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Pyroprocessing and the optimum mix ratio of rice husks, broken bricks and spent bleaching earth to make pozzolanic cement

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ABSTRACT

This paper presents the findings of an experimental investigation on optimizing pozzolanic activity of a blend of Rice Husks (RH), Spent Bleaching Earth (SBE) and Broken Bricks (BB) to form pozzolana that would have pozzolanic activity comparable to natural pozzolanas. Four ratios of RH, BB, and SBE were burnt in the Fixed Bed Kiln (FBK). The starting ratio had 20 kg of RH, 0 kg of BB and 4 kg of SBE. The amount of BB was increased by 2 kg each to a maximum of 6 kg as the mass of SBE was kept constant. The resultant ashes were subjected to various pozzolanic tests. This included; saturated lime test and compressive strength analysis. It was observed that the calcined blend with 10: 1: 2 mix of RH: BB: SBE exhibited the highest pozzolanic activity. This sample was mixed with acetylene lime sludge (ALS) in the ratio of 2:1 pozzolana: ALS. The compressive strengths for these cements were tested at 2 and 28 days of curing. The compressive strengths of this cement met the required EN standards for Portland pozzolana cement.

1. Introduction

Convectional building materials in most developing countries are unaffordable to a majority of the population. In addition to the escalation in the cost of building materials, there are rising environmental concerns due to the extensive exploitation of natural resources related to general construction and other housing development activities. These urge the search for alternative technological options. The most commonly used type of cement in Kenya is Portland cement. This is made by heating calcareous and argillaceous materials in a rotary kiln at temperatures in excess of 1300 °C (Marangu et al., 2018). In Kenya, Portland cement is expensive to a majority of the population. This is due to the rising cost of production especially the high energy demands associated with the making of clinker, where the temperatures are in excess of 1300 °C (Wachira et al., 2019). Cement manufacturers have been innovating new strategies to reduce the cost of cement worldwide. This has been done by mainly shifting from the wet process of manufacturing cement to the dry process. Other strategies involve partial replacement of cement by natural pozzolanas and making of pozzolana-lime binders blended with a small amount of OPC (Middendorf et al., 2005). There are now a wide variety of blended cements available (Marangu et al., 2014). The inorganic materials that are used to reduce cement quantities can be blended and/or ground intimately with clinker and/or cement during manufacture, or blended while preparing the concrete or mortar. The most commonly used minerals are fly ash, granulated slag, micro silica (silica fume) and various natural and calcined pozzolanas (Khan et al., 2014). In Kenya, natural pozzolanas for use in blended cements are plenty. They have been investigated by (Wachira et al., 2019) among many others. Muthengia (2003) investigated the possibility of using a calcined blended mix of rice husks (RH), broken bricks (BB) and spent bleaching earth (SBE) as a pozzolana. A blend of the materials in the ratio of 5:1:1 was tried and it gave very promising results. However the optimization of ratios especially the amount of BB in the blend was not done. If an optimized workable ratio is determined, the production of the pozzolana for cement will be applicable. It will help lower the cost of cement when replaced in OPC at higher proportions (Marangu et al., 2014).

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2. Materials and methods

2.1. Materials

The test materials were sampled from several places. SBE was sampled from dump sites around BIDCO Oil Refineries in Thika, RH from Mwea Rice Mills Limited in Mwea, and BB from Clay Works Manufacturers Limited in Ruiru, ALS from Welding Alloys Limited (WAL) in Nairobi, ordinary Portland cement (OPC) from East African Portland Cement Company (E.A.P.C.C) at Athi River around Nairobi, Kenya. Each of the samples as received from the respective sources was thoroughly mixed before being subjected to treatment and/or analysis.

2.2. Methods

2.2.1. Characterization of raw SBE

Calorific value of the raw SBE was done in accordance to EN 14214 (2013). A bomb calorimeter model CAB101.AB1.C was used. Determination of oil content was done in accordance to the BS EN ISO 659 (1999). Soxhlet apparatus were used.

