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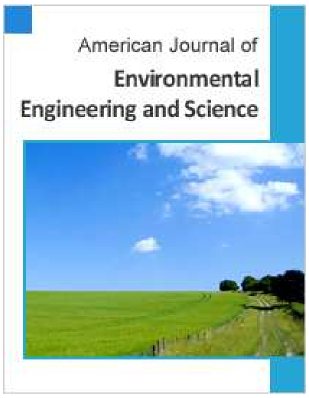

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# Ceramic Wall and Floor Tiles Containing Local Waste of Cement Kiln Dust - Part I: Densification Parameters

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## Abstract
The effect of using the local electrostatic precipitator cement kiln dust waste (EPCKD) collected from Tourah Portland cement factory on the production of ceramic floor tiles was studied. The EPCKD as received from the factory was used to replace gradually a part of the total batch composition of a traditional floor tile composition (clay, feldspar, limestone, quartz). The results showed that the EPCKD can be used with an amount of 5 up to 25 wt.% from the total batch. By using lower amounts, it needs higher firing temperature of 1180°C to reach the actual ceramic parameters (bulk density of 2.126 g/cm$^3$, porosity of 2.211% and water absorption of 1.041%). On the other side, with higher amounts of EPCKD waste, the prepared tiles reach the optimum ceramic characteristic parameters at lower firing temperature of 1150°C (bulk density of 2.23 g/cm$^3$, porosity of 0.33% and water absorption of 0.15%). So, the use of EPCKD waste in the production of wall or floor tiles has an additional advantage of lower fuel and energy consumption. The appropriate firing conditions of ceramic batches were also estimated and the variation of the ceramic properties was indentified.

## 1. Introduction

During the last fifty years, rapid scientific and technological advances in industrialization have been occurred all over the world. As a result, new health problems were created particularly those come from air pollution. Air pollution has excited from many countries but recently, it becomes much more severe and has adverse effects on man's health, animals and plants, and also on materials or other environmental media. Nowadays, because of growing industrialization, population and prosperity, the intensity and type of air pollution has globally increased [1, 2]. During the last two decades, a problem has been arose in the cement industry in Egypt consisting of the accumulation of large quantities of cement kiln dust waste behind the kiln filters reached to more than 2.5 million tons yearly, particularly after the conversion of production techniques from the wet process to the dry process (Fig. 1). The dry process with a precalciner represents the most up to date technique for the manufacture of Portland cement clinker with high productivities and low specific heat consumption [2, 3]. However, such process is characterized by the high intensity of the circulation phenomena of secondary constituents as alkalies, chlorine and sulfur between the preheater - calciner and the kiln if compared to other techniques such as semi-dry, semi-wet, long-dry and Lepol kiln techniques [4, 5]. During these processes, a large amount of airborne dust (1000 tons/day) in Assiut cement company as an example may be generated as a waste. The bypass dust is produced as a solid waste material from the preheater bypass systems by using the dry process as a result of some minor volatile constituents in the kiln feed and fuel (Fig. 1). The properties of this bypass dust waste depend mainly on the kind of raw materials and the fuel used as well as on the production method and the kiln type [6].

