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# Firing Resistance of Ordinary Portland Cement Pastes Incorporated a Waste of Building Bricks

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**Abstract:** Addition of fine materials, as clay bricks or Homra (H) to Ordinary Portland cement (OPC) is evidently increases resistance to firing. Effect of firing temperature on physical, mechanical properties and phase composition of cement pastes is investigated. H can react with lime liberated from hydration of OPC. This improves morphology and microstructure of formed hydration products. OPC cement was partially substituted by 0, 5, 15, 25 and 35 wt.% H. Cement pastes were subjected to firing for 3 hours at temperatures 100, 200, 300 400, 500 and 600°C. Results showed that loss on ignition of various cement pastes increased with temperature as well as with H. Density decreased with temperature up to 400°C, while porosity increased, Above 400°C, density increased and porosity decreased. Compressive strength of OPC pastes increased with temperature up to 500°C and then decreased. Those incorporated H increased only up to 400°C and then decreased. Hardened cement pastes with 15% H recorded highest compressive strength value at 400°C while the OPC at 500°C. SEM images showed a microstructure of well-developed crystals of CSH and CAH.  $\text{Ca}(\text{OH})_2$  are spreading in matrix at temperatures up to 400°C. There are some cracks at 600°C.

**Keywords:** Homra, Pozzolanic, Temperature, Density, Porosity, Strength, Microstructure, SEM

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## 1. Introduction

### 1.1. Scope of the Study

When the building materials are exposed to fire, some deterioration effects can occur. This can often reach to a level at which the structure may have to be thoroughly renovated or completely replaced. Heat resistance materials are usually used for structural purposes. The need for such building materials is particularly important in the chemical and metallurgical industries, and also for the thermal shielding of nuclear power plants. The hydrated Portland cement pastes contain a large amount of  $\text{Ca}(\text{OH})_2$ , which dehydrated to CaO between 500°C and 600°C. Exposure to moisture, the CaO is rehydrated to  $\text{Ca}(\text{OH})_2$  with a volume expansion of 97% accompanied with the formation of cracks [1, 2]. The pozzolanic materials can react with liberated lime from ordinary Portland cement (OPC) forming additional amounts of calcium silicate hydrates. The replacement of OPC by 10 wt.% silica fume improves the physicochemical properties as well as the microstructure of concrete when exposed to

elevated temperatures [3, 4].

The Portland cement containing 20±30 wt.% fly ash thus possesses a good fire resistance and dimensional stability when exposed to high temperature and then high humidity or wetting [5, 6]. The replacement of OPC by 20 wt.% of thermally activated kaolinite in cement paste increases its thermal stability against temperature up to 600°C [7].

Homra (crushed clay bricks) is a solid waste material, which is constituted mainly of silica quartz, aluminosilicate, anhydrite, and hematite. Therefore, it acts as a pozzolanic material [6, 8]. The effect of Homra on the main hydration characteristics of Portland cement at early ages by means of electrical conductivity measurements as well as the physicochemical and mechanical characteristics up to 90 days was studied [9-11].

### 1.2. Objectives of the Study

The objective of his work is to investigate the effect of elevated temperatures on the physical and mechanical properties as well as the microstructure of thermally treated cement pastes containing Homra.

## 2. Experimental Work

### 2.1. Raw Materials

The used raw materials in this research study was Ordinary Portland cement (OPC) and Homra (H) which is a broken waste of fired bricks (H) having a blaine surface area 3500 and 4100 cm<sup>2</sup>/g, respectively. The XRD patterns of H are shown in Figure 1. It is mainly composed of Quartz, Albite

and Hematite. The OPC cement was partially substituted by H with ratios of 0, 5, 15, 25 and 35 wt.% which were given the symbols H0, H1, H2, H3 and H4. The chemical compositions of raw materials are recorded in Table 1. The water of consistency and setting times (initial and final) of each cement batch [12, 13] were determined using Vicat Apparatus (Figure 2).

