

## **UTILIZATION OF PARTIALLY DE-ALUMINATED METAKAOLIN SOLID WASTE AS A SOURCE OF SILICATE INSTEAD OF SAND IN PRODUCTION OF LIGHTWEIGHT BRICKS**

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### **ABSTRACT**

Partially de-aluminated metakaolinite (PDK) is a by-product of Aluminum Sulphate Co. of Egypt (ASCE) plant. PDK contains considerable amounts of active silicate. This study, examines the production of light-weight, made out of the PDK and lime. Cubic brick samples were produced through steam curing of the blend (70gm PDK, 15 gm cement, 10 gm quick lime, 2 gm gypsum, and 0.085 gm aluminum powder) in an autoclave in order to give the highest compressive strength (5 mPa) and water absorption 63%.

The findings of this paper suggest that the PDK solid waste can be used instead of the sand for the production of light weight bricks, resulting in reduction of the environmental pollution of that solid waste. The energy consumed in the production of lightweight bricks is only a fraction compared to the production of conventional bricks.

**Key words:** Partially de-aluminated metakaolin, lightweight brick, quick lime, tobermorite, aerated autoclaved concrete.

## INTRODUCTION

In view of the worldwide interest of having a clean and healthy environment, the search for useful application of waste by-product materials from different industrial sectors has become a necessity, as the amount of waste produced by industry is increasing all the time, while the Earth's capacity to decompose these residues is declining (Abdel Aziz *et al.*, 2010). For this reason, policies encouraging the utilization of waste products are becoming increasingly popular to avoid the negative impact on the environment and human health.

Since the past few decades, there has been raising concern over the inefficient use of resources for building construction in most countries leading to the need for the use of the alternative building materials. The United Nations Commission on Human Settlements report, for instance, emphasized the need to promote "appropriate technology" in the construction industry in developing countries as one of the ways of promoting sustainable construction. (UNCHS, 1993).

So, a popular trend by researchers has been to incorporate wastes into fired clay bricks to assist the production of normal and lightweight bricks. The utilization of these wastes reduces the negative effects of their disposal. Many attempts have been made to incorporate waste in the production of bricks including rubber, limestone dust, wood sawdust, processed waste tea, fly ash, polystyrene and sludge. Recycling the wastes by incorporating them into building materials is a practical solution to pollution problem. The

utilization of wastes in clay bricks usually has positive effects on the properties, although the decrease in performance in certain aspects has also been observed. (Tang *et al.*, 2020), The positive effects such as lightweight bricks with improved shrinkage, porosity, thermal properties and strength can be got by incorporating the recycled wastes. (Kadir & Mohajerani, 2011).

In general, bricks are categorized into various groups according to their major mineral composition such as silica, zirconia, alumina, mullite, magnesite and dolomite bricks. (Najar, 2016).

PDK is considered as a synthetic pozzolanic character that is thought to be provided by the aluminate sand amorphous silicate minerals that contains. Pozzolanic material has the ability to react with slaked lime and water. The reaction occurring between lime and silica occurs based upon  $\text{CaO-SiO}_2\text{-H}_2\text{O}$  (C-S-H) formulation (Ball & Carroll, 1999), (Baoju *et al.*, 2001), (Ma & Brown, 1997), (Chan *et al.*, 1999). Hydration reactions can also take place to form  $\text{CaO-Al}_2\text{O}_3\text{-SiO}_2\text{-H}_2\text{O}$  (C-A-S-H) phases contributing to the strength of the final product (Cicek & Tanriverdi, 2007). In order to achieve that and to accelerate the reaction kinetics, curing process should be conducted under pressurized steam at 125–200 °C in an autoclave. This property of PDK provides an important advantage for the utilization of PDK in production of construction materials.

The strategies aim to employ simple building blocks manufacturing technology, which not only reduce the building cost but also restrict the environmental effects. Subsequently, policy and regulatory strategies have

been made by decision makers in most countries based on the international proposals to promote sustainable development since the early 1990s (Mpakati-Gama *et al.*, 2012).

