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## ORIGINAL ARTICLE

# Hydration behavior and mechanical properties of blended cement containing various amounts of rice husk ash in presence of metakaolin



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## KEYWORDS

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**Abstract** This paper reports an experimental study carried out to investigate the effects of metakaolin and rice husk ash on the hydration behavior and mechanical properties of blended cement. The results showed that the combination of metakaolin and rice husk ash provides a positive effect on mechanical properties. The samples incorporating the ternary blends of cement with 20–15% metakaolin and 5–10% rice husk ash showed better compressive strength than that of the normal sample without rice husk ash. These blends proved to be the optimum combination for achieving maximum effect.

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## 1. Introduction

In recent years, many researchers have established that the use of pozzolanic materials like blast furnace slag, silica fume, metakaolin (MK), fly ash (FA) and rice husk ash (RHA) etc. can, not only improve the various properties of concrete, but also can contribute to economy in construction costs (Amrutha et al., 2009). The MK is a valuable pozzolanic and thermally activated aluminosilicate material obtained by

calcining kaolin clay within the temperature range of 700–850 °C. MK is usually added to concrete in amount of 5–15% by weight of cement. Addition of MK causes an increase in mechanical strength, enhancement of long term strengths, decrease of permeability, porosity, reduction of efflorescence, increase of resistance to soluble chemicals like sulfates, chlorides and acids (Ambroise et al., 1994; Kostuch et al., 1993; Sabir et al., 1996). The addition of MK decreases workability of fresh concrete mix. This disadvantage can be reduced by superplasticizers (SP) or increasing water to binder (W/B) ratio. However, rheological properties of fresh concrete mix depend on the type of (SP) Okan et al., 2012. The replacement with 30% of metakaolin leads to a substantial improvement in strength and transport properties of blended concrete when compared to that of unblended concrete. The replacement with 30% of metakaolin leads to a substantial improvement in strength and transport properties of blended concrete when compared to that of unblended concrete. This is due to the fact that MK is finer than OPC and producing of an

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additional calcium silicate hydrate (C-S-H) gel, blocking existing pores and altering pore structures (V, 2278).

The use of RHA as a partial replacement to cement will provide an economic use of the by product and consequently produce cheaper materials for low cost construction materials. RHA are very high in silica content but the silica content depends on the type of rice husk, method of firing and period of combustion (Fadzil et al., 2008). RHA can produce a pozzolanic activity but the pozzolanicity of RHA depends on its chemical and physical properties. RHA with highly content silica in amorphous phase reported to react with cementitious binders to perform pozzolanic activity. According to Poon et al. (2006), at temperatures around 40 °C and in the presence of water, the amorphous silica contained in rice husk ash (RHA) can react with Ca(OH)<sub>2</sub> to form more C-S-H gel.

The employment of RHA in cement and concrete has gained considerable importance because of the requirements of environmental safety and more durable construction in the future (Naji Givi et al., 2010).

The use of RHA in concrete has been associated with many advantages, such as increased compressive and flexural strengths (Rodriguez, 2006), reduced permeability (Ganesan et al., 2008), increased resistance to chemical attack (Chindapasirta et al., 2007), reduced effects of alkali-silica reactivity (ASR) (Nicole et al., 2000), reduced shrinkage due to particle packing, making concrete denser, enhanced workability of concrete (Habeb and Fayyadh, 2009), reduced heat gain through the walls of buildings (Lertsatitthanakorn et al., 2009) and reduced amount of super plasticizer (Sata et al., 2007).

Alkali activation of biomass fly ash – an industrial waste and biomass fly ash – metakaolin blends was investigated. Mixtures of biomass fly ash and commercially available metakaolin were used as solid components. Different molar volumes of NaOH and sodium silicate solutions were used for the preparation of alkali activators. The mechanical strength was determined after 10 days of curing. The mineral and microstructural properties of the alkali activated binders were carried out by X-ray diffraction (XRD), thermogravimetric/differential thermal analysis (TG/DTA) and scanning electron microscopy (SEM) methods. The mortar samples of biomass fly ashes gave a compressive strength of 18 MPa. The metakaolin incorporated samples showed a higher strength. The highest value was obtained for 40% metakaolin incorporated mortars for around 38 MPa. The biomass fly ash metakaolin blend is working well in the alkali activation application resulting in the contribution of both aluminosilicates and hydrated phase formation (Rajammaa, 2012).

