

ENHANCING THE REACTIVITY OF CLAY POZZOLONA THROUGH
MECHANICAL ACTIVATION

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by

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Master of Philosophy in Materials Engineering

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DECLARATION

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ABSTRACT

Inadequate provision of shelter in Ghana due to the over-dependence on imported clinker has rendered about 12.5% of the population homeless. This has led to the springing up of slums, increase in communicable diseases and generally a decrease in productivity. Alternative and cheaper locally manufactured materials to Portland cement clinker such as clay pozzolana can be employed to reduce the cost of construction. The study focused on improving the reactivity of four clay deposits through mechanical activation. These deposits are at Mankranso, Mfensi, Tanoso and Mankessim.

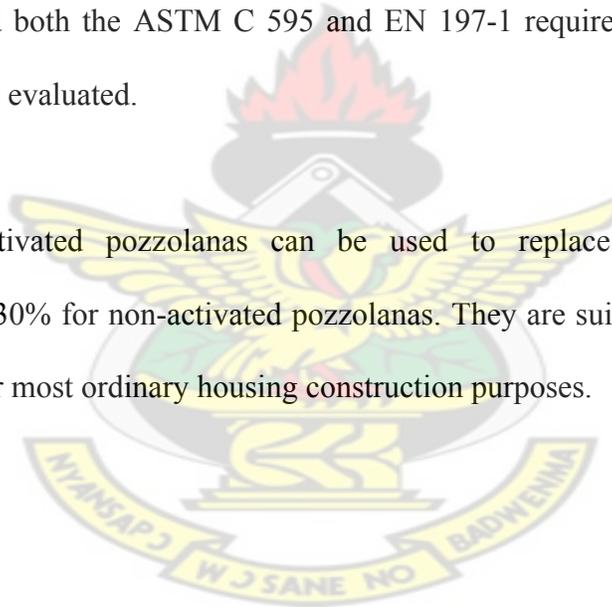
Pozzolana was produced from the clays by calcining them at 800°C. They were subjected to various degrees of milling using a hammer mill, ball mill and a Raymond type ring roll mill. After chemical analysis, the activated pozzolanas were used to replace 30-50% of ordinary Portland cement (OPC) and the physical properties such as standard consistence, setting times and compressive strength at various ages of curing of mortars determined.

All the pozzolanas were chemically suitable with total $\text{SiO}_2 + \text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ oxide contents greater than 70%, the minimum required by ASTM C 618 of natural or calcined pozzolanas. The 28-day compressive strengths of blended cements containing various amounts of the pozzolanas showed that after 36 h ball milling, most of the activated pozzolanas could be used to replace 40-50% OPC. Tanoso pozzolana attained the highest strength of 35.8 MPa followed by Mfensi (30.5 MPa); Mankranso (27.7 MPa) and Mankessim (25.6 MPa) pozzolanas at 40% cement replacement. Although lower compressive strengths were obtained at 50% cement replacement relative to strengths attained at 30-40% replacement, values were mostly above the 24.1 MPa minimum as

stipulated in ASTM C 595. The lowest value at 50% replacement of 23.1 MPa was attained by Mankessim pozzolana.

Roll milling gave higher 28-day compressive strengths than ball milling with Tanoso pozzolana gaining the highest of 36.9 MPa compressive strength; followed by Mfensi (32.1 MPa); Mankessim (30.3 MPa) and Mankranso (29.6 MPa) at 40% replacement. The highest 28-day compressive strength at 50% replacement was attained by Tanoso pozzolana at 34.2 MPa whereas the lowest was gained by Mankessim pozzolana at 23.9 MPa. The compressive strengths attained by 36 h ball milled and roll milled pozzolanas after 90 days curing satisfied both the ASTM C 595 and EN 197-1 requirements for all the pozzolana cement mortars evaluated.

Hence the activated pozzolanas can be used to replace more than the currently recommended 30% for non-activated pozzolanas. They are suitable for up to 50% of OPC replacement for most ordinary housing construction purposes.



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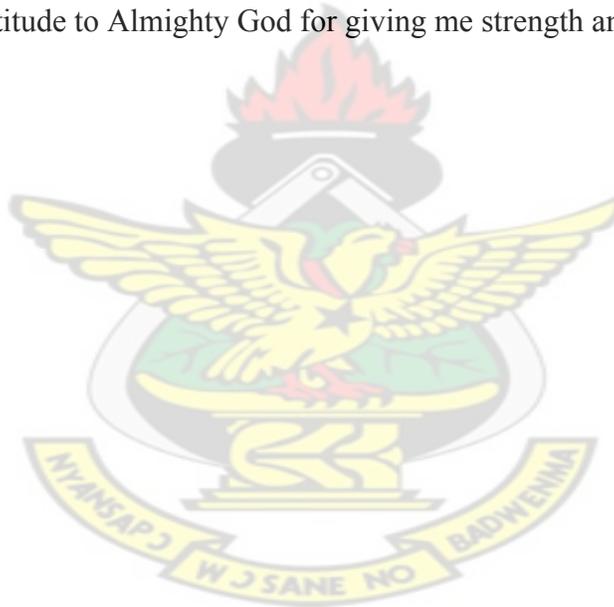


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CHAPTER ONE

1 INTRODUCTION

The problem of inadequate shelter provision in Ghana has been escalating over the years. Statistics released by the Ministry of Works and Housing in Ghana in the year 2005 showed that about 2.5 million Ghanaians had been rendered homeless due to an increase in the population without a corresponding increase in housing provision in Ghana. This figure represented 12.5% of the population of Ghana.

The statistics further revealed that the annual demand for housing was between 300,000 - 500,000 units. With a rate of between 25,000 - 40,000 units per annum, there was a housing deficit of over 200,000 units per annum (Ministry of Works and Housing, 2005). Attendant environmental and health problems such as the springing up of slums, decrease in productivity, an increase in communicable diseases and crime have all compounded the problem of housing (Ministry of Works and Housing, 2005).

To alleviate the problem, the government of Ghana proposed to put in place a housing scheme where one- and two-bedroom flats would be constructed to cater for the housing needs of the average-sized family of five members (Ministry of Works and Housing, 2005). However, the cost of these buildings at GH¢ 92,000 and GH¢ 120,000 for one- and two-bedroom flats respectively is quite high and beyond the reach of the majority of the populace in a country where the minimum wage is \$2.25 (Ministry of Finance, 2008).

The problems with housing have arisen principally due to the over reliance on imported building inputs — especially Portland cement, which is the most costly input into buildings.

It is estimated that Ghana spends over US\$100 million per annum to import clinker (Ministry of Trade and Industry, 2007). Table 1-1 gives the consumption of cement in Ghana for the period of 2000-2007 excluding small-scale imports.

Table 1-1: Cement consumption in Ghana

Year	Cement Consumption in million tonnes
2000	1.950
2001	2.050
2002	2.150
2003	2.330
2004	2.500
2005	2.700
2006	2.858
2007	3.009

(Source: Atiemo, 2006; Ministry of Trade, Industry and PSI, 2007)

Table 1-1 shows that the average year-on-year increase in cement consumption is over 5%. With the rate of population growth in Ghana at about 2.7%, the high rural-urban drift and the need to rehabilitate old houses, the demand for cement for construction will greatly increase. These factors have contributed to the high price of cement on the market — currently selling at an ex-factory price of over GH¢10 per 50 kg bag. As a solution to the above problems, the Building and Road Research Institute (BRRI) of the Council for Scientific and Industrial Research (CSIR) in Ghana has been undertaking research into developing earth-based materials as either alternatives or partial substitutes to Portland cement and its derivatives. Products such as lime-stabilized laterite blocks, lime from limestone and clamshells, burnt bricks as well as clay and bauxite waste pozzolana have thus been developed (Gogo, 1993; Hammond, 1974; Hagan, 1997; Atiemo, 1998).

Pozzolanas have been used with Portland cement to impart several desirable properties to such pozzolana-Portland cement mixes (Lea, 1970a). Work done with clay pozzolanas in Ghana indicate that by replacing approximately 30% by mass of ordinary Portland cement with burnt clay pozzolana through intimate mixing, the resulting Portland pozzolana cement (PPC) exhibits compressive strength values good for both load-bearing and non load-bearing structural applications (Atiemo, 1998, 2005). Since the production of pozzolanas is based entirely on local raw materials such as clay, the cost of a 50 kg bag of pozzolana at GH¢ 5 is much lower than the corresponding cost for a similar amount of Portland cement.

Atiemo (2005) states that the use of pozzolana to replace 30% of Portland cement results in 25% savings in housing construction. Portland pozzolana cements are noted for their slow strength development resulting in low early strengths. They also exhibit a slow rate of setting and hardening. These undesirable properties arise mainly from the slow reaction rate of the active pozzolana constituents with the liberated Ca(OH)_2 from the Portland cement (Lea, 1970a).

The reaction of the active pozzolana constituents with lime in Portland cement has been shown to depend on the degree of fineness to which they are milled. Mechanical activation (activation milling) has thus been proposed as a remedy to the slow strength development in Portland pozzolana cements. Mechanical activation is a form of mechanochemical processing – the use of mechanical energy to activate or accelerate chemical reactions and to cause changes in chemical structure of solid substances (Gaffet *et al.*, 1998). It has been employed to activate various artificial and natural pozzolanas such as fly ash and volcanic tuffs (Glinicki, 2002; Uzal and Turanli, 2003).

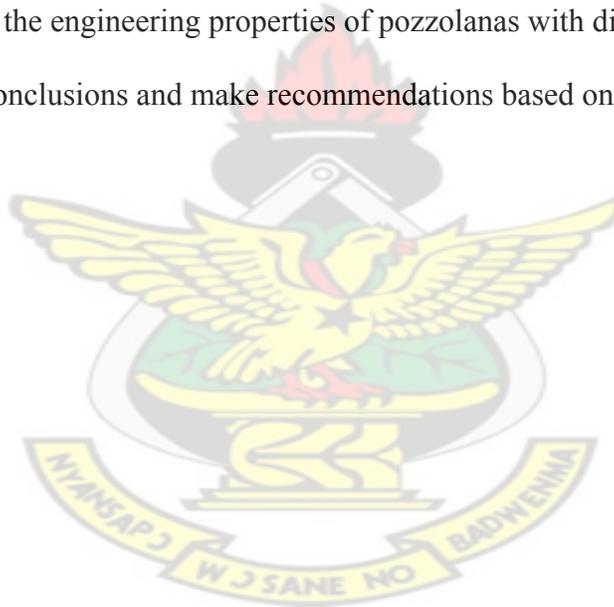
1.1 Main objective

The main objective of this study is to find out how burnt clay pozzolana can be activated by milling and hence made more effective as a partial substitute to ordinary Portland cement.

1.2 Specific objectives

To achieve the main objective, the following specific objectives were set:

- i. review literature relevant to the subject area
- ii. produce and chemically characterize pozzolanas from the clays
- iii. mechanically activate and characterize particle sizes of the pozzolanas and
- iv. test for the engineering properties of pozzolanas with different milling times.
- v. draw conclusions and make recommendations based on the results.



CHAPTER TWO

2 LITERATURE REVIEW

The term pozzolana is derived from Puzzuoli, an area in the Naples region of Italy, where the ancient Romans obtained reactive ashes for construction. The use of pozzolanas either as a partial replacement of ordinary Portland cement or with limes has gained widespread popularity in construction.

Pozzolanas have been defined as materials which though not cementitious in themselves, contain constituents which will react with lime at ordinary temperatures in the presence of water to form compounds possessing cementitious properties (Lea, 1970a). Pozzolanas can be divided into two categories namely natural and artificial pozzolanas.

2.1 Natural Pozzolanas

Natural pozzolanas are of two types: the true natural pozzolanas and the pseudo natural pozzolanas. The true natural pozzolanas are ashes and lavas originating from alkalitrachytic, leucitic, leucotephritic and hauynophric types of magma. These ashes result from explosive eruptive volcanoes and are forced to solidify as a pyroclastic glass (glass fragments formed by rapid quenching of magma produced by volcanic explosions) (Malquori, 1960).

Examples of true natural pozzolanas are the alkalitrachytic Phlegraean and leucitic Latian pozzolanas in Italy as well as Santorin earth found in Greece. On account of the high surface area, resulting from the forced expulsion of gases due to rapid quenching of magma, the true natural pozzolanas can be used in their natural state as pozzolanas without further processing and are highly reactive toward lime (Lea, 1970a; Malquori, 1960, 1964).

In the pseudo natural pozzolanas, the pyroclastic glassy minerals in the original lava have undergone hydrothermal alteration (auto-metamorphism) leading to zeolitization and sometimes argillization (Malquori, 1960; Steopoe, 1964; Kremser, 1964). These tuffaceous rock deposits when finely ground and known as trass, yield highly reactive pozzolanas such as the Romanian trass (Steopoe, 1964) or the Bavarian trass (Kremser, 1964; Lea, 1970a). Other known pseudo natural pozzolanas are the Danish “moler”, the French “gaize” as well as diatomaceous earths (Malquori, 1960; Lea, 1970a).

2.2 Artificial Pozzolanas

Artificial pozzolanas are those materials in which the pozzolanic property is not well developed and hence usually have to undergo pyro-processing before they become pozzolanic. Examples of such materials are fly ash, burnt clay, spent oil shales, calcined bauxite waste and suitably burnt fibrous agricultural residues such as rice husk ash (Hammond, 1983). Fly ash, collected from the flue gases of thermal plants and boilers that use powdered coal as fuel, is the most widely used artificial pozzolana in the world. (Lea, 1970a; Chopra *et al.*, 1964).

2.2.1 Burnt clay pozzolanas

Burnt clay pozzolanas are produced by burning suitable clays at temperatures between 600-900 °C, depending on the nature of the clay and the conditions of burning. The product is milled usually to cement fineness before it fully develops pozzolanic activity. Burnt clay pozzolana has been used to execute several construction projects such as the Bonville dam in the USA (Lea, 1970a) and the Vanivilas Sagar and Krishnarajar Sagar dams among many others in India, where the clay pozzolana was used under the name “Surkhi” (Puri and Srinivasan, 1964). An important criterion for a good burnt clay pozzolana as well as

most other pozzolanas in terms of constituents is that the sum of SiO_2 , Al_2O_3 and Fe_2O_3 contents should exceed 70% (Lea, 1940; Oza, 1964; ASTM C 618).

Research done with various Ghanaian clay deposits has shown that when milled to cement fineness, clay pozzolanas can replace up to 30% of ordinary Portland cement in structural applications. These clay pozzolana-cement mixes have been successfully used for various housing construction projects in Ghana (Atiemo, 1998; 2005). Table 2-1 shows the properties of some Ghanaian clay pozzolana cement mortars.

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Table 2-1: Engineering properties of Ghanaian clay pozzolana cement mortar

Engineering Property	Clay Sample					
	Asokwa	Hwereso	Mankranso	Mfensi	Mankessim	Nkonsia
Pozzolana content, %	30	25	30	30	30	30
Initial setting time, min	83	92	85	212	90	75
Final setting time, min	208	262	225	310	250	194
Water absorption, %	1.8	2.5	1.9	2	2.2	1.8
28-day compressive strength, MPa	31.4	24.1	29.9	30.5	29.1	28.4

(Source: Atiemo, 2005)

The minimum 28-day compressive strength as specified in EN 197-1 for OPC class 32.5N is 32.5 MPa.

2.3 Clays

Clays are defined as earthy materials with plastic properties when moist, of very fine particle size, the upper limit of which is 2 μm and mainly composed of hydrous aluminium and magnesium silicates (Gilliot, 1968).

2.3.1 The composition of clay minerals

Clays are multi-component systems in which solid, liquid and gaseous phases are commonly present. The solid phase is usually inorganic and polycrystalline with minerals being defined by their crystal chemistry. The most important crystalline components are the secondary clay minerals which have layered structures and are known as phyllosilicates (Gilliot, 1968). The structures of the phyllosilicates are composed of two fundamental crystal sheets—the tetrahedral silica and the octahedral alumina sheets. Isomorphism, polymorphism and polytypism are common in the phyllosilicates principally due to the octahedral sheet (Gilliot, 1968; Holtz and Kovacs, 1981). The many clay minerals are a result of the different ways in which the tetrahedral silica and the octahedral aluminium or magnesium sheets are bonded (Holtz and Kovacs, 1981).

The first structural variety in the phyllosilicates is one in which two-thirds of the hydroxyl ions in one plane of the octahedral layer are replaced by the apical oxygen ions of the tetrahedral layer. The remaining OH^- ions in this layer are in the centers of the hexagons formed by the oxygen ions of the tetrahedral layer. Such a combination of one octahedral and one tetrahedral layer generates a 1:1 type of structure. This is the basic structure of the kaolinite-serpentine group. The principal kaolinite group minerals are kaolinite, dickite, nacrite — all with the general formula $\text{Al}_4\text{Si}_4\text{O}_{10}(\text{OH})_8$ and halloysite $[\text{Al}_4\text{Si}_4\text{O}_6(\text{OH})_{16}]$ or $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$. Each species differs from the other in the manner in which the

structural layers are arranged with respect to each other as can be recognized through X-ray powder diffraction (Brindley, 1955).

A second tetrahedral layer may be added in a similar manner with replacement of hydroxyl ions in the sheet on the opposite side of the octahedral cation. Such an arrangement results in the formation of a 2:1 type of layer structure typical of the montmorillonite and mica groups. The montmorillonite-saponites or smectites as they are also known have the general formula $\text{Al}_4\text{Si}_8\text{O}_{20}(\text{OH})_4 \cdot n\text{H}_2\text{O}$ (or $2\text{Al}_2\text{O}_3 \cdot 8\text{SiO}_2 \cdot (2+n)\text{H}_2\text{O}$). Montmorillonite, beidellite, nontronite, saponite, hectorite and saunonite are important minerals in this group. From the viewpoint of properties and analytical recognition, the most distinctive feature of the smectites is that water and organic liquids may penetrate between the layers so that the spacing between two successive layers is variable with water uptake leading to large increase in volume (Gilliot, 1968). Another important 2:1 mineral associated with the argillaceous hydrous micas is illite ($\text{K}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 3\text{H}_2\text{O}$) which has a structure similar to montmorillonite. The major difference between montmorillonite and illite is that in illite, unlike montmorillonite, the interlayers are bonded together with a potassium atom. Additionally, there is isomorphous substitution of aluminum for silicon in the silica sheet in illites (Holtz and Kovacs, 1981).

In yet another group of minerals, known as the chlorites, there is an additional octahedral layer which is located between successive 2:1 units. Such a structure is often classified as 2:1:1 (Holtz and Kovacs, 1981; Gilliot, 1968). In the chlorites, the alumino-silicate structure is similar to that found in the 2:1 layer silicates. The chlorites however have 2:1 layers which are regularly intergrown with a brucite-like sheet of magnesium-ions which are octahedrally co-ordinated by hydroxyl ions (Gilliot, 1968).

2.3.2 Mode of occurrence of clay minerals

The formation of the clay minerals is due to two main processes namely hypogene and supergene processes. Clay minerals formed by the hypogene processes result from the action of subsurface gases, vapours or solutions that force their way upward through rocks of the earth's crust. The principal materials removed from the crustal rocks are alumina, silica, alkali or alkaline earth elements and iron. These are transformed into clay minerals at temperatures ranging between 100-450 °C in environments that may be acidic, neutral or alkaline depending on the pH of the invading vapours from the magma (Kerr, 1955). Supergene processes take place on or near the earth's surface and may require long periods unless some accelerating conditions prevail. However, even under slightly favourable conditions supergene activities such as leaching and deposition, together with weathering and soil formation take place (Kerr, 1955; Gilliot, 1968).

According to Ayetey (1977), the most important clay deposits of Ghana belong to the supergene (weathering) product type. He concluded that the clays weathered from phyllites and schists are usually very plastic with the main clay minerals being kaolinite and illite; the clays from granite are non-plastic containing predominantly quartz, a non-clay mineral with a little kaolinite; whilst the clay from shales are very plastic with montmorillonite and illite as the main clay minerals. By 1985, the Geological Survey Department of Ghana had conducted extensive survey into clay deposits and found out that clay exists in commercially exploitable quantities in every district in the country (Kesse, 1985). Table 2-2 lists the major evaluated clay deposits in the various regions.

Table 2-2: Major clay deposits in Ghana

Region	Area	Reserve, t
Greater Accra	Ada	51 242 553
	Kasseh	42 661 830
	Afiencya east	24 194 681
Eastern	Somanya	34 862 223
	Akim Swedru	33 173 335
	Abepotia	7 614 793
Central	Winneba-Kaso	51 702 127
	Ajumako	15 441 702
	Afrankwa	12 000 000
Western	Nzima-East	241 190 113
	Bokazo	221 600 000
	Essiama-Kakam	113 550 239
	Nkroful	74 456 122
Ashanti	Obuasi-Asokwa	33 865 955
	Nkawie-Aferi	2 055 900
	Kasi(Kumasi)	1 086 993
Brong Ahafo	Tanoso	16 200 200
	Sunsan valley	661 188
	Adantia	530 665
Northern	Gambibigo	12 419 998
	Koblimahago	9 455 892
	Tono	8 477 333
Volta	Kadjebi	97 742 979
	Adutor	35 854 085
	Adidome	7 755 319

(Source: Kesse, 1985).

The crystalline clay minerals in their natural state are stable, non-reactive and thus non-pozzolanic. Reactivity and hence pozzolanicity is induced by the introduction of imperfections in the crystal structure due to disorderliness and huge internal strains. This is achieved by calcining clays (Uppal and Singh, 1960).

2.3.3 Calcination of clays

When heated, the clay minerals lose most of their surface adsorbed water in the 100-120 °C temperature range. This is followed by the endothermic dehydroxylation of the clay

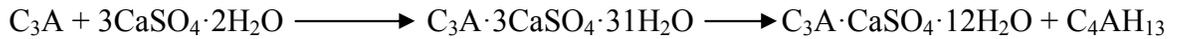
minerals. The temperature at which water is lost by dehydroxylation varies with the nature of the mineral, impurities present, the heating rate and other conditions. Illites dehydroxylate between 350-600 °C, montmorillonites dehydroxylate at about 500 °C, the reaction going to completion by about 750°C and kaolinites dehydroxylate in the 500-600 °C temperature range (Grim,1962).

Above the temperature of dehydroxylation, the clay minerals change form into other structures in exothermic processes. Kaolinite retains a two-dimensional crystal structure as metakaolinite up to about 925 °C where it changes to an Al-Si spinel phase. Above 1050 °C mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) and cristoballite (SiO_2) are formed. Illites and montmorillonites change to an anhydrous modification at about 600 °C up to 850 °C where spinel ($\text{MgO} \cdot \text{Al}_2\text{O}_3$) is formed with mullite forming at elevated temperatures above 1200 °C (Gilliot, 1968). According to the Indian Standards (IS 1334:59), kaolinites are best calcined at temperatures between 600-800 °C, montmorillonites at temperatures between 600- 800 °C, and illites at 900 °C for optimum pozzolanicity.

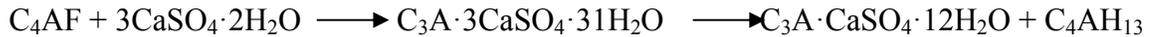
2.4 Hydration of Cement and the Pozzolanic Reaction

The reaction of cement with water is a reaction of the individual constituents of cement. These constituents are tricalcium aluminate ($3\text{CaO} \cdot \text{Al}_2\text{O}_3$ or C_3A); tetracalcium-aluminoferrite ($4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$ or C_4AF); tricalcium silicate ($3\text{CaO} \cdot \text{SiO}_2$ or C_3S) and dicalcium silicate ($2\text{CaO} \cdot \text{SiO}_2$ or C_2S). In contact with water, C_3A and C_4AF react almost instantaneously leading to the setting of the cement. Both C_3A and the C_4AF react with calcium sulphate to produce ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 31\text{H}_2\text{O}$), which hydrates further to form a solid solution of the low-sulphate sulphotoaluminate —

$3\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot\text{CaSO}_4\cdot 12\text{H}_2\text{O}$, usually existing in a solid solution with C_4AH_{13} . The overall non-stoichiometric reactions and their products can be represented as:



and



(Lea, 1970b).

Concurrently, C_3S and C_2S react with water, albeit more slowly, producing afwillite ($3\text{CaO}\cdot 2\text{SiO}_2\cdot 3\text{H}_2\text{O}$) and lime $\text{Ca}(\text{OH})_2$. This process continues up to more than one year and leads to hardening of the cement paste though there are bound to be unhydrated cement cores (Ashby and Jones, 1986). The overall non-stoichiometric reactions that lead to hardening of the cement products can be represented as:



The afwillite and the lime produced by these reactions remain in solution and in the presence of water, further hydrolyze to produce more lime in solution (Lea, 1970b). The lime thus produced is a source of weakness in cement products, especially concrete, for several reasons. The free lime has a low strength and poor stability — leading to lower strength and less durability of cement paste and concrete (Pu, 1999). The free lime, on account of its low stability, is also easily attacked by sulphate solutions.

Additionally, by a preferential growth in one direction in the presence of water, the free lime crystals may cause unsoundness in cement products especially in mass concrete

leading to disruptions and failure of structures, several years after setting (Lea, 1970b). With the addition of pozzolanas, the active silica and alumina present in the pozzolanas will have secondary reactions with the free lime, removing it from solution and producing stable calcium hydrosilicates and calcium hydroaluminates (Pu, 1999). Various reaction products, which have been identified from different pozzolanas in lime and ordinary Portland cement (OPC), are summarized in Table 2-3.

Table 2-3: Reaction products of various pozzolanas in lime and ordinary Portland cement

Pozzolana	Base	Major reaction product
Italian tuff	Lime	$4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{aq}$
Burnt clay	Lime	$3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$
Bacoli trass	Lime	$3\text{CaO} \cdot 2\text{SiO}_2$
Rhenish trass	Ordinary Portland cement	$3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4$

(Source: Lea, 1970a).

On account of the increased stability of the pozzolanic reaction products, the mechanical properties of pozzolanic cements are enhanced appreciably as compared to plain Portland cement products. A classic example of the influence of pozzolanas on mechanical properties of cement products is seen in pozzolana-cement concrete. Bentz and Garboczi (1991) showed that in plain concrete, as with other composites, there is a transition zone near the aggregate surface of up to 50 μm width. This transition zone is characterized by high porosity due to the presence of large calcium hydroxide particles.

According to Bentz and Garboczi (1991) who cite Goldman *et al.* (1989), this high porosity near the aggregate leads to low bonding near the aggregate surface than in the bulk cement

paste matrix thus introducing weaknesses in the concrete. They conclude that the formation of the calcium silicate hydrate in the pozzolanic reaction has a significant effect on the aggregate-cement paste bond improvement. Thus, the ultimate strength (obtained 365 days or beyond after production) of concrete containing fine pozzolanic materials may be higher than corresponding neat cement concrete.

Aside the increase in strength due to the pozzolanic reaction products, the removal of the free lime in set Portland cement by the active pozzolana constituents results in increased resistance to sulphate attack (Uppal and Singh, 1964). Pozzolanas, which contain little or no alkalis such as burnt clay, have been shown to improve the resistance of concrete to the alkali-aggregate reaction, which occurs when reactive aggregate such as opal, chalcedony and tridymite is used with cement containing more than 0.6% of alkali (R_2O) in concrete structures (Malquori, 1960). Pozzolanas also improve the workability, plasticity and ability to retain water in masonry cement mortars. Consequently, the extent of bond between mortar and masonry units is higher and the masonry is more resistant to cracking and rain penetration (Rehsi, 1979).

The substitution of pozzolanas for portions of Portland cement reduces the strength obtained at the earlier ages, especially up to 28 days. This effect is attributed to the low surface activity of the pozzolana at early ages. The low activity of fly ash and other pozzolanas can be attributed to two factors:

- a) The glassy surface layer of pozzolanas is dense and chemically stable. This layer prevents the interior constituents, which are porous and amorphous and therefore more reactive, from taking part in the pozzolanic reaction.

- b) The silica-alumina chain of pozzolanas is firm and must be broken if activity is to be enhanced (Yueming *et al.*, 1999).

2.5 Activation of Pozzolanas

The early strength of pozzolana cement mortar and concrete can be substantially raised by chemical, thermal or mechanical activation. Yueming *et al* (1999), working with fly ash, noted that, alkalis such as Na_2SiO_3 , when added to a mixture of fly ash and lime hydrolyze and form NaOH , increasing the hydroxide concentration and hence the pH and thus facilitating greatly the corrosion of the silica-alumina glassy chain in the pozzolana. They postulated the reactions as taking the following course:

- a) Neutralization of surface silanol groups:



This neutralization repeats on new surfaces resulting in the corrosion of the silica-alumina glassy chain of the pozzolana.

- b) The gradual destruction of the interior silane chain, resulting in $[(\text{Si},\text{Al})\text{O}_4]_n$ disintegration:



Owing to the solubility of $-\text{Si}-\text{ONa}$, they stated that, Na^+ is replaced by Ca^{2+} , forming sedimentary calcium silicate hydrate. By several repetitions of the above reactions, Na_2SiO_3 can accelerate the pozzolana activation (Yueming *et al.*, 1999). Other alkalis which have been used to excite the activity of pozzolanas, especially fly ash are $\text{Ca}(\text{OH})_2$ and NaOH as well as alkali salts such as Na_2CO_3 (Wang and Wang, 2003).

Palomo *et al.* (1999) working with high concentrations of alkalis produced cementitious compounds with a 24-hour compressive strength of 60 MPa from fly-ash at elevated temperatures of around 85°C. In their investigations, they found out that, if all other factors remained constant, temperature increase tends to result in a gain of mechanical strength in the alkaline fly ash cement. Similar hydrothermal and/or thermo chemical methods have been used to produce highly reactive pozzolanas; activation usually taking place in autoclaves (Wang and Wang, 2003).

2.6 Mechanical Activation of Pozzolana

Two distinct processes can be identified under mechanochemistry — the subject area that deals with the use of high-energy milling to change the physicochemical properties of materials. These are mechanical alloying and mechanical activation. Mechanical alloying, which is a dry and high energy-milling process, employs the transfer of mechanical energy to finely milled solid particles to effect chemical reactions at temperatures where such reactions would otherwise not be possible. Various kinds of materials from ionic to metallic have thus been synthesized including extended solid solutions, alloys of immiscible elements, quasicrystals, amorphous phases, and all sorts of compounds and composites. The synthesized materials, often with non-equilibrium structures, have typical nanometer-sized grains usually averaging 10 nm (Beke, 1981).

Besides materials synthesis, high-energy milling is a way of modifying the conditions in which chemical reactions take place by changing the reactivity of milled solids; a process known as mechanical activation (Gaffet *et al.*, 1998). In mechanical activation, the absorption of mechanical energy by fine solid particles results in the damage of grain surface and atomic structure leading to an increase in dislocation energy, which results in

increased reactivity (Beke, 1981). Most often the average size of the milled powder particles mechanically activated is not in the nanometer size range but typically in the micrometer range. In the micrometer size range, reactivity increase results through high stresses imposed by the grinding bodies acting on only a small powder-volume (Chen *et al.*, 1999; Heegn *et al.*, 2002).

Neville (1996) states that it is essential that pozzolana be in a very finely divided state as it is only then that silica can combine with calcium hydroxide in the presence of water to form stable calcium silicates which have cementitious properties. He cites the work of Kohno *et al.*, (1990) who produced a highly reactive clay pozzolana by fine-milling burnt clay to a specific surface (as determined by nitrogen adsorption) of between 4000-12000 m²/kg to support this claim. Several other investigators have reported the increased activity at early ages of various pozzolanas when finely ground to the micrometer range of particle size with Blaine index exceeding 500 m²/kg.

Puri and Srinivsan (1964) worked with three different clay pozzolanas milled to different degrees of fineness with Blaine indices ranging between 700-1200 m²/kg. They found that between 7-28 days, concrete cubes made from the finely milled clay pozzolanas attained compressive strengths greater than or equal to that of plain cement concrete designed for a 28-day compressive strength of 31.7 MPa. They thus confirmed that increasing fineness of pozzolanas resulted in increasing compressive strength.

In an investigation on a mechanically activated fly ash from fluidized bed boilers in Poland, Glinicki (2002) reported substantial increases in early age compressive strength, when a mechanically activated fly ash with patent name Flubet[®] was used to replace about 20% of

cement. The 28-day compressive strength of the 20% fly ash concrete was good enough for pavement of a 400-meter road stretch. In the same investigation it was observed that when the mechanically activated fly ash was used to replace 50% of class 32.5N cement, though the compressive strength at 7 days was affected, the strength almost equalized that of plain cement at 32.5 MPa between 28-90 days and exceeded that of plain cement concrete after two years by 18%.

Similar results have been obtained with blended cement containing volcanic tuff (Naceri and Benia, 2006). It was found out that between ages 2-90 days, compressive strength increased with increasing fineness of blended cement containing 30% of volcanic tuff. The compressive strength at 28 days of the blended cement at a fineness of over 350 m²/kg Blaine index was well above the standard 32.5 MPa (EN 197-1).

Uzal and Turanli (2003) also conducted research into blended cements containing 55% of two types of volcanic tuffs from Turkey. They milled the pozzolanas with ordinary Portland cement for 90 and 120 min respectively in a laboratory vibrating mill and obtained Blaine indices ranging between 290-598 m²/kg. Compressive strengths which they obtained, agreed with values obtained by Glinicki (2002) with 50% fly ash addition: the blended cements obtaining lower strengths than the reference Portland cement at ages up to 28 days while strength values beyond 90 days almost equalized to that of ordinary Portland cement.

The fineness of cements was also investigated by Beke (1973) who concluded that in order to produce a cement of optimum strength development a maximum percentage of the particle size fraction between 3-30 μm known as the quality control fraction is always

essential. He claimed that the percentage of the quality control fraction should be a minimum of 70%.

