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Investigation of Effect of Mineral Additives on Hydration Behavior of Portland Cement Using Electrochemical Technique

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The current study examined the effects of rice husk ash (RHA) and quarry dust (QD) as mineral additives on the setting time, compressive strength, and hydration behavior of Portland cement (PC). When compared to control (PC0), blending QD (20-60%) and RHA (10-30%) into PC speeds up the setting. QD addition reduced the strength of PC and a maximum strength of 55.1 MPa was attained after 4 weeks of curing with 20% of QD (QD20). The addition of RHA increased the strength and a maximum strength of 68.1 MPa was obtained with 20% of RHA (RHA20) blended cement. XRD and SEM analyses confirmed the formation of various crystalline phases of hydration products and their effects on hydration behavior of blended cement. The electrochemical studies illustrated that the values of charge-transfer resistance increase with increasing hydration time. In addition, QD and RHA can affect the charge transfer impedance. Results showed after 4-weeks hydration time, QD and RHA additives can reduce the value of charge transfer impedance and hydration degree of PC paste.

Keywords: Mineral Additives; Hydration Behavior; Rice Husk Ash; Quarry Dust; Portland Cement; Setting Time; Compressive Strength; Electrochemical Technique

1. INTRODUCTION

During the construction of a structure, concrete is used to provide strength, durability, and versatility [1, 2]. Concrete's excellent properties have made it a dependable and long-lasting choice of construction companies for both commercial and residential construction [3]. One of the most important properties of concrete and mortar is its compressive strength [4, 5]. The strength of the binder (cement) has a significant impact on the mixture's performance characteristics. Concrete's compressive strength is its ability to withstand failure in the form of cracks and fissures when a specific amount of load is applied to it [6]. As a result, earthquake-resistant building configuration considerations can help maintain a building, and concrete strengthening will become necessary [7, 8].

Mineral additives are among the most promising concrete components for new building material development [9, 10]. Concrete density, compressive strength, freeze-thaw resistance, and setting time can all be increased by replacing cement with mineral additives [11, 12]. Mineral additives can be used to improve cementation systems. Mineral additives are widely available and can be used to replace portland cement in concrete [13, 14]. Fly ash, granulated blast-furnace slag, silica fume, natural pozzolans, rice-husk ash, and metakaolin are among them [15, 16]. Although calcium chloride is the most common accelerator component, it can promote corrosion activity in steel reinforcement [17, 18].

Because agricultural wastes produce millions of tons of agricultural waste each year, agricultural waste ashes can be an eco-friendly, economical, and appropriate mineral additive [19]. As a result, numerous studies have been conducted to identify and optimize the level of agricultural waste ashes as additives in concrete [20-24]. Reusing waste rice husk ash as pozzolanic mineral additives and quarry dust as nonpozzolanic mineral additives plays an important role in sustainable solid waste management because it not only saves natural resources but also significantly reduces environmental pollution [25, 26].

Some researchers have used rice husk ash and quarry dust as cement ingredients in concrete matrixes [27-29]. However, just a little research has been focused on the application of rice husk ash and quarry dust as binary cementitious mineral additive materials in concrete structures [30, 31]. Furthermore, studies of rice husk ash and quarry dust have focused solely on compressive power, ignoring properties such as cement hydration and electrochemical properties. To our best knowledge, this study is the first report on the electrochemical evaluation of corrosion resistance of steel rebar incorporated into concrete, which was prepared with Portland cement blended with RHA and QD as mineral additives in a 3.5 wt% NaCl solution. As a result, the current study was conducted to evaluate the effect of rice husk ash and quarry dust as mineral additives on Portland cement setting time, compressive strength, and hydration behavior.

2. EXPERIMENT

In this study, Portland cement (PC) was used as the binder, along with rice husk ash (RHA; 80μ m, Lingshou Chuangwei Mineral Products Processing Factory, China) and quarry dust (QD; 10μ m, Jinyuan Stone Co., Ltd., Hebei, China) and basalt macro-fibers (BMF; Shandong Federal Fiber Co., Ltd., China). Table 1 exhibits the chemical composition and physical properties of PC and RHA. In addition, QD (20 to 60%) and RHA (10 to 30%) were used as mineral additives in varying concentrations and dry blended with PC. Table 2 shows the seven mixtures that were created and prepared for this project. The water-to-cement ratio was kept constant in all mixtures at 0.4.

