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Research Article

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Rice Husk Ash for Enhancing Salts Attack Resistance of Blended Cement Containing Metakoalin

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Abstract: The effects of addition rice husk ash on the mechanical properties of blended cement containing metakaolin have been investigated experimentally as well as sulphate and chloride resistance of these blends. The influence of aggressive media such as 5% $MgSO_4$ and 5% $MgCl_2$ on the mechanical properties of the prepared pozzolanic cement pastes was studied. The pastes were made from ordinary Portland cement containing 25 wt % metakaolin with partial replacement of metakaolin by different ratios (0, 5, 10, 15 and 20 wt %) of rice husk ash. All cement pastes immersed in tap water for 28 days (zero time) then immersed in aggressive media for 1, 3, 6, 9 and 12 months. The mechanical properties were measured by determination of the compressive strength. Total sulphate and chloride contents at each curing time in the aggressive mediums up to one year as well as their physico-chemical properties were monitored periodically and analyzed by XRD, DTA and visual inspection. The results showed that, the partial replacement of metakaolin by 10 wt % rice husk ash improves the resistance of hardened cement pastes against 5% $MgSO_4$ attack. Whereas 15 wt % rice husk ash improves the resistance against 5% $MgCl_2$ attack.

Keywords: Rice Husk Ash; Metakaolin; Blended Cement; Salt Attack

1. INTRODUCTION

Recently, there has been a high interest in using metakaolin (MK) as a supplementary cementitious material [1-3]. MK is an ultra fine pozzolana, which consists predominantly of silica and alumina can produced by calcining kaolin at temperatures between 700 and 900 ºC. MK has high ability to increase the concrete strength especially during the early ages of hydration [4, 5]. This is due to filling the space between cement particles, acceleration of cement hydration and pozzolanic reaction of MK. This is similar to silica fume effect. Although the pore volume slightly increases in pastes containing MK, the pore structure of paste, is found to be refined [6]. As a result of the reaction between protland cement (PC) hydration product and MK, the portlandite content in MK paste and mortar is reduced. Therefore,

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the increasing in sulphate resistance of mortars containing MK was attributed to the reduction in portlandite in the system [7]. The refinement in pore structure is considered as the other reason for the improved sulphate resistance, which would hinder the ingress of sulphate ions. The incorporation of MK in concrete increases the penetration resistance of chloride ions [4]. Pastes containing MK showed higher capacity to bind chloride ions compared with PC pastes [8]. The concrete corrosion resistance influences its durability and finally its performance. The concrete performance depends on the microstructure, the chemistry of the concrete and the environmental conditions. It is obvious that the presence of MK affects the concrete performance.

Rice husk ash (RHA) is an agricultural waste and possesses a very high amount of high surface area amorphous SiO_2, so it is classified as a highly active pozzolan [9-12]. RHA is consider as the alternative source of silica fume which is high cost and not available. The addition of RHA to a concrete mixture has been proven to increase corrosion resistance. It has a higher early strength than concrete without RHA [13]. RHA forms a calcium silicate hydrate gel around the cement particles which is highly dense and less porous. This will prevent the cracking of the concrete and protect it from corrosion by preventing any leeching agents to break down the material. The presence of RHA in concrete shows lower ratio of soluble/total chloride ions content than those of OPC concretes have been reported [14].

The effect of RHA on corrosion resistance of Portland cement concrete and concluded that RHA is very effective in improving the corrosion resistance of mortars indicating better contribution of RHA to corrosion resistance [15]. sulphate ions transported into the pore and reacted with hydration products to form ettringite and gypsum, which could block the pore, micro-crack and interfacial transition zone [16]. As previously mentioned, there are huge quantities of RHA found as waste material; these waste materials make environmental disposal problems hence there is current interest in this work to investigate the suitability of using rice husk ash in combination with metakaolin which is abundant in Egypt in preparation of pozzolanic cement with improved properties.

This work aims to study the effect of the aggressive solutions $MgSO_4$ and $MgCl_2$ on the durability of prepared pozzolanic cement pastes containing RHA. Moreover, the enhancements in Portland cement resistance to the aggressive solutions as result of adding RHA were discussed. The investigations are carried out by the determination of compressive strength as well as total sulphate and chloride content of hardened cement pastes cured in the aggressive medium for different periods.

2. MATERIALS AND METHODS

Raw materials used in the present work are: ordinary Portland cement (OPC, from Assuit Cement Co., Egypt), rice husk ash (prepared by burning of rice husk for 2 h at 650 ◦C), and kaolinite clay (collected from Kalabsha, Aswan, Egypt). Kaolinite clay was calcined in an electrical muffle furnace at 800 °C for 3 h with a heating rate 10 °C/min to give metakaolin (MK). Both of RHA and metakaolin recharged from the muffle furnace, cooled to room temperature in desiccators and ground (less than 90 μm). Various mixtures from OCP, RHA and MK have been prepared with different weight ratios and given the codes S0, S5, S10, S15 and S20 in case of 5% $MgSO_4$ and C0, C5, C10, C15 and C20 in case of 5% $MgSO_4$ as shown in Table 1. The mixtures were homogenized for one hour in porcelain ball mill then kept in airtight containers until the time of cement paste preparation. The dry mixtures were mixed for three minutes with the suitable water/solid ratio required to attain the standard consistency.

