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Valorization of phosphate waste rocks and sludge from the Moroccan phosphate mines: Challenges and perspectives

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Abstract

Sedimentary phosphate mines produce millions of tons of waste rocks during their open-pit mining. In addition, during ore phosphate beneficiation, fluorapatite is separated from associated gangue minerals by a combination of successive mineral processing steps that involve crushing / screening, washing, and flotation. These operations generate large volume of tailings (called phosphate sludge) that are deposited in large surface ponds and waste rocks stockpiled within the mining site. The potential reuse of these phosphate mine by-products (waste rocks and sludge) has been investigated in the last 10 years.
The first investigated option consisted in using the alkaline waste rocks (APW) to control the acid mine drainage (AMD). Indeed, these alkaline mine wastes contain significant quantities of calcite (46 wt%) and dolomite (16 wt%) that help in neutralizing the acidity generated by the wastes from the closed Kettara mine , located near Marrakech, Morocco. The addition of 15 wt% APW to the coarse Kettara tailings produced leachates with significantly lower acidity and metal loads in comparison to the un-amended control sample. Secondly, the efficiency of APW was assessed in the laboratory as an alternative alkaline material for passive AMD water treatment. In semi-arid climate, the oxic passive treatment has been proven to be the most suitable. The pH of the water and its quality were significantly improved. As a third option, the hydrogeotechnical characterization of original and screened phosphate limestone waste rocks as well as the phosphate sludge showed their suitability for use as a component of store-and-release (SR) covers for industrial mine site reclamation. Lab tests (columns) and field tests (instrumented columns and experimental cells) showed that water infiltration can be controlled, even for extreme rainfall events (150 mm/day), by 1 m thick of a SR cover made with APW. Further research is currently being investigated around the recycling and valorization of phosphate sludge from phosphate mines as ceramics. Furthermore, the overburden of the phosphates sedimentary basins are mainly composed of marls; limestones blocks; silex bed; silex nodule; marls and clays; silicified limestone; which have a significant reuse potential as marble-mosaic floor, mortars and concrete, and natural stone products slabs for floors and stairs.

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1. Introduction

In general, the mining industry is facing many environmental challenges resulting from the huge quantities of wastes that mines engender such as waste rocks, concentrator tailings and phosphate sludge. These wastes are deposited or stockpiled within the mine site and constitute a potential source of pollution because of their chemical characteristics and grain size.

Morocco, with its large share of the world's phosphate reserves, is the leading exporter of phosphate and its derivatives. The country's global market share is more than 30%. Morocco's currently known reserves are sufficient to meet several centuries of demand. A large tonnage of the phosphate reserves are sedimentary type rock containing appreciable amounts of carbonate minerals. Phosphate deposits in Morocco occur in three areas - the Khouribga area (Oulad Abdoun Plateau), the Gantour area (Youssoufia area) and Layoune-BouCraa. Exploitable phosphate beds were deposited in the Upper Cretaceous, Paleocene and Eocene.

The open pit mining of phosphate deposits in the OCP mines consists of cutting the deposit in panels; the panel is then cut in trenches of 40m large into a block with 40mx100m [1]. These open-pit phosphate mines produce large quantities of rocks that are excavated without sorting and stored all around the mines (stockpiles).The phosphate deep layer below the ground surface is open-pit mined whereas the soils on top of(overburden) are halded to the side in spoil haldes. The phosphate ore, high in clay content, is transported by train or trucks to the beneficiation plant (Fig. 1). As example, the "Exploitations Minières du Gantour Company" 'Recette VI' site (Fig. 2) alone produces million tons of waste rocks which constitute the overburden removed systematically to reach the phosphate ore. Trucks transport the ore to a dry screening unit for phosphate separation (fines) and disposal of the oversize (>40 mm) low-grade material on site. Thus, as for the other OCP phosphate mines, the "Recette VI" mine produces two types of wastes: the overburden waste rocks and the wastes produced by the pre-concentration (screening) process. Large quantities of these wastes are stockpiled in waste rock dumps.

During ore phosphate beneficiation (washing and flotation unit), fluorapatite is separated from associated gangue minerals (clay, limestone, silicates,…) by a combination of various mineral processing units involving crushing and screening, washing, and/or flotation. Enrichment plants of phosphate ores generate huge amounts of sludge and flotation tailings deposited together (what is called phosphate sludge) in basins over an area of several dozen hectares. This represents a significant management challenge for the operator.