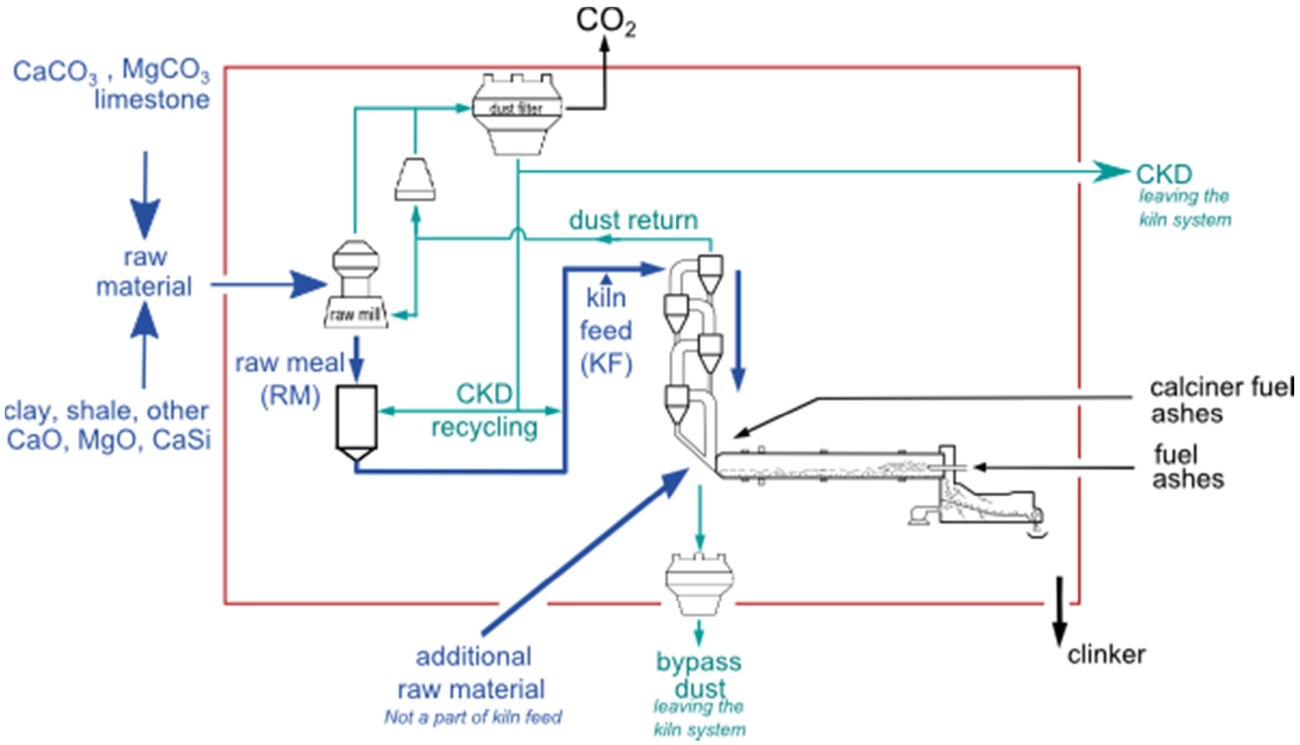


***Fig. 1.*** *A systematic diagram showing the discard of CKD or bypass dust waste.*

The EPCKD which is exhausted from the cement industry has very fine particles and a tendency to suspend in the atmosphere for a long time. Therefore, it can fly and spread over a wide area very easily even by means of very weak winds. Also, the EPCKD has high alkali content. Accordingly, it causes deleterious effects for the various bioecosystems [1, 6, 7]. Not only does it cause air pollution, but also it causes a very serious economic problem to all cement production companies. This is due to the accumulation of very huge amounts like hills of that EPCKD behind the kiln filters which approximately reach about 2.5 million tons yearly or may be little more. Table 1 shows the daily production of cement kiln dust waste (EPCKD) in some of the various cement factories in Egypt [1, 8].

As it is clear, the total amount of cement kiln dust waste in some of the cement factories as shown in Table 1 is about 5700 Tons /day. In USA for an example, the total amount of EPCKD waste is about 12 million tons yearly. Generally, about 18 wt.% of the feed kiln materials may be purged as a waste [9, 10].

It is very important to note that the use of CaO sources in ceramic field and specially wall and floor tiles is recommended as a whiting, flux and bleaching agent [11]. Accordingly and due to the nature and composition of EPCKD waste which contains high CaO content together with alkali oxides ($Na_2O$ & $k_2O$) with a considerable part of $Fe_2O_3$, the EPCKD waste has often a powerful fluxing effect. So, it would be more suitable to use as a component of ceramic bodies [12, 13]. So, the main objectives of the current study are the exploitation of EPCKD waste as a source of CaO and a replacing material of the main ceramic raw materials (clay, quartz, feldspar) to produce wall and/or floor tiles with the acceptable ceramic parameter sto study the influence of this waste material on the densification properties of wall and floor tiles. This will eliminate or at least reduce the environmental pollution with this dangerous waste material and also will lighten the heavy burden on the cement factories concerning its disposal.

***Table 1.*** *The daily amounts of cement kiln dust waste (EPCKD) produced in some of the Egyptian cement factories.*

| No. | Factory | Tons/day |
|---|---|---|
| 1 | Tourah Cement Company | 350 |
| 2 | Helwan Cement Company | 550 |
| 3 | National Cement Company | 650 |
| 4 | Alexandri Cement Company | 200 |
| 5 | Suez Cement Company | 300 |
| 6 | Titan Cement Company | 650 |
| 7 | Cemix Cement Company | 350 |
| 8 | El-Amria Cement Company | 300 |
| 9 | El-Menia Cement Company | 250 |
| 10 | Al-Arabia for Cement | 350 |
| 11 | Al-Sweedy for Cement | 300 |
| 12 | South Valley for Cement | 400 |
| 13 | Arabian Cement Company | 250 |
| 14 | Assiut Cement Company | 1000 |
| | Total | 5700 |