To determine ignition and self combustion temperatures, 1.000 g of SBE in a silicon carbide crucible was slowly heated by use of a Bunsen burner flame until it ignited. Temperature measurement was done by use of two digital thermometers number GTH 1160 fitted with heat sensors probe model RS Number 610-067. After ignition, temperature was then monitored to determine the maximum self-combustion temperature. The resulting product was preserved for determination of oil content after self-combustion. This product was weighed and heated at 1000 °C to a constant weight in a furnace model FSE-520-210P. The difference in weight before heating and after heating at 1000 °C was expressed as a percentage of the original weight of the sample. This was done to determine the amount of oil withheld after self-combustion.

To determine ash content, 1.000 g of SBE was heated at temperature of 1000 °C in a pre-weighed platinum crucible in a furnace model FSE-520-210P. It was then cooled in a desiccator and weighed. The crucible and its content were reheated, cooled and weighed to a constant weight. The ash content was calculated using Eq. (1).

$$A = \frac{W_f}{W_o} \times 100 \quad (1)$$

where

A = % ash content
$W_{o=}$ original mass of sample
W_f = mass of sample after heating

2.2.2. Incineration of the pozzolana

RH and SBE as obtained from their respective sources were incinerated in the form they were sampled. Some of the BB was first crushed using a laboratory crusher, and then finely ground to pass through 90 μm sieve (Kenya Bureau of Standards, 2001) using a laboratory ball mill. Other broken bricks were not finely milled after crushing. It was used in the crushed form where some particles were as large as 3 mm in diameter.

The RH, ground BB and SBE were then fed into the fixed bed kiln (FBK) (Ochung'o, 1993). This was done in accordance to Muthengia (2003) but with variation in the feed ratios. 5 kg of RH was spread on the floor of the FBK, followed by 2 kg of BB, then 5 kg of RH, then 4 kg of SBE and finally 10 kg of RH was spread over the whole mass. A little paraffin was put at the window of the kiln and the whole mass ignited. The window was closed. The temperature of the kiln was controlled below 700 °C through the action of opening and closing the ventilation windows. The temperature of the kiln was monitored using a thermocouple with a long probe. The resulting ash was collected after cooling for a period of 3 h. It was then ground to pass through 90 μm sieve using a laboratory ball mill and taken for other tests.

The procedure above was repeated for making other samples but in each successive arrangement more BB was added. These gave test ashes with the ratios of RH: BB: SBE of 20: 0: 4, 20:1:4, 20:2:4, 20:4:4, 20:6:4. These were labeled as sample 1, 1.5, 2, 3 and 4 respectively. The procedure was repeated but this time using 2 and 4 kg of BB. The BB in these two samples had particles as of about 3 mm in diameter. The resulting ashes were labeled sample 5 and 6 respectively. The resulting labeled ashes were preserved in airtight plastic containers for further tests.

2.2.3. Chemical constituent analysis of pozzolana samples

This was done using atomic absorption spectroscopy, Flame photometry and gravimetric analysis.

2.2.4. Pozzolanicity tests

A quick way to check the pozzolanic reaction was done by determining the change in electrical conductivity of a saturated $Ca(OH)_2$ solution. This was done in accordance to Luxan (Luxan et al., 1989) and Bui (2001). Pozzolanicity by use of the standard pozzolanicity curve was done in accordance with the ISO 863(E), (ISO, 1990).

2.2.5. Compressive strength analysis

Compressive strength analysis of Pozzolana-ALS-OPC cement was determined in accordance with the KS EAS 148-1 (KEBS, 2000). Three mortar prisms each of dimensions 40 × 40 × 160mm were used. The ASTM 593 part C (ASTM, 1991) was adopted for analysis of compressive strength for the Pozzolana-ALS cements. However this was done with slight modifications. 70.7 mm mortar cubes instead of the 50 mm mortar cubes prescribed in the standard were used.

3. Results and discussion

3.1. Raw spent bleaching earth (SBE)

SBE as obtained from Bidco Oil Refineries at Thika-Kenya was light grey in color, oily and finely pulverized. Characterization test results are given in the Table 1.

Oil content of the SBE used in this study was slightly lower than those used in the study by Muthengia (2003), but considerably higher than others (Moshi Anselm, 2017; Sahafi et al., 2016). This may be attributed to the differences in the filtering processes of the different oil manufacturing industries. For this reason, most oil processing companies will prefer processes that would withhold as little oil as possible (Hayder et al., 2011). In the oil manufacturing industry, the lower the oil content in the SBE the more the profit for the manufacturer as less oil is wasted in the filtering process. The amount of oil withheld content was comparable to other SBE studied elsewhere (Huang and Chang, 2010; Park et al., 2001).