Supplementary cementing materials (SCM) have become an integral part of high strength and high performance concrete mix design. These may be naturally occurring materials, industrial wastes, or byproducts or the ones requiring less energy to manufacture. Some of the commonly used supplementary cementing materials are fly ash, silica fume (SF), granulated blast furnace slag (GGBS), rice husk ash (RHA) and metakaolin (MK), etc. Metakaolin is obtained by the calcination of kaolinite. It is being used very commonly as pozzolanic material in mortar and concrete, and has exhibited considerable influence in enhancing the mechanical and durability properties of mortar and concrete. This paper presents an overview of the work carried out on the use of MK as partial replacement of cement in mortar and concrete. Properties reported

in this paper are the fresh mortar/concrete properties, mechanical and durability properties (Rafat and et al., 2009).

The results prove that it is possible to obtain RHA concrete with comparable or better properties than those of the control specimen (without RHA) with a lower consumption of cement (Chao-Lung et al., 2011).

The change occurring in the phase composition and microstructure of burnt kaolinite cement pastes after being exposed to high temperatures was studied. The kaolinite clays were thermally activated by firing at 850 °C for 2 h. The ordinary Portland cement (OPC) was partially substituted by 0%, 10%, 20%, and 30% of activated kaolinite clay by weight. It was concluded that, the improvement in compressive strength of pozzolana-cement paste containing burnt kaolinite clay is 27% (Morsy and et al., 1998). The suitable firing temperature of the clay mixes was 800 °C for 2 h (El-Didamony et al., 2000).

## 2. Materials and experimental techniques

Raw materials used in the present work are: OPC from Assuit Cement Co., Egypt, and rice husk ash (RHA) was prepared by burning of rice husk (RHA) for 2 h at 600 °C, and kaolinite clay was collected from Kalabsha, Aswan, Egypt. Kaolinite clay was calcined in an electrical muffle furnace with a heating rate 10 °C/min at 800 °C for 3 h, to give metakaolin (MK). Both of RHA and metakaolin recharged from the muffle furnace, cooled to room temperature in desiccators and ground to pass 90 µm sieves. The various mixes and the chemical compositions of the starting materials are shown in Tables 1 and 2 respectively. Each dry mix was homogenized for 1 h in porcelain ball mill provided with four balls to obtain complete homogeneity then kept in airtight containers until the time of cement paste preparation. The mixing of each dry cement mix was carried out with the required water of standard consistency which is previously determined according to ASTM Designation C 187–98 (ASTM Designation: C 187–98, 2002). The resulting paste was then pressed by hand molding pressure into stainless steel cylindrical molds of 3.14 cm<sup>2</sup> cross-section and 2 cm height. The molds were vibrated for 1 min to remove any air bubbles and voids. Immediately after molding, the cylindrical specimens were cured in humidity cabinet at about 100% relative humidity at room temperature (23 ± 2 °C) for 24 h in order to attain the final setting of the specimens. The specimens then were demolded and cured under tap water for various hydration periods namely: 3, 7, 28, 90 and 180 days.

Bulk density was determined using Archimedes principle (Gennaro et al., 2004). The compressive strength was measured using a manual compressive strength machine for a set of three cubes according to ASTM designation (ASTM designation:

**Table 1** Mix composition in wt.% of blended cements.

Symbol	OPC	MK	Rice husk ash
B	100	0	0
R0	75	25	0
R1	75	20	5
R2	75	15	10
R3	75	10	15
R4	75	05	20

**Table 2** Chemical composition of the starting materials, wt%.

Oxide contents	Portland cement	Rice husk ash	Kaolinite clay
SiO <sub>2</sub>	20.48	86.00	44.18
Al <sub>2</sub> O <sub>3</sub>	4.76	1.16	36.75
Fe <sub>2</sub> O <sub>3</sub>	4.69	1.85	1.36
CaO	62.15	0.64	0.26
SO <sub>3</sub>	2.63	0.32	–
MgO	1.47	0.77	0.16
Na <sub>2</sub> O	0.46	1.14	0.18
K <sub>2</sub> O	0.27	2.54	0.25
TiO <sub>2</sub>	0.58	–	2.94
P <sub>2</sub> O <sub>5</sub>	0.15	0.06	–
L.O.I	1.85	4.77	13.55
Total	99.51	99.25	99.63

