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Characterizations and potential recovery pathways of phosphate mines waste rocks

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A B S T R A C T

The phosphate ore production is steadily increasing due to its high demand for agriculture, medicine, and others. Ore extraction generates a considerable quantity of waste rocks that are generally stocked in piles. The current research aims to investigate the characterization of phosphate mine waste rocks (PMWR) generated in Benguerir, Morocco mine site. As a part of a wide project aiming to recycle those stockpiles, sensor-based ore sorting will be employed to separate the different lithologies. As a prior investigation before implementing this technology, two samples of 25 tons from the destoning and screening PMWR piles were sampled and submitted to manual sorting. The latter operation revealed the presence of different lithologies including indured phosphate, different types of siliceous, carbonate, phosphorus, and marly rock types. Those waste rocks were characterized physically, chemically, and mineralogically. Their potential uses for recycling or upcycling was investigated and addressed. About 25% of indured phosphate was found, which can be first recovered by ore sorting. This reserve of residual phosphate could be recovered using beneficiation methods. The flintstone, phosphated flintstone, and dolomitic limestone exhibit good physical and mechanical properties that meet the requirements to produce concrete. The silexite and siliceous marls have a low density and could be used as lightweight aggregate for non-structural concrete blocks production. The investigation on tender marls corroborates the literature and their suitability as alternative binders or as supplementary cementitious materials. Those marls could be used as well as lime binder for repairing historical buildings or as hydraulic lime binder for road construction. PMWR valorization as road construction materials was already proved. However, studying this remediation pathway after the recovery of phosphate and other lithologies by ore sorting is highly recommended. The recycling of those by-products will decrease the natural resources consumption in the civil engineering field alongside with resolving their environmental problems.

1. Introduction

The demand on phosphorus is increasing due to its highly added value for agriculture, medicine, etc. (Gharabaghi et al., 2010; Mohammadkhani et al., 2011). According to the last statistics released in January 2021, the US Geological Survey's estimated the phosphate ore production in 2020 of 223 million metric tons where: China (90), Morocco (37), the United States (24) are the top three (*Mineral commodity summaries, 2021*). Despite China is the leader in phosphate ore production, the estimated worldwide reserves are located in Morocco (70%), China (4.5%), Egypt (3.9%), followed the rest of the world (21%). Sedimentary phosphate in Moroccan context came from the decomposition of sea animals, since about 75 million years (My). The main phosphate fields in Morocco are in four large basins: Oulad Abdoun Basin, Gantour Basin, Meskala Basin, and Oued Eddahab Basin as presented in the Fig. 1. Phosphatogenesis took place during the geological period between the Maastrichtian (Late Cretaceous) and Lutetian (Middle Eocene) (Armand Boujou, 1976). The phosphatic series in the Gantour Basin is produced based on the collected data from exploitation operation and borehole recognition. Its start by Maastrechtian (66.0–72.1 My) and end by Lutetian (41.2–47.8 My) (Anjjar et al., 2018; Ihbach et al., 2020). Sandy phosphate, phosphatic marls,

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phosphatic limestones, and clay sandstone constitute the main layers of the Maastrechtian (Late epoch). The Danien (61.6–66.0 My) and Thanetian (56.0–59.2 My) from Paleocene epoch, are mainly composed by an alternate of the phosphatic limestones, sandy phosphate, and phosphatic marls with the presence of flint and phosphated flint (Phos-flint). The Ypresian (47.8–56.0 My) as lower Eocene epoch is elaborated by phosphatic marls with intercalation of silicified marls, phosphatic limestones, and silexite. The base of Ypresian is marked by the impermeable Ypresian clay. The end of the phosphatic series is marked by the Lutetian. Which characterized by marly-siliceous limestones known as Thersitae limestones and the presence of the clayed layers (Boujou, 1976; El Bamiki et al., 2021; El Haddi et al., 2014).

OCP-SA, the holding in charge of phosphorus extraction in Morocco, adopt a combination of different processing units including destoning, screening, washing and then flotation to maximize the extracted phosphate ore. This last as sandy phosphate layers are intercalated between waste rock levels including marlstone, limestone, etc. (Mouflih, 2015). Thus, the value chain of phosphate extraction generates different by-products i.e., destoning, screening, and washing waste rocks. The production of those by-products is susceptible to increase with the increase of the phosphate production. Idrissi et al. (2021), have investigated the properties of nine different wastes from phosphate ore mine. Their study suggested that those materials are rich of different minerals such as cristobalite, dolomite, and calcite. Therefore, they could be valorized in several applications to generate additional revenue. More recently, Taha et al. (2021) have proposed several solutions to achieve a sustainable mining while maintaining circularity. The main identified circularity opportunities were the residual phosphorus recovery alongside with other critical minerals and metals. Also, considering the phosphate mine waste rocks (PMWR) as alternative resources and raw materials especially for civil engineering.

In Morocco, the open-pit phosphate mines extract the sedimentary phosphate using the discontinuous panel extraction process. During this operation, the overburden and intercalation layers are extracted and stored in piles. The sandy phosphate is extracted using bulldozers and transported to treatment and beneficiation plants. However, some of the intercalation layers are also extracted with phosphate due to imprecision of the extraction equipment and the geometric definition of the phosphate layers. To separate the sandy phosphate from the waste rocks, the extracted mixture is subject to crushing and sizing. The resulting fine sandy phosphate is transported to treatment and beneficiation plants while coarse rocks are discarded as waste rocks in PMWR piles.

Attempts were done to recycle those different by-products of phosphate extraction to be used in the civil engineering field. Extracted from phosphate interlayers, red clays were used to synthesis lightweight aggregate (0.80–0.95 g/cm^3) that achieved in concrete ~77 MPa at 28-d compressive strength (fc_{28}) (Bayoussef et al., 2020). Also they are used to produce alkali activated material of 39 MPa fc_{28} (Moukannaa et al., 2020), and even used as a supplementary cementitious material (SCMs), after treatment, up to 20 *wt*% which results 22 MPa fc_{28} (Bahhou et al., 2021a). Calcined tender marls were used as a compound of binary system and proved to be used as SCMs up to 40 *wt*% (Safhi et al., 2022a, 2022b). From the same provenance, yellow clays were tested for geopolymers manufacturing that generated 25 MPa fc_{28} (Mabroum et al., 2020). Dolomitic limestone and flintstone were crushed and recycled as aggregates for the production of 25 MPa fc_{28} ordinary concrete (El Machi et al., 2020, 2021). Furthermore, washing waste tailings were tested to develop a geopolymer mortar (Dabbebi et al., 2018). A manufactured geopolymer based on phosphate sludge (60 *wt*%) and metakaolin (40 *wt*%) has achieved a compressive strength of 40 MPa (Moukannaa et al., 2019). Calcined marls at 750 °C was used to manufacture geopolymers of 38 MPa fc_{120} (Mabroum et al., 2021). Mixed waste rocks were successfully used for road construction (Amrani et al., 2019, 2020b). The conducted studies revealed that Moroccan PMWR could be used as a mineral resource and raw materials.