# 2. Experimental

## 2.1. Raw Materials

The clay sample was taken from Toshka region (TC). Toshka region is located on latitude $20^{o}$ $30^{-}$ N and longitude $31^{o}$ $53^{-}$ E at 250 km south of Aswan which was related to the Upper Cretaceous age. The selected clay deposit is belonging to El-Dakhla Shale Formation (Fig. 2). About 20 kg clay was collected from the 85th km north of Aswan/Abu-Semple asphaltic road. It is a dark yellowish grey. The clay sample was first well dried in an open air for 2 days and in a suitable furnace at 110 °C for another 2 days, then crushed, ground and quartered to have a representative sample which was fine ground to pass 200 mesh sieves. Feldspar (F), quartz (Q) from El-Hekma firm, limestone (L) from Samalout district were obtained from the Arab Ceramic Company which is commercially known as Aracemco, Cement kiln dust (EPCKD) which was provided by Tourah Portland Cement Company and a broken fired ceramic waste with the commercial name Grog (G) from the Aracemco ceramic factory, Egypt. The chemical analysis using the X-ray fluorescence technique (XRF) and the particle size distribution of the starting raw materials are shown in Tables 2 and 3, respectively. The suggested ceramic batch composition from the above mentioned raw materials is given in Table 4. The base batch composition was well mixed in an agate ball mill for one hour using the wet method, let to dry and thoroughly crushed and ground well again to pass through 200 mesh sieve to be the stock base ceramic tile batch.

*Fig. 2. The map indicates the area from which the clay sample was taken.*

*Table 2. The chemical composition of the raw materials, wt.%.*

| Materials Oxides | T-Clay (TC) | Feldspar (F) | Quartz (Q) | Limestone (L) | Homra (H) | C. kiln dust (EPCKD) |
|---|---|---|---|---|---|---|
| L. O. I | 9.72 | 0.67 | 1.78 | 42.63 | - | 24.51 |
| $SiO_2$ | 53.47 | 75.37 | 93.63 | 0.08 | 58.22 | 12.84 |
| $Al_2O_3$ | 26.78 | 13.62 | 3.64 | 0.03 | 28.25 | 1.86 |
| $Fe_2O_3$ | 3.99 | 0.41 | 0.08 | 0.04 | 8.16 | 1.53 |
| CaO | 0.60 | 0.53 | 0.18 | 56.84 | 0.79 | 52.51 |
| MgO | 1.38 | --- | --- | 0.10 | 0.46 | 1.84 |
| MnO | 0.03 | 0.03 | 0.02 | --- | --- | --- |
| $K_2O$ | 1.18 | 5.84 | 0.14 | 0.05 | 1.46 | 1.65 |
| $Na_2O$ | 1.15 | 3.44 | 0.17 | 0.12 | 1.32 | 0.83 |
| $TiO_2$ | 1.12 | 0.05 | 0.16 | 0.01 | 1.34 | --- |
| $SO_3$ | --- | 0.02 | 0.14 | 0.02 | --- | 2.43 |
| $P_2O_5$ | 0.51 | --- | --- | --- | --- | --- |
| $Cl^-$ | --- | 0.02 | 0.06 | 0.08 | --- | --- |

***Table 3.*** *The particle size distribution of the raw materials, wt.%.*

| Range Raw materials | < 71 µm | 100-71 µm | >100 µm |
|---|---|---|---|
| Clay (TC) | 87.33 | 7.78 | 4.89 |
| Feldspar (F) | 89. 41 | 9.52 | 1.07 |
| Quartz (Q) | 98.67 | 1.21 | 0.12 |
| Limestone (L) | 96.78 | 2.93 | 0.39 |
| Grog (G) | 99.42 | 0.36 | 0.22 |
| EPCKD | 93.25 | 5.21 | 1.54 |

***Table 4.*** *The base batch composition, mass%.*

| Materials | Clay (TC) | Quartz (Q) | Feldspar (F) | Limestone (L) | Grog (G) |
|---|---|---|---|---|---|
| Wt.% | 46.04 | 18.71 | 18.71 | 9.35 | 7.19 |

Fig. 3 demonstrates the DTA-TGA thermograms of T-Clay sample. The endothermic peak at the temperature range of 700-900°C is due to the calcination of limestome. The two endothermic peaks at the temperature range 100-120 and 500-600°C are due to the evaporation of the absorbed and structural or hygroscopic water, respectively. The endothermic peak at the temperature range 500-600°C is due to the conversion of kaolinite ($AS_2H_2$) to metakaolin ($A_2S_2$), which in turn is converted to mullite phase ($A_2S_2$) at 980°C as follows:-