Table 1. Chemical composition and physical properties of used PC and RHA in this study

| Properties | PC | RHA |
|------------|-------|------|
| CaO (%) | 60.45 | 4.11 |

| Properties | PC | RHA |
|--|-------|--------|
| CaO (%) | 60.45 | 4.11 |
| SiO ₂ (%) | 19.72 | 78.52 |
| Al ₂ O ₃ | 2.14 | 5.47 |
| SO ₃ (%) | 2.09 | 1.10 |
| Mg(OH) ₂ (%) | 1.3 | 0.6 |
| Fe ₂ O ₃ | 0.8 | 1.75 |
| Na ₂ O (%) | 0.16 | 0.28 |
| Alkalies (%) | 0.06 | 0.74 |
| Cl ⁻ (%) | 0.011 | _ |
| Surface area (m ² /kg) | 310 | 19,000 |
| Specific gravity (kg/cm ³) | 3.01 | 1.98 |
| Insoluble residue (%) | 1.02 | _ |

Table 2. Mixture designs in this study.

| Sample name | Mixture designs |
|-------------|-------------------|
| PC0 | PC only (control) |
| QD20 | PC + QD (20%) |
| QD40 | PC + QD (40%) |
| QD60 | PC + QD (60%) |
| RHA10 | PC + RHA (10%) |
| RHA20 | PC + RHA (20%) |
| RHA30 | PC + RHA (30%) |

 Table 3. The chemical composition of steel

| С | Р | S | Ni | Mn | Si | Cr | Fe |
|------|------|------|-------|-----|------|-----|----------|
| 0.22 | 0.04 | 0.03 | 0.001 | 1.6 | 0.56 | 0.1 | residual |

The initial and final setting times of binders with PC at different concentrations were measured using a Vicat Needle in accordance with IS: 4031 (1999). The needle's penetration into the samples was monitored every 5 minutes, and the average values were recorded. To assess sample compressive strength, 20cm cube steel molds of all designed mixtures were prepared. For 24 hours, the demolded samples were kept in an airtight curing room at 27 °C with a relative humidity of not less than 95%. The chemical composition of steel rebar is shown in Table 3. The compressive strength was determined using IS: 4031 (1999). X-ray diffractometry (XRD, Bruker D8 Advanced difractometer, AXS, Karlsruhe, Germany) was used to study the hydration process of a sample blended with QD and RHA after 4 weeks of curing. For investigation of the change in structure of a hydrated sample, the field emission scanning electron microscopy (FE-SEM, S-4800, Hitachi, Japan) was used. The hydration behavior of Portland cement was studied using the electrochemical impedance spectroscopy (EIS) technique which contains the system with steel rebar as working electrode incorporated into the concrete samples, a Pt foil as counter electrode and a KCl saturated Ag/AgCl electrode as reference electrode. The EIS experiments were done in a frequency range between 10^{+4} Hz and 10^{-2} Hz in a 3.5 wt% NaCl solution.

3. RESULTS AND DISCUSSION

Figure 1 depicts the results of all PC samples' initial and final setting times. As can be seen, the addition of QD and RHA speeds up the setting. The control sample (PC0) achieves the setting in 359 minutes, mixed PC samples with QD (QD20, QD40, and QD60) achieve the setting in 354, 343, and 336 minutes, and mixed PC samples with RHA (RHA10, RHA20, and RHA30) achieve the setting in 304, 338, and 349 minutes, respectively.



Figure 1. The findings of initial and final setting times of all PC samples

Figure 1 also shows that increasing the content of quarry dust in the PC mixture accelerates the setting time, whereas RHA retards the setting time of cement, with higher replacement levels causing

even more retardation. The reason for this retardation is the negative effects of the reduction of superplasticizer dosage with the reduction in cement content, and the converse effect on silicate hydration which occurs with increasing the replacement levels for longer setting [32-34].

Figure 2 shows the average compressive strength of all PC samples. As seen from Figure 2, the compressive strengths increased with increasing curing times (up to one year). Figure 2a indicates that the hydration reactions begin with the addition of quarry dust. The main hydration products are ettringite and calcium silicate hydrate (C–S–H) that the ettringite crystals lead to high strengths [35]. Although, as compared to PC0 samples, QD blended cement samples (QD20, QD40 and QD60) exhibit a decrement in strength. It can be related to QD containing calcium carbonate which are often serves as a filler component within cement [36, 37]. The QD20 sample demonstrates a maximum strength of 55.1 MPa for 4 weeks which is approximately 8% lower than the PC0 sample (60.2 MPa).