C109–80., 1983). Free water content was determined using a domestic microwave oven (Olympic electric model KOR-131G, 2450 MHz, 1000 W) (Pavlik et al., 2003). The combined water content was determined using hydration stopped specimen after being ignited in porcelain crucibles at 1000 °C for 1 h in a muffle furnace. The total porosity of the hardened cement paste was calculated from the values of bulk density, free and total water contents as described elsewhere (Copeland and Hayes, 1956). Free lime CaO were determined at the various curing times after stopping the hydration of the hardened pastes (Grim, 1962; Searl and Grimshaw, 1971). X-ray diffraction (XRD) analyses were carried out by Philips X-ray diffractometer PW 1370; Co. with Ni filtered CuK $\alpha$  radiation (1.5406Å).

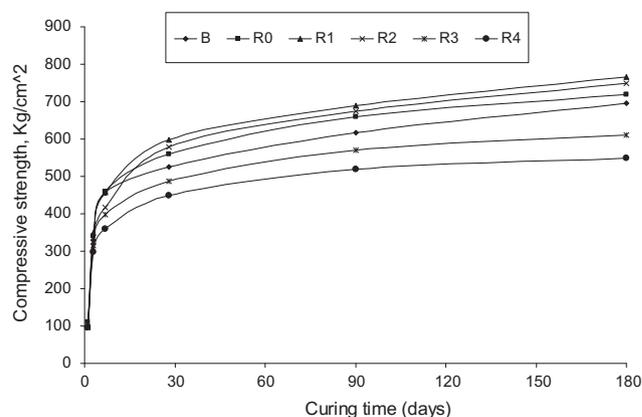
### 3. Results and discussion

#### 3.1. Hydration characteristics of blended cement pastes

##### 3.1.1. Compressive strength

The effect of pozzolana on the strength of the pozzolanic cement pastes depends on number of factors such as the content, type and surface area of pozzolana and the individual characteristics of the OPC (Hewlett, 1998). It is well known that substitution of Portland cement with pozzolana materials reduces the initial rate of strength development at early ages. This may be explained by the slow rate of the pozzolanic reaction (Massazza, 1993). At early ages, pozzolana acts as filler which dilutes the Portland cement. Hence the strength of pozzolanic cement pastes is less than that of OPC at early ages. At later ages the situation is reversed and pozzolanic cement paste attains the same or even a higher compressive strength than the corresponding OPC paste because as the hydration proceeds more hydration products and more cementing materials are formed such as CSH as well as CAS hydrates leading to an increase in compressive strength of hardened cement pastes (Commit 232, 1994; Taha et al., 1981).

The results of compressive strength of the blended cement pastes made from OPC-MK blends with/without rice husk ash at various curing times up to 180 days are illustrated graphically in Fig. 1. The compressive strength of the all pastes increases with curing time. As the hydration proceeds, more hydration products and more cementing materials are formed leading to an increase in compressive strength of cement pastes. This is mainly due to the fact that the hydration prod-



**Figure 1** Compressive strength (kg/cm<sup>2</sup>) of hardened specimens made from PC and MK with/without rice husk ash as function of curing time (days).

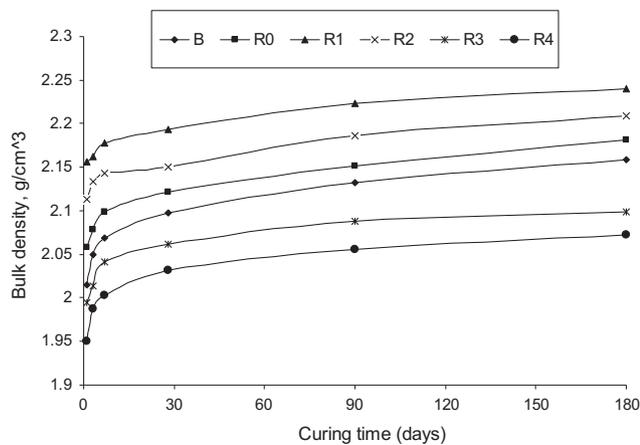
ucts possess a large specific volume than the unhydrated cement. Therefore, the accumulation and compaction of these hydrated products give higher strength. The compressive strength values of the hardened pastes made from PC-MK blends including rice husk ash, increase continuously with increasing age of hydration it is clear that the best mixes are those with 5% and 10% rice husk ash (mixes R1&R2). Thus, 5–10% addition of rice husk ash to PC-MK blends may be considered as the optimum limit. The increase in compressive strength in the presence of 5–10% rice husk ash may be both due to the pozzolanic reaction between calcium hydroxide and silica and the hydration of silica itself will be responsible for the increased compressive strength (Yu et al., 1999).