The resulting paste was then pressed by hand molding pressure into stainless steel cylindrical moulds of 3.14 cm^2 cross-section and 2 cm height. The moulds were vibrated for one minute to remove any air bubbles and voids. Immediately after molding, the cylindrical specimens were cured in humidity cabinet at approximately 100% relative humidity at room temperature (23 $\pm$ 2 °C) for 24 hours in order to

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attain the final setting of the specimens. The specimens then were demoded and cured under tap water for 28 days (zero time).

Two sets of samples were cured under 5% $MgSO_4.7H_2O$ and 5% $MgCl_2.6H_2O$ to investigate their resistance to this aggressive medium at the desired testing times 1, 2, 3, 9 and 12 months. The aggressive solution was renewed monthly to maintain constant concentration. The aggressive attack was determined through the measurements of compressive strength, total sulphate and total chloride at each time of testing.

The total sulphate content was estimated gravimetrically by taking 1 g of powdered sample and dissolving it in 5 mL concentrated HCl. 100 ml of distilled water was added, boiled for 5 minutes, filtered and washed several times with distilled water. 10 mL of 10% $BaCl_2$ was added to the filtrate, which then digested, filtered and ignited. The total sulphate content was calculated from the weight of ignited samples.

Total chloride content was determined in the hardened pastes by adding 2 g sample into a stopper conical flask. The specimen has dispersed in 25 mL water and then 10 mL of nitric acid has added. 50 mL of hot water has been added, heated to near boiling and kept warm for 10 to 15 min. Filtration step may be performed if the supernatant liquid is turbid. An excess of standard 0.1 N $AgNO_3$ and 2–3 mL of nitrobenzene have been added, then shaken vigorously to coagulate the precipitate. 1 mL ammonium ferric alum as indicator has been added and titrated against standard 0.1 N ammonium thiocyanate (NH_4SCN).

X-Ray florescence (XRF, Philips X-ray PW1370) was used to verify the oxide content of the starting materials (OPC, RHA and MK). X-ray diffraction (XRD, Philips PW1700 diffractometer system using Cu-K_α radiation) was used to identify the phase and structure of the samples. Differential thermal analysis (DTA, Schimaduz DTA-50 thermal analyzer Co-Kyoto, Japan) was used to study the thermal behavior of the samples during the treatment processes. The sample also observed by visual inspection to flow the apparent changes.

Table 1. Mixture composition (wt %) of blended cements.

Code*	OPC	MK	RHA
S0 C0	75	25	0
S5 C5	75	20	5
S10 C10	75	15	10
S15 C15	75	10	15
S20 C20	75	5	20

* S and C in case of 5% $MgSO_4$ and 5% $MgCl_2$, respectively.

3. RESULTS AND DISCUSSION

The chemical composition of OPC, RHA calcined at 650 °C for 2 h and MK calcined at 800 °C for 3 h were tested using XRF and the data are shown in our recent publication [3].

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3.1 Effect of 5% $MgSO_4$ on Properties of OPC-MK and OPC-MK-RHA Mixtures

3.1.1 Compressive Strength

The results of compressive strength of pozzolanic cement pastes made from OPC and MK with/without RHA immersed in 5% $MgSO_4$ as a function of curing time up to 12 months are shown in Figure 1. The compressive strength of pozzolanic cement pastes increases with curing time up to 3 months and then decreases gradually with increasing the curing time. The increase of compressive strength is due to the progress of hydration and sulphate ions activate the hydration of cement pastes. After 3 months, the MK mortar specimen stored in $MgSO_4$ solution showed a complete transformation of the C-S-H to M-S-H as well as formation of secondary gypsum and ettringite. The latest one expands causing cracks and deterioration of hardened cement paste. Similar results were found in case of curing in sodium sulphate solution [17, 18].