The effect of RHA on the compressive strength of PC is displayed in Figure 2b. As seen, as RHA content in PC is increased from 10–20%, the compressive strength is increased toward the control sample. A maximum strength of 68.1 MPa is obtained for RHA20 sample after 4 weeks. It is approximately 11% higher than the control sample (60.2 MPa). The improvement of strength in RHA blended samples may be related to pozzolanic reactions of RHA [38, 39]. As observed, the strength is continuously increased with increasing the hydration period, and the remarkable increase in strength (74.5 MPa) is achieved at one year of hydration. This strength enhancement can be associated with the generation of C–S–H due to the high pozzolanic activity of RHA [40, 41].



Figure 2. Effect of (a) QD and (b) RHA on the compressive strength of PC samples

3.2. XRD and SEM analyses

Figure 3 depicts the XRD patterns of unhydrated cement, control cement sample (PC0), QD20 (20% QD blended cement) and RHA20 (20% RHA blended cement). XRD patterns of unhydrated cement in Figure 3a shows the most prominent diffraction peaks of tricalcium silicate (C₃S) at $2\theta = 29.38^{\circ}$, 32.65° , 34.35° , 41.29° , 51.68° , 56.58° , and the presence of dicalcium silicate (C₂S) due to diffraction peaks at $2\theta = 26.38^{\circ}$ and 32.19° [32, 42].



Figure 3. XRD patterns of (a) unhydrated cement, (b) control cement sample (PC0), (c) QD20 (20% QD blended cement) and (d) RHA20 (20% RHA blended cement).



Figure 4. FE-SEM images of (a) PC0 after 4 weeks hydration, (b) QD20 sample after 4 weeks of hydration, (c) QD60 after 4 weeks of sample.hydration, (d) 4 weeks of hydration of RHA20 sample and (e) one year of hydration of RHA20.

The XRD patterns of PC0 after 4 weeks of hydration process in Figure 3b indicate a decrease in the diffraction peak intensity of C₂S and C₃S because of formation of various crystalline phases such as calcium hydroxide (CH) with diffraction peaks at $2\theta = 18.12^{\circ}$ and 47.28° , and ettringite (E) with diffraction peaks at $2\theta = 28.58$, 34.05° and 50.68° [43, 44]. Figure 3c depicts the XRD patterns of QD20 after 4 weeks of hydration. It shows the additional diffraction peaks at $2\theta=29.07^{\circ}$ and 32.01° due to existence of the calcium carbonate (CaC) phase in cement, and diffraction peaks at $2\theta=23.18^{\circ}$ and 33.48° because of the formation of monocarboaluminate phase [43, 45].

The addition of QD can accelerate the production of ettringite as a stable hydration product in early ages in the presence of a sufficient supply of sulfates. When the sulfate source is insufficient and is not able to readily supply enough sulfate ions before the alumina content has completely hydrated, ettringite is converted to monosulphoaluminate [46-48]. RHA as a pozzolanic material is reported to contain a high percentage of silicon dioxide (SiO₂), cristobalite and sylvite [49, 50]. The XRD pattern of RHA20 after 4 weeks of hydration in Figure 3d displays the formation of ettringite (E) with a diffraction peak at 44.69°, presence of sylvite (S) and cristobalite (C) of RHA, and the existence of other crystalline phases of C_3S , C_2S and CH.

Figure 4 shows the FE-SEM images of PC0, RHA and QD blended cement samples. According to reports after hydration for 1 day nearly half the cement has reacted in the water-activated paste and hydration products include hydrated grains, the amorphous or poorly C-S-H, tobermorite (T) and calcium hydroxide (CH) in needle-like or short-acicular on cement [51, 52]. After one week, the dense cement microstructure could be formed with distinct needle crystals [51, 53]. At 4 weeks, Figure 4a shows that the continuous hydrated structure of PC0 is generated by outgrowth and interconnection of the reacted grains. Figures 4b and 4c exhibit the surface morphology of QD20 and QD60 samples after 4 weeks of hydration, respectively. These surface morphologies indicate needle and prismatic crystals of ettringite and euhedral-like C–S–H (T) crystals along with cubic calcium hydroxide (CH) crystals are formed which makes the samples denser. FE-SEM image of QD60 in Figure 4c illustrates that unused calcium carbonate (UCC) becomes aggregated on the surface and act as a filler. The FE-SEM image of QD20 in Figure 4b for sample with lower quarry content shows traces of calcium carbonate which demonstrates that a certain amount of QD could participate in hydration reaction and result in formation the monosulfate and carboaluminate [54, 55]. Moreover, the FE-SEM images of 4 weeks and one year of hydration of RHA20 samples are displayed in Figures 4d and 4e, respectively, which reveal the formation of several phases of calcium hydroxide (CH), tobermorite (T) and ettringie (E) on the surface of samples. The FE-SEM image of one year of hydration of the RHA20 sample also indicates the densification of the cement microstructure with the formation of secondary C-S-H gel which would delay strength [56]. RHA contains amorphous silica, which has a higher pozzolanic activity and reacts with calcium hydroxide [57, 58]. These results are in agreement with the XRD results.

