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Beneficiation of the huge waste quantities of barley and rice husks as well as coal fly ashes as additives for Portland cement

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A B S T R A C T

This study aims at benefit from barely husk ash (BHA), rice husk ash (RHA), and coal fly ash (CFA) as a replacement of ordinary Portland cement (OPC) to eliminate the problems associated with cement industry involving energy consuming and air pollution. Three series of cement mixes were prepared from OPC with different contents (0–30 wt.%) of each additive. Different cementing and mechanical properties of the prepared mixes were tested according to the international standard specifications. It was concluded that OPC blended with 15–20 wt.% of BHA, RHA, or CFA show outstanding cementing properties.

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1. Introduction

The current cement production rate of the world, which is approximately 1.2 billion tons/year, is expected to grow exponentially to about 3.5 billion tons/year by 2015. Most of the increase in cement demand will be met by the use of supplementary cementing materials, as each ton of Portland cement clinker production is associated with a similar amount of CO_2 emission. Nowadays, pozzolanic materials are widely used as supplementary cementing material in Portland cements and may replace part of the clinker in order to enhance the performance of the hydrated cement. Such composite or blended cements are employed for their economic, ecological, and technological benefits i.e., reduction of energy consumption as well as CO_2 emission. Supplementary cementing materials reduce lime content in hydrated Portland cements and replace it with pore-filling cement hydrates, which are known to improve the ultimate strength, impermeability, and durability to chemical attack of cement. Different types of additions are used such as pozzolanic (natural pozzolan, low calcium fly ash, and silica fume), auto pozzolanic (high calcium fly ash and blast furnace slag), and crystalline (generally known as filler). Pozzolanic activity or hydraulicity of pozzolanic material is

mainly associated with their vitreous and/or amorphous structure [1,2]. Among of theses additive is the ash resulted from the composition of agricultural byproducts such as rice and barley husks. Rice and Barely husks are recognized as two of the most economic and important cereals in the world. By area and production, rice and barley is the second and fourth of most important cultivated crop. On average 20% of the rice and barley paddy are husks, the majority of these husks are either burnt or dumped as waste [3–5]. Unlike other residues, rice and barley husks also contain large proportions of ash and silica, which to a large extent have been reported to affect the property and application of husks [6,7]. By burning husks under a controlled temperature and atmosphere, a highly reactive ash is obtained [8,9]. Ash containing little or no residual carbon has many potential uses ranging from soil amendment to abrasive component of tooth paste [10,11]. The most important property of husk ashes that determines pozzolanic activity is the amorphous phase content. RHA and BHA are highly reactive pozzolanic material suitable for use in lime-pozzolana mixes and for Portland cement replacement. RHA and BHA contain high amounts of silicon dioxide, and their reactivity related to lime depends on a combination of two factors, namely the non-crystalline silica content and their specific surface [12]. The ash being very light is easily carried by wind and water in its dry state [13]. Prior to 1970, husk ash was usually produced by uncontrolled combustion, [14] and the ash so produced was generally crystalline and had poor pozzolanic properties. In 1973, Mehta studies have shown that burning rice husks at 600 °C

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produces an ash with an optimum composition for pozzolanic materials [15]. Several researchers have studied the durability variation of the concrete with pozzolanic materials (after complete combustion) [16–18]. Among the family of other agro-wastes, rice husk is a popular boiler fuel, and the ash generated usually creates disposal problems. The chemical process discussed not only provides a solution for waste disposal but also recovers a valuable silica product, together with certain useful associate recoveries [19]. In this century, the utilization of rice husk ash (RHA) as cement replacement is a new trend in concrete technology. Besides, as far as the sustainability is concerned, it will also help to solve problems otherwise encountered in disposing of the wastes. Disposal of the husks is a big problem, and open heap burning is not acceptable on environmental grounds, and so the majority of husk is currently going into landfill. The disposal of rice husks create environmental problem that leads to the idea of substituting RHA for silica in cement manufacturing [20]. Numerous investigations on the use of RHA in concrete production have been done and all produced positive results [21,22]. The main advantage of using RHA as a mineral admixture in concrete is the significant reduction in the permeability of the concrete [23–25]. The use of rice husk ash in concrete was patent in the year 1924 [26]. Up to 1972, all the researches were concentrated to utilize ash derived from uncontrolled combustion [27]. Controlled combustion influences the surface area of RHA, so that time, temperature and environment were to be considered to produce ash of maximum reactivity [28]. Fly ash material is composed primarily of complex alumino-silicate glass, mullite, hematite, magnetite spinel, and quartz. The proportion of quartz (crystalline silica) in the fly ash varies depending on the quartz content of the coal. Class C fly ash may have 1–7% free CaO and calcium sulfate as well as calcium alumino-silicate glass [29]. Fly ash is useful in many applications because it is a pozzolan, meaning it is a siliceous or alumino-siliceous material that, when in a finely divided form and in the presence of water, will combine with calcium hydroxide (from lime, Portland cement, or kiln dust) to form cementations compounds [30] and is found to have numerous advantages for use in the concrete industry. Some of the advantages include improved workability, reduced permeability, increased ultimate strength, reduced bleeding, better surface finishing, and reduced heat of hydration [31]. Mineral admixtures or pozzolans are used to improve strength, durability, and workability in concrete [32–35]. Freshly mixed concretes are generally more workable when a portion of the cementitious material is fly ash, in part because of the spherical shape of fly ash particles. Smoother mixtures are typically produced if the mineral admixture is substituted for sand rather than cement, but highly reactive or cementitious pozzolans can cause loss of workability through early hydration [36,37]. Very finely divided mineral admixtures, such as silica fume, can have a very strong negative effect on water demand and hence, workability, unless high-range water-reducing admixtures are used [38,39]. This work aims at benefit from the huge wasted quantities of barley, rice husks, and coal fly ashes as additives for Portland cement and study their effect on different physico-mechanical, chemical, and cementing properties of its hardened pastes.

2. Materials and experimental

2.1. Materials

The starting materials used in this investigation are:

1. Ordinary Portland cement (OPC) Type I.
2. Barley husk ash (BHA).
3. Rice husk ash (RHA)

4. Coal fly ash (CFA)
5. Some chemicals namely; methyl alcohol and diethylether were used for stopping the hydration of the cement pastes at different curing times of cement hydration 3, 7, 28, and 90 days.