$$AS_2H_2 \rightarrow AS_2 \rightarrow A_2S_2.$$

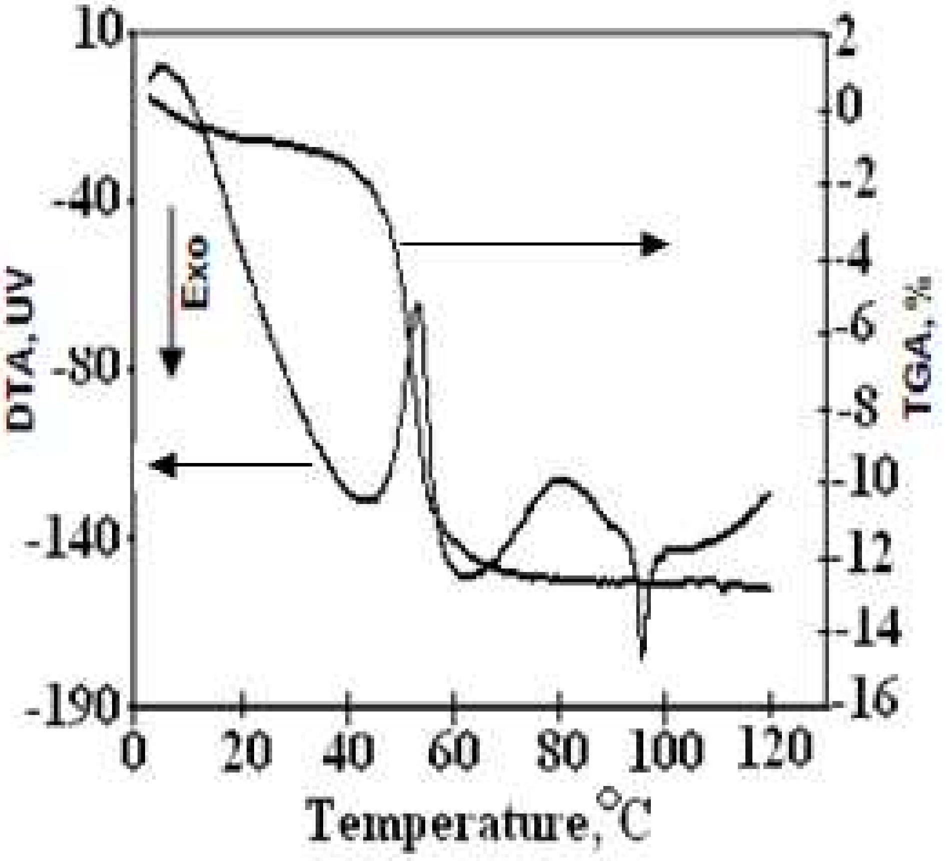


***Fig. 3.*** *The DTA and TGA thermograms of T-clay.*

## 2.2. Preparation and Methods

Six ceramic batches containing TC, F, L, Q, G and EPCKD were prepared as 100:0, 95:5, 90:10, 85:15, 80:20 and 75:25mass% having the symbols C0, C1, C2, C3, C4 and C5, respectively. The batches were mixed well in a gate ball mill for one hour using the wet method, dried at 105°C for 72 hours and then ground to pass a 200 mesh sieve to obtain the same homogeneity of all batches. Five disc-shaped samples of 2 cm diameter and 2 cm thickness were prepared for the physical properties in terms of water absorption (WA), bulk density (BD) and apparent porosity (AP), five rod-shaped samples of 2.5 x 2.5 x 7 $cm^3$for dry and firing bending strength and five cubes of 2.5 x 2.5 x 2.5 $cm^3$ for crushing strength were moulded. The molding of specimens were carried out under a shaping pressure of 20 $KN/mm^2$ using water as a binder. After demoulding, the samples were let to dry in air (23 ± 2ºC) for 48 hours and then dried to a constant weight at 105ºC in a suitable oven to ensure the complete elimination of the free water and to avoid the cracks during firing. The firing process was carried out by a slow rate furnace Mod. VECSTAR with a heating rate of 5ºC/min. The firing temperatures were in the range of 1000 and 1250ºC with one hour soaking time. The fired specimens were left to cool slowly inside the furnace over night to room temperature. The optimum firing temperature of each ceramic batch was estimated.

