



Effect of Magnesium Sulphate on Calcined Clay Brick Powder Cement Replaced Mortar

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ABSTRACT

*This paper determines the resistance to Magnesium sulphate ($MgSO_4$) of Mortar produced with calcined clay brick powder (CCBP) as partial cement replacement at 0 %, 5 %, 10 %, 15 %, and 20 %. A total of ninety (90) 160*40*40 Prisms were cast and cured in water for 7, 14, and 28 days. 45 prisms were transferred into 0.5 M $MgSO_4$ solution after these days and compressive strength test conducted after 28 days in the solution, the remaining 45 prisms were immediately subjected to the test on these days as control. The slump test results indicated that the workability decreases as the percentage addition of CCBP content increases. The cement paste consistency test and the initial and final cement setting times increases with an increase in CCBP content. The compressive strength results of the CCBP-mortar showed an increase in the strength with curing age and a decrease with CCBP content (%) addition. Compressive strength development of samples cured for 7 days in water continued under the $MgSO_4$ solution while a reduction in compressive strength was observed for samples cured in water for 14 and 28 days, moreover, compared with control, samples containing CCBP possess higher resistance to the $MgSO_4$ solution because the control samples showed more deteriorations after exposure. It was found that 5% CCBP is the optimum as it has the highest compressive strength and showed the highest resistance to the $MgSO_4$ solution. It was concluded that powdered calcined clay brick could provide a valuable source of mineral replacement for cement for increased mortar durability under an aggressive environment.*

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INTRODUCTION

Sulphate attack is one of the most aggressive environmental factors that affect the long term durability of mortar and concrete structures. It can result in cracking, expansion, and deterioration of cement products such as mortars and concrete (Ding *et al.*, 2002). Sulphate attack in cement mortars and concrete made with ordinary Portland cement is the reaction of sulphate ions with calcium hydroxide and calcium aluminate to form ettringite and gypsum, these products are voluminous and lead to expansion,

cracking, deterioration, and deformation of concrete structures when formed after it has hardened (Charlotte and Taylor, 2001).

Sulphates present internally in concrete and mortars due to the use of sulphate rich aggregates, cement containing excess gypsum and sulphate bearing water, can also cause deteriorations; however, appropriate screening of ingredients and test procedures before cement mortar or concrete production can avoid such internal sulphate attack of the different



sulphates such as sodium, magnesium, calcium, and ammonium sulphate that cause deterioration in cement products, the first two are the most common ones, with the magnesium sulphates causing more destructions than any other (Cohen and Benter, 1988). However, the resistance of the blend to deteriorations depends greatly on the chemistry of the pozzolan used and its replacement level. In particular, the ability of the pozzolan to reduce the permeability of the matrix is essential to improving the resistance to an aggressive environment. The mechanism of attack of Portland cement binders by the sodium and magnesium sulphates is primarily due to two reasons: (1) the formation of gypsum due to the reaction of sulphates with the calcium hydroxide produced from cement hydration, and (2) the formation of ettringite due to reaction between the gypsum and monosulphates or calcium aluminate hydrate (Cohen and Benter, 1988).

The sulphate resistance of Portland cement mortars can be provided by using Portland cement that has low in their C_3A content, such as ASTM C150 Type II cement (moderate sulphate resistance) or Type V cement (high sulphate resistance). In situations in which such sulphate resistance Portland cement are not readily available, ordinary Portland cement can be used along with pozzolans such as fly ash, silica fumes, slag, or metakaolin at appropriate cement replacement levels to minimize deteriorations due to aggressive environmental factors (Hooton *et al.*, 1999). Because the effect of sulphate attack is the weakening of the cement paste, thereby leading to premature failure of mortars and concrete, the sulphate resistance of Portland cement mortars is an important durability consideration. Aliyu *et al.*, (2020) carried out a study on the effect of sulphuric acid on the compressive strength of concrete with quarry dust as partial replacement of fine aggregate and the result of the compressive strength of Quarry Dust-

concrete indicated that the compressive strength increased with the curing age and also increased with the addition of Quarry Dust. On the other hand, the resistance of the concrete to acid increases as the percentage addition of Quarry Dust increases. Groundnut shell ash (GSA) addition of up to 4 % increased the compressive strength and resistance of concrete exposed to a sulphuric acid environment (Wazumtu and Ogork, 2015).