2.2. Experimental

2.2.1. Processing of rice and barley husk ashes

Rice and barley husks were processed and washed separately by passing running water on them for a few times and then washed with distilled water, drying in a sunny place, burning at 600 °C for 8 h and screening through 0.125 mm standard sieve. Coal fly ash (CFA) was also screened through 0.125 mm standard sieve.

2.2.2. Preparation of cement pastes

Different cement mixes were prepared from OPC with different wt.% of BHA, RHA, or CFA ash as given in Table 1 using the optimum amount of water. After 2 min of well dry mixing for 10 min in an electrical cement mixer, the mixes were separately gauged with the suitable amount of water with a constant hand mixing using stainless steel spatula. During 4 min from the beginning of water addition, the pastes were molded into $1 \times 1 \times 1$ inch cubic samples in steel mold. The molded pastes were left in a 100% relative humidity cupboard. After 1 day, the hardened samples were de-molded obtaining cubic samples of the hardened cement pastes which were kept under water until time of investigation. Three representative samples from each cement mix were tested for their bulk density and apparent porosity at each curing time; 3, 7, 28, and 90 days. At each curing time, the hydration of the tested samples was stopped by grinding with 1:1 methanol:ether mixture. The chemically combined water was determined for the hydrated sample after stopping its hydration through ignition of 1 g of the hydrated cement sample at 800 °C using the following equation:

$$\% \text{ of chemically combined water} = \frac{\text{Wt. before ignition} - \text{Wt. after ignition} \times 100}{\text{Wt. before ignition}}$$

2.2.3. Instrumentation

Chemical compositions of OPC, BHA, RHA, and CFA were investigated using X-ray fluorescence (XRF) technique. The mineralogical compositions of the hydrated cement mixes were investigated through X-ray diffraction (XRD) technique, A Phillips PW 1710 diffractometer with Ni filtered Cu Kα radiation operating at 30 mA and 40 kV was used. Thermal analysis, thermo gravimetry (TG) and differential thermo gravimetry (DTG) as well as differential scanning colorimetry (DSC), of some hydrated samples was also investigated using NETZSCH STA 409 analyzer, with Al_2O_3 as a reference material over a range of temperature from room temperature up to ≈1000 °C.

Table 1
The prepared mixes from OPC with either BHA, RHA, or CFA.

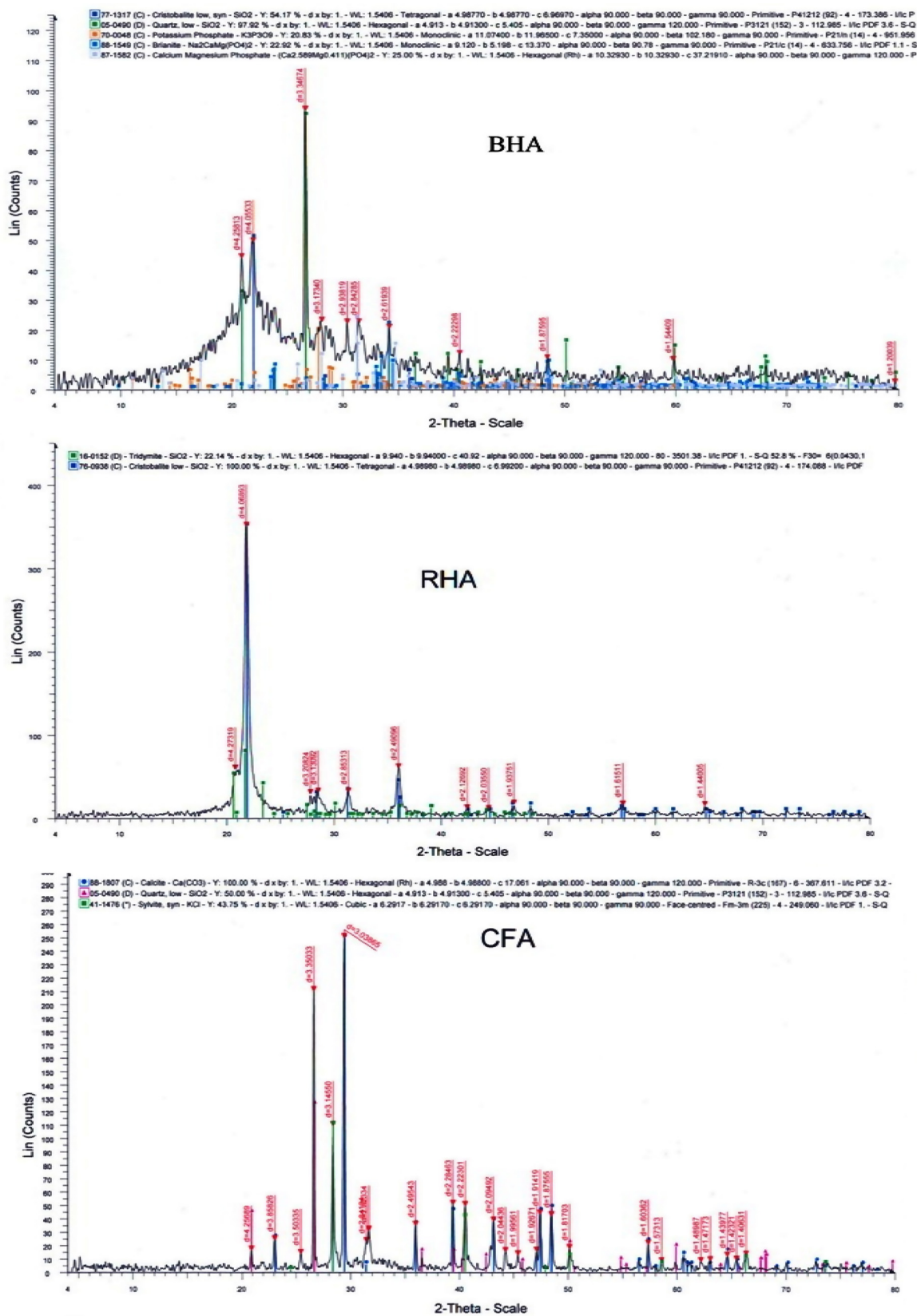| Mix no.	OPC (wt.%)	BHA, RHA, or CFA ash (wt.%)
1	100	–
2	95	5
3	90	10
4	85	15
5	80	20
6	75	25
7	70	30