In order to be used as crop nutrients or animal feed, apatite (the main phosphate mineral) must be converted to a water-soluble form. The OCP production facilities in chemical complexes coastal sites at Safi and Jorf Lasfar (Fig. 2) utilize a wet process in which the phosphate concentrate is mixed with sulfuric acid and filtered to make phosphoric acid which is the starting point in the production of most phosphate derivative products. Phosphoric acid is used for ferilizer production and purified phosphoric acid used in the food industry. So, the main reaction of phosphate with sulfuric acid produces phosphoric acid to be valorized and phosphogypsum (dihydrate or hemihydrate) to be rejected. The weight of gypsum per weight of phosphate rock concentrate is about 1.5. This byproduct is not addressed in this paper.

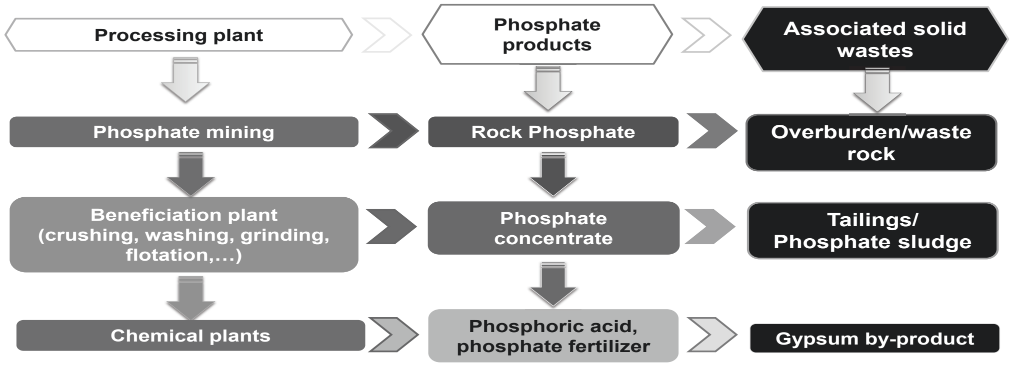

Fig. 1.Schematic diagram of processing phosphate rock: products and associated solid wastes.

The main environmental implication of the phosphate waste rocks dumps and phosphate sludge impoundments is the physical and aesthetic changes on the environment surrounding as well as the huge footprints of the mine site areas. Given the high level of carbonate contents in these waste rocks and phosphate sludge, they are not polluting water (surface and underground) as it was proven by recent research works [2,3, 4]. However, the accumulation of these phosphate by-products is a serious problem in terms of storage capacities. They reduce arable lands, modify topography and disfigure the landscapes. In addition during open pit mining, topsoils are lost because they are mixed with other materials in waste rocks and overburden piles since no waste sorting is implemented.

Successful mine closure becomes a complex and multi-faceted process, particularly in the case of mines that have been in operation for several decades without a specific closure plan that integrates mining activities with closure goals and requirements. One of the major challenges facing OCP is the use of appropriate technologies to reduce negative environmental impacts of phosphate mine wastes and improve site rehabilitation techniques. A commitment to leading practice in a sustainable development framework is critical for a mining company to gain and maintain its 'social license to operate' in the community. In addition, successful implementation of rehabilitation provides credibility for the mine operator towards its partners, investors and competitors. Rehabilitation planning should be undertaken in the early stages of a project development and frequently adapted in the context of the overall mine site closure objectives. During mining, research and field trials enable the rehabilitation scenario to be modified to reflect site specific parameters.

Approaching the ecological and cost-effective techniques for the rehabilitation of an open-cast phosphate mine sites in Morocco during the post-operational phase present some challenges. Thus, the recycling of the phosphate waste rocks and/or phosphate sludge could be a useful alternative to limit their negative impacts. This could permit to the OCP to switch from a linear industrial patron to the circular one, with care of the environment and searching additional value of some of the by-products. OCP S.A group is in the process of developing an environmental strategy to fit perfectly into the challenges of sustainable development by considering the phosphate waste management as one of their priority projects. However, to date, there is no sufficient research done on the potential re-use of wastes after open-pit exploitation.

The potential reuse of phosphate mine by-products has been investigated by the authors and their teams since 2008. The studies were carried out with support of the IDRC (Canada) Research Chair in Management and Stabilization of Mining and Industrial Wastes (www.gesrim.com). The chair is a joint project between UCA (University Cadi Ayyad, Morocco), UQAT (University of Quebec in Abitibi-Temiscamingue, Canada) and the Centre de Développement de le Région de Tensift (CDRT-Marrakech).

