Characteristics of Blended Cements Produced from Selected Hardwood Ashes

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Abstract—In an attempt to find an alternative binding material for construction industry, this study considered the use of wood ash from three different hardwoods namely: Tectonagrandis, Cassia siamea and Vitellariaparadoxa as a pozzolan in cement production. The study investigates the chemical composition (silica (SiO₂), aluminium oxide (Al₂O₃), ferric oxide (Fe₂O₃), calcium oxide (CaO), magnesium oxide (MgO), sulphur trioxide (SO₃), sodium oxide (Na₂O) and potassium Oxide (K₂O)) of the ashes and the clinker. The production of blended cements were carried out in the factory by replacing 5- 50% by weight of Ordinary Portland Cement Clinker with the ashes during the manufacturing process. The cement without wood ash serves as the control. The physical characteristic (fineness, initial and final setting times, heat of hydration and residue on 45µm sieve), and the chemical composition of the blended cements were also investigated. The results showed that all the wood ashes (Tectonagrandis ash (TGA), Cassia siamea ash (CSA) and Vitellariaparadoxa ash (VPA)) were suitable material for use as pozzolan since they satisfied the requirement for such a material by having a combined SiO₂, Fe₂O₃ and Al₂O₃ of more than 70%. The TGA, CSA and VPA blended cements satisfied standard requirements for up to 20% replacement level. It was concluded that all the wood ashes were suitable for use in the production of blended cements.

Keywords—Tectonagrandis ash, Cassia siamea ash, Vitellariaparadoxa ash, Pozzolan, Blended cement

1. INTRODUCTION

Cement is the only factory made standard component in concrete (Shetty, 2005). Other ingredients namely, water and aggregates are natural materials. Cement is a finely ground, non-metallic, inorganic powder which when mixed with water forms a paste that sets and hardens. The search for alternative binder or cement replacement materials led to the discovery of the potentials of using agricultural residue as cementitious materials.

The use of agricultural residue in cement production is an environmental friendly method of disposing of large quantities of materials that would otherwise pollute land, water and air. The agricultural residue which possess pozzolanic properties and have been studied for use in blended cement are Rice Husk Ash (Chungsangunsi et al., 2007; Coutinho, 2003), Bamboo Leaf Ash (Dwivedi et al., 2006), Palm Fruit Ash (Olonode, 2010), Locust Bean Pod Ash (Adama and Jimoh, 2011), Cement Cassava Peel Ash (Salau et al., 2012), Corn Husk Ash (Raheem et al., 2012), Corn Cob Ash (Adesanya and Raheem, 2009, 2010) and wood ash (Raheem and Adenuga 2013; Abdullahi, 2006). Pozzolans are siliceous material, which by itself possesses no cementitious properties but in processed form and finely divided form, react in the presence of water with lime, to form compounds of low solubility having cementitious properties (Arthanari et al., 1981).

Previous studies showed that many researchers have worked on the mixture of wood ash and ordinary portland cement at the point of need, without considering the type of wood (either from softwood or hardwood) and without considering blending of wood ash with clinker and gypsum during factory production of the cement.

Therefore, this study focuses on determination of chemical composition of wood ashes from three different types of hardwood, as well as the physical and chemical properties of the blended cements produced at varying percentage replacement of each wood ash during the manufacturing process.

2. MATERIALS AND METHODS

2.1 Materials

Three types of hardwood were used to produce the wood ash namely:-Tectonagrandis (gedu), Cassia siamea (Kasia) and Vitellariaparadoxa (Emi). Each wood was burnt separately in a bread bakery that uses wood as fuel for their operation and their ashes were collected using clean sacks. The wood ashes were sieved and only those retained on a 0.425µm sieve was used for the study. The ashes were taken to Lafarge Cement, West Africa Portland Cement Company (WAPCO) Sagamu, Ogun State, Nigeria, for chemical analysis. The analyses of the ashes were carried out using X-ray Fluorescence analyser (Model QX 1279). The clinker used for producing the blended cement was obtained from Lafarge Cement, West Africa Portland Cement Company (WAPCO) Sagamu, Ogun State, Nigeria. This is the clinker used by the company to produce Ordinary Portland Cement (OPC) and the chemical analysis was also carried out.