The oil present in SBE has lead to research in best ways of recycling it. Such oil has been found to be of low quality, but can be used for biodiesel production (Park et al., 2001). It has also been investigated as a possible component in making lubricating grease (Hayder et al., 2011). However due to economic, ecological and/or logistical issues, these methods have not been widely used by the industry. Incineration of SBE remains the main disposal method (Shanks, 2007).

The high oil content and its spontaneous auto ignition is the main

Table 1
Characterization of raw SBE.

Property	Results
Calorific value	13.23 ± 0.0259 J/g
Oil content	29.52 ± 0.5022 wt %
Ignition temperature	235.0 ± 0.8165 °C
Maximum self-combustion temperature	438.0 ± 1.633 °C
Ash content	61.25 ± 0.0867 wt %

cause of fire hazards associated with handling of SBE especially when dumped in open sites (Pollard et al., 2003). Therefore SBE is classified as a hazardous waste, since the oil in the SBE contributes to its pollution aspect. Untreated SBE cannot be used as land fill material. This is due to its high organic content which exceeds the limit of waste acceptance criteria (WAC) for hazardous waste landfills under the EU land fill Directive (Environmental Agency, 2010).

The calorific value of the Bidco Oil refineries SBE was also found to be lower than those obtained by Muthengia (2003). These calorific values can be attributed to the amount of oil content present in the SBE samples. In line with the high calorific value, SBE has been investigated as a source of energy in the activation of RH and BB in the production of combined pozzolanic materials (Muthengia, 2003). It has also been investigated as a possible source of making SBE fuel briquettes. Other options include using SBE as an alternative fuel in cement kilns or as a feedstock for the production of clay bricks (Surhartini et al., 2011).

When heated with a Bunsen burner in a silicon crucible, the oil in the SBE started to volatilize at a temperature of 160 °C. The volatiles ignited at 235 °C to a pale yellow flame. The flame continued to burn on its own to a temperature of 438 °C. These self-ignition temperatures are important in the production of artificial pozzolana. Reactive silica in the pozzolana is formed in the temperature range of 550 °C to 700 °C (Chandaresekha et al., 2006). A maximum ignition temperature of 438 °C, would therefore not affect the pozzolanicity of the resulting pozzolanic material when the SBE is used to make pozzolana.

After complete combustion of the oil from the SBE, the ash content was quite high in comparison with what other authors have investigated (Surhartini et al., 2011; Muthengia, 2003). High ash content would be due to low amount of volatiles. It is also important in that it would increase the amount of pozzolana after incineration.

3.2. Chemical constituents

The results of the chemical constituent analysis of the pozzolana samples are given in Table 2. The results shown are those for AAS, gravimetric analysis and Flame Photometry. For sodium and potassium, Flame photometry was used, for LOI, gravimetric analysis was used. For all the others, AAS was used.

From Table 2, the results show that all the pozzolana samples except SBE, had the sum of SiO_2, Al_2O_3 and Fe_2O_3 above the Kenya Standard KS 02 1263 (KEBS, 1993) as well as the ASTM C 618 (ASTM, 2006). These oxides are considered as the major components of pozzolanas. A pozzolanic material must contain a minimum of 70 % of the sum of the oxides of Aluminium, silicon and iron (KEBS, 1993). This avails the required glassy content to react with added lime in the presence of water at room temperature to produce the cementing materials according to Eqs. (2) and (3) respectively (Igor et al., 2010; Cook, 1983).

$$Al_2O_3 + 4Ca(OH)_2 + 9H_2O \rightarrow 4CaO.Al_2O.13H_2O \quad (2)$$

$$3Ca(OH)\ \ 2 + 2SiO\ \ 2 \rightarrow 3CaO.2SiO\ \ 2.3H2O \quad (3)$$

The alkali content (Na_2O) varied from 1.47 % in BB to 0.45 % in sample 6. These values are lower than the maximum alkali content of 1.5% required for pozzolana (KEBS, 2001). Higher levels of the alkalis may result to expansion of cured mortar through alkali aggregate reactions (Garcia et al., 2007). The alkali levels are responsible for the pore water in cured cement paste for maintaining a pH of above 12. This is important in passivating the rebar if embedded and also availing the medium for cement hydration (Diamond, 1981).