The use of clay brick powder as a cement addition improved mortar compressive strength (Aliabdo *et al.*, 2014). The alternative waste materials such as recycled clay brick powder (CBP) hold great promise for improving the properties of Magnesium oxychloride cement (MOC) cost effectively because CBP is crushed from recycled clay bricks, and a typical pozzolanic material, and has similar properties to grade II fly ash (Cheng, 2016). Replacing cement with recycled clay-brick-powder (RCBP) could reduce CO_2 emission, enhance conservation of natural resources, and decrease the cost of waste disposal sites, however, the recycled clay brick powder could be used as partial replacement of cement in mortar without reducing its properties (Zheng *et al.*, 2011). Crushed clay brick powder (CCBP) improves the cement paste structure. It reduces the volumes and numbers of pores, and the paste sample containing 25 % CCBP has the smallest pore size and the best pore structure (Aliabdo *et al.*, 2014). Chen *et al.*, (2016) carried out a study on effect of agriculture and construction wastes on the properties of magnesium oxychloride cement mortar with Tourmaline powder, the result showed that the incorporation of CBP in Magnesium Oxychloride Cement (MOC) mortar improved the water resistance, but reduced the negative ion-including ability, due to the denser structure induced by the pore-filling effect and pozzolanic reaction.

Hence, the main aim of this paper is to investigate the effect of Magnesium sulphate on some properties of calcined



clay brick powder (CCBP) - mortar (i.e. compressive strength and flow (workability)).

MATERIALS AND METHODS

Materials

The materials used for this study include:

Cement: The Ordinary Portland Cement used is that of Dangote 3X brand and obtained from Samaru market in Zaria, Kaduna State.

Fine aggregate: The fine aggregate used was river sand obtained from a nearby river Samaru, Zaria, Kaduna State, Nigeria with a specific gravity of 2.57 and a bulk density of 1632 kg/m³. The sieve analysis test conducted in accordance with BS 882, Part 2.

Calcined clay brick powder (CCBP): A recycled brick obtained from demolished construction and remnants in a production plant around Zaria. The bricks were ground manually to powdered form and passed through sieve number 75µmm to obtain the required CCBP.

Water: Clean tap water fit for drinking was used for the preparation of both the cement mortar and magnesium sulphate solution as specified by BS3148 (1980)

Preparation of Magnesium Sulphate Solution

From the equation, concentration (C) = $\frac{M_o}{V_t}$
 $C = \frac{n}{v} \Rightarrow n = CV \quad (1)$

For a 0.5 molar concentration of magnesium sulphate solution,

n = 0.5mol x 15 litres

7.5 moles of MgSO₄ is required

1 mole of MgSO₄ \longrightarrow 120.3g

7.5 mole \longrightarrow x

X = 902.25 g of magnesium sulphate salt was diluted in 15 litres of water to have magnesium sulphate solution of 0.5 M.

Specimen Preparation and Curing

The preparation of mortar was conducted per BS EN 196-1, a prescribed mortar mix of 1:3 and water-cement ratio 0.5 were used. A total of ninety (90) prisms were cast for compressive strength. The moulds were oiled before casting and clamped to the vice, casting of mortars

Methods

Test on fine aggregates

The tests conducted on the fine aggregates were the specific gravity BS 812-1995, and sieve analysis (BS 812-103.2) analysis,

Test on CCBP-cement paste

Standard Consistency Test

This test was done to determine the quantity of water required to produce a CCBP-cement paste of standard consistency based on BS EN 197-1:2009 standard.

Initial and Final Setting Times Test

The test was conducted to evaluate the initial setting time and final setting time of CCBP-cement paste following BS EN 196 part 3:1995.

Soundness

This soundness of CCBP-cement paste was measured in accordance with BS EN 196-3: 1995.

Flow (Slump) Test

The ease at which the fresh mortar flow was assessed using the flow or slump per BS EN 12350-2:2009 standard.

were done in three layers, and each layer has given 25 blows of tamping rod. The specimens were left in the laboratory covered at room temperature for 24 hours then were de-moulded and transferred to the curing tank for curing.



