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Setting time and standard consistency of Portland cement binders blended with rice husk ash, calcium carbide and metakaolin admixtures

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Setting time and standard consistency of Portland cement binders blended with rice husk ash, calcium carbide and metakaolin admixtures

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Abstract

The use of pozzolanic materials to reduce carbon dioxide (CO₂) emissions and enhanced properties of mortar and concrete has received increased interests in the last decades. In this study, admixtures such as metakaolin (MK), rice husk ash (RHA) and calcium carbide waste (CCW) partially replaced Portland cement (PC) at 5, 10, 20 % in multiple combinations of aforementioned admixtures as binders and the effect of setting times (initial and final) and standard consistency on the binders were evaluated. The Department of Environment (DOE) approach for mix proportions was employed. The mixes are in six groups consisting of control PC, each admixture replaced PC at 5, 10 and 20 % replacement level in binary mixes, then two of the admixtures replaced PC at same replacement level in ternary and the three admixtures replaced PC in quaternary mixes respectively. Results indicated that RHA significantly influenced the standard consistency of the binders incorporating the three admixtures as compared with CCW and MK. The requirement of water in the binder increased with increase in percentage replacement level of RHA within the binder due to its porous nature. At 5 % replacement level, it was 9 % above control value, 21 % and 39 % at 10 and 20 % replacement level subsequently. Similarly, setting times at the aforementioned replacement levels are 27 % and 6 % above the control value for initial and final setting time. For 20 % replacement level, it was 24 % and 38 % above the control value for the initial and final setting time respectively. This is due to reduction in PC content leading to less content of Tricalcium silicate aluminate (C₃A) and amount of carbon content within the pozzolanic materials which retard the rate of reactions. By and large, the admixtures retard the setting times of quaternary binder, and increased its water of consistency which can be overcome by activation. However, they are useful in the construction of concrete structural elements or structures that requires longer time



for placement. Furthermore, it will be worthwhile to investigate same properties at the nanoscale since these have been established at the micro level.

Keywords: Portland cement, pozzolans, rice husk ash, calcium carbide waste, metakaolin, consistency, setting times

1 Introduction

The use of concrete is over centuries and has retained its popularity because of its properties. Setting time as one of the fresh properties of concrete is vital in concrete construction [1]. In concreting, this property (setting time) find application in its development in terms of transportation, placing, compacting and finishing. The placement of concrete in formwork depends on the setting time for it to harden [2, 3]. With the advent of new types of concrete such as high strength concrete (HSC), self-compacting concrete (SCC) and high performance concrete (HPC), application of Pozzolans is imperative so as improve their performance, achieve better engineering properties and most especially reduce cost and lower the carbon foot print. Mineral admixtures such as rice husk ash (RHA), calcium carbide waste (CCW), metakaolin (MK), fly-ash (FA) and others are applied to replace cement partially for more efficacy of blended concrete.

Since these mineral admixtures differ in chemical, mineralogical composition and particle characteristics, their effects on concrete properties including setting time characteristics is also likely to differ. It is not out of place to say that knowledge of the setting characteristics of an admixture is imperative as this will enhance the scheduling of stages in concrete construction operations vis-a-vis transporting, placing, compacting and finishing of concrete work. Such vital knowledge cannot be over emphasized in the choice for a retarder or an accelerator admixture.

The mixing of water with cement triggers-off hydration products formation. Through the rigid behavior of the matrix, its setting times is determined. The beginning of the hardening of mortar or concrete is termed the initial setting time, while its sufficient hardness is termed final setting time [3]. Previous work [4] on cementing material from calcium carbide residue-rice husk ash showed that the product of the mixture is pozzolanic. Also, similar work on activated CCW indicated that it has prolonged setting times compared with control values [5].

When calcium carbide (CaC_2) reacts with water, CCW and acetylene gas is obtained (equation 1).



It is a by-product from acetylene gas (C_2H_2) production [6]. Acetylene gas find application in ripening of fruits, a veritable choice for heating processes such as flame heating, flame gouging, welding, flame hardening, thermal spraying and other heating applications [7]. However, in Nigeria, acetylene gas finds application in oxy-acetylene gas welding. The carbide residue (CCW) is disposed as waste which is becoming a health hazard [8]. Also, CCW contains Ca(OH)_2 and is alkaline with pH greater than twelve [9]. To mitigate this negative effect on the environment, attempts were made to use CCW positively in both building and construction industry. When CCW is mixed with rice husk ash (RHA), pozzolanic reaction occurs and mortar produced from this mix has a compressive strength of 15 MPa at 28 days [10].