PDK is obtained from the Aluminum Sulphate production factories, is derived from acid leaching of Metakaolin (MK) (daily production is about 200 tons of wet material) this waste causes a great environmental problem, as a result of the de-alumination process, the  $\text{SiO}_2/\text{Al}_2\text{O}_3$  ratio and Brunauer-Emmett-Teller (BET) surface area analysis values are increased, the increasing of specific surface area observed as consequence of acid attack was found due to the increasing of the pore volume without an appreciable increasing of the mean pore size distribution. (Mostafa & Brown, 2005, and Mostafa *et al.*, 2001).

The pozzolanic activity of PDK in blended cements was also demonstrated by the same method. They also reported that DK exhibits higher BET values than silica fume. Other researchers have also shown that the porous structure of calcined kaolin particles doesn't collapse owing to the acid attack (Macedo *et al.*, 1994). It was found that, the isothermal calorimetric tests of different mixes of PDK and lime, show that PDK has an appreciable pozzolanic activity (Abdelaziz *et al.*, 2010).

According to ASTM C595, a pozzolan is defined as "a siliceous or siliceous and aluminous materials which in itself possesses little or no cementitious value but in the presence of moisture, chemically react with

calcium hydroxide at ordinary temperature to form compound possessing cementitious properties”.

Production of sand/lime or Silica/lime bricks is based mainly on caustic lime (CaO), Silica and water. These components form certain compound called calcium silicate hydrate (C-S-H). This formed under pressurized steam at 125-200° C. at the beginning, lime-rich tobermorite gel is formed. It is composed of  $C_7S_4H_n$  this phase reacts with residual silica to form  $C_5S_4H_n$  and finally the low lime  $C_2S_3H_2$  phase is formed. (Stepien *et al.*, 2019).

The steam autoclaved (PDK) based partially on the formation of calcium silicate hydrate (C-S-H) phase. Hydration reaction can also take place to form Ca-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-H<sub>2</sub>O (C-A-S-H) the presence of Al<sub>2</sub>O<sub>3</sub> contributes to the strength of the final product and hardening of PDK/Lime mixture (Cicek & Tanrıverdi, 2007). The type of materials and firing temperature are two major factors affecting the final product quality. Additives are frequently used in brick production for gaining special characteristics such as lightweight, better durability and good insulation. One way to increase the insulation capacity of bricks is to generate porosity in the brick structure.

**Table (1):** Chemical composition of autoclaved light weight brick (Mestnikov *et al.*, 2016).

Parameter	D500
Ingredients of the raw materials per 1 m <sup>3</sup> of light weight brick, Kg	
Cement	190
Lime	40
Sand	290
Water	210
The average density of dry materials ( kg/m <sup>3</sup> )	
536	
The compressive strength (MPa)	
2.6	

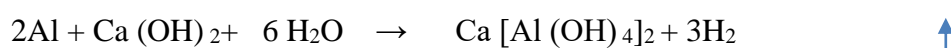
The composition of autoclaved light weight brick is presented for the class D500 in Table (1).

This study investigated the usage of the solid waste of aluminum sulphate industry commonly known as partially De-aluminated metakaolin (PDK) as an alternative of sand in the previous ingredients. To produce a special class of lightweight brick.

## MATERIAL AND METHODS

Production of light weight bricks: In this study, the Partially De-Aluminated Metakaolin (PDAMK) was collected from a large storage area at the Aluminum Sulphate Co. of Egypt (ASCE) located in Abu Zaabal district, about 40 km from Cairo. The sample was ground to avoid large particles and to be more reactive in the reactions.

The quick lime was prepared by calcinations of calcium carbonate at 950 °C for two hour. Quick lime reacts with silica of PDK and water forming certain compound called calcium silicate hydrate (C-S-H). This compound is formed under pressurized steam at 125-200° C. Fine aluminum powder also was used to produce hydrogen gas forming fine pores, according in this equation:



In this part of the study, different mixes were prepared and poured in standard moulds 100\*100\*100 mm cubic regarding different temperature and steam pressure. Lightweight brick samples, were produced using a full scale autoclave, for steam autoclaving. ELE autoclave with automated steam pressure control was conducted. A hydraulic press with max. 5 tons capacity was employed for brick forming and mechanical strength tests assessing of the different proportions of the ingredients (PDK, cement (OPC 52), quick lime, gypsum, aluminum powder). Gypsum was used to increase the brick strength and reduction water absorption.

In order to find the optimum parameters for brick production, the test results given in Table 3 was followed. The quick lime powder used with grain size < 45 µm and the quick lime (CaO) content is 66%.