Compressive Strength Test

The compressive strength test was conducted on hardened mortar samples following BS EN 12390, Part 3 specifications. A total of ninety (90) prisms were cast and cured in water. Forty-five (45) cubes were tested for compressive strength after 7, 14, and 28 days curing. The remaining forty-five (45) samples were transferred to twenty-five (25) liters of $MgSO_4$ solution with a molar concentration of 0.5 M. These samples were then tested after 28 days in $MgSO_4$ solution.

RESULTS AND DISCUSSION

Oxide Composition Test

Table 1 presents the chemical (Oxide) composition of the cement and the CCBP, the cement oxides satisfy the requirements of BS EN 197, Part 1. The CCBP with a sum of SiO_2 , Al_2O_3 , and Fe_2O_3 as 91.77 % also met the minimum target of 60 % and 70 % for BS and ASTM C618 respectively and hence could be used as a pozzolan. These oxides are considered to be the main components of pozzolans because they react with $Ca(OH)_2$ during OPC hydration of blended cement and/or lime to form cementitious materials (Taylor, 1997).

Table 1: Chemical composition of OPC and Calcined Clay Brick Powder (CCBP)

| Oxides | OPC | CCBP |
|-------------------------|-------|-------|
| SiO_2 | 13.84 | 58.94 |
| Al_2O_3 | 3.56 | 20.82 |
| Fe_2O_3 | 3.73 | 12.01 |
| SO_3 | 2.40 | 1.01 |
| MgO | 0.1 | 1.52 |
| Cl | 0.14 | 0.08 |
| K_2O | 0.45 | 1.26 |
| CaO | 73.95 | 1.62 |
| $SiO_2+Al_2O_3+Fe_2O_3$ | 21.13 | 91.77 |

Source: Laboratory work

Particles Size Distribution

The particle size distribution of fine aggregate shown in figure 1, the results indicated that the fine aggregate

used was well-graded and falls into zone 2 based on BS 882, Part 2, grading limits for fine aggregate and hence good for cement mortar