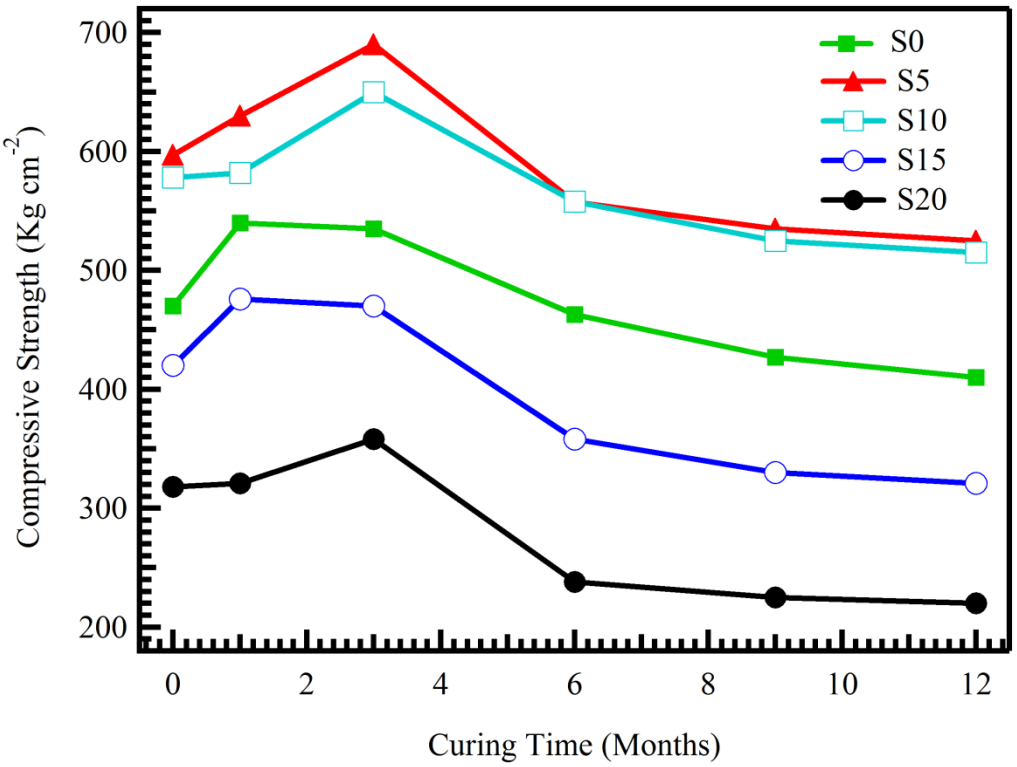

Figure 1. Compressive strength of hardened specimens immersed in 5% $MgSO_4$ as function of curing time.

It was observed that the samples S5 and S10 containing 5% and 10% RHA, respectively show higher compressive strength. This can attributed to that RHA consumes CH formed during hydration which consequently reduces the content of ettringite and gypsum hence the penetration of sulphate ion is reduced. Increased amounts of RHA (15% and 20%) enhance the water demand associated with its high surface areas resulting in the penetration of sulphate ion increases and then the compressive strength decreases.

3.1.2 Total sulphate Content

Figure 2 shows the total sulphate content pozzolanic cement pastes made from OPC and MK

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with/without RHA immersed in 5% $MgSO_4$ as a function of curing time up to 12 months. One can observe that the total sulphate contents increase with curing time for all cement pastes, especially at early ages of hydration. This is due to the migration of the sulphate ions to the cement paste, which reacts with aluminate and $Ca(OH)_2$ of the cement paste, forming calcium trisulphoaluminate hydrates (ettringite), which have 227% volume, leading to the expansion of the resulting products. Sulphoaluminate hydrates are formed at early ages of hydration, which need high amount of sulphate ions, precipitate, close the pores and hinder the penetration of more sulphate ions [17].

Therefore, the total sulphate content increases sharply up to 6 months and gradually at the later ages of hydration. The blended cement pastes containing RHA show lower values of the total sulphate content than those of only MK; this is due to the high pozzolanic activity of RHA which participates MK in the consuming of CH. In the other hand, Figure 2 shown that the total sulphate content increasing with increase RHA content up to 15 wt % due to increasing the water demand associated with its high surface areas resulting in the penetration of sulphate.

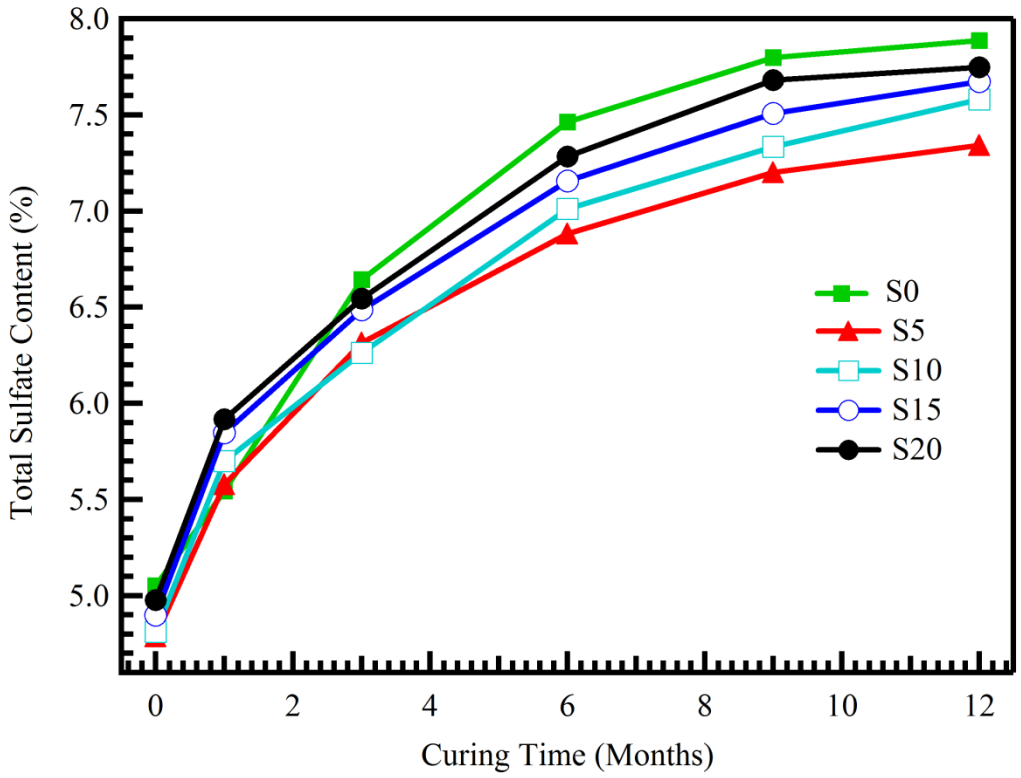

Figure 2. Total sulphate content of hardened specimens immersed in 5% $MgSO_4$ as function of curing time.