Up to 10% replacement of rice husk ash, the compressive strength decrease. This may be due to the fact that the quantity of RHA present in the mix is higher than the amount required to combine with the liberated lime during the hydration process thus leading to excess silica leaching out causing a deficiency in strength as it replaces part of the cementitious material but does not contribute to strength (Al-Khalaf and Yousift 1984). In addition to the high surface areas of RHA, that consumes more water for hydration (Eberemu et al., 2013).

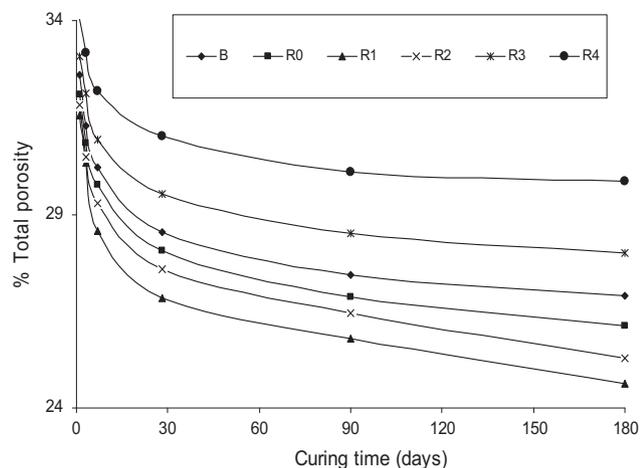
##### 3.1.2. Bulk density and total porosity

The density is an important factor in the determination of porosity, assessment of durability and strength and estimation of lattice constants for the CSH phase in hydrated Portland cement. As the hydration of cement progresses the hydration products fill some of pores because the volume of hydration products is more twice than that of the anhydrous cement; this decreases the porosity and increases the bulk density of hardened cement paste. The bulk density of all hardened cement pastes increases with curing time as a result of the hydration of clinker phases and formation of further hydration products that fill some of pores in the cement paste as well as due to the pozzolanic reaction of MK with liberated CH.

Figs. 2 and 3 show bulk density and total porosity of blended cement pastes PC-MK with/without rice husk ash hydrated up to 180 days, respectively. The bulk density increases while total porosity decreases for all hydrated cement pastes with curing time. As the hydration progresses, hydration products fill some of pores tending to decrease the porosity



**Figure 2** Bulk density ( $\text{kg}/\text{cm}^3$ ) of hardened specimens made from PC and MK with/without rice husk ash as function of curing time (days).



**Figure 3** % Total porosity of hardened specimens made from PC and MK with/without rice husk ash as function of curing time (days).

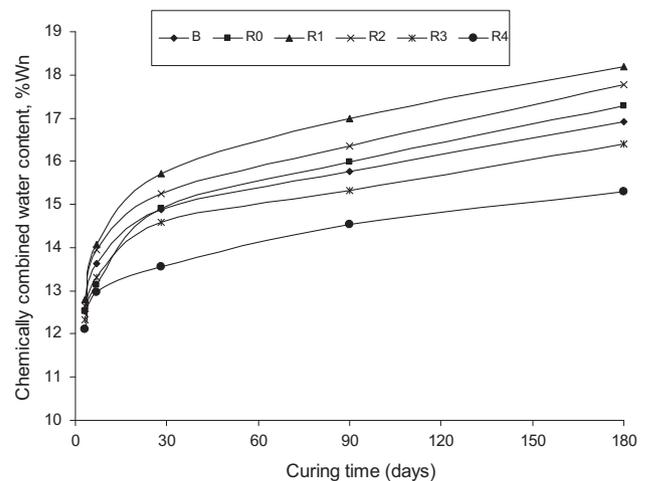
and increase the bulk density of hardened cement paste. The bulk density decreases while the total porosity increases with rice husk ash content due to that rice husk ash lower specific gravity as well as an increase of water of consistency resulting in opening of pore system of the hardened samples.