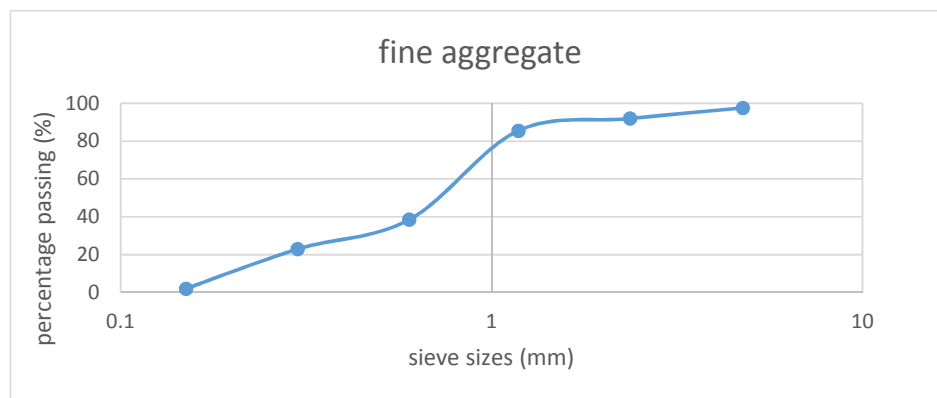


Figure 1: Sieve analysis for fine Aggregates



CCBP-Cement Consistency

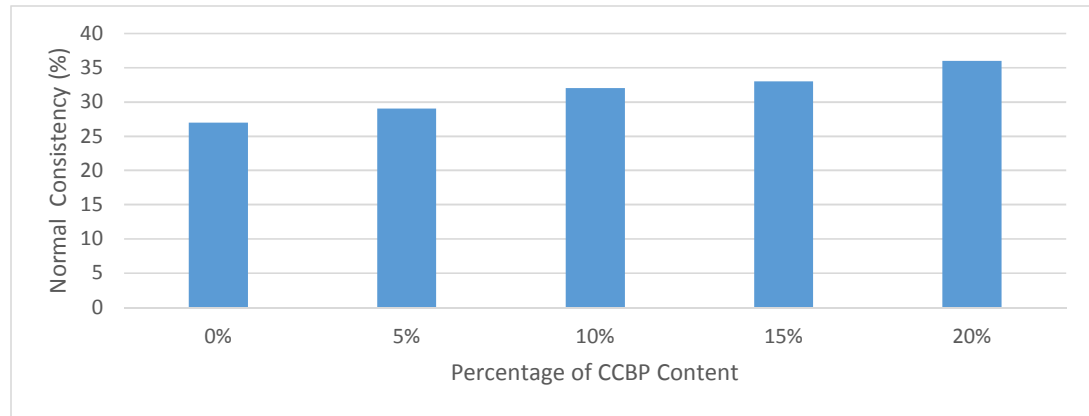


Figure 2: Normal Consistency versus Percentage of CCBP Content

The result of the normal consistency of CCBP-cement paste is presented in figure 2. It was observed that the normal consistency of CCBP-cement increased as the percentage replacement of CCBP content increased, this means with CCBP desired plasticity can be achieved with increased water content. The same behaviour was observed by Wazumtu and Ogork, (2015) and Sulaiman *et al.*, (2020), also the more water required might be to wet the increased surface area of particles (Marthong, 2012).

CCBP-Cement Paste Soundness

Figure 3 showed the soundness of CCBP-Cement paste. It can be seen that the cement soundness decreased from 0% to 10% CCBP content and then increased. However, the values of soundness obtained at each replacement levels fall within the range of the acceptable limit specified by BS EN 196-3 (1995). Which stated that cement is sound only if the soundness value falls within the range of 0-10 mm and unsound above 10 mm.

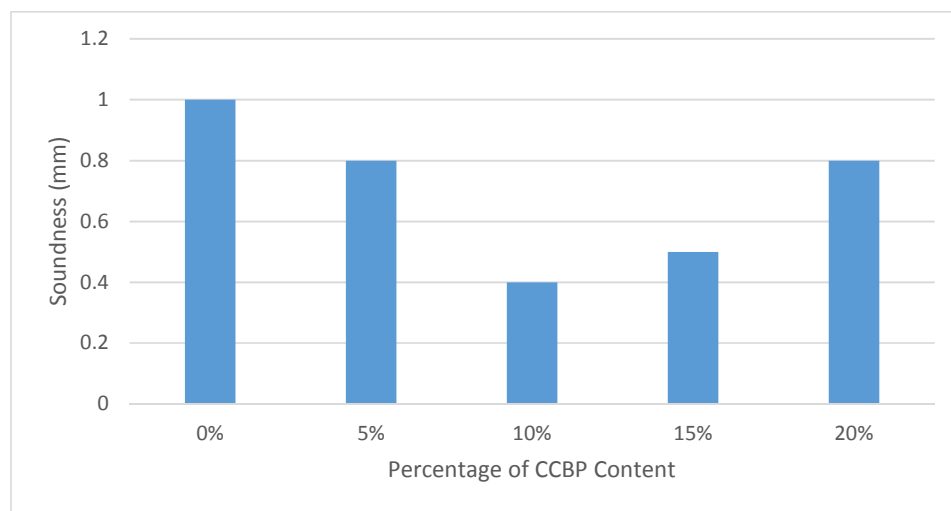


Figure 3: Soundness of CCBP-Cement

CCBP-Cement Initial and Final Setting Times

The results of setting times presented in Figure 4 revealed that the result of setting times of CCBP-cement paste increased with the increment of CCBP content. This may be a result of the potassium oxide (K_2O) presents in CCBP that obstructs the total formation of lime

and induces setting anomalies effect on setting as explained by Sulaiman *et al.*, (2020). It may also be a result of the formation of magnesium silicate ($MgSiO_3$) in the cement paste which was cognised to be a retarder thereby creating a time lag in the setting time of cement (Wazumtu and Ogork, 2015).