Chemical analysis was carried out using [Axios (PW4400) WD-XRF Sequential Spectrometer]. XRD analysis was recorded using [a Philips PW 3050/60 Diffractometry] with a Cu-Kα source with a post sample Kα filter.

XRD patterns were collected from 5 to 50 2 $\Theta$ . Silica was used as an internal standard. Data were identified according to the XRD software.

**Determination of density:** The density of both fresh and hardened concrete is important as it can give an idea related to concrete durability, strength and resistance to permeability. Hardened concrete density is determined either by simple dimensional checks, followed by weighing and calculation or by weight in air/water buoyancy methods. (Turney *et al.*, 2009) In this research simple method to determine the density of lightweight concrete sample was using the formula given below: Density = Average Weight of Samples (kg) / Volume of Sample (m<sup>3</sup>).

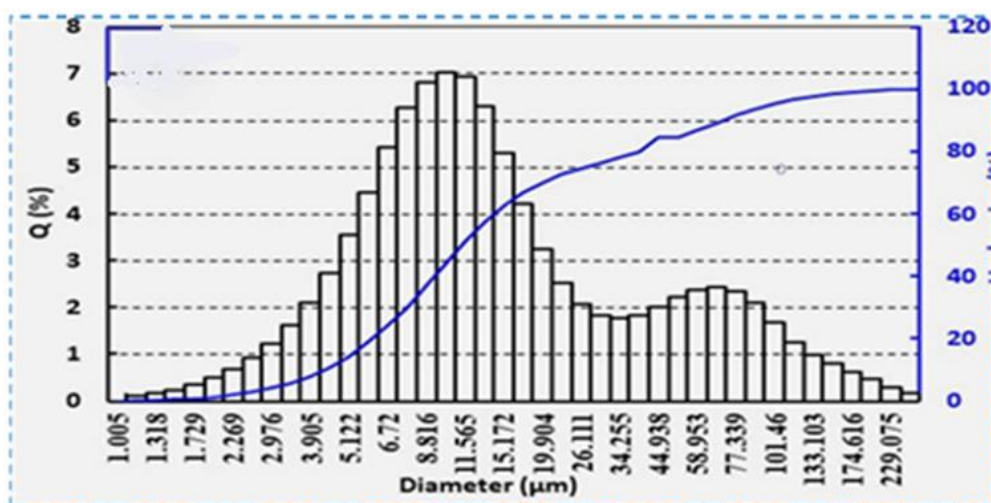
**Determination of water absorption:** Water absorption is defined as the transport of liquids in porous solids caused by surface tension acting to the capillaries (Khatib & Mangat, 2002).

This test was needed to determine the capability of concrete cubes to adsorb water. The method of determination water absorption capacity is determined by finding the weight of surface-dry sample after it has been soaked for 24 hr. and again finding the weight after the sample has been dried in an oven the difference in weight, expressed as a percentage of the dry sample weight, is the absorption capacity. Absorption capacity can also be determined using British Standards (BS) absorption test. Result for each sample for every batch will be compared with controlled sample (Ibrahim *et al.*, 2013).



## RESULTS AND DISCUSSIONS

Characterization of partially de-aluminated metakaolin: PDK samples were obtained from ASCE plant, it was adopted as a source of activated silicate. The samples were dried in an oven at 100°C for 12 h. The dried material was ground and dispersed in water as slurry (ground-slurry). The B.E.T. analysis revealed a high fineness for PDK (43 m<sup>2</sup>/g for PDK) and the specific gravity was 1.36. PDK as being coarse, it was ground and plotted in Fig.1.



**Fig. (1):** Particle size distribution of Partially De-Aluminated Metakaolin

The particle-size distribution of the PDK sample shows that the diameter of PDK in cumulative Percent is 90 % of ~70.33 μm and 10 % of ~4.329 μm with an average diameter of ~9.445 μm. This might be attributed to the surface texture of particles characterized by high roughness already observed