The management of the PMWR is a major concern regarding their environment footprint and the urban planning of the region. In a sustainable development point of view, and for reducing the negative

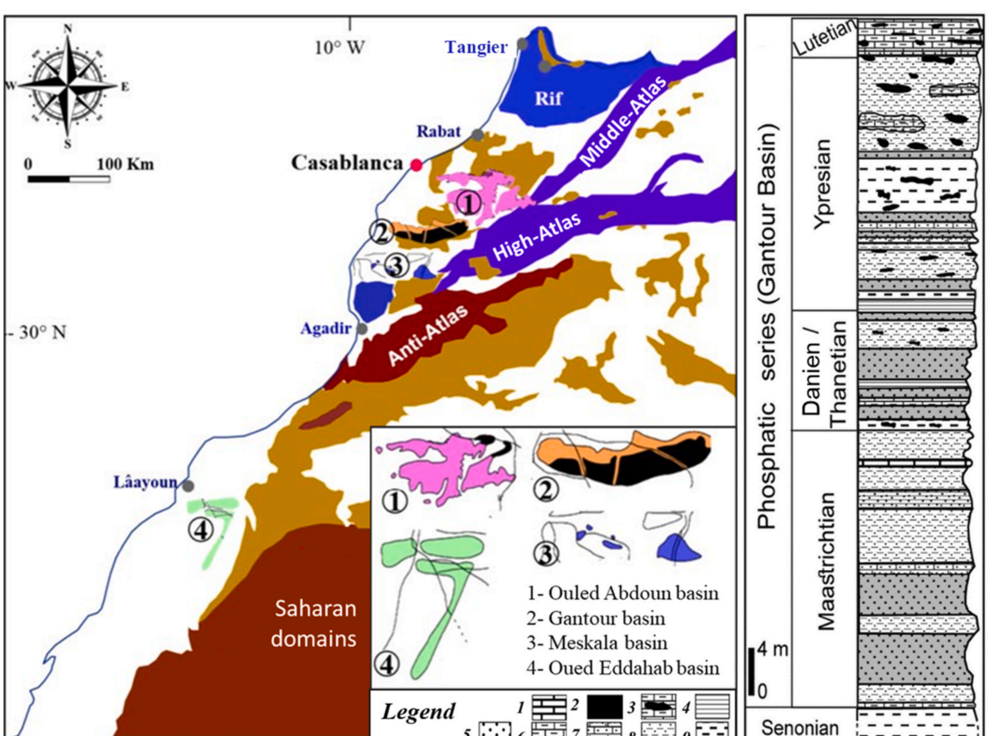


Fig. 1. Location of phosphate basins in Morocco including the one of Benguerir mine site and it stratigraphic log, adapted from (El Haddi, 2014; Ihbach et al., 2020); *Legend of the lithologic column: 1) Limestone, 2) Flintstone, 3) Marley-silicious limestone, 4) Clay, 5) Uncemented phosphate, 6) Marley limestone, 7) Phosphatic limestone, 8) Phosphatic marl, 9) Marl.*

impact of mining, this paper aims to characterize the screening and destoning PMWR piles of the Benguerir, Morocco mine site and thus propose potential reuses of each lithology. The study revealed the existence of 25% of indured phosphate which could be used as a new reserve. Also, the study characterizes new lithologies that were not studied before. The recovery of those by-products will be with a great environmental and economic interests.

2. Materials and methods

2.1. Open pit mine of Benguerir, Morocco

The Gantour deposit is one of the eight Morocco's phosphate deposits and the Benguerir mine is located at the center of that deposit. The intercalation layers are composed of siliceous, carbonate, marly, and phosphatic rocks (Boujou, 1976). In fact, the stratigraphic log of the panel seven on the Northern Benguerir mine shows that the lithologies of the intercalation layers are dolomitic limestones, phosphatic limestones, marly and carbonate phosphate, and flintstone beds (Anjjar et al., 2018). To stock the PMWR in the vicinity of the mine, the Benguerir mine has two different piles: the destoning PMWR of 90–150 mm (32° 15′ 33.8"N, 7° 51′ 22.3"W) and the screening PMWR of 30–90 mm (32° 14′ 09.6"N, 7° 51′ 54.0"W). The 600 m length with a height of 60 m piles with top-down configuration are formed by the same rocks as the intercalation layers of the mine and the particle size of the rocks vary from fine to coarser fraction. These piles are characterized by high heterogeneity (large blocks to fine particles) and high segregation effect of the particles due to the disposition method.

3D modeling constitutes an efficient tool for decision-making of the mine designers and operators in mining industry. It serves to conduct industrial development to define priorities and improve production performance. A 3D modeling for PMWR piles was conducted to estimate their precise volume. To achieve this goal, a Professional drone equipped by GPS RTK was used to collect data points with high ground resolution (2.75 m/Px), flight altitude of 100 m, camera with high resolution 16 Mpx, and 70% overlap of images. The collected data was processed using Lidar module of the Global Mapper software for data cleaning to optimize the database size. Then, the cleaned data was exported to *Datamine RM* software to create the 3D modeling (Fig. 2). The analyses showed that the PMWR screening piles are about 7.43 Mm^3 and those of destoning are about 7.63 Mm^3. Considering an approximative density of 1.60 kg/m^3, the tonnage is around 11.89 and 12.18 Mt for screening and destoning PMWR piles, respectively. In other manuals, the coarse fraction (>30 mm) for potential uses constitutes around 45–50% of the PMWR piles. Based on that proportion, the tonnage of the coarse material is about 5.65 and 5.79 Mt for screening and destoning PMWR piles, respectively.

The PMWR was produced with three quality that are i) overburden and extraction waste rock dumps, ii) PMWR destoning piles and iii) PMWR screening piles. Around 0.8–1.0 Mt/yr of extracted PMWR are disposed in the destoning and screening piles. Stripping ratio in the context of Benguerir site is around 3:1, for one ton of phosphate rocks production, three tons of waste rocks were produced (Taha et al., 2021).

2.2. Materials sampling and manual-sorting

The PMWR piles are characterized by high heterogeneity due to the segregation effect (Amrani et al., 2019). The top-down disposal consists of discharging PMWR over an advancing face (Zevgolis, 2018). The fine materials are disposed on the crest and the coarse materials move to the bottom (Blight, 2009). The adopted sampling method in the PMWR piles takes in consideration the segregation effect by collecting a composite sample with real grain size distribution from the top, middle and the base of the piles. Tweeny five tons of destoning and screening PMWR piles was sampled (Fig. 3). For this, different samples were collected from the bottom, middle and the top of the piles to have a homogeneous representative sample. A manual-sorting was conducted on the coarse fraction of those samples (>30 mm) based on the surface texture, the color, and the general aspect of rocks under the supervision of geologists. The Fig. 3.c presents the manual sorting of the screening PMWR.

2.3. Characterization methods

Identification and classification of PMWR were done using a macroscopic inspection and a petrographic observation on thin sections using Leica DM2700 standard optical microscope. Chemical characterization was performed utilizing X-ray fluorescence (XRF) by employing Epsilon 4 XRF Spectrometer. The mineralogical characterization was performed using X-ray diffraction (XRD) by employing D2 PHASER diffractometer, monochromatic CuKα radiation at $\lambda = 1.54$ Å (40 kV, 40 mA). The identification of the mineral phases was made by HighScore software using COD crystallography database and the quantification of the abundance of the identified phases was realized by *powdR* package using rockjock library from the original RockJock program R (Butler and Hillier, 2020; Eberl, 2003). Physical characterizations were done using the volumetric displacement method for the measure of the specific gravity according to the ASTM D6473 (2015). Helium pycnometer of Micromeritics: AccuPyc II 1340 was utilized to quantify the absolute gravity. The unconfined compressive strength (UCS) was conducted on phos-flint and dolomitic limestone cylinder rock specimens according to the ASTM D7012 (2014) standard on intact rock core specimens of one cylinder of 62Ø124 mm and triplicate cubes of 50 mm^3. Intact rock core

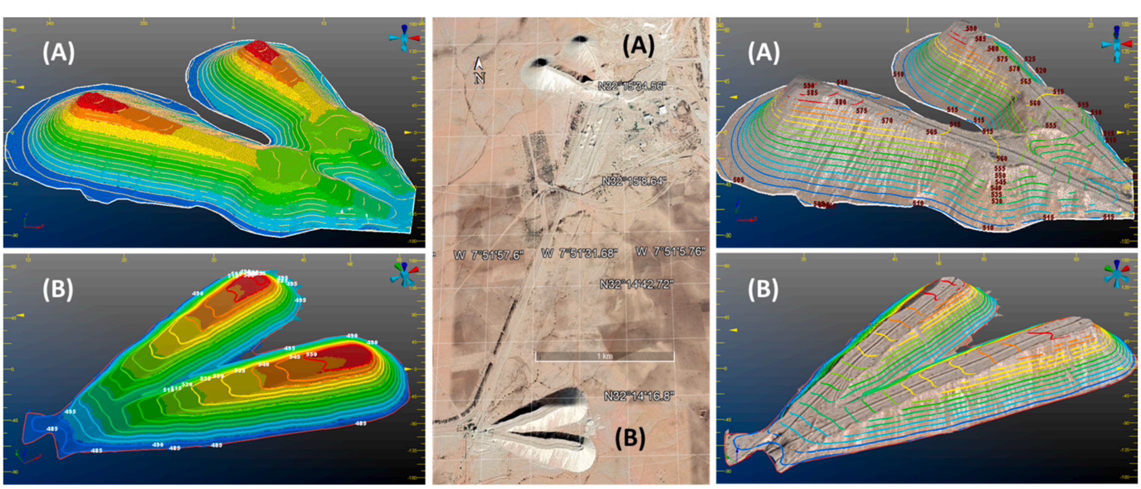


Fig. 2. 3D models for destoning (A) and screening (B) PMWR piles.