This paper aims in showing some of promising results obtained from investigations already done on the reuse of phosphate rock wastes and phosphate sludge. Calcareous phosphate rocks from "Recette VI" mine were investigated as both an amendment and a hydrogeological system cover called store divert and release cover (SDR), at the Kettara abandoned mine site to control Acid Mine Drainage (AMD) problems [2, 4]. Secondly, the efficiency of phosphate waste rocks was also assessed in the laboratory where used asan alternative alkaline material for passive

AMD water treatment. Experiments were done in both anoxic and oxic conditions[3].The potential use of washing sludge, from Youssoufia phosphate mine in the field of ceramic have been also assessed [5].

Since the 1970s, mine wastes management has made quite a large progress in the developed countries by replacing the conventional methods of storage with more integrated management techniques *in situ*; which reduces the environmental damage of mining. The main practices inside mine sites are cemented mine backfilling; construction of roads and embankments with waste rocks; rehabilitation techniques using wastes as a barrier to water and/or oxygen, etc.). However, outside mine sites reuse remains very limited, even more and more incentive measures take place in many developing countries. Indeed, some mine wastes (with little or no metal sulfide or sulfur free) may have characteristics similar to the aggregates used in civil engineering (embankments, mortars, concretes, road engineering, etc.) [6].Some perspectives on the reuse of waste rocks and phosphate wash sludge will be discussed in this paper. The reuse of these wastes as concrete, mortars and in road engineering, as aggregates treated with alternative hydraulic binders is a promising ways to be adopted help reduce the risks of pollution from phosphate mines on the environment.

2. Materials: sampling and characteristics

Phosphate waste rocks are sampled in the dumps situated near, the "Recette VI" open pit phosphate mine, belonging to "Mining Operations of Gantour, OCP group"(Fig.2). The investigated sample of phosphate sludge was taken from the storage ponds belonging to a Moroccan phosphate plants (Gantour, Youssoufia). Sampling was carried out in order to have representative specimens [5]. Samples of marls; limestones blocks; silex in the form of large blocks are collected at the Bengurir phosphate mine.

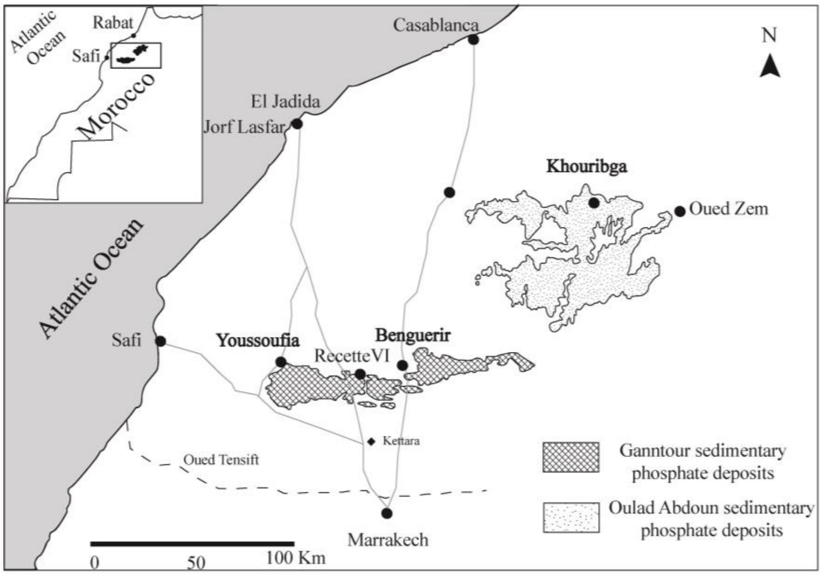

Fig. 2.Local situation of Gantour and Oulad Abdoun sedimentary phosphates deposits.

Materials to be characterized were ground using an agate mortar. The obtained powder was analyzed using a PerkinElmer Optima 3100 RL ICP-AES after a total $HNO_3/Br_2/HF/HCl$ digestion. Dilute HCl was used to extract sulfate from the samples, and the solution obtained was analyzed by inductively coupled plasma and atomic emission spectrometry (ICP-AES). Total carbon and total sulfur content were measured using an ELTRA CS 800 sulfur/carbon analyzer. Pulverized samples were analyzed by x-ray diffraction (XRD) analysis using a Bruker AXS D8 Advance diffractometer equipped with a scintillation detector and Co Kα radiation (λ= 1.54 Å). The data were collected at 5-70° by steps of 0.005° and a counting time of about 0.5 s per step.