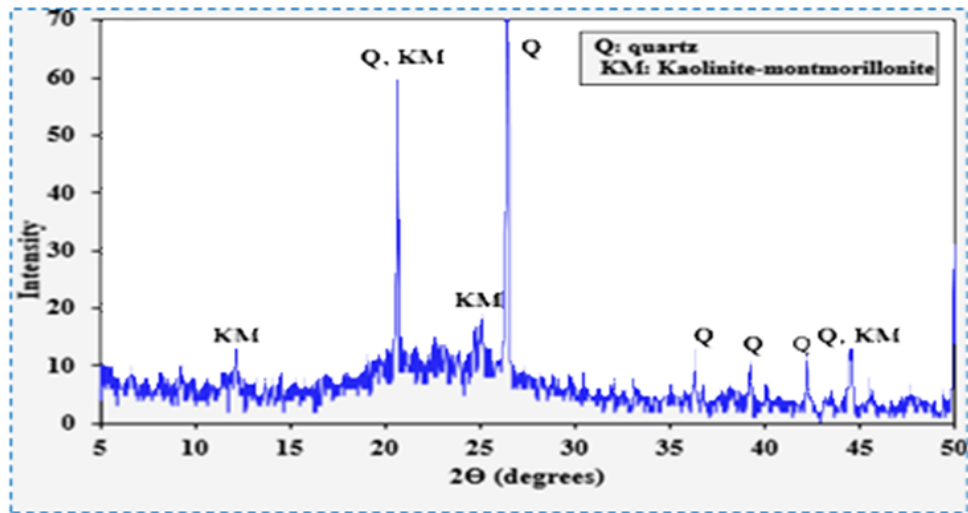
by (Vollet *et al.*, 1994). The raw material was also subjected to chemical analysis for determining its composition and the results are tabulated in Table 2.

**Table (2):** The chemical composition of Partially De-Aluminated Metakaolin

Parameter	Concentration (%)	EN 450-I Limits
SiO <sub>2</sub>	83	Not less than 25 %
Al <sub>2</sub> O <sub>3</sub>	5.5	-
Fe <sub>2</sub> O <sub>3</sub>	0.5	-
SiO <sub>2</sub> + Al <sub>2</sub> O <sub>3</sub> + Fe <sub>2</sub> O <sub>3</sub>	89	Not less than 70 %
TiO <sub>2</sub>	3.2	
MgO	0.09	Less than 4 %
CaO	0.15	-
Na <sub>2</sub> O	0.03	Less than 5 %
K <sub>2</sub> O	0.05	
SO <sub>3</sub>	0.85	Less than 3 %
P <sub>2</sub> O <sub>5</sub>	0.01	Less than 5 %
SrO	0.04	
Cl <sup>-</sup>	0.06	Less than 0.1 %
L.O.I	5.6	Category A not greater than 5 % Category B not greater than 7 % Category C not greater than 9 %
Total	99.08	

The composition of PDK of SiO<sub>2</sub>+Al<sub>2</sub>O<sub>3</sub>+Fe<sub>2</sub>O<sub>3</sub> is about 89.33% and CaO ratio was determined as 5.34%. Based on this values, the PDK can be classified as F type of pozzolan according to ASTM C 618 (C618-12a, 2019) standard and as indicated in Table 2 for EN 450 I (BS EN, 2012), hence according to IS3812:2003 (IS, 2003) this PDK can be used as a pozzolanic material.

The mineralogical characteristics of PDK are shown in Fig. 2. XRD pattern of PDK shows quartz peaks and amorphous background.



**Fig. (2):** XRD traces of Partially De-Aluminated Metakaolin

**Table (3):** Test results for light weight brick production with autoclave, using 2 gram of gypsum and 0.085 gram of aluminum powder and the drying Time was 6 Hours

Test No	Sand (g)	PDK (g)	Cement (g)	Caustic lime (g)	Water Added (g)	Compressive strength (MPa)		Water absorption (%)	Density (g/cc)
						Dry	Wet		
Ref. AAC	70	0.0	15	13	53	5.0	4.5	50	0.5
B1	0.0	70	5	13	90	3.6	3.2	81	0.48
B2	0.0	70	10	13	90	4.3	3.6	72	0.48
B3	0.0	70	15	13	90	4.5	4.1	66	0.50
B4	0.0	70	15	10	90	5.0	4.6	64	0.51
B5	0.0	70	15	14	90	4.9	4.3	73	0.50
B6	0.0	70	15	18	90	4.0	3.3	82	0.49
B7	0.0	50	15	10	90	3.7	3.1	90	0.48
B8	0.0	60	15	10	90	4.2	3.7	83	0.50
B9	0.0	70	15	10	90	5.0	4.6	63	0.51
B10	0.0	70	18	10	90	4.4	4.0	60	0.52
B11	0.0	70	20	10	90	4.8	4.2	61	0.50
B12	0.0	70	22	10	90	4.9	4.6	63	0.50
B13	0.0	70	18	12	90	5.0	4.5	71	0.49
B14	0.0	70	18	14	90	5.0	4.3	75	0.40