3.3. Electrochemical analyses

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The Nyquist plots of steel rebar incorporated into the PC0 concrete (control sample) with various hydration periods and the corresponding equivalent circuit model used for fitting the EIS results are presented in Figures 5 and 6. The equivalent circuit model Figure 6a contains the classical Randles model with Rs as electrolyte resistance and Cdl as capacitance of the electric double layer. The EIS measurements are conducted on the Faraday impedance. Due to the electrochemical reactions, Faraday's procedure includes charge transfer procedure and ion diffusion procedure. The Faraday impedance can be demonstrated by the charge transfer impedance (Rct) and the diffusion procedure impedance (W) [59]. Figure 6b shows the equivalent circuit model for PC which contains the PC pore solution resistance (Rs), double-layer capacitance between liquid/solid phases (CPE1), resultant resistance for ion transfer procedure in the cement (R1), and the Warburg resistance (W1) due to the ion diffusion procedure in the cleater [60, 61]. Furthermore, R2 denotes the resistance generated by the charge-transfer procedure on the electrode surface, CPE2 denotes the double-layer capacitance between cement and electrode, and W2 denotes the resistance generated by the ion diffusion procedure on the electrode surface [62].



Figure 5. The Nyquist plots of PC samples with various hydration periods, and (b) Bode diagrams of the steel rebar incorporated into the PC0 concrete (control sample) at various hydration periods in 3.5 wt% NaCl solution.



Figure 6. The corresponded equivalent circuit model used for fitting the EIS results (a) classical Randles model and (b) equivalent circuit model for PC.

Figure 5b depicts the Bode diagrams of the steel electrode incorporated into the PC0 concrete (control sample) at various hydration periods. As seen in high frequency range, Bode |Z| diagrams exhibit a slope close to zero while the phase angle approaches 0 implying to the resistance of mortar (bulk material and pore solution) [63]. At intermediate frequencies, value of the phase angle in the Bode diagram is close to 70, reflecting to capacitive activities at the interface of steel rebar/concrete. For low frequencies, Bode diagrams display that the slope is decreased to 0° which can be related to the resistance of the charge-transfer procedure [64].

Figure 5a also depicts hydration in one day. The Nyquist curve shows a straight line and there does not appear any semicircle in the high-frequency range which competently is different from the presented classical Randles model in Figure 5a. Results indicate that the chemical reactions of the cement are very fast, and controlled by a diffusion process [65, 66]. After one week, the Nyquist plot shows an incomplete semicircle at a high frequency region that it is suggested that it attributed to the contribution of OH^- and SO_2^{-4} in hydration reactions and generation of hydrated phases [67]. For more hydration time, samples exhibit the classical Nyquist curve, and the reactions are controlled by kinetic and diffusion processes [68]. In long hydration periods, the water absorption contributes to the continuous hydration to the extent that it slightly enhances the charge-transfer resistance which leads to generating the capillary porosity network in PC0, and shifting the Nyquist curve toward the right. The fitting results of ZSimpWin are presented in Table 4. As observed, for the sample that was hydrated for one day, the low vale of Rct indicates a low hydration degree of PC0. In addition, the cement liquid phase hydration process resulted in the formation of a few hydration products and a few diffuse of these products in the PC sample [69]. In general, the cement hydration process resulted in more stable products than the granulation process according to FE-SEM and XRD results [70]. As seen, the value of Rct increases with increasing hydration time. It is proposed that as the hydration time increases, so does the amount of hydration products, resulting in an increase in chemically combined water. Hence, the free water is slowly decreased with time due to the water consumption by hydration reactions [71]. It causes an increase in the total mass of solids with time as hydration products [72, 73]. Since the bulk solid resistance is always much larger than that of the liquid phase in cement, the ion-transfer resistance is enhanced by hydration time. The initial reaction products are formed by the dissolution and precipitation, whereas, at later ages, a solid state mechanism is followed where the reaction takes place on the surface of the formed particles, dominated by the slow diffusion of the ionic species into the unreacted core [74]. As a result, the rate of hydration is faster in the early hydration period than later [75]. Moreover, a fast enhancement in Rct value indicates that hydrationreactions are strong during the early stages of hydration and can be accomplished with a high population of electron hydration [76].

