

# Index Properties of Alkali-Activated Cement Mortar Affected by the Addition of Phosphatic Clay

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## Abstract

Phosphatic clay (PhC) is a by-product waste released from beneficiation processes during phosphatic mining. This study presents an analysis of the effects of PhC on the characteristics of the alkali-activated aluminosilicate mixture to formulate a cement mortar. The mix proportion of cement mortar was prepared from aluminosilicate materials (kaolin and silica sand). PhC was added to the mix proportion with an appropriate ratio. The quality-indicating parameters were tested. The results indicated that, the bulk density of hardened mortar increased from 2 g/cm<sup>3</sup> to 2.21 g/cm<sup>3</sup>. The compressive strength accounted for the 12.5% improvement. The cement mortar absorbed less amount of water and showed less shrinkage behavior. This study found that using PhC as admixture in activated aluminosilicate cement mortar shows a tendency to improve the index properties of the activated mortar.

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**Keywords:** Phosphatic clay, by-product materials, mining wastes, activated aluminosilicates, cement mortar

## 1. Introduction

Phosphate is one of the most essential commodities mined in large quantities yearly around the world. It is considered as the main source of the element phosphorus used essentially in the agricultural sector as a form of fertilizer (Abu-Hamatteh, 2007). The phosphate layer, known as the matrix, is made up of nearly equal parts of sand, clay, and phosphatic minerals (Beavers et al., 2015). Jordan has huge resources and reserves of phosphate with high quality P<sub>2</sub>O<sub>5</sub> content in the middle of country; namely in Al Hassa area, Al-Abiad area, and Eshidiya area (Sadaqah, 2000). The mines require processing of flotation and beneficiation to reduce the content of accessory minerals, including quartz, clay, calcite, and dolomite (Abouzeid & Seifalnassr, 1996). The process of beneficiation depends upon the grain size. The Phosphate pebble is separated by a series of screens and washers, then the concentrates are utilized directly. The finer material is separated from the final phosphate concentrate as waste tailings, thus so-called phosphatic slime (Pittman and Sweeney, 1983). A typical mine can generate millions of tons of phosphate clay tailings annually. On the whole, for each ton of phosphate produced, approximately one ton of clay must be disposed (El-Shall, 2009). In Jordan, almost 10,000 tons of clay waste are produced daily during the phosphate beneficiation and flotation processes at the Eshidiya mine (Al-Hwaiti et al., 2015). This product is dispersed in water as a dilute colloidal suspension. The use of a conventional clay settling areas is the dominant method of storing phosphatic clay during the phosphate mining operations (Beavers et al., 2015). The PhC creates one of the most difficult disposal problems in the mining industry, due to the pollution of streams by turbid or toxic effluents (Abouzeid and Seifalnassr, 1996). The PhC of the Jordanian phosphate mines was characterized by several

work studies (Khoury et al., 1988); (Al-Hwaiti, 2000); (Al-Hwaiti et al., 2005); and (Slaty, 2005). The solubility of PhC indicated that PhC can be used as a source material for the immobilization process of heavy metals.

Very little research has been done in the past on the utilization of PhC around the world. Goode and Sadler (1975) tested the PhC as a substitute for bentonite in geothermal drilling fluids. The results indicated that, PhC base drilling fluids have more favorable rheological behavior than bentonite base fluids but poorer filtration properties. Wissa et al. (1983); Hardianto & Ericson (1993), and Boyd et al. (2007) investigated the stabilization potential of PhC with different materials as a lime and gypsum. Srinivas et al. (2106) studied the possibility of recovering phosphate minerals from the rejects of the Eshidiya beneficiation plant. He found that PhC can be used for direct application in soils as phosphate-rich organic manure in highly saline soil. Khoury et al. (1988) estimated the possibility of utilizing PhC from El-Hasa and Russifaha phosphate mines in Jordan. He concluded that PhC is more suitable as a lightweight for thermal insulation due to the small density, low water absorption, and good thermal conductivity. In Jordan, a low-cost building material with an acceptable compressive strength (32 MPa) was developed through the alkali activation of Jordanian Hiswa kaolinite, and was assessed in several research papers by Khoury and Al-Shaer (2009); Al-Shaer et al. (2009); and Slaty et al. (2013). Physical, thermal, mineralogical and microstructural results indicate that the activation process has decreased the porosity due to the formation of sodium zeolite phases and feldspathoids that fill the pore spaces and bind the matrix.