**Determination of the optimum materials blend ratio:** According to the test design and the obtained results (Table 3), the reference sample represents the current production of the light weight brick or the Aerated Autoclaved Concrete (AAC) using the sand which was exchanged by PDK in the tested blends. For the blends B1, B2, B3, constant masses of PDK, quick lime, gypsum and aluminum powder: 70, 13, 2, 0.085 g respectively. The cement was used with different masses 5, 10, 15 g for the three trials respectively. The Compressive Strengths (CS), water absorption and density were determined and depicted in Table 3 and represented in Fig. (5, 6, and 7). It was found that highest compressive strength (4.5 mPa) was recorded for the brick which was prepared using highest mass of cement (15 gm) in (Fig 3), this value was complied with that reported by (Mestnikov *et al.*, 2016) for class D500 lightweight bricks (Table 1), but it was relatively lower than that obtained from the reference sample (AAC) (5.0 mPa). Different matrices were done to optimize the ratios of the ingredients till getting the highest recorded CS with low water absorption maintaining the targeted density. The blend B9 achieved a CS of 5 mPa as that of the reference light weight brick, but the water absorption was relatively higher (63 %) than the reference (50%).

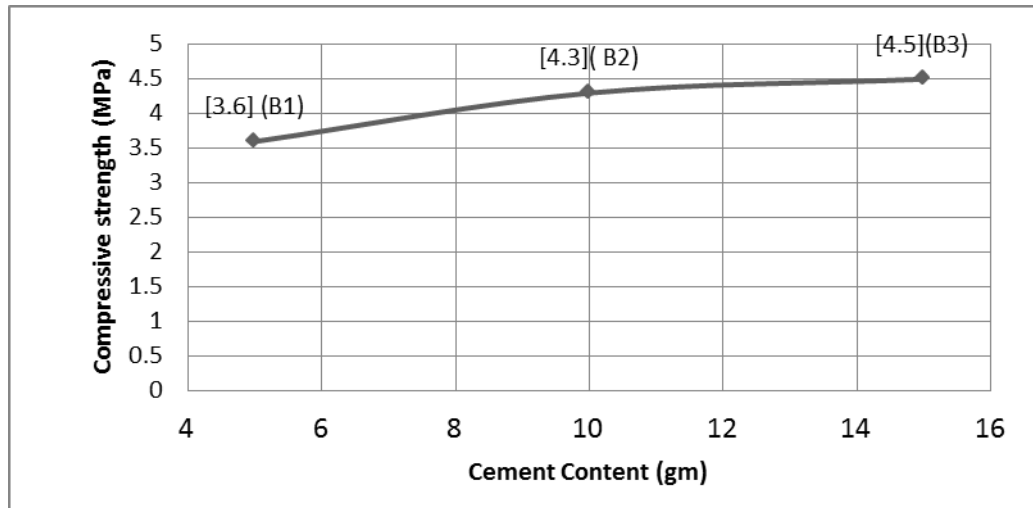


Fig. (3): The effect of cement content on the compressive strength

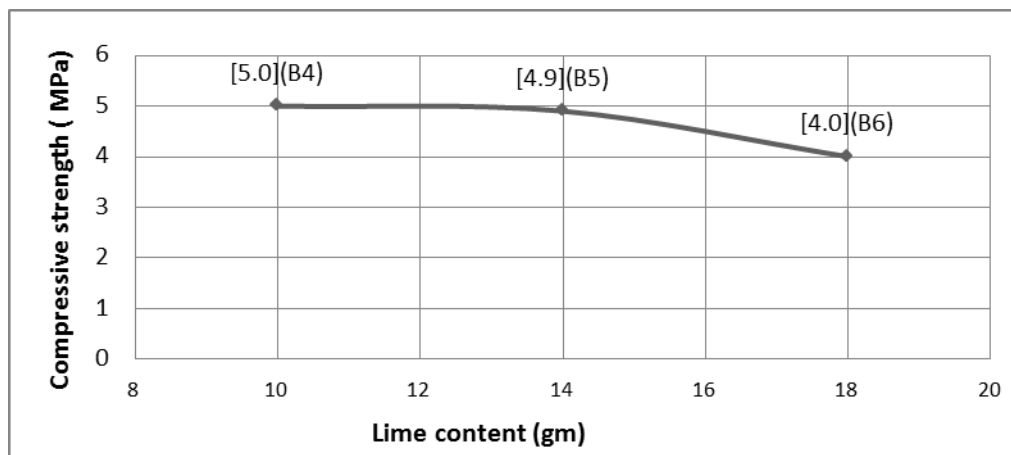


Fig. (4): The effect of lime content on the compressive strength

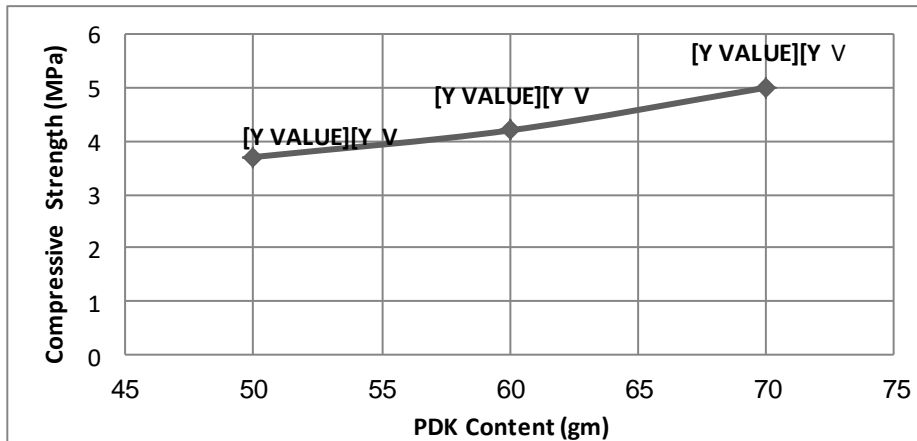