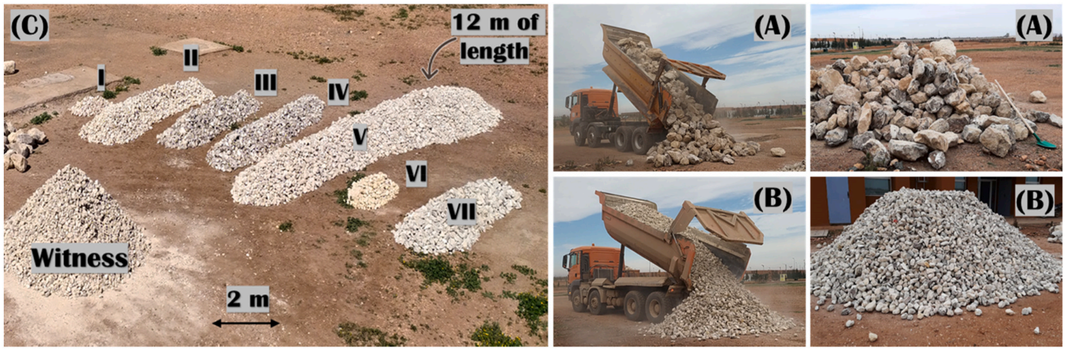


Fig. 3. 25-tons samples of destoning (A) and screening (B) PMWR each, subjected to manual sorting (C).

specimens of flint samples were not produced because the rock exhibited triboluminescence during the sample preparation. Gamma-ray (γ-ray) densitometry (GRD) was applied on a fraction of the rocks of ~1 cm thickness of each to estimate the linear attenuation for each sample which could be of a benefit for enhanced concrete radiation shielding that is used widely in nuclear, industry, health, and agricultural sectors. The in-house developed GRD consists of a sealed Cesium-137 source of about 190 mCi with a collimator of 1 mm opening and a collimated NaI scintillation detector of 1 mm opening. The sealed source and the collimated detector are placed in front of each other with alignment of their opening. The distance between the source and the detector is adjustable. A line beam of gamma ray passes from the source e to the detector through the specimen placed in the center distance between them. Thus, the attenuation of the gamma ray radiation line of 1 mm thickness that passes through the sample rock placed in the center distance between the source and the detector was calculated using Beer--Lambert's law of Eq. *1* (Farid et al., 2022).

$$I = I_0 . e^{-\mu x} \quad (1)$$

where I is the γ-ray intensity after attenuation (passed through the sample), I_0 is the initial γ-ray intensity, μ is the linear attenuation coefficient (cm^{-1}), and x is the physical thickness of absorber (cm). From this equation, the linear attenuation μ can be estimated for each sample rock used in this study to assess the suitability for radiation shielding as concrete aggregates and binders for the potential use of PMWR.

3. Results and discussion

Screening PMWR have a global mixed bulk composition, and each single rock particle is composed of one rock type that was sorted. In fact, phosphate sedimentary deposits present great heterogeneity as shown in the stratigraphic log and in the PMWR piles with high segregation degree due to the top-down pile configuration. Particularly, the panel phosphate extraction method leads to bulk deposition of waste rocks. The manual-sorting revealed the existing of eight lithologies, each has a different specific potential use for phosphate beneficiation or construction materials.

3.1. Manual-sorting

As shown in Fig. 3 C, the manual-sorting revealed that the PMWR screening piles are mainly composed of indured phosphate (V, 30%), flintstone (III, 14%), phos-flint (IV, 14%), silicious marls (II, 20%), dolomitic limestone (VII, 14%), silexite (I, 5%), tender marls (VI, 2%), and some clays. The manual sorting of the destoning PMWR revealed the presence of indured phosphate (25%), flintstone (25%), Phos-flint (25%), silicious marls (10%), dolomitic limestone (10%), and silexite (5%). The tender marls were not found on those late piles because of their fragility. Overall, the two piles are composed of the approximately the same lithologies with a small different proportion. The fine fraction (<30 mm) was not subjected to manual-sorting which found to present about 50–55% of the PMWR piles, respectively.

3.2. Chemical characterization

Table 1 summarizes the main chemical oxides composition of the founded lithologies. The phosphate rocks are composed of 24% P_2O_5 carbonated in a matrix that contains 16% of silica. The flintstone is mainly composed of silica (94% of SiO_2) which corroborates the findings of El Machi et al. (2021). A different type of flintstone was found, called in the following Phos-flint, contains around 35% of silica, 25% of phosphorus pentoxide, and carbonated (34% of CaO). The silexite is mainly composed of silica (80%) with a remarkable low density that indicates a high porosity and higher absorption. The silicious marls have a very similar chemical composition of the silexite. The found limestone has a high loss on ignition (LOI) around 38% and mainly composed of dolomite (24% of MgO) and calcite (28% of CaO). Tender marls have a high LOI (32–34%), mainly composed of MgO and CaO.

3.3. Petrographic macroscopic characterization

Table 2 summarizes the petrographical properties of the existing lithologies founded in the PMWR piles. The sedimentary rocks are formed through chemical and biochemical reactions. The macroscopic examination revealed distinctive properties of the rocks. Flint is a siliceous rock with smooth surface texture, conchoidal break, and a high hardiness. Phosphate flint is similar to flint but contains phosphate particles. Dolomitic limestone is a carbonate rock of a rough surface texture that has a positive reaction with HCl. The indured phosphate composed of two types, one based on consolidated fine grains and the other with indured coarse grains. Both have a certain important hardness and are not easily friable. Regular flintstone and the phos-flint both have a massive surface texture and a high hardness. The silexite distinguished with its purplish-pink color and its lower density compared to the other flintstones. Silexite is a sedimentary siliceous rock with a fine texture formed from chemical, biochemical or volcanic interactions (Foucault and Raoult, 2010). The silicious marls are compact and lightweight like the silexite but with lesser hardness. The tender marl is fragile and comes in different colors.

3.4. Mineralogical characterization

Fig. 4 represents the XRD analysis of the main lithologies. The mineralogical composition shows one major peak of quartz in the flintstone, carbonate major peak in the limestone with minor peaks of quartz which corroborate the XRF analyses. Flint is a chert siliceous rock composed of silica oxides (94%) and quartz minerals. It occurs as nodules or beds in the phosphate deposits, and the quartz can be microcrystalline or cryptocrystalline. Boujou (1976) investigated siliceous rocks of the Gantour deposit under the microscope and stated the

Table 1
Chemical properties of the founded lithologies.