2.2 Cement Mixtures

Ten (10) series of Tectonagrandis ash (TGA), Cassia siamea ash (CSA) and Vitellariaparadoxa ash (VPA) were prepared for cement milling. The milling involved the replacement of 5%, 10%, 15%, 20%, 25%, 30%, 35%, 40%, 45% and 50% by weight of Ordinary Portland Cement Clinker with the ashes during the manufacturing process. Ordinary Portland Cement (OPC) without ash content served as the control. As it is the practice in WAPCO Sagamu works, 5% gypsum was used for the cement milling. The milling machine used is Laboratory Ball Mill (Model R. PM 1400/50, Serial Number...
The ball mill was charged with the required weight of clinker, TGA, CSA, VPA and gypsum. After which it was firmly tightened and put in operation for 55 minutes. The milling was done in batches of 4000g. Table 1 shows the mix proportion for each batch and the percentage ash replacement.

Table 1. Mix proportion for changing ball mill

| % of Ash | % of Clinker | Weight of Ash (g) | Weight of Clinker (g) | Weight of Gypsum (g) |
|----------|-------------|------------------|-----------------------|----------------------|
| 0        | 95          | 3800             | -                     | 200                  |
| 5        | 90          | 3600             | 200                   | 200                  |
| 10       | 85          | 3400             | 400                   | 200                  |
| 15       | 80          | 3200             | 600                   | 200                  |
| 20       | 75          | 3000             | 800                   | 200                  |
| 25       | 70          | 2800             | 1000                  | 200                  |
| 30       | 65          | 2600             | 1200                  | 200                  |
| 35       | 60          | 2400             | 1400                  | 200                  |
| 40       | 55          | 2200             | 1600                  | 200                  |
| 45       | 50          | 2000             | 1800                  | 200                  |

2.3 Testing of Blended Cement

The physical characteristics of the blended cements determined are fineness, initial and final setting times, residue on 45μm sieve and heat of hydration. Also, the chemical characteristics of each of the blended cements determined are the chemical composition, loss on ignition (LOI), free lime and insoluble residue (IR). All the tests were carried out according to BS EN 196-6:1995, BS EN 196-3:1995, BS EN 196-2:1995, BS EN 196-1:1995.

3. RESULTS AND DISCUSSION

3.1 Chemical Composition of Hardwood Ashes and Clinker

Table 2 shows the mean value of elemental oxides present in the TCA, CSA and VPA sample. The results indicate that all the samples of ashes from TCA, CSA and VPA had percentage of silica (SiO₂) of 64.00, 65.88 and 64.78 respectively with CSA having the highest. These values are higher than the value (61.18) reported by Raheem and Adenuga (2013) in which the type of wood that produces the ash was not known. The results also indicate that all the ashes are good pozzolanic material in accordance with ASTM C 618 requirements.

Table 2. Percentage Composition of Hardwood Ashes

| Chemical Constituents | TGA    | CSA    | VPA    |
|-----------------------|--------|--------|--------|
| SiO₂                  | 64.00  | 65.88  | 64.78  |
| Al₂O₃                 | 6.16   | 7.52   | 6.40   |
| Fe₂O₃                 | 2.70   | 4.48   | 2.91   |
| CaO                   | 10.67  | 11.10  | 10.52  |
| MgO                   | 3.76   | 2.53   | 3.79   |
| SO₃                   | 1.05   | 1.37   | 1.12   |
| K₂O                   | 5.73   | 5.87   | 6.08   |
| Na₂O                  | 0.37   | 0.42   | 1.06   |

Table 3 shows the chemical composition of the clinker used. The values obtained for the elemental oxide are in agreement with range of values reported by Adesanya and Raheem (2009). The composition also satisfied the requirement in ASTM C 150 and NIS 439:2000. Thus the clinker is quite suitable for cement production.