The MgO levels of the pozzolana samples were below the maximum limits of 5 percent as per ASTM standard. For commercial cements the ISO standard recommends a maximum of 2.8 % (ASTM, 1991). The MgO is limited because of it being associated with destructive expansion of concrete. This is because in cured mortar and concrete, MgO forms expansive $Mg(OH)_2$ on reaction with water. This may further react with the silica in the pozzolana to form non cementitious magnesium silicate hydrates. This is shown by Eqs. (4) and (5).

$$MgO + H_2O \rightarrow Mg(OH)_2 \quad (4)$$

$$3Mg(OH)_2 + 2SiO_2 \rightarrow 3MgO.2SiO_2.3H_2O \quad (5)$$

The LOI of the samples increased with the increase in the amount of total clay available in the sample. This was due to increased compaction of BB and SBE. This might have caused reduction of air spaces for complete incineration of the RH and activation of the SBE (Muthengia, 2003). It was noted that LOI was highest with the samples that had crushed BB. However all the samples tested were within the limits of the standard of pozzolanas (KEBS, 2001). High LOI decreases pozzolanicity (Shihembetsa and Waswa-Sabuni, 2006; Sensale, 2006). LOI signifies the amount of carbon content in the samples. High carbon content in pozzolana may affect the workability of concrete (Payá; et al., 2001).

3.3. Pozzolanicity tests

3.3.1. Change in electrical conductivity of saturated $Ca(OH)_2$ solution

The results of the change in electrical conductivity of the pozzolana samples in saturated calcium hydroxide are shown in the Fig. 1.

From Fig. 1 it is observed that the sample with the RH: BB: SBE ratio of 20:2:4 by weight, showed the highest change in conductivity. According to Luxan et al. (1989), the sample which shows the highest change in conductivity has the highest value of pozzolanicity. Luxan et al. (1989) further noted that sample which give a conductivity change of above 1.4, are potentially very good pozzolanic materials. In this case, the pozzolanicity increased as the amount of BB was increased in the RH: BB: SBE ratio. Muthengia (2003) observed that the pozzolanic activity of RHA, ashed SBE and BB increased in that order respectively. It was therefore expected that increase in the amount of BB in the pozzolana mixes would increase the pozzolanicity of the samples. However from the above conductivity tests this was not the case. When the amount of BB added was beyond 2 kg in the 20 kg RHA and 4 kg SBE mix, pozzolanic activity of the resultant ash decreased. This implied a decrease in the

Table 2
Chemical Constituent of BB, SBE and the pozzolana samples.

Oxide	% by weight							
	BB	SBE	Sample1	Sample2	Sample 3	Sample 4	Sample 5	Sample 6
CaO	0.46	2.30	0.87	0.65	0.55	0.45	3.03	2.01
SiO_2	64.84	66.0	74.3	80.5	81.20	82.50	76.90	75.92
Al_2O_3	21.54	6.20	7.68	7.80	7.92	8.00	6.73	6.04
Fe_2O_3	7.87	3.40	6.80	6.90	7.90	3.67	4.50	3.98
SO_3	0.23	-	0.31	0.13	0.12	0.30	0.12	0.52
MgO	1.94	3.20	1.70	1.90	1.74	1.70	1.72	2.24
K_2O	3.62	1.90	1.99	1.59	1.70	2.20	1.80	3.20
Na_2O	1.47	2.80	1.44	0.45	0.55	0.54	0.55	0.55
LOI	0.88	14.0	3.99	2.99	4.01	4.70	4.20	5.54
$SiO_2 + Al_2O_3 + Fe_2O_3$	94.07	71.6	88.78	91.00	89.82	88.27	88.33	85.94