Lithologies		SiO_2	Al_2O_3	MgO	CaO	P_2O_5	Other	LOI
Tender marls	*White*	11.3	1.13	13.7	36.4	2.35	0.8	34.4
	Yellow	18.3	3.35	18.2	24.1	0.99	3.28	31.7
	Green	16.2	2.38	11.5	34.1	7.89	2.06	25.7
Dolomitic limestone		7.84	0.37	23.6	28.4	0.98	0.60	38.1
Siliceous marls		74.0	1.49	4.52	7.23	1.88	0.80	10.1
Flintstones	*Flintstone*	93.6	0.28	0.43	1.55	1.85	0.46	1.84
	Silexite	80.1	1.03	3.70	4.54	0.70	0.81	9.14
	Phos-flint	34.8	0.00	0.42	33.6	25.0	0.76	5.52
Phosphate rocks		15.6	0.30	4.63	42.6	23.6	0.94	12.4

dominance of microcrystalline calcedony in the mineralogical composition of flint. Dolomitic limestone is a carbonate rock composed of calcite and dolomite minerals. In a previous study by Idrissi et al. (2021), chemical characterization of a dolomitic limestone (S4) from the Benguerir mine revealed the presence of SiO_2 (4.6%), MgO (11%), CaO (36%), P_2O_5 (1.4%), and 46% LOI, with a corresponding mineralogical composition of quartz (8%), calcite (35%), and dolomite (52%). The chemical composition of the sample is close to the studied dolomitic limestone, and the mineralogical composition confirms the classification of this rock as dolomitic limestone. The phos-flint shows peaks of apatite and quartz, and the silexite is mainly composed of quartz. Sidibé (1995) also found that the Senegalese silexite contains 91% of flint, 6% of indured phosphate, 3% of fine phosphated, and clayey elements.

The mineralogical observation on the thin sections was done by the optical microscopy of the major lithologies stored manually is illustrated in Fig. 5 and the quantification in Table 3. The indured phosphate is constituted by phosphate grain as fluorapatite, bioclasts and coprolites rich in *P* element. All these elements are cemented by micrite dominated by carbonates. The phosphate grains are presented by an ovoid/elliptical morphology of a size of 100–600 μm as encapsulated in the gangue (Mouflih, 2015). The dolomitic limestone appears to be finely grained with a micritic matrix (93–95%) that bonds some quartz particles (5–7%). The size of the quartz particles is ranged in 10–100 μm and the micritic cement of 5–7 μm. The texture can be classified as finely crystalline micrite according to Folk (1959) and a mudstone according to Dunham (1962). The phos-flint are characterized by the presence of ovoid morphology of the phosphate grains and the abundance of coprolites and bioclasts. The quartz is the main element that cements the phosphate grains. The size of the phosphate grain is around 100–400 μm. Generally, the phosphate grains are encapsulated in the gangue minerals. The silexite is formed mainly by quartz and dolomite minerals. The quartz in the silexite is presented by fine grain size and the dolomite with size of 20–70 μm. Yellow marls and white marls contain mainly dolomite and calcite in micrite matrix, the only difference is that the latter composed of coarse grain compared to yellow marls. While the green marls characterized by laminated layers that contain coprolite and fluorapatite cemented by micrite. Several quartz lodes were observed in the silicious marls with a considerable presence of calcite and dolomite. Overall, the observations corroborate the chemical and mineralogical characterization.

3.5. Gamma-ray radiation

A Gamma ray densitometry (GRD)(consisting of γ-ray collimated sealed source with a single line radiation and a collimated single detector facing the sealed source with alignment) was used to measure the linear attenuation of the samples rocks by locating each sample rock in the center distance between the source and the detector (Baalamurugan et al., 2019; Farid et al., 2022). Fig. 6 represents the estimated linear attenuation coefficient of the different lithologies. The waste rocks of phos-flint and limestone can be used as aggregate with mortar for enhanced concrete nuclear shielding as per the study of Farid et al. (2022) particularly for no segregation of a high strength self-consolidating concrete (HSSCC). Farid et al. (2022) used a crushed coarse and fine limestone aggregates of no segregation HSSCC that showed ~25% enhanced in γ-ray radiation shielding over the mortar sample. From the results of Fig. 6, waste rock of phos-flint could further enhance the radiation shielding of HSSCC over limestone aggregate.

4. Potential uses of the by-products

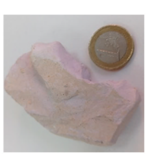
The wide range and variety of the PMWR properties (physio-chemical and mineralogical) encourage several remediation pathways including the phosphate beneficiation since those PMWR are mainly composed of indured phosphate. The other lithologies could be recycled or upcycled as raw material resources for the different civil engineering sub-fields. In order to recycle each lithology separately an ore sorting is necessary.

The ore sorting is used to upgrade the low-grade ores using non-destructive sensors based on the physio-chemical properties (dos Santos et al., 2017). It was used successfully to concentrate different ores such us tungsten, diamonds, sulfides, and minerals bearing heavy rare earth elements (Robben and Wotruba, 2019; Veras et al., 2020). It offers many advantages related to the reduction of environmental footprint and quantity of the generated waste (Lessard et al., 2014). The feed sample can be separated in two products (accepted and ejected) using many techniques including color, X-rays, near infrared (NIR). The ore sorting applied for coarse phosphate separation was implemented by Saudi Arabia and china (Li et al., 2020; Robben and Wotruba, 2019). The particle size sorted using X-rays in the Umm Wu'al Phosphate Mine (Saudi Arabia) was between 12 and 75 mm with total capacity of 1,800 t/h (Robben and Wotruba, 2019). The objective of the ore sorting was to remove the silica gangue of around 700,000 t/yr from the phosphate. The final product was about 2% SiO_2 compared to the feed samples with 10–30% SiO_2. The X-ray sorting was also applied on the Yichang phosphate layer ore (China) to recover the coarse fraction 10−30 mm (Li et al., 2020). The ore was upgraded from 20% to 27% P_2O_5. The yield and recovery rate were 36% and 86%, respectively. The hand sorting was applied in that context to identify the existent lithologies in the PMWR piles and to prove the separation feasibility of the coarse phosphate for the future installation of ore sorting machine onsite.

4.1. Phosphate recovery

The phosphate mine waste rock piles located in the Benguerir site are generated in the exploitation phase to concentrate the fine fraction (<30 mm) by classification operation such as destoning and screening (El Machi et al., 2020; Hakkou et al., 2016). The coarse fraction (>30 mm) constitutes around 45–50% of the PMWR piles, with considerable amount of indured phosphate. The recovery of the residual phosphate in the fine fraction can be done by gravity separation as a preconcentration phase (Carlson et al., 2012; Kawatra and Carlson, 2013; Khan et al., 2019; Liu et al., 2016), flotation (Aleksandrova et al., 2020; Boujlel et al., 2019; Elgillani and Abouzeid, 1993; Mohammadkhani et al., 2011), leaching (Gharabaghi et al., 2010; Soltani et al., 2018; Zafar and Ashraf, 2007), and calcination (Al-Fariss, 1993; El-Jallad et al., 1980;

Table 2
Petrographical properties of the existing lithologies founded in the piles.

Lithology	*Phosphate*	*Flintstones*			*Dolomitic limestone*	*Red clays*	*Marls*	
		Flintstone	*Phos-flint*	*Silexite*			*Silicious and calcareous*	*Tender*
Picture								
General aspect	Fine/coarse grains and consolidated	Sharp angle ribs slicers	Sharp angle ribs slicers	Very light and sticky to the tongue	Very dense	Very tender and crumbly	Compact, lightweight, and sticky to the tongue	Tender
Surface texture	Granular welded	Crystalline	Packstone	Massive	Mudstone	Sprayed	Laminated to massive	
Break	Irregular	Conchoidal and irregular	irregular	Irregular	Regular net	None	Irregular	Sprayed tender
Color	Greyish	Dense brown	Black with greyish elements	Purplish pink	Whitish beige	Brick red	Whitish and Whitish beige	Yellow, white, and green
Sparkle	No shine	Weak	Weak	No shine	Low	No shine	No shine	
Effervescence at HCL	Weak	Negative	Very weak	Very weak	Positive and strong	Very weak	Weak to moderate	
Estimated density	+++	++++	++++	++	+++++	+	++	+
Estimated hardness	+++	++++	++++	+++	++++	+	+++	+

Guo and Li, 2010). The gravity separation will concentrate the dense particles, the direct and/or inverse flotation will eliminate the gangue minerals such as carbonates and silicates, leaching will dissolve the gangue minerals using the weak acids, and calcination to reduce the carbonate component and to dry the concentrate ore.