Table 3. Average Percentage Composition of Clinker

| Chemical Constituents | Percentage composition (%) |
|-----------------------|-----------------------------|
| SiO₂                  | 20.30                       |
| Al₂O₃                 | 5.47                        |
| Fe₂O₃                 | 3.47                        |
| CaO                   | 65.90                       |
| MgO                   | 2.02                        |
| SO₃                   | 2.17                        |
| K₂O                   | 0.15                        |
| Na₂O                  | 0.28                        |
| P₂O₅                  | 0.17                        |
| LOI                   | 1.31                        |
| LSF                   | 95.90                       |
| AR                    | 1.55                        |
| SR                    | 2.25                        |
| Free lime             | 1.03                        |

3.2 Chemical Composition of Blended Cements

The chemical composition of TGA, CSA, and VPA blended cements produced are presented in Table 4 to 6. For TGA, the silica content of the blended cements increased from 20.35% for 5% replacement to 22.46% for 50% replacement. For CSA, the silica content of the blended cement also increased from 20.15% for 5% to 22.00% for 50% replacement. VPA blended cements behaved in the same manner with increasing from 20.35% for 5% replacement to 21.45% for 50% replacement. This is in line with the findings of Adesanya and Raheem (2009) that reported increase in Silica contents of corn cob ash (CCA) blended cement as the percentage of CCA increases. All the blended cements satisfied the minimum of 20% silica content requirements for Type II and Type IIA Portland Cement as specified by ASTM C 150-02.
### Table 4. Percentage composition of TGA-blended cement

| Constituent | 0% | 5%  | 10% | 15% | 20% | 25% | 30%  | 35%  | 40%  | 45%  | 50%  |
|-------------|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| SiO₂        | 20.10 | 20.35 | 20.45 | 20.55 | 20.75 | 21.00 | 21.45 | 21.95 | 22.24 | 22.54 | 22.46 |
| Al₂O₃       | 5.09  | 4.64  | 4.57  | 4.25  | 4.07  | 3.99  | 3.74  | 3.50  | 3.42  | 3.10  | 3.02  |
| Fe₂O₃       | 2.99  | 2.86  | 2.76  | 2.60  | 2.52  | 2.45  | 2.31  | 2.15  | 2.07  | 1.92  | 1.82  |
| CaO         | 64.18 | 62.36 | 60.74 | 59.35 | 57.93 | 56.25 | 55.23 | 53.50 | 51.20 | 45.25 | 47.46 |
| MgO         | 1.73  | 1.93  | 2.04  | 2.25  | 2.43  | 2.60  | 2.72  | 3.00  | 3.13  | 3.30  | 3.44  |
| SO₃         | 2.18  | 2.33  | 2.38  | 2.45  | 2.56  | 2.62  | 2.71  | 2.80  | 2.85  | 2.95  | 3.05  |
| K₂O         | 0.15  | 0.59  | 0.79  | 1.00  | 1.50  | 2.00  | 2.28  | 3.25  | 3.59  | 4.35  | 4.61  |
| Na₂O        | 0.03  | 0.02  | 0.02  | 0.02  | 0.01  | 0.01  | 0.02  | 0.01  | 0.03  | 0.04  | 0.04  |
| P₂O₅        | 0.00  | 0.08  | 0.12  | 0.25  | 0.35  | 0.45  | 0.53  | 0.60  | 0.66  | 0.75  | 0.82  |
| Free lime   | 1.47  | 1.45  | 1.43  | 1.40  | 1.39  | 1.37  | 1.35  | 1.32  | 1.30  | 1.27  | 1.25  |
| LSF         | 96.54 | 98.81 | 98.48 | 100.02| 102.57| 104.50| 106.40| 108.00| 108.67| 112.50| 114.18|
| SR          | 2.49  | 2.53  | 2.54  | 2.56  | 2.58  | 2.55  | 2.56  | 2.56  | 2.55  | 2.50  | 2.52  |
| AR          | 1.70  | 1.64  | 1.66  | 1.65  | 1.62  | 1.63  | 1.62  | 1.64  | 1.66  | 1.65  | 1.66  |
| CaS         | 65.92 | 69.34 | 67.17 | 70.00 | 71.68 | 72.05 | 74.34 | 73.25 | 72.22 | 73.00 | 73.51 |
| CaA         | 8.44  | 7.55  | 7.44  | 6.00  | 6.54  | 6.45  | 6.01  | 5.35  | 5.57  | 4.10  | 4.93  |
| CaAF        | 9.09  | 8.69  | 8.38  | 8.10  | 7.65  | 7.25  | 7.03  | 6.10  | 6.28  | 5.25  | 5.53  |