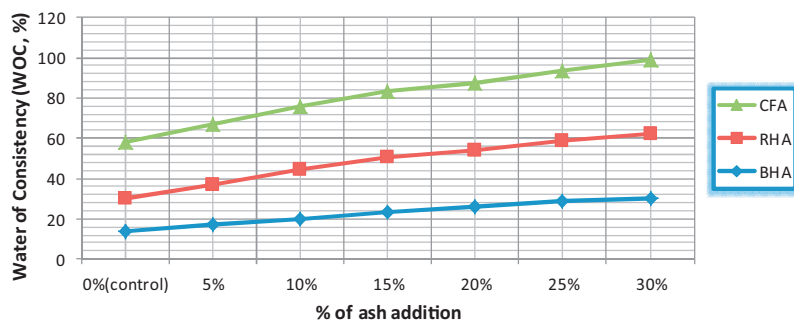**Fig. 1.** XRD patterns of BHA, RHA, and CFA.

Fig. 2. Water of consistency (WOC) of different cement mixes.

2.2.4. Characterization of the prepared cements

(a) Water of consistency and setting time was determined using Vicat apparatus [40,41].
(b) Bulk density (B.D) and apparent porosity (A.P) were measured using kerosene displacement method [42].
(c) Mechanical properties cold crushing strength (c.c.s) was determined using a compressive strength testing machine (ELE International compression tester 1881 A0030) [43].

3. Results and discussion

3.1. Chemical composition of the starting materials

The XRF analysis of the starting materials indicates that the cement is composed mainly of CaO (64.62%) and SiO_2 (20.63%) with considerable contents of Fe_2O_3 (6.88%) in addition to little contents of other constituents e.g., Al_2O_3 (1.54%), MgO (1.62%), SO_3 (1.21%), $Na_2O + K_2O$ (0.27%), free lime (1.09%), and ignition loss (3.01%). According to the American classification, the cement used in this research work is ordinary Portland cement, Type I [44]. Three investigated ash samples BHA, RHA, and FCA composed mainly of silica (SiO_2); 79.298, 64.948, and 60.402%, respectively. They possess variable contents of alumina (Al_2O_3); 8.775, 11.830, and 11.00% in addition to little contents of Fe_2O_3; 0.415, 0.785, and 0.730%. Some other constituents are also present in variable amounts e.g., Na_2O, MgO, K_2O, P_2O_5, SO_3, K_2O, CaO, MnO, CuO, ZnO, SrO, ZrO_2, and (Cl^-). The ignition loss in each one of the three samples is less than 1%. According to ASTM (1978), if the sum of SiO_2, Al_2O_3, and Fe_2O_3 is more than 70% then the material would be considered as a pozzolanic material [45]. In our samples the sum of SiO_2, Al_2O_3, and Fe_2O_3 is more than 70%. The sum of these oxides in BHA is 88.488%, in RHA is 77.263% while in CFA is 72.132%. So according to the ASTM (1978), the three ash samples are pozzolanic in nature, and the pozzolanity order is BHA > R-RHA > CFA. So, they could be used successfully as a partial replacement for ordinary Portland cement (OPC) to obtain blended cement.

3.2. Mineralogical composition of the ashes samples (BHA, RHA, CFA)

Fig. 1 shows the X-ray diffraction (XRD) patterns of the used BHA, RHA, and CFA powders. Based on the XRD results, the three investigated samples are crystalline silica with different crystalline forms. The data show that BHA, RHA, and CFA composed mainly of quartz in addition to cristoblite or tridymite mineral. Some intense peaks characterizing calcite and KCl minerals could also be detected. The calcite mineral is a result of carbonation of CaO present in CFA as indicated from its chemical analysis. The presence of some peaks characterizing KCl is an indication on its presence in a considerable content as impurities in CFA.

3.3. Cementing properties of the prepared cement pastes

3.3.1. Water requirement (water of consistency)
The percentages of standard consistency as a function of BHA, RHA or CFA content as a replacement of cement are represented in Fig. 2 It is shown that the water demand for standard consistency linearly increases with an increment of cement replacement level by BHA, RHA, or CFA, this is due to the relatively higher specific surface area of the ashes (11,000–12,000 cm^2/g) than the OPC (3100 cm^2/g) in addition to the hygroscopic nature of the ashes which consume relatively higher amounts of mixing water, these observations are limited to that observed with other several researchers [46,47].

3.3.2. Setting time
Figs. 3–5 show that the initial setting time reaches 45 min in the control sample OPC (0% ash additives). It increases gradually as the ash content increases reaching 5 h: 20 min, 5 h: 23 min, and
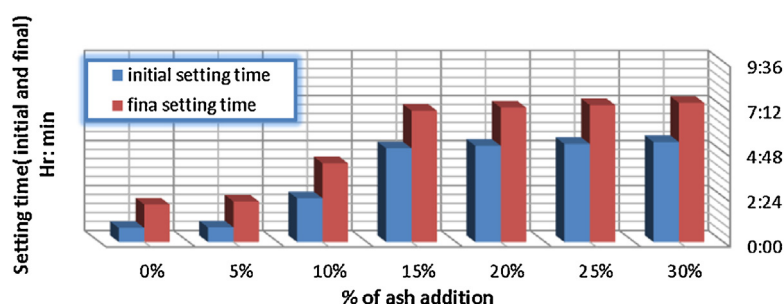