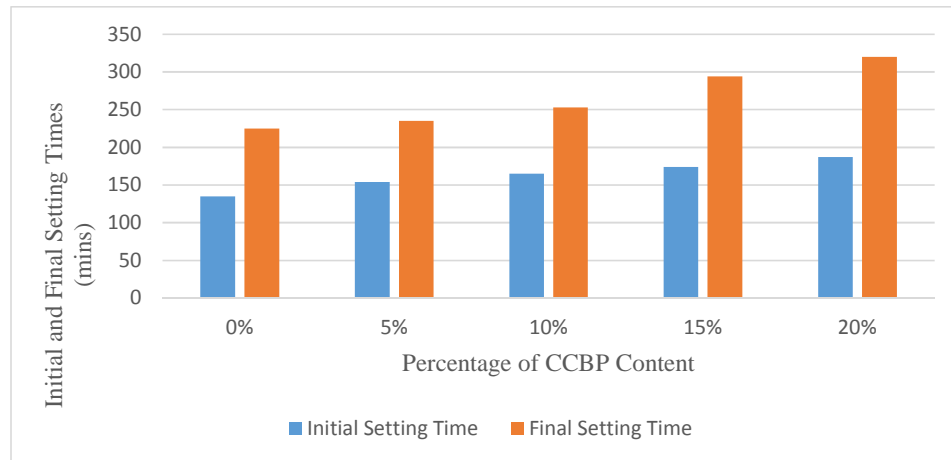


Figure 4: Setting Time of CCBP-Cement paste

Flow (Slump) Test

The results of the (flow) slump test conducted on 1:3 cement/sand mortar with different percentages of calcined clay brick powder (CCBP) are presented in figure 5. It has been observed

that as the percentage of CCBP increases the slump value decreases, the same pattern was observed by Aliyu *et al.*, (2020). The decrease may be due to the high specific surface of CCBP for constant water content.

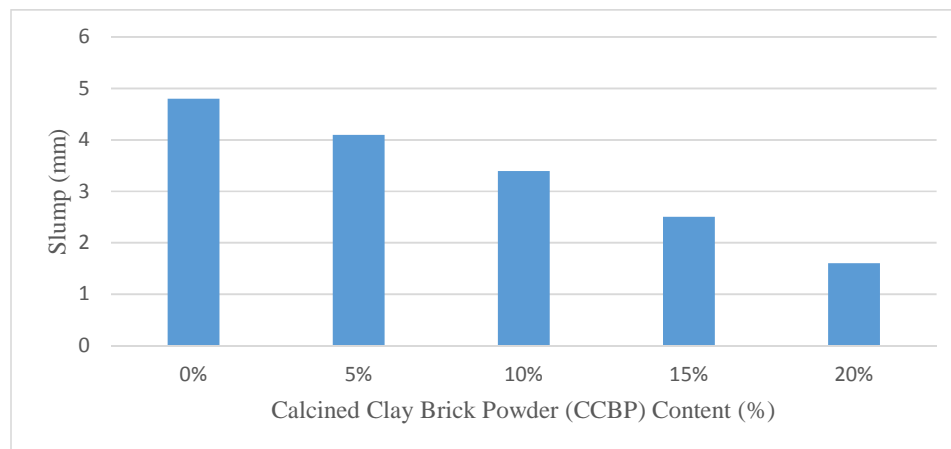


Figure 5: Slump Value against the percentage of CCBP content.

Compressive Strength of CCBP

The results of the compressive strength of CCBP-mortar in Figure 6 indicated that the compressive strength increased with curing age but decreased with the addition of CCBP content. The increase in compressive strength with curing age is as a result of continued

cement hydration, while the reduction in compressive strength with the addition of CCBP content might be because of a limited pozzolanic activity of the CCBP leaving unreactive CCBP within the mortar matrix be due to saturation of the cement similar to observation by Rao *et al.*, (2015)