### 2.3. Densification Parameters

The ceramic or densification parameters (14) in terms of water absorption (WA), bulk density (BD) and apparent porosity (AP) could be determined from the following equations:

$$\text{W. A,\%} = (W1\text{-}W2) / (W3) \times 100 \quad (1)$$

$$\text{B. D, g/cm}^3 = (W3) / (W1\text{-}W2) \quad (2)$$

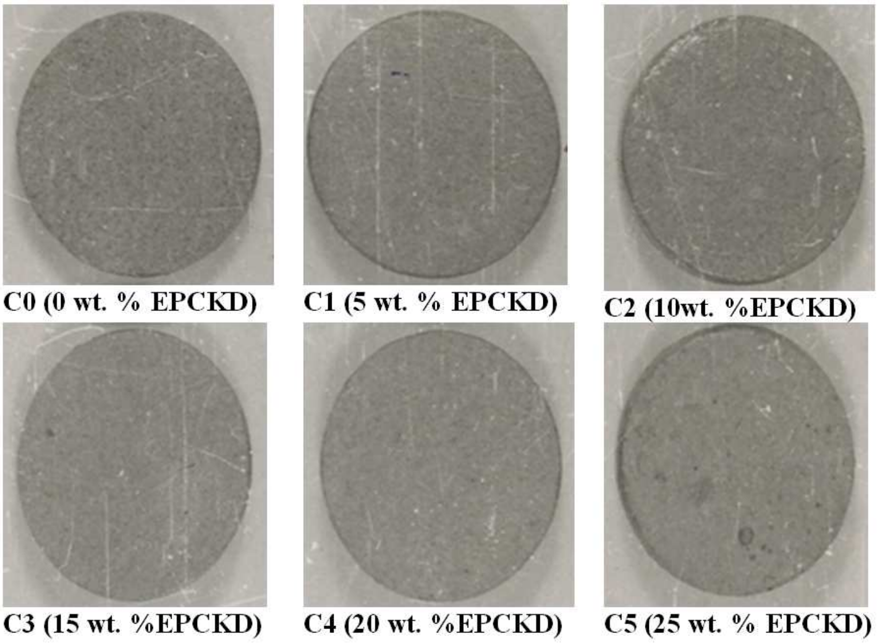
$$\text{A. P,\%} = (W1\text{-}W3) / (W1\text{-}W2) \times 100 \quad (3)$$

The XRF analysis is equipped by a modern wavelength dispersive Spectrometer (WD-XRF, 2005, Netherlands), provided with an integrated preparation unit including grinding, pressing and automatic bead preparation Instrument (Perl' X3, SAVOILOR S.A.S, France). Seven powdered samples as received were prepared for analyses in two forms: Fused beads for the determination of major elements which were carried out by melting one gram of the powder using 10 gram element flux. The latter is composed of a mixture of Lithium tetra-and metaborates in a ratio of 66:34, respectively. Complete melting was achieved using an automatic bead preparation Instrument (Perl' X3, Savoilor S.A.S, France). The second is pressed pellets for the determination of trace elements. The pellets were prepared by mixing 7 grams of the sample with 1.6 gram of binding wax through the computerized Mini-Mill-II grinding machine for one minute on 380 rpm speed. Then, the mixture was pressed in a standard aluminium cup using an automatic HTP 40 H, HERZOG pressing machine under 130 KN. The particle size distribution of the starting raw materials was carried out using the sieve analyzing method. The DTA-TGA thermograms of T-Clay sample which was carried out using NETZSCH Geratebae GmbH Selb, Bestell No. 348472c at a heating rate 10°C/min. up to 1000°C. The XRF analysis was carried out in the National Research Centre while the particle size distribution was achieved in the Metals Institute, El-Tabine, Cairo, Egypt.

## 3. Results and Discussion

### 3.1. Densification Parameters

Fig. 4 shows the different six ceramic batches containing various ratios of EPCKD after firing. It is well known that the densification of the resulted bodies after firing is evident from the values of water absorption where the floor tiles are known as dense and fully vitrified tiles, frequently with water absorption from 0-5% whereas wall tile are more than this range. The water absorption of the fired articles is graphically represented as a function of firing temperature in Fig. 5. In the lights of the results, it was found that the addition of EPCKD waste causes a gradual decrease in the water absorption of the fired articles which was a general trend displayed by all ceramic batches at all firing temperatures (Fig. 5. Also, the maturing temperature was lowered at which the minimum value of water absorption was recorded. It was found to be 0.366 and 0.15% for batches containing 20 and 25 wt.% EPCKD fired at 1160 and 1150ºC, respectively without any deformation or surface bloating on the body shape. This represents a high degree of densification and could be applied as a heavy duty as floor tiles according to international and Egyptian specifications [1, 15, 16].