Fig. 3. Initial and final setting time of cement pastes containing barley husk ash.
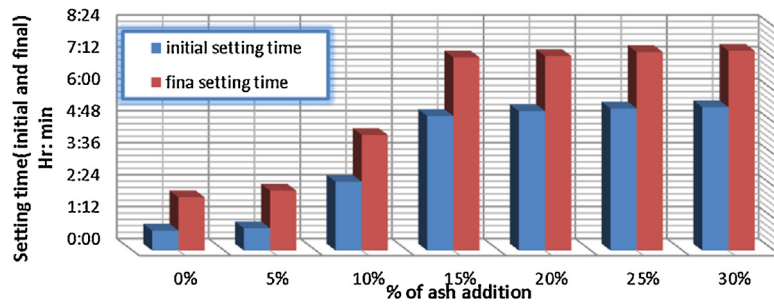
Fig. 4. Initial and final setting time of cement pastes containing rice husk ash.
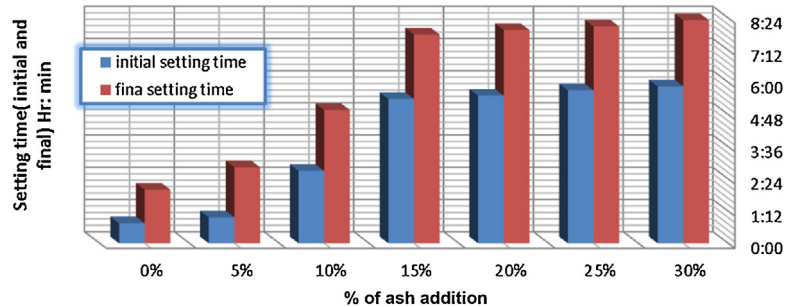

Fig. 5. Initial and final setting time of cement pastes containing coal fly ash.

5 h: 51 min at 30% of BHA, RHA, and CFA addition, respectively. The final setting time of the control OPC paste is reached after 2 h: 00 min which is also prolonged with the increment of ash content reaching 7 h: 25 min, 7 h: 30 min, and 8 h:19 min at 30% addition of BHA, RHA, and CFA, respectively. These values are well within the permissible limits as per ASTM C191-04 and also in a good agreement with other several observations [48,49].

3.3.3. Chemically combined water

Figs. 6–8 show a gradual increase in the content of combined water as the dose of the ash increases, reaching its maximum value with 15 wt.% addition of either BHA, RHA, or CFA. On increasing the ash dose to 20% and up to 30% a noticeable gradual decrease in the chemically combined water content is observed. A noticeable feature is the relatively higher combined water content with BHA addition than RHA which in turn is higher than CFA. On the other hand, in the control sample (0%) at a fixed dose of ash, a noticeable increase in the water content is observed as the curing time (age of hydration) increases from 3 to 7 to 28 days beyond which (at 90 days of hydration) the increase in water content is less pronounced which means that after 28 days, the hydration reactions are mostly completed. However, the hydration of cement is not 100%
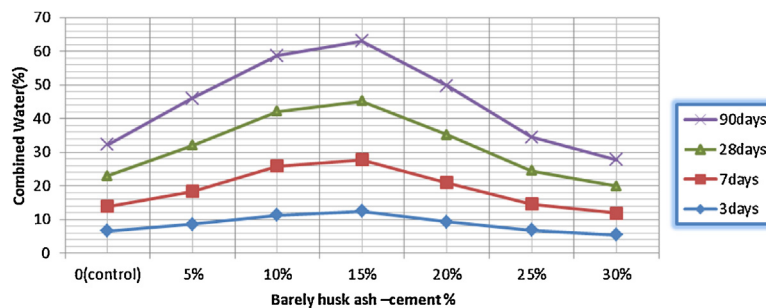

Fig. 6. Chemically combined water of the hardened cement pastes containing different doses of BHA.
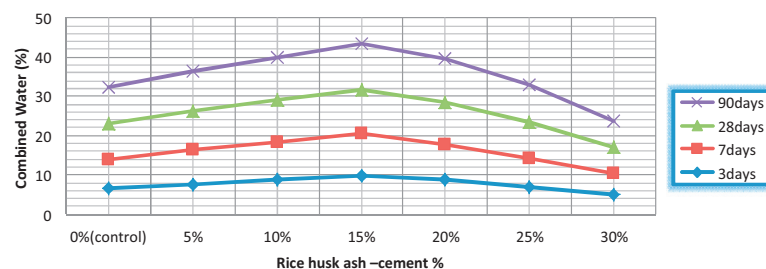

Fig. 7. Chemically combined water of the hardened cement pastes containing different doses of RHA.
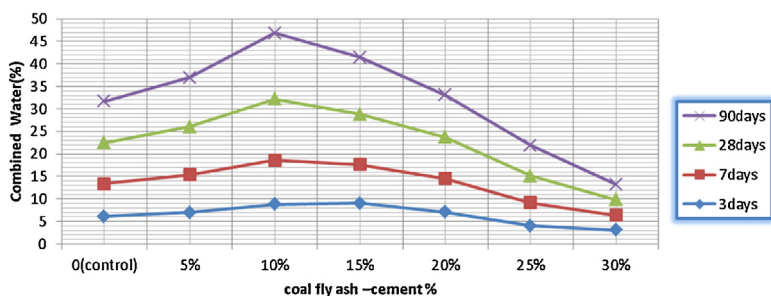
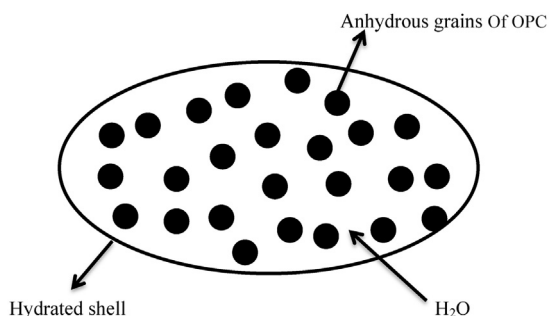Fig. 8. Chemically combined water of the hardened cement pastes containing different doses of CFA.

Fig. 9. Encapsulation of the hardened cement.

completed even after many years due to the encapsulation phenomena in which the hydration products encapsulate the anhydrous grains of the cement slowing water to reach them delaying further hydration [50], as proposed by Fig. 9.

3.3.4. Bulk density and apparent porosity

Figs. 10–12 show the data of bulk density values of the hardened cement pastes with different ash contents while Figs. 13–15 show their apparent porosity percent. The results show that the bulk density is linearly decreased while the apparent porosity is linearly increased as the content of BHA, RHA, or CFA increases; this behavior is correlated with the relatively lower

density of BHA, RHA, or CFA compared with OPC [51]. Regarding the hardened cement pastes containing ashes, the bulk density increases in the following order, cement containing BHA > R-RHA > CFA while the porosity behaves oppositely i.e., in the order BHA < RHA < CFA.