Grain size analysis was performed in two steps: First, by sieving for the granular fractions with diameter greater than 80 µm and second, by using a Malvern Mastersizer laser particle size analyzer for the grain fraction smaller than 80 µm. The specific gravity was estimated with a Micromeritics Accupyc 1330 helium pycnometer. Standard tests for liquid limit, plastic limit, and plasticity index were performed according to ASTM D 4318-10 (2010). Optimum moisture content and maximum dry density of each SR material were also determined using the modified Proctor compaction test in accordance with ASTM D 1557-12 (2012).

The chemical, mineralogical compositions and some of the physical characteristics of phosphate wastes are presented respectively in Tables 1 and 2.Theobtainedresults show high contents of CaO (43.0 wt%), P_2O_5 (16.9 wt%), SiO_2 (11.6 wt%), MgO (3.23 wt%), C (5.20 wt%), and potential contaminants, such as F (1.73 wt%), Cr (137 ppm), and Zn (195 ppm). However, Hakkou et al. [2] showed through kinetic tests that there is no significant contaminant generation from the phosphate limestone wastes. The chemical analysis confirms the mineral composition (Table 1) with four main phases: calcite, fluorapatite, dolomite and quartz.

Table 1. Chemicaland mineralogical composition of the phosphate waste.

Major elements (wt%)														
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	P_2O_5	F		S		C	
11.6	0.89	0.38	43.0	3.28	0.46	0.12	0.07	16.9	1.73		0.30		5.20	
Traces elements (ppm)														
As	Ba	Be	Cd	Cl	Cr	Cu	Mn	Ni	Pb	Sr	U	V	Y	Zn
8	79.2	0.82	33	108	137	31.8	31.8	34	13	769	00	56	97.8	195
XRD mineralogical quantification, wt%. (within 0.5% error)														
		Calcite ($CaCO_3$)	Dolomite ($CaMgCO_3$)	Apatite ($Ca_{10}(PO_4)_6F_2$	Quartz (SiO_2)									
		40.7	25.2	25.9	8.2									

The chemical and mineralogical composition of the phosphate sludge are reported in Table 2. The sludge consisted of quartz (17 wt.%), calcite (15 wt.%), dolomite (7 wt.%), fluorapatite (44 wt.%) and a smectite clay mineral (7 wt.%).

Table 2. Chemical and mineralogical composition of the phosphate sludge.

Major elements (wt%)														
SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	P_2O_5		Cr_2O_3		V_2O_5		
22.8	2.48	0.91	34.2	4.11	0.77	0.4	0.17	14		0.1		0.04		
Traces elements (ppm)														
Bi	Cd	Co	Cu	Li	Mo	Ni	Pb	Sb	Se	Sn	Sr	U	Y	Zn
<20	35	<4	37	<7	8	84	20	<10	<30	<20	670	82	86	375
XRD mineralogical quantification, wt%. (within 0.5% error)														
		Calcite	Dolomite	Fluoroapatite	Quartz		Moisture (%)							
		15	7	44	17		40							

The main geotechnical properties of the phosphate waste rocks and phosphate sludge are summarized in Table 3.According to USCS classification, the grain-size distributions of the phosphate limestone waste are typical of non-plastic silty sand with gravel, whereas the distributions of the phosphate sludge are typical of low-plastic lean clay with sand (Table 3). For more details, the reader can consult [2, 4, 9].

3. Valorization of phosphate waste rock to control Acid Mine Drainage

In order to control the AMD generated at Kettara pyrrhotine closed mine [7], Ouakibi et al. [3,8] tested different

Table 3. Hydro-geotechnical properties of the phosphate rock waste and phosphate sludge

Classification (USCS)			Particle Size Distribution										
Material	Symbol	Name	Gravel (%)	Sand (%)	Silt (%)	Clay (%)	*Gs*	*LL*	*PL*	*PI*	Υ_{dmax}(kN/m3)	W_{opt} (%)	k_{sat} (cm/s)
Phosphate sludge	CL	Lean clay with sand	0.0	15.5	72.5	12.0	2.78	35.4	26.0	9.4	16.6	17.5	$1.9x10^{-6}$
Phosphate rock waste	SM	Silty sand with gravel	15.1	48.5	28.3	7.1	2.85	26.4	25.6	0.8	17.1	14.5	$3.7x10^{-5}$