The recovery of the phosphated lithologies in the coarse fraction will be focused on the indured phosphate and phos-flint. Those lithologies can be recovered using the ore sorting technologies. These phosphated lithologies are presented with large quantities of the coarse fraction. This coarse fraction must be destoned at 90 mm to recover the fraction <90 mm for screening at 30 mm. The large rocks (+90 mm) must be crushed to serve as feed (30–90 mm) with the screened fraction (30–90 mm) of the ore sorting machine. A screening of the fine fraction <30 mm is done on the beneficiation plant entry to remove the coarse fraction (>3 mm) for crushing operation. The product of the ore sorting operation such as the phosphated lithologies will be crushed to join the phosphate beneficiation plant with the screened fraction (<3 mm). The ore sorting on PMWR piles will demonstrate it efficiency to reduce the noneconomic rocks and to maximize the phosphate resources (Lessard et al., 2014; Robben and Wotruba, 2019). The other lithologies can also be separated using ore sorting according to the application vocations as illustrated in Fig. 7.

4.2. Road construction materials

The construction of roads is a worldwide consumer of geomaterials. Employment of industrial by-products in such field can beneficiate the conservation of non-renewable natural resources and the reduction of produced wastes. PMWR are good candidates for a potential alternative secondary raw materials use. Ahmed and Abouzeid (2009) tested the PMWR from Egypt to be used as a subbase aggregate in road construction. The geotechnical findings corroborate such valorization, and a good dry density was achieved of 1.95 g/cm^3. Ahmed et al. (2014a), studied the Egyptian PMWR for the same use and reached 2.02 g/cm^3 dry density with an optimum moisture content of 12%.

In fact, the feasibility of PMWR valorization from Benguerir mine as materials for road construction was investigated and found to be a good remediation pathway. Amrani et al. (2019), demonstrated that those PMWR have satisfying properties i.e., a specific density >26 kN/m^3, Los Angeles abrasion in range of 45–58%, methylene blue value < 1 g/100 g, organic matter <1% and a plasticity index <20%. All the tested PMWR corroborate that they possess the needed geotechnical characterizations for an employment as embankments materials. Furthermore, environmental evaluation by leaching tests showed no risk of any contaminants. Later, an experimental testing of collapsible behavior of dry compacted PMWR in road embankment was conducted by Amrani et al. (2021). They found that dried PMWR can be utilized in embankment under total pressure <200 kPa. The same group of research studied the feasibility of recycling phosphate wastes (phosphogypsum and sludge) to enhance high-temperature rheological properties of asphalt binder (Amrani, 2020; Amrani et al., 2020a). The results showed that phosphogypsum ameliorates the mechanical performances of asphalt binder including its resistance against rutting more than phosphate sludge wastes and fly ash. Applying the ore sorting will lead to recover several lithologies that are candidates for potential uses (Fig. 7). Nevertheless, the feasibility study of those ore sorting residues is to be tested as road construction materials in case the content of phosphate is very low, and the investment revenue does not worth it.

4.3. Alternative binders

The recovery of industrial by-products as SCMs or as alternative binder is a great preoccupation of the scientific and industrial community. Interests on such topic is increasing due to the environmental issues related to the cement production responsible of 0.73–0.99 t_{CO2}/t_{Cement} (Latawiec et al., 2018). Calcined materials such as clays, sediments, and

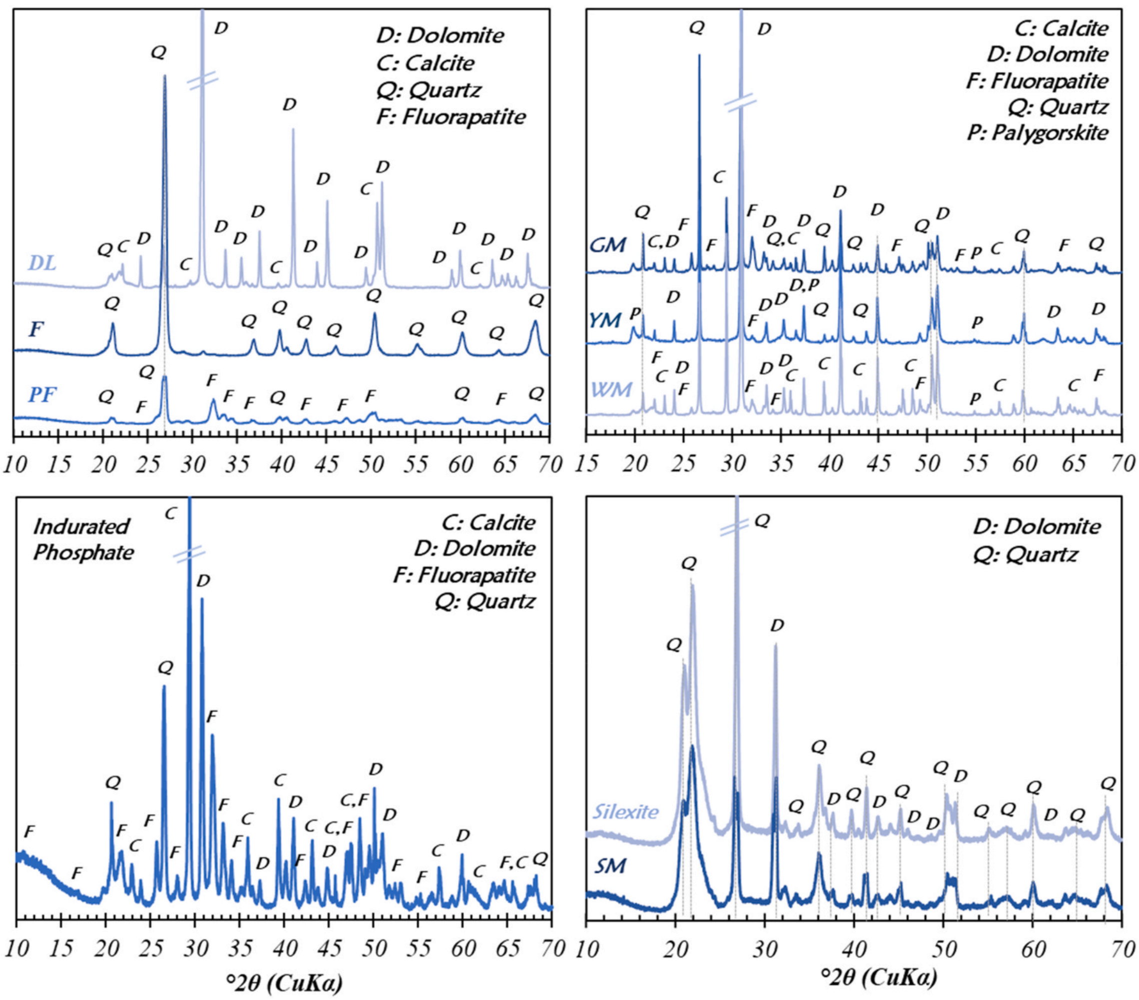


Fig. 4. XRD analysis of the founded lithologies: dolomitic limestone (DL), flint (F), phos-flint (PF), green marls (GM), yellow marls (YM), white marls (WM), silexite, silicious marls (SM), and indured phosphate.

marls proved to be a good raw material for such application (Bahhou et al., 2021b; Hanein et al., 2021; Safhi, 2022). Those materials are known as carbonated clay composed of magnesium or calcium carbonates ($MgCO_3$ and $CaCO_3$) that decomposes to MgO and CaO, and dioxide carbon (CO_2) after calcination. However, the reported emitted CO_2 after calcination was 73% which is lower than those of cement (Mo et al., 2010). Several studies and investigations were conducted on the hydraulicity and pozzolanicity of calcined marls. These studies showed that calcination of marlstone is generally in range of 650–850 °C, composed mainly of dolomite, palygorskite, and smectite among others. The investigated incorporation rates were ambitious, and the results revealed a good reactivity up to 50 *wt*% replacement (Akgün, 2020; Danner and Justnes, 2018; Justnes, 2015; Østnor and Justnes, 2014; Poussardin et al., 2022; Rakhimov et al., 2017; Shaaban, 2021; Soltani et al., 2018).