It has been shown that cement paste and concrete containing CCW at 1 % addition of CCW had initial and final setting times of 78 % and 57 % of control values, but increased in consistency by 14 % [11]. Also 5 % addition of CCW to PC mix, increased consistency but decreased the setting times [12]. From these results, CCW can influence properties of PC blended cement such as consistency and setting times. Consistency can be said to be the extent of wetness or dryness; an indication that a concrete sample is workable or not through the entire process of concreting operations (transportation, placement, finishing) without segregation. RHA mortar paste has higher consistency compared with control paste values. The consistency increases with increase in replacement level of PC with RHA [13-15]. This is not unconnected with the natural structure of RHA with large surface area [16]. The adsorption capacity, high fineness and specific surface area require more water [17, 18] which could be as high as 100 % [19]. The use of RHA of high fineness also leads to increased water demand as opined in earlier studies [20-22]. From the effect of RHA and RHA-CCW in concrete, the setting time of RHA mortar decreased compared with that for RHA-CCW mortar [23].

Rice husk ash (RHA) is a pozzolanic material which is produced by incineration of (RH) under controlled temperature (550- 800 °C), time and rate of burning. When it is incinerated under these conditions, highly reactive amorphous silica (SiO_2) is obtained. At incineration temperature above 700 °C, the amorphous SiO_2 changes to a crystalline SiO_2 (Cristoballite, Quartz and Tridymite) and below 500 °C, the product has high LOI which affects rate of reaction. In the aforementioned cases, the reactivity of RHA is greatly decreased. It has been shown to have low specific gravity [24], hence finds application in the making of light weight building materials. It

has been reported that when PC is replaced at 10 % level, optimum workability and strength is obtained [25]. Its usage improves concrete properties; mitigate global warming and waste disposal problems [26, 27]. In a study [28], the physico-chemical and hardened properties of blended cement binders containing RHA and (MK), there was an increase in water demand and setting times. MK is a highly reactive alumino silicate and alumina. These oxides combine with slake lime ($\text{Ca}(\text{OH})_2$) at ambient temperature with moisture to form compounds that were virtually identical to the compounds in hydrated Portland cement. MK is widely used for its highly pozzolanic properties and considered to have twice the reactivity of most other pozzolans and considered a very viable admixture. It is not like other SCMs like fly ash, silica fume which are by-products of an industrial process. When kaolin clay is calcined within a temperature range of 650-800 °C, metakaolin is obtained; a relatively new material in the concrete industry, effective in strength development and mitigates sulphate attack. The pozzolanic reactions changed the microstructure of concrete and chemistry of hydration products by consuming the released CH to form more calcium silicate hydrate (C-S-H), thus enhancing properties of mortar and concrete [29]. When PC is replaced with MK, up to 20 % increase in strength and good results in other variables were obtained when compared with control values. The use of MK as a supplement in concrete up to 20 % can reduce cost of cement and also mitigates negative environmental pollution due to cement production.

Results so far analyzed indicated prolonged consistency and setting times. Since pozzolans enhance both fresh and hardened properties and ensure the production of concrete at minimum cost, there has been increasing use of pozzolans both in the building and construction industry. Although CCW as a pozzolan improves strength of concrete, it prolongs setting due to its low pozzolanicity. However, (MK) though improves early strength development, lowers workability due to high heat of hydration which is detrimental to hardened properties of mortar and concrete. It is also weak in magnesium sulphate environment as well as at high temperatures. But when RHA replaced PC, it accelerates the early hydration of Tri-calcium silicate (C_3S) and increase in the early hydration rate of C_3S which is attributed to its high fineness [30]; but due to its porous nature, absorbs more water. Thus, extent of scope of application of binary blends of CCW, MK, RHA and the ternary blends of CCW-MK, and MK-RHA in building and construction industry may be limited. However, the multiple applications of these admixtures in the form of blended binders have the potential to compensate for the differences due to their synergistic interactions.

Hence, this study was set out to evaluate the fresh properties of blended binders containing RHA, CCW and MK with a view to determine its suitability as a retarder or accelerator in concrete production.