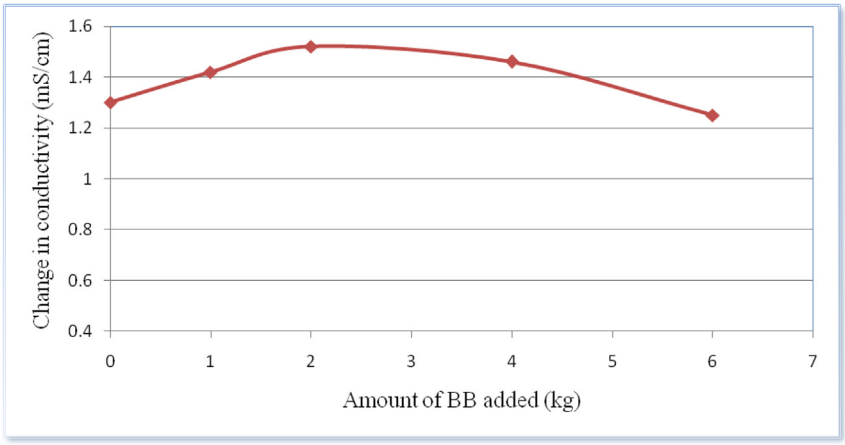

Fig. 1. Change in conductivity with increase in amount of BB in RH: BB: SBE.

available reactive silica in the samples. This could have been due to compaction of the whole mix mass. Compaction of the samples by an increased amount of BB may have reduced effective burning of the materials being incinerated (Muthengia, 2003). This must have resulted in less pozzolanic material being formed. Compared to the chemical constituents of the samples in Table 2, it can also be seen that the LOI of the samples increased with an increase in the amount of BB in the samples. This confirms the reduction in the burning as more organic materials in the pozzolanas show less burning.

Fig. 2 shows the change in conductivity of the pozzolana samples 3 and 4 compared to those of sample 5 and 6 respectively. t values for the two sets of changes in conductivities were calculated to determine whether a significant difference in their means existed. For samples 5 and 3, t calculated was 4.648, while t critical at 95 % confidence level was 2.776. This meant that the change in fineness of the BB as opposed to the amount of added BB as 4 kg does not have a significant effect on pozzolanicity of the materials at this stage. However when amount of BB added was 6 kg as in sample 6, t calculated was 0.775 compared to t critical of 2.776, meaning that there was a significant change in pozzolanicity when less fine BB was used. This could be attributed to the uniformity of the amount of milled BB and that not milled. Since the actual amounts of the different sizes of BB were not considered during incineration, sample 6 could have had finer particles of BB (90 μm) than the larger ones of about 3 mm.

3.3.2. *Pozzolanicity by use of the standard pozzolanicity curve*

Figs. 3 and 4 give the results of CaO and OH^- concentrations of the various pozzolana samples at the 8^{th} and 15^{th} day of curing (ISO, 1990). All the coordinates of the pozzolana samples were below the solubility isotherm. This is because as the cement hydrates, it releases calcium hydroxide as a by-product. This sets the right pH value for $Ca(OH)_2$ to react with the pozzolana as well as availing the $Ca(OH)_2$ for the reaction (Takemoto and Uchikawa, 1980). In Portland pozzolana cement (PPC), the released $Ca(OH)_2$ react with the pozzolana incorporated in the cement to produce more cementitous material. This lowers the concentration of CaO in the pore solution. For all the samples, the OH^- remains fairly constant at the 8^{th} day of curing. This is because the pozzolanic reaction involves only the $Ca(OH)_2$ and not the K_2O and Na_2O (Takemoto and Uchikawa, 1980).

The alkali oxides are responsible for the pore solution pH with $Ca(OH)_2$ acting as a buffer store for OH^- (Diamond, 1981).

All the samples had a 40 percent replacement of the pozzolana containing RH: BB: SBE. The reduction in the Ca^{2+} and OH^- was more pronounced compared to the same replacements for the individual pozzolana of RHA, BB and SBE as observed by Muthengia (2003). This indicated that the incineration of the three pozzolanas results in a more reactive sample. The 15^{th} day pozzolanicity diagram indicates even lower coordinates for all the PPC samples made. The coordinates are low because the pozzolana samples in solution release reactive silica that reacts with the released $Ca(OH)_2$. The sample number 2 had the lowest coordinates. This means the sample had the highest concentration of the silica that reacted with the $Ca(OH)_2$, meaning it was the most pozzolanic.

3.4. *Compressive strength evaluation*

3.4.1. *Compressive strength of Pozzolana-ALS cements*

Results of the 7^{th} day and 28^{th} day compressive strengths of pozzolana-ALS cements are shown in Fig. 5.