Fig. (5): The effect of PDK content on the compressive strength

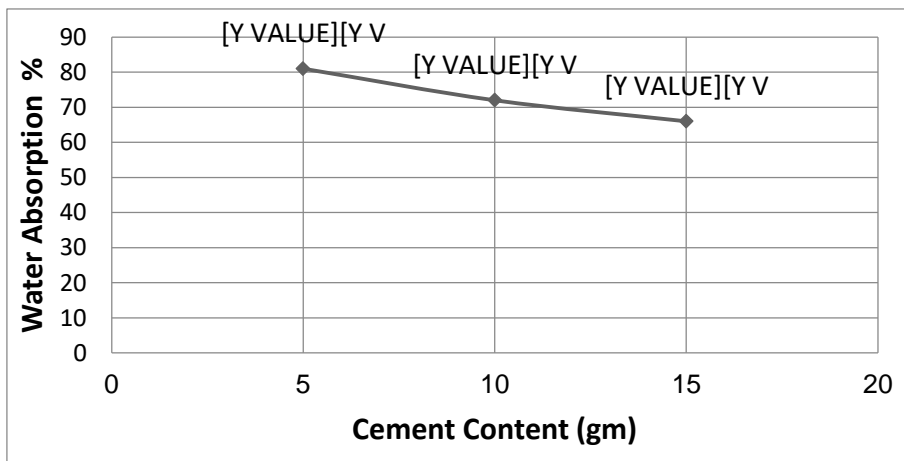


Fig. (6): The effect of cement content on the water absorption

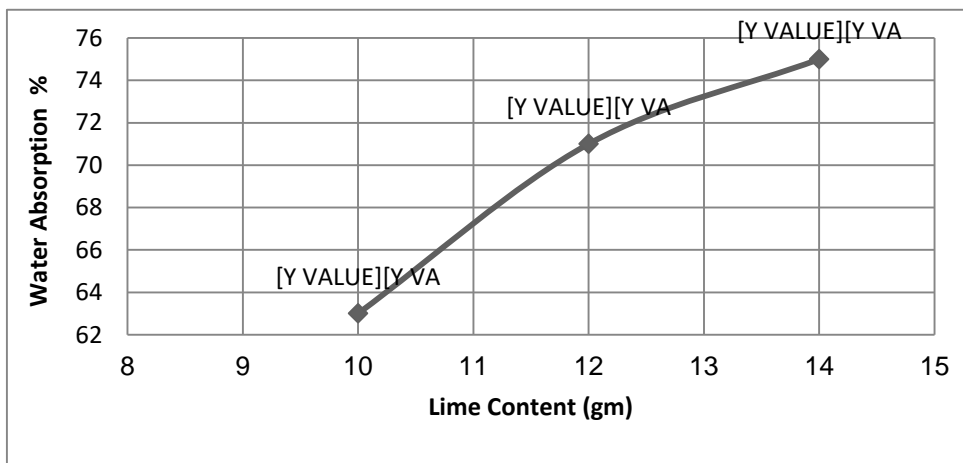


Fig. (7): The effect of lime content on the water absorption

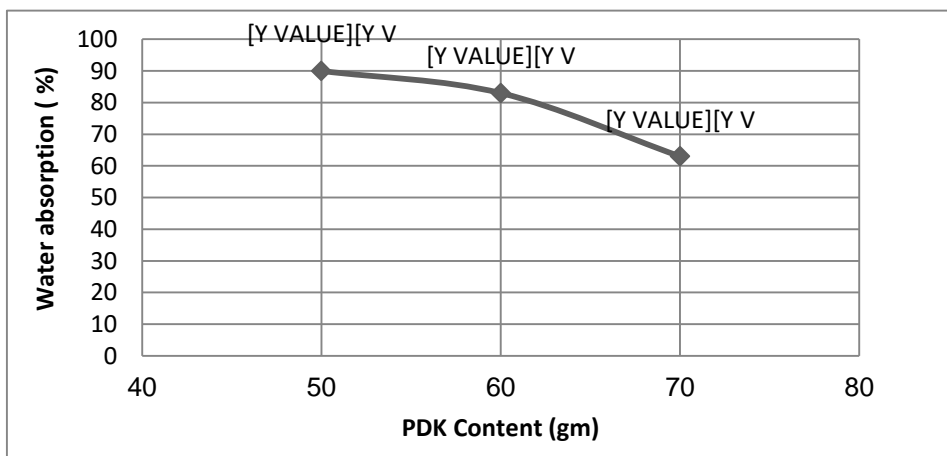


Fig. (8): The effect of PDK content on the water absorption



**Mineralogical composition of the brick:** The mineralogical investigation of the prepared brick showed that the brick are made of quartz ( $\text{SiO}_2$ ), anhydrite ( $\text{CaSO}_4$ ), calcite ( $\text{CaCO}_3$ ), tobermorite ( $\text{Ca}_5\text{Si}_6(\text{O.OH.F})18.5\text{H}_2\text{O}$ ) and calcium silicate hydrate ( $2\text{Ca}_{1.5}\text{SiO}_{3.5}\cdot x\text{H}_2\text{O}$ ).