2 Materials and experimental investigation

2.1 Materials

PC, CCW, RHA and MK were used in the production of quaternary paste binders. Tests were carried out on the physical and chemical properties of PC, CCW, RHA and MK to determine their suitability (Table 1). The mix proportions of the binder were divided into six groups: Group 1a (100 % PC), Group 1b BM (PC + MK), Group 2 BM (PC + RHA), Group 3 BM (PC + CCW), Group 4 TM (PC + CCW+ MK), Group 5 QM (PC + CCW + RHA + MK) as indicated in Table 2. A local dealer of Dangote Cement Company, Nigeria provided the PC (CEM 1-42.5 N). RH was incinerated in a local furnace for 3 h at 600 °C, and cooled to room temperature in a desiccator and then ground using a local mill and sieved through a 75 µm sieve to obtain RHA. Kaolin was obtained from Madagali, Yobe state, Nigeria and prepared and calcined at 700 °C for 2 h at a rate of 10 °C/min at the Central Services Laboratory, National Cereals Research Institute, Badeggi, Niger State, Nigeria to obtain MK that was used for the study. It was then cooled to room temperature in a desiccator, ground with a local mill and sieved through a 75 µm sieve. CCW was obtained from a local panel beater at Katerin-Gwari, Minna, Nigeria. The CCW was air dried, sieved through a 75 µm sieve. Preliminary tests were carried out on the materials.

2.2 Preparation of samples

Six groups of paste samples were prepared using the admixtures RHA, CCW and MK which replaced PC at 5, 10 and 20 %. The constituents were thoroughly mixed on a glass sheet. The mixtures include 1 control sample of plain PC, 3 binary, 1 ternary and 1 quaternary mixture. PC was partially replaced by RHA, CCW, and MK at replacement levels of 0, 5, 10 and 20 %. Maximum replacement of PC by supplementary materials was 20 % by mass for all mixtures (Table 2).

2.3 Methods

For the determination of consistency and setting times of PC, PC-RHA, PC –CCW and PC-MK pastes, six groups of samples were tested. The normal consistency and setting time tests were conducted in accordance with BS EN 196-3 provisions [31]. The normal consistency, setting times (initial and final) of PC pastes were determined using Vicat apparatus in accordance with ASTM C 191 [32]. The water of consistency and setting times of the prepared pastes were directly determined from the relation in equation (2)

$$WC (\%) = L/W \times 100 \quad (2)$$

Where WC is consistency water, L is water required to produce a suitable paste and W is the mass of cement sample (300g).

The initial setting time is the time taken to reach the initial set, while the final setting time is the time taken to attain final set of paste. Using the Vicat apparatus, a needle of 1 mm square penetrated the paste at every 10 min interval till the index scale showed 5 + 0.5 mm from bottom of the mould. For the final setting time, the needle was replaced with an annular attachment. The needle was released every 30 min interval until it makes no visible indentation on the test specimen surface. For each mixed sample, three tests were carried out and the mean result was recorded.

3 Results and discussion

3.1 Physical and chemical properties of constituent materials

3.1.1 Physical Properties

Table 1 showed the physical and chemical properties of PC, CCW and MK respectively. The average specific gravity of RHA, CCW and MK showed that CCW with a value of 2.19 is very light and fine compared with other constituents like PC with a value of 3.15. RHA and MK are both lighter than PC with a value of 3.15. The specific surface area of CCW, RHA and MK are higher compared with PC value which means that they are much finer than PC particles and thus occupy more space than PC. Generally, the specific gravity of mineral admixture is less than that of PC therefore more volume is obtained when it replaces PC.

Table 1. Physical and Chemical Properties of Materials

| Physical Properties | Materials | | | |
|--|-----------|------------|------------|-----------|
| | PC (wt %) | RHA (wt %) | CCW (wt %) | MK (wt %) |
| Specific gravity (g/cm ³) | 3.16 | 2.25 | 2.19 | 2.52 |
| Blaine Fineness (cm ³ /g) | 3045 | - | - | 7483 |
| Loss on ignition (LOI) | 2.98 | 15.76 | 31.70 | 1.22 |
| Chemical composition | | | | |
| Calcium Oxide, CaO % | 64.19 | 1.30 | 93.69 | 0.39 |
| Silicon dioxide, SiO ₂ % | 19.57 | 73.60 | 6.49 | 54.30 |
| Aluminum Oxide, Al ₂ O ₃ % | 5.47 | 0.68 | 2.00 | 38.30 |
| Ferric Oxide, Fe ₂ O ₃ % | 3.24 | 0.93 | 1.87 | 4.28 |
| Magnesium Oxide, MgO % | 2.01 | 0.35 | 0.70 | 0.08 |
| Manganese Oxide, MnO % | 1.25 | - | 0.47 | - |
| Sodium Oxide, Na ₂ O % | 0.26 | 0.12 | 0.10 | 0.12 |
| Potassium Oxide, K ₂ O % | 0.43 | 2.37 | 0.18 | 0.50 |
| SO ₃ | 2.74 | - | 0.36 | 0.22 |