The marls from the PMWR of Benguerir mine site were already investigated for such use. Bahhou et al. (2021a), investigated the feasibility of using yellow marls as SCM and revealed that up to 20 *wt*% substitution rates led to achieve 80% of the strength of the reference (22 MPa at 28-d). Moreover, in their review, the authors stated that those materials would react in a hydraulic and pozzolanic reaction with 25–75% of carbonate content (Bahhou et al., 2021b). Safhi et al. (2022a) evaluated the reactivity of the same marls from PMWR. The sieved marls (white, yellow, and green) were treated by calcination at 850 °C. The loss of ignition was in range of 26–34%, the equivalent alkalis in range of 0.3–0.4% and increased to 0.8–1.5% after calcination. After treatment, the concentration of oxides responsible of pozzolanic properties (Fe_2O_3+SiO_2+Al_2O_3) was in range of 18–40% which is lower than the chemical requirements of ASTM C618 (2019), for pozzolan. The concentration of carbonates was much higher (57–69% of CaO + MgO) which implies important hydraulic reactivity. However, the hydraulicity modulus was in a wide range (0.8–2.9%). The composition of those three marls (red marks) was plotted in a CaO–SiO_2–Al_2O_3 ternary diagram as shown in Fig. 8.

The substitution of cement with 20–100% of an optimized calcined marl (mixture of three marls) was evaluated (Safhi et al., 2022b). The authors suggested that the blended binder with 20 *wt*% of marls had the highest strengths at all curing age with a strength activity index of 153%, 138%, and 127% at 7, 28, and 91 days, respectively. The blended binder with 40 *wt*% of marls results in a comparable compressive strength to that of the reference. The calcined marls alone revealed a good self-reactivity that achieved 10 MPa fc_{28} and can be used as a binder for repairing historical building. The use of those marls as an alternative binder will reduce CO_2 emissions and release the stress and demand on natural resources. Furthermore, using red clay as a binder could further enhance the radiation shielding property (Fig. 6) of the material that is mixed with.

4.4. Potential utilization of PMWR as alternative aggregates for concrete

It is widely known that concrete is the most consumed construction materials worldwide and the aggregates occupy most of the concrete

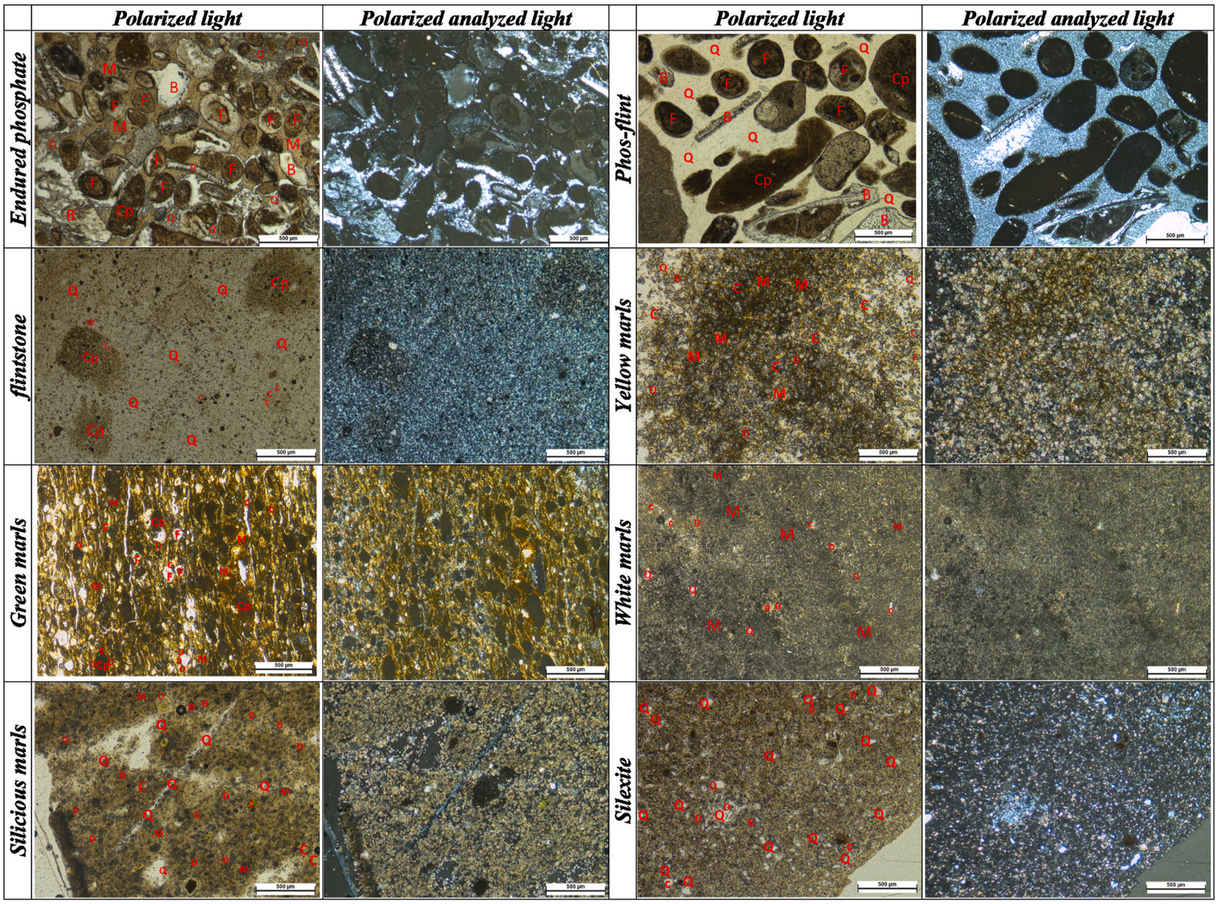


Fig. 5. Mineralogical observation (× 50 magnification) of PMWR lithologies. Legend: M: micrite, F: fluorapatite, C: calcite, D: dolomite, Cp: coprolite, Q: quartz.

Table 3
Quantification of the mineral phases using RockJock program R.

Lithologies		Palygorskite	Dolomite	Apatite	Calcite	Quartz
Tender marls	*White*	38.6	35.9	12.3	10.4	2.79
	Yellow	47.9	33.5	5.66	8.20	4.69
	Green	49.1	15.8	22.1	10.7	2.79
Dolomitic limestone		–	67.7	7.18	16.5	8.55
Siliceous marls		–	48.8	–	–	51.2
Flintstones	*Flintstone*	–	1.94	1.20	1.78	95.1
	Silexite	–	37.6	–	–	62.4
	Phos-flint	–	3.91	38.3	10.3	47.5
Indured Phosphate		–	30.4	47.7	13.0	8.81

volume. Aggregate production is estimated to be 48 billion ton in 2015 with an estimated annual growth rate of 5%, which means that its production could double in the next one or two decades (Xing et al., 2022; Zhang et al., 2019). The increasing quarrying of aggregates creates pressure on the primary sources, and it is necessary to use alternative resources of aggregate. The use of PMWR local alternative aggregates will help to reduce the stress on the primary aggregates. The region of Benguerir produces 0.70 Mm^3 of aggregates each year. On the other hand, the PMWR of Benguerir mine site have a total volume of ~15 Mm^3 and a total of reserve of hard rocks (~20–30% in the total piles) equal to ~4 Mm^3. PMWR are processed using mechanical operations that are executed on quarry natural concrete aggregates. The use of rocks from PMWR as aggregates for concrete would additionally save resources in terms of extraction and pre-processing operations. Very few studies have investigated the use of phosphate wastes aggregates for concrete. From Sebaeya in Egypt, phosphate wastes were used as coarse aggregates for concrete production, and produced a 24 MPa fc_{28} on 150 mm^3 specimens (Ahmed and Abouzeid, 2011) and 20 MPa fc_{28} on 100 mm^3 specimens (Ahmed et al., 2014b). From the phosphate PMWR of Benguerir, “flint” was used as coarse aggregates to produce ordinary concrete and substitutes completely the natural coarse aggregates (El Machi et al., 2021). In this study PMWR contains three hard rocks: flintstone, phos-flint, dolomitic limestone. The screening PMWR possess a maximum dimension equal to 150 mm. The three rocks were crushed using a laboratory jaw crusher to produce concrete coarse aggregates respecting the n°7 mesh of ASTM C33 (2018) standard. The physio-mechanical properties of the rocks and produced aggregates were tested. Potential, limitations, and strategies were discussed to valorize these rocks as aggregates for concrete. Table 4 presents the properties of rocks (Fig. 9), and Table 5 presents the properties of produced coarse aggregates.