2.6.1 Effect of fineness on standard consistence of cements

The introduction of finely milled pozzolanas into ordinary Portland cement affects other properties of cement apart from the increase in early strength. According to Neville (1996), the degree of fineness to which cement should be milled is not only constrained by energy considerations but also other factors such as the normal consistence and the setting time of the cement. This is also true with the introduction of finely milled admixtures into ordinary Portland cement as was concluded by Uzal and Turanli (2003). They found that the water/cement ratio for normal consistence of ordinary Portland cement was increased by about 50% when up to 50% pozzolanas milled to a fineness of about 600 m²/kg Blaine index was added to the cement.

Naceri and Benia (2006) who also milled pozzolana together with ordinary Portland cement to a specific surface (by the air permeability method) of about 500 m²/kg also came to similar conclusions. In their investigation they found out that the increase in fineness not only increased the water/cement ratio for normal consistency, it also resulted in a decrease in both initial and final setting times.

2.7 Principles of Fine Grinding

The purposes for which solids are comminuted are:

- a) to decrease the size of materials,
- b) to increase the surface area of materials and
- c) to free material from its interstices (Lowrison, 1974).

Two distinct phases can be identified in comminution. These are the coarse phase of size reduction, often known as crushing, in which particles of millimeter to centimeter size are produced; and the fine phase of size reduction, known as grinding, in which product sizes range from the micrometer to millimeter range (Beke, 1981). In general, all that is necessary to break a solid is to change its shape beyond certain limits when it is subjected to tension, compression, torsion, flexure, shear or attrition beyond the elastic limit (Lowrison, 1974).

2.7.1 Energy requirement for grinding

Three classical theories have been propounded to explain the energy demand for comminution: Rittinger's theory, Kick's theory and Bond's theory.

Rittinger's theory

The first of these theories, attributed to Rittinger, deals with comminution by imaginary slicing. According to him, the energy consumed for size reduction is directly proportional to the new surface produced (Snow *et al.*, 1973).

Mathematically, Rittinger's theory is summarized as

$$E = c \left(\frac{6v^3 x_2^2}{v^3 x_2^3} - \frac{6x_1^2}{x_1^3} \right) = c_R \left(\frac{1}{x_2} - \frac{1}{x_1} \right)$$

where

x_1 = initial particle size, cm

x_2 = final particle size, cm

v = the reduction ratio defined as $v = x_1/x_2$

c and c_R = constants, and

E = energy consumed, J/cm^3 (Beke, 1981).

Kick's theory

The second theory, attributed to Kick states that for plastic deformation within the elastic limit, the size reduction from x_1 to x_2 will require the energy of

$$E = c_K \log \frac{x_1}{x_2}$$

where

c_K = the Kick's constant.

Other symbols retain definitions as stated before (Snow *et al.*, 1973).

Bond's theory

In the third theory attributed to Bond, the total energy related to a unit volume is $\frac{1}{\sqrt{x}}$. Size reduction from x_1 to x_2 will then require the energy

$$E = c_B \left(\frac{1}{\sqrt{x_1}} - \frac{1}{\sqrt{x_2}} \right)$$

where

c_B = the Bond's constant and other symbols retain their meanings.

To make the third theory more practicable, Bond defined the work index E_i , a constant for a given material as

$$E_i = E \frac{\sqrt{x_1}}{\sqrt{x_1} - \sqrt{x_2}} \sqrt{\frac{x_2}{100}}$$

where

x_1 = initial particles size, μm and

x_2 = final particles size, μm

E = the energy expended in size reduction from x_1 to x_2 , J/cm^3 (Beke, 1981).

The work index E_i is defined as the work required to reduce a unit weight of material from a theoretically infinite size to a product with 80 % passing 100 μm (Snow *et al.*, 1973).

2.7.2 Particle size distribution

Since neither the feed nor the product of milling are composed of regularly-shaped particles, the meaning of particle size in all three theories, must be given some practical significance. Two values are used in practice — x_{80} defined as the particle size giving an undersize of 80% on a particular sieve, and \bar{x} defined as the particle size giving an oversize of $100/e = 36.8$ %. This later definition, known as size module is favored because it is more practicable (Beke, 1981).

To measure the particle size distribution, various methods have been adopted such as:

- a) The wedge gauge method, which determines the largest particle present in a fine paste. It consists essentially of putting a film of the paste on the surface of an accurately ground stopper of a small taper and pushing the stopper home into the neck of an accurately ground hole of the same taper. The extent to which the stopper goes is a measure of the size of the largest particle.
- b) Sieving, in which a sample of the powder is passed through a series of sieves each made of two sets of wires of uniform cross-section which are woven at right angles to each other in such a way as to produce a square mesh of holes of uniform size. In sieving, the proportion of the original sample passing one sieve and retained on the next sieve is

determined, the complete set of such proportions being a measure of the cumulative size distribution of the particles in the sample. The smallest particle size which can be identified by this method is about 45 μm .

c) Sedimentation methods, in which the Stoke's diameter distribution of the powder is deduced from a study of concentration changes occurring within a settling suspension. The method is based on Stokes law

$$D = \sqrt{\frac{18\mu u \times 10^8}{(\rho_s - \rho_f)g}}, \text{ (Lowrison, 1974).}$$

where

μ = the viscosity of the dispersing fluid, poise;

u = the settling velocity, cm/s;

ρ_s = density of solid particles, g/cm^3

ρ_f = density of dispersing fluid, g/cm^3

D = diameter of particles, μm and

g = the acceleration due to gravity, cm/s^2

d) The pipette method, in which the concentration changes within the suspension are monitored by taking samples using a pipette. The particle-size distribution is calculated from the measured concentration changes.

e) Hydrometer method, in which a hydrometer is inserted into the suspension at time t , where it sinks to a depth h . Theoretically, the density of a settling suspension at a depth h after a time t is related to the particle size as

$$Y_{ht} = \frac{\rho_{ft} - \rho_L}{\rho_{fi} - \rho_L}, \text{ (Snow } et al., 1973)$$

where

ρ_{ft} = density of suspension at depth h and time t, g/cm³;

ρ_{fi} = initial density of suspension, g/cm³;

ρ_L = density of dispersing fluid, g/cm³

Y_{ht} = the fraction of solid particles smaller than x_{ht} which is the Stoke's diameter
obtained by substituting h and t into the Stokes equation.

Other methods such as centrifuging, elutriation and chromatography have been developed to determine particle-size distribution of powders (Snow *et al.*, 1973).

To characterise the particle-size distribution, three formulae have customarily been adopted:

Gaudin-Schumann relation

$$D(x) = \left(\frac{x}{k}\right)^m \quad (\text{Snow } et \text{ al.}, 1973).$$

Rosin- Rammler- Bennet equation

$$1 - D(x) = \exp\left[-\left(\frac{x}{x_0}\right)^n\right] \quad (\text{Beke}, 1981; \text{Snow } et \text{ al.}, 1973).$$

Logarithmic - normal function

$$D(x) = \text{erf}\left[\ln\left(\frac{x}{d}\right)^p\right]$$

where

x = particle size, μm

$D(x)$ = mass of material passing a designated sieve, g

k , \bar{x} , and d = constant parameters of the dimension lengths representing a characteristic particle size;

m , n and p = dimensionless constants related to the scatter of distribution, and

$\text{erf}(x)$ = the error integral (Gauss function).

By plotting $\ln D$, $\ln \ln \frac{1}{(1-D)}$ and $\text{erf}(x)$ as ordinates respectively, against a common abscissa of $\ln x$, all three functions can be plotted as straight lines, their slopes being m , n and p respectively.

The Rosin-Rammler-Bennet equation gives the best approach to the particle-size distribution by mass of ground products. The two constants of the Rosin-Rammler-Bennet equation are the size module, \bar{x} and the uniformity coefficient, n . The size module \bar{x} is defined as the size giving a sieve residue of 36.8% whilst the uniformity coefficient is the slope of the straight line obtained by plotting the logarithms of particle size as abscissa and $\ln \ln \frac{1}{(1-D)}$ as ordinate. On the basis of the logarithm of the particle-size as abscissa one can determine the mode of the distribution as

$$x_{md} = \bar{x} \quad (\text{Beke, 1981})$$

More importantly, the logarithmic basis for the Rosin-Rammler-Bennet equation allows a calculation of the very important value of the uniformity coefficient as:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

where R is the percentage by mass of particles retained for a particular size, x (Beke, 1973).

2.7.3 Specific surface area

Frequently, the surface area of a unit mass of the powder, known as the specific surface area is used to characterize the physical condition of powders by one value. The most important methods of determining the specific surface area of powders are:

- a) The adsorption method, especially as developed by Brunauer, Emmett and Teller popularly known as the B.E.T method. By this method the surface of the powder is made to adsorb a gas and the area accessible to the gas is calculated from the quantity of gas adsorbed. The area so determined includes all accessible cracks, crevices and pores down to 2 nm.
- b) Air permeability method, in which a closely packed bed of particles is considered as a complex system of capillaries and thus the frictional resistance to the laminar flow of air through the capillary is proportional to the area of the walls of the capillary. By measuring the pressure drop of air through the bed of particles, the related surface area can be calculated. In one type of equipment known as the Blaine apparatus, air passes through the bed of particles at a diminishing rate which causes the oil in the u-tube (manometer) to fall steadily over a prescribed length. The period of fall is timed and used to calculate the specific surface as:

$$S = K\sqrt{t}$$

where

S = the specific surface, m²/kg;

K = calibration constant of the apparatus, and

t = the time, s (Lea, 1970c).

Other methods of specific surface area determination deal with the rate of dissolution of the solid particles in solvents, the magnetic coercivity and proton beam energy loss when this is passed through a thin section of the material (Lowrison, 1974).

The specific surface can also be estimated once the particle size distribution is known by using Anselm's or Kihlsted's formulae (Lea 1970c; Beke, 1981):

Anselm's equation

$$S = \frac{368000}{\bar{x}n\rho}$$

where

S = the specific surface area, cm²/g

\bar{x} = the characteristic particle size (mode of the Rosin-Rammler distribution), μm ,

ρ = the density, g/cm³ and

n = the uniformity coefficient (gradient of the Rosin-Rammler distribution).

The Anselm's equation holds true for n=0.85-1.5

Kihlsted's formula

$$S = \frac{C}{\rho\sqrt{x_{80}}}$$

where

S = the specific surface area, cm^2/g

C = constant ranging between 500 -1000

ρ = the density, g/cm^3 and

x_{80} = the sieve aperture with 80% of the particles passing, μm (Beke, 1981).

The use of the specific surface area to characterize particle size often is too simplistic and leads to a misjudgment of the actual particle size distribution of the powder. The final strength of cement is also not determined, contrary to widely accepted opinions, by the absolute value of specific surface since above a certain value of specific surface; (approximately $5000 \text{ cm}^2/\text{g}$ Blaine index), the 28-day strength of cement is reduced (Beke, 1973).

2.8 Agglomeration Due to Fine Grinding

Two phenomena — aggregation and agglomeration — play critical roles in determining strength development when attempts at fine-milling cements are made. These phenomena can be fully investigated by considering the actual particle-size distribution of the milled products. Agglomeration is a serious hindrance to fine grinding and occurs through the welding of fissures between two successive impacts due to the presence of surface tension on both sides (Beke, 1981). Agglomeration is signaled by an increase in the size module, \bar{x} with increasing specific surface whilst the onset of aggregation is indicated by a decrease in the uniformity coefficient. Aggregation usually precedes agglomeration (Beke, 1981).

CHAPTER THREE

3 MATERIALS AND TEST METHODS

Four clay samples taken from deposits at Mankranso, Mfensi, Mankesim and Tanoso were used for the study. Ordinary Portland cement (OPC) of class 42.5N manufactured by Ghacem Company Limited, a subsidiary of Heidelberg Cement Group was used. The OPC had a Blaine fineness of 420 m²/kg. Ordinary pit sand from Fumesua, near Kumasi, was also used.

3.1 Topographical and Geological Setting of Study Areas

The map of Ghana, showing the location of the study areas referred to as M for Mankranso, Mf for Mfensi, Ma for Mankessim and T for Tanoso clay deposits is shown in Figure 3-1.

3.1.1 Mfensi clay

The clay sample from Mfensi is a grey plastic one and is deeply weathered from igneous or metamorphic rocks found in the humid zone of Ghana. Mfensi in the Ashanti Region lies about 24 km west of Kumasi on the Kumasi - Sunyani road. The clay deposit, lying along Offin river, covers an estimated area of 110,860 m² with an average thickness of 2 m and 0.2 m overburden.

3.1.2 Mankranso clay

The clay sample was obtained from Mankranso in the Ashanti Region, 36 km from Kumasi on the Kumasi-Sunyani road. The clay is derived from the weathering of the Lower Birimian (Middle Precambrian) Phyllites and is greyish in colour. The deposit area is drained by the Mankran river and has a proven reserve of about 1 million m³ with an overburden thickness of less than 0.2 m.

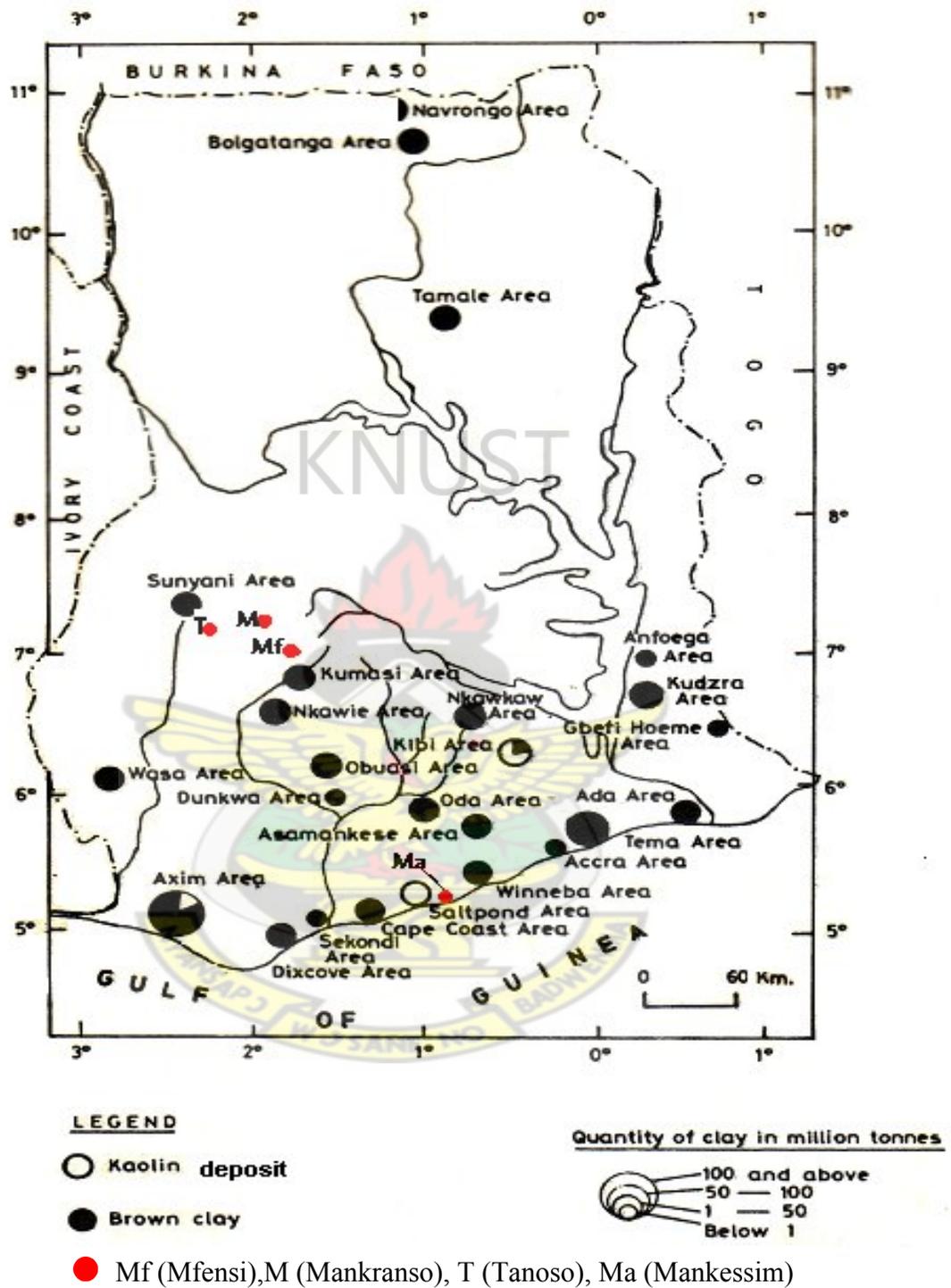


Figure 3-1: Topographical map of Ghana showing clay deposits

(Source: Kesse, 1985)

3.1.3 Mankessim clay

The clay from Mankessim is derived from the complete weathered Lower Birimian Schists and is also grey coloured. The deposit lies along the Mankessim Cape - Coast road. The deposit is drained by the Ochi river and occupies a vast area on both sides of the road. The clay deposit has a thickness of 2.5 m with an overburden thickness of 0.2 m.

3.1.4 Tanoso clay

The fourth clay was sampled at Tanoso, a village 19 km east of Sunyani on the Kumasi-Sunyani road in the Brong-Ahafo Region. The rocks in the area belong to the Voltaian Formation. The clay deposit is the residual product of the weathering of siltstone and shale in sandstone. It is brownish red in colour. The area is drained by the Tano River and extends laterally by about 49,053 m² with an average thickness of 1.2 m and 0.1 m overburden (Kesse, 1985).

3.2 Pozzolana Preparation

The clay samples were air dried and milled with a jaw crusher to an average product size of 150 µm. The milled clay samples were then thoroughly mixed with powdered palm kernel shells in a ratio of 8 parts of clay to 1 part of palm kernel shells by volume (Atiemo, 2005) in a mechanical mixer. The clay and palm kernel shell mixture was then formed into nodules in a rotating pan nodulizer by adding water as a fine spray. The nodules were air-dried and fed into a shaft kiln at the Building and Road Research Institute (BRRI) fitted with blowers. The nodules were calcined at 800°C by transferring heat from a bed of previously ignited charcoal with a stream of air blown through the kiln with electric blowers. The average temperature of the bed of nodules was determined with a

thermocouple. Calcination automatically stopped once the palm kernel shell content of the nodules had been consumed.

3.3 Milling of Pozzolanas

The fired nodules were first crushed with a hammer mill into an average particle size of 80% passing a 100 μm sieve and divided into two portions. One portion of the crushed pozzolana was milled for batch periods of 6 h in a 1 m long, 30 cm inner diameter ball mill with pebbles after which samples were taken from the ball mill and analyzed for significant change in the fineness as determined by hydrometer sedimentation. After 36 h milling, the samples were analyzed every 2 h until agglomeration was observed. The other portion of crushed pozzolana was milled in a Raymond type ring-roll mill and the particle size distribution of product also determined.

3.4 Physical Properties

3.4.1 Specific gravity

The specific gravity of the pozzolanas prepared were determined as specified by the British Standard BS 1377:90. A 50 ml density bottle with the stopper on was dried in an oven and weighed (m_1). 25g of the pozzolana samples was oven-dried at 105°C for 24 h. The pozzolana was transferred into the density bottle and the bottle with its contents and the stopper weighed again (m_2). Then the pozzolana in the bottle was covered with kerosene of known specific gravity. With the stopper removed, the container was placed in a vacuum desiccator, and the air gradually evacuated. When no more air was seen to be released, the bottle was removed from the dessicator and filled with de-aired kerosene. The stopper was again inserted and the bottle immersed in a water bath until a constant temperature of 25°C was attained. The bottle was then wiped dry and weighed (m_3). The bottle was afterwards

emptied of its contents, cleaned and completely filled with kerosene, stoppered and reweighed (m_4).

The specific gravity (G_s) of the sample was calculated as:

$$G_s = \frac{G_L(m_2 - m_1)}{(m_4 - m_1) - (m_3 - m_2)}$$

where G_L = specific gravity of kerosene = 0.80

3.4.2 Bulk density

The bulk densities of the milled pozzolanas were determined with a Ravenhead Kilner[®] density cylinder. The cylinder had a volume of 1000 ml. Approximately 1 kg of the sample was weighed and poured into the previously weighed density jar. The top of the sample was leveled off with a straight edge, covered and re-weighed. The bulk density was calculated as

$$\rho_b = \frac{W_D - W_C}{1000}$$

where

W_D = mass of jar and sample, g

W_C = mass of empty jar

ρ_b = bulk density, g/cm³.

3.4.3 Particle size distribution

The particle size distribution of the clay pozzolanas was determined by the hydrometer method of sedimentation as specified by British Standard BS 1377:90. 200 g of pozzolana

was dried in an oven at 105°C for 4 h. The dry sample was then quartered and 50 g of it was transferred into a 600 ml brass container. 100 ml of a dispersant solution, made from 7 g of anhydrous sodium metahexaphosphate (NaPO_3)₆ and 33 g of anhydrous sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$) dissolved in 1 litre of distilled water, was added to the pozzolana and made up to 250 ml with distilled water.

The suspension was agitated with a mechanised stirrer for 15 min and later transferred to a 1 litre measuring cylinder. The contents were made up to 1 litre with distilled water and left to stand undisturbed for 24 h to effect the decoagulation of the various particles. The cylinder was agitated manually by holding the measuring cylinder containing the suspension between the palms and turning it upside-down for a minute to disperse the particles and placed on a bench. The timer was immediately switched on. The hydrometer readings were first taken at 30 s and then at 1, 2 and 4 min and specific intervals for 8 h and at 24 h. After the hydrometer readings, the sample was washed through a 75 μm sieve and the material retained, if any, were dried at 105°C for 24 h. The dry sample was passed through 0.6, 0.4 and 0.2 mm, 150 and 75 μm standard sieves and the retained on each sieve recorded.

3.4.4 Blain index

The Blaine indices of the milled pozzolana samples and OPC were determined with an air permeability apparatus at the laboratories of Ghacem Co. Ltd. according to methods outlined by ASTM C 204. The apparatus consists of a manometer filled with a non-volatile light grade mineral oil with a cell for holding the samples attached at the top. The apparatus had a calibration constant of 39.813. The cell was filled with 2.715 g of the sample to be analyzed for specific surface area and a filter paper disk placed on top of the bed. The

sample was compacted with a plunger after which the cell was placed on top of the manometer. Air was evacuated from one arm of the manometer until the liquid reached the top mark and allowed to drop to the second mark next to the top. The timer was started and the duration of travel of the liquid from the second to the bottom marks determined in seconds. The Blaine index was calculated as:

$$S = K\sqrt{t}$$

where

S = specific surface area or Blaine index, m²/kg;

K = calibration constant of the apparatus = 39.813

t = time, s

3.5 Chemical composition

The chemical analysis of the clay pozzolanas and Portland cement was carried out according to methods recommended by Standard Methods of Chemical Analysis (Welcher, 1963) and by the European Standard EN 196-4:2000, both of which employ the gravimetric methods. 100 g of each of the pozzolana samples was oven dried at 105°C for 4 h and two parallel determinations were made for each constituent. The results were averaged and reported.

3.5.1 SiO₂ content

Approximately 1 g of the milled pozzolana sample was weighed out on an analytical balance and placed into a beaker. 1 g of ammonium chloride (NH₄Cl) was added to it and mixed intimately. 10 ml of concentrated HCl (specific gravity of 1.19) and 10 drops of

concentrated HNO_3 (specific gravity of 1.42) was added to the mixture and the solution stirred to effect dissolution.

The beaker was heated over a hot water bath for a period of about 30 min and covered with a watch glass to avoid spurting. The gelatinous material formed was then transferred to a filter paper and washed with hot HCl (1+99). The filtrate was reserved for the determination for the total Al_2O_3 and Fe_2O_3 content. The filter paper and precipitate from the filtration were transferred into an ignited and weighed platinum crucible. The crucible was heated gradually on a hot plate to char the paper and later ignited in a muffle furnace at 1200°C for 30 min. The hot crucible was transferred into a dessicator to cool and then weighed (m_1).

The contents of the cold crucible were moistened with water, 5 drops of H_2SO_4 (1+1) and 10 ml of 40% HF solution. The contents were then evaporated to dryness. The crucible and residue were then heated and ignited in the furnace. The crucible was cooled in a dessicator and weighed (m_2). The difference in mass, $m_1 - m_2$, gave the mass of silica (SiO_2) in the sample. 0.5g of sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) was added to the residue from the hydrofluoric acid treatment and heated below red heat to dissolve the melt. The mixture was then cooled, dissolved in water, added to the filtrate reserved for the total Al_2O_3 and Fe_2O_3 determination and made up to 200 ml.

3.5.2 Combined Fe_2O_3 and Al_2O_3 content

To the filtrate obtained in the SiO_2 determination 10 ml of concentrated HCl and 3 drops of methyl red indicator were added and boiled. Ammonia solution, NH_4OH (1+1) was added to it drop wise while stirring until precipitation appeared to be complete. One drop of

NH_4OH (1+1) was added to make the solution just alkaline and to produce a distinctly yellow coloration. The alkaline solution was digested for 2 min on a steam bath, allowed to stand for 5 min for the precipitate to settle and filtered through an ashless no. 40 Whatman filter paper.

The precipitate was washed four times with hot 20 g/l NH_4Cl solution. The filtrate was reserved and the precipitate transferred back to the precipitation beaker. The paper was moistened and the precipitate dissolved with hot HCl (1+3) and washed thoroughly with water, adding these washings to the precipitation beaker. This solution was re-precipitated as before. The precipitate was filtered through another ashless no. 40 Whatman filter paper and washed free from chlorides with hot NH_4Cl solution as before. The precipitate and filter papers were reserved for ignition.

The combined reserved filtrate was acidified with concentrated HCl and evaporated to about 100 ml. It was then made just alkaline to methyl red with 50% NH_4OH solution. Any precipitate was filtered off, re-precipitated, washed as before and reserved for ignition. The solution or the filtrate and washings was acidified with concentrated HCl and reserved for the determination of CaO and MgO . The precipitates and papers were placed in a weighed platinum crucible and heated slowly on a hot plate to dry the precipitates and char the papers. It was finally ignited at 1050°C for 30 min in a muffle furnace. The mass of combined Fe_2O_3 and Al_2O_3 was thus obtained.

3.5.3 Fe_2O_3 content

Approximately 1 g of the oven-dried pozzolana was weighed into a 500 ml beaker. 40 ml of distilled water and 10 ml of HCl (1+1) were added to the sample and heated to dissolve it.

When the solution started boiling, 50 g/l stannous chloride (SnCl_2) solution was added drop by drop from a pipette with constant stirring until the solution became colorless. One drop of SnCl_2 solution in excess was added and the solution was cooled quickly under tap water. Then 10 ml of saturated mercuric chloride (HgCl_2) solution was added at a go. The solution was stirred vigorously for 1 min and 10 ml of concentrated phosphoric acid (H_3PO_4) and 2 drops of barium diphenylamine sulphonate indicator added to it. The solution was made up to 100 ml and titrated with 0.008 M standard potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) solution, which was added very slowly while stirring constantly, until a permanent purple end point was obtained (1 ml of the $\text{K}_2\text{Cr}_2\text{O}_7$ is equivalent to 0.004g Fe_2O_3). A blank determination following the same procedure and using the same amounts of reagents was also made.

The amount of ferric oxide present was calculated as:

$$\text{Fe}_2\text{O}_3, \% = 0.004 (V-B) \times 100$$

where

V = volume of $\text{K}_2\text{Cr}_2\text{O}_7$ used, ml

B = volume of $\text{K}_2\text{Cr}_2\text{O}_7$ for blank determination, ml

3.5.4 Al_2O_3 content

The difference between the mass of the combined oxides determined in 3.4.2 and the mass of ferric oxide gave the mass of alumina.

3.5.5 CaO content

The filtrate reserved after determination of the combined ferric oxide and alumina was neutralized to the methyl red end point with concentrated HCl and made acidic with 6 drops of concentrated HCl in excess. The solution was then evaporated to 100ml and 40 ml of

saturated bromine water added to it. Concentrated ammonia solution was then added until the solution became distinctly alkaline. The solution was then boiled for 5 min and the precipitate allowed to settle. It was allowed to cool and stand for 2 h, and then filtered through a no. 40 ashless Whatman filter paper. The precipitate, impure MnO_2 , was discarded and the filtrate acidified with conc. HCl and boiled to expel the bromine. Then 5 ml of concentrated HCl was added, diluted to 200 ml and 30 ml of warm 50 g/l ammonium oxalate, $[(\text{NH}_4)_2\text{C}_2\text{O}_4]$ solution added to the solution. The solution was heated to 80°C and precipitated with ammonia solution as before. The solution was allowed to cool for 1 h and then filtered through a no. 40 Whatman ashless filter paper. The precipitate was washed thoroughly with cold 1 g/l ammonium oxalate solution and the filtrate reserved.

The filter paper was transferred to a beaker and the precipitate dissolved in 50 ml of hot HCl (1+4) and the paper macerated. The solution was diluted to 200 ml with water and 20 ml of 50 g/l ammonium oxalate solution, heated and precipitated with concentrated ammonia solution as before. The filtrate and washings were combined with the previous filtrate reserved for the MgO determination and acidified with concentrated HCl. The precipitate and papers were ignited in a weighed platinum crucible, slowly at first to burn off the carbon and finally at 1100°C for 30 min. The crucible was cooled in a desiccator and weighed to obtain the mass of CaO.

3.5.6 MgO content

The acidified filtrate from the previous determination was concentrated to about 250 ml. 10 ml of 250 g/l ammonium hydrogen phosphate $[(\text{NH}_4)_2\text{HPO}_4]$ solution was added to the solution and cooled to room temperature. After cooling, concentrated ammonia solution was added drop by drop while stirring constantly until the precipitate began to form, and

then the reagent added in moderate excess while stirring continued for several minutes. The solution was set aside for at least 2 h in a cool atmosphere and then filtered through an ashless no. 42 Whatman filter paper. The filter paper was unfolded and the precipitate washed with hot water into a beaker. It was later rinsed with hot HCl (1+4) to dissolve the precipitate and again with hot water. The solution was diluted to 100 ml, precipitated as before and washed with 100 g/l ammonium nitrate (NH_4NO_3) solution. The filter paper was placed in a weighed platinum crucible, slowly charred on a hot plate, then ignited at 1100°C for 30 minutes in a muffle furnace, cooled in a desiccator and weighed to give the mass of MgO.

3.5.7 Sulphur content (expressed as SO_3)

Approximately 1 g of the oven-dried clay pozzolana sample was weighed into a beaker and 25 ml of cold distilled water was added to it. While the mixture was being stirred vigorously 5 ml of concentrated HCl was added to it. The solution was diluted to 50 ml and digested for 15 min at a temperature just below boiling. It was then filtered through a no. 40 Whatman filter paper and the residue washed thoroughly with hot water. The filtrate was diluted to 250 ml and heated on a hot water bath with a stirrer. On boiling, 10 ml of hot 100 g/l barium chloride (BaCl_2) solution was added slowly, drop wise and left to boil until the precipitate, barium sulphate (BaSO_4), was well formed.

The solution was then digested for 12 h at a temperature just below boiling and filtered through a no. 42 Whatman ashless filter paper. The precipitate was washed with hot water, placed with the paper in a weighed platinum crucible, slowly charred on a hotplate to consume the paper without inflaming and ignited at 800°C in a muffle furnace for 30 min. The crucible was placed in a desiccator to cool and weighed.

The amount of sulphur present expressed as SO₃ was calculated as:

$$\text{SO}_3 = W \times 34.3, \%$$

where

W = weight of BaSO₄ precipitate, g

34.3 = molecular ratio of SO₃ to BaSO₄

3.5.8 Loss on ignition

4 g of the finely ground pozzolana sample was weighed and oven dried at 110°C for 18 h. Approximately 1 g of the dry sample was then transferred into a weighed platinum crucible. The crucible was placed in a muffle furnace and slowly heated and ignited to 1000°C for 30 min. The crucible was then transferred into a desiccator to cool and weighed afterwards. The difference in mass represented the loss on ignition.

3.6 Engineering Properties

3.6.1 Compressive strength

The tests for the determination of the compressive strength of the pozzolana cement mortar cubes were carried out according to methods specified by the European Standard EN 196-1:2000. 4 different milled pozzolanas were used for the experiments: the product of the hammer mill, the roll mill and 2 products from the ball mill (one after 24 h milling and the other after 36 h milling). The milled clay pozzolanas were blended with ordinary Portland cement using cement replacement of 30, 35, 40 and 50% by mass.

River sand was used to prepare mortar cubes for the compressive strength determination. The sand satisfied the requirements of the European Standard EN 196-2:2000, that is it

passed through a 0.8 mm sieve and not more than 10% passed through a 0.6 mm sieve. Pozzolana cement to sand ratio of 1:3, water to pozzolana-cement ratio of 0.5 for all levels of Portland cement replacement were used to prepare the mortar test cubes. The same 1:3 water to cement ratio was used to produce the control cubes using only ordinary Portland cement. The materials for each mortar cube were mixed separately. The pozzolana and the cement were mixed first in a motorized mixer and then mixed with the measured quantity of sand. Afterwards, the quantity of water needed was added and mixed for about 4 min comprising 3 min at low speed and 1 min at high speed mixing.

The mortar cubes were moulded in 75×75 mm metal moulds as well as in 70×70 mm metal moulds at an average room temperature of 27°C and 80% relative humidity. The moulding involved tamping the mortar in the moulds and then vibrating them on a vibrating machine for 2 min. After gauging, the moulds were covered with a metal plate and placed under damp jute sacks. The pozzolana cement cubes were removed from their moulds after 24 h and immersed in a pond of water of average temperature of 22°C and kept for the specified curing periods of 2, 7, 28 and 90 days. The cubes after curing were tested for compressive strength by loading the sides of the cubes uniformly with a compressive strength testing machine until fracture occurred. For each crushing test 5 cubes were used and the average taken for the compressive strength. The maximum load in kN at which fracture occurred was recorded and used to calculate the compressive strength as

$$R_c = \frac{1000F_c}{A_r}$$

where

R_c = compressive strength, MPa

F_c = maximum load at which fracture occurs, kN

A_r = area of a face of the cube, mm^2 (EN 196-1: 2000).