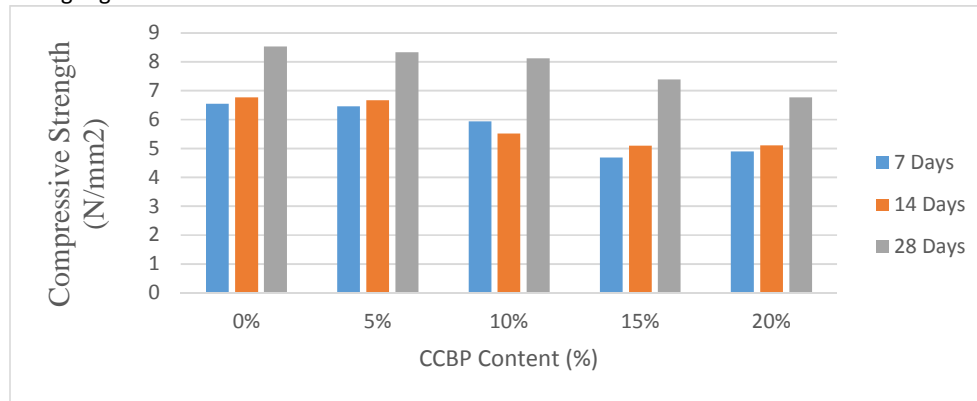


Figure 6: Compressive strength versus % Addition of CCBP Content

Effect of Magnesium Sulphate Solution on the Compressive strength CCBP-Mortar. Compressive strength at 7 days water curing and after 28 days in MgSO₄ solution

Figure 7 showed the compressive strength results of the mortar at 7 days water curing, and after the same specimens cured in water for 7 days were transferred to MgSO₄ solution for 28 days. The samples cured in water for 7 days showed more resistance to the sulphate attack because strength development continues even after the immersion into the solution, with 5 % CCBP replacement recording the highest strength after the 28 days in MgSO₄ solution. The strength increase maybe because after 7 days

water curing, the hydration of the cement is incomplete which continued but at a slower rate under the MgSO₄ solution, the compressive strength of the control remained relatively unchanged even after curing with MgSO₄ solution, this may be because the sulphates attack the hydration product of Tri-calcium aluminate (C₃A) phase of Portland cement (Slag Cement Association, 2002) and the control samples containing a higher quantity of C₃A hydrates at a higher rate compared to the samples containing CCBP and also because the samples containing ground brick are less porous making it harder for sulphate ions to penetrate the mortar and attack the hydration process.

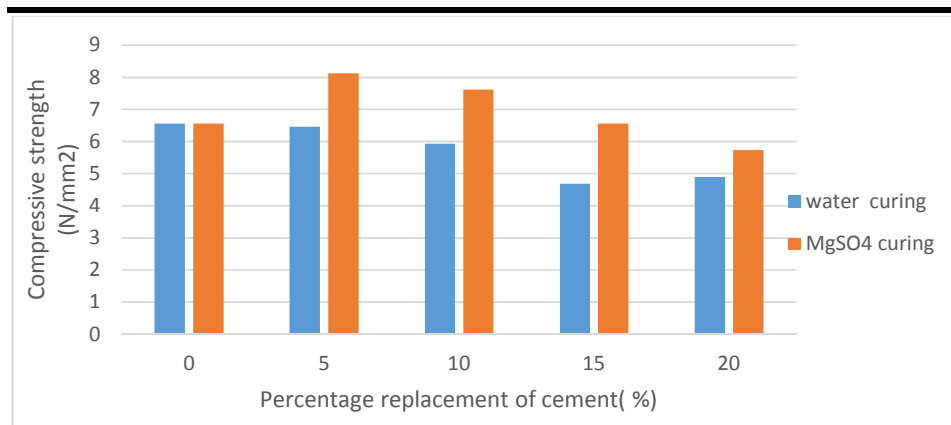


Figure 7: Compressive strength at 7 days water curing and after 28 days in MgSO₄ solution

Compressive strength at 14 and 28 days water curing and after 28 days in MgSO₄ solution

The compressive strength results of the mortar after 14 and 28 days water curing, and later 28 days in MgSO₄ solution is shown in figures 8 and 9. A decline in the compressive was recorded when transferred to the MgSO₄ solution after the specimens were cured in water for 14 and 28 days. The decrease is because a reaction takes place between the sulphates and the products of cement hydration leading to the decay of the hydrated product (shehata *et al.*, 2008 and Wee *et al.*, 2000), it may also be because most of the cement hydration had taken place after 14 days in water and the sulphate attack dominates the process. It was also observed that the control (0 % CCBP replacement) recorded 26.14 % (highest) decline in compressive strength,

compared with 5 %, 15 %, and 20 % which respectively recorded 7.8 %, 0.0 %, and 8.2 % (figure 8) also from figure 9, the control mix recorded the highest decrease after insertion in the MgSO₄ solution, this showed that adding mineral admixtures improves the resistance of the cement-mortar to the sulphate attack in agreement with Wee *et al.*, (2000); the increased resistance might be because CCBP improves the Calcium Silica Hydrate (CSH) content which improved the mortar pore structure and makes the product more impermeable than the control specimen, this concurs with Elinwa and Abdulkadir (2011) who attributed it to a reduction in the free lime content through pozzolanic reaction with the free lime in cement paste which reduces the lime content which reacts with chemicals causing cement-mortar deterioration.