Table 1. Chemical composition of raw materials, mass%.

| Oxide Material | LOI  | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | CaO   | MgO  | Na <sub>2</sub> O | K <sub>2</sub> O | SO <sub>3</sub> | TiO <sub>2</sub> | Fineness, cm <sup>2</sup> /g |
|----------------|------|------------------|--------------------------------|--------------------------------|-------|------|-------------------|------------------|-----------------|------------------|------------------------------|
| OPC            | 2.64 | 20.12            | 5.25                           | 3.38                           | 63.13 | 1.53 | 0.55              | 0.31             | 0.3             | 1.86             | 3500                         |
| H              | ---  | 58.22            | 28.25                          | 8.16                           | 0.79  | 0.46 | 1.32              | 1.64             | ---             | 1.34             | 4100                         |

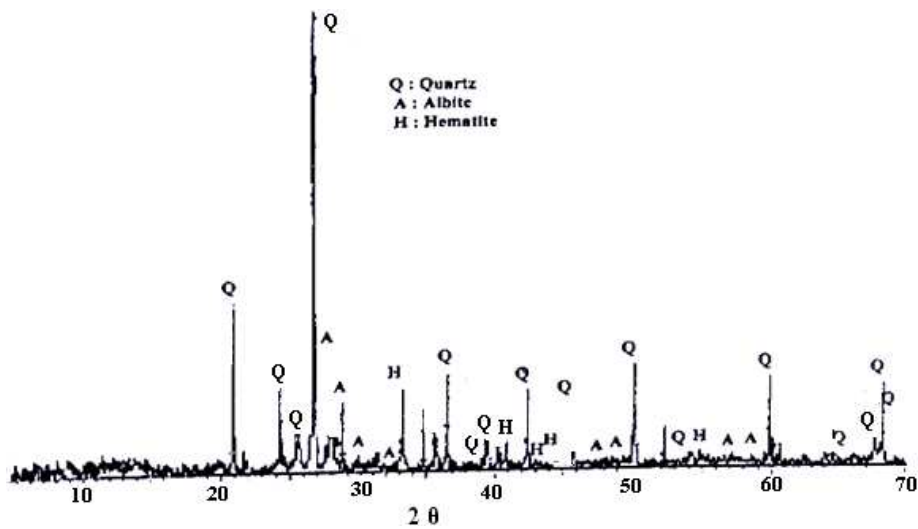


Figure 1. The XRD patterns of Homra waste (H). (Q: Quartz, A: Albite, H: Hematite).

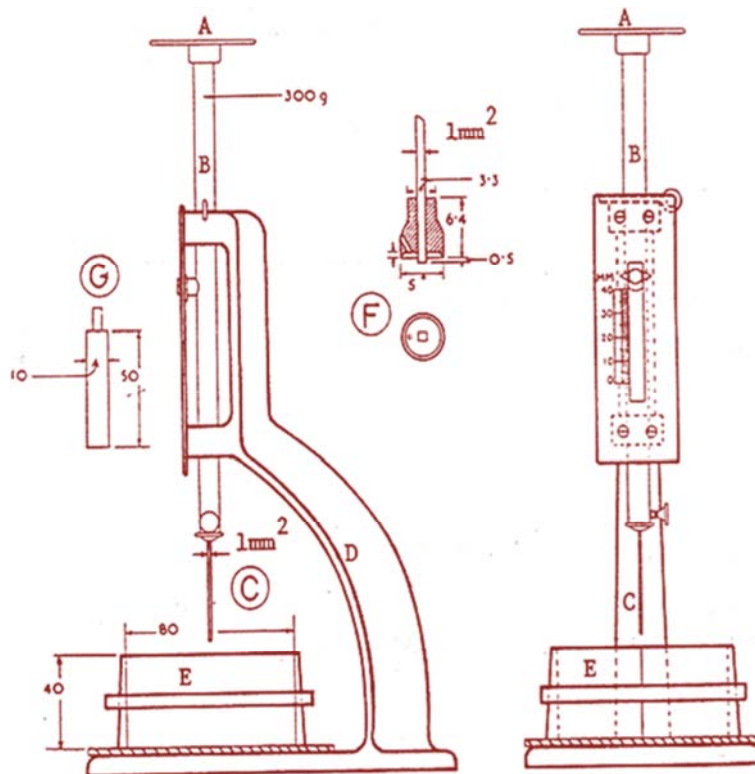


Figure 2. Vicat apparatus for determining the standard consistency water of and setting times of Portland cement pastes.

## 2.2. Preparation and Methods

The cement pastes of each batch were prepared using the measured amount of water of consistency. The cement pastes were mixed and molded into one inch cubic stainless steel molds. The molds were vibrated manually for 2 minutes to get rid of any air bubbles or voids. Then, it vibrated mechanically for another 2 minutes to ensure the complete elimination of all air bubbles. The surfaces of the pastes were smoothed using a suitable spatula. At once after molding, the specimens were kept in a humidity chamber at room temperature and 100% Relative humidity for 24 hours. In the following day, it was demolded and soon cured under water up to 28 days taking into consideration that water was daily changed. The density, apparent porosity and compressive strength were then measured, which were considered as zero readings. The hardened cement pastes were then dried at 105°C for 24 hours. The hardened cement pastes of all batches were subjected to temperature for 3 hours at 100, 200, 300, 400, 500 and 600°C. They let to cool over night till room temperature. After cooling, the ignition loss, apparent

porosity, bulk density and compressive strength were determined [14-16].