| Sample | Rs(Ω) | Rct(kΩ) | CPE1 (μF. cm ⁻²) | n | $R_2(k\Omega)$ | CPE2 (μF. cm ⁻²) |
|---------|-------|---------|---------------------------------|------|----------------|---------------------------------|
| 1 day | 65 | 4.021 | 7.4 | 0.71 | 0.544 | 10.3 |
| 3 days | 55 | 4.711 | 5.5 | 0.73 | 1.067 | 9.2 |
| 1 week | 49 | 7.579 | 4.2 | 0.77 | 2.071 | 8.2 |
| 2 weeks | 59 | 12.301 | 2.7 | 0.81 | 2.821 | 6.0 |
| 4 weeks | 44 | 22.658 | 1.6 | 0.86 | 5.198 | 4.4 |

Table 4. The fitting results of ZSimpWin for obtained EIS data from Figure 5 for steel rebar incorporated into the PC0 concrete (control sample) at various hydration periods in 3.5 wt% NaCl solution.

The Nyquist curves of steel rebar incorporated into the PC0 concrete and PC0 blended with various additives (QD20, QD60 and RHA20) after 4-week hydration periods are depicted in Figure 7. The data in Table 5 is obtained from Nyquist curves in Figure 7 using the equivalent circuit model in Figure 2b. As seen, by the addition of QD and RHA to cement, the semicircle radius decreases at high frequency. It is possible that the water film wraps around the main active substance during the hydration process, reducing its activity [77]. Therefore, the QD and RHA do not cooperate in the hydration reactions, and the quantity of reacted cement is dropped. When the hydration reaction begins, the addition of QD and RHA leads to a decrease in the reactions. As a result of the significant loss of chemically bound water from hydrated cement and increased pore connectivity, the Rct value is reduced [78].



Figure 7. The Nyquist curves of steel rebar incorporated into the PC0 concrete and PC0 blended with various additives (QD20, QD60 and RHA20) after 4 weeks hydration periods in 3.5 wt% NaCl solution.

The compressive strength of RHA samples is higher than that of PC0 samples after a 4-week hydration period, which can be attributed to the fact that RHA does not participate in the initial hydration process and contributes only as a filler. Therefore, the cement has the main role in

microstructural evolution. Thus, after initial curing, QD and RHA particles are distributed into the cement paste, and then secondary hydrations begin. These reactions form a hard hydration gel film. RHA shows the maximum compressive strength and maximum Rct of the other mixture. Consequently, the RHA can effectively improve the Rct value and the compressive strength of blended samples [35].

Table 5. The fitting results of ZSimpWin for Nyquist curves from Figure 7 for steel rebar incorporated into the PC0 concrete and PC0 blended with various additives (QD20, QD60 and RHA20) after 4 weeks hydration periods in 3.5 wt% NaCl solution.

| Sample | Rs(Ω) | Rct(kΩ) | CPE1 (μF. cm ⁻²) | n | $R_2(k\Omega)$ | CPE2 (μF. cm ⁻²) |
|--------|-------|---------|---------------------------------|------|----------------|---------------------------------|
| PC0 | 44 | 22.658 | 1.6 | 0.86 | 5.198 | 4.4 |
| RHA20 | 58 | 12.022 | 2.7 | 0.80 | 2.102 | 5.9 |
| QD20 | 47 | 7.402 | 4.3 | 0.76 | 1.871 | 8.3 |
| QD60 | 54 | 3.621 | 5.6 | 0.73 | 0.977 | 9.4 |

4. CONCLUSION

The purpose of this study was to investigate the effects of RHA and QD as mineral additives on setting time, compressive strength, and hydration behavior of PC. As a control cement sample, the blending of both QD (20-60%) and RHA (10-30%) in PC accelerates the setting. QD addition reduced PC strength, with a maximum strength of 55.1 MPa achieved after 4 weeks of curing with 20% QD (QD20). The addition of RHA increased the strength, and a maximum strength of 68.1 MPa was obtained with a blended cement containing 20% RHA (RHA20). The formation of various crystalline phases of hydration products and their effects on the hydration behavior of blended cement were confirmed by XRD and SEM analyses. The electrochemical studies illustrated that the values of charge-transfer resistance increase with increasing hydration time. In addition, QD and RHA can affect the charge transfer impedance. Results showed after 4-week hydration time, QD and RHA additives can reduce value of charge transfer impedance and hydration degree of PC paste.

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