3.1.3 DTA Results

Figure 3 shows the DTA thermograms of control and pozzolanic cement pastes S0 (75 wt % OPC with 25 wt % MK) and S10 containing 10 wt % RHA immersed in 5% $MgSO_4$ solution for 12 months. The DTA peaks for sample containing RHA are more intense and slightly shifted to higher temperatures [5]. It is observed, the aggressive attack is characterized by the appearance of endothermic peaks around 200 °C and near 500 °C. The broad peak at around 200 °C is due to the dehydration of calcium sulfoaluminate hydrates (ettringite), monosulphate as well as CSH and CAH phases. While the endothermic peak near 500 °C is due to the dehydration of CH. When MK was replaced by RHA, the

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intensity of the endothermic peak due to the dehydration of CH decreases markedly due to that RHA participates MK to consume CH forming CSH. This reflects the high pozzolanic activity of RHA. It is known, during $MgSO_4$ attack, the Mg^{+2} forms a layer of brucite ($Mg(OH)_2$) at the exposed surface. The brucite formation consumes a high amount of $Ca(OH)_2$ released from hydration. In other words, due to that $MgSO_4$ attacks CSH to form silica gel, brucite and gypsum. This is confirmed from the appearance of brucite and gypsum as a white coat in hardened cement pastes made from OPC, MK and RHA immersed in $MgSO_4$ solution for 12 months. The peak around 830 °C is due to the recrystallization of wollastonite formed due to the decomposition of CSH.

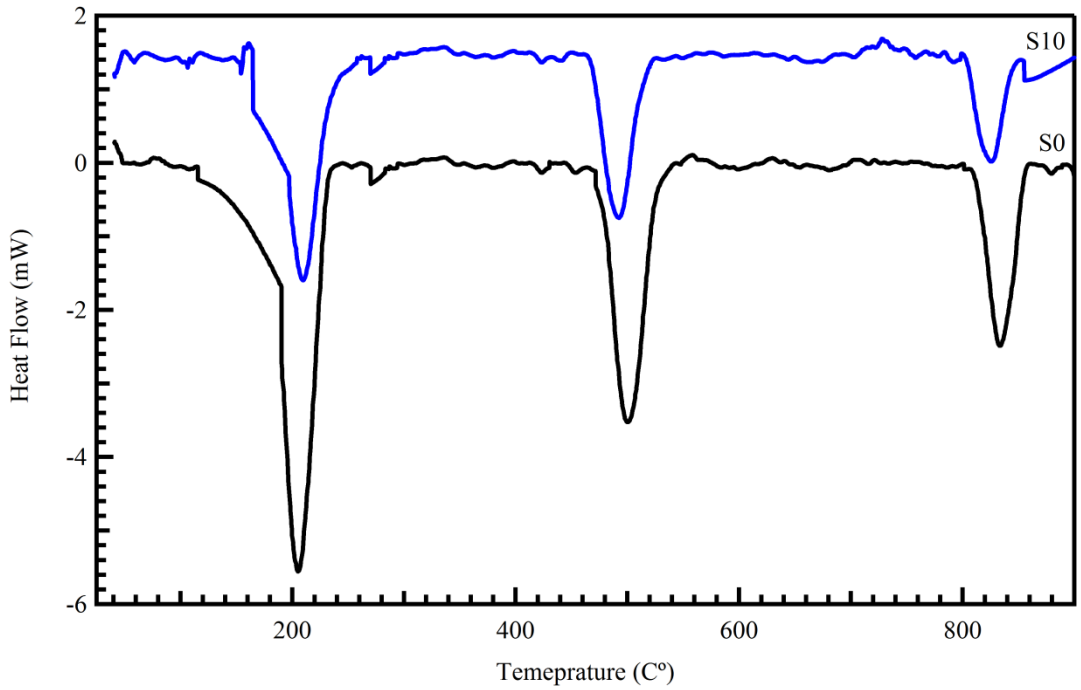

Figure 3. DTA thermograms of hardened pozzolanic cement pastes immersed in 5% $MgSO_4$ solution for 12 months.

3.1.4 Visual Inspection

Figure 4 shows the surface appearance of the hardened pozzolanic cement paste cylinders made of OPC and MK with/without RHA after one year of immersion in 5% $MgSO_4$. It is clearly shown that hardened cement pastes containing high ratio of RHA have been damaged. The aggressive attack is characterized by formation of a white coat of brucite and gypsum on sample surface which peeled off leaving porous damaged surface. The formation of the white coat is due to the attack of $MgSO_4$ solution on CSH which enhanced and accumulated as a result of the pozzolanic reaction of RHA with CH liberated from the hydration of cement. The damage in sample S10 (10% RHA) is less than in samples S20 (20% RHA) indicating replacement of MK by 10% RHA leading to enhancement the resistance to sulphate attack.