Scanning electron microscopy (SEM) of the fractured surfaces, coated with a thin layer of gold by JEOL-JXA-840 electron probe microanalyzer at accelerating voltage of 30 KV was used to identify the changes that are occurring in the microstructure of the formed or decomposed phases during hydration and heating to study the crystal morphology.

## 3. Results and Discussion

### 3.1. Water of Consistency and Setting Times

The water of consistency and setting times of the different OPC/Homra cement mixes (H0, H1, H2, H3 and H4) are shown in Figure 3. The water of consistency increased as the content H increased and also did the setting times (Initial and final). This is essentially attributed to the nature of H which is voracious for water and it is necessary to proceed pozzolanic reactions between H and the evolved  $\text{Ca}(\text{OH})_2$  from the hydration process [6, 7].

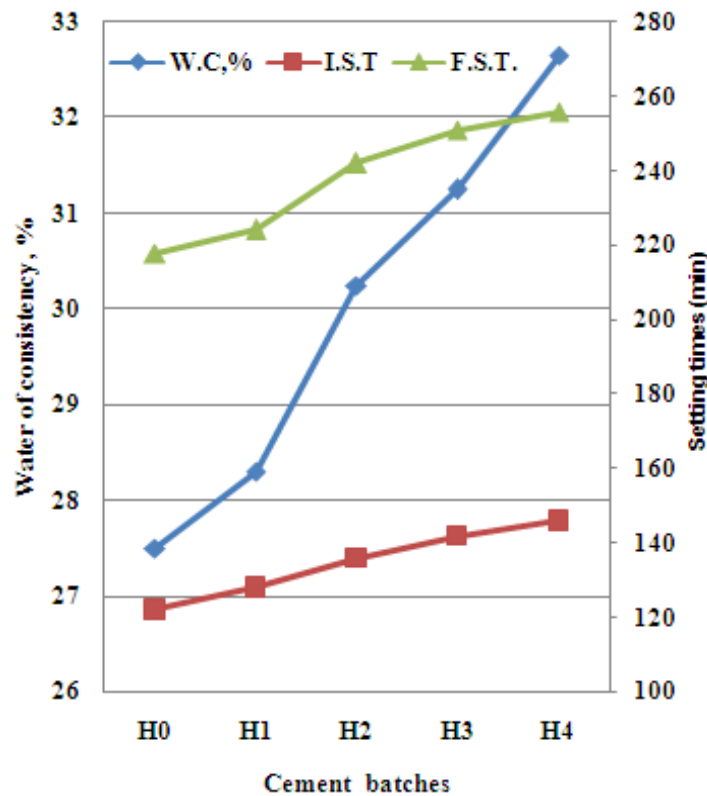


Figure 3. Water of consistency and setting times of the OPC/ Homra cement mixes.

### 3.2. Loss on Ignition Loss

Figure 4 illustrates the relation between the loss on ignition of the hardened cement pastes and temperature was investigated. It is clear that the loss on ignition increased as the temperature increased. This is mainly attributed to the occurrence of some chemical reactions in the usual phases of cement pastes that stimulated on firing as:- 1-The free water content found in the intermolecular spaces was removed nearly at 110°C. 2- A slight

decomposition of calcium silicate and/or aluminate hydrates (CSH and CAH) occurred at about 200-250°C. 3- The braking down of gehlenite hydrate phase ( $\text{C}_2\text{ASH}_8$ ) at about 300°C or little more. 4-The dehydration of calcium hydroxide,  $\text{Ca}(\text{OH})_2$  at 400-600°C also occurred. Addition of H increased the loss on ignition which is essentially contributed to the relatively high water consistency of the cement pastes containing H. As it is obvious, the loss on ignitions took place in three steps.