3.6.2 Standard consistence

The Vicat method as specified by the European Standards EN 196-3:2000 was used to determine the standard consistence of both pozzolana and ordinary Portland cement pastes. In this method a measured quantity of the cement is gauged with water until the resistance to penetration of a plunger reached a specified level. The ratio of the water required to the weight of cement and expressed as a percentage is taken as the standard consistence of the cement. The plunger, made of brass, is of at least 45 mm effective length and 10 mm diameter. The Vicat mould, also made of brass, is in the form of a truncated cone with a depth of 40 mm and an internal diameter of 75 mm.

The Vicat mould, resting on a non-porous plate, was filled completely with the cement paste in one layer and the surface smoothed off level with the top of the mould as quickly as possible. The test block, confined in the mould and resting on the plate was placed under the rod bearing the plunger. The plunger was lowered gently into contact with the surface of the test block and quickly released and allowed to sink in.

Trial cement pastes, made with varying amounts of water, were used until the plunger penetrated a distance of not more than 5 mm from the bottom of the mould. The amount of water used was recorded and expressed as a percentage by mass of the dry pozzolana cement to give the standard consistence of the pozzolana cement (EN 196-3: 2000).

3.6.3 Setting time

Initial setting time

The Vicat method was also used for the determination of the initial setting time. The pozzolana cement paste, using the percentage of water recorded for the standard consistence, was gauged into the Vicat mould. The paste, confined in the mould and resting on the base plate, was placed under the Vicat apparatus with the needle provided for the initial setting time determination in place.

The needle was lowered gently into contact with the surface of the test block and quickly released and allowed to sink in. This process was repeated until the needle, when brought into contact with the test block and released as described above, did not penetrate beyond a point approximately 4 mm from the bottom of the mould. The period elapsing between the time when the water was added to the cement and the time at which the needle ceased to pierce beyond 4 mm from the bottom of the test block was noted as the initial setting time (EN 196-3: 2000).

Final setting time

For the determination of the final setting time, the needle used for the initial setting time was replaced by the needle with an annular attachment. The cement block was also turned upside-down for the determination. The cement paste was considered as finally set when, upon applying the needle gently to the surface of the test block, only the needle made an impression, while the attachment failed to do so. The time from gauging, at which the needle ceased to pierce the test block, as described above, was noted as the final setting time (EN 196-3: 2000).

CHAPTER FOUR

4 TEST RESULTS AND DISCUSSION

4.1 Chemical Composition

Table 4-1 shows the chemical composition of the clay pozzolana samples and ordinary Portland cement (OPC).

Table 4-1: Chemical composition of pozzolana samples and OPC

Constituent,%	Mankranso Pozzolana	Tanoso Pozzolana	Mfensi Pozzolana	Mankessim Pozzolana	OPC
SiO ₂	62.03	62.34	61.77	65.85	19.70
Al ₂ O ₃	16.66	18.36	18.71	18.34	5.00
Fe ₂ O ₃	12.98	10.98	11.68	9.48	3.16
CaO	0.21	0.25	0.25	0.72	63.03
MgO	1.75	1.40	1.46	0.13	1.75
SO ₃	0.16	0.19	0.19	0.04	2.80
LOI	2.97	3.64	2.75	3.82	2.58
Others*	3.24	2.84	3.19	1.62	1.98
TOTAL	100.00	100.00	100.00	100.00	100.00

* These are the oxides of trace elements such as Na₂O, K₂O, TiO₂ and MnO which needed atomic absorption spectrophotometry to be determined. The values indicated were obtained as the difference between 100 and the sum of the determined constituents.

The SiO₂ content of all the pozzolanas exceeded the minimum limit of 25.0% as prescribed by EN 197-1: 2000. The sum of SiO₂, Al₂O₃ and Fe₂O₃ also exceeded the minimum of 70% prescribed by ASTM C 618 for all the pozzolana samples. Thus the materials are chemically suitable as pozzolanas. The loss on ignition was also below the maximum limit

of 5.0% required of pozzolanas as specified in EN 197-1:2000. The OPC used had a loss on ignition of 2.58% which also satisfied the maximum 3.5% allowed by EN 197-1:2000. Since the ratio of Al_2O_3/Fe_2O_3 for the OPC ≥ 0.64 (i.e. 1.58), the modified Bogue equation as presented in ASTM C 150: 2000 can be used to calculate the mineralogical composition of the OPC as follows:

$$C_3S = (4.071 \times CaO) - (7.600 \times SiO_2) - (6.718 \times Al_2O_3) - (1.430 \times Fe_2O_3) - (2.852 \times SO_3)$$

$$= 60.78\% \approx 61\%$$

$$C_2S = (2.867 \times SiO_2) - (0.7544 \times C_3S) = 10.62\% \approx 11\%$$

$$C_3A = (2.650 \times Al_2O_3) - (1.692 \times Fe_2O_3) = 7.90\% \approx 8.0\%$$

$$C_4AF = 3.043 \times Fe_2O_3 = 9.62\% \approx 10\%.$$

4.2 Physical Properties

4.2.1 Specific gravities

The specific gravities of the pozzolanas and OPC are presented in Table 4-2.

Table 4-2: Specific gravities of pozzolanas and OPC

Sample	Specific gravity
Mfensi pozzolana	2.56
Mankranso pozzolana	2.56
Mankessim pozzolana	2.54
Tanoso pozzolana	2.82
OPC	3.21

4.2.2 Particle size distributions

Tables 4-3 – 4-6 give the particle size distribution data for the pozzolanas for the various types of milling. The particle size distribution curves drawn from these data are presented in Figures 4-1, 4-3, 4-5 and 4-7 whilst Figures 4-2, 4-4, 4-6 and 4-8 show the Rosin-Rammler distribution curves. Based on the particle size distributions, the specific surface areas were calculated using Anselm's formula as presented in appendices 1-4. The uniformity coefficients, n and the characteristic particle sizes, \bar{x} were read from the Rosin-Rammler distributions and are presented together with the particle size data in Tables 4-3 - 4-6. These tables contain the Blaine indices and bulk densities as well.

Mfensi pozzolana

For ball milling, the highest Blaine index achieved for Mfensi pozzolana was 1128 m²/kg at 36 h, after which agglomeration started and the fineness reduced in the mill. Roll milling gave a finer particle size with a Blaine index of 1405 m²/kg. The characteristic particle size obtained by roll milling was also much smaller, than the smallest attained after 36 h of ball milling (7 µm and 12 µm respectively).

Hammer milling was the least efficient, giving a Blaine index of only 325 m²/kg and a characteristic particle size of 45 µm. The calculated specific surface area using Anselm's formula was always smaller than the measured value using the air permeability (Blaine) apparatus for all the milled pozzolana samples.

Table 4-3: Particle size distribution, bulk density, specific surface area, uniformity coefficient and characteristic particle size for Mfensi pozzolana

X, μm	Hammer milling, D, %	Roll milling, D, %	Ball milling, D, %		
			24 h	36 h	42 h
300	100	-	-	-	100
150	99.8	-	-	-	98.6
75.0	88.4	100	100	100	90.6
60.8	76.1	-	-	-	-
54.0	-	-	-	99.9	-
53.5	-	-	98.9	-	-
52.8	-	99.8	-	-	-
44.5	62.8	-	-	-	72.8
39.0	-	-	95.6	96.9	-
38.2	56.7	-	-	-	65.9
37.8	-	96.9	-	-	-
32.6	-	-	-	-	58.6
29.8	-	93.4	-	-	-
28.6	-	-	86.5	89.0	-
24.2	-	88.5	-	-	-
23.9	46.2	-	-	-	50.6
21.0	-	-	78.6	83.0	-
19.3	-	84.3	-	-	-
17.4	40.4	-	-	-	44.7
15.0	-	-	68.5	72.5	-
13.5	36.0	76.0	-	-	38.7
12.0	-	-	60.2	66.5	-
9.5	29.4	-	-	58.1	33.3
8.5	-	62.5	52.6	-	-
6.5	22.9	-	42.6	48.2	26.5
5.2	-	52.0	-	42.6	-
5.0	18.0	-	36.1	-	19.7
3.5	13.2	44.4	28.0	32.1	13.2
1.5	0.6	25.2	20.0	20.6	0.6
Bulk Density, kg/m^3	<i>1083</i>	<i>453</i>	<i>964</i>	<i>824</i>	<i>987</i>
Blaine Index, m^2/kg	<i>325</i>	<i>1405</i>	<i>897</i>	<i>1128</i>	<i>760</i>
Anselm's Fineness, m^2/kg	<i>293</i>	<i>1317</i>	<i>879</i>	<i>1079</i>	<i>364</i>
Characteristic particle size (\bar{x}), μm	<i>45</i>	<i>7.2</i>	<i>15</i>	<i>12</i>	<i>38</i>
Uniformity coefficient (n)	<i>1.1</i>	<i>1.5</i>	<i>1.1</i>	<i>1.0</i>	<i>1.1</i>

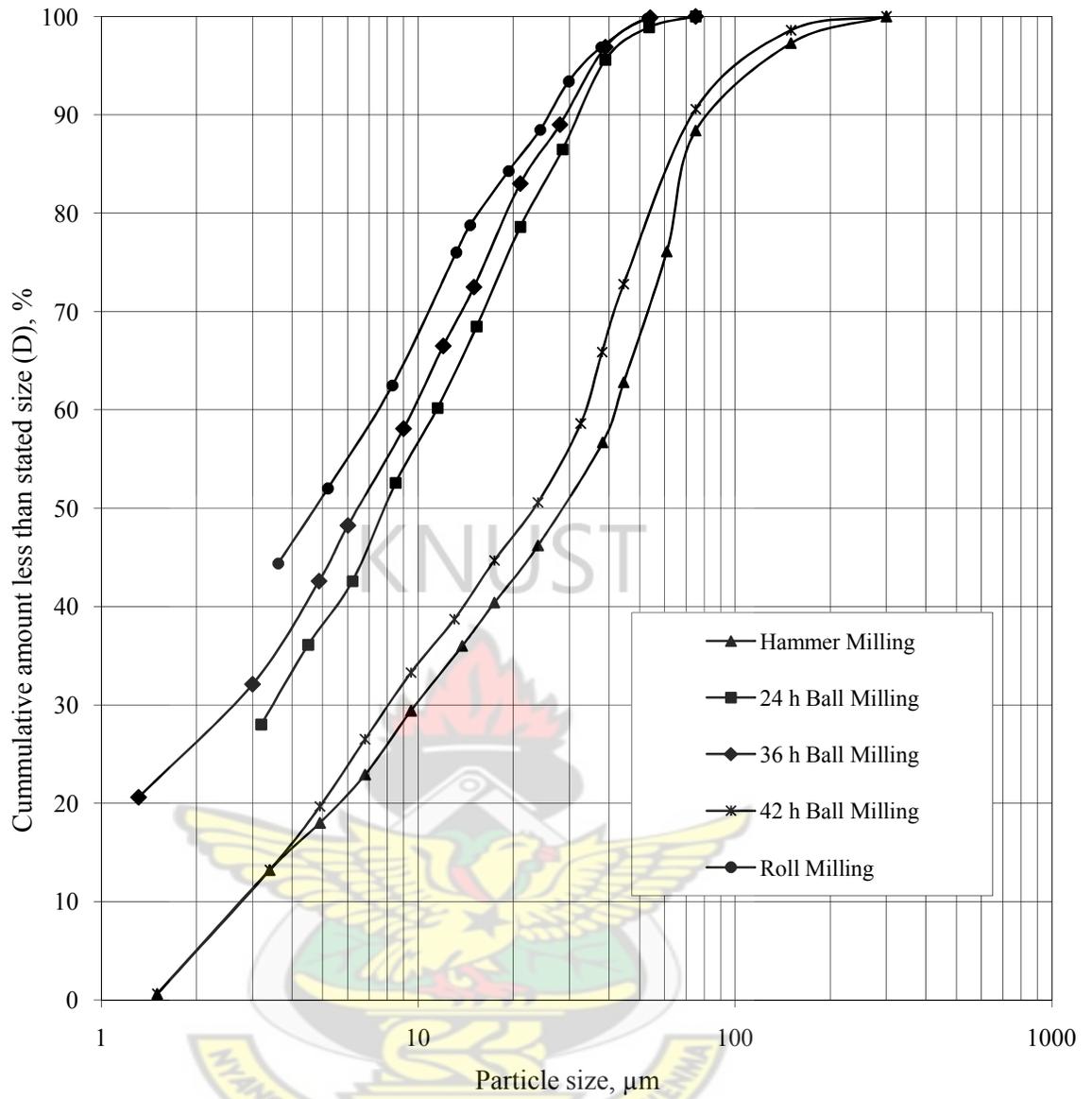


Figure 4-1: Particle size distribution curves of Mfensi pozzolana

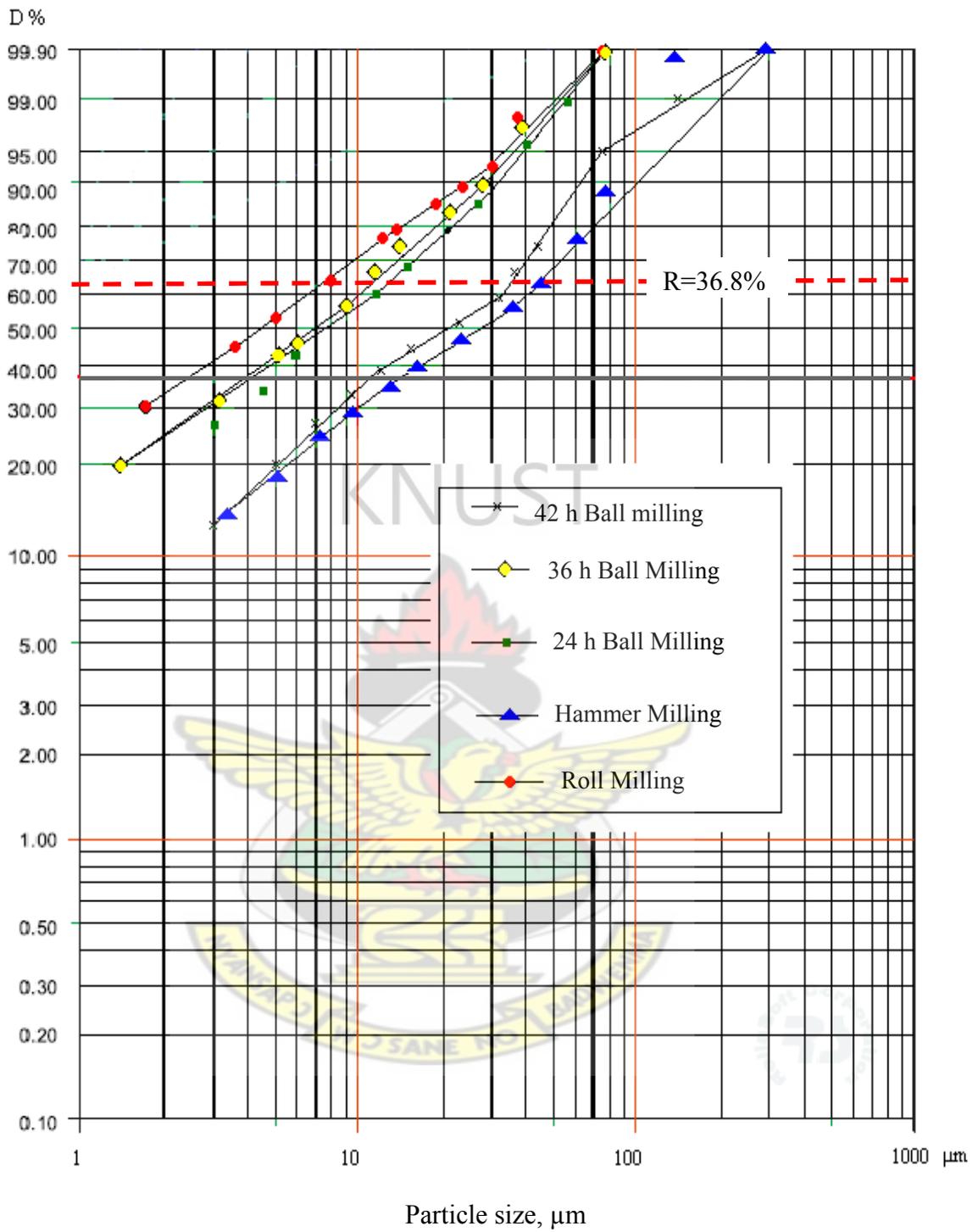


Figure 4-2: Rosin – Rammler distribution curves for Mfensi pozzolana

Mankranso pozzolana

Table 4-4: Particle size distribution, bulk density, specific surface area, uniformity coefficient and characteristic particle size for Mankranso pozzolana

X, μm	Hammer milling, D,%	Roll milling, D,%	Ball milling, D, %		
			24 h	36 h	42 h
300	100	-	100	-	100
150	-	-	99.6	-	-
75.0	-	100	95.2	100	-
57.0	75.6	-	-	-	78.4
53.5	-	-	90.0	-	-
52.8	-	98.6	-	-	-
51.3	66.8	-	-	-	62.8
44.8	-	-	-	96.0	-
39.0	-	-	82.3	-	-
37.5	54.9	90.6	-	-	58.9
34.0	-	-	-	92.0	-
29.8	-	72.8	-	-	-
29.0	-	-	74.1	87.6	-
23.0	45.1	65.9	-	-	48.1
21.0	-	58.6	64.9	-	-
18.5	40.6	-	-	76.9	42.6
16.9	-	44.7	55.7	-	-
15.0	-	50.6	60.3	69.6	-
12.0	-	-	-	62.5	-
9.5	27.6	-	46.2	56.1	23.6
8.3	-	38.7	-	-	-
7.0	20.8	-	38.5	45.6	20.8
5.2	-	33.3	-	42.2	-
4.9	16.0	-	32.6	-	18.0
3.5	10.3	26.5	27.0	31.2	13.3
1.5	0.3	-	16.3	19.5	0.4
Bulk Density, kg/m^3	<i>1052</i>	<i>425</i>	<i>953</i>	<i>685</i>	<i>984</i>
Blaine Index, m^2/kg	<i>349</i>	<i>1395</i>	<i>892</i>	<i>1025</i>	<i>743</i>
Anselm's Fineness, m^2/kg	<i>349</i>	<i>1123</i>	<i>814</i>	<i>943</i>	<i>371</i>
Characteristic particle size(\bar{x}), μm	<i>18</i>	<i>10</i>	<i>19</i>	<i>12</i>	<i>38</i>
Uniformity coefficient (n)	<i>1.0</i>	<i>1.1</i>	<i>0.9</i>	<i>1.1</i>	<i>1.0</i>

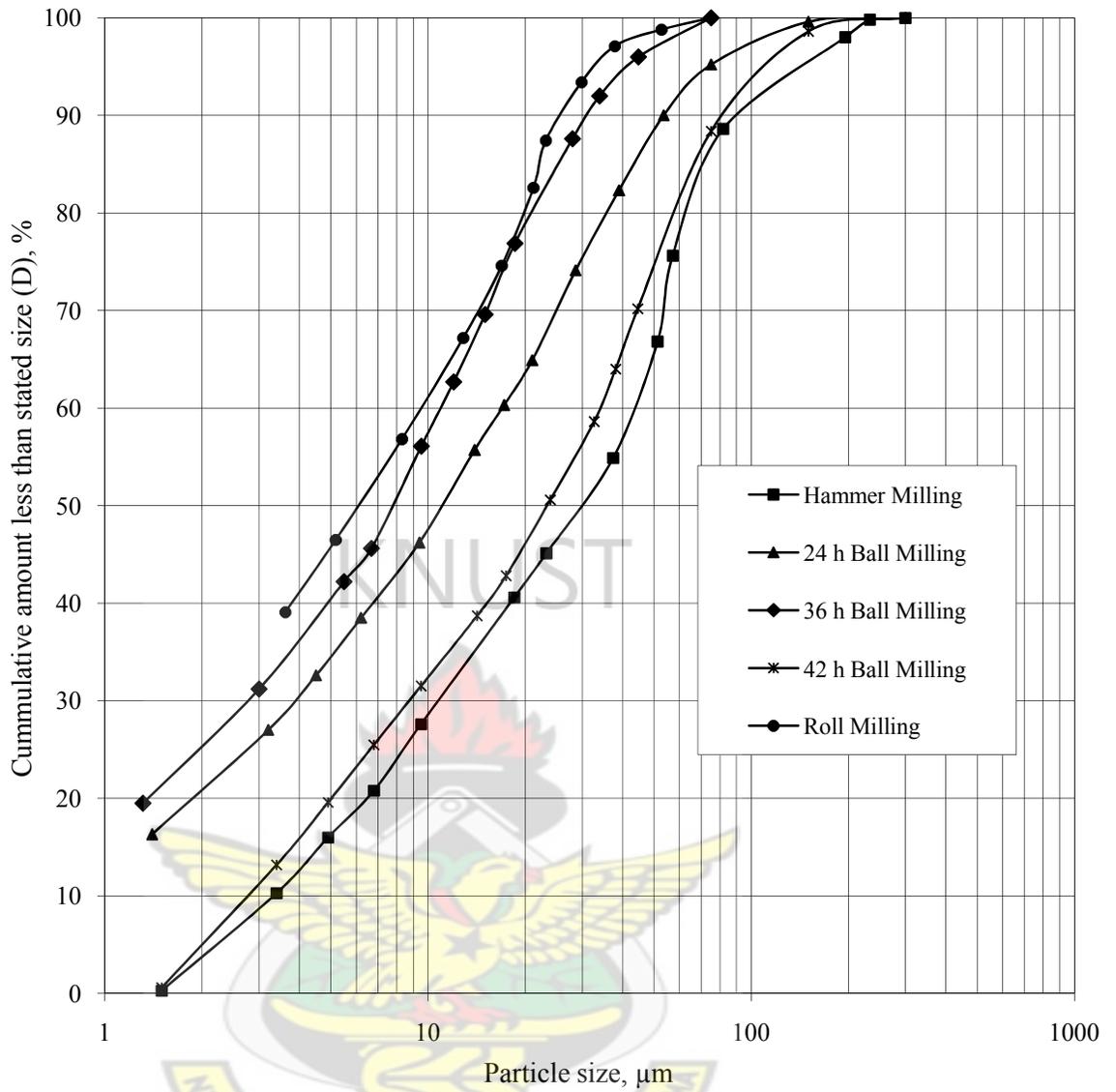


Figure 4-3: Particle size distribution curves for Mankranso pozzolana

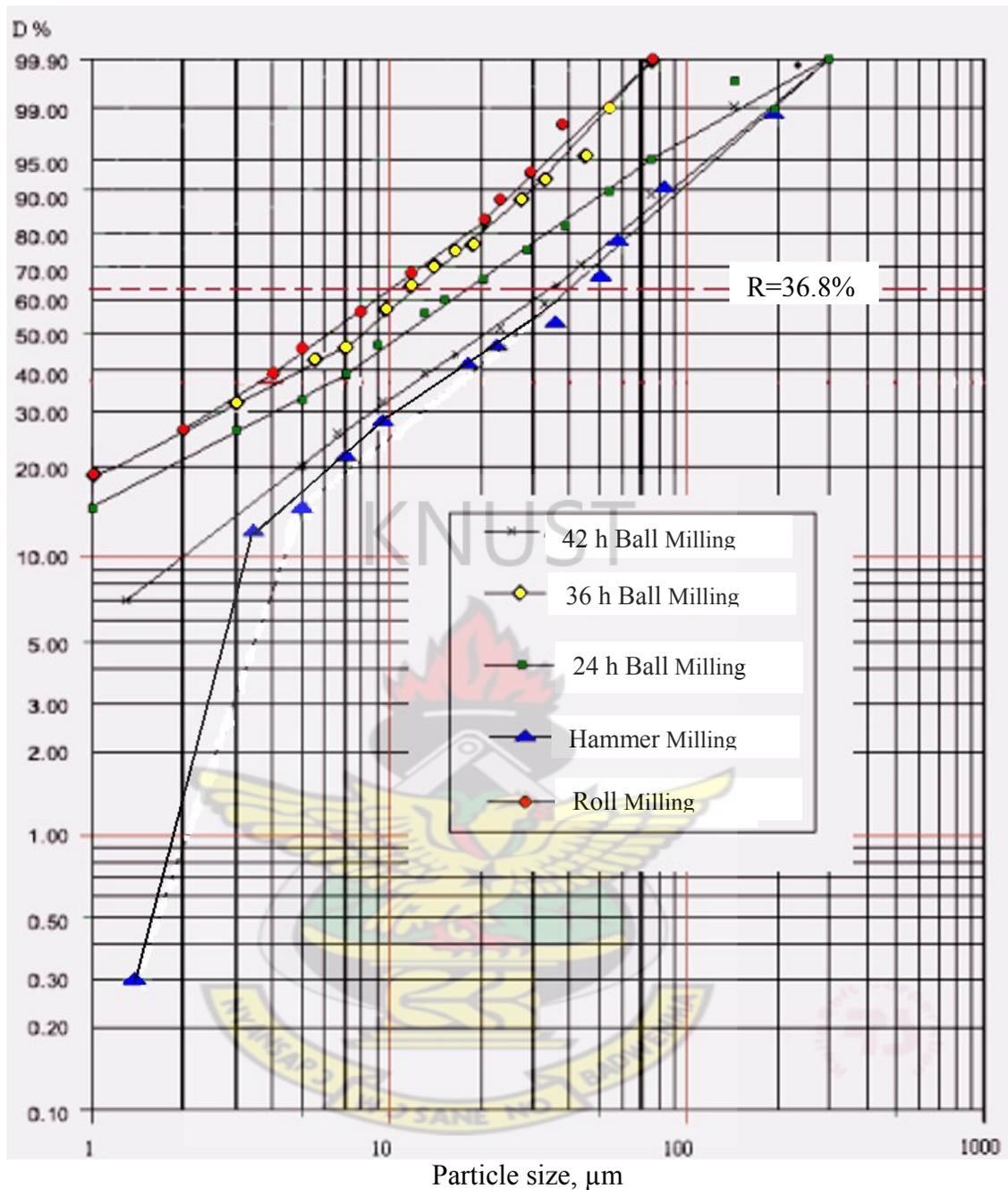


Figure 4-4: Rosin – Rammler distribution curves for Mankranso pozzolana

Mankranso pozzolana obtained a Blaine index of $1025 \text{ m}^2/\text{kg}$ after 36 h ball milling. Agglomeration in the ball mill reduced the specific surface area and increased the characteristic particle size from $12 \text{ }\mu\text{m}$ to $38 \text{ }\mu\text{m}$ after 42 h of ball milling.

Roll milling proved to be the most efficient, producing a characteristic particle size of 10 μm and a specific surface area determined by the Blaine method of 1123 m^2/kg . Hammer milling produced a specific surface area of only 349 m^2/kg as determined by the Blaine method.

Mankessim Pozzolana

For Mankessim pozzolana, the finest particle size for ball milling was obtained after 36 h milling, producing a Blaine index of 1128 m^2/kg and a characteristic particle size of 11 μm . Continued milling resulted in significant agglomeration, reducing the Blaine index to 696 m^2/kg and increasing the characteristic particle size to 38 μm .

Roll milling resulted in a much finer product with a Blaine index of 1311 m^2/kg and a characteristic particle size of 6 μm — the smallest of all the four pozzolana samples. Hammer milling produced a rather coarse pozzolana with a Blaine index of 96 m^2/kg and a characteristic particle size of 80 μm . This represented the coarsest sample produced of all the milling experiments.

Table 4-5: Particle size distribution, bulk density, specific surface area, uniformity coefficient and characteristic particle size for Mankessim Pozzolana

X, μm	Hammer milling, D, %	Roll milling, D, %	Ball milling, D, %		
			24 h	36 h	42 h
600	100	-	-	-	-
420	99.8	-	-	-	-
300	99.4	-	-	-	100
150	-	-	-	-	98.6
110	92.8	-	-	-	-
100	76.8	-	100	-	-
92.6	72.3	-	-	-	-
82.6	68.5	-	-	-	-
75	56.2	100	-	100	88.4
70.3	48.4	-	-	-	-
68.8	38.6	-	-	-	-
57.5	29.1	-	-	-	-
54.0	24.0	98.6	92.9	-	-
48.6	18.0	-	-	-	-
44.6	-	-	-	-	70.2
39.2	-	-	88.8	-	-
38.0	-	88.4	-	-	64.0
32.6	-	-	-	-	58.6
28.3	-	-	84.2	-	-
24.0	-	64.0	-	88.7	50.6
19.3	0.33	58.6	-	-	-
17.5	-	50.6	-	-	42.8
14.0	-	-	62.4	-	38.7
13.0	-	-	46.7	78.9	-
11.6	-	38.7	-	-	-
10.3	-	-	-	66.9	-
9.5	-	-	37.2	-	31.5
8.5	-	-	-	57.8	-
7.0	-	-	28.6	40.4	25.5
5.0	-	31.5	-	25.7	19.6
3.5	-	25.5	-	21.8	13.2
1.5	-	-	3.7	9.6	0.6
Bulk Density, kg/m^3	<i>1043</i>	<i>384</i>	<i>864</i>	<i>528</i>	<i>920</i>
Blaine Index, m^2/kg	<i>96</i>	<i>1311</i>	<i>798</i>	<i>1128</i>	<i>696</i>
Anselm's Fineness, m^2/kg	<i>59</i>	<i>1072</i>	<i>584</i>	<i>1062</i>	<i>374</i>
Characteristic particle size (\bar{x}), μm	<i>80</i>	<i>6</i>	<i>17</i>	<i>11</i>	<i>38</i>
Uniformity coefficient (n)	<i>3.1</i>	<i>1.3</i>	<i>1.5</i>	<i>1.2</i>	<i>1.0</i>

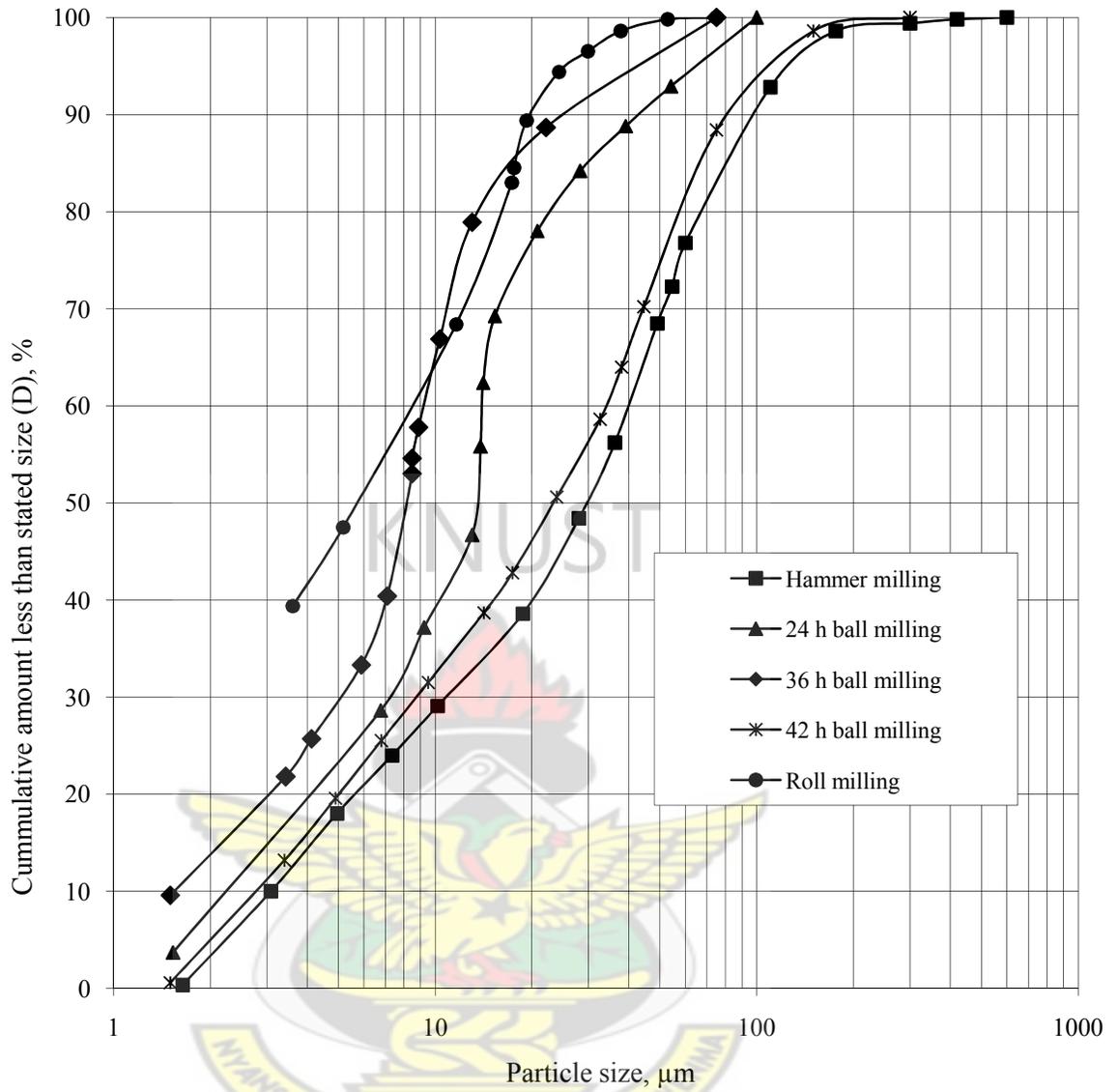


Figure 4-5: Particle size distribution curves for Mankessim pozzolana

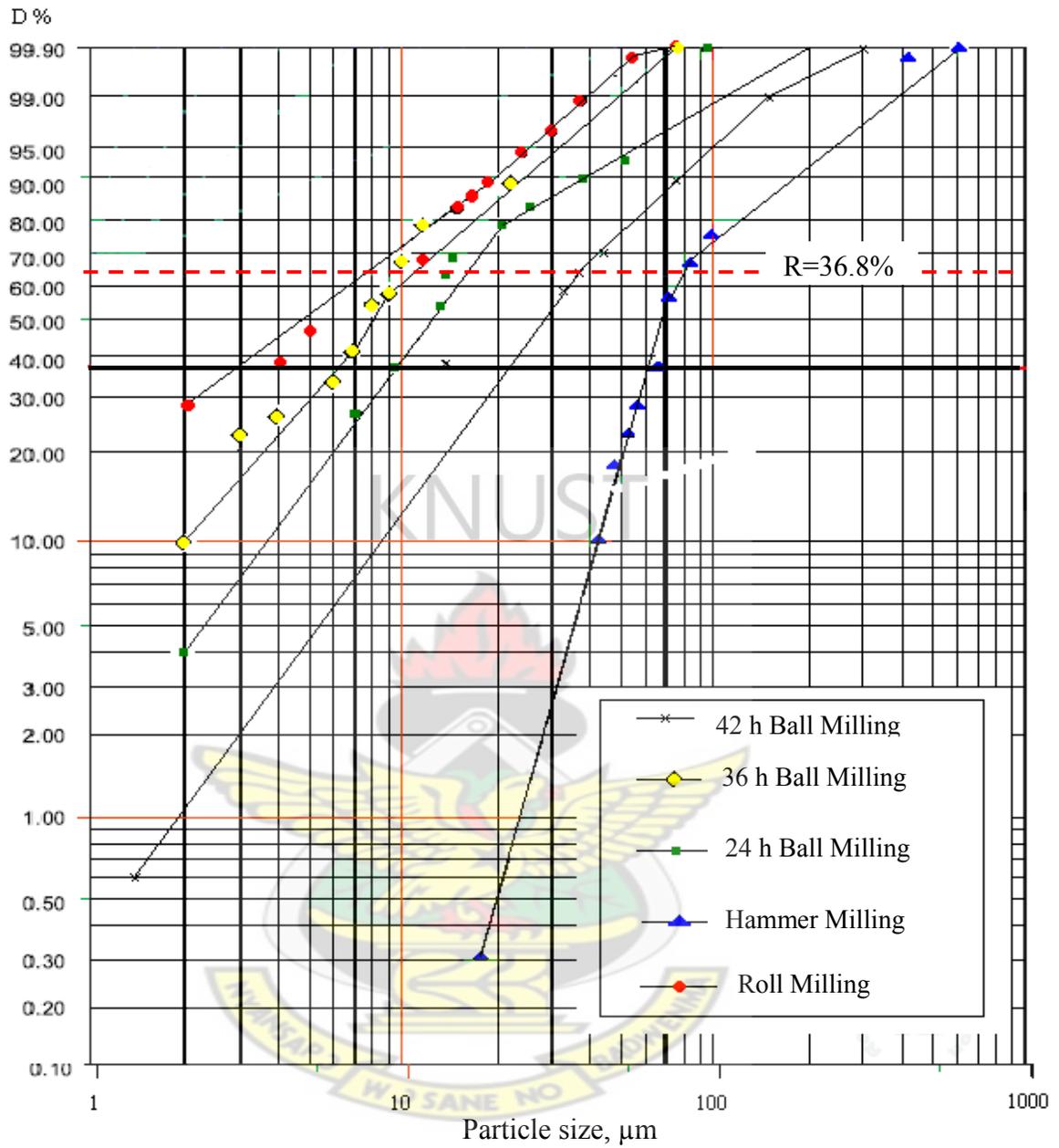


Figure 4-6: Rosin-Rammler distribution curves for Mankessim pozzolana

Tanoso pozzolana

Table 4-6: Particle size distribution, bulk density, specific surface area, uniformity coefficient and characteristic particle size for Tanoso pozzolana