**C0 (0 wt. % EPCKD)** **C1 (5 wt. % EPCKD)** **C2 (10wt. %EPCKD)**

**C3 (15 wt. %EPCKD)** **C4 (20 wt. %EPCKD)** **C5 (25 wt. % EPCKD)**

***Fig. 4.*** *The prepared ceramic body batches containing0-25 wt.% EPCKD waste after firing.*

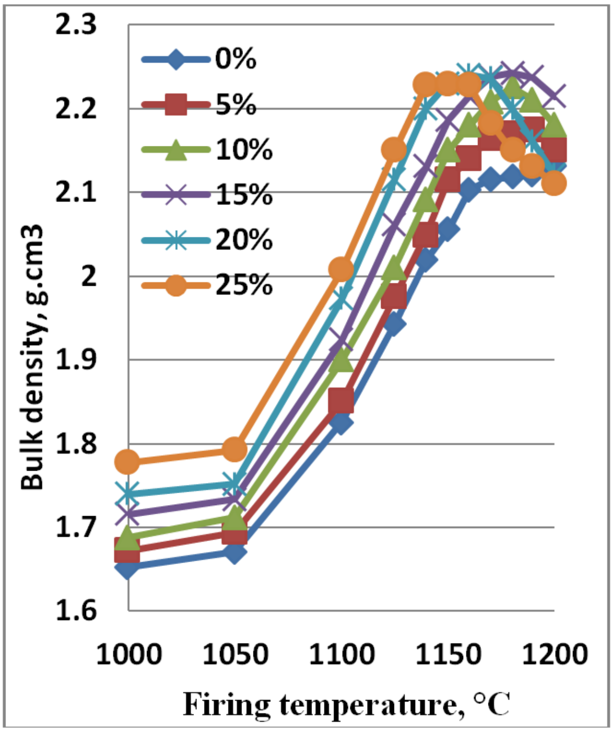


*Fig. 5. Water absorption of the fired ceramic bodies containing different EPCKDcontents.*

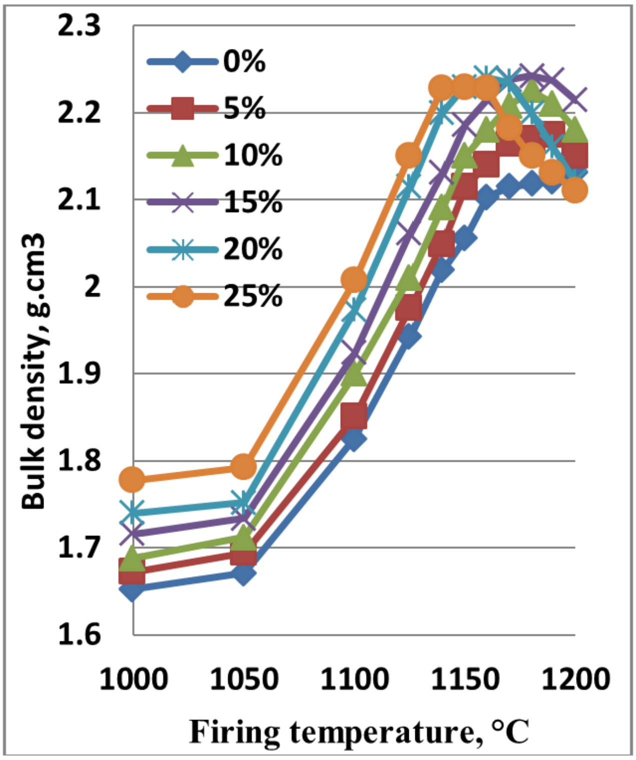


*Fig. 6. Bulk density of ceramic bodies containing different EPCKD contents.*

The bulk density of the fired articles is graphically represented as a function of firing temperature in Fig. 6. The increase of firing temperature improves and enhances the bulk density. The maximum value of bulk density for each batch was found to be at a temperature different than others, but coincides with that obtained before in case of minimum water absorption and ranged also between 1200-1150ºC. This means that the bulk density decreased by the increase of EPCKD content. The maximum bulk density was found to be 2.24 g/cm$^3$ for the batch containing 20 wt.% EPCKD fired at 1160ºC. With 25 wt.% EPCKD, the bulk density was slightly decreased indicating the creation of more pore structure.