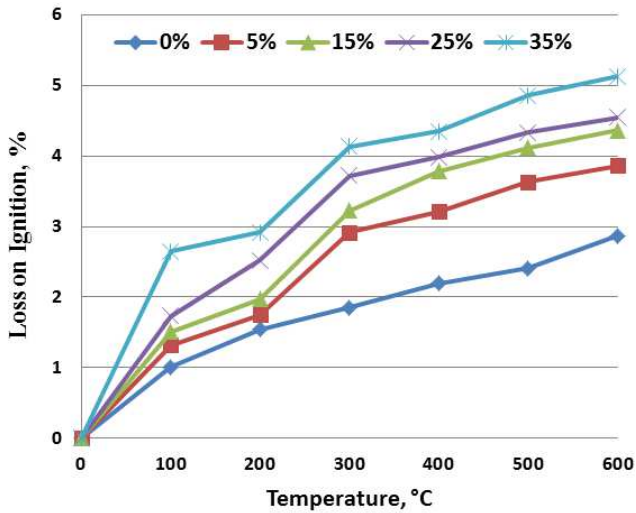


Figure 4. Loss on ignition of cement pastes as a function of temperature up to 600°C.

3.3. Bulk Density and Apparent Porosity

Figures 5 and 6 show the density and porosity of the hardened cement pastes as a function of temperature, respectively. The density decreased as the H content increased and also as the degree of temperature enhanced up to 400°C, and then increased if the temperature raised more than 400°C. On contrast, the porosity increased up to 400°C and then decreased. The decrease of bulk density and the increase of apparent porosity are mainly due to the primarily high w/c-ratios used to prepare the original and suitable cement pastes and also, to the relatively low density of H if compared to that of the OPC that may lead to an open pore structure in the cement pastes [3, 14]. On the other side, the increase of density and the decrease porosity is often due to the development and improving of the crystal nature and also may be due to the pozzolanic character of H in the cement pastes in the beginning of the hydration process which later initiated by thermal treatments [15-17].

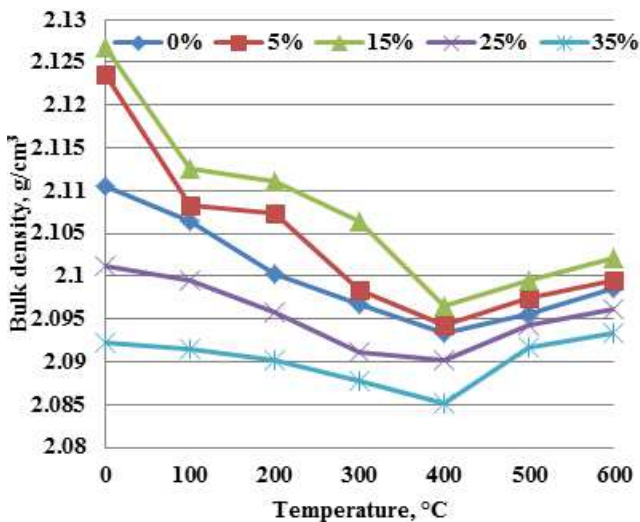


Figure 5. Bulk density of cement pastes as a function of temperature up to 600°C.

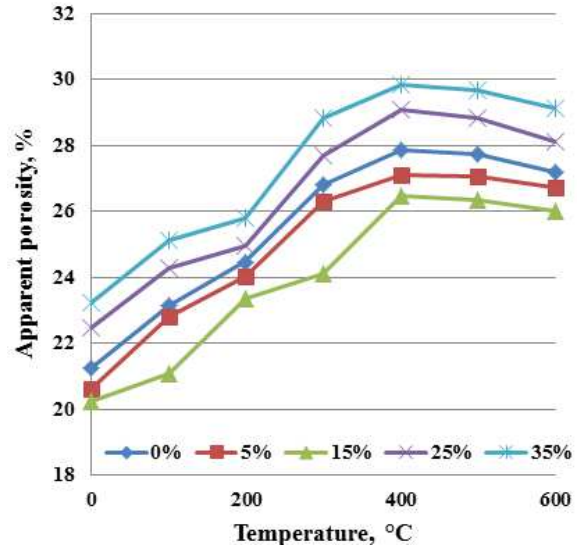


Figure 6. Apparent porosity of cement pastes as a function of temperature up to 600°C.

The changes in density as well as porosity due to the exposure to temperature from 0-600°C were happened in three steps. The density decreased from room temperature until 400°C which is due to the removal of free or crystallized water existed in the intermolecular spaces leading to create more open pore volume. As a result, the porosity increased. From 300°C till 400°C, the breaking down of the formed hydrates and/or minerals started. From 400°C to 600°C, it was accompanied by an increase in the density for all cement pastes and a decrease in the porosity. This is mainly attributed to the decomposition of Ca(OH)<sub>2</sub>. Moreover, the associated shrinkage due to the exposure to high firing temperature was leading to the formation of denser structures as well as higher density and lower porosity. Accordingly, the changes in the porosity are in a good conformity with those of density [15, 16].