X, μm	Hammer milling, D,%	Roll milling, D,%	Ball milling, D, %		
			24 h	36 h	42 h
600	100	-	-	-	-
420	99.8	-	-	-	-
300	98.2	-	-	-	100
150	91.0	-	-	-	98.6
110	-	-	100	-	-
75.0	80.2	100	-	100	88.4
55.4	-	-	91.5	-	-
52.8	-	98.8	-	98.8	-
50.5	67.9	-	-	-	-
44.6	-	-	-	-	70.2
40.6	-	-	84.0	-	-
38.0	-	-	-	-	64.0
37.8	-	97.1	-	97.1	-
33.1	54.8	-	-	-	-
32.6	-	-	-	-	58.6
30.0	-	93.4	74.8	93.4	-
25.9	-	-	-	-	-
24.0	45.9	-	69.7	-	50.6
23.1	-	87.4	-	87.4	-
21.2	-	82.6	-	82.6	-
18.9	-	-	59.5	-	-
17.5	39.1	-	-	-	42.8
16.9	-	74.6	-	74.6	-
14.2	-	-	-	-	38.7
13.0	33.3	67.2	51.0	67.2	-
9.5	27.9	-	44.7	-	31.5
8.3	-	56.8	-	56.8	-
7.0	21.8	-	38.6	-	25.5
5.0	16.2	46.5	32.8	46.5	19.6
3.5	1.0	39.1	26.2	39.1	13.2
1.5	0.33	-	12.9	-	0.6
Bulk Density, kg/m³	852	337	836	560	843
Blaine Index, m²/kg	382	1354	889	985	701
Anselm's Fineness, m²/kg	330	1124	666	740	364
Characteristic particle size (\bar{x}), μm	42	9	20	12	35
Uniformity coefficient (n)	0.9	1.3	1.0	1.5	1.0

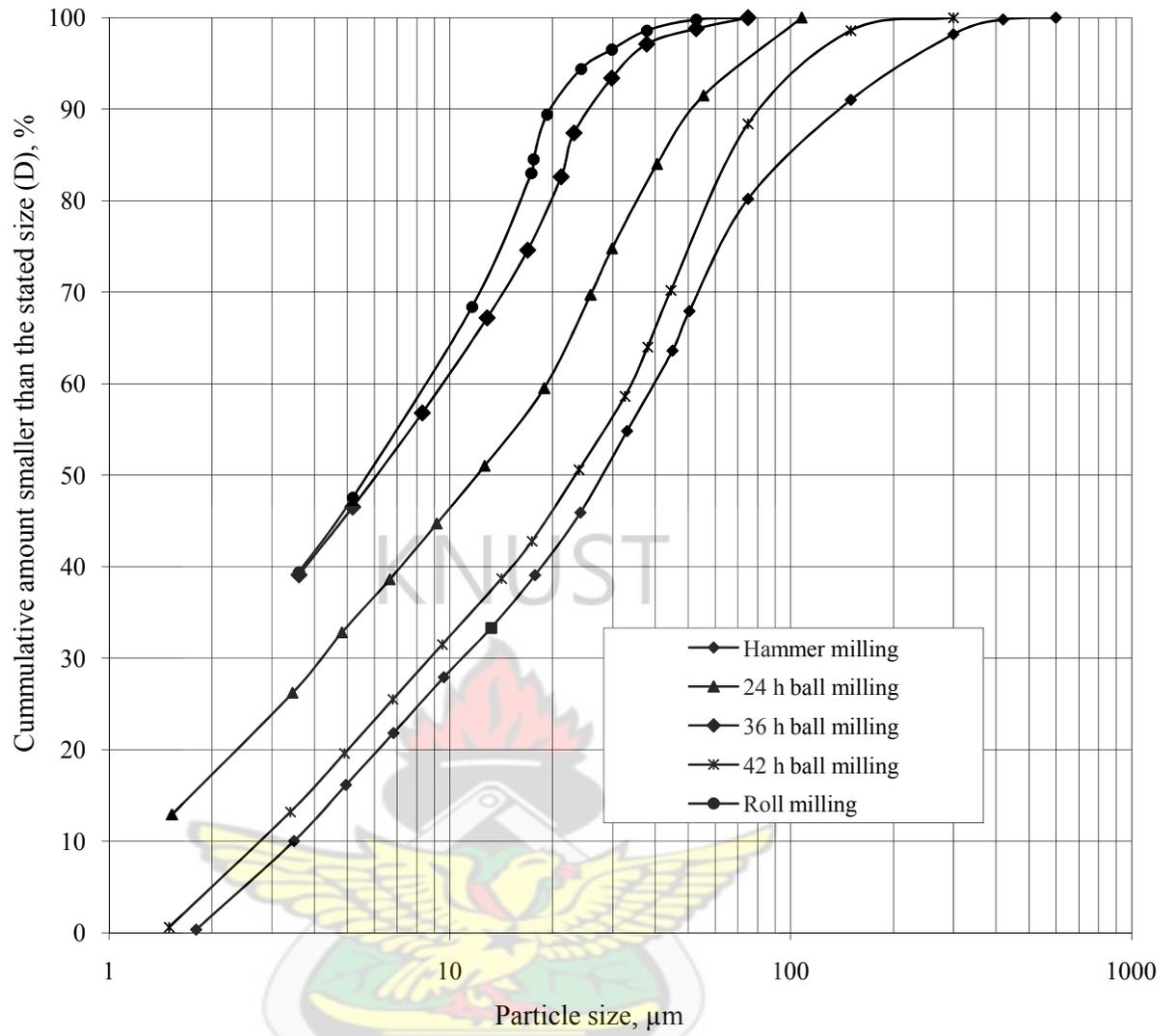


Figure 4-7: Particle size distribution curves for Tanoso pozzolana

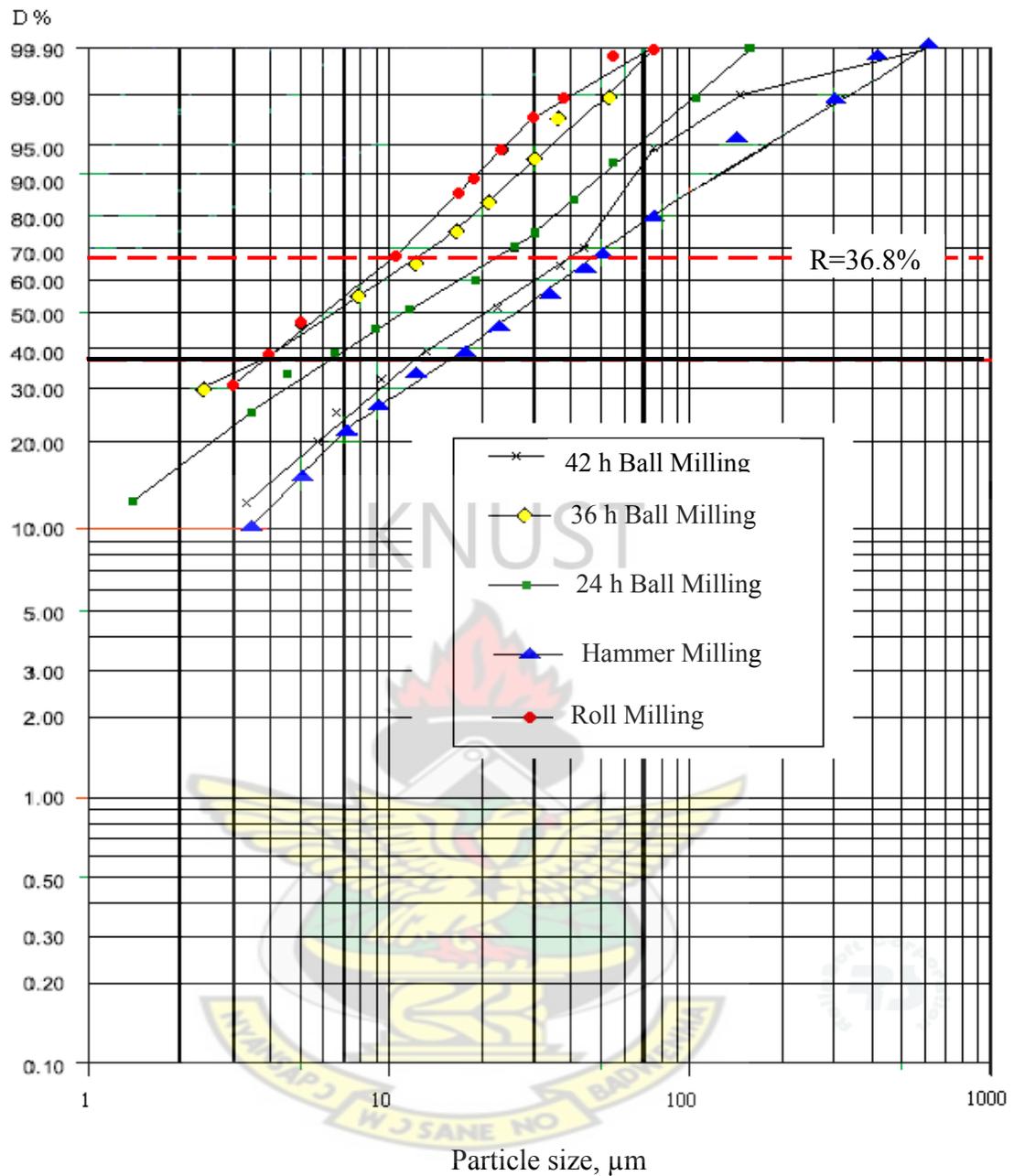


Figure 4-8: Rosin – Rammler distribution curves for Tanoso pozzolana

Tanoso pozzolana, like all the others, started agglomeration in the ball mill after 36 h grinding. The finest particle size was achieved in the ball mill for 36 h milling, which resulted in a specific surface area of 985 m²/kg, the lowest of all the four pozzolana samples for the same grinding time.

Roll milling, however, produced a comparable fineness to the other pozzolanas with a Blaine index of 1354 m²/kg and a characteristic particle size of 9 µm. Hammer milling was much less efficient as was the case for all the pozzolana samples.

4.3 Compressive Strength

The results for the compressive strength determinations are given in Tables 4-8 – 4-11. The data are also presented graphically in Figures 4-9 – 4-40. The minimum standard requirements of compressive strength for cement mortar cubes by both the European Union and the American Society for Testing and Materials standards are given in Table 4-7.

Table 4-7: Standard requirements for compressive strength of cements

Standard	Curing period, days		
	2	7	28
	Compressive strength, MPa		
EN 197-1	10.0	16.0	32.5
ASTM C 595	-	-	24.1

4.3.1 Mfensi pozzolana

Table 4-8 gives the compressive strengths of ordinary Portland cement (OPC) and activated pozzolana cement mortar cubes containing Mfensi pozzolana at 2, 7, 28 and 90 days curing periods for 30, 35, 40 and 50% replacement of OPC respectively. The data are also presented graphically in Figures 4-9 – 4-12.

Table 4-8: Compressive strengths of Mfensi pozzolana cement mortar cubes

Milling type and period	Curing period, days			
	2	7	28	90
Compressive strength, MPa				
30% cement replacement				
Hammer milling	14.7	18.2	24.5	29.4
24 h Ball milling	16.6	30.7	35.4	39.6
36 h Ball milling	17.0	32.3	39.9	43.4
Roll milling	18.0	32.8	40.8	45.4
35% cement replacement				
Hammer milling	12.7	17.4	21.6	28.8
24 h Ball milling	14.3	27.4	29.2	38.7
36 h Ball milling	14.9	30.1	33.3	42.6
Roll milling	16.3	30.9	35.6	43.3
40% cement replacement				
Hammer milling	11.0	15.9	21.0	27.9
24 h Ball milling	12.2	20.0	27.3	36.4
36 h Ball milling	13.2	20.4	30.5	40.5
Roll milling	14.6	22.4	32.1	41.3
50% cement replacement				
Hammer milling	8.0	14.9	20.6	27.1
24 h Ball milling	9.9	18.3	23.9	36.5
36 h Ball milling	10.3	19.3	24.4	39.0
Roll milling	11.2	18.8	25.4	40.1
0% cement replacement (OPC)				
	19.0	36.3	43.9	47.1

Generally, compressive strengths increased as curing age increased. Strength losses were more pronounced for samples containing 40-50% activated pozzolana for ages 2-28 days. However, these samples improved in strength rapidly as curing continued to 90 days.

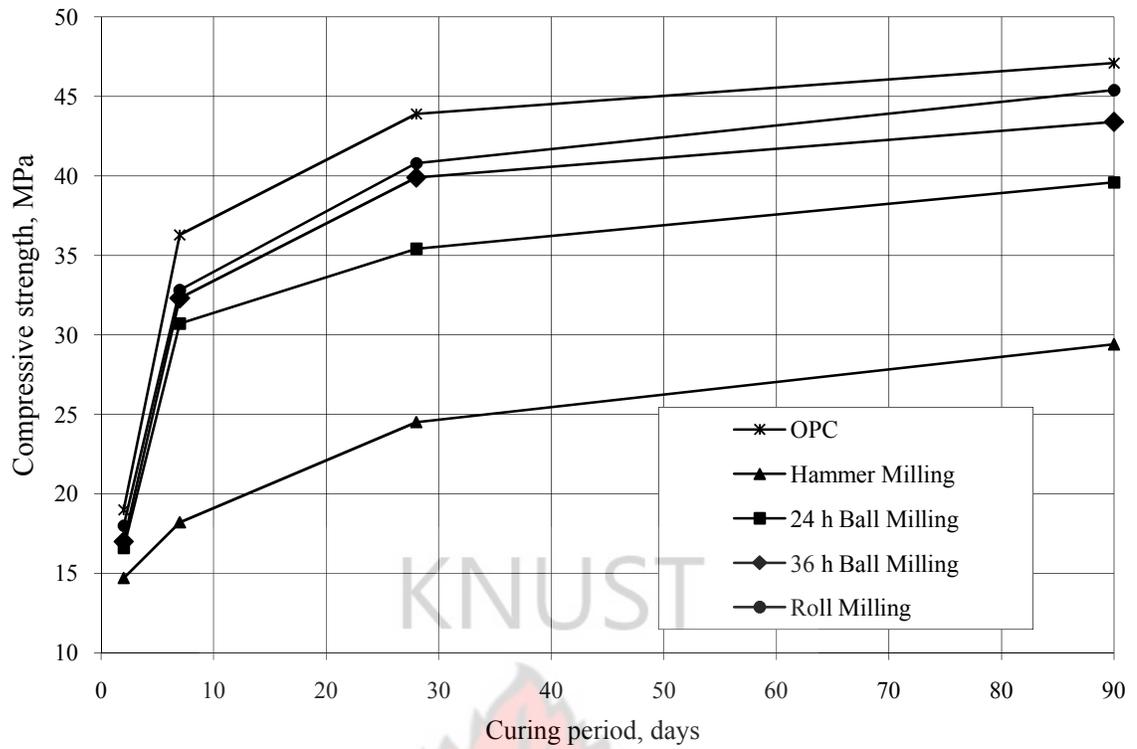


Figure 4-9: Compressive strength development of 30% Mfensi pozzolana cement mortar cubes

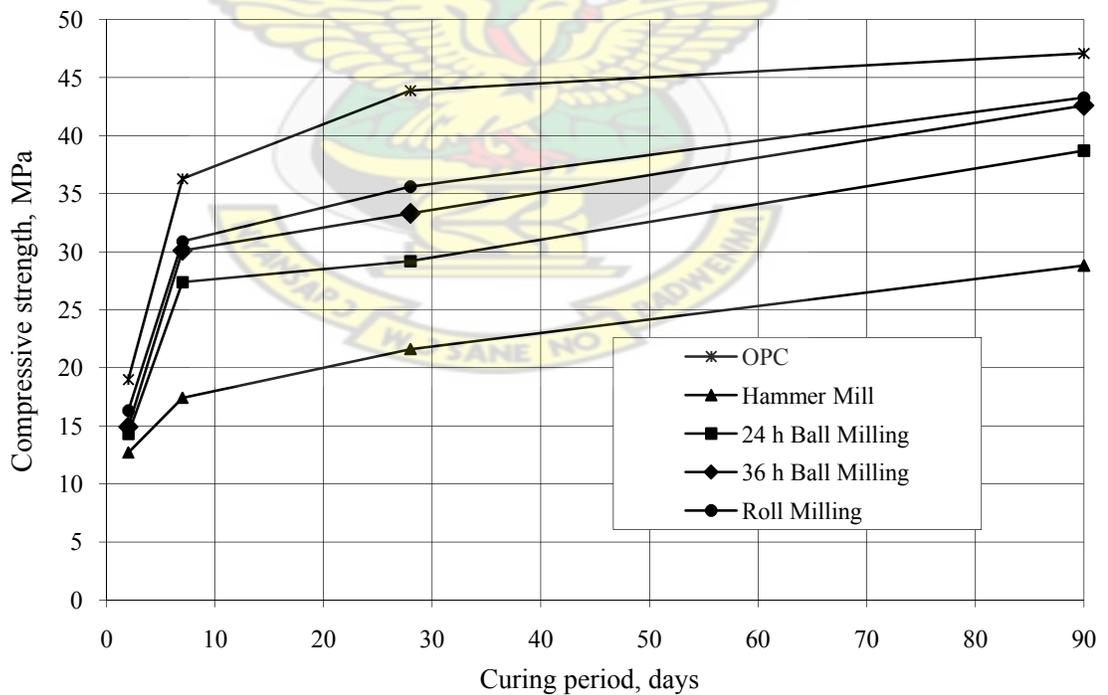


Figure 4-10: Compressive strength development of 35% Mfensi pozzolana cement mortar cubes

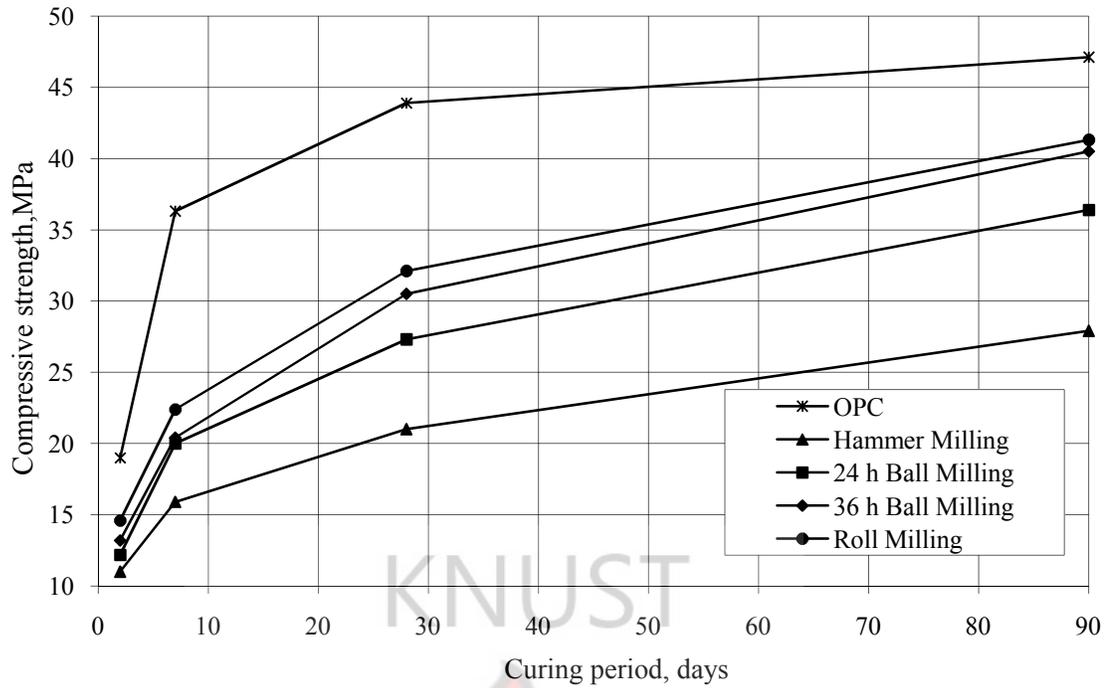


Figure 4-11: Compressive strength development of 40% Mfensi pozzolana cement mortar cubes

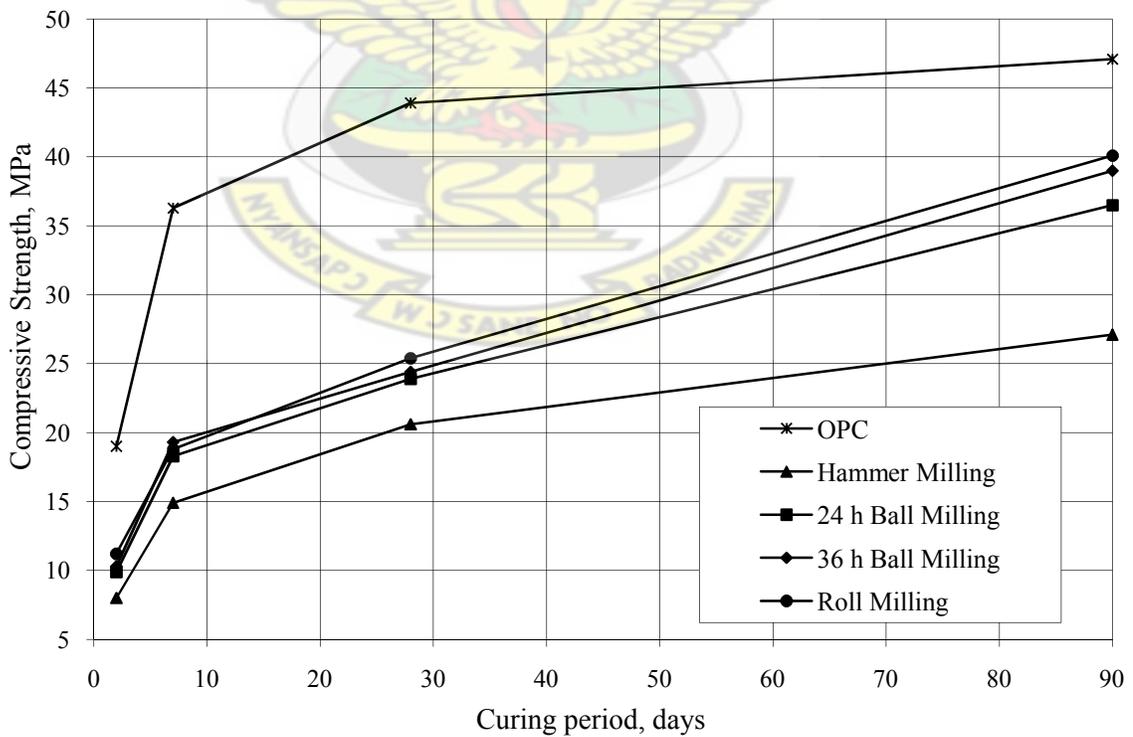


Figure 4-12: Compressive strength development of 50% Mfensi pozzolana cement mortar cubes

Generally, compressive strengths increased with curing period, as was expected. Although at all replacement ratios the pozzolana mortar cubes were weaker than OPC mortar cubes, their compressive strengths increased more rapidly after 28 days curing, than that of OPC. Milling of the Mfensi pozzolana substantially increased the compressive strength at all replacement ratios. The finer the pozzolana particles were, the higher the compressive strengths became. Thus the highest compressive strength values were obtained for the roll milled pozzolanas, followed by the 36 h ball milled pozzolana. The lowest compressive strengths were obtained using hammer milling.

Comparing compressive strengths at 28 days, with hammer milling as a basis, roll milling resulted in 65-66% strength increase at 30 and 35% cement replacement, 52% increase at 40% cement replacement and 23% increase at 50% cement replacement. The 90-day compressive strengths were generally 45-55% higher for both 36 h ball milling and roll milling above that of hammer milling.

Early age strength

The early age strengths are shown in Figures 4-13 and 4-14. As expected, the compressive strengths decreased as the pozzolana content increased in the pozzolana cement mortars. However, except for hammer milling, all the Mfensi pozzolana samples satisfied the early strength requirement of the European standard EN 197-1 for 2 and 7 days curing periods of 10.0 and 16.0 MPa up to 50% cement replacement.

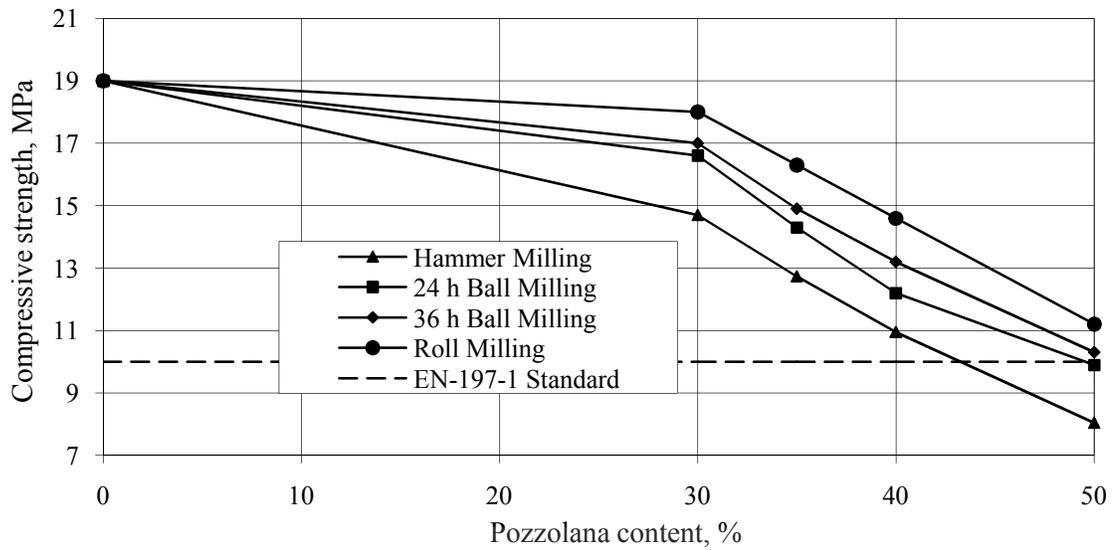


Figure 4-13: 2-day compressive strength of Mfensi pozzolana

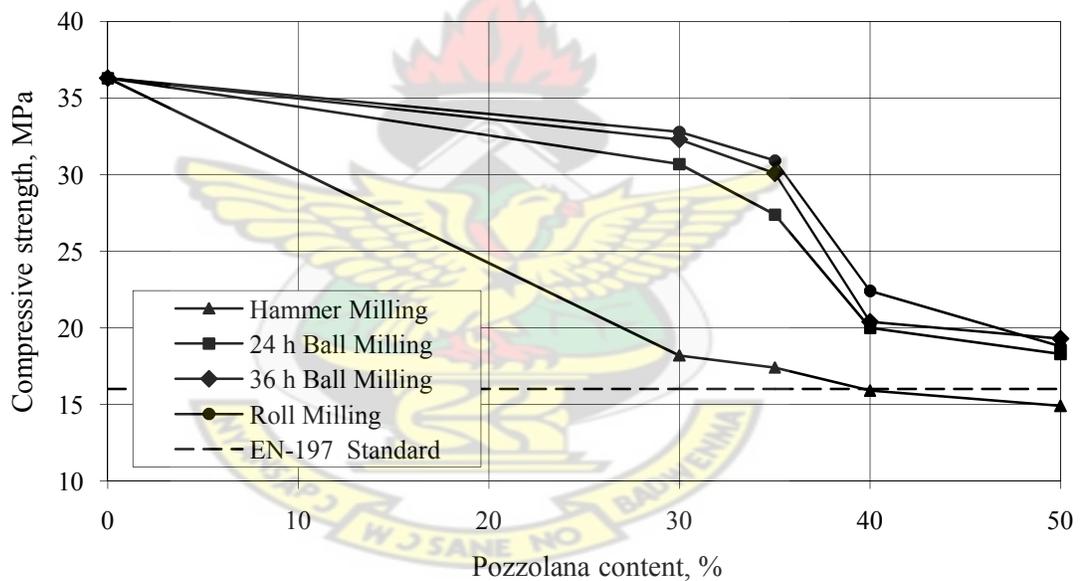


Figure 4-14: 7-day compressive strength of Mfensi pozzolana

Ultimate strength

The ultimate strength of the activated Mfensi pozzolana cement mortar cubes were lower than that of ordinary Portland cement mortar cubes at all cement replacement levels. However, except for the hammer milled pozzolana, they satisfied the ASTM standard

requirement of minimum 24.1 MPa at 28 days up to 50% cement replacement. The European standard is higher—32.5 MPa, but even that was satisfied up to 35% cement replacement by the roll milled and 36 h ball milled pozzolanas.

The standards do not specify compressive strength limits for curing periods longer than 28 days. The 90-days compressive strengths were therefore compared to the existing 28-day requirements. Figure 4-16 clearly shows that the activated pozzolana cement mortar cubes had significantly gained strengths after 28 days and except for hammer milling, all the pozzolana samples satisfied even the higher EN 197-1 requirement up to 50% cement replacement. Furthermore, the difference between the compressive strength of the ordinary Portland cement mortar cubes and that of the pozzolana cement mortar cubes reduced significantly as curing was continued beyond 28 days. The best activated pozzolana obtained by roll milling was 42% weaker after 28 days and only 15% weaker after 90 days than OPC even at 50% cement replacement. At 30% cement replacement, the difference was not significant, being only 4% weaker after 90 days curing.

The hammer mill product, being the worst in compressive strength of all the Mfensi pozzolana samples, could only satisfy the ASTM C 595 requirement for 28 days strength up to 30% cement replacement but not the European standard requirement. It gained strength by the 90 days curing, but it still did not satisfy European standard; however, the ASTM C 595 standard was satisfied up to 50% cement replacement.