### Table 5. Percentage composition of CSA-blended cement

| Constituent | 0% | 5%  | 10% | 15% | 20% | 25% | 30%  | 35%  | 40%  | 45%  | 50%  |
|-------------|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| SiO₂        | 20.10 | 20.15 | 20.35 | 20.65 | 20.85 | 20.99 | 21.25 | 21.65 | 21.85 | 21.95 | 22.00 |
| Al₂O₃       | 5.09  | 5.00  | 4.95  | 4.88  | 4.80  | 4.70  | 4.25  | 3.95  | 3.75  | 3.55  | 3.25  |
| Fe₂O₃       | 2.99  | 2.97  | 2.85  | 2.72  | 2.65  | 2.65  | 2.45  | 2.35  | 2.35  | 2.15  | 2.00  |
| CaO         | 64.18 | 63.15 | 62.00 | 59.15 | 58.00 | 57.15 | 55.95 | 53.35 | 50.00 | 48.35 | 47.95 |
| MgO         | 1.73  | 1.83  | 2.00  | 2.05  | 2.35  | 2.55  | 2.60  | 2.65  | 2.72  | 2.82  | 3.00  |
| SO₃         | 2.18  | 2.28  | 2.35  | 2.45  | 2.57  | 2.65  | 2.85  | 2.92  | 2.95  | 2.97  | 3.15  |
| K₂O         | 0.15  | 0.20  | 0.35  | 0.50  | 0.65  | 0.70  | 1.05  | 1.95  | 2.25  | 2.35  | 2.95  |
| Na₂O        | 0.03  | 0.03  | 0.03  | 0.02  | 0.21  | 0.01  | 0.01  | 0.02  | 0.01  | 0.03  | 0.03  |
| P₂O₅        | 0.00  | 0.01  | 0.08  | 0.12  | 0.14  | 0.20  | 0.30  | 0.35  | 0.38  | 0.42  | 0.5   |
| Free lime   | 2.00  | 1.98  | 1.92  | 1.87  | 1.85  | 1.75  | 1.55  | 1.45  | 1.34  | 1.24  | 1.14  |
| LSF         | 96.54 | 97.00 | 98.15 | 98.55 | 98.95 | 99.00 | 95.35 | 100.00| 102.05| 104.35| 108.05|
| SR          | 2.49  | 2.50  | 2.52  | 2.53  | 2.55  | 2.55  | 2.56  | 2.56  | 2.55  | 2.56  | 2.56  |
| AR          | 1.70  | 1.68  | 1.68  | 1.67  | 1.67  | 1.67  | 1.64  | 1.64  | 1.64  | 1.62  | 1.64  |
| CaS         | 65.92 | 66.00 | 68.15 | 69.15 | 69.95 | 70.15 | 71.35 | 71.92 | 72.15 | 72.55 | 72.15 |
| CaA         | 8.44  | 8.35  | 8.30  | 8.15  | 8.00  | 7.85  | 7.55  | 7.45  | 7.24  | 6.95  | 6.65  |
| CaAF        | 9.09  | 8.95  | 8.70  | 8.50  | 8.20  | 7.00  | 6.89  | 6.55  | 6.35  | 6.25  | 6.00  |
A different trend was noticed for the alumina and ferric oxide contents which decreased from 4.64% to 3.02% and 2.86 to 1.82 for 5% and 50% respectively, for TGA blended cement. Similar trends were observed for CSA and VPA with the alumina and ferric oxide contents decreasing from 5.00% to 3.25% and 2.97 to 2.00 as well as 5.09% to 4.29% and 2.99 to 2.70, respectively. This shows that as the percentage of all the wood ashes is increases, the alumina and ferric oxide is reducing. It was noticed that CSA blended cement had the highest values of alumina and ferric oxides. However, all the blended cements satisfied the maximum of 6.00% alumina content requirements for Type II and Type IIA Portland cement specified by ASTM C 150-02.