The pozzolanic activity of the test pozzolana samples increased as the amount of BB was increased in the RH, BB and SBE blend up to 2 kg.

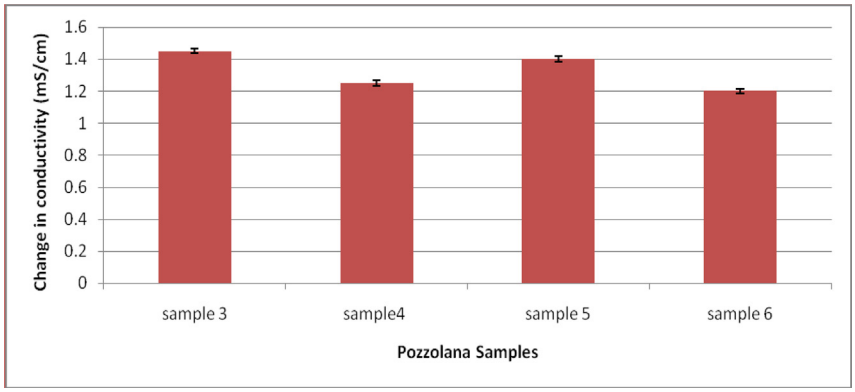

Fig. 2. Change in Conductivity of pozzolana samples equal masses of BB.

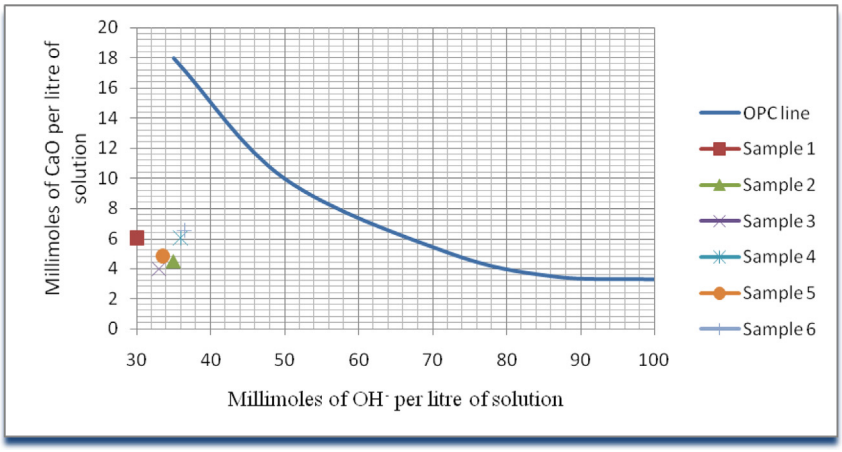

Fig. 3. Pozzolanicity diagram for pozzolan-ALS cements on the 8th day of curing.

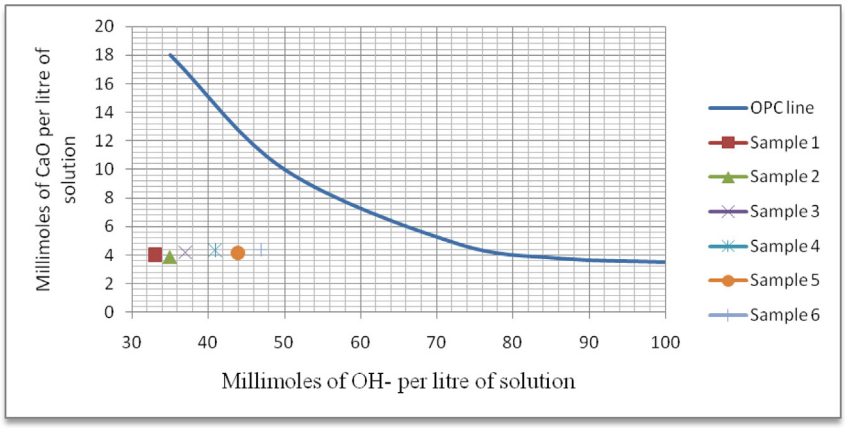

Fig. 4. Pozzolanicity diagram for pozzolana-ALS cements on the 15th day of curing.