The results of the characterization were compared to properties of natural coarse aggregates that are presented in NF EN 12620, ASTM C33 and ACI 211.1R-96 standards to produce ordinary concrete. F and PF

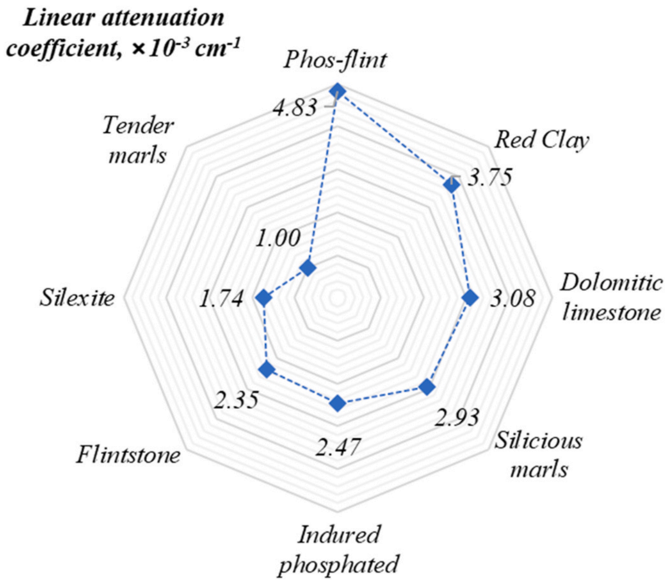


Fig. 6. Calculated linear attenuation coefficient of the different lithologies.

and DL conform to the specifications to use as coarse aggregates for concrete, and they seem adequate candidates to produce ordinary concrete.

Aggregates are an inert filler in concrete to reduce the cost, but they have a major influence on many properties of fresh, hardened and durability of concrete (Alexander and Mindess, 2010). The presence of deleterious substances, the alkali-aggregate reaction (AAR), and the porosity of aggregates are a major concern for concrete. Chert and finely grained dolomite or finely grained limestone aggregates are widely known to be potentially reactive with the alkaline pore solution of concrete (Rønning et al., 2021). The effect of apatite on the AAR has not been reported previously.

The recycling of mining by-products presents several technological challenges (Taha et al., 2021). The valorization of PMWR as alternative aggregates for concrete needs a sorting to separate the different rocks. The preparation of aggregates needs to be efficient to produce aggregates with adequate properties. The produced concrete need to present mechanical and durability performances similar to natural aggregates. Mining wastes have been successfully valorized as coarse aggregates for ordinary concrete (André et al., 2014; Kumar et al., 2016; Ostrowski et al., 2020), it is also recommended to produce concrete with higher performance (Rana et al., 2016). In addition, utilizing flint, phos-flint and limestone as aggregates enhance the radiation shielding property with phos-flint provides more enhanced shielding (Fig. 6) that is important for a wide range of peaceful applications of nuclear technology from industry to health and agricultural sectors.

4.5. Potential use of lightweight aggregates

Silexite and silicious marls found to have similar properties including the low density and could be used as lightweight aggregate (LWA) for lightweight concrete production. Nevertheless, the latter has pros and cons i.e., higher porosity and higher absorption, thus it results poor resistance. However, these properties are beneficial to reduce the total dead load of the structure, while relatively lowering thermal conductivity and increasing fire resistance with good sound absorption (Agrawal et al., 2021). Several studies encourage to use natural LWA to produce concrete for blocs due to their thermal and sound insulation properties. In this study, the thermal properties of four samples of silexite (about 82×42×10 mm) were tested. The findings result in an average thermal conductivity of 0.894 ± 0.113 W/m.K and an average thermal diffusivity of 0.153 ± 0.039 mm^2/s.

Silexite disposal in Senegal was mainly recovered as road construction material in foundation layer. Sidibé (1995) worked on recycling 0/40 mm silexite disposal, while BA (2008) conducted a geotechnical identification of 0/31[5] mm crushed silexite. Mbengue et al. (2019) have studied the performance of the same granular fraction in a seat layer. The results were conclusive and certifying a good potential use in the foundation layer. A California bearing ratio (CBR) index of 149 with a density of 2.01 meeting the specifications needed for its use in bedding. Cisse et al. (1999) Studied four compacted concretes made from fine aggregates exclusively using 0–3 mm filled sands produced from the crushing of limestone, sandstone, silexite, and basalt. The study presents a promising result depending on the type of addition and the road classification.

A 100 kg sample of silexite and silicious marls from the screening PMWR was crushed in a jaw crusher to have a d_{max} less than 14 mm. About 39% and 61% of fine (0/5 mm) and coarse aggregates (5/12[5] mm) was generated, respectively. The Table 6 summarizes the physical and geotechnical properties of those materials alongside with those

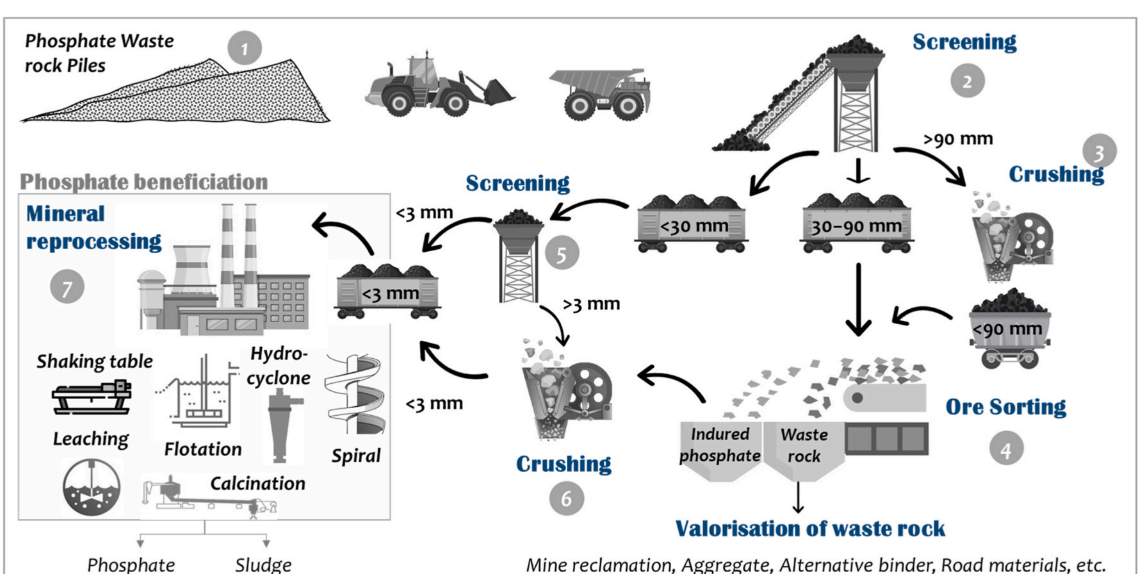


Fig. 7. Phosphate recovery and valorization of waste rocks from the PMWR piles.