3.1.5. XRD Results

Figure 5 illustrates the XRD patterns of the investigated pozzolanic cement pastes made from PC and MK (S0) and PC, MK and RHA (S10) after one year of immersion in 5% $MgSO_4$. The results show that, after 12 months of immersion, the diffraction peaks of ettringite and gypsum are clearly detected

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indicating the ettringite and gypsum indeed formed in the concrete [16]. This is due to the migration of the sulphate ions to the cement paste, which reacts with aluminate and $Ca(OH)_2$ forming calcium trisulphoaluminate hydrates (ettringite) leading to the expansion of the resulting products. However, one can observe that the amount of unhydrated clinker phases such as C_2S (β-dicalcium silicate, β-$2CaO.SiO_4$) and C_3S (tricalcium silicate, $3CaO.SiO_4$) decreases with adding RHA because of dilution of cement by RHA on the expense of OPC. Portlandite also decreases with RHA content. The pozzolanic activity of rice husk of ash consumes portlandite forming additional C-S-H. There is low intensity peak is due to the recrystallization of wollastonite formed as result to the decomposition of CSH.

Figure 4. Typical surface appearances of hardened pozzolanic cement pastes immersed in 5% $MgSO_4$ for 12 months.

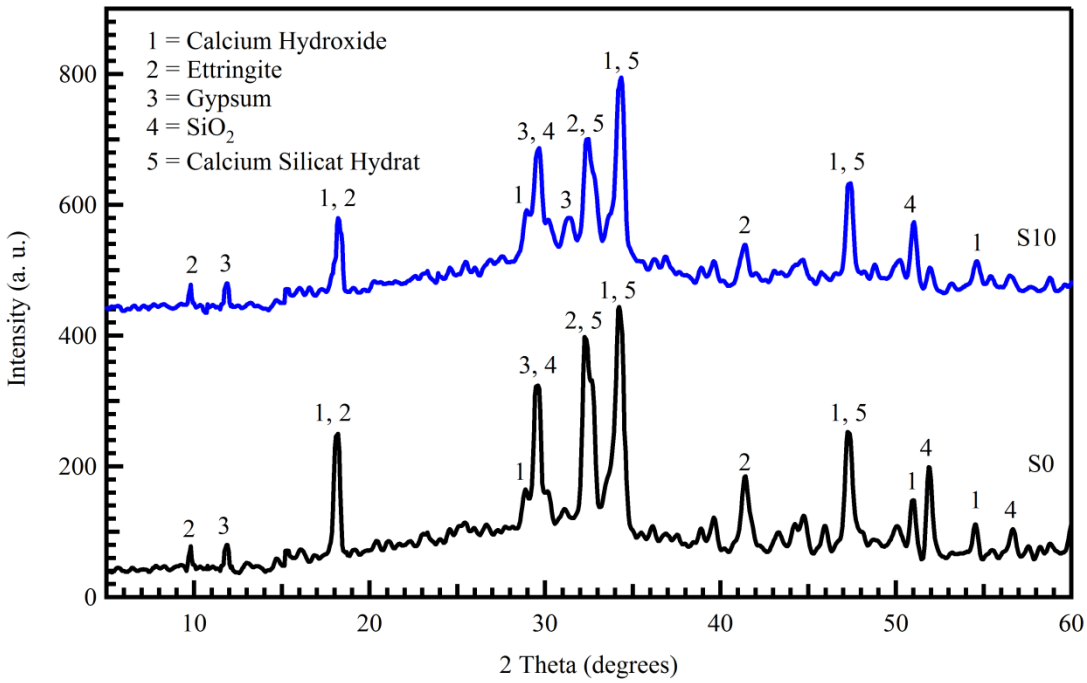

Figure 5. XRD patterns of hardened pozzolanic cement pastes immersed in 5% $MgSO_4$ solution for 12 months.

3.2. Effect of 5% $MgCl_2$ Solution on Properties of OPC-MK and OPC-MK-RHA Mixtures

3.2.1 Compressive Strength

The compressive strength of pozzolanic cement pastes made from OPC and MK with/without RHA after one year of immersion in 5% $MgCl_2$ solution are shown in Figure 6. The compressive strength

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of all cement pastes increases gradually with curing time up to 3 months then decreases up to 12 months of curing in $MgCl_2$ solution. The compressive strength increases due to that the chloride ions activate the hydration of cement and formation of hydration products which fill some of pores enhancing the compressive strength. Also, Mg^{2+} ions adsorbed and incorporated into the particles of CSH; enhance the crystallization of CSH and the strength of cement pastes. Generally, based on the obtained results, adding MK to mortar may be lead to lower its chloride ion permeability [19]. Although partially attributable to the modification of the microstructure resulting from the pozzolanic addition discussed earlier, this effect due to the high alumina content in MK, in as much as this component can bond to the chloride ions. The compressive strength decreases for all pozzolanic cement pastes due to the diffusion and aggressive attack of $MgCl_2$ on CH, C_3A and CSH leading to formation of chloroaluminate hydrates as well as M-S-H which are accompanied by softening and loss of strength, consequently, the strength of hardened cement pastes decreases. Moreover, the compressive strength increases with RHA content up to 15 wt % (C15) and then decreases for higher RHA content. The increasing in compressive strength is due to RHA consumes CH susceptible for attack by chloride ions. RHA also refines the pore structure of cement paste and delays the diffusion of chloride ions thereby reduces deterioration of cement paste by $MgCl_2$ solution. Further addition of high ratio of RHA more than 15 wt % (C15) increases the water demand and the total porosity as well as the penetrability of chloride ions through hardened cement paste increase and as a result the compressive strength decreases.

