadmium	mg/L	0.1
Total Nitrogen	mg/L	15
Ammonia	mg/L	10
Total Metals	mg/L	10

CONCLUSIONS AND RECOMMENDATIONS

Phosphate fertilizers production and their agricultural use have gained bad reputation for their impacts on the environment and human health. Ongoing additions of these fertilizers to agricultural land will result in accumulative impacts to the land and agricultural products. These impacts can potentially result in elevated levels of many of these contaminants in crops grown on that land. Chemical fertilizers cause increase in annual exposure dose which should be taken into account with the continuing use of chemical fertilizers. The high content of uranium radionuclide in the Iraqi granular NPK fertilizer must be taken into account. It is recommended that, whenever possible, radioactive elements and heavy metals should be removed before they are used for farming. The process of removing radionuclides including uranium, thorium, and other heavy metals is very important action to mitigate the contamination of agricultural soil and waters in Iraq. From the point of view of general conservation also, it is far better to extract uranium from the rock than spread it over the fields where it will never be recovered. The cost of extraction and marketing uranium together with phosphoric acid purification compensates much of the cost of purifying the acid. In other words, for a fertilizer plant recovering uranium, the resulting clean acid could be used for a number of downstream products, while much of the cost of producing such clean acid would be borne by uranium which is a good value product today.

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