3.4. Compressive Strength

Figure 7 demonstrates the compressive strength of the various hardened cement pastes as a function of temperature. The compressive strength of the OPC cement pastes gradually increased when from 100°C up to 500°C and then decreased, whereas that of the hardened cement pastes containing H increased only up to 400°C and then decreased. The compressive strength of cement pastes containing 5 and 15 wt.% H are higher than that of OPC. This is principally due to the pozzolanic reactions that occurred between H and the released Ca(OH)<sub>2</sub> that was coming during the hydration process to produce additional calcium silicate and/or aluminate hydrates (CSH & CAH) [12, 13, 17]. These hydrates often deposited inside the pore structure of the hardened cement pastes. The decrease of the compressive strength of cement pastes containing 25 and 35-wt.% H is mainly contributed to the deficiency of the OPC portion which is the main binding material. This means that the higher addition of H is undesirable [18, 19]. Moreover, the increase of the compressive strength of cement pastes when



heated up to 400 or 500°C may be due to that fact the thermal treatment strengthened the pastes and increases its hardness. The thermal treatment more than 400-500°C came with the opposite result, i.e. the compressive strength decreased. This is certainly contributed to the breakdown of the formed CSH and/or CAH, particularly with the decomposition of the combined water content which in turn was accompanied with internal cracks [17-19]. The cement pastes containing any pozzolanic materials are more sensitive if exposed to fire. Accordingly, the cement pastes made with 15-25 wt.% H

represent the most suitable fire resistant cements up to 600°C if compared with other types even that of the OPC pastes taking into consideration that the values of compressive strength of all studied cements are close or near to each others. In a general sense, it could be concluded that although the decrease of density and the increase of porosity up to 400 and 500°C, the compressive strength increased. On contrast, in spite of increasing the density and the decrease of porosity from 400 to 600°C, the compressive strength decreased.

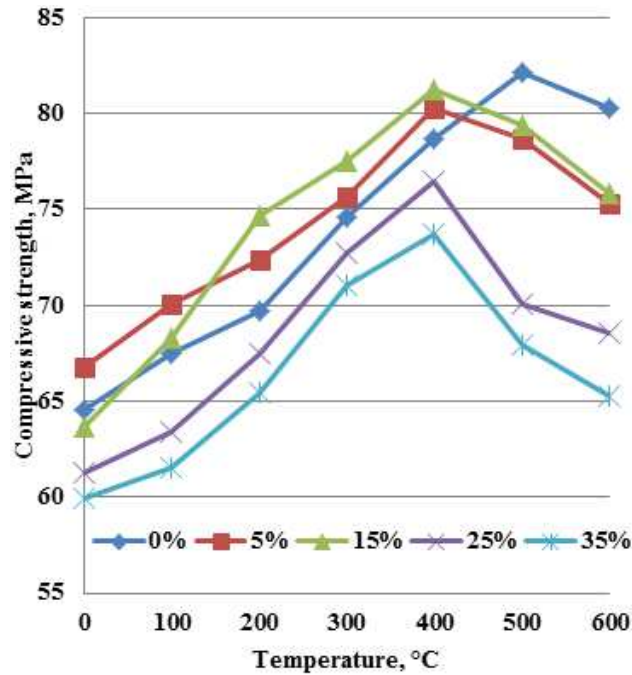


Figure 7. Compressive strength of cement pastes as a function of temperature up to 600°C.

### 3.5. Microstructure

The SEM images provide us with a useful knowledge on the morphology and crystals of the hydration products. The SEM images of the OPC cement pastes (H0) exposed to 400 and 600°C are shown in Figure 8, whereas those of H4 exposed to 400 and 600°C are seen in Figure 9. Figure 8A shows a microstructure of well-developed crystals of CSH

and/or CAH, while the  $\text{Ca}(\text{OH})_2$  are spreading in the matrix. At 600°C (Figure 8B), there are clearly some or few microcracks. This was leading to a decomposition of the bonds between particles to cause a decrease in density and an increase in the porosity which in turn causes of a sharp decrease in compressive strength.