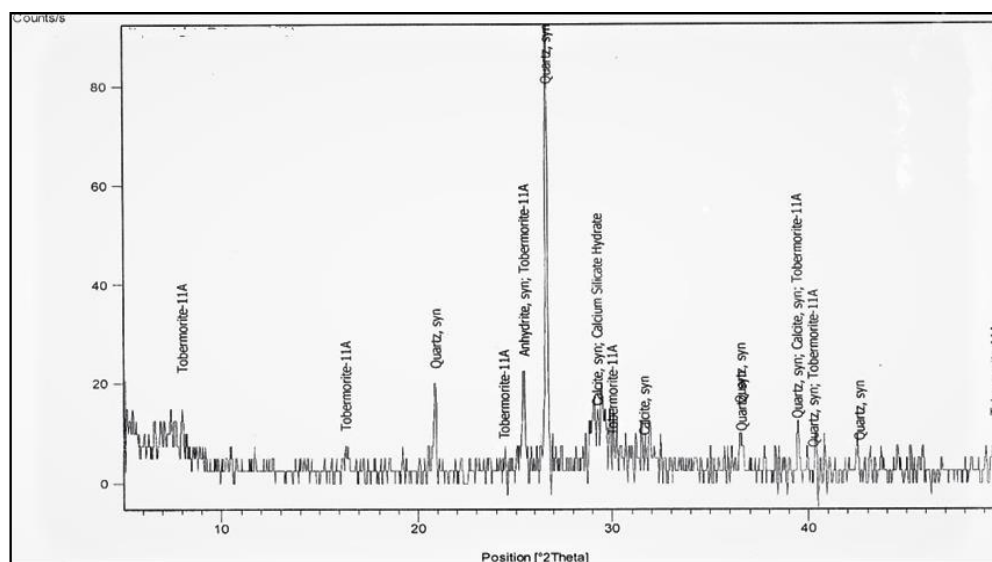


Fig. (9): XRD for light weight brick

In order to confirm the crystalline structure and the mineral existence in the cooked brick, XRD analysis gives the fingerprint of the crystalline contents. Figure (9) shows the presence of two binders that formed during autoclaving process. Tobermorite and Calcium silicate hydrate, tobermorite binder formation enhance the mechanical characteristics of the brick, it was formed from the hydrothermal reaction between silicate and lime at high temperature (150 – 200 °C) and pressure (10 – 12 bar). (Kovler, 2010)

Tobermorite structure is stable at the ambient temperature up to 150 °C. From the above XRF pattern calcium silicate hydrate (C-S-H) compound is present in the brick matrix it was formed through two steps: in the first step C-S-H will be generated on the surface of SiO<sub>2</sub> by the reaction with Ca(OH)<sub>2</sub>, in the second step, curing of the crystalline product, it is very important step because it develops the internal structure to form the stable, hard and durable compound (Ungkoon *et al.*, 2007). In this study curing was done by autoclaving at high temperature and pressure. The presence of anhydrite mineral is attributing to presence of gypsum added in the raw materials. The action of heating with pressure converted limestone to calcite and this product has no impact on the strength of the brick. The formation of tobermorite and calcium silicate hydrate binders is agreeing with that reported for lightweight brick as a fingerprint (Strokova *et al.*, 2013). The presence of quartz (SiO<sub>2</sub>) in the figure refers to impurities in the raw materials.

## CONCLUSION

Based on the results of the study the following can be concluded:

- PDK can used as a silicate source for the production of light weight brick with sufficient mechanical strength (5 MPa) and this would help in the mitigating the negative impacts of PDK on the environment.
- The binding component of the PDK, cement, lime brick are tobermorite (Ca<sub>5</sub>Si<sub>6</sub> (O.OH.F) 18.5H<sub>2</sub>O) as clearly shown in the XRD print.

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## استخدام الميتاكاولين المنزوع الألمنيوم جزئياً (مخلف طوب) كمصدر للسيليكات بدلاً من الرمل في إنتاج الطوب خفيف الوزن

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### المستخلص

يعتبر ميتاكاولين منزوع الألمنيوم جزئياً هو منتج ثانوي لمصنع شركة الشبة المصرية. يحتوي الميتاكاولين منزوع الألمنيوم على كمية كبيرة من السيليكات النشطة. يتناول هذا البحث دراسة إنتاج الطوب الخفيف باستخدام هذا المخلف مع إضافات أخرى. حيث تم إنتاج مكعبات من الطوب المعالجة الحرارية داخل الأوتوكلاف. وكانت نسب الإضافات كالتالي: (٧٠ جم ميتاكاولين منزوع الألمنيوم، ١٥ جم أسمنت، ١٠ جم من الجير، ٢ جم من الجبس، و ٠,٠٨٥ جم من مسحوق الألمنيوم) لإعطاء أعلى مقاومة للضغط (٥ ميجا باسكال) وامتصاص الماء ٦٣٪. تشير نتائج هذه الورقة إلى أنه يمكن استخدام تلك النفايات الصلبة بدلاً من الرمل لإنتاج الطوب خفيف الوزن، مما يؤدي إلى تقليل التلوث البيئي الناشئ عن تلك النفايات الصلبة، كما أن الطاقة المستهلكة في إنتاج الطوب الخفيف ليست سوى جزء بسيط مقارنة بإنتاج الطوب التقليدي. كلمات دالة: ميتاكاولين منزوع الألمنيوم جزئياً - الطوب خفيف الوزن - الجير الحي - توبرمورايت - خرسانة خفيفة الوزن.