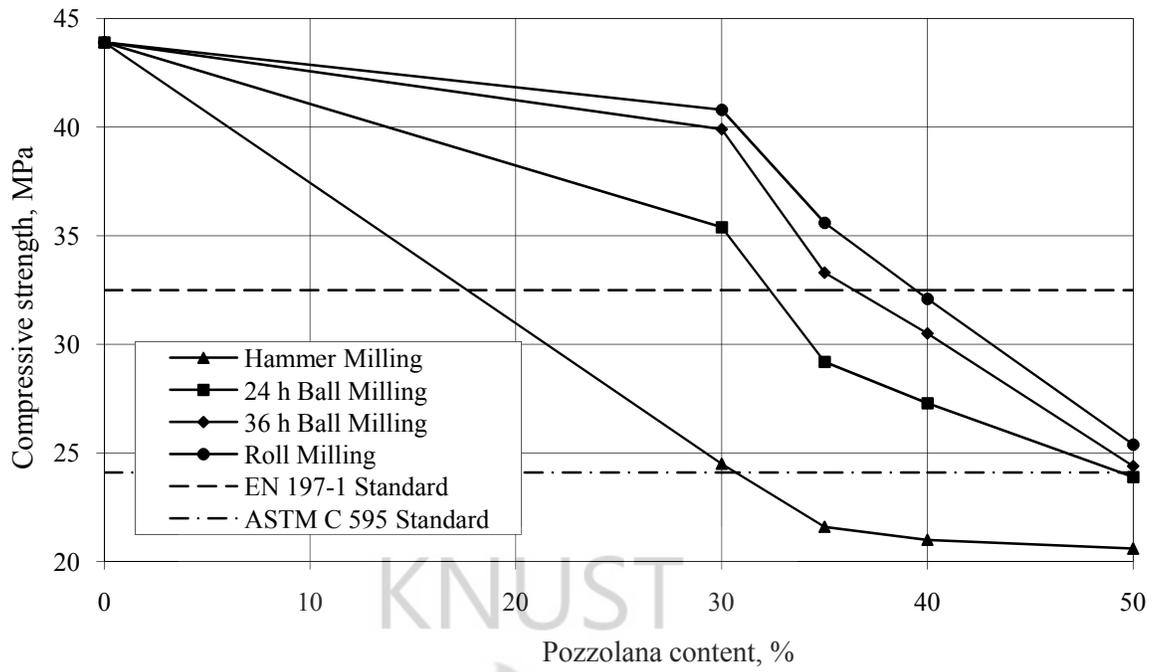


Figure 4-15: 28-day compressive strength of Mfensi pozzolana

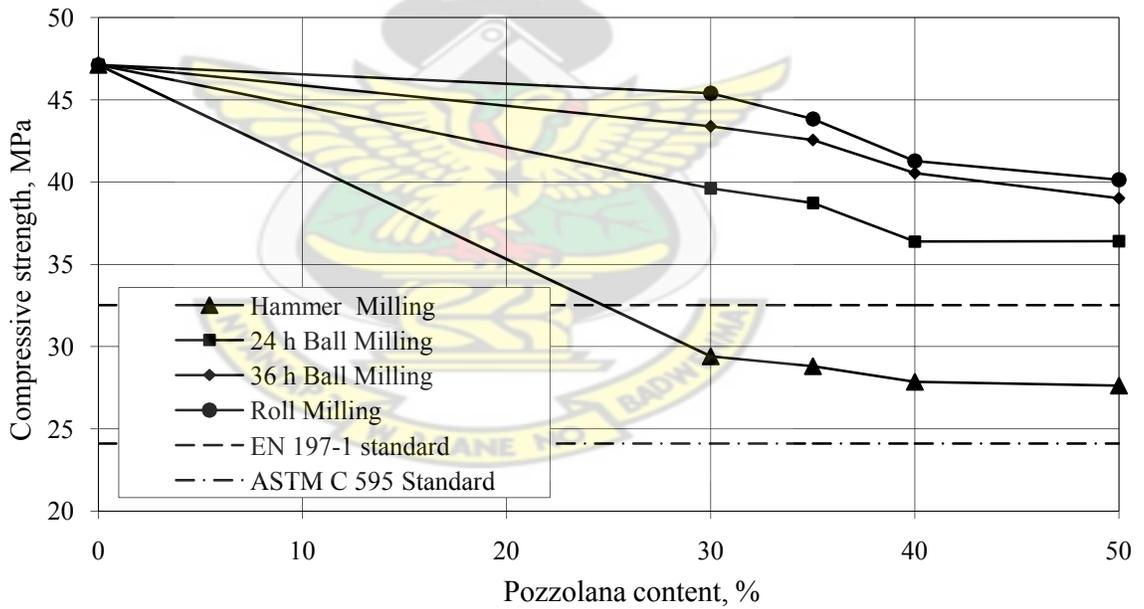


Figure 4-16: 90-day compressive strength of Mfensi pozzolana

4.3.2 Mankranso pozzolana

Table 4-9: Compressive strengths of Mankranso pozzolana cement mortar cubes

Milling type and period	Curing period, days			
	2	7	28	90
	Compressive strength, MPa			
30% cement replacement				
Hammer milling	10.3	22.0	27.5	32.3
24 h Ball milling	13.8	26.9	33.6	40.3
36 h Ball milling	14.8	27.5	33.8	44.6
Roll milling	16.2	28.6	35.8	46.3
35% cement replacement				
Hammer milling	7.2	18.0	26.5	30.6
24 h Ball milling	12.2	23.5	28.6	38.6
36 h Ball milling	12.8	26.4	32.6	42.5
Roll milling	13.6	26.8	34.1	42.4
40% cement replacement				
Hammer milling	6.9	16.5	19.9	28.6
24 h Ball milling	10.6	21.2	26.8	36.5
36 h Ball milling	11.0	21.9	27.7	41.8
Roll milling	11.8	23.4	29.6	40.6
50% cement replacement				
Hammer milling	6.0	14.5	18.1	26.7
24 h Ball milling	8.8	18.8	24.7	34.6
36 h Ball milling	9.9	18.9	25.2	40.3
Roll milling	10.2	19.6	24.8	40.0
0% cement replacement(OPC)				
	19.0	36.3	43.9	47.1

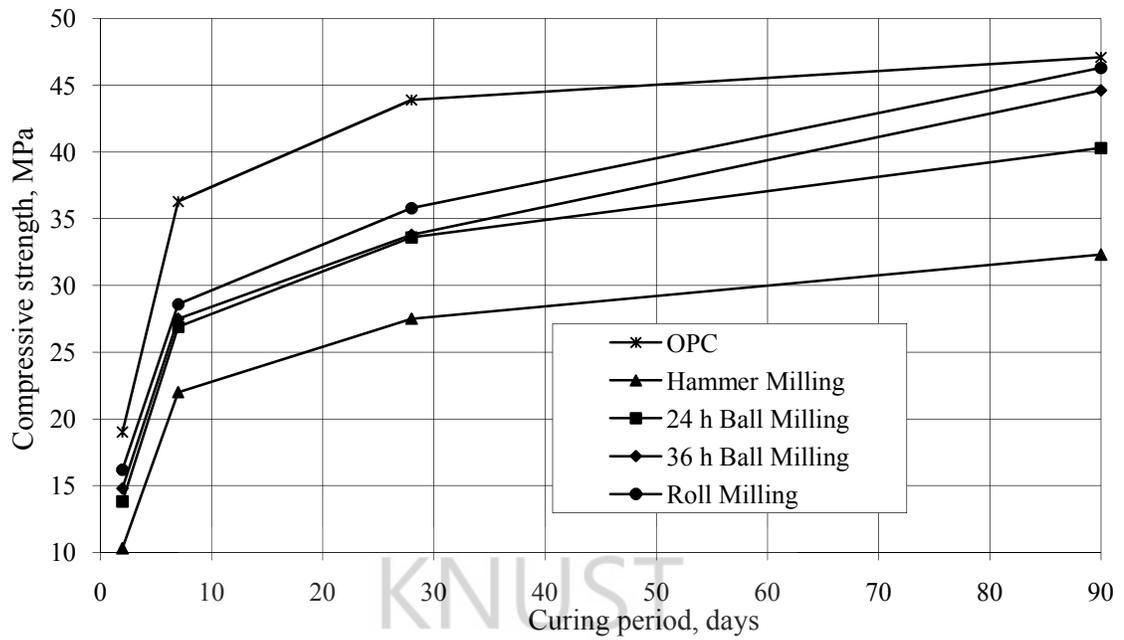


Figure 4-17: Compressive strength development of 30% Mankranso pozzolana cement mortar cubes

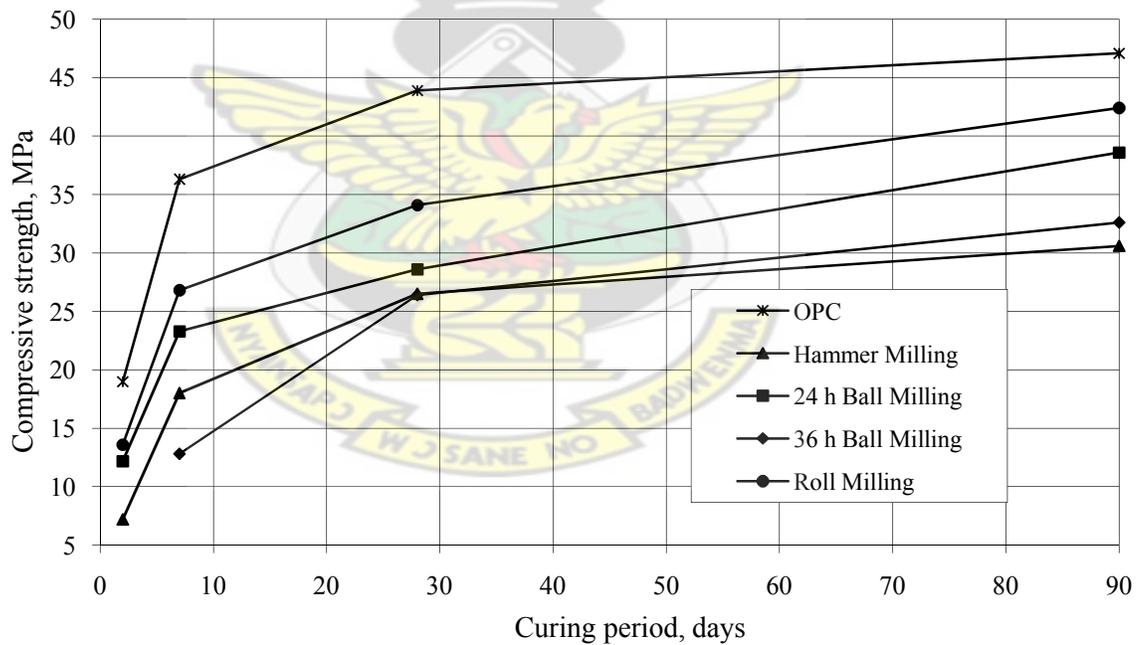


Figure 4-18: Compressive strength development of 35% Mankranso pozzolana cement mortar cubes

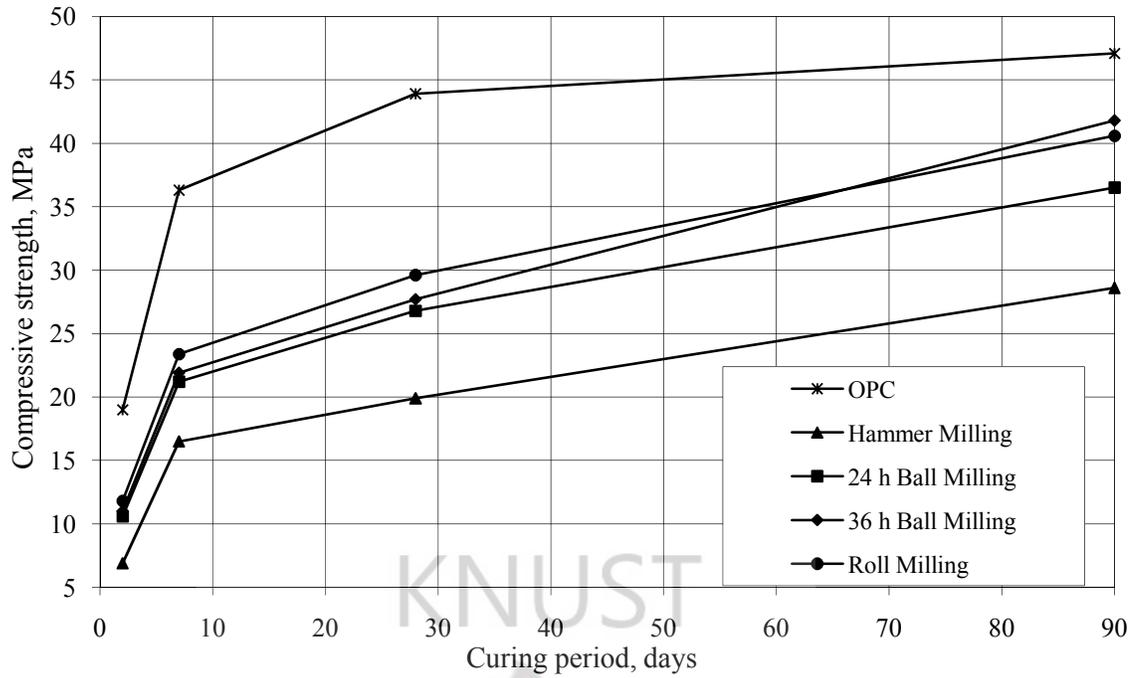


Figure 4-19: Compressive strength development of 40% Mankranso pozzolana cement mortar cubes

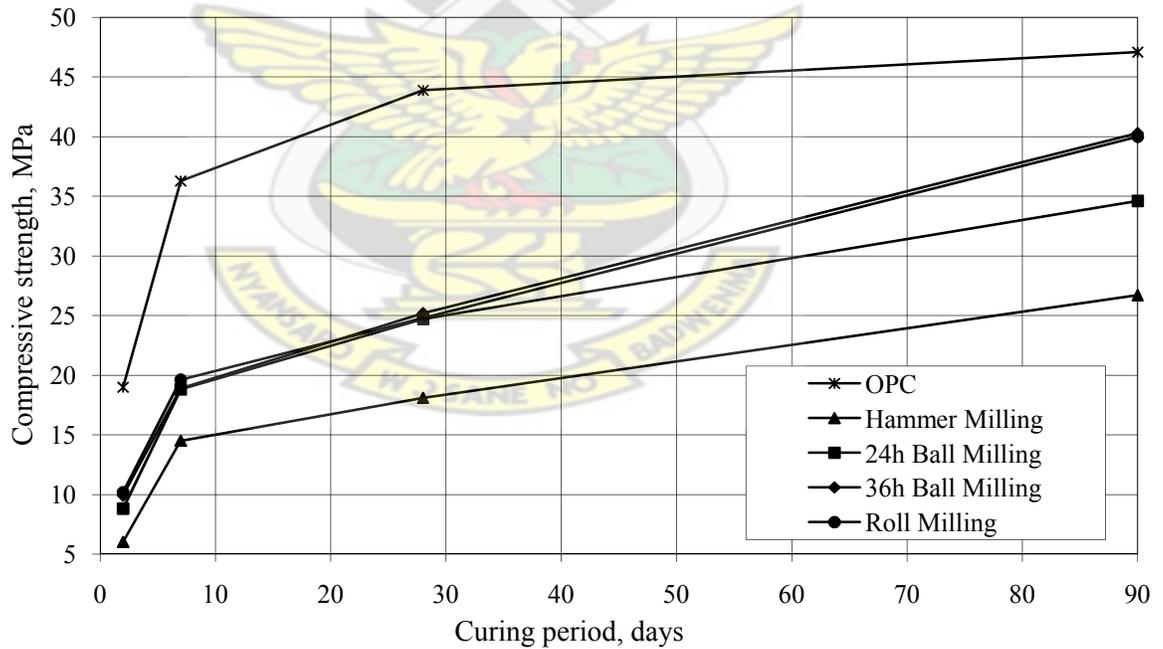


Figure 4-20: Compressive strength development of 50% Mankranso pozzolana cement mortar cubes

Generally, the compressive strength of Mankranso pozzolana cement mortar cubes increased faster 28 days curing than that of OPC at all cement replacement levels, even

though the pozzolana cubes were weaker. Milling the pozzolanas increased the compressive strength at all replacement. The highest values were again obtained for the roll milled pozzolana, followed closely by the 36 h ball milled pozzolana. The least strengths were obtained with hammer milling.

Using hammer milling as a basis, at 28 days roll milling resulted in 23-29% strength increase at 30 and 35% cement replacement, 49% increase at 40% cement replacement and 37% increase at 50% cement replacement. The 90-day compressive strengths were 40-50% higher for roll milling than for hammer milling. Figures 4-17 – 4-20 also show, that the compressive strength development of the pozzolana cement mortar cubes is faster than OPC, since the curves are steeper for all the samples than for OPC.

Early age strength

The early strengths of Mankranso pozzolana cement are shown in Figures 4-21 and 4-22.

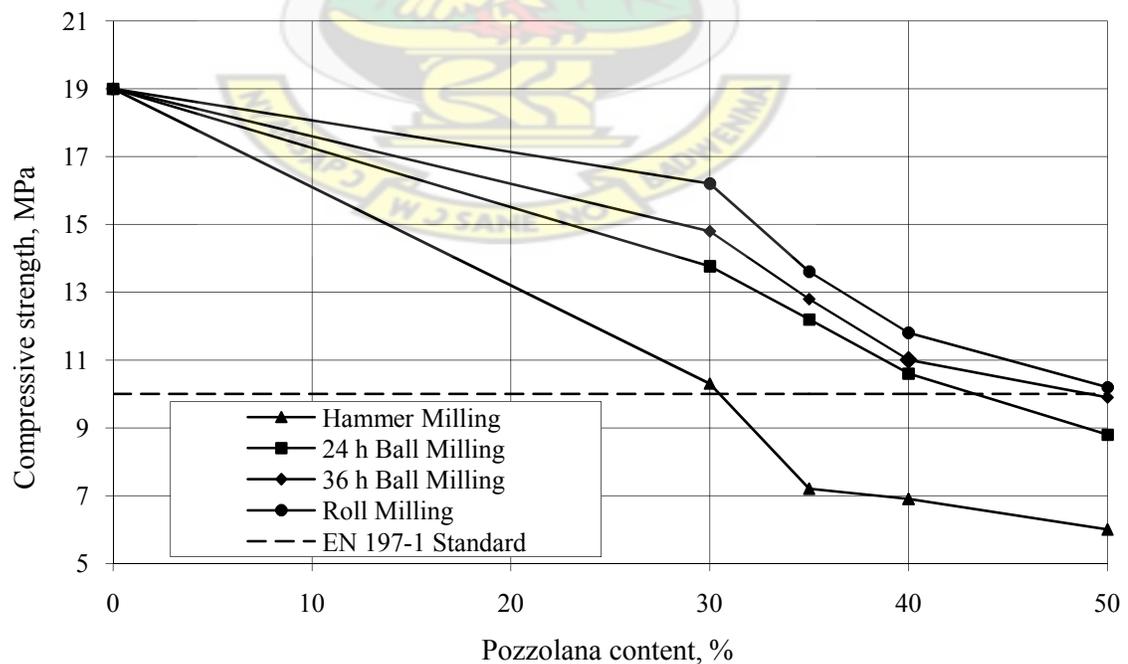


Figure 4-21: 2-day compressive strength of Mankranso pozzolana

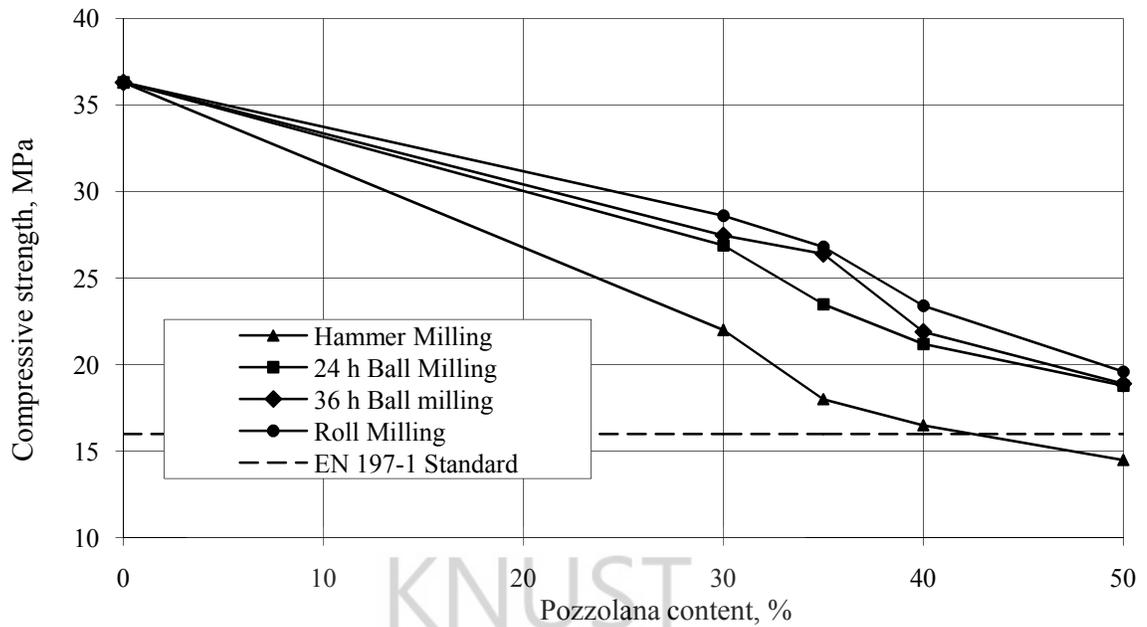


Figure 4-22: 7-day compressive strength of Mankranso pozzolana

As the pozzolana content increased, the compressive strength of the mortar cubes decreased. Except for hammer milling, all the samples satisfied the EN 197-1 2-day compressive strength requirement of minimum 10 MPa up to 40% cement replacement and the 7-day compressive strength requirement of 16 MPa up to 50% cement replacement. The hammer mill product was sufficiently strong at 2 days only up to 30% cement replacement and at 7 days up to 40% cement replacement.

Ultimate strength

Figures 4-23 and 4-24 show the compressive strengths of the pozzolana cement mortar cubes after 28 and 90 days curing respectively. The 28-day compressive strength of all the Mankranso pozzolana samples satisfied the European standard EN 197-1 up to 30% cement replacement, except for the hammer milled pozzolana.

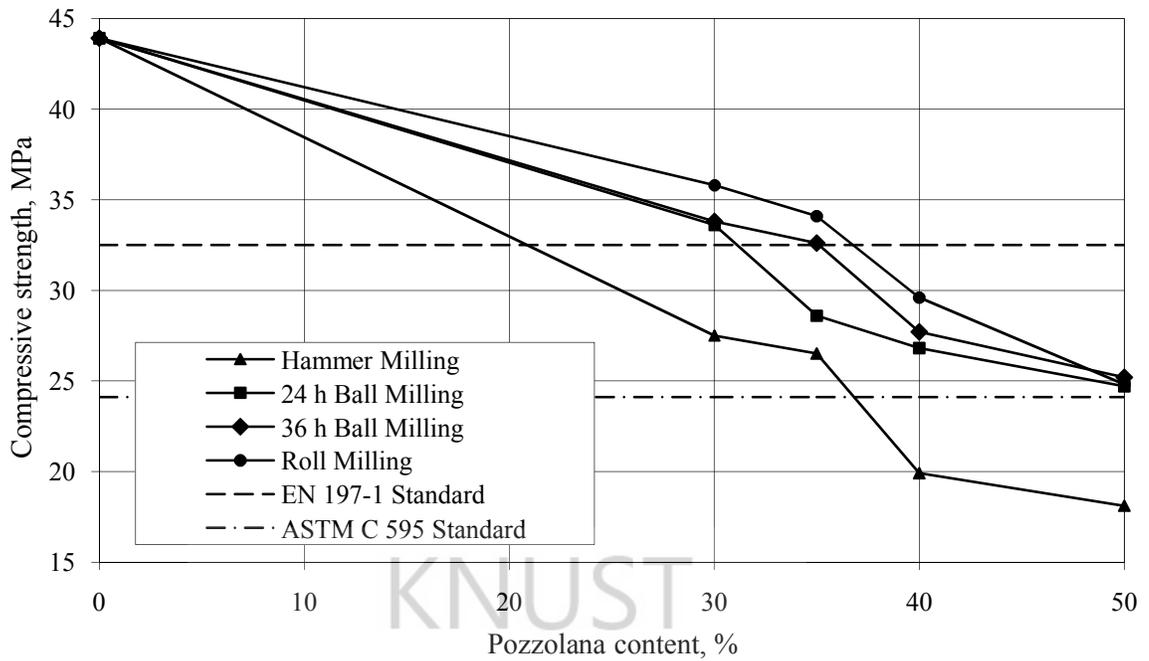


Figure 4-23: 28-day compressive of Mankranso pozzolana

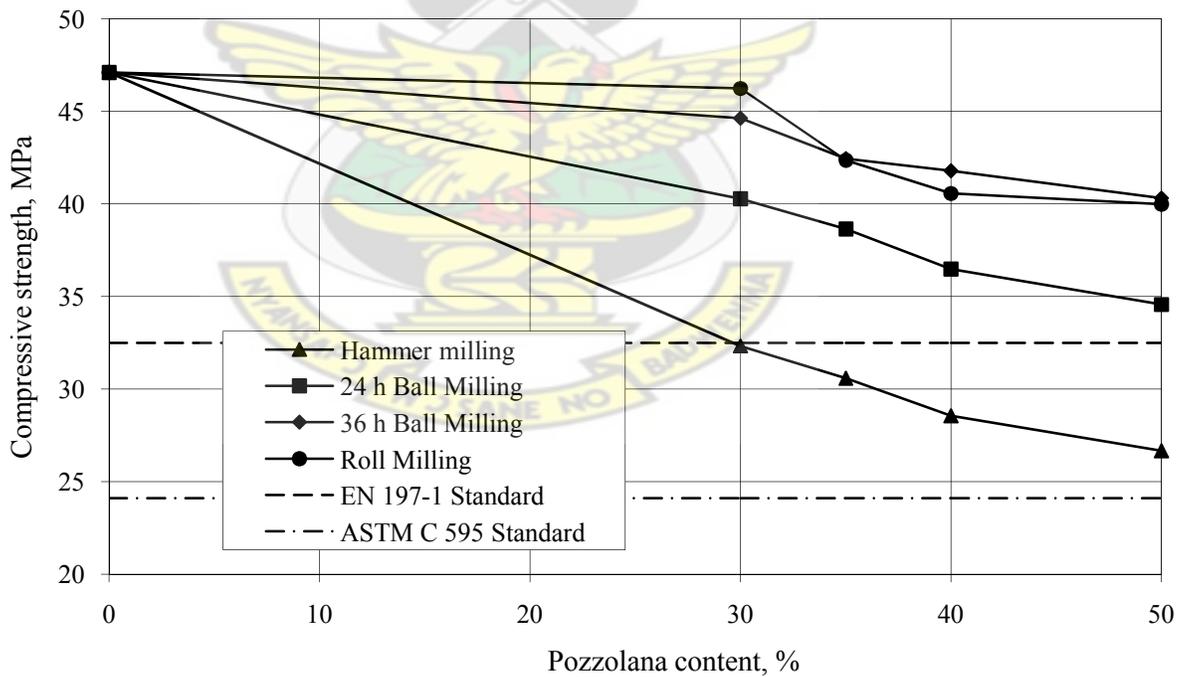


Figure 4-24: 90-day compressive strength of Mankranso pozzolana

The roll milled pozzolana satisfied the EN 197-1 standard up to 35% cement replacement.

The ASTM C 595 standard requirement was met at all cement replacement levels except

for the hammer milled pozzolana. Similarly, the 90-day compressive strengths attained satisfied the 28-day requirement of EN 197-1 up to 35% for the roll milled and 36 h ball milled samples. The ASTM C 595 standard was however satisfied at all cement replacement levels except for the hammer milled pozzolana.

4.3.3 Mankessim pozzolana

Data for compressive strength results are presented in Table 4-10 and illustrated on Figures 4-25 – 4-32.

Table 4-10: Compressive strengths of Mankessim pozzolana cement mortar cubes

Milling type and period	Curing period, days			
	2	7	28	90
Compressive strength, MPa				
30% cement replacement				
Hammer milling	12.6	16.6	21.8	32.6
24 h Ball milling	13.2	17.0	23.2	35.4
36 h Ball milling	16.1	31.3	35.2	42.9
Roll milling	17.6	32.6	40.1	43.2
35% cement replacement				
Hammer milling	10.8	15.4	20.4	31.8
24 h Ball milling	12.4	16.7	21.8	33.9
36 h Ball milling	15.5	29.4	28.6	36.2
Roll milling	16.0	30.3	33.2	38.5
40% cement replacement				
Hammer milling	8.6	14.8	19.3	28.4
24 h Ball milling	10.9	15.5	21.6	30.1
36 h Ball milling	12.4	18.0	25.6	33.8
Roll milling	13.6	19.8	30.3	35.2
50% cement replacement				
Hammer milling	7.4	12.6	18.6	28.6
24 h Ball milling	8.9	14.3	20.2	28.6
36 h Ball milling	9.1	16.0	23.1	32.7
Roll milling	10.4	19.1	23.9	33.4
0% cement replacement (OPC)				
	19.0	36.3	43.9	47.1

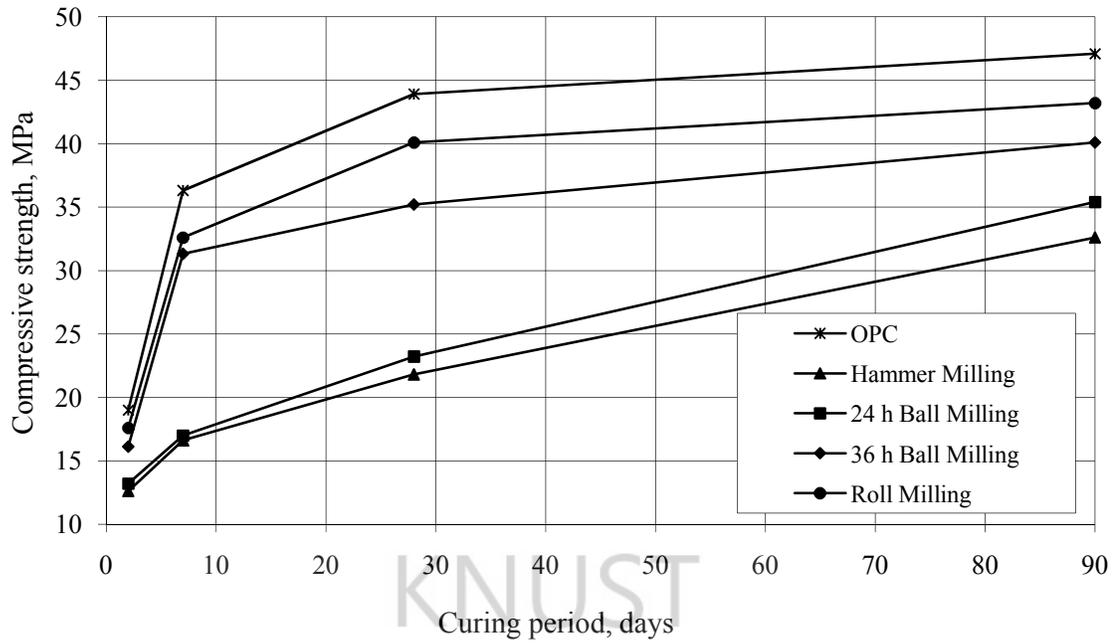


Figure 4-25: Compressive strength development of 30% Mankessim pozzolana cement mortar cubes

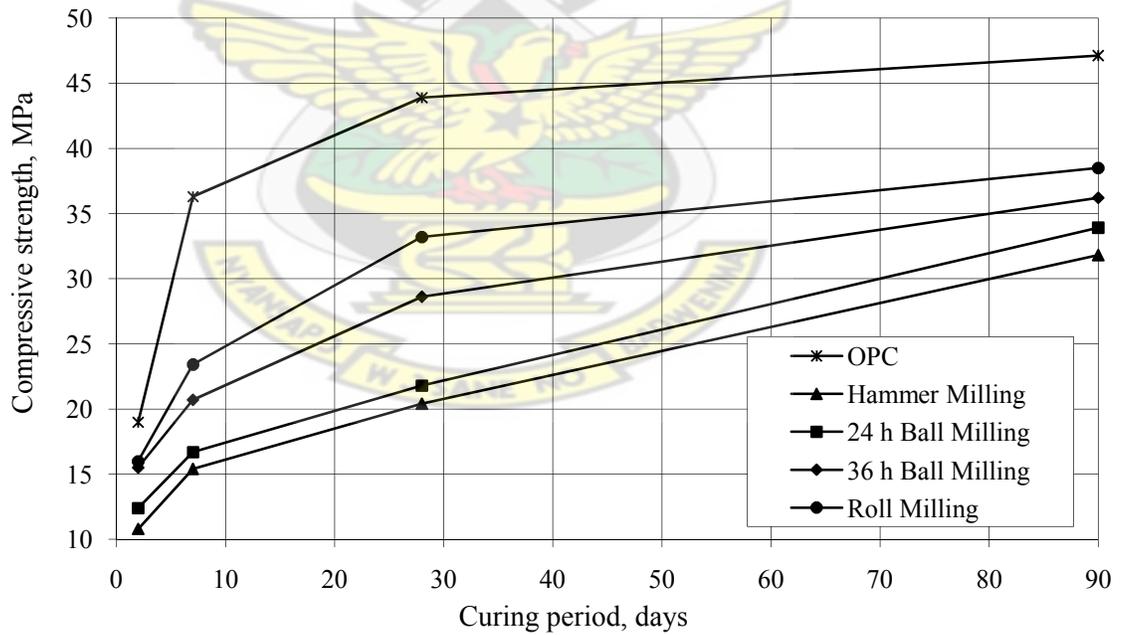


Figure 4-26: Compressive strength development of 35% Mankessim pozzolana cement mortar cubes