On the other hand either in the control sample (0% ash) or at any fixed content of BHA, RHA, or CFA, a gradual increase in the bulk density values corresponded with a gradual decrease in apparent porosity percent is observed as the curing time (age of hydration) increases from 3 to 7 to 28 days, beyond which i.e., at 90 days of hydration the change in the density or porosity is less pronounced, these results are in accordance with the chemically combined water contents from the point of age of hydration.

3.3.5. Compressive strength of the hardened cement pastes

The results shown in Fig. 16 indicate a noticeable improvement in the compressive strength of the hardened cement as either BHA, RHA, or CFA content increases up to 15 wt.% at which the compressive strength reaches its maximum value, beyond which i.e., starting from 20 wt.% and up to 30 wt.%, a noticeable decrease in the compressive strength value is observed. However, with 20 wt.% addition of BHA, RHA, or CFA, the decrease in compressive strength value is less pronounced and the hardened cement pastes still maintain a value of compressive strength comparable with the control sample (0% ash addition). These results indicate that 15–20 wt.% of BHA, RHA, or CFA are considered as an optimum
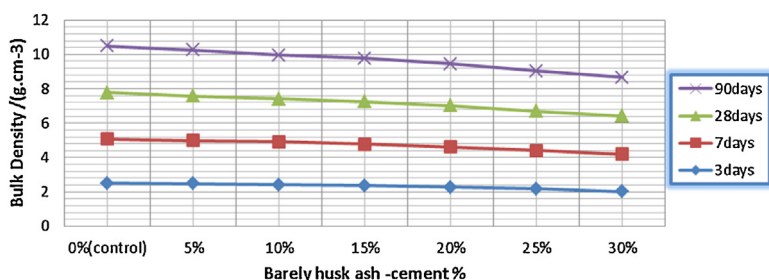

Fig. 10. Bulk density of the hardened cement pastes containing different contents of BHA at different curing times.
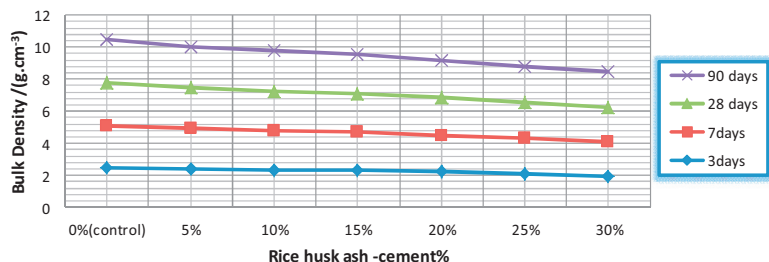

Fig. 11. Bulk density of the hardened cement pastes containing different contents of RHA at different curing times.
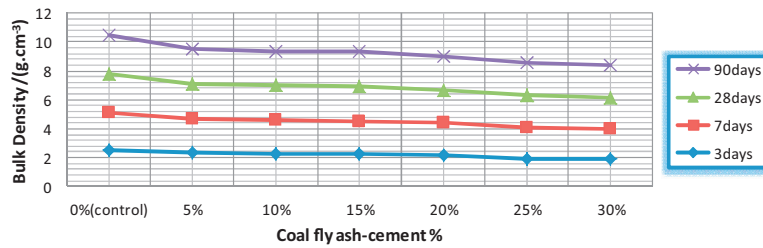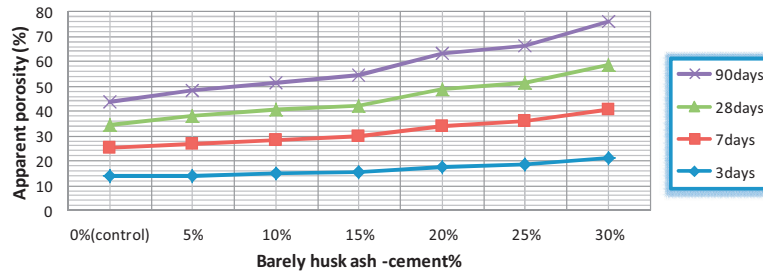
Fig. 12. Bulk density of the hardened cement pastes containing different contents of CFA at different curing times.

Fig. 13. Apparent porosity of the hardened cement pastes containing different contents of BHA at different curing times.
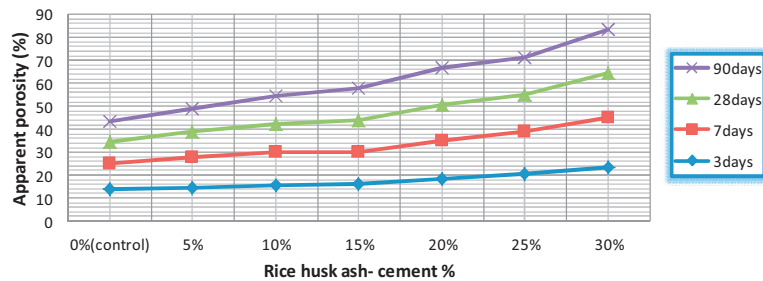

Fig. 14. Apparent porosity of the hardened cement pastes containing different contents of RHA at different curing times.

addition for OPC with an improvement in compressive strength or at least keeping it within the range of the control sample. The results also show that samples with BHA exhibit the highest compressive strength values followed by those containing RHA which in turn are relatively higher than those containing CFA. Also, at a constant addition of ash a noticeable improvement in compressive strength is observed as the curing time (age of hydration) increases from 3 to7 to 28 days, beyond which (at 90 days) the improvement in strength is less significant. The results of the compressive strength are in a well accordance with those of chemically combined water. The improvement in compressive strength with the addition of BHA, RHA, or CFA is due to the pozzolonic nature of these additives. When these pozzolanic additives are added to the cement they react with lime released by the hydration of the cement, to create compounds which improve the strength and other properties of the hardened cement pastes [52,53]. Some authors attributed the improvement in compressive strength to filler effect in addition to the higher pozzolanity of the ashes [54,55]. Zhang et al. [56] suggested that $Ca(OH)_2$ and calcium silicate hydrates (C-S-H) are the main hydration product in the ash containing pastes, Qijun et al. [57] support the findings concluded that the reaction between ash and $Ca(OH)_2$ is responsible for the improvement of cement properties. Cisse and Laquerbe [58] strengthen the fact that pozzolanic activity of ashes was liable for the increasing of strength of the hardened paste, this explains the relatively better improvement of the cement containing ashes on the following order, cements with BHA > RHA > CFA which is the same order of the pozzolanic nature. Rukzon et al. [59] attributed
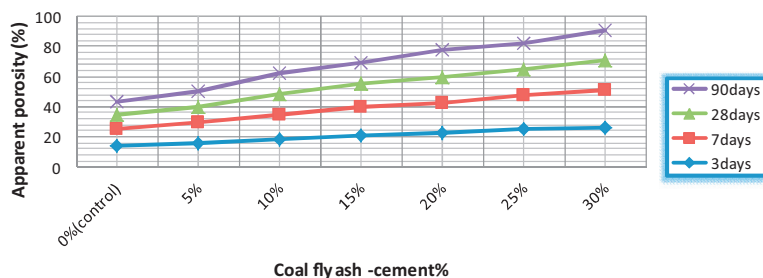