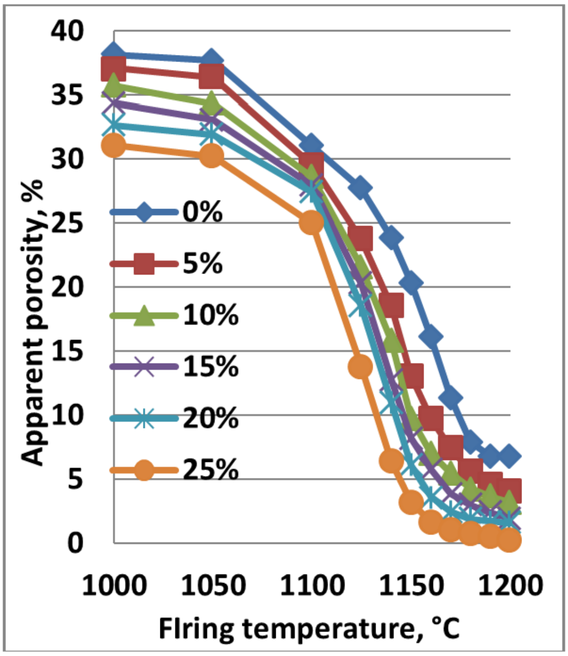


*Fig. 7. Apparent porosity of Ceramic bodies with different EPCKD contents.*

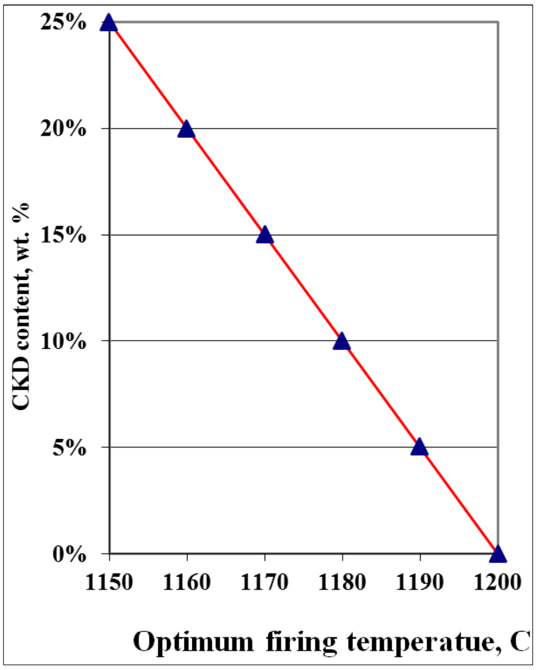


*Fig. 8. The optimum firing temperature in relation to EPCKD content.*

Fig. 7 shows the representation of the apparent porosity of the fired bodies as a function of firing temperature. It is obvious that the gradual addition of EPCKD decreased the apparent porosity slowly up to 1050ºC while stepwise decreased rapidly up to 1150ºC to reach its lower value for each batch at the suitable maturing temperature, and then increased up to 1200ºC. The maturing temperatures of the prepared fired bodies were decreased from 1200ºC with the base batch (C0) to 1150ºC with the batch (C5) containing 25 wt% EPCKD as shown in Fig. 8. So, whether the liquid phase is formed or not at these maturing temperatures, the firing was started with a compact powder of high porosity and finished with a fired body with porosity of 6.83%for the basic batch (C0) and 0.82 or 0.33% for batches containing 20 or 25 wt.% EPCKD, respectively.

The values of apparent porosity represent the relics of open pore system when the tested bodied reaching the maturing conditions. Since, the content of both open or closed pores governs and determines the degree of densification at these maturing temperatures, the minimum apparent porosity does not mean the maximum densification, except when these closed pores reach their minimum existence. However, the results of either apparent porosity or both water absorption and bulk density revealed that the tested floor tile bodies displayed to some extent a very low content of closed pore structure.

### 3.2. Relation Between Densification Parameters

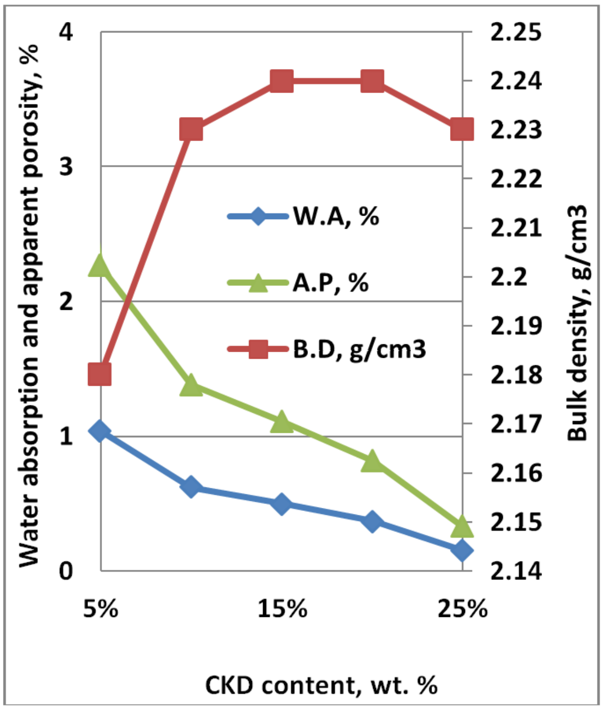


*Fig. 9. Relationship between water absorption, bulk density and apparent porosity of ceramic bodies with different CKD contents.*

Fig. 9 illustrates the relation between the water absorption, bulk density and apparent porosity of fired articles to compare between the base batch (C0) and the various composites containing 5-25 wt.% EPCKD. As it is clear, the apparent porosity decreased gradually with EPCKD content up to 25 wt.% which reflected positively on both bulk density and water absorption. This means that the addition of EPCKD to the base batch has some advantages. The first is to reduce the amount of EPCKD behind the kiln filters so as to avoid the environmental pollution with it. The second is the reduction of the expensive main raw ceramic materials. The third is to reduce the energy consumption.