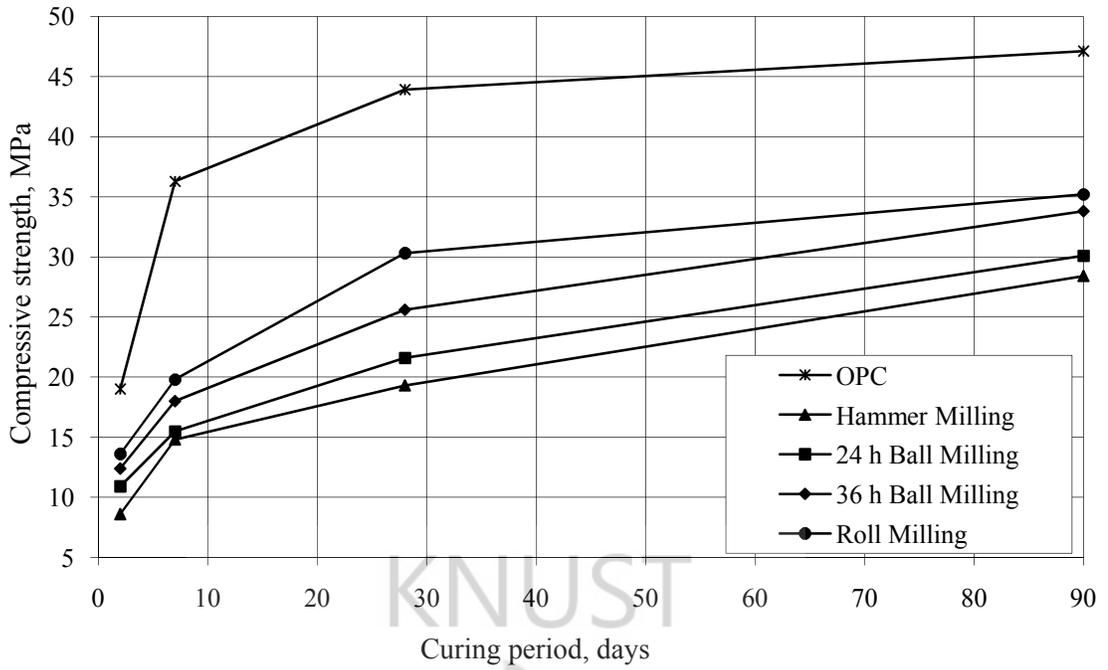


Figure 4-27: Compressive strength development of 40% Mankessim pozzolana cement mortar cubes

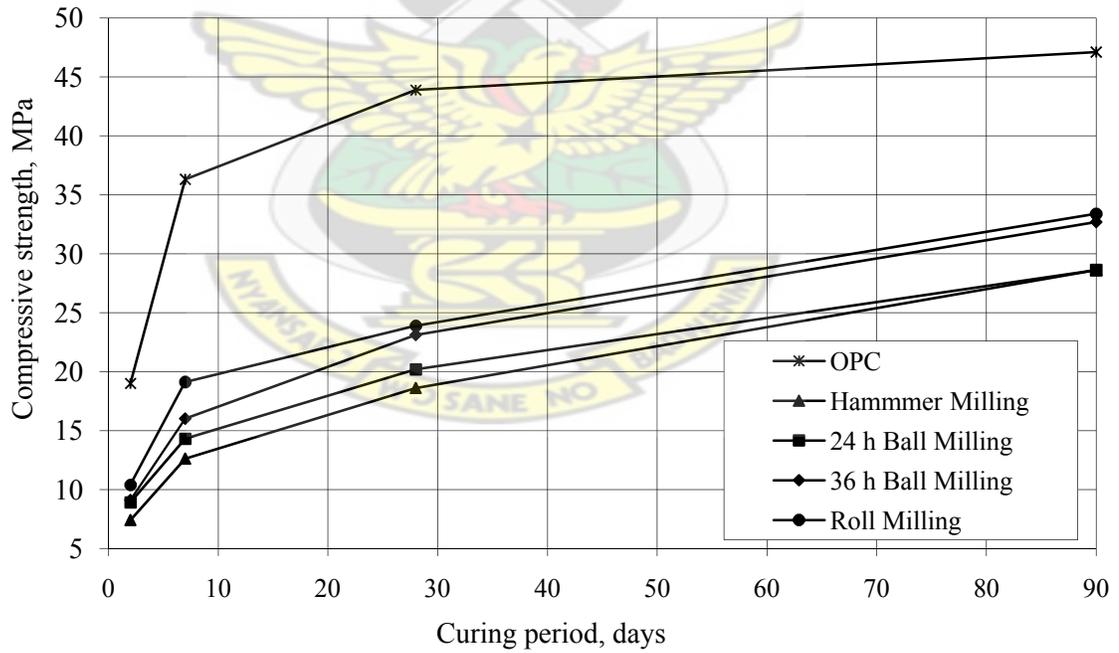


Figure 4-28: Compressive strength development of 50% Mankessim pozzolana cement mortar cubes

As with the other pozzolana samples evaluated, compressive strengths decreased with increasing pozzolana content. Compressive strengths also increased with increasing curing period as expected for all cement replacement levels. Figures 4-25 – 4-27, however, show that Mankessim pozzolana cement mortar cubes gained in compressive strength more rapidly than ordinary Portland cement mortar cubes, especially after 28 days curing. The finer pozzolana cement mortar cubes (36 h ball milled and roll milled) obtained substantially higher compressive strengths than the less fine (hammer milled and 24 h ball milled) pozzolana cement mortar cubes for all cement replacement levels. Thus highest strengths were obtained with the roll milled product whereas the least compressive strengths were obtained with the hammer milled product.

With the hammer milled product as a basis, the 36 h ball milled pozzolana obtained an increase in compressive strength 61.5 and 40% after 28 days for 30 and 35% cement replacement respectively, whilst the roll milled pozzolana cement mortar cubes showed an even improved strength increment of 83.9 and 62.7% for 30 and 35% cement replacement respectively. At 90 days, however, the compressive strength differences had reduced considerably. The roll milled product obtained strength increments in the range of 21-33% whilst the 36 h ball milled product obtained increments in the range of 13-32%.

Lower compressive strength gains were obtained at 40 and 50% cement replacement as compared to 30 and 35% cement replacement. The 36 h ball milled product obtained 24-33% compressive strength increment whilst that of the roll milled pozzolana cement obtained 28-57% after 28 days curing. At 90 days, as was the case for 30-35% cement replacement, the differences in strengths dropped. The 36 h ball milled Mankranso

pozzolana showed strength gains between 14-19% whilst the roll milled product obtained 16-24% strength increment over the hammer milled product.

Early age strength

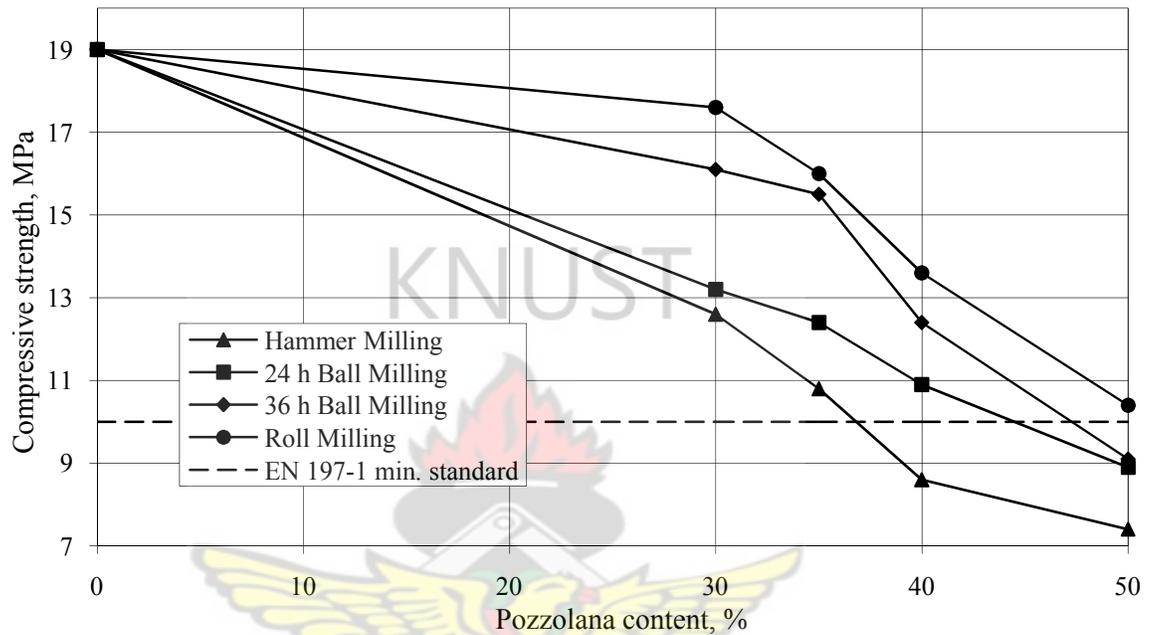


Figure 4-29: 2 - day compressive strength of Mankessim pozzolana cement

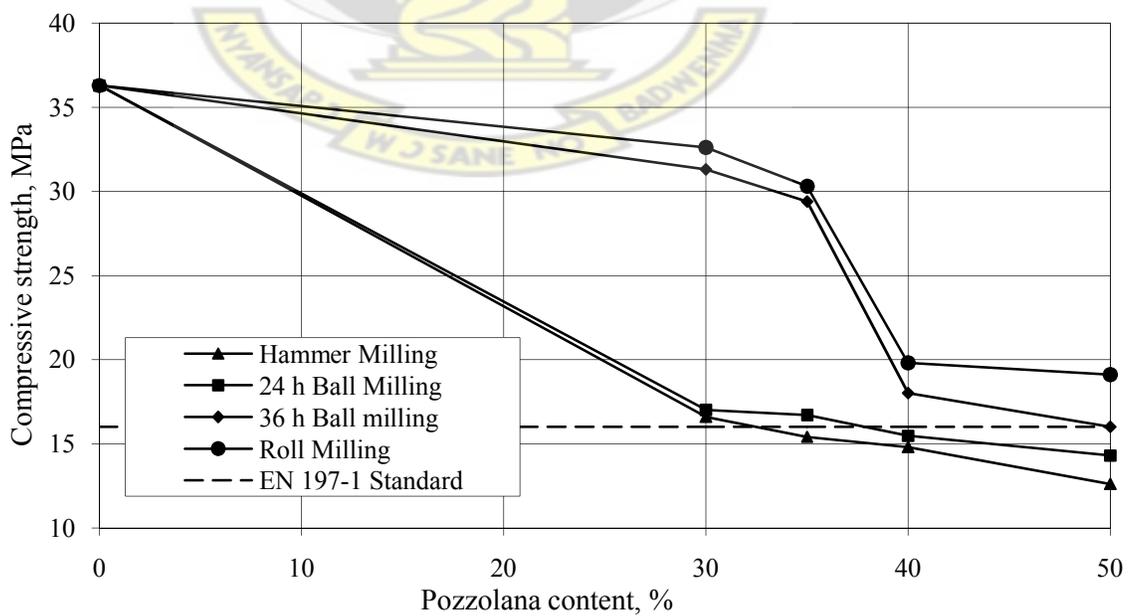


Figure 4-30: 7 - day compressive strength of Mankessim pozzolana cement

At two days the 36 h ball milling and roll mill products attained compressive strengths equal to or greater than the EN 197-1 minimum standard of 10 MPa except at 50% pozzolana content where the 36 h ball milled pozzolana cement had a lower compressive strength. Strengths much lower than that of OPC (19.0 MPa) were however obtained for all levels of OPC replacement. Compressive strengths of the activated pozzolana cements at 36 h ball milling and the roll milled product decreased with increasing pozzolana content with the highest strengths being obtained at 30% cement replacement. The 24 h ball milled pozzolana cement satisfied the EN 197-1 minimum strength up to a pozzolana content of 40% whilst the hammer mill product only satisfied the European standard only up to 30% cement replacement.

At 7 days as shown in Figure 4-30, the pozzolana cement mortar cubes had gained significantly in compressive strength such that the 24 h and 36 h ball milled and the roll milled products had compressive strengths greater than the EN 197-1 minimum standard of 16.0 for class 32.5N cements at all cement replacement levels. Compressive strengths of the pozzolana cement mortars generally decreased as pozzolana content in the blended cement increased with the roll mill product obtaining the highest strengths at all levels of pozzolana content. Thus the finer the pozzolana, the greater the compressive strength obtained up to 50% pozzolana content.

Ultimate strength

Figures 4-31 and 4-32 show the compressive strengths of the pozzolana cement mortar cubes containing 30-50% of Mankessim pozzolana after 28 and 90 days curing respectively.

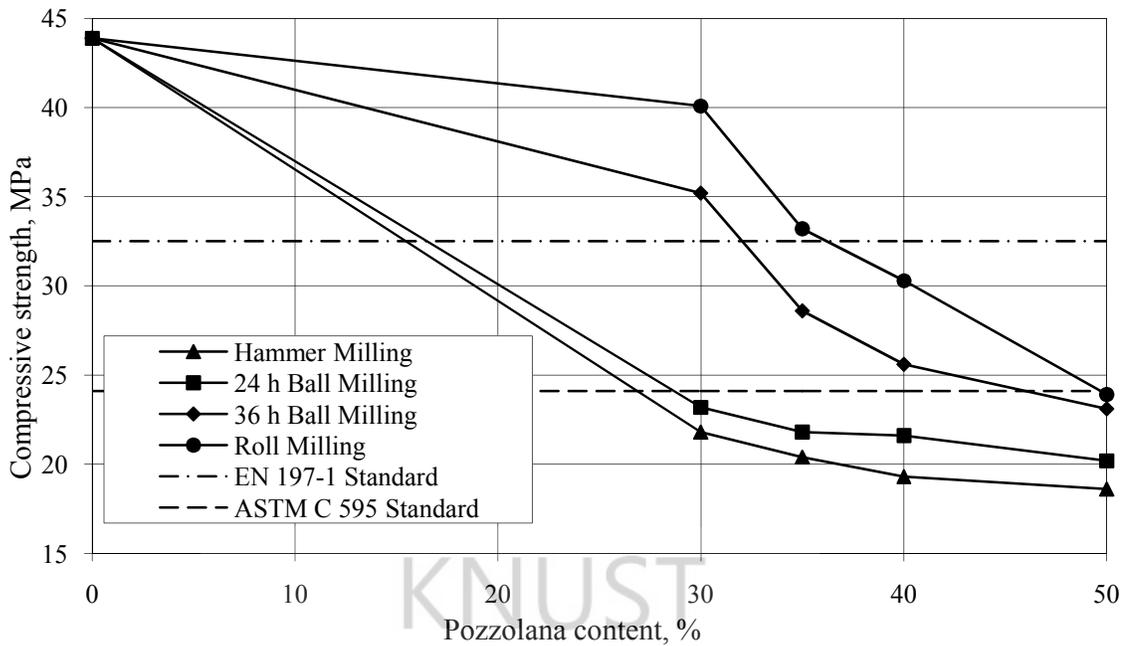


Figure 4-31: 28-day compressive strength of Mankessim pozzolana

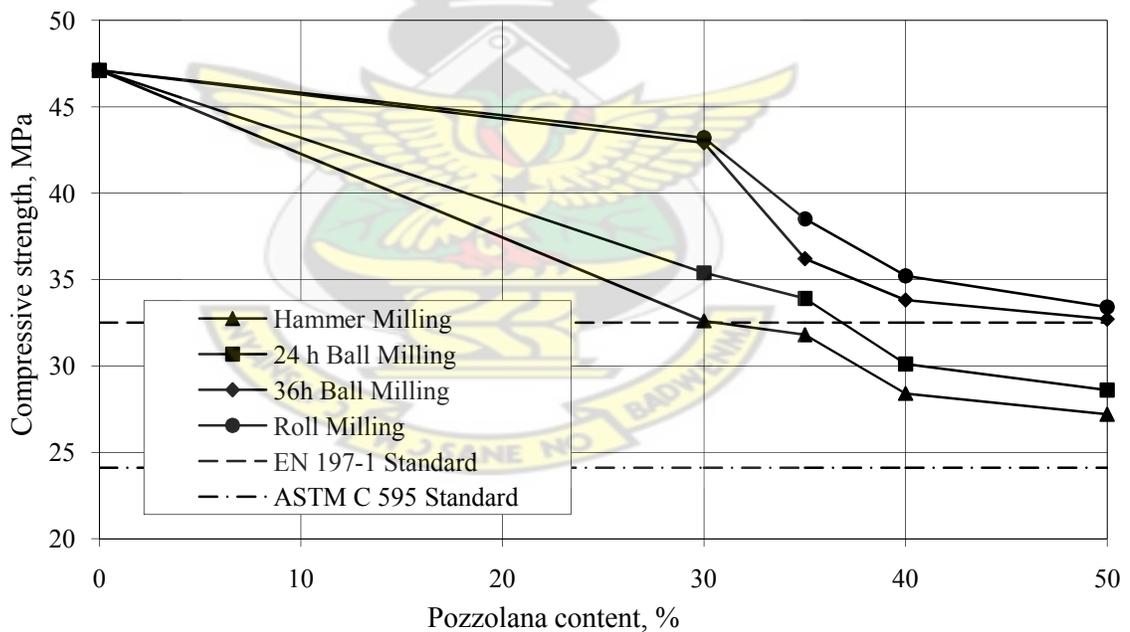


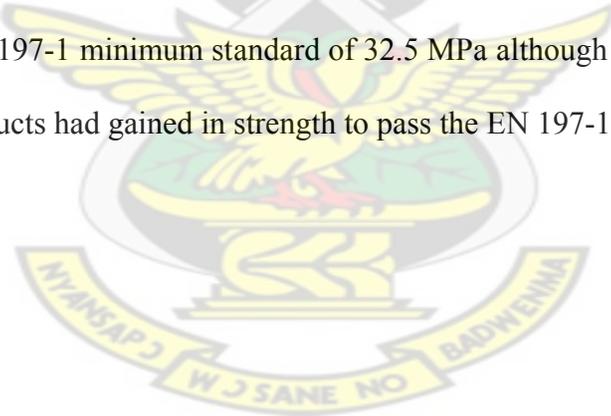
Figure 4-32: 90-day compressive strength of Mankessim pozzolana

The 36 h ball milled and roll milled pozzolana cement mortar cubes attained strengths equal to or greater than the EN 197-1 minimum standard of 32.5 MPa at 28 days for 30% cement replacement. Beyond 30% replacement of OPC, the compressive strengths obtained fell

short of the EN 197-1 minimum standard except for the roll milled product which satisfied the European standard up to 35% cement replacement. However the Mankessim pozzolana cement containing after 36 h ball milling and by roll milling attained compressive strengths above the ASTM C 595 minimum standard of 24.1 MPa except at 50% OPC replacement where the 36 h ball milled pozzolana cement obtained 23.1 MPa compressive strength. The 24 h ball milled and the hammer milled pozzolana cement mortar cubes after 28 days curing could neither satisfy the EN 197-1 of 32.5 MPa nor the ASTM C 595 minimum standard of 24.1 MPa for any of the cement replacement levels tested.

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After 90 days curing, all the Mankessim pozzolana cement mortar cubes attained strengths equal to or greater than 24.1 MPa — the ASTM C 595 minimum standard for all cement replacement levels. The hammer milled and 24 h ball milled pozzolana cements still failed to meet the EN 197-1 minimum standard of 32.5 MPa although the 36 h ball milled and the roll milled products had gained in strength to pass the EN 197-1 standard.



4.3.4 Tanoso pozzolana

Table 4-11: Compressive strengths of Tanoso pozzolana cement mortar cubes

Milling type and period	Curing period, days			
	2	7	28	90
Compressive strength, MPa				
30% cement replacement				
Hammer milling	15.4	18.4	32.4	33.1
24 h Ball milling	16.0	19.4	33.7	39.4
36 h Ball milling	18.4	28.1	41.0	44.2
Roll milling	18.9	32.2	42.0	45.0
35% cement replacement				
Hammer milling	14.2	16.6	28.2	31.8
24 h Ball milling	14.7	17.2	31.2	36.6
36 h Ball milling	17.4	24.6	36.2	41.1
Roll milling	18.2	30.5	39.6	42.6
40% cement replacement				
Hammer milling	11.6	14.8	24.6	26.5
24 h Ball milling	12.4	15.5	26.1	32.5
36 h Ball milling	15.7	22.0	35.8	36.6
Roll milling	17.0	23.2	36.9	39.7
50% cement replacement				
Hammer milling	9.5	12.0	22.4	25.0
24 h Ball milling	10.0	14.3	23.0	28.6
36 h Ball milling	13.9	19.9	32.9	34.2
Roll milling	15.0	20.0	34.2	36.4
0% cement replacement (OPC)				
	19.0	36.3	43.9	47.1

The 36 h ball milled pozzolana obtained between 73.2-96.8% of the strength of the OPC for the cement replacement levels studied whilst the roll milled pozzolana attained 78.9-99.5% of the OPC compressive strength at 2 days. At 7 days, the 36 h ball milled pozzolana gained between 54.8-77.4% of the compressive strength of OPC for all cement replacement levels whilst the roll milled pozzolana, which always performed better, attained between 55.1-88.7% of the OPC strength.

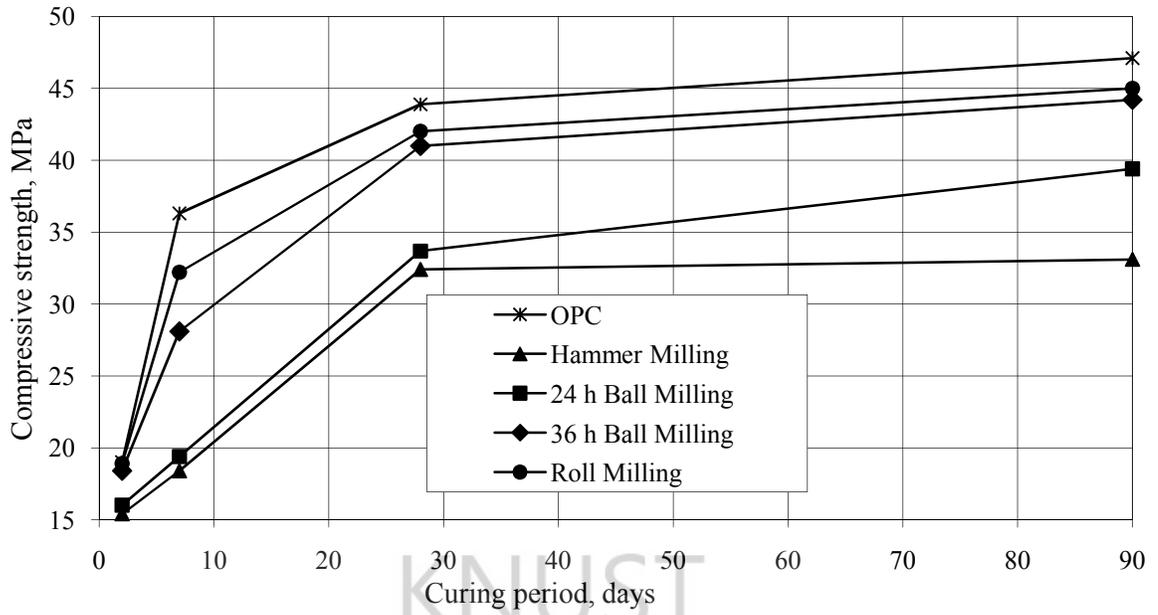


Figure 4-33: Compressive strength development of 30% Tanoso pozzolana cement mortar cubes

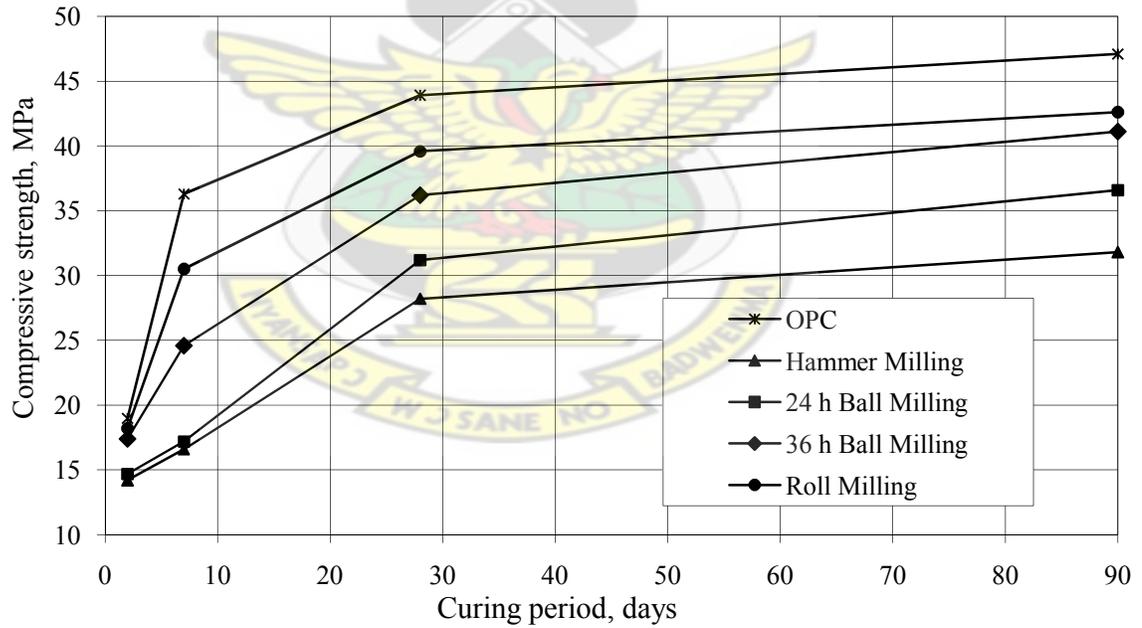


Figure 4-34: Compressive strength development of 35% Tanoso pozzolana cement mortar cubes

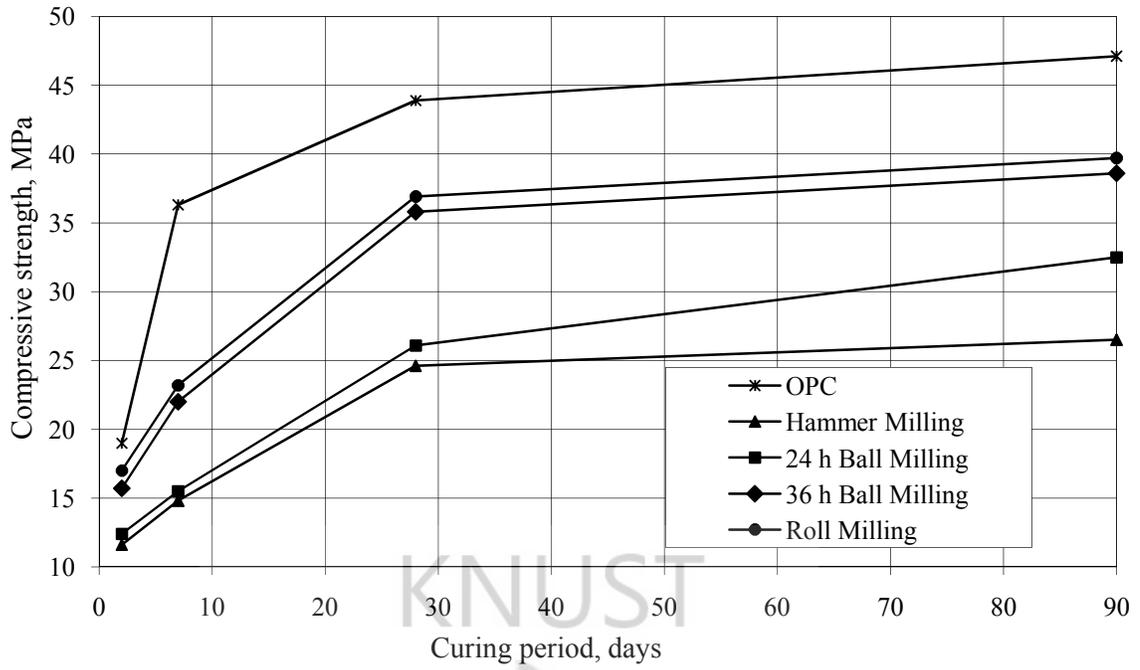


Figure 4-35: Compressive strength development of 40% Tanoso pozzolana cement mortar cubes

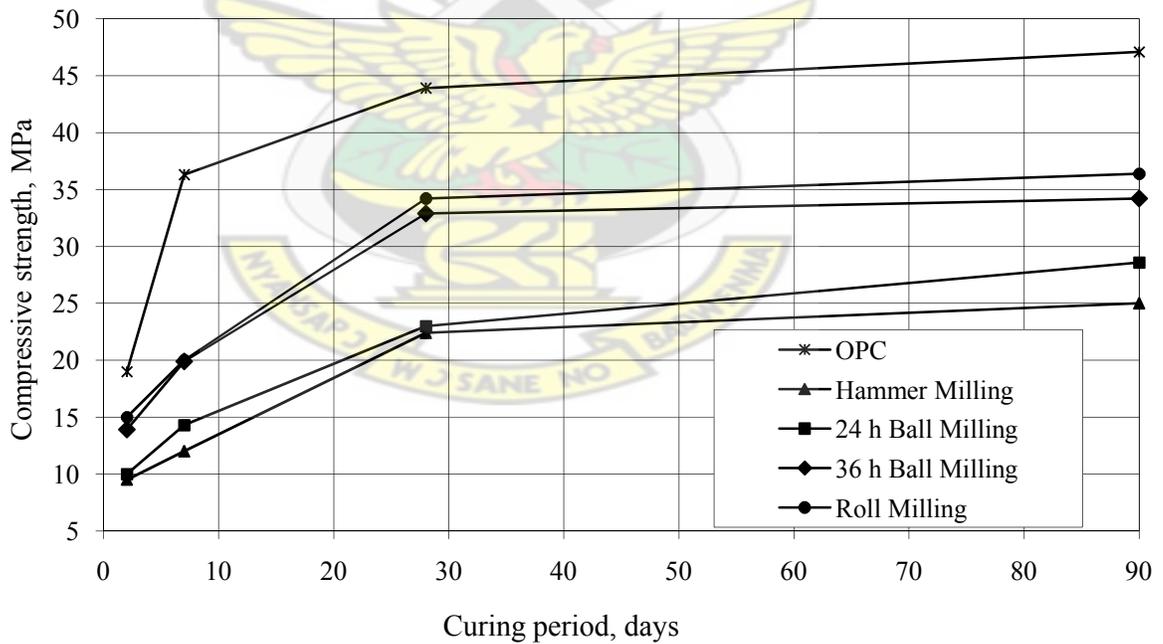


Figure 4-36: Compressive strength development of 50% Tanoso pozzolana cement mortar cubes

Better strengths were even recorded after 28 days with the 36 h ball milled pozzolana obtaining between 74.9-93.4% of the OPC strength whilst the roll milled pozzolana attained between 77.9-95.7% of the OPC compressive strength for all replacement levels tested. Using hammer milled pozzolana which had the least strengths as a basis, finer particle sizes resulted in substantial increments in compressive strengths. The 36 h ball milled pozzolana attained increments in compressive strength between 26.5-49.9% at 28 days whilst the roll milled pozzolana had even greater increments between 29.6-59.7% over the strengths of the hammer milled pozzolana at 28 days for the cement replacement levels tested.

Early age strength

The 36 h ball milled and roll milled products attained compressive strengths equal to or greater than the EN 197-1 minimum standard of 10 MPa after 2 days for all cement replacement levels. Compressive strengths of the 36 h ball milled and roll milled activated pozzolana cements decreased with increasing pozzolana content with the highest strengths being obtained at 30% pozzolana content. The 24 h ball milled pozzolana cement satisfied the EN 197-1 minimum strength after 2 days up to a pozzolana content of 50% whilst the hammer mill product satisfied the standard only up to 40% cement replacement.

After 7 days, compressive strengths of the 36 h ball milled and roll milled pozzolana cement mortar cubes were greater than the EN 197-1 minimum standard of 16 MPa although sharp losses in strengths were observed as compared to the strength obtained by the control OPC (36.3 MPa). The 24 h ball milled and hammer milled pozzolana cements satisfied the EN 197-1 minimum standard only up to 35% pozzolana content.