Gravel (>4.75 mm), Sand (4.75-0.075 mm), Silt (0.075-0.002 mm), Clay (<0.002 mm), GS gravity specific, LL Liquid Limit, PL Plastic Limit, PI Liquid Index, ϒdmax Proctor maximum dry unit weight, wopt Proctor optimum water content, ksat saturated conductivity hydraulic (geometric mean)..

passive treatment systems that prevent water from reacting with mine tailings. Lab tests indicated that in semi-arid climate, the oxic passive treatment has been proven to be the most suitable. The initial pH of DMA increased from 3 to 5 and 6.5 during tests in batch and from 6.5 to 8 for the column tests. For batch tests, both in oxic and anoxic conditions, a significant decrease in the concentration of Fe (500 to 120 mg/L), Al (160 to 1.7 mg/L), Zn (15-5 mg/L) and Cu (23 to 0.002 mg/L) was noticed. In the column tests, Al and Cu decreased (from 177 to 2.5 and from 26 to 0.002 mg/L, respectively), while Fe decreased significantly (618 to 300 mg/L). Therefore, the availability and low cost of sterile phosphate limestones make its use in passive treatment of AMD potentially efficient.

Additional testing by Hakkou et al. and Bossé et al.[2, 4,9] confirmed the ability of phosphate waste rocks to prevent water infiltration and oxidation of mine wastes at Kettara. On the basis of these important findings, the research team designed a store-and-release (SR) cover that used capillary barrier effects and that prevented water percolation by storing and evaporating water during wet and dry climatic periods.

A field investigation was conducted on the effectiveness of store-and-release (SR) covers made with different phosphate mine wastes to reduce water infiltration and to control AMD. Four instrumented experimental cells and one inclined field experimental cell were constructed with a SR layer placed over a capillary break layer made of acidic coarse-grained materials. Cover performance and hydrogeological behavior under natural and extreme climatic conditions were monitored using lysimeters, tensiometers, suction and soil moisture sensors [4, 9, 10]. The important finding of this research is that the store-and-release cover made of phosphate limestone waste can limit deep water infiltration even during extreme simulated rainfall (150 mm/d). Not only is this method effective, but it is also cost-effective as it uses a phosphate mining by-product that is widely available in Morocco to control AMD at the Kettara mine.

After five years of laboratory and field research, the rehabilitation scenario of the abandoned Kettara mine) is almost finished. The reclamation business plan is believed to be cheap (0.7 million US$ to restore 30ha) because of the use of phosphate by-products as material for the covers. The findings could be applicable to other mines located in semi-arid climates, especially those in others countries in Africa.

4. Assessment of the potential use of phosphate waste rocks and sludge in ceramics

Considering the mineralogical composition of the two raw materials (Tables 1 and 2), it appears that they may be suitable for ceramic manufacturing. The presence of carbonates serves to stabilize the acid-base environment and can lead to refractory monolithe formation which is required for thermal resistance. Quartz allows extending the sintering temperature range and participates in the melt formation which ensures the mechanical strength for the ceramic bodies. However, since the plasticity index (Table 3) of the raw materials is very low respectively Ip= 0.8 for phosphate waste rocks (not plastic) and Ip= 9.4 for phosphate sludge (slightly plastic), additives are required in order to improve the targeted plasticity. Local clay from phosphate mines containing plasticizing clay minerals could be a very good solution for both the proximity and compatibility with the base materials for ceramic manufacture.

The potential use of phosphates sludge in the field of ceramic has been assessed by Loutou et al [5]. The preliminary results showed that OCP sludge can be used for various applications such as lightweight aggregates to improve its plasticity by mixing them with local clay material.

Thermal behavior and physical properties of phosphate sludge originated from Moroccan phosphate industries were investigated at the range 900-1200 °C using X-ray diffraction, scanning electron microscopy, thermal analysis and Fourier-transform infrared spectroscopy. The sludge consists chiefly of quartz, dolomite, calcite and fluroapatite. Because of its low plasticity, the sludge was blended with swelling clays (up to 30 %.mass). Properties of the resulting mixture (shrinkage, density, water absorption and compressive strength) were measured as a function of temperature and clay addition. The results showed that the formation process involved lime of decomposed carbonates and breakdown products of clay minerals. The measured properties were mainly controlled by temperature, and the effect of clay addition was less regular. The mechanical properties (compressive strength) were enhanced by the addition of the swelling clay (3.7 MPa for clay sludge free and up to 15 MPa when mixed). Lightweight aggregates (d=1.44-3.02 g/cm^3) were obtained.