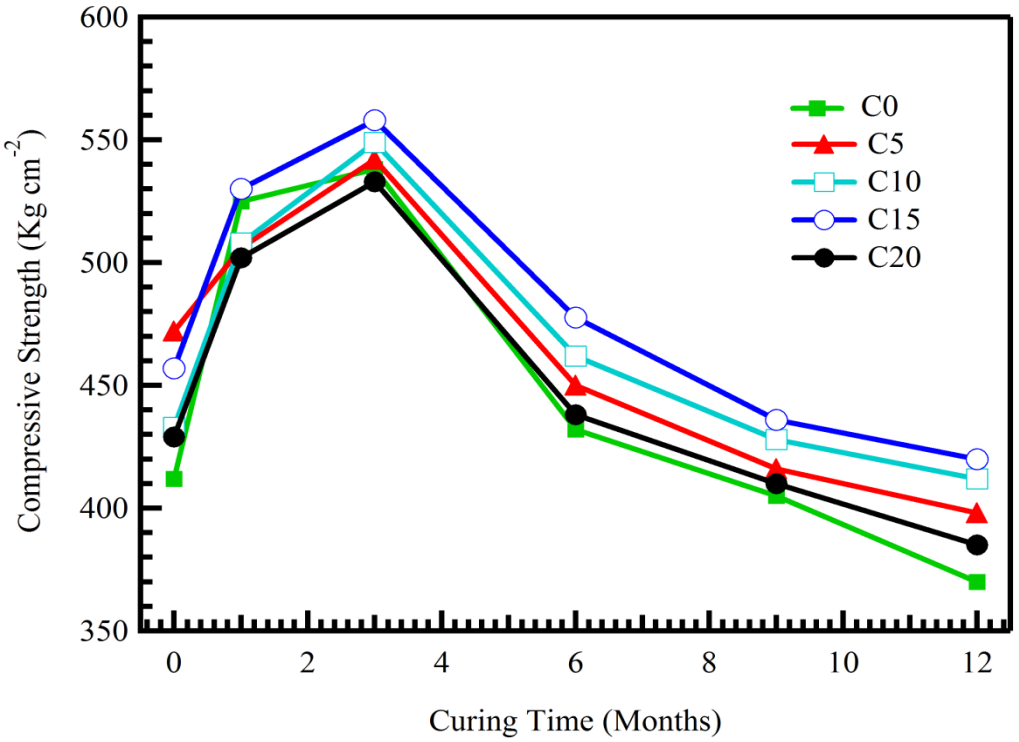

Figure 6. Compressive strength of hardened specimens immersed in 5% $MgCl_2$ as function of curing time.

3.2.2 Total Chloride Content

The total chloride contents of control and pozzolanic cement pastes made from OPC, MK and RHA after one year of immersion in 5% $MgCl_2$ solution are illustrated in Figure 7. The results show that

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the total chloride content increases gradually with curing time for all hardened cement pastes. This is due to the reaction of chlorides with $Ca(OH)_2$ to produce $CaCl_2$, and with tricalcium aluminate as well as calcium aluminoferrite hydrates, giving chloroaluminate hydrate. The total chloride contents decrease with RHA content up to 15 wt % (C15); that attributed to the reduction in the average pore diameter of cement paste caused by the incorporation of RHA in the mixture will effectively reduce the pore sizes, permeability and diffusivity of chloride ions in concrete. This blending leads to the formation of more compact and finer pore structure [13]. Moreover, addition of high ratio of RHA (more than 15 wt %) increases the water demand and the total porosity as well as the penetrability of chloride ions through hardened cement paste increase and as a result the total chloride contents increases.