The bulk density values of pozzolanic cement pastes made from OPC-MK blends including rice husk ash mixes; (R1 and R2) give the highest values of bulk density and the lowest values of porosity which is in a good agreement with the previous studies (Rodrigues et al., 2006; Toutanji et al., 2004; Feng et al., 2004).

### 3.1.3. Chemically combined water content ( $W_n, \%$ )

Fig. 4 shows the combined water content of MK-blended cement pastes with/without rice husk ash hydrated up to 180 days.

The combined water content for all hydrated cement pastes increases with curing time as a result of the progress of hydration of anhydrous clinker phases as well as pozzolanic reaction and formation of hydration products. The combined water



**Figure 4** Chemically combined water content ( $W_n, \%$ ) of hardened specimens made from PC and MK with/without rice husk ash as function of curing time (days).

content of blended cement pastes containing 5–10% rice husk ash (mixes R1&R2) gives higher combined water content as compared with those made of cement containing MK alone. The additional formation of (C–S–H) during the interaction between RHA, as an active silica, with free lime released accounts for higher strength values of all the hardened PC–MK–RHA paste. In other words, that rice husk ash at lower substitution level acts as a nucleating agent for hydration products enhancing the cement hydration while increasing rice husk ash content make a dilution effect (Mostafa and Brown, 2005). Also results show that the variations of combined water content with increasing age of hydration are almost parallel to the changes in the compressive strength.

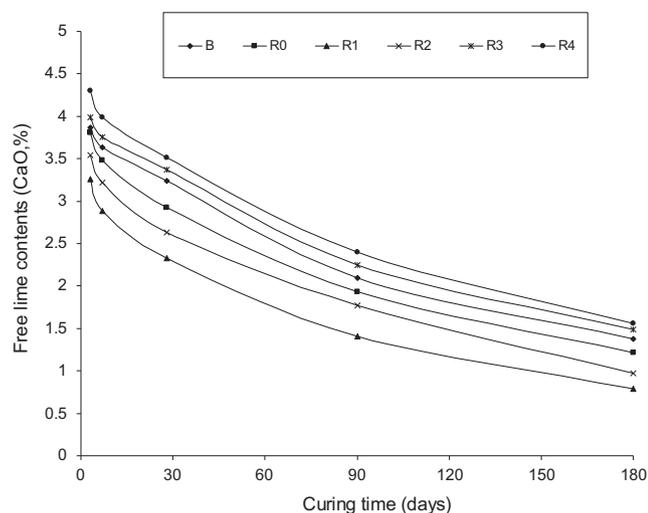
### 3.1.4. Free lime content ( $\text{CaO}, \%$ )

The results of free lime content ( $\text{CaO}, \%$ ) of the hardened blended cement pastes made from OPC-MK in the absence and presence of various ratios of rice husk ash at different hydration ages are illustrated graphically in Fig. 5.

All mixes show that the free lime content ( $\text{CaO}, \%$ ) of all mixes decreases as the curing age increases this is due to the pozzolanic reaction of metakaolin (MK) with free lime forming more calcium silicate hydrates. Such behavior can be explained to the high pozzolanic activity of rice husk ash which participates in the consumption of free lime. Obviously, the quantity of free lime increases with increasing rice husk ash content in the blended cement pastes. This may be attributed to the leaching of  $\text{Ca}^{2+}$  ions from rice husk ash, in addition to the lime liberated due to the hydration of OPC. Free lime content of blended cement pastes containing 5–10% rice husk ash (mixes R1&R2) gives lower value as compared with those made of only Portland cement or containing MK alone which is in a good agreement with the compressive strength values.

### 3.1.5. -DTA results

The differential thermal analysis (DTA) is a very useful technique to identify the phases coexisting during the hydration of cement pastes. Fig. 6 shows the DTA thermograms of the investigated pozzolanic cement pastes made from PC and MK with/without rice husk ash cured in water for 180 days.