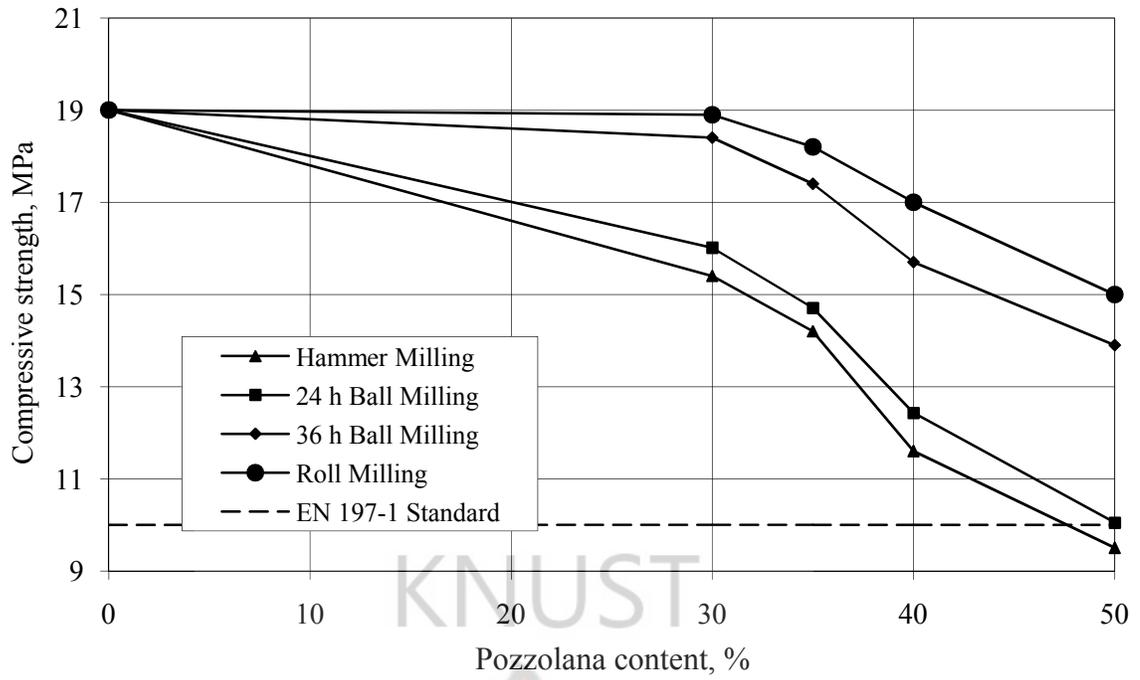


Figure 4-37: 2-day compressive strength of Tanoso pozzolana

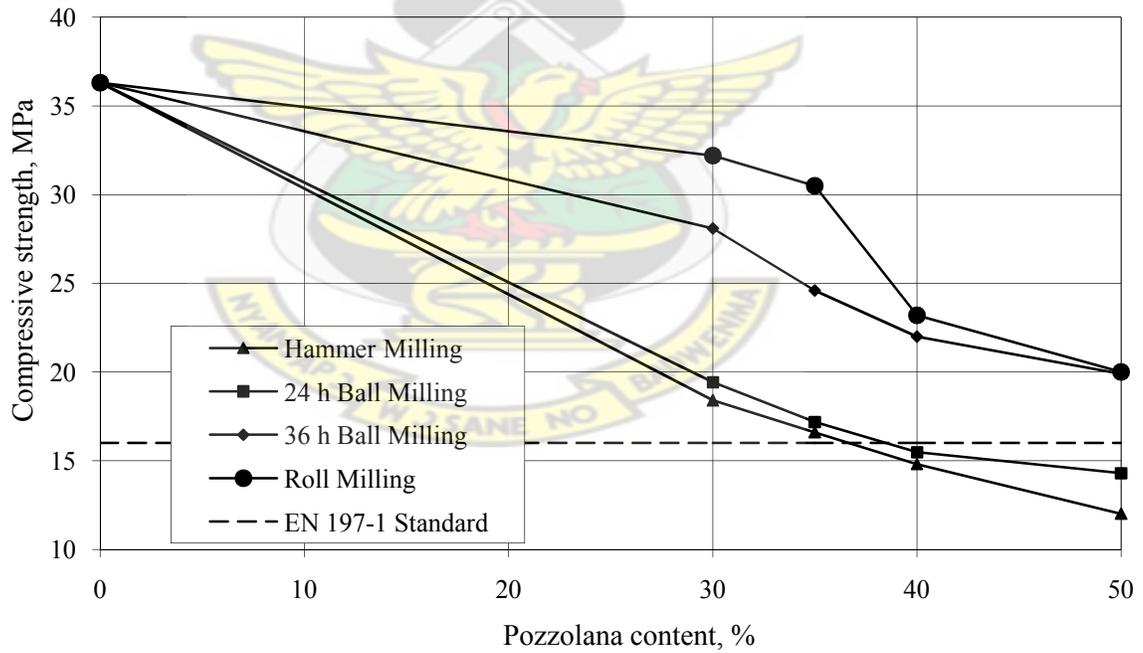


Figure 4-38: 7-day compressive strength of Tanoso pozzolana

Ultimate strength

The compressive strengths attained by the activated pozzolana cement mortar cubes after 28 and 90 days curing are shown in Figures 4-39 and 4-40.

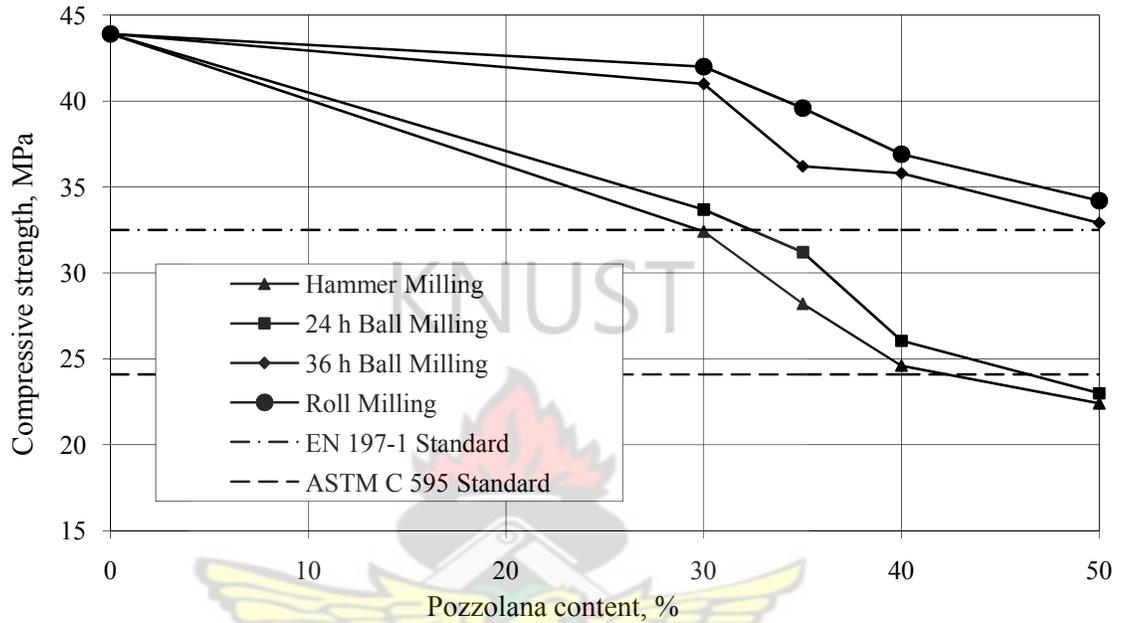


Figure 4-39: 28-day compressive strength of Tanoso pozzolana

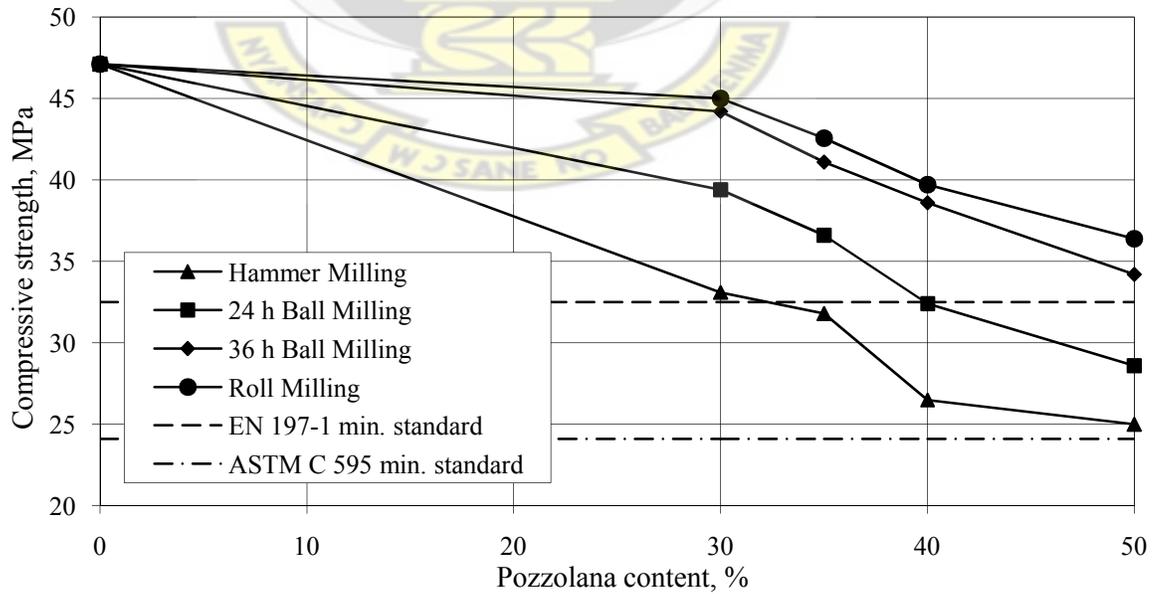


Figure 4-40: 90-day compressive strength of Tanoso pozzolana

After 28 days curing, the pozzolana cement mortar cubes attained strengths lower than those of the ordinary Portland cement mortar cubes. However, the 36 h ball milled and roll milled products attained strengths greater than 32.5 MPa which is the EN 197-1 minimum standard for all pozzolana replacement levels. Above 30% replacement, the pozzolana cement mortar cubes made from the 24 h ball milled and hammer milled products failed to meet the EN 197-1 standard.

However, the pozzolana cement mortar cubes satisfied the minimum strength requirement of 24.1 MPa as stipulated in ASTM C 595 for replacement levels up to 50% although strengths reduced with increasing pozzolana content in the blended cement. At 90 days curing, the 36 h ball milled and the roll milled products attained compressive strengths equal to or greater than 32.5 MPa — the EN 197-1 minimum standard for cement at all cement replacement levels. The 24 h ball milled pozzolana cement mortar had also gained in strength to satisfy the EN 197-1 minimum standard of 32.5 MPa up to 40% pozzolana content.

The 90-day compressive strengths of roll milled products for the four pozzolana samples are shown in Figure 4-41. The roll milled mortar cubes of the four pozzolanas studied attained compressive strengths greater than the EN 197-1 minimum of 32.5 MPa. Mankranso pozzolana had the highest compressive strength of 46.3 MPa at 30% pozzolana content. Mfensi pozzolana had the highest compressive strengths beyond 35% pozzolana content with a compressive strength of 40 MPa at 50% pozzolana content.

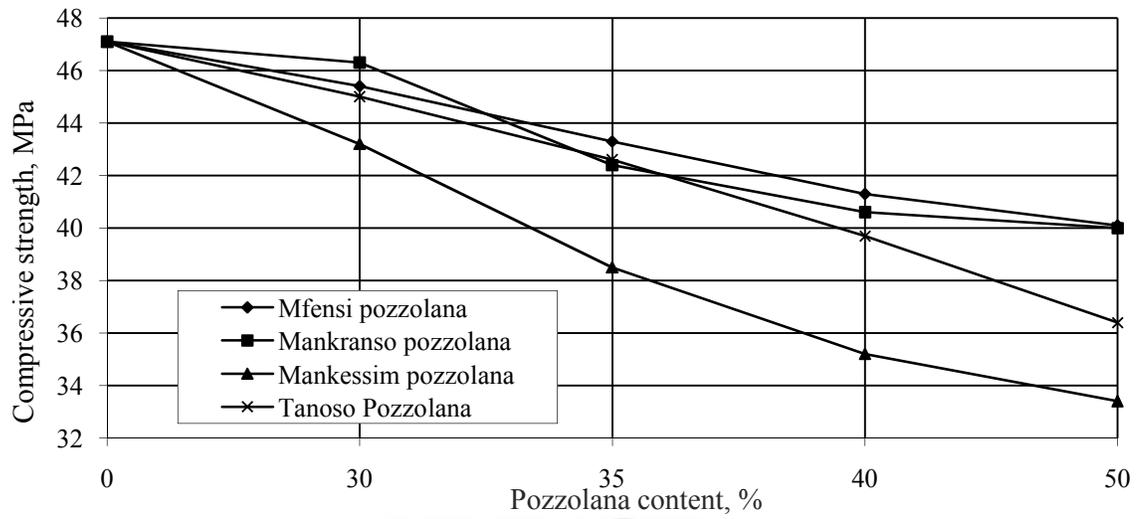


Figure 4-41: 90-day compressive strength of roll milled pozzolanas



4.4 Standard Consistence and Setting Times

4.4.1 Mfensi pozzolana

The results for standard consistence as well as initial and final setting times for Mfensi pozzolana samples are given in Table 4-12. The data are also presented in Figures 4-42 – 4-44.

Table 4-12: Setting times and standard consistence for Mfensi pozzolana

Milling type and period	Setting time, min		Standard consistence, %
	Initial	Final	
30% cement replacement			
Hammer milling	135	251	40.6
24 h ball milling	123	240	43
36 h ball milling	110	237	47.6
Roll milling	98	228	47.8
35% cement replacement			
Hammer milling	112	246	44.6
24 h ball milling	86	230	47.4
36 h ball milling	70	225	49.2
Roll milling	64	219	50.1
40% cement replacement			
Hammer milling	87	240	46.4
24 h ball milling	64	218	48.2
36 h ball milling	60	207	52.6
Roll milling	56	198	53.5
50% cement replacement			
Hammer milling	79	225	48
24 h ball milling	61	168	52
36 h ball milling	50	155	57.2
Roll milling	48	142	58.2
0% cement replacement (OPC)			
	185	280	28.2

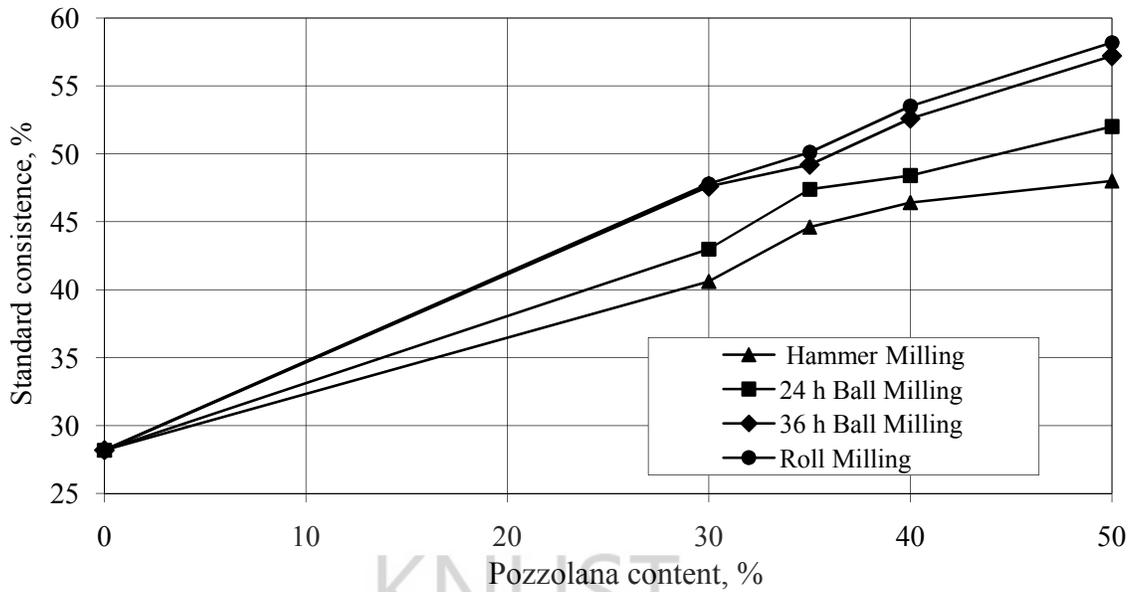


Figure 4-42: Standard consistence of Mfensi pozzolana

Standard consistence

The standard consistence (or water requirement) of the Mfensi pozzolana cement pastes increased steadily as the pozzolana content of the paste increased for all types of grinding. The standard consistence also increased with increasing fineness. Thus, the lowest standard consistence was obtained for hammer milling whilst the highest was recorded for roll milling. OPC paste had a standard consistence of 28.2% which increased to 58.2% when 50% of it was replaced by the roll mill product. This behavior can be explained by the fact that decreasing the particle size during milling results in a lower bulk density of the powdered pozzolana. This implies that larger volumes would be required to replace an equal mass of OPC, therefore the amount of water needed to get a workable paste also increased.

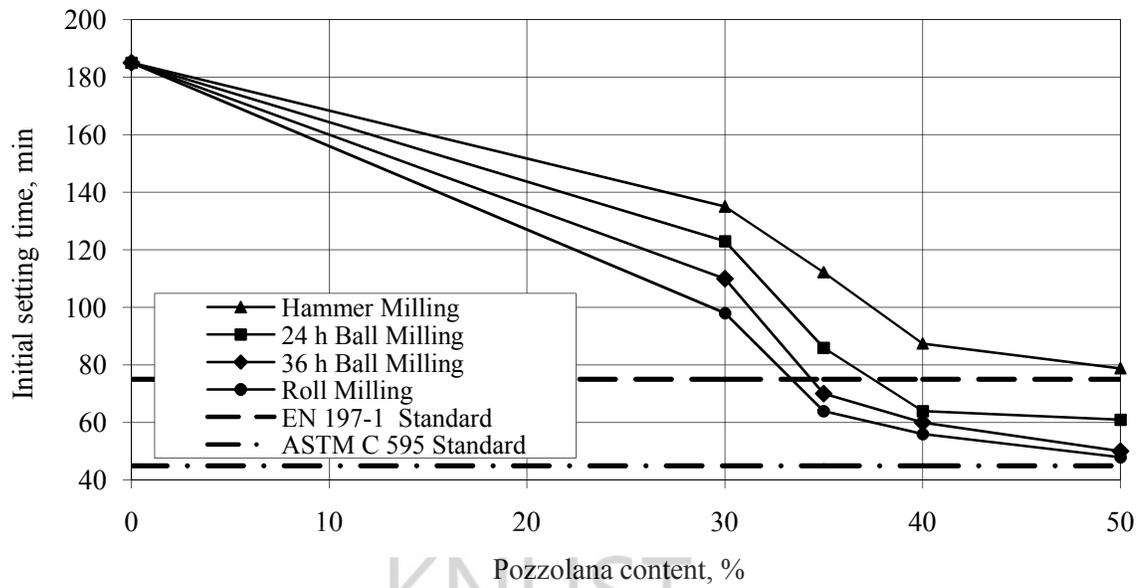


Figure 4-43: Initial setting time of Mfensi pozzolana

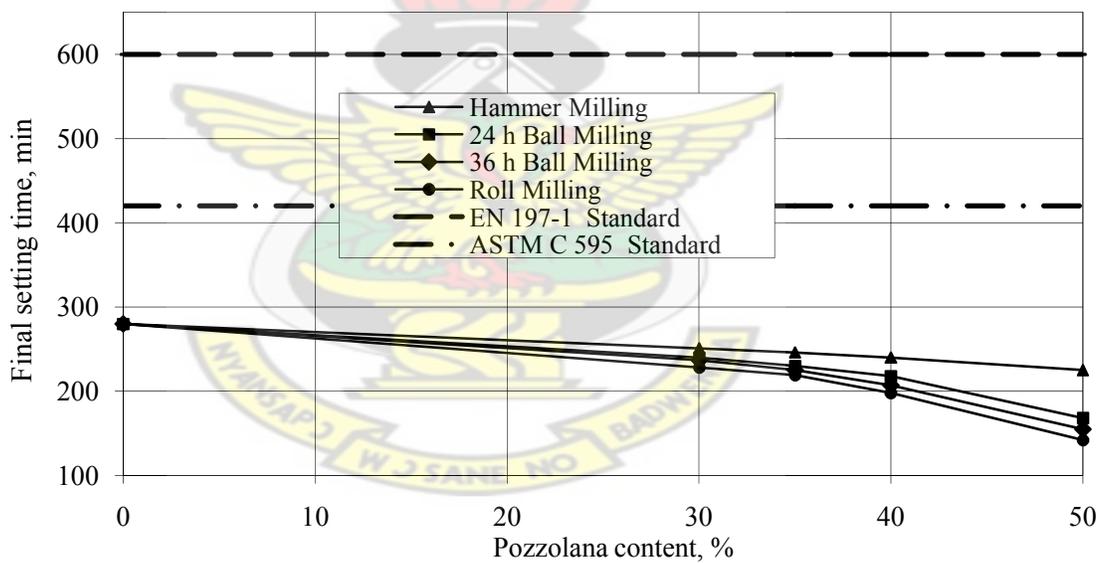


Figure 4-44: Final setting time of Mfensi pozzolana

Initial setting time

Initial setting times decreased significantly as pozzolana content in the blended cements increased. The decrease was pronounced for all milled products. This was because at such

high fineness levels the pozzolana reacted faster with the ordinary Portland cement leading to faster setting. Although above 30% pozzolana content the activated pozzolana cements set faster than the EN 197-1 minimum standard of 75 min they all set well above the ASTM C 595 minimum standard of 45 min.

Final setting time

Final setting times for all degrees of milling decreased with increasing pozzolana content. All the Mfensi pozzolana samples attained final sets below 5 h which satisfied both the EN 197-1 and ASTM C 595 maximum standards of 600 min (10 h) and 420 min (7 h) respectively as is shown in Figure 4-44. As the pozzolana content of the pastes increased, the final setting times decreased for all the different types of milling. For a particular pozzolana content of the paste, the coarser pozzolana obtained by hammer milling had the highest final setting time and the finest sample obtained by roll milling gave the lowest final setting time.

4.4.2 Mankranso pozzolana

The standard consistence of the activated pozzolana cement with Mankranso pozzolana is shown in Figure 4-45 whilst the initial and final setting times are shown in Figures 4-46 and 4-47. The data are also presented in Table 4-13.

Table 4-13: Setting times and standard consistence of Mankranso pozzolana

Milling type and period	Setting time, min		Standard consistence, %
	Initial	Final	
30% cement replacement			
Hammer milling	181	275	28.8
24 h ball milling	128	255	29.2
36 h ball milling	67	230	46.4
Roll milling	63	226	48.2
35% cement replacement			
Hammer milling	160	256	32.2
24 h ball milling	96	240	40.8
36 h ball milling	56	218	48.0
Roll milling	50	204	52.5
40% cement replacement			
Hammer milling	148	242	35.0
24 h ball milling	75	228	41.0
36 h ball milling	50	206	50.0
Roll milling	47	198	56.0
50% cement replacement			
Hammer milling	130	225	36.8
24 h ball milling	68	186	46.0
36 h ball milling	47	180	59.0
Roll milling	41	174	60.0
0% cement replacement (OPC)			
	185	280	28.2

Standard consistence

Figure 4-45 shows that the activated pozzolana cements required a high water content to form a workable paste. The standard consistence increased as fineness and pozzolana content increased and was always much higher than that attained by OPC. The highest standard consistence, obtained for cement paste containing 50% roll milled pozzolana was 60% which was 31.8% higher than the OPC standard consistence. As expected, increasing pozzolana content in the paste also resulted in increasing standard consistence.

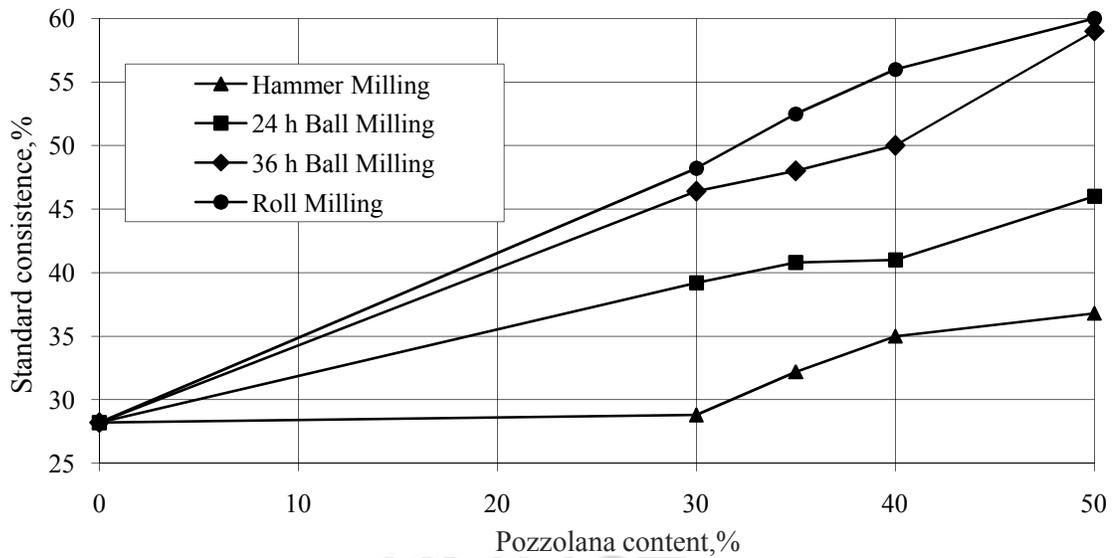


Figure 4-45: Standard consistence of Mankranso pozzolana

Initial setting time

The hammer mill product satisfied both the EN 197-1 and ASTM C 595 minimum standards of 75 min and 45 min respectively for pozzolana contents up to 50%. As fineness increased, the initial setting time decreased with increasing pozzolana content. Thus the 36 h ball and roll milled activated pozzolana cements failed to meet the EN 197-1 minimum standard at all replacement levels up to 50%. However they complied with the ASTM C 595 minimum standard of 45 min for pozzolana contents up to 40%. The low initial setting times at high fineness as obtained for the 36 h ball milled and roll milled products indicate enhanced reactivity of the activated pozzolana cements upon hydration.

Final setting time

Figure 4-47 shows the final setting times of Mankranso pozzolana cements. The pozzolana cements attained final sets faster as fineness increased and as pozzolana content increased up to 50%. The final setting times were lower than that obtained for the parent OPC. Final setting times attained at all pozzolana content and fineness levels however satisfied both the

ASTM C 595 and EN 197-1 maximum setting time for cement of 420 min and 600 min respectively.

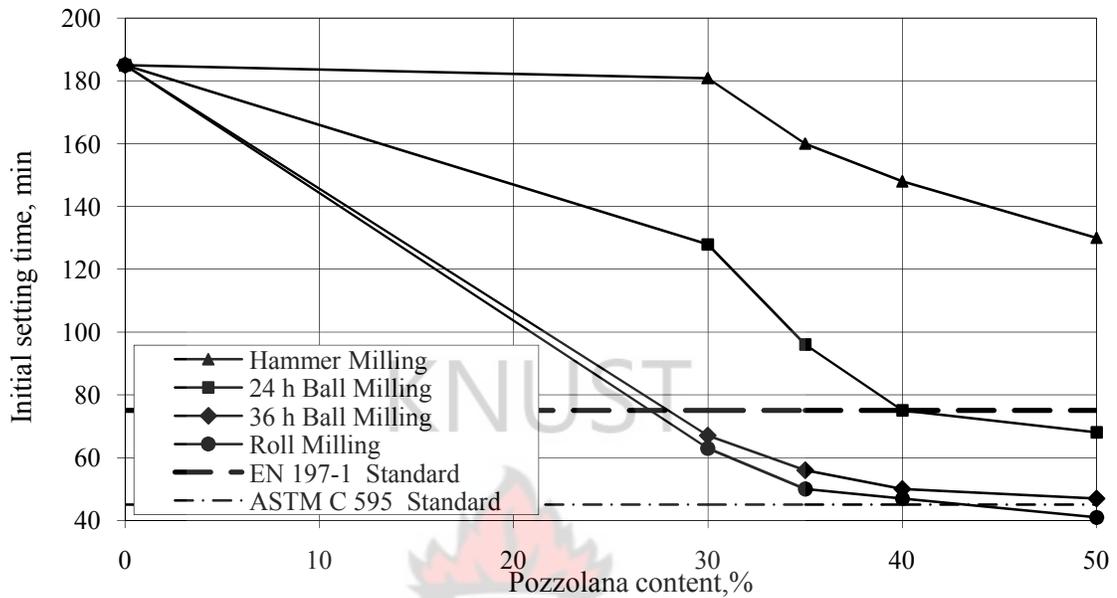


Figure 4-46: Initial setting time of Mankranso pozzolanas

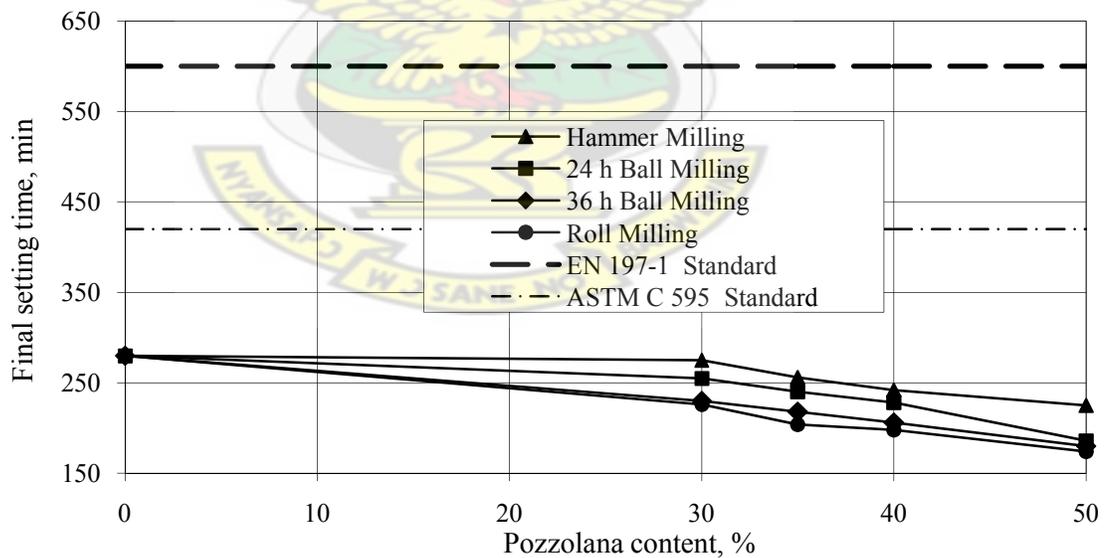


Figure 4-47: Final setting time of Mankranso pozzolanas

4.4.3 Mankessim pozzolana

Table 4-14: Setting times and standard consistence of Mankessim pozzolana

Milling type and period	Setting time, min		Standard consistence, %
	Initial	Final	
30% cement replacement			
Hammer milling	162	265	32.0
24 h ball milling	135	272	38.0
36 h ball milling	98	218	48.4
Roll milling	96	211	50.1
35% cement replacement			
Hammer milling	150	252	36.8
24 h ball milling	108	258	43.2
36 h ball milling	65	207	49.6
Roll milling	62	199	51.8
40% cement replacement			
Hammer milling	132	241	40.4
24 h ball milling	92	240	46.4
36 h ball milling	58	196	52.0
Roll milling	54	190	57.2
50% cement replacement			
Hammer milling	120	232	43.2
24 h ball milling	68	202	48.6
36 h ball milling	45	152	57.6
Roll milling	45	150	57.5
0% cement replacement (OPC)			
	185	280	28.2

Standard consistence

The standard consistencies of the Mankessim pozzolana cement samples are presented in Figure 4-48. Standard consistence increased with increasing fineness and pozzolana content. All the pozzolana cement samples tested had higher standard consistencies than the OPC mortar.

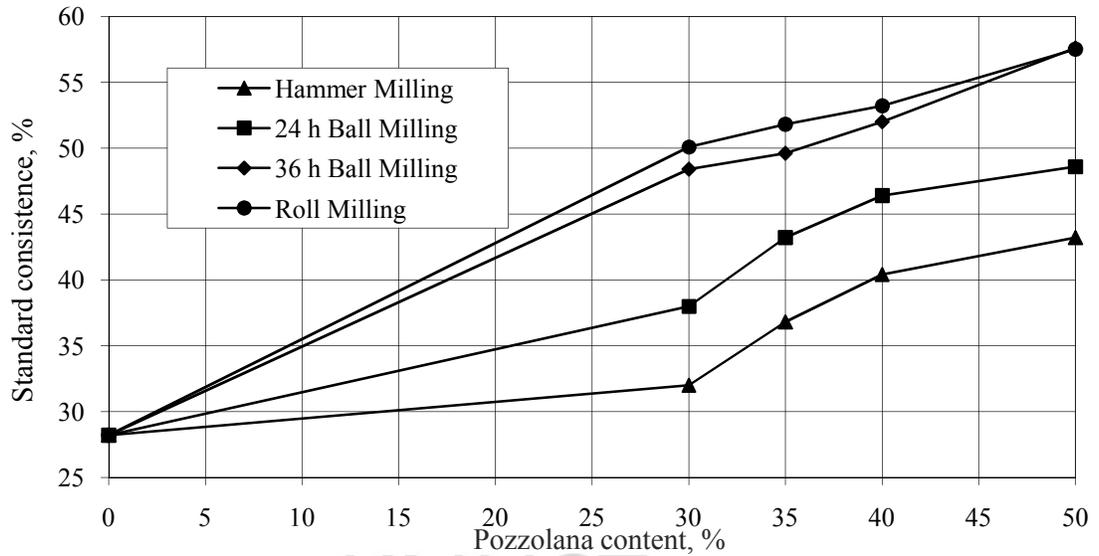


Figure 4-48: Standard consistence of Mankessim pozzolanas

Pozzolana cements containing the 36 h ball milled and roll milled products had very high consistencies ranging between 48-60% for pozzolana contents between 30-50%. This high water demand is probably due to the enhanced reactivity of the activated pozzolanas with OPC on hydration since the 24 h ball milled and hammer milled pozzolanas which were relatively coarser had comparable standard consistencies to OPC mortar.

Initial setting time

Figure 4-49 is the graphical representation of initial setting times obtained for mortars of Mankessim pozzolana. Setting times decreased with increasing fineness and increasing pozzolana content. Cement samples containing 36 h ball milled and roll milled pozzolana had initial setting times lower than the EN 197-1 minimum standard of 75 min for all pozzolana content levels above 35%.