For TGA blended cements, the calcium oxide content decreased from 62.36% for 5% to 47.46% for 50% replacement, while for CSA blended cements, it decreased from 63.15% to 47.95% and for VPA blended cements from 64.00% to 42.00%. All the blended cements have lower value of CaO contents than the control. This shows that addition of all the wood ashes led to a reduction in the lime content of the blended cements. Calcium oxide is responsible for the formation of C3S (3CaO.SiO2) and C2S (2CaO.SiO2) in cement which are responsible for the strength in the late or early part of the concrete (Taylor, 2004).

Table 6. Percentage composition of VPA-blended cement

| Constituent | 0% | 5% | 10% | 15% | 20% | 25% | 30% | 35% | 40% | 45% | 50% |
|------------|----|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| SiO2       | 20.10 | 20.35 | 20.45 | 20.75 | 20.85 | 20.95 | 21.00 | 21.05 | 21.35 | 21.45 | 21.55 |
| Al2O3      | 5.09 | 5.07 | 5.05 | 5.00 | 4.95 | 4.85 | 4.72 | 4.65 | 4.55 | 4.35 | 4.25 |
| Fe2O3      | 2.99 | 2.99 | 2.96 | 2.80 | 2.85 | 2.82 | 2.80 | 2.76 | 2.74 | 2.72 | 2.70 |
| CaO        | 64.18 | 64.00 | 63.15 | 63.00 | 62.95 | 62.75 | 62.55 | 62.35 | 62.15 | 62.00 | 62.00 |
| MgO        | 1.73 | 1.85 | 1.95 | 2.00 | 2.15 | 2.25 | 2.30 | 2.32 | 2.40 | 2.45 | 2.50 |
| SO3        | 2.18 | 2.20 | 2.28 | 2.32 | 2.45 | 2.55 | 2.57 | 2.60 | 2.65 | 2.70 | 2.85 |
| K2O        | 0.15 | 0.16 | 0.18 | 0.20 | 0.22 | 0.24 | 0.30 | 0.34 | 0.36 | 0.38 | 0.50 |
| Na2O       | 0.03 | 0.03 | 0.02 | 0.02 | 0.03 | 0.03 | 0.01 | 0.01 | 0.02 | 0.30 | 0.02 |
| PO4        | 0.00 | 0.01 | 0.04 | 0.06 | 0.07 | 0.10 | 0.12 | 0.15 | 0.18 | 0.18 | 0.20 |
| Free lime  | 2.00 | 1.95 | 1.92 | 1.85 | 1.75 | 1.72 | 1.68 | 1.61 | 1.55 | 1.50 | 1.45 |
| LSF        | 96.54 | 96.75 | 96.85 | 97.00 | 97.50 | 98.00 | 100.00 | 102.00 | 102.50 | 103.00 | 104.00 |
| SR         | 2.49 | 2.50 | 2.51 | 2.52 | 2.55 | 2.56 | 2.58 | 2.58 | 2.56 | 2.58 | 2.58 |
| AR         | 1.70 | 1.68 | 1.68 | 1.66 | 1.67 | 1.65 | 1.65 | 1.64 | 1.62 | 1.62 | 1.64 |
| CaS        | 65.92 | 65.92 | 65.95 | 65.98 | 70.15 | 70.25 | 70.35 | 70.55 | 71.55 | 71.75 | 72.00 |
| CaA        | 8.44 | 8.43 | 8.43 | 8.4o | 8.40 | 8.38 | 8.20 | 8.15 | 7.95 | 7.75 | 7.55 |
| CaAF       | 9.09 | 9.09 | 9.06 | 9.04 | 9.00 | 8.85 | 8.80 | 8.50 | 7.95 | 7.85 | 7.55 |

The minor compounds of Na2O and K2O knowns as alkalis for TGA, CSA and VPA blended cements ranges from 0.02% to 0.04%, 0.01% to 0.03%, 0.20% to 2.95% and 0.56% to 4.61%, 0.01% to 0.03%, 0.16% to 0.50% for 5% to 50%, respectively. All the values are higher than control values which are 0.03% for Na2O and 0.15% for K2O. The losses on ignition (LOI) of the blended cements (TGA, CSA and VPA) were higher than that of the control. For TGA the values ranges from 2.97% to 18.42% for 5% to 50% while for CSA it ranges from 2.87% to 18.00% and for VPA, from 2.90% to 18.35% as against 1.68% for the control. This indicates that the addition of TGA, CSA and VPA increases the organic content which has a negative effect on the binding properties of cement. However, the recommended limit of 5% by NIS 439:2000 requirements was not exceeded.