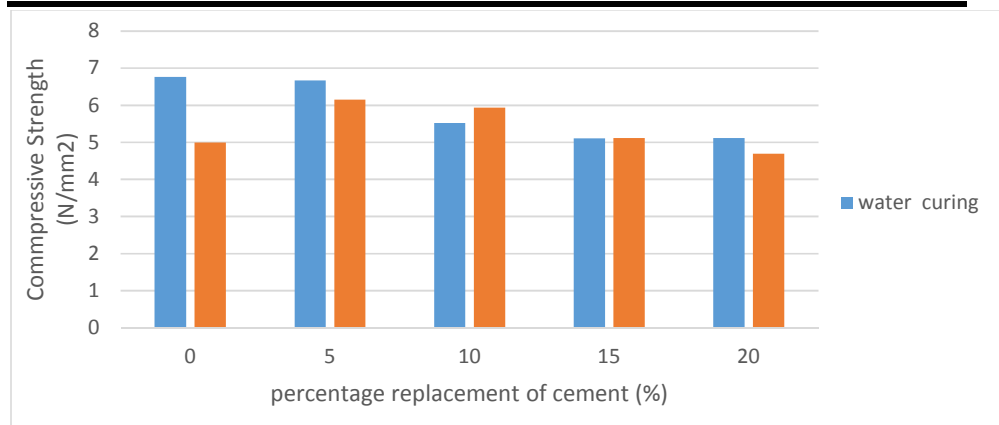


Figure 8: Compressive strength at 14 days water curing and after 28 days in MgSO₄ solution

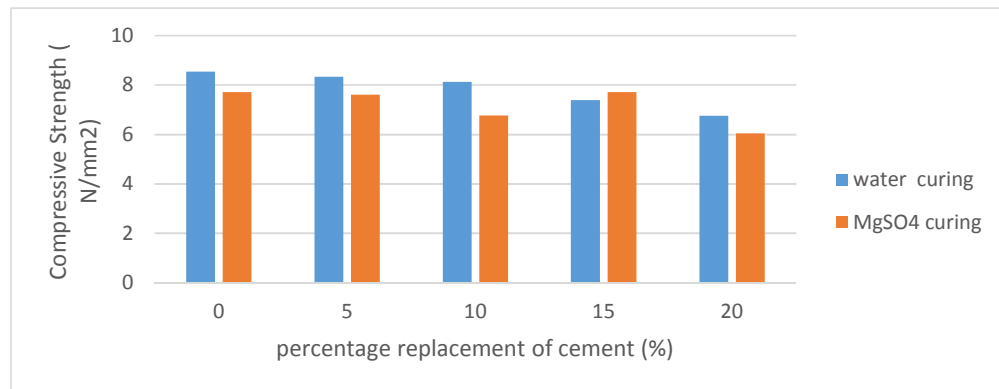


Figure 9: Compressive strength at 28 days water curing and after 28 days in MgSO₄ solution

CONCLUSIONS:

At the end of the study, it was concluded that

- 1) The CCBP having 91.77 % as a net sum of SiO₂, Al₂O₃, and Fe₂O₃ satisfies the minimum oxide composition of 70 % by ASTM C618 (2008)
- 2) The addition of CCBP decreases the flow (slump) of mortar but increases the setting times and the standard consistency of CCBP-cement paste.
- 3) The addition of CCBP reduces the compressive strength of the mortar but does not change the trend of compressive development with age.
- 4) Compressive strength development of samples cured for 7 days in water continued under the MgSO₄ solution and samples cured in water for 14 and 28 days recorded a strength decrease.
- 5) Mortar specimens containing CCBP possess higher resistance (durability) to the MgSO₄ solution when compared with control specimens
- 6) The best results were between 5-15 % CCBP content and the optimum CCBP content is 5%
- 7) Powdered calcined clay brick provides a valuable source of mineral replacement for cement for increased mortar durability under an aggressive environment.



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