3.1.2 Chemical Properties

From Table 1, the sum of SiO₂ + Al₂O₃ + Fe₂O₃ (SAF) exceeds 70 % and thus met provisions for pozzolanic materials [33, 34]. Amount of SiO₂ and Al₂O₃ in RHA and MK depicts their reactive capability with the primary hydrate of cement. It is interesting to note that CCW with less SiO₂ and Al₂O₃ has the highest CaO content of 95.69 % ensuring that the Ca/Si ratio is maintained (kept at not less than pH 12) for reaction to continue.

3.2 Standard consistency and setting times

3.2.1 Standard Consistency of PC-CCW-RHA-MK

The result of standard consistency tests for the quaternary binder, PC-CCW-RHA-MK at different percentage replacement levels is shown in Figure 1. In a study on ternary binder with OPC-FA-SF at 20FA 10SF and 30FA 10 SF as replacement of OPC, it was shown that requirement of water increased with increase in percentage replacement level due to its fineness [35]. From this study, it was found that as CCW, RHA and MK replaced PC to make quaternary binder; the normal consistency became higher as replacement level increased. This can be attributed to the high fineness of RHA and MK compared with PC value. Their specific surface value is much higher compared with PC value (Table1). At 5 % replacement level of PC with CCW-RHA-MK binder, its water demand increased by 9 %, and 21 %, 39 % at 10 % and 20 % replacement levels, when compared to PC value (Figure 1). For the binary binders (PC-CCW,

PC-RHA, PC-MK), consistency is same with that of PC (control) value (Table 3) at 5 % and 10 % replacement levels but increased by about 5 % at 20 % replacement level except for PC-CCW binder whose consistency is same with that of PC at all replacement levels. This showed that CCW does not demand much water due to its low pozzolanicity compared with RHA and MK values. The increase in consistency for the quaternary binder is fruitful as it facilitates more reactions between constituents and the primary hydrate of PC.

Table 2. Mix proportion of the materials

| S/NO | Mix ID | Materials (gm) | | | |
|--------------|-----------------------|----------------|-----|-----|----|
| | | PC | RHA | CCW | MK |
| Group 1a | 100 % PC | 300 | - | - | - |
| Group 1b, BM | 95PC- 5MK | 285 | - | - | 15 |
| | 90PC- 10MK | 270 | - | - | 30 |
| | 80PC- 20MK | 240 | - | - | 60 |
| Group 2, BM | 95PC- 5RHA | 285 | 15 | - | - |
| | 90PC- 10RHA | 270 | 30 | - | - |
| | 80PC- 20RHA | 240 | 60 | - | - |
| Group 3, BM | 95PC- 5CCW | 285 | - | 15 | - |
| | 90PC- 10CCW | 270 | - | 30 | - |
| | 80PC- 20CCW | 240 | - | 60 | - |
| Group 4, TM | 95PC- 5CCW-5MK | 285 | - | 15 | 15 |
| | 90PC- 10CCW-10MK | 270 | - | 15 | 15 |
| | 80PC- 20CCW-20MK | 240 | - | 30 | 30 |
| Group 5, QM | 95PC-5CCW-5RHA-5MK | 255 | 15 | 15 | 15 |
| | 90PC-10CCW-10RHA-10MK | 210 | 30 | 30 | 30 |
| | 80PC-20CCW-20RHA-20MK | 120 | 60 | 60 | 60 |