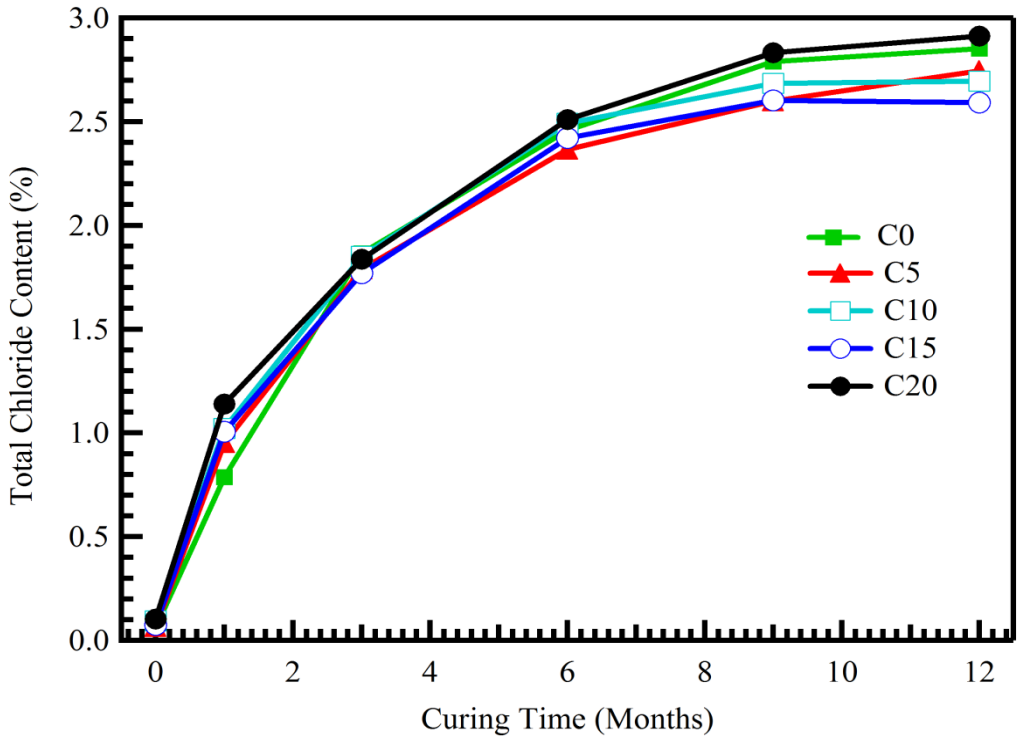

Figure 7. Total chloride content of hardened specimens immersed in 5% $MgCl_2$ as function of curing time.

3.2.3 DTA results

Figure 8 shows the DTA thermograms of the control paste made from 75 wt % OPC with 25 wt % MK (C0) and pozzolanic cement paste made from 75 wt % OPC with 10 wt % RHA and 15 wt % MK (C15) immersed in $MgCl_2$ solution for 12 months. The endothermic peaks located at 200°C can be attributed to the dehydration of amorphous and crystallized calcium silicate hydrates, calcium aluminate hydrate as well as calcium sulfoaluminate hydrate. It is obvious that when MK has been replaced by RHA, the endothermic peaks due to the dehydration of chloroaluminate hydrate near 450 °C were very small. This is due to that RHA consumes the most of CH which is susceptible for attack by chloride ions. One can conclude, RHA improves the resistance against $MgCl_2$ attack because it consumes CH liberated from hydration which is susceptible for attack of chloride ions. However, using finely ground RHA reduced the rapid chloride penetrability of concrete from a moderate rating to low or very low ratings depending on the type and addition level of RHA [9].

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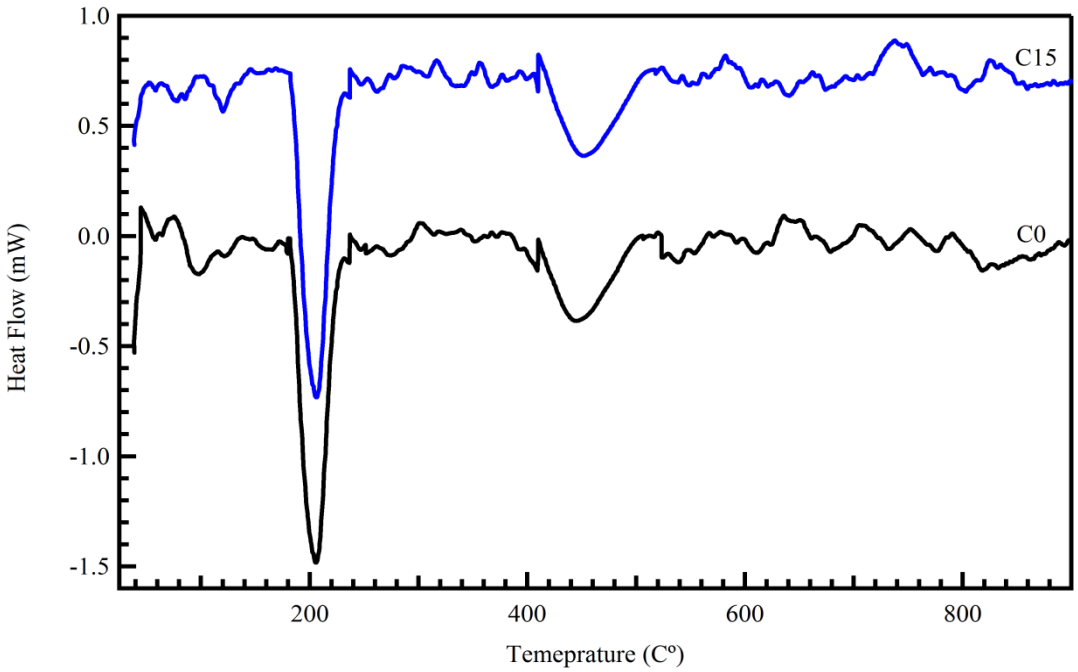

Figure 8. DTA thermograms of hardened pozzolanic cement pastes immersed in 5% $MgCl_2$ solution for 12 months.

3.2.4 Visual Inspection

Figure 9 shows the surface appearance of the hardened pozzolanic cement paste cylinders made of OPC and MK with/without RHA after one year of immersion in 5% $MgCl_2$. It is clear that in case of $MgCl_2$ aggressive attack, the hardened cement pastes immersed in $MgCl_2$ solution have suffered severe loss of strength in comparison with that immersed in $MgSO_4$ solution. This is mainly due to the attack of $MgCl_2$ solution upon most of all constituents of hardened cement paste especially CH, C_3A and CSH. This attack is accompanied with the formation of calcium chloroaluminate hydrates as well as MSH gel which possess no binding characteristics and are accompanied by loss of strength. This assumption is in agreement with the compressive strength results. Chloride ion occupies lower specific volume and hence has higher permeability through the hardened cement paste than sulphate ions.