3.3 Physical Properties of the Blended Cements

Figure 1 shows the fineness of all the blended cements. The fineness of TGA blended cements ranges from 340m²/kg for 5% SDA to 531m²/kg for 50%. For CSA blended cements the fineness also ranges from 340m²/kg for 5% SDA to 528m²/kg for 50%. VPA blended cements behaved in the same manner, the fineness ranges from 345m²/kg for 5% SDA to 528m²/kg for 50%. The fineness of all the blended cements increases as the percentage of TGA, CSA and VPA (which had lower density compared to OPC-clinker and gypsum) increases. This factor contributes to the higher fineness of all the blended cements because the cements are of lower density compared to the control sample (369 m³/kg). All the blended cements satisfied the 250m²/kg and 280m²/kg minimum fineness specified by NIS 439:2000 and ASTM C 150-02, respectively.
Figure 2 shows the effect of ash replacement on residue on 45µm sieve for all the blended cements. For TGA blended cement the residue on 45µm sieve increases from 22.00% to 42.50% for 5% to 50% replacement, For CSA blended cement the residue on 45µm sieve also increases from 21.00% to 44.50% for 5% to 50% replacement and VPA blended cements behaved in the same manner, the residue on 45µm sieve increases from 20.95% to 42.50% for 5% to 50% replacement.

Figure 3 shows the initial and final setting time for all the blended cements. The initial and final setting time of the TGA blended cement increased from 45 min to 76 min and 130 min to 250 min, respectively as the percentage of TGA replacement increases from 5% to 50%. For CSA, the value increased from 46 min to 80 min and 130 min to 270 min, respectively while for VPA the value increased from 55 min to 70 min and 135 min to 268 min, respectively. This is in line with the finding of Adesanya and Raheem (2009). All the blended cements satisfy the NIS 439:2000 requirement of 45 min minimum initial setting time and maximum of 10h final setting time.

The heat of hydration of all the wood ash blended cement for six hour duration has been presented from Figures 4 to 6. It was revealed that the temperature of control sample increases consistently while all wood ash replacements levels were constant for some hours before showing slight changes in temperature. This means that, the control sample generated more heat of hydration at early stage while all other wood ash blended cements produced low heat of hydration at early stage. It can also be seen that as the percentage replacement of the wood ash increases, the heat of hydration of all the wood ash blended cements reduces.
industry: bamboo leaf ash.

Cement and Works, Ogun African Portland Cement Company (WAPCO) Sagamu

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The main conclusions derived from this investigation are as follows:

- All the hardwood ashes (TGA, CSA and VPA) are suitable material for use as pozzolan.
- The addition of TGA, CSA and VPA as pozzolan in blended cement increases marginally the oxide composition of SiO$_2$ and decrease slightly that of Al$_2$O$_3$, Fe$_2$O$_3$ and CaO.
- The TGA, CSA and VPA blended cement satisfied BS 12:1991, ASTM C 150:1994 and NIS 439:2000 requirement for up 20% replacement of ash.
- All the TGA, CSA and VPA blended cement have higher setting time than the control hence, they are most applicable where low rate of heat development is required. This shows that all the blended cement are good as low heat cement.

4. CONCLUSION
The main conclusions derived from this investigation are as follows:

- The TGA, CSA and VPA blended cement increases marginally the oxide composition of SiO$_2$ and decrease slightly that of Al$_2$O$_3$, Fe$_2$O$_3$ and CaO.
- The TGA, CSA and VPA blended cement have higher setting time than the control hence, they are most applicable where low rate of heat development is required. This shows that all the blended cement are good as low heat cement.
- All the wood ash blended cements are recommended for use where low heat cement is applicable.

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