In this study the potential utilization of PhC in constructions as a cement admixture is examined by studying

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the effects of adding PhC on the index properties of hardened aluminosilicate mortar through alkali activation.

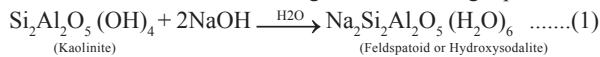
**2. Methodology**

*2.1. Characterization of PhC*

The PhC sample of this study was collected from Al-Hasa Phosphate Mine plant (about 130 km south of Amman, Jordan). The sample was collected directly from hydrocyclones after the beneficiation process. Filtration was done to separate the fine particles of PhC from water. The sample was oven-dried at 105 °C for twenty-four hours. Its color was determined by visual observation. The specific gravity was measured by the standard testing methods ASTM D854-00, using the pycnometer. The particle size distribution was determined using sieve analysis and hydrometer methods (for particle sizes pass sieve No. 200), according to ASTM D 422. The Atterberg limits were determined according to ASTM D4318-17. The chemical and mineralogical composition of PhC was determined using X-ray diffractometers (XRD) by Philips 2KW model and SHIMADZU Sequential X-ray Fluorescence Spectrometer (XRF) at the University of Jordan.

*2.2. Preparation of cement mortar*

An activated cement mortar from Jordanian kaolinite was developed and characterized previously by the researchers (Khoury and Al-Shaer, 2009); (Al-Shaer et al., 2009); and (Slaty et al., 2013). The activated cement mortar was prepared by the alkali activation of untreated kaolinite (HK) from a deposit in Hiswa area (South Jordan). HK consists mainly of 74% of silica (SiO<sub>2</sub>) and alumina (Al<sub>2</sub>O<sub>3</sub>) of the total content. The selected grain size was less than 425µm after the crushing and sieving of the oven-dried sample (at 105 °C). Silica sand (SS) was used as an inert filler (with purity being about 99% and the grain size between 90 and 250 µm) to improve the workability of the mixture and its mechanical strength. NaOH pellets of 99% purity (Merck) and distilled water were used to prepare 16 M of alkaline activator for dissolution aluminosilicate phases. The mixing process of the constituents was done according to the following equation.



In order to investigate how PhC affects the activated mortar characteristics, two mixtures of raw materials were prepared. The first mix was composed of 2HK: 1SS proportion, and acted as the reference mixture. The second mix was composed of 2HK:1SS:1PhC proportion, to investigate the potential influence of PhC on the final quality of the product. The water ratio was added according to the plasticity limit of each mixture. The prepared mixture was mixed for 10.0 minutes to obtain a homogenized paste. The produced cement paste was molded and processed at the 60.0 °C for twenty-four hours. The final activated mortars were dried at room temperature in lab conditions for seven days and characterized. The unconfined compressive strength was tested by the CBR Tester device according to ASTM C39/C39M-18.

*2.3. Characterization of cement mortar*

The effect of adding PhC to the activated cement mortar was evaluated by calculating the mortars density (the ratio between the weight and volume of the cement mortar after curing). The amount of water absorption after immersing the mortars in de-ionized water for seven days (ASTM C187-16), according to equation 2:

$$A = ((W_w - W_d)/W_d) \times 100 \dots\dots\dots(2)$$

Where A is the absorption of water expressed in percentage, W<sub>w</sub> is the weight of the immersed specimen (g), and W<sub>d</sub> is the weight of the dry specimen after curing (g).