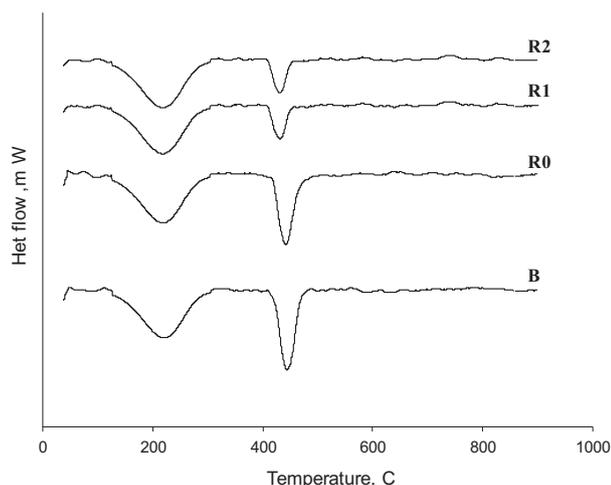


**Figure 5** Free lime content (CaO,%) of hardened specimens made from PC and MK with/without rice husk ash as function of curing time (days).

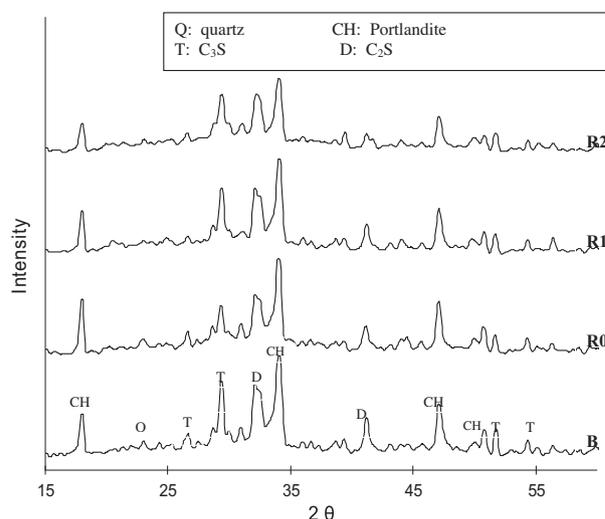
The broad peaks in thermograms below 300 °C are attributed to the dehydration of calcium silicate hydrate (CSH), calcium aluminate hydrate (CAH) as well as calcium sulfoaluminate hydrate (CSAH). The endothermic peak at 450 °C is due to the decomposition of CH. In the blended cement pastes made from OPC and MK blends in the absence of rice husk ash, the intensity of CH peak at about 450 °C indicates that MK reacts with CH liberated from the hydration of cement and forms additional calcium silicate, aluminate as well as sulfoaluminate hydrates, this peak (at about 450 °C) decreases with increasing rice husk ash content up to 10% (mixes R1&R2). Therefore, rice husk ash, due to its very high surface area interacts rapidly with the free calcium hydroxide, liberated from the hydration, leading to the formation of excessive amounts of calcium silicate hydrates (CSH) [Abdel Rahman and et al. \(2016\)](#).

### 3.1.6. XRD results

The XRD technique is a very useful technique to identify the phase composition which forms during the hydration of the



**Figure 6** DTA thermograms of the hardened specimens made from PC and MK with/without rice husk ash for 180 days.



**Figure 7** XRD patterns of the hardened specimens made from PC and MK with/without rice husk ash for 180 days.

investigated OPC-MK blends, as well as blended hardened cement pastes containing rice husk ash. The XRD patterns of the investigated hardened OPC (mix. B), and MK paste without rice husk ash cured in water for 180 days (mix R0) show the main peak characteristics to Portlandite (CH). This is due to the reaction of MK with CH liberated from the hydration of cement to form additional calcium silicate hydrates. The XRD patterns of the investigated hardened OPC-MK paste with rice husk ash, there is a noticeable decrease in the intensity of the main peak of CH.

**Fig. 7** illustrates the XRD patterns of the investigated pozzolanic cement pastes made from PC and MK with/without rice husk ash cured in water for 180 days. It was observed that the amount of portlandite decreases with increasing rice husk ash content up to 10% (mixes R1&R2) because of pozzolanic activity of rice husk ash which consumes portlandite forming additional C-S-H.

## 4. Conclusion

The main conclusions derived from this study could be summarized as follows: At the early ages of hydration rice husk ash acts as filler whereas at later ages it acts as pozzolana. Increasing rice husk ash content makes a dilution effect, requires higher water demands and forms a layer of rice husk ash particles around anhydrous cement grains which delays the hydration of cement. Accordingly it is recommended to use the pozzolanic cement mix containing 75 wt% OPC, 20–15 wt% MK and 5–10 wt% rice husk ash, respectively instead of OPC and control cement for general construction purposes. In addition, the utilization of rice husk ash in construction purposes solves the problem of its disposal thus keeping the environment free from pollution.

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