BM, binary mix; TM, ternary mix; QM, quaternary mix; PC, Portland cement

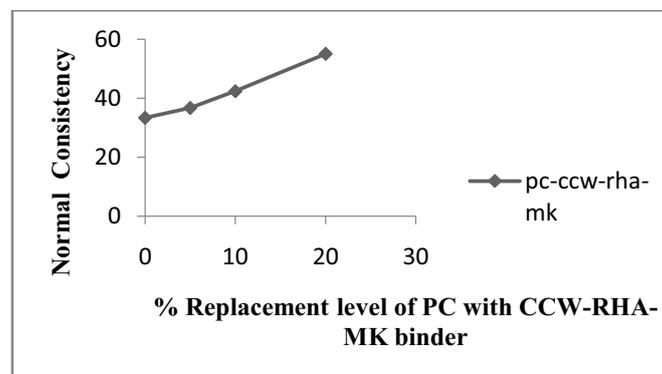


Figure 1. Consistency of PC-CCW-RHA-MK

3.2.2 The initial and final setting times of PC-CCW-RHA-MK binders

For quaternary binders containing PC, CCW, RHA and MK, the setting times, initial (IS) and final (FS) are shown in Figures 2 and 3. It can be observed that the effect of the admixtures prolonged the setting times of the quaternary binders when compared with control value. The observed retardation in setting times is mainly due to the effect of lower cement content. It can also be observed that the setting times for binary and ternary binders increased as percentage replacement level of PC with the admixtures increased. This shows that the admixtures used in this study retards the setting times of binary, ternary and quaternary binders [36, 37]. If quaternary binder (PC-CCW-RHA-MK) is compared with ternary binder (PC-CCW-MK), (Table 3 and Figure 3) at 5 % replacement level, IS decreased by 27 %, at 10 % replacement level, increased by 8 % and then decreased again at 20 % replacement level by 24 %.