The shrinkage percentage was tested by measuring the change in the height of cement mortars after storage in dry conditions for twenty-one days (ASTM C827/C827M-16). According to the S equation:

$$S = [(H_0 - H_1)/H_0] * 100 \dots\dots\dots(3)$$

Where S is the difference in specimens high after curing (H<sub>0</sub>) and after twenty-one days of storage at room temperature (H<sub>1</sub>).

**3. Results and discussion**

*3.1. Characterization of PhC*

The PhC sample was characterized physically by testing the color appearance, specific gravity of solids (Gs), Atterberg limits (LL, PL, and PI) and hydrometer analysis to obtain the physical properties of PhC as presented in table 1. The collected sample of the PhC appeared as fine particles with a light creamy yellow color. The Gs was 2.43 g/cm<sup>3</sup>. The particle size analysis indicated that about 68% of PhC particles are in the range of silt fraction size (< 0.075 mm). The plasticity indices of LL, PL, and PI, are 33%, 21%, and 12%, respectively. According to USCS classification, the plasticity index of the present PhC is medium plastic sediments. The clay activity depending on the ratio between the plasticity index and clay size particle percentage indicated that PhC is a less active material. According to Skempton (1953), when the activity is less than 0.75 the material has low-volume change when wetted and low shrinkage when dried.

Chemically, PhC consists mainly of about 34.40% silica (SiO<sub>2</sub>), 32.35% lime (CaO), and 19.46% phosphorous (P<sub>2</sub>O<sub>5</sub>), as shown in table 2. PhC contain also a high concentration of some trace elements like, Cr, Ni, Sr, V, Zn, and U. Most of the trace elements are substituted in apatite and clay minerals or are adsorbed by organic matter in the phosphatic ore; being washed out after the beneficiation process causes a concentrate of these elements in the PhC (Dudka et al., 1994); (Al-Hwaiti, 2000).

**Table 1.** Physical properties of PhC

Color	Yellow or creamy
Gs	2.43 g/cm <sup>3</sup>
Particle size distribution Texture USCS	7% Fine sand, 68% silt, 24% clay Silt clay loam CL
Plasticity Limits Degree of plasticity Activity = PI / (% clay particles)	33% LL, 21% PL, 12% PI Medium plasticity 0.5%

Table 2. Major element composition of PhC

Major oxides	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SiO <sub>2</sub>	SO <sub>3</sub>	LOI
%	1.45	33.35	1.02	0.09	0.71	0.86	19.46	34.40	0.51	6.22
Traces	Cd	Cr	Cu	Ni	Sr	V	Zn	Zr	U	
PPm	3.23	231.80	15.41	97.21	575.31	127.94	132.58	18.27	45.72	

The X-Ray Diffractogram of the PhC is shown in Fig.1. The result indicated that the PhC is composed mainly of quartz, calcite, and carbonate apatite minerals as well as a minor amount of kaolinite and montmorillonite. PhC is represented as an argillaceous matrix that intercalated with all phosphate beds associated with pellets, teeth, and bones in the phosphatic ore (Khoury et al., 1988). The main conclusion from previous studies is that PhC should not be directly discharged into the environment (Slaty, 2005); (Al-Hwaiti et al., 2014). The utilization of PhC in industrial applications is considered as an effective waste management plan.

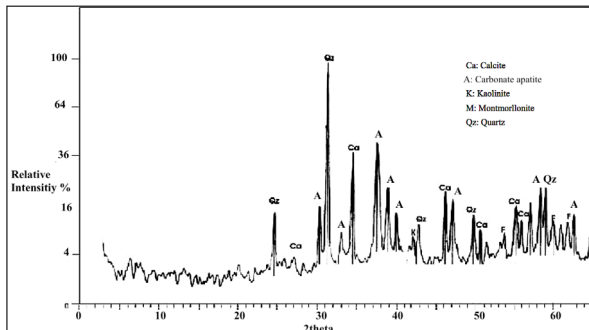


Figure 1. X-ray diffractogram of PhC

### 3.2. Characterization of the cement mortar

The effects of the addition of PhC to the aluminosilicate mixture were investigated by studying the changes in the index properties of cement mortars as presented in table 3. The workability of the cement mixture is largely dependent on the amount of added water. The ratio of water was measured according to the plasticity limits for solid components over which the mixture is in the plastic behavior. The plastic limit of HK as a raw material was around 30%.