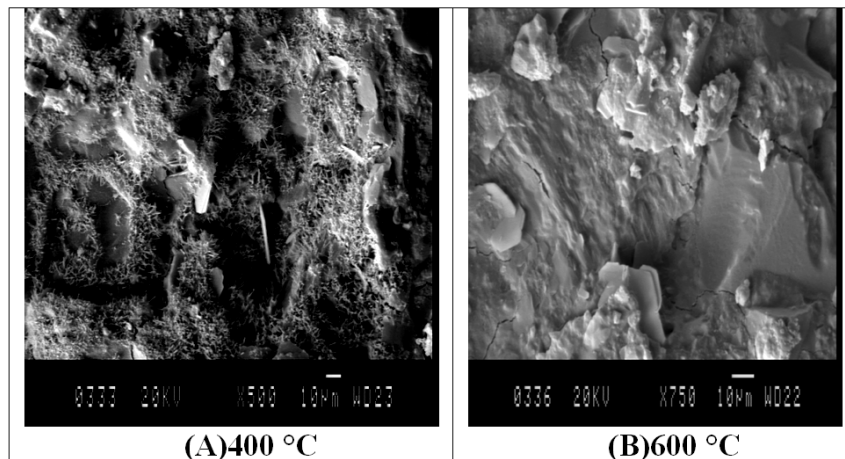


Figure 8. The SEM microscopy of the OPC cement pastes exposed to 400 and 600°C.

The CSH from the pozzolanic reactions between H and  $\text{Ca}(\text{OH})_2$  resulted in a consumption of a large quantity of lime (Figure 9). This was reflected positively on the density, porosity and compressive strength, i.e. the density increased and the porosity decreased. This was leading to increase the compressive strength when compared with those of the OPC

cement pastes. At 400°C (Figure 9A), the microstructure indicated the formation of dense masses of hydration products having micro- and narrow pores except that there are some cracks in the matrix which caused the decrease in the compressive strength.

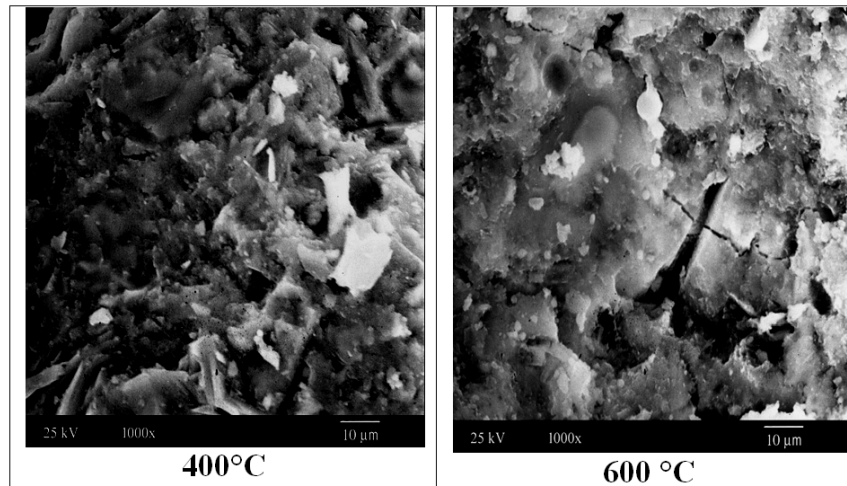


Figure 9. The SEM microscopy of the different cement pastes exposed to 300 and 600 °C for three hours.

## 4. Conclusions

- The loss on ignition of the various hardened cement pastes increased with temperature as well as with Homra content.
- The density decreased with temperature up to 400 or 500°C while the apparent porosity increased. More than 400 or 500°C, the density increased and the porosity decreased.
- The compressive strength of the OPC pastes gradually increased with temperature up to 500°C with those of the OPC pastes and then decreased while those incorporated H increased only up to 400°C and then decreased.
- The hardened cement pastes with 15% H recorded the highest compressive strength value at all firing temperatures.
- The SEM images showed that a microstructure of well-developed crystals of CSH and/or CAH, while the  $\text{Ca}(\text{OH})_2$  are spreading in the matrix at temperatures up to 400°C, while there are some cracks if heated up to 600°C.
- It could be concluded that with the addition of 15 wt.% H at most, there is no adverse effects. Therefore, the higher content of H is undesirable. The pastes containing H are much stable at temperature up to 400°C and the decay of  $\text{Ca}(\text{OH})_2$  started at more than that up to 600°C.
- The pure OPC cement pastes could withstand against thermal exposure up to 500°C while those incorporated H could withstand only up to 400°C.

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