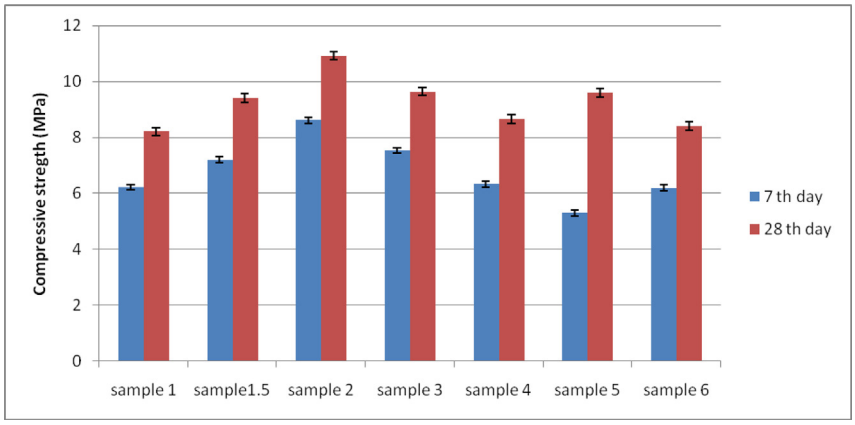

Fig. 5. Compressive Strength of the Pozzolana-ALS cements.

There after the pozzolanic activity reduced. Studies had shown that BB had the highest pozzolanicity, followed by RHA then SBE had the least activity(Muthengia, 2003). It was therefore expected that the compressive strength would increase with the increase in the amount of BB added. However this was not the case. A similar trend as the variation of compressive strength with amount of BB added was observed in pozzolanicity tests (Fig. 2). This could be attributed to pozzolanic activity changes with the available amount of amorphous or crystalline silica. This could perhaps be attributed to the increase in compaction of pozzolana samples as a result of addition of more BB. Compaction of the whole mass of the pozzolana being incinerated would increase the possibility of production of crystalline phases of silica in the sample. The pozzolanic activity is enhanced by the presence of non-crystalline silica in a pozzolana (Boateng and Skeete, 1990; Mehta, 1979). More so LOI was high in samples 3, 4 and 5 which lower pozzolanic activity. The (SiO_2+ Al_2O_3+Fe_2O_3) was also low in these samples. These constituents

are important in pozzolana-lime strength development (Muller, 2005).

From Fig. 5, it is clear that both the 7th and 28th day compressive strength of all the pozzolana samples was higher than the minimum standard requirements. ASTM 593 C (1991) standard prescribes a minimum of 4.1 Mpa at the 7th and/28th day curing. The difference between the 7th and 28th day compressive strengths is small. This is because the pozzolana-ALS cements were cured at 54 ± 2 °C. This is an accelerated hydration (Lea, 1973). The test samples would therefore be ideal for the making pozzolana-lime cement. Sample 2, which had the optimum pozzolanic activity, was used to formulate a blend of pozzolana-ALS-OPC cement for further investigation.

4. Conclusions

In view of the results and analysis, the following conclusions were drawn:

1. When blends RH, BB and SBE were incinerated in the FBK in varying ratios, the resulting materials had varying pozzolanic activities. Pozzolanic activity increased with increase in the amount of BB in the raw blended mix up to 2 kg. The optimum mix ratio for RH: BB: SBE was 10:1:2.
2. The chemical composition of the resulting blends of the pozzolanas, varied with increased amount of BB in it. More BB increased the amount of total silica in the blends. However this did not necessarily increase the pozzolanic reactions of the resulting ash. This amount of the silica, alumina and iron which directly influence the reactivity of the pozzolana were found to be in optimum proportion when the ratio of RH: BB: SBE was 10:1:2.
3. Pozzolanic activity of the raw calcined blended mix of RH, BB and SBE did not considerably change with a variation in fineness of the BB used. This means that BB fineness varying from 90 μm to 3 mm in diameter would be suitable for use in making calcined raw blended mix pozzolana of RH, BB and SBE.
4. Strength development of the pozzolana-ALS cement increased with increase in the amount of BB in the pozzolana blends up to the ratio 10: 1:2, when it started reducing with each extra addition of the BB. However in all samples both the 7th day and 28th day compressive strengths were above the minimum standard requirements of 4.1 MPa.

Declarations

Author contribution statement

P. Nalobile: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

J. M. Wachira, J. K. Thiong'o & J. M. Marangu: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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