The plasticity limit of 2HK: 1SS and 2HK: 1SS: 1PhC mixtures were 22% and 28%, respectively. The Addition of SS to HK already decreased the plasticity limit due to the increased porosity and air content. On the other hand, the adding of PhC to the aluminosilicate mixture resulted in increasing the limit of plasticity for the cement mixture. The increase of water content provides greater lubrication and helps increase the workability of the cement paste (Johnson, 1926); (Yen et al., 2012). The addition of PhC to the cement mixture resulted in increasing the mortar density (2.21 g/cm<sup>3</sup>) because of the increased plasticity limit. This has immensely affected the strength of the cement mortar giving the mortar more stiffness (Yen et al., 2012). After adding the PhC content, the activated cement mortar absorbed less water in immersing conditions for seven days of about (8.4%) compared to (10%) in the reference mortar. The shrinkage test indicated that the addition of PhC caused less shrinkage in the cement mortar of about (0.08%) compared to (1,5 %) in the reference mortar. The compressive strength test showed increase in the mortar strength after adding the PhC (36.48 MPa), compared to (32.53 MPa) of the reference mortar. This study found

that the addition of PhC to aluminosilicate mixture could simultaneously accelerate the activation process and achieves better performance.

Table 3. The index properties of the activated cement mortars

	2HK:1SS (Reference)	2HK:1SS: 1PhC
Pl%	22.0	28.0
Bulk Density (g/cm <sup>3</sup> )	2.0	2.21
Water absorption%	10.0	8.4
Shrinkage %	0.15	0.08
Compressive Strength (MPa)	32.53	36.48

### Conclusion

PhC is a medium plastic moldable material. In accordance with the results of this study, adding PhC with an appropriate ratio to the aluminosilicates mixture improve the characteristics of the cement mortar. The results showed an increase in the mortar density and the compressive strength of the cement mortar and a decrease in the percentage of water absorption and shrinkage. On the basis of these results, PhC could be a suitable by-product waste to be used in constructions as a cement admixture. After all, it can be truly useful for managing the huge amounts of PhC produced daily from the phosphate mines. However, further experiments on the quality and permanence of the cement mortar are recommended.

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### تأثير اضافة مادة الطين الفوسفاتي في خواص قوالب الاسمنت المضاعل قلوبيا

فاتن مصطفى السليتي

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### خلاصة

يهدف هذا البحث الى دراسة تأثير اضافة مادة الطين الفوسفاتي - التي تلحق من عملية تركيز الفوسفات الاردني- في خواص القوالب الاسمنتية المصنعة من مواد قليلة التكلفة. يهدف البحث في امكانية اعادة استخدام هذه المادة في البناء عوضا عن طرحها في برك طينية حيث تتراكم بكميات ضخمة مع زيادة عملية التعدين ويزداد تركيز العناصر الثقيلة فيها مما يجعلها تشكل خطرا على النظام البيئي في منطقة التعدين. خلال الدراسة تم تحضير خليط اسمنتي من مواد المنيوسيليكاتية ومفاعله قلوبيا لتشكيل قوالب اسمنتية قوية ومتماسكة. ومن ثم تشكيل قوالب اسمنتية من نفس الخليط الاول مضافا له نسبة 25% من مادة الطين الفوسفاتي. وقد دلت الفحوصات المخبرية للخواص الاساسية للقوالب الاسمنتية المحضرة، ان كثافة القوالب الاسمنتية قد زادت بعد اضافة الطين الفوسفاتي لها مما ادى الى زيادة في قوة القوالب الاسمنتية. وقد اشارت النتائج الى حدوث انخفاض في قابلية العينات لامتصاص الماء والانكماش في حجم العينات. وقد اوصت الدراسة بان امكانية استخدام هذه المادة مع خليط من المواد الالومنيوسيليكاتية يعتبر مجدي لتحسين خصائص الخليط الاسمنتي.

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