Fig. 15. Apparent porosity of the hardened cement pastes containing different contents of CFA at different curing times.
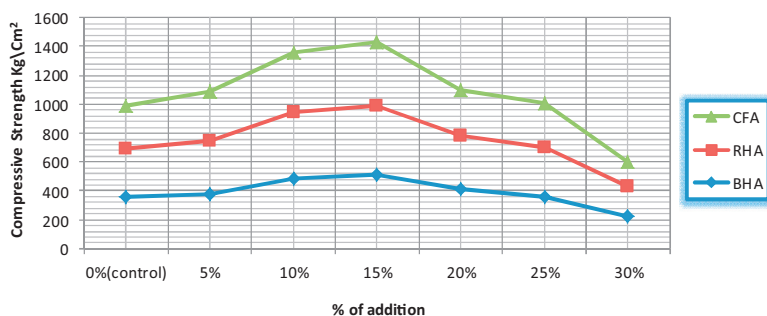
Fig. 16. 90 day's compressive strength of the hardened modified cement pastes compared with the non-modified one.

the improvement in strength to the finer particles of the ashes than OPC which causes the segmentation of large pores and increases nucleation sites for the precipitation of pozzolanic reaction products in cement paste, this increases the pozzolanic reaction and refines the pore structure of the paste, the increase in hydration leads to reduction of $Ca(OH)_2$ in paste, the incorporation of pozzolan such as BHA, RHA, and CFA reduces the average size and results in a permeable paste. Farah Alwani et al. [60] attributed the improvement in strength of the hardened cement containing ash to the pozzolanic effect or pore size refinement that takes place when an addition amount of calcium silicate hydrates form in the hydrated cement. This occurs due to BHA, RHA, and CFA which are known as highly siliceous materials reacting with $Ca(OH)_2$ in the present of moisture or water. At early ages of hydration, slow pozzolanic reaction occurs which increases as the age of hydration increases. The decrease in compressive strength on increasing the ash content to 20 wt.% and up maybe certainly due to the complete consumption of the $Ca(OH)_2$ resulting from the hydration of OPC, in the pozzolanic reaction with BHA, RHA, or CFA additives.

Accordingly, the excess contents of ash additives (20 wt.% or more) are not involved in a pozzolanic reaction as a result of the marked decrease of OPC content and hence, the decrease of $Ca(OH)_2$ resulting from its hydration. The relatively lower compressive strength value of CFA compared with BHA or RHA is due to its relatively lower pozzolanic nature (as seen from its chemical analysis (XRF)) as well as its relatively higher content of

$CaCO_3$ and KCl impurities (as seen from its phase composition (XRD), Fig. 1) with no hydraulic properties.

3.4. Phase composition of the hydrated cement pastes

Fig. 17 shows the phase composition of the hydrated control sample of OPC (0% ash) compared with the anhydrous OPC sample. The figure shows that the control sample (OPC) composed mainly of hatrunite mineral (Ca_3SiO_5) with a considerable contents of larnite mineral $Ca_2(SiO_4)$ in addition to iron aluminum calcium oxide and olivine mineral (Ca_2SiO_4). In the hydrated sample, some new sharp lines characterizing portlandite mineral ($Ca(OH)_2$) could be detected with a marked decrease in the intensities of the hatrunite mineral (Ca_3SiO_5) which confirms its reaction with water (hydration) producing portlndite mineral ($Ca(OH)_2$). Fig. 18 shows the phase composition of the hardened cement pastes containing 15 wt.% of BHA, RHA, or CFA. In the three samples the lines characterizing the portlandite mineral ($Ca(OH)_2$), the hydration product of OPC, predominate together with lines characterizing hatrunite mineral (Ca_3SiO_5) and sometimes larnite mineral (Ca_2SiO_4) which are the constituents of the anhydrous OPC.

Comparing with the control OPC sample, Fig. 17, the intensity of the peaks characterizing the anhydrous minerals (hatrunite and larnite) has been clearly decreased. Also, the peaks characterizing quartz, cristobalite, or tridymite (the main constituents of BHA, RHA, or CFA) have been disappeared or decreased which confirm their consuming in the pozzolanic reaction, this is also approved by
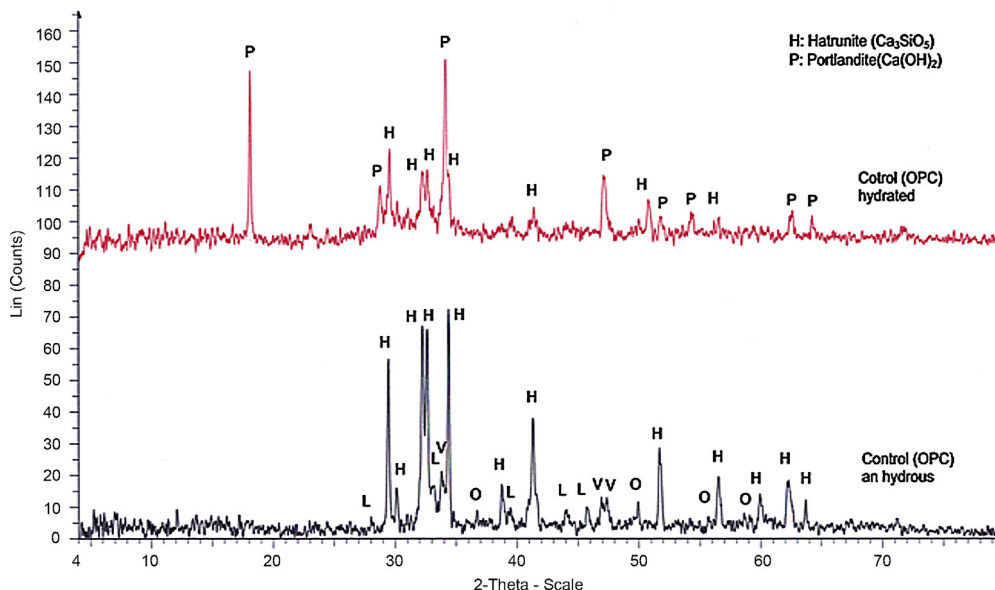