5. Valorization of waste rock and phosphate sludge from phosphate mines as aggregates in civil engineering: natural stone products, embankments , mortars, concretes

The Moroccan phosphate series is characterized by a wide variety of lithological formations both in space and age dimensions (from the Maastrichtian to the Lutetian) [11].The Moroccan phosphate deposits occur as sub-horizontal beds along with limestone, marls, and clays in which there are 3 to 4 beds up to 2.5 m thick.The phosphate formations have an age Upper Cretaceous – Eocene and consist of alternating layers of phosphate separated by silica-carbonate layers (Fig. 3). The most interesting series is located in Oulad Abdoun basin (Fig.2). According to Kchikach et al [12] the phosphate series (Fig. 3) begins by Maastrichtian where alternating limestone horizons, phosphate marls and sand-phosphate. The Paleocene is represented by un-cemented phosphates and limestone phosphates. It ends with a layer of limestone with coprolite and siliceous nodules. This layer constitutes a horizon of reference during the mining exploitation. It is surmounted by a big Eocene formation which consists of alternating layers of un-cemented phosphates with coarse grains, regular fine layers of phosphatic marl limestone and siliceous horizons. The phosphate series ends with an Eocene bar of limestone with shells called "bar with thersites''. The overburden of the phosphates sedimentary basins is mainly composed of marls; limestones blocks; silex bed or in the form of larger blocks; silex nodule; marls and clays; silicified limestone (Fig. 3).

Even though they have characteristics similar to the aggregates used as raw construction materials in many application the phosphate waste rocks are considered currently as mine wastes. It seems a priori that phosphate mine by-products beneficiate from promising geotechnical properties and are considered to be an excellent alternative secondary raw material to conventional borrow pits. The rocks contained in the overburden of the phosphates sedimentary basins may have a significant potential reuse as marble-mosaic floor, mortars and concrete, natural stone products, slabs for floors and stairs, wallboard, and roofing tiles. This practice may limit the quantities of wastes to be stored on the surface and will help create new sectors consisting of the exploitation of these mine wastes in national road networks, and ornamental stones (Fig.3). Significant savings in the exploitation of non-renewable natural resources (sand, gravel, soils …) are expected to be made and a response should be given to the increasing demand from major construction projects in Morocco (roads , highways, bridges , etc.) in terms of raw materials.

During the initial planning of mine exploitation the focus should be on identifying parts of the overburden strata containing these rocks. It is important that leading practice be flexible and innovative in developing solutions that match site-specific requirements. Issues that need to be systematically addressed include:

- Characterization of topsoils and overburden should start as early as the exploration phase and continue through the pre-feasibility and feasibility phases as a basis for mine planning
- Selection of the soil horizons to be conserved
- Identification and selection of overburden strata overlying the phosphate layers containing limestones blocks; silex bed, silicified limestone; which have a significant potential reuse as marble-mosaic floor, mortars and concrete, natural ornamental stone products, slabs for floors and stairs, wallboard, and roofing tiles
- Include the exploitation of the interesting overburden lays with the phosphate extraction