### 3.3. General Discussion

There are two successive processes namely, sintering and vitrification [15, 16]. The reactions could be taken place between particles on heating as solid phase reactions which are the first step towards densification even when it is followed by the formation of liquid or not at all. This process is a mutual diffusion of atoms among the touching particles, which is known as “sintering” [15, 16]. As a result, the particles become fewer and larger, and then the pores are eliminated or decreased. The vitrification (i.e. glass formation) may start to form in the ceramic bodies above 900-1000ºC depending on the composition of these bodies. The particles of feldspar which used as a flux and EPCKD waste which used as a secondary or modifying flux could react with the constituents of other ingredients which they are in contact to form the liquid phase. The quantity of this liquid phase increases with the increase of firing temperature. Often, the fired ceramic bodies contract during firing due to the formation of that liquid phase which is known as “firing shrinkage”. So, the porosity of the bodies as well as water absorption reduced while the bulk density increased [15, 17, 18].

New phases may crystallize particularly as the temperature increased as needle-like crystals of mullite ($3Al_2O_3$. $2SiO_2$) which usually separated from the melt. On cooling, more other crystals may be separated and the liquid itself solidifies to a glass after cementing together the unmelted particles. This often finished by the formation of a glass matrix with crystal aggregates. The presence of MgO, CaO, $K_2O$ and $Na_2O$ is the main reason controlling the maturing temperature, liquid phase formation, crystallization of the new phases and the resulting densification properties. The MgO in EPCKD waste shares in the formation of cordierite (2MgO. $2Al_2O_3$. $2SiO_2$) and favors the conversion of quartz to into cristobalite [19- 21].

The presence of alkali oxides ($Na_2O$ and $K_2O$) from feldspar and EPCKD waste enhance the liquid phase content and lowered the viscosity at higher firing temperatures to improve the vitrification process. The formed liquid phase always favors the conversion reaction of quartz to cristobalite rather than the dissolution of MgO into it. However, the quantity of $SiO_2$ dissolved in the glassy matrix only has a small effect on the amount and composition of the liquid phase [16, 22].

The major influence of the alkalies is to alter the relative amounts of cristobalite and liquid phase up to the maturing temperature. Furthermore, the temperature dependence of the vitrification rate of a certain composition as that used in the current study is greater than that of viscosity alone. This is to be expected in the presence of EPCKD waste due to the increased liquid content at the firing temperatures to reach the suitable maturing conditions.

Also, the addition of CaO, as that existed in the EPCKD waste in forms of carbonates, sulphates or silicates, the formation of gehlenite ($2CaO. Al_2O_3. SiO_2$), wollastonite ($\beta$-$CaO.SiO_2$), anorthite ($CAS_2$ or $CaO. Al_2O_3. 2SiO_2$), spurrite ($2C_2S. CaCO_3$) as well as $C_3A$ was enhanced. So, the crystallization of these solid phases was increased which in turn enhances the densification properties of the ceramic body containing EPCKD waste [22, 23].

The improvements of the densification parameters, namely water absorption, bulk density and apparent porosity by increasing the EPCKD content seems to be due to the enhancement of the vitrification and sintering processes together to reach the best conditions that regulating and governing the function of the liquid phase to close the pores and not to open new ones as well as its role to conduct bonding of solid phases present together resulting in higher densities [24, 25].

## 4. Conclusion

Though not every city and land are interested in the use of EPCKD waste, it is now possible and practical to develop batch composition for the most ceramic wall and floor tiles applications having EPCKD up to a limited ratio of 25 wt.%. The water absorption and bulk density of the prepared ceramic products increased gradually, while the apparent porosity decreased as the EPCKD content as well as the firing temperature increased. The maturing temperature of the prepared ceramic articles was decreased by the increase of EPCKD content to reach 1150ºC instead of 1200ºC. This means that the densification parameters were improved and enhanced and all prepared samples displayed the same trend as EPCKD content and firing temperature increased. The bulk density was increased to 2.23 g/cm$^3$ with a porosity of 0.33% and a minimum water of absorption of 0.15%. Accordingly, the resulting ceramic products containing 20-25 wt.% EPCKD waste could be used as floor tiles due to its low water absorption and apparent porosity in addition to the high bulk density compared with those of the blank ceramic body.

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