Figure 9. Typical surface appearance of hardened pozzolanic cement pastes immersed in 5% $MgCl_2$ for 12 months.

3.2.5 XRD Results

Figure 10 illustrates the XRD patterns of the investigated pozzolanic cement pastes made from PC and MK with/without RHA after one year of immersion in 5% $MgCl_2$. The results show that, after 12 months immersion of specimens in $MgCl_2$ solution there are clear diffraction peak of chloroaluminate.

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This is mainly due to the attack of $MgCl_2$ solution upon most of all constituents of hardened cement paste especially CH, C_3A and CSH. This accompanied with the formation of calcium chloroaluminate hydrates as well as MSH gel which possess no binding characteristics and are accompanied by loss of strength. It can be concluded that partial replacement of MK by 15 wt % RHA (C15) improves the resistance against $MgCl_2$ attack because RHA consumes CH liberated from hydration which is susceptible for attack of chloride ions. The peaks corresponding ettringite and gypsum are not detectable. Also, it was observed that the amount of unhydrated clinker phases C_2S and C_3S decreases with RHA content. Portlandite decreases also with RHA content because of the previous reasons in addition to pozzolanic activity rice husk of ash which consumes portlandite forming additional C-S-H. This result is in agreement with previous studies [10, 19, 20].

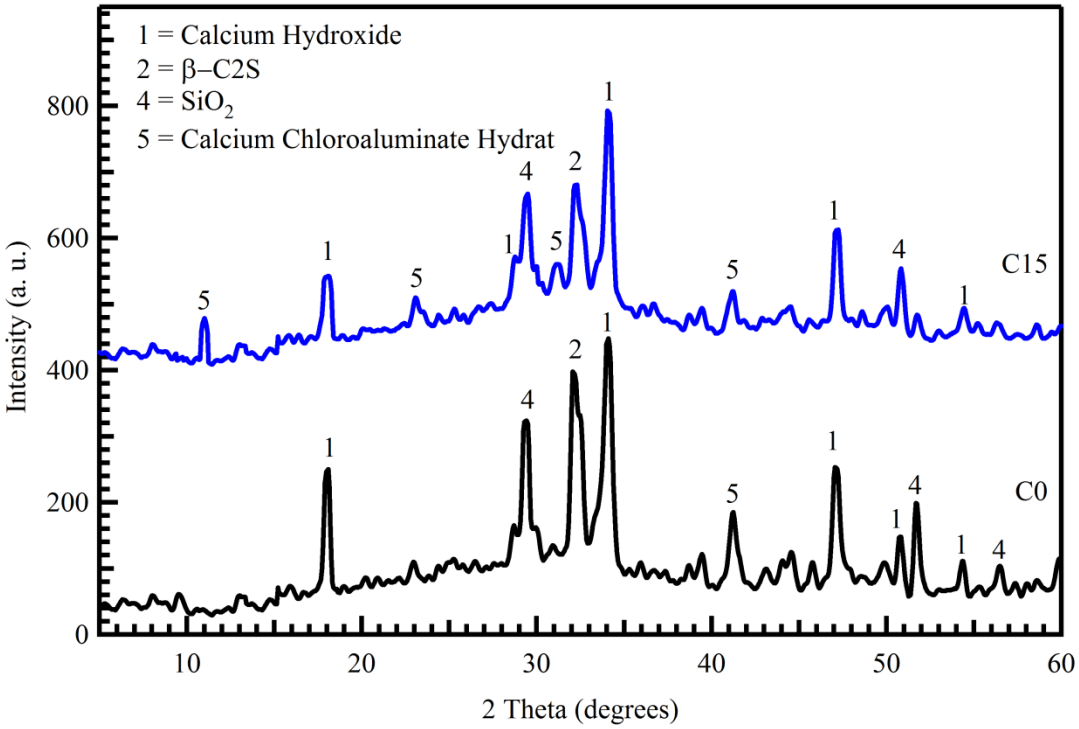

Figure 10. XRD patterns of hardened pozzolanic cement pastes immersed in 5% $MgCl_2$ solution for 12 months.

4. CONCLUSION

Addition of rice husk ash up to 10 wt % to blended cement containing metakoalin enhances the compressive strength. Characterization data such as XRD, DTA and other measurements confirmed that the partial replacement of MK by RHA improves the resistance of hardened cement pastes against 5% $MgSO_4$ and 5% $MgCl_2$ attack. Whereas RHA improves the resistance against 5% $MgCl_2$ attack even up to 15 wt % replacement levels because RHA consumes CH liberated from hydration which is susceptible for attack of chloride ions. It can be concluded that the mixture containing 75 wt % OPC and 25 wt % MK and partial replacement of MK by 10-15 wt % RHA is the suitable durable mixture for using under action of aggressive media. Generally RHA can be considered as a supplementary cementitious material using for producing ultra high performance concrete.

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The authors declare no conflict of interest

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