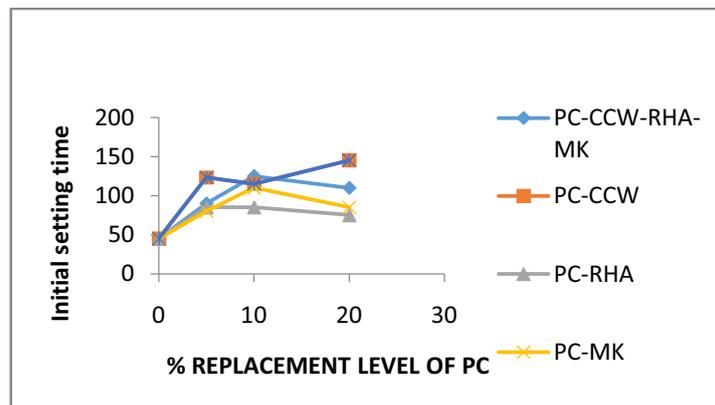


Figure 2. Initial setting times of binders

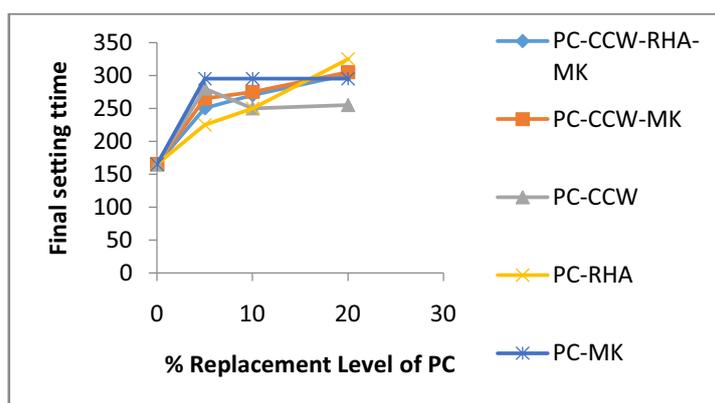


Figure 3. Final setting times of binders

Table 3. Consistency, initial and final setting time results

| S/NO | Mix ID | PC (g) | Materials | | | Std Consistency | IS (min) | FS (min) |
|--------------|------------------------|--------|-----------|-----|----|-----------------|----------|----------|
| | | | RHA | CCW | MK | | | |
| Group 1a | 100 % PC | 300 | - | - | - | 33.33 | 45 | 165 |
| Group 1b, BM | 95PC- 5MK | 285 | - | - | 15 | 33.00 | 80 | 295 |
| | 90PC- 10MK | 270 | - | - | 30 | 34.00 | 110 | 295 |
| | 80PC- 20MK | 240 | - | - | 60 | 34.33 | 85 | 295 |
| Group 2, BM | 95PC- 5RHA | 285 | 15 | - | - | 32.67 | 85 | 225 |
| | 90PC- 10RHA | 270 | 30 | - | - | 33.67 | 85 | 250 |
| | 80PC- 20RHA | 240 | 60 | - | - | 35.00 | 75 | 325 |
| Group 3, BM | 95PC- 5CCW | 285 | - | 15 | - | 31.67 | 105 | 280 |
| | 90PC- 10CCW | 270 | - | 30 | - | 32.00 | 60 | 250 |
| | 80PC- 20CCW | 240 | - | 60 | - | 32.33 | 70 | 255 |
| Group 4, TM | 95PC- 5CCW- 5MK | 285 | - | 15 | 15 | 33.00 | 123 | 265 |
| | 90PC- 10CCW- 10MK | 270 | - | 15 | 15 | 34.67 | 115 | 275 |
| | 80PC- 20CCW- 20MK | 240 | - | 30 | 30 | 39.00 | 145 | 305 |
| Group 5, QM | 95PC-5CCW- 5RHA-5MK | 255 | 15 | 15 | 15 | 36.67 | 90 | 250 |
| | 90PC-10CCW- 10RHA-10MK | 210 | 30 | 30 | 30 | 42.33 | 125 | 270 |
| | 80PC-20CCW- 20RHA-20MK | 120 | 60 | 60 | 60 | 55.00 | 110 | 301 |

This effect of RHA on IS depict disorder; but on FS, at 5 % level of replacement, it decreased by 6 %; at 10 % level by 2 % and by 1 % at 20 % replacement level which showed that RHA progressively decreased FS of binders studied. This can be attributed to pozzolanic reaction of RHA at later stage. Similarly, when PC-CCW-MK is compared with PC-CCW, at 5 % level of replacement, decreased FS by 5 %, at 10 % level of replacement decreased by 1 % and then increased by 26 %. The effect of MK decreased FS by 5 % and 1 % at 5 and 10 % replacement levels but increased it by 26 % at 20 % replacement level. Furthermore, comparison between PC-CCW-MK with PC-MK revealed that CCW prolonged IS by 35 %, 4 % and 41 % but decreased FS by 10 %, 7 % and 3 % at 5, 10 and 20 % replacement levels respectively. These results

showed that as % replacement level increased, IS is increased and then reduced FS which implies that reaction becomes faster at later stage of hydration.

4 Conclusion

From the analysis and discussion, results showed that the effect of the admixtures on standard consistency and setting times of the binders differs. The following conclusions are hereby drawn from the study:

- i. At 5 % replacement of PC with RHA, CCW, MK, influence on consistency is limited (< 9 %) but becomes significant (21 %) at 10 % and (39%) at 20 % replacement levels which are still within standard limits.
- ii. Standard consistency in quaternary binder (PC-CCW-RHA-MK) increased with increase in replacement level of PC with RHA due to high demand for water and its high specific surface area. This suggests that if percentage level of RHA is checked in binder, standard consistency requirements and water issues can be addressed.
- iii. Replacement of PC by CCW, RHA, MK at 5 % level has negligible influence on consistency but at 20 % replacement level increased consistency which is at limits and similar to that of PC. This suggests that absorption and adsorption properties of CCW and MK are similar to that of PC.
- iv. When ternary (Group 4) binder, PC-CCW-MK is compared with quaternary, PC-CCW-RHA-MK, with the addition of RHA, setting properties increased, suggesting that increase of pozzolanic materials increase hydration process leading to increase in time period. Delayed setting times is advantageous in that concrete can be worked for longer periods resulting in time delays in transport and between mixing and using concrete becomes less critical. This type of binder finds application in reduction of cold joints in larger concrete pours.
- v. Setting time of quaternary binder increases at lower replacement level (5 %) of RHA but decreases at higher replacement level (20%) of RHA in the binder. This is due to the high specific surface area of RHA and MK which facilitates pozzolanic reaction with $\text{Ca}(\text{OH})_2$ to speed up the hydration process.
- vi. Retardation of setting time is the overall effect of CCW, RHA and MK on standard consistency and setting times (initial and final) of the quaternary binder. Since there is

less tri-calcium-aluminate (C_3A) due to lower cement content, the influence of increasing levels of RHA is to provide greater retardation in the setting time. This type of binder (PC-CCW-RHA-MK) can be used in the manufacture of special types of concrete such as reinforced marine concrete (RMC) and also in construction works in marine areas with RMC where longer period is required for its placing.

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