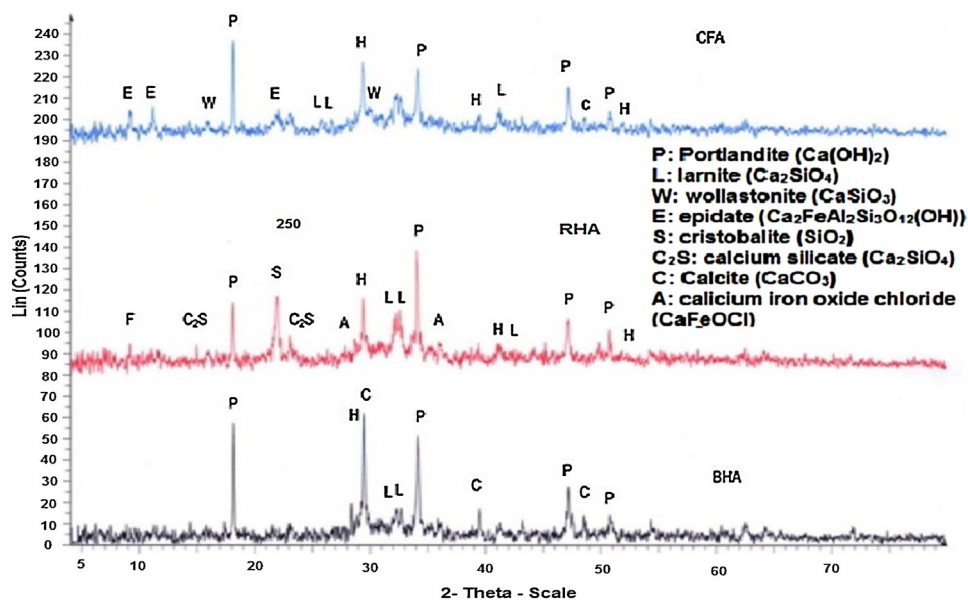**Fig. 17.** XRD patterns of anhydrous and hydrated control OPC samples.

Fig. 18. XRD patterns of hydrated cement samples containg 20 wt.% of BHA, RHA, or CFA.

the decrease in the intensity of the portlandite $(Ca(OH)_2)$ lines in the cement containing BHA, RHA, or CFA (Fig. 18), compared with their intensities in the hydrated control sample (Fig. 17). The relatively lower intensity of the lines characterizing portlandite mineral in samples containing BHA than those containing RHA which in turn are relatively lower than those containing CFA, is a good evidence that the pozzolanic reaction (between portlandite and pozzolanic ash) was greater in case of cement containing BHA than one containing RHA and this in turn was greater than one containing CFA, this explains and confirms the improved properties of cement containing BHA, RHA, and CFA in the following order; BHA > RHA > CFA. In addition some lines characterizing calcite mineral $(CaCO_3)$ could be detected in the cement samples containing BHA resulting from the carbonation of a part of the portlandite mineral $(Ca(OH)_2)$. Also, this sample shows few lines characterizing wollastonite mineral $(CaSiO_3)$ and epidate mineral $(Ca_2FeAl_2Si_3O_{12}(OH))$, which enhance the strength of such cement sample. In cement sample containing RHA, some lines with relatively lower intensity characterizing calcium silicate (Ca_2SiO_4), cristobalite (SiO_2), and calium iron oxide chloride $(CaFeO_2Cl)$ could also be detected. In cement sample containing CFA, in addition to the main constituents; portlandite and larnite, some lines

characterizing calcite could also be detected which were present in the XRD patterns of CFA sample (Fig. 1).

3.5. Thermal analysis of the hydrated cement pastes

The results of TG, DTG, and DSC of the hydrated cement pastes containing BHA, RHA, and CFA, Figs. 19–21 respectively, show three steps of weight loss on TG, corresponded with three endothermic peaks on DTG and DSC. At temperature up to 150 °C, an evaporable water loss within the pore structure occurs in every cement paste. This effect is less pronounced with RHA or CFA, these peaks will eliminate if the samples are oven dried at 105 °C before the heating process. When the temperature varies at the range of 105–1000 °C the chemically combined hydrated water from the gel structure is then lost.

Dehydrolaxation of calcium hydroxide appears in the range of temperature between 400 and 450 °C. The weight loss in this stage is relatively lower, and the DTG and DSC peaks are relatively less pronounced in the sample containing BHA (Fig. 19) but increase in the sample containing RHA (Fig. 20) and increase more and more in the sample containing CFA (Fig. 21). This reflect the relatively lower content of portlandite mineral $(Ca(OH)_2)$ in the cement