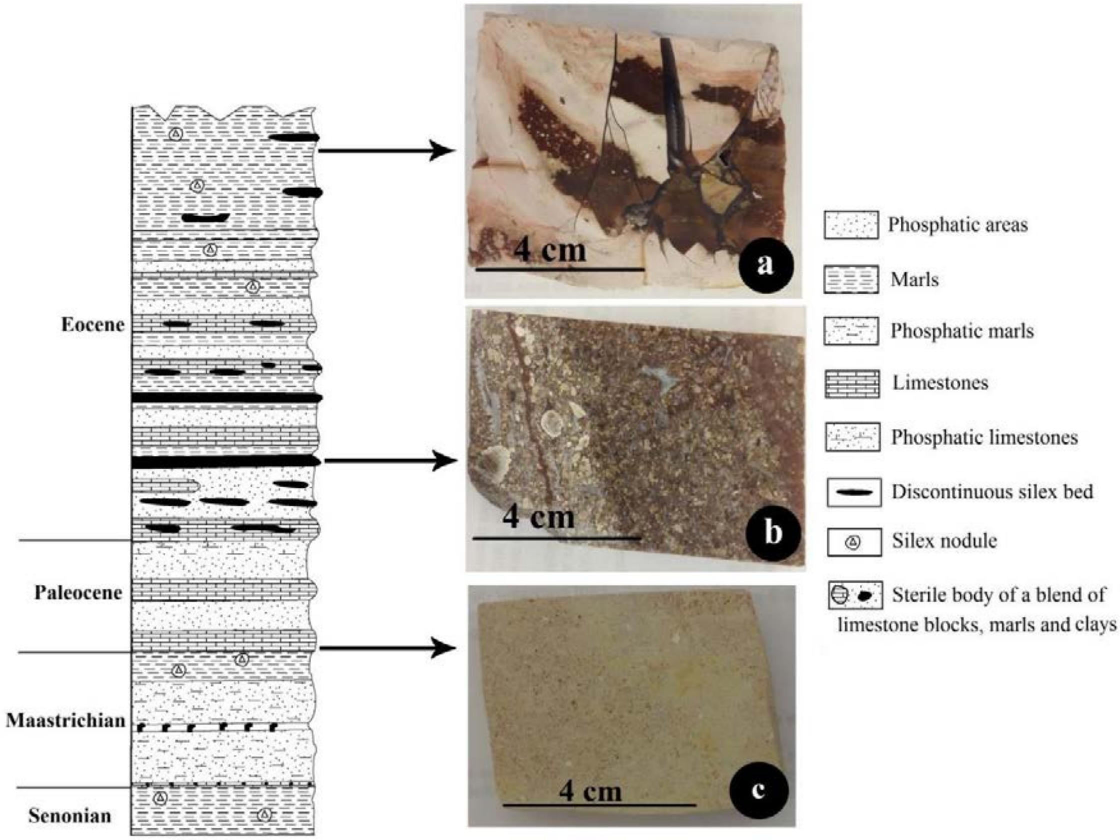

Fig.3. Example of Moroccan phosphate deposits series and photos of natural stone productswhich could be obtained from the overburden Photos of samples from Bengurir mine: **a**: silicified marls, **b:**nougat silex, **c**: limestone.

Another challenge facing OCP is to implement, wherever possible, a progressive rehabilitation of disturbed mining land as they progress in the panel exploitation in all its mining sites and piloted research for new technologies to accelerate the pace of piles and tailings pond reclamation. Progressive rehabilitation during the life of the mine will help to reduce the overall liability for rehabilitation works. The other benefits include reduction of double handling for waste materials and topsoils and reducing the disturbed land areas, the early establishment of re-vegetation, which reduces dust levels (wind erosion) and improves visual mine surroundings. In addition this also results in a significant reduction in the amount of rehabilitation costs required when mining is completed. This cost effective approach will allow gradual reclamation of the land within years, rather than decades. In addition, progressive rehabilitation provides opportunities for testing rehabilitation practices, and for the gradual development and improvement of rehabilitation methods.

6. Conclusion

Phosphate waste rocks and phosphate sludge should be valorized to minimize environmental damage and to reduce OCP mining footprint. The recycling and reuse of these by-products could create opportunities for domestic value-adding.

The hydrogeological characterization of original and screened phosphate limestone waste rocks and phosphate sludge showed their suitability to be used a component of store-and-release (SR) covers for industrial site reclamation. Considering their mineralogical composition, phosphate waste rocks and phosphate sludge mixed with local clay from phosphate mines have been proven to be suitable for ceramic manufacturing. Furthermore, the phosphate barren material have the potential to become a significant source of raw construction material because of their varied components and their ornamental potentials: marls, limestones blocks, silex bed, silex nodule, marls and clays silicified limestone. To achieve those targets, instead of pursuing with the conventional mining technologies, selective surface mining with proper planning could be implemented for both overburden and phosphate removal. These actions are essential for successful mine closure, reclamation and rehabilitation of mined disturbed lands While reducing the footprint.

The implementation of a progressive mine site rehabilitation could be implemented by OCP group in all its mining exploitations. This cost effective approach offers a number of advantages: improving the visual appearance of the landscape and establishing a cover to provide erosion control, dust control and re-vegetating support.

Acknowledgements

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