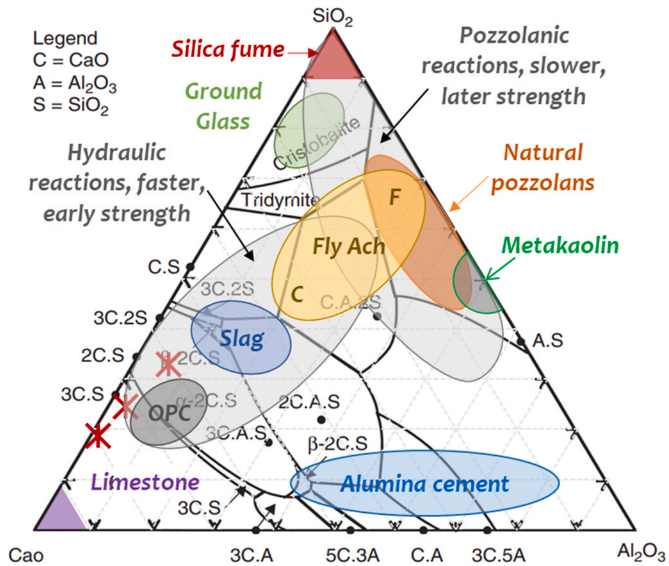


Fig. 8. Composition of three marls (red marks) superimposed on mainly used SCMs, adapted from Safhi et al. (2021). For the relevance of the diagram the $\Sigma(CaO + SiO_2+Al_2O_3)$ is in range of 70–72%.

Table 4
Physio-mechanical characterization of the three rocks.

Rock type/Properties		Flintstone	Phos-flint	Dolomitic limestone
Absolute gravity, t/m³		2.61	2.76	2.87
UCS, MPa	***Cubes***	Not tested	208 ± 27	167 ± 48
	Cylinder	Not tested	70	>120
Hardness, Mohs scale		7	$6^{1/2}$	6

properties of similar materials from the literature. It can be noticed that the silexite have a low specific density and lower apparent density compared to that of the other aggregate discussed in the previous section (4.4). Like the flintstone, the crushing of the silexite led to a high flakiness coefficient of 25–33%. The coarse aggregates had a good hardness property (LA < 30).

The fine aggregate (FA) were used in a mortar mix design with the calcined marls with a ratio of FA:CM of 1:3 according to the EN 196-1 (AFNOR, 2016). The water-to-binder was 0.8, and an additional water for the absorption of FA was considered. Nine cylindrical specimens of 75Ø150 mm were prepared, casted in sealed molds, and left in it (internal curing) until the age of testing. The UCS was tested on triplicate samples at each curing age. The samples achieved a UCS of 1.04 ± 0.06 MPa, 3.09 ± 0.43 MPa, and 4.94 ± 0.56 MPa at 7, 28, 90 days of curing age, respectively. The apparent volumetric mass of the tested cylinders was 1684 ± 48 kg/m³. Despite the achieved low compressive strength, this preliminary study showed that achieving lightweight concrete blocks using the LWA from PMWR is possible.

The one should keep in mind that many siliceous rocks are alkali-reactive, hence the importance of testing silexite. Silexite could be potentially reactive material due to opal and chalcedony present in the material. Sidibé (1995) tested the potential reactivity to cement alkalis comparing silexite, limestone and sandstone aggregates according with the ASTM C289–71 standard to assess the potential AAR of aggregates by a chemical method (withdrawn in 2016). The results show that

Table 5
Characterization of aggregates compared to different specifications.

Property	Standard	Values range	Flintstone	Phos-flint	Dolomitic limestone
Specific density, t/m³	ASTM C33	2.3–2.9	2.59	2.59	2.60
	ACI 211.1	2.5–3.0			
	NF EN 12620	2.0–3.0			
Water Absorption, %	ASTM C33	0.5–4.0	1.7	2.7	2.9
	ACI 211.1	0.2–4.0			
Bulk density, t/m³	ASTM C33	1.28–1.92	1.30	1.30	1.43
Water content, %	ASTM C33	0–2.0	0.5	0.8	0.3
Los Angeles value, % (NF EN 1097-2)	ACI 211.1	25–50	21	30	26
	NF EN 12620	–	(21%) LA25	(30%) LA30	(26%) LA30
Micro-Deval value, %(NF EN 1097-1)	NF EN 12620	–	(5%) MDE10	(9%) MDE10	(15%) MDE15
Flakiness index, % (NF EN 933-3)	NF EN 12620	–	(32%) FI35	(26%) FI35	(23%) FI35

Fig. 9. Some pictures of the compressive strength test specimens.

Table 6
Physical properties of the silexite from PMWR compared to previous studies.

Properties	Silexite				
	From PMWR		*Sidibé (1995)*	*Cisse et al. (1999)*	*BA (2008)*
Granular fraction	0/5 mm	5/12[5] mm	0/31[5] mm	0/3 mm	0/40 mm
Los Angeles, %	–	28.0	9	–	21
Micro Deval, %	–	–	21.4	–	17
Flakiness coefficient, %	–	32.8	–	–	24.9
Specific density	2.48		2.63	2.73	2.40
Apparent density	1.68	1.73	1.56	–	–
Absorption, %	17.4	16.5	–	1.47	–
Porosity, %	–	–	–	–	15.6
Property, %	–	–	3	–	–
Volumetric coefficient, %	–	–	0.18	–	–
Proctor – W_{OPT}, %	–		–	6	9.5
Proctor – γ_{dmax}, t/m^3	–		–	1.94	2.40

silexite maybe alkali-reactive while limestone and sandstone are non-alkali-reactive.

5. Conclusions, recommendations, and perspectives

This paper characterized the PMWR of Benguerir mine site and suggested several recovery pathways for those materials. Valorization of those by-products would have a great environmental impact on the urban planning. The following conclusions could be drawn:

- **Phosphate recovery:** The phosphate waste rock piles contain around 45–50% of the coarse material (>30 mm) that can be separated by screening for ore sorting. The recovered indured phosphate constituting around 25% of the PMWR can be valorized by joining the conventional mineral processing with the fine fraction (<30 mm).
- **Road construction materials:** Valorization of the PMWR as construction road material was already tested and proved for the sub-layer foundation. However, after recovery of the indured phosphate by ore sorting alongside with flint, phos-flint, and dolomitic limestone, the rest of this process will lead to a new disposal. Investigating the recycling of this rest is required.
- **Concrete aggregates:** Flint, phos-flint, and dolomitic limestone from PMWR have good properties to be used as aggregates. However, these lithologies could present several difficulties in terms of deleterious substances presence, the chemical stability, and the shape for use as concrete aggregates. The research perspective of this characterization study would consist of the production of regular coarse aggregates and the recovery of sands. The study of the AAR of aggregates and concrete production is to be investigated. The study of the effect of those aggregates on the properties of ordinary and high-performance concretes is recommended.
- **Alternative binders:** The tender marls from the PMWR have an interesting hydraulicity and medium pozzolanicity reaction that qualifies them to be used as alternative binder. Previous studies revealed that upcycling tender marls as alternative binder would have a great ecological impact by reducing the CO_2 footprint. The calcined marls alone could be used as repairing binder for historical buildings and monuments, nevertheless, more testing is to be done such as drying shrinkage, water absorption, etc. Moreover, the calcined marls could be used as an eco-friendly hydraulic road lime binder which can be used in mine site itself for the road's construction.
- **Lightweight aggregate:** The silexite and silicious marls have a considerable lightweight density to be used for the production of lightweight concrete blocks. The preliminary mix design with calcined marls and the thermal conductivity properties confirmed such a feasibility. The studied aggregates are suspected to be reactive to alkali-silica reaction. Study of reactivity proves to be an essential element of the potential valorization.

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CRediT authorship contribution statement

Amine el Mahdi Safhi: Conceptualization, Investigation, Methodology, Visualization, Data curation, Writing – original draft. **Hicham Amar:** Investigation, Visualization, Writing – original draft. **Yahya El Berdai:** Investigation, Writing – original draft. **Mustapha El Ghorfi:** Visualization, Validation, Investigation. **Yassine Taha:** Project administration, Validation. **Rachid Hakkou:** Project administration, Validation. **Muthanna Al-Dahhan:** Investigation, Validation, Writing – original draft, Writing – review & editing. **Mostafa Benzaazoua:** Funding acquisition, Project administration, Supervision, Validation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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