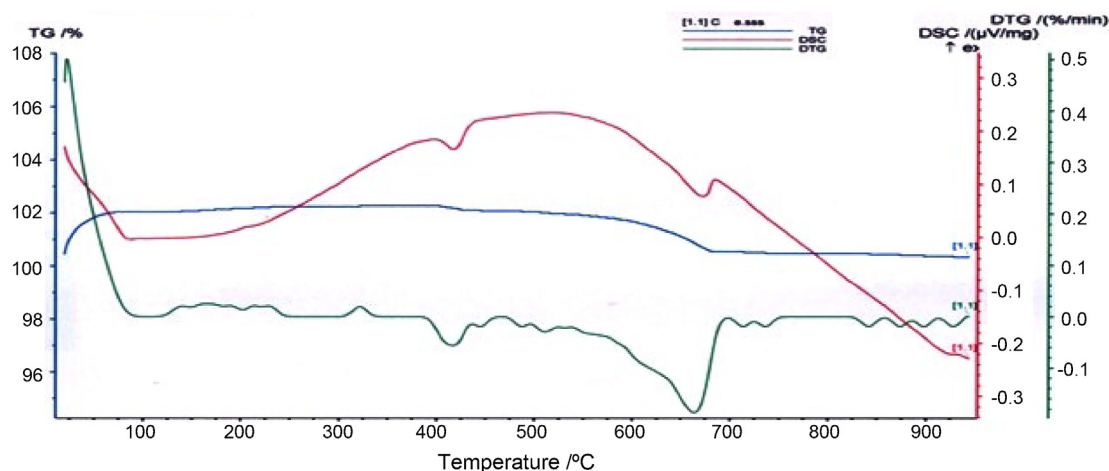**Fig. 19.** TG, DTG, and DSC of hydrated cement containing 20 wt.% of BHA.

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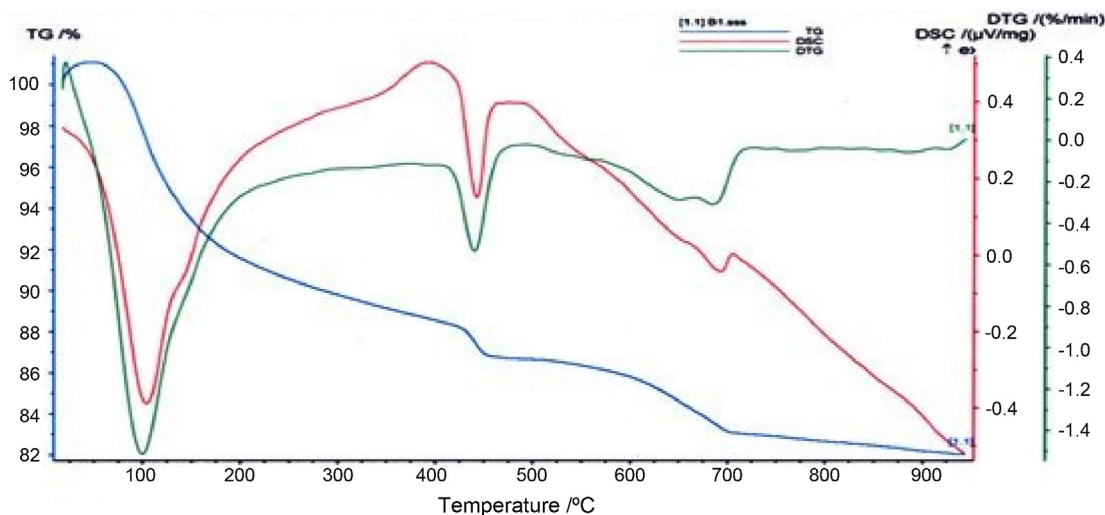

Fig. 20. TG, DTG, and DSC of hydrated cement containing 20 wt.% of RHA.
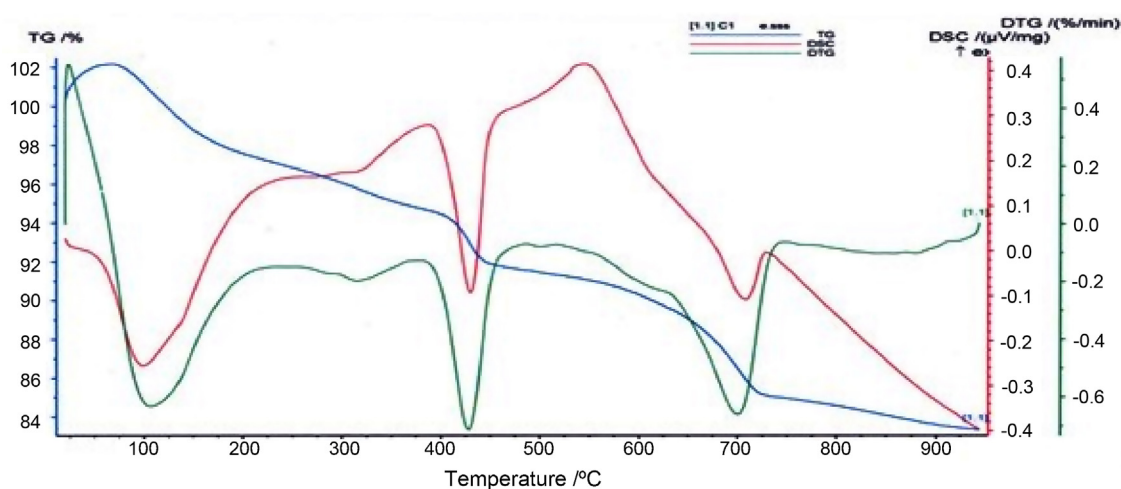

Fig. 21. TG, DTG, and DSC of hydrated cement containing 20 wt.% of CFA.

containing BHA due to its consuming in the pozzolanic reaction, these result explain the better cementing properties in cement samples containing BHA than those containing RHA. Also the relatively lower weight loss (TG) and less pronounced peaks (DTG and DSC) of cement sample containing RHA than those containing CFA explain the relatively better cementing properties of the first than those of the second. The third effect at the temperature range 650–700 °C is due to the decomposition of calcite mineral ($CaCO_3$) to produce CO_2 and CaO. The effect is more pronounced in cement samples containing RHA and CFA than one containing BHA, due to their relatively higher contents of calcite that proved before in XRD results. Therefore, attention has to be paid in order to prevent carbonation of portlandite mineral ($Ca(OH)_2$). Based on these evaluations the thermal analyses agree qualitatively with XRD results on the consuming of portlandite mineral ($Ca(OH)_2$) in the pozzolanic reaction in the order; cement containing BHA > RHA> CFA supporting the improvement in the compressive strength results in the same order.

4. Conclusion

A partial replacement (15–20 wt.%) of BHA, RHA, and CFA to OPC results in improved cementing properties. The improvement takes the following order BHA > RHA > CFA which correlated with their pozzolonic nature.

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