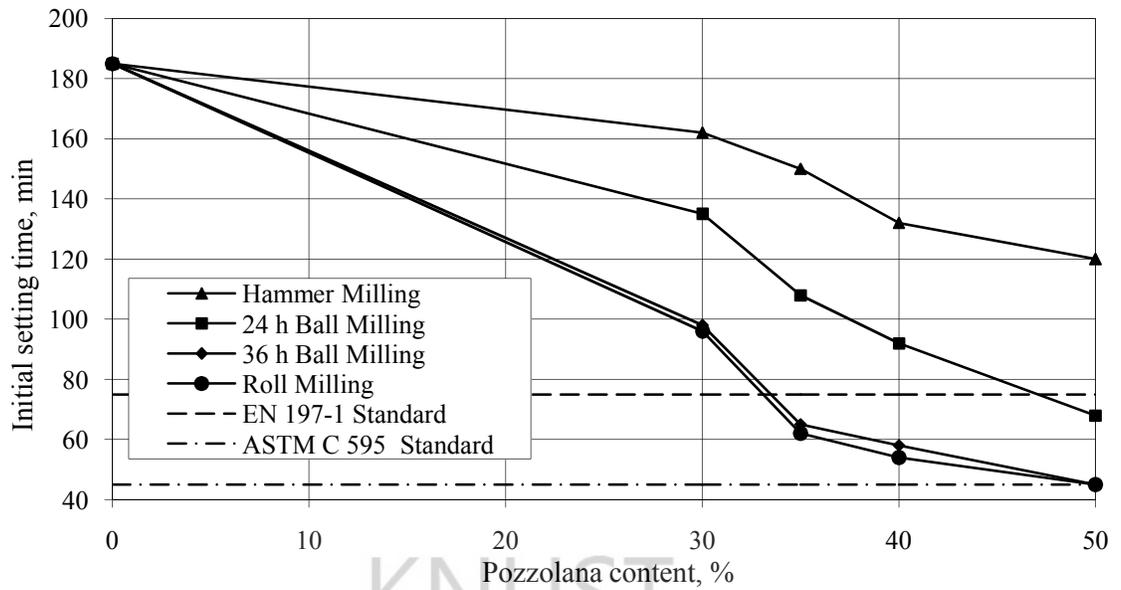


Figure 4-49: Initial setting time of Mankessim pozzolanas

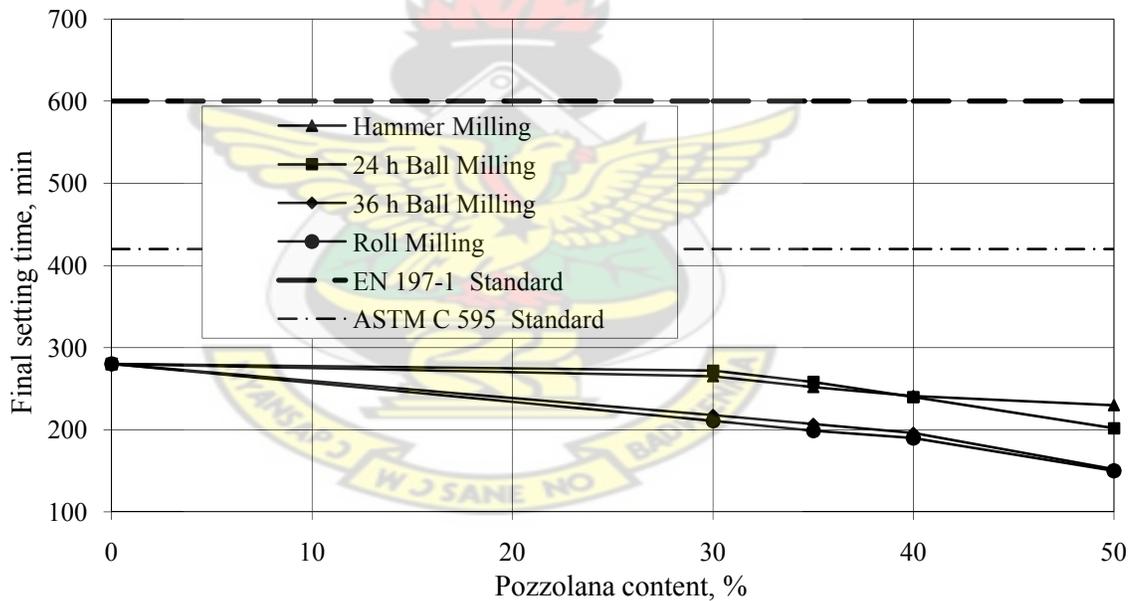


Figure 4-50: Final setting time of Mankessim pozzolanas

All the pozzolana cement samples irrespective of the degree of milling however satisfied the ASTM C 595 minimum standard of 45 min. The low setting times attained by the activated roll milled and 36 h ball milled pozzolana cement samples is an indication of increased reactivity with the increased fineness.

Final setting time

The activated pozzolana cement samples containing Mankessim pozzolana all attained final sets well below both the EN 197-1 and ASTM C 595 maximum standards of 10 and min 7 h respectively as is shown in Figure 4-50. Final setting times for all degrees of activation also decreased with increasing pozzolana content

4.4.4 Tanoso pozzolana

Table 4-15: Setting times and standard consistence of Tanoso pozzolana

Milling type and period	Setting time, min		Standard consistence, %
	Initial	Final	
30% cement replacement			
Hammer milling	158	281	36.0
24 h ball milling	132	272	40.0
36 h ball milling	97	214	49.8
Roll milling	94	208	52.4
35% cement replacement			
Hammer milling	145	269	38.2
24 h ball milling	104	258	42.2
36 h ball milling	64	202	51.6
Roll milling	60	194	53.2
40% cement replacement			
Hammer milling	126	252	41.4
24 h ball milling	85	240	46.8
36 h ball milling	56	192	52.8
Roll milling	50	185	55.2
50% cement replacement			
Hammer milling	114	222	44.6
24 h ball milling	64	202	48.8
36 h ball milling	45	150	56.4
Roll milling	45	146	59.4
0% cement replacement (OPC)			
	185	280	28.2

Standard consistence

At all pozzolana contents, the activated pozzolana cement mortars had higher standard consistencies than the control OPC. This is possibly due to the lower bulk density of the pozzolana which meant that higher volumes had to be used to replace portions of the OPC. The increased reactivity of the pozzolana at higher specific surface areas may also be a contributory factor to the higher than expected consistencies.

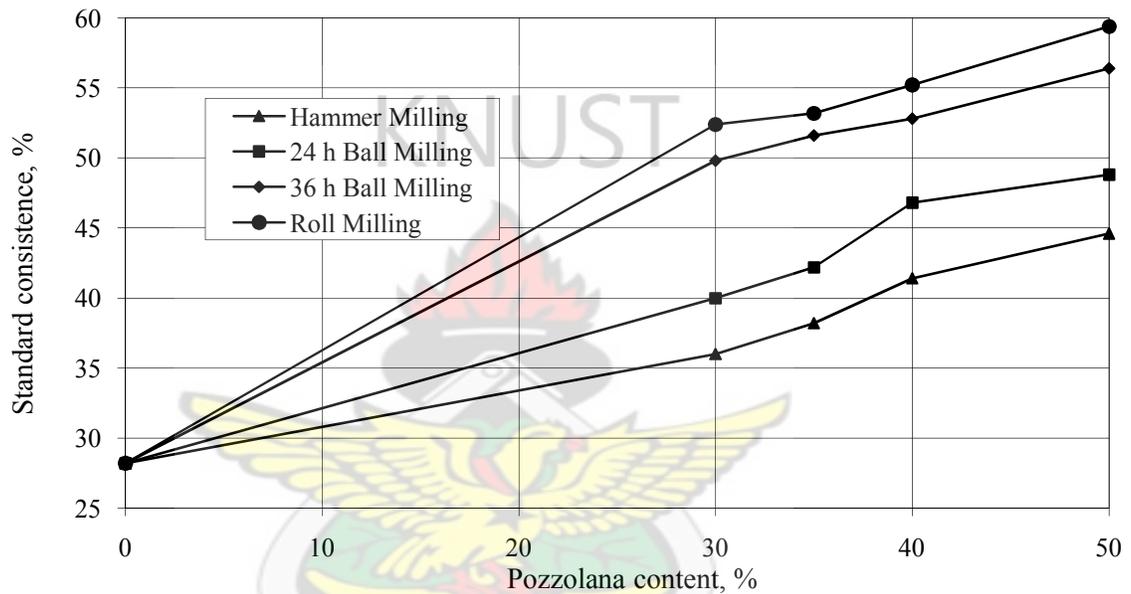


Figure 4-51: Standard consistence of Tanoso pozzolanas

Initial setting time

The initial setting times of the activated pozzolana cement mortars and OPC are shown in Figure 4-52. At all pozzolana contents, the pozzolana cement mortars set faster than the OPC mortar with the rate of setting increasing as fineness increased. Thus there is greater reactivity of the pozzolana with OPC as fineness of the pozzolana increased. The 36 h ball milled and roll milled pozzolana cement mortars set faster than the EN 197-1 minimum standard of 75 min after 35% pozzolana was added to the OPC although the ASTM C 595 minimum standard of 45 min was satisfied at all pozzolana content levels.

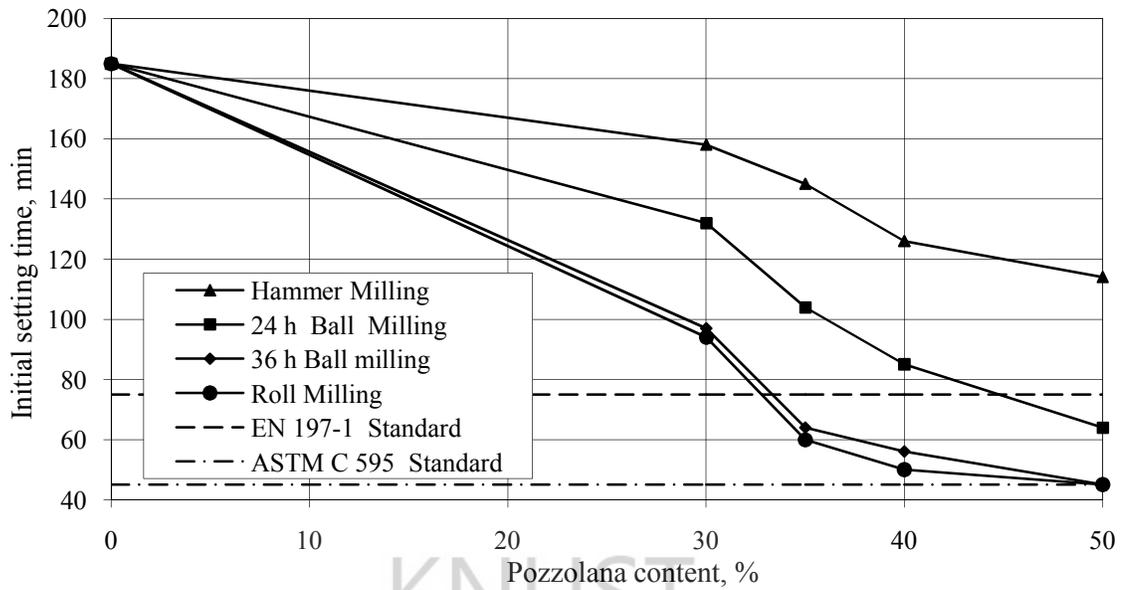


Figure 4-52: Initial setting time of Tanoso pozzolanas

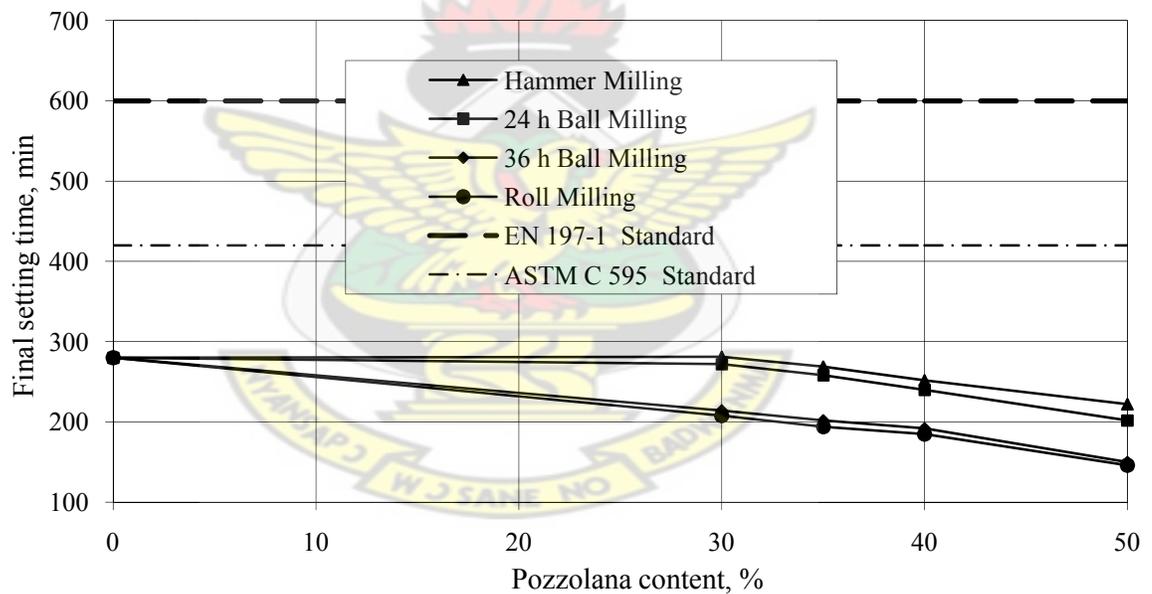


Figure 4-53: Final setting time of Tanoso pozzolanas

Final setting time

The activated pozzolana cement mortars attained final sets well below both the EN 197-1 and ASTM C 595 maxima of 10 and 7 h respectively. Final setting times of the activated pozzolana cement mortars as shown in Figure 4-53 were lower than that of OPC.

CHAPTER FIVE

5 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The following conclusions are drawn on the pozzolana samples based on the degree of activation and the results of the various tests performed on the samples.

5.1.1 Mechanical activation

Mechanical activation of the pozzolana samples can be achieved up to 36 h of ball milling beyond which agglomeration will prevent activation or by roll milling. The Blaine indices obtained after 36 h ball milling and by roll milling were at least 3 times higher than that of the OPC used. which Thus, the four pozzolanas evaluated can be made more reactive and hence used to replace up to 50% OPC for concrete works, which is more than the current 30% as recommended by CSIR-BRRI.

Hammer milling without classification is inappropriate for mechanical activation of the burnt clays studied as specific surface areas attained with the four pozzolana samples were too low to effect any significant changes in the physical properties of the pozzolana cement samples. After 24 h ball milling, the particle sizes of the pozzolana samples were not very fine and as such, compressive strengths obtained with such pozzolana cement samples mostly failed to satisfy both the EN 197-1 and ASTM C 595 minima at 28 days at OPC replacement beyond 35%. Hence mechanical activation cannot be achieved after 24 h ball milling for the four pozzolanas studied.

Roll milling, which employed classification, as well was the best alternative in milling the pozzolanas as it achieved activation at shorter time and hence with lower energy

consumption rates. The highest Blaine specific surface area for roll milling was attained with Mfensi pozzolana followed by Mankranso, Tanoso and Mankessim pozzolanas.

5.1.2 Chemical/physical properties

Chemically, all the pozzolana samples studied contained adequate amounts of reactive silica and alumina as specified by EN 197-1: 2000 with all the samples attaining silica contents above the minimum limit of 25%. The sum of SiO_2 , Al_2O_3 and Fe_2O_3 contents of all the samples was above the ASTM C 618 minimum standard of 70%.

The setting times and standard consistencies of the pozzolana cement samples were all within acceptable standards (ASTM C 595 and EN 197-1), although at high specific surface areas, such as for the 36 h ball milled and roll milled products, the pozzolana cement mortars set much faster than the OPC.

The lower bulk densities of the milled pozzolana samples, which implied that larger volumes would be needed to replace an equal mass of OPC, accounted for the higher standard consistencies for the pozzolana cement mortars. The highest standard consistencies were obtained for the roll milled pozzolanas whilst the lowest consistencies were obtained by the hammer milled products. At the highest extreme was the paste containing 50% of the roll milled Mankranso pozzolana with a consistence of 60%.

Compressive strengths of the pozzolana cement mortar cubes increased with increasing fineness. The highest values for all ages of tests were obtained for the roll milled pozzolana cement followed closely by the 36 h ball milled pozzolanas. At 40 and 50% pozzolana content, the pozzolana cement mortars from the roll mill and 36 h ball milling attained

lower compressive strengths at 2 and 7 days compared to the OPC although both the ASTM C 595 and EN 197-1 minimum standards were met.

Compressive strengths increased for all the pozzolana cements obtained from both the 36 h ball milling and roll milling at all ages and replacement levels. The highest 2- and 7- day compressive strength at 40 and 50% cement replacement was for the roll milled Tanoso pozzolana, whilst the hammer milled product from Mankessim had the lowest compressive strength.

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After 28 and 90 days curing, the 40 and 50% pozzolana cement mortars improved in compressive strength in relation to the OPC. At 40 and 50% replacement, the 28-day compressive strength of the 36 h ball milled and roll milled Tanoso pozzolana cement was the highest followed by that of Mfensi pozzolana cement, Mankranso pozzolana cement and Mankessim pozzolana cement.

Compressive strengths attained for both the 36 h and roll-milled products of all the pozzolana cement samples at 40% pozzolana content were higher than the ASTM C 595 minimum strength of 24.1 MPa recommended for blended cements for general construction. At 50% pozzolana content all but the Mankessim pozzolana cement met the above standard. Hence, at high specific surface areas the pozzolana content in pozzolana cement mortars can be increased beyond 30% without compromising the strength and other physical properties of the cement.

The roll milled and the 36 h ball milled pozzolanas from all the four clay deposits studied can be used to replace up to 40% of ordinary Portland cement and classified as class 32.5 N

Portland Pozzolana cement which meets the EN 197-1 standard. Ordinary Portland cement can be replaced up to 50% with any of the four pozzolana samples to meet the ASTM C 595 standard for blended cements.

Based on the compressive strength results, the best technique available locally to mechanically activate clay pozzolanas is roll milling followed by long hours of ball milling.

5.2 Recommendations

It is recommended that further studies be undertaken with regards to the structural properties of the pozzolana cement at pozzolana contents beyond 30%, since compressive strengths obtained from mortar cubes alone cannot be used to judge the behaviour of the cement in structural concrete.

Further investigations should be conducted into the energy consumption and costs with regards to milling of the pozzolana to achieve activation. This will allow for holistic investor decision making in opting for activated pozzolana production.

It is also recommended that the preparation and adoption of the Ghana Standard on pozzolanas be vigorously pursued by stakeholders in the construction industry to enable safe use of pozzolanic products such as the ones that have been investigated in this study and elsewhere.

It is also recommended that CSIR-BRRI, which is currently producing pozzolana on commercial scale, improves the quality of pozzolana by mechanically activating its product.

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APPENDICES

Appendix 1

Calculation of Anselm's specific surface for Mankessim pozzolana

Table A1-1: Calculation of the uniformity coefficient, n for the roll mill product of Mankessim pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnx ΔR	ln ² x ΔR
75	100	0	1.4	4.317488	18.6407	6.044483	26.09699
52.8	98.6	1.4	10.2	3.965753	15.7272	40.45068	160.4174
37.8	88.4	11.6	18.2	3.63178	13.18982	66.09839	240.0548
29.8	70.2	29.8	6.2	3.395179	11.52724	21.05011	71.4689
24.2	64	36	5.4	3.187592	10.16074	17.21299	54.86799
19.3	58.6	41.4	8	2.95803	8.749944	23.66424	69.99955
17.6	50.6	49.4	7.8	2.866762	8.218324	22.36074	64.10293
17.3	42.8	57.2	4.1	2.852439	8.136409	11.695	33.35928
11.6	38.7	61.3	7.2	2.451867	6.011651	17.65344	43.28389
5.2	31.5	68.5	6	1.644805	2.705384	9.86883	16.2323
3.6	25.5	74.5	25.5	1.283708	1.647906	32.73455	42.02159

Therefore, from Table A1-1, $\sum \ln x \Delta R = 2.688335$, $(\sum \ln x \Delta R)^2 = 7.227144$ and $\sum \ln^2 x \Delta R = 8.219057$. The uniformity coefficient, n can be calculated from

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

Thus $n = 1.287215$. From Anselm's formula the specific surface area, S can be calculated as:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg}; \quad S = 1071.7 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 6 \mu\text{m}; \quad \delta = 2.54 \text{ g/cm}^3.$$

Table A1-2: Calculation of the uniformity coefficient, n for the 36 h ball milling product of Mankessim pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnx ΔR	ln ² x ΔR
75	100	0	11.34	4.317488	18.6407	48.96032	211.3856
22.1	88.66	11.34	9.75	3.096934	9.591001	30.19511	93.51226
13	78.91	21.09	12.02	2.567254	6.590795	30.8584	79.22136
10.3	66.89	33.11	9.1	2.335052	5.452469	21.24898	49.61747
8.9	57.79	42.21	3.19	2.182675	4.764069	6.962733	15.19738
8.6	54.6	45.4	1.6	2.136531	4.564763	3.418449	7.30362
8.5	53	47	12.6	2.135349	4.559716	26.9054	57.45242
7.1	40.4	59.6	7.1	1.960095	3.841972	13.91667	27.278
5.9	33.3	66.7	7.6	1.773256	3.144437	13.47675	23.89772
4.1	25.7	74.3	3.9	1.415853	2.00464	5.521827	7.818097
3.4	21.8	78.2	12.2	1.23256	1.519205	15.03724	18.5343
1.5	9.6	90.4	9.6	0.405465	0.164402	3.892465	1.578259

From Table A1-2, $\sum \ln x \Delta R = 2.203943$, $(\sum \ln x \Delta R)^2 = 4.857366$ and $\sum \ln^2 x \Delta R = 5.927965$.

The uniformity coefficient, n can be calculated from

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

Thus $n = 1.23901$. From Anselm's formula, the specific surface area, S can be calculated as:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 1062.2 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 11 \mu\text{m}; \delta = 2.54 \text{ g/cm}^3.$$

Table A1-3: Calculation of the uniformity coefficient, n for the 24 h ball milling product of Mankessim pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnx ΔR	ln ² x ΔR
100	100	0	7.1	4.60517	21.20759	32.69671	150.5739
54.1	92.9	7.1	4.1	3.991019	15.92823	16.36318	65.30575
39.2	88.8	11.2	4.6	3.667911	13.45357	16.87239	61.88643
28.3	84.2	15.8	6.2	3.341801	11.16764	20.71917	69.23934
20.8	78	22	8.75	3.03351	9.202181	26.54321	80.51908
15.3	69.25	30.75	6.89	2.729812	7.451872	18.8084	51.3434
14.1	62.36	37.64	6.57	2.645465	6.998487	17.38071	45.98006
13.8	55.79	44.21	9.09	2.627563	6.904087	23.88455	62.75815
13	46.7	53.3	9.55	2.567254	6.590795	24.51728	62.94209
9.3	37.15	62.85	8.53	2.224624	4.94895	18.97604	42.21454
6.8	28.62	71.38	24.92	1.913977	3.663308	47.69631	91.28964
1.5	3.7	96.3	3.7	0.425268	0.180853	1.573491	0.669155

From Table A1-3, $\sum \ln x \Delta R = 2.660314$, $(\sum \ln x \Delta R)^2 = 7.077272$ and $\sum \ln^2 x \Delta R = 7.847216$.

The uniformity coefficient, n can be calculated from:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

Thus n = 1.461028.

From Anselm's formula, the specific surface area, S, is calculated as:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 583.7 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 17 \mu\text{m}; \delta = 2.54 \text{ g/cm}^3.$$

Table A1-4: Calculation of the uniformity coefficient, n for the 42 h ball milling product of Mankessim pozzolana

x μm	D %	R %	ΔR %	$\ln x$	$\ln^2 x$	$\ln x \Delta R$	$\ln^2 x \Delta R$
300	100	0	1.4	5.703782	32.53313	7.985295	45.54639
150	98.6	1.4	10.2	5.010635	25.10647	51.10848	256.086
75	88.4	11.6	18.2	4.317488	18.6407	78.57828	339.2608
44.6	70.2	29.8	6.2	3.797061	14.41767	23.54178	89.38957
38	64	36	5.4	3.638112	13.23586	19.64581	71.47365
32.6	58.6	41.4	8	3.484312	12.14043	27.8745	97.12346
23.9	50.6	49.4	7.8	3.173878	10.0735	24.75625	78.57333
17.4	42.8	57.2	4.1	2.85647	8.159422	11.71153	33.45363
14.2	38.7	61.3	7.2	2.650421	7.024732	19.08303	50.57807
9.5	31.5	68.5	6	2.251292	5.068315	13.50775	30.40989
6.8	25.5	74.5	5.9	1.916923	3.674592	11.30984	21.68009
4.9	19.6	80.4	6.4	1.589235	2.525669	10.17111	16.16428
3.4	13.2	86.8	12.6	1.223775	1.497626	15.41957	18.87009
1.5	0.6	99.4	0.6	0.405465	0.164402	0.243279	0.098641

From Table A1-4, $\sum \ln x \Delta R = 3.149365$, $(\sum \ln x \Delta R)^2 = 9.9185$ and $\sum \ln^2 x \Delta R = 11.48708$.

Hence the uniformity coefficient, n is:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

$$n = 1.023611.$$

From Anselm's formula, the specific surface area, S, is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 373.8 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 38 \mu\text{m}; \delta = 2.54 \text{ g/cm}^3.$$

Table A1-5: Calculation of the uniformity coefficient, n for the hammer mill product of Mankessim pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnx ΔR	ln ² x ΔR
600	100	0	0.2	6.39693	40.92071	1.279386	8.184142
420	99.8	0.2	0.4	6.040255	36.48468	2.416102	14.59387
300	99.4	0.6	0.8	5.703782	32.53313	4.563026	26.02651
176.2	98.6	1.4	5.8	5.17162	26.74565	29.99539	155.1248
110.2	92.8	7.2	16	4.701843	22.10733	75.22949	353.7173
99.8	76.8	23.2	4.5	4.603168	21.18916	20.71426	95.35121
92.6	72.3	27.7	3.8	4.528289	20.5054	17.2075	77.92053
82.6	68.5	31.5	12.3	4.413768	19.48134	54.28934	239.6205
72.9	56.2	43.8	7.8	4.288814	18.39393	33.45275	143.4726
70.3	48.4	51.6	9.8	4.252203	18.08123	41.67159	177.196
68.8	38.6	61.4	9.5	4.230477	17.89693	40.18953	170.0209
57.5	29.1	70.9	5.1	4.05248	16.4226	20.66765	83.75524
54.3	24	76	6	3.994892	15.95917	23.96935	95.755
48.6	18	82	8	3.8828	15.07614	31.0624	120.6091
42.4	10	90	9.67	3.745968	14.03228	36.22351	135.6921
18.2	0.33	99.67	0.33	2.901422	8.418247	0.957469	2.778022

From Table A1-5, $\sum \ln x \Delta R = 4.338887$, $(\sum \ln x \Delta R)^2 = 18.82594$ and $\sum \ln^2 x \Delta R = 18.99818$.

The uniformity coefficient, n is:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

n = 3.089078.

From Anselm's equation, the specific surface area, S, is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 58.7 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 80 \mu\text{m}; \delta = 2.54 \text{ g/cm}^3$$

Appendix 2

Calculation of Anselm's specific surface for Tanoso pozzolana

Table A2-1: Calculation of the uniformity coefficient, n for the Roll mill product of Tanoso pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnx ΔR	ln ² x ΔR
75	100	0	1.2	4.317488	18.6407	5.180986	22.36884
52.8	98.8	1.2	1.7	3.965753	15.7272	6.741781	26.73624
37.8	97.1	2.9	3.7	3.63178	13.18982	13.43759	48.80235
29.8	93.4	6.6	6	3.395179	11.52724	20.37108	69.16346
23.1	87.4	12.6	4.8	3.140698	9.863984	15.07535	47.34712
21.2	82.6	17.4	8	3.053057	9.321159	24.42446	74.56927
16.9	74.6	25.4	7.4	2.827314	7.993702	20.92212	59.1534
12.9	67.2	32.8	10.4	2.553344	6.519565	26.55478	67.80347
8.3	56.8	43.2	10.3	2.116256	4.478537	21.79743	46.12894
5.2	46.5	53.5	7.4	1.644805	2.705384	12.17156	20.01984
3.6	39.1	60.9	39.1	1.283708	1.647906	50.19297	64.43311

Thus from Table A2-1, $\sum \ln x \Delta R = 2.688358$, $(\sum \ln x \Delta R)^2 = 7.227271$ and $\sum \ln^2 x \Delta R = 8.219192$. The uniformity coefficient, n is calculated as:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

n = 1.28721.

Anselm's equation gives the specific surface as:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg}; S = 1124.0 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 9 \mu\text{m}; \delta = 2.82 \text{ g/cm}^3.$$

Table A2-2: Calculation of the uniformity coefficient, n for the 36 h ball milling product of Tanoso pozzolana

X μm	D %	R %	ΔR %	ln X	ln2x	lnX ΔR	ln2x ΔR
75	100	0	1.2	4.317488	18.6407	5.180986	22.36884
52.8	98.8	1.2	1.7	3.965753	15.7272	6.741781	26.73624
37.8	97.1	2.9	3.7	3.63178	13.18982	13.43759	48.80235
29.8	93.4	6.6	6	3.395179	11.52724	20.37108	69.16346
23.1	87.4	12.6	4.8	3.140698	9.863984	15.07535	47.34712
21.2	82.6	17.4	8	3.053057	9.321159	24.42446	74.56927
16.9	74.6	25.4	7.4	2.827314	7.993702	20.92212	59.1534
12.9	67.2	32.8	10.4	2.553344	6.519565	26.55478	67.80347
8.3	56.8	43.2	10.3	2.116256	4.478537	21.79743	46.12894
5.2	46.5	53.5	7.4	1.644805	2.705384	12.17156	20.01984
3.61	39.1	60.9	39.1	1.283708	1.647906	50.19297	64.43311

From Table A2-2, $\sum \ln x \Delta R = 2.168701$, $(\sum \ln x \Delta R)^2 = 4.703264$ and $\sum \ln^2 x \Delta R = 5.46526$.

The uniformity coefficient, n is:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

$$n = 1.468627$$

From Anselm's equation, the specific surface, S is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg}; S = 739.8 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 12 \mu\text{m}; \delta = 2.82 \text{ g/cm}^3.$$

Table A2-3: Calculation of the uniformity coefficient, n for the 24 h ball milling product of Tanoso pozzolana

x μm	D %	R %	ΔR %	$\ln x$	$\ln^2 x$	$\ln x \Delta R$	$\ln^2 x \Delta R$
107.7	98.3	1.7	6.8	4.678853	21.89166	31.8162	148.8633
55.4	91.5	8.5	7.5	4.014941	16.11975	30.11205	120.8981
40.6	84	16	9.23	3.702536	13.70877	34.17441	126.532
29.9	74.77	25.23	5.07	3.397858	11.54544	17.22714	58.53539
25.9	69.7	30.3	10.2	3.25347	10.58507	33.1854	107.9677
18.9	59.5	40.5	8.5	2.94022	8.644891	24.99187	73.48157
12.6	51	49	6.3	2.536866	6.435691	15.98226	40.54485
9.2	44.7	55.3	6.1	2.213754	4.900706	13.5039	29.89431
6.7	38.6	61.4	5.8	1.896119	3.595269	10.99749	20.85256
4.8	32.8	67.2	6.6	1.572774	2.473618	10.38031	16.32588
3.5	26.2	73.8	13.3	1.238374	1.533571	16.47038	20.39649
1.5	12.9	87.1	12.9	0.425268	0.180853	5.485954	2.332999

From Table A2-3, $\sum \ln x \Delta R = 2.443274$, $(\sum \ln x \Delta R)^2 = 5.969586$ and $\sum \ln^2 x \Delta R = 7.666252$.

The uniformity coefficient, n is calculated as:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

n = 0.984215 and from Anselm's formula, the specific surface area is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg}; S = 665.8 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 20 \mu\text{m}; \delta = 2.82 \text{ g/cm}^3$$

Table A2-4: Calculation of the uniformity coefficient, n for the hammer mill product of Tanoso pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnxΔR	ln ² xΔR
600	100	0	0.2	6.39693	40.92071	1.279386	8.184142
420	99.8	0.2	1.6	6.040255	36.48468	9.664408	58.37548
300	98.2	1.8	7.2	5.703782	32.53313	41.06723	234.2386
150	91	9	10.8	5.010635	25.10647	54.11486	271.1498
75	80.2	19.8	12.26	4.317488	18.6407	52.9324	228.535
50.5	67.94	32.06	4.34	3.921379	15.37721	17.01879	66.73711
45	63.6	36.4	8.79	3.807329	14.49575	33.46642	127.4177
33.1	54.81	45.19	8.91	3.499231	12.24462	31.17815	109.0996
24.1	45.9	54.1	6.84	3.18387	10.13703	21.77767	69.33728
17.8	39.06	60.94	5.76	2.878074	8.283311	16.57771	47.71187
13.2	33.3	66.7	5.4	2.580217	6.657519	13.93317	35.9506
9.6	27.9	72.1	6.1	2.261763	5.115572	13.79675	31.20499
6.8	21.8	78.2	5.65	1.921325	3.691489	10.85548	20.85691
5	16.15	83.85	6.15	1.599388	2.558041	9.836234	15.73195
3.5	10	90	9.67	1.249902	1.562254	12.08655	15.107
1.8	0.33	99.67	0.33	0.587787	0.345493	0.19397	0.114013

From Table A2-4, $\sum \ln x \Delta R = 3.397792$, $(\sum \ln x \Delta R)^2 = 11.54499$ and $\sum \ln^2 x \Delta R = 13.39752$.

The uniformity coefficient, n can be calculated as:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

$$n = 0.941901.$$

Hence from Anselm's formula, the specific surface area, S is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg}; S = 329.8 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 42 \text{ } \mu\text{m}; \delta = 2.82 \text{ g/cm}^3$$

Table A2-5: Calculation of the uniformity coefficient, n for the 42 h milling product of Tanoso pozzolana

x μm	D %	R %	ΔR %	$\ln x$	$\ln^2 x$	$\ln x \Delta R$	$\ln^2 x \Delta R$
300	100	0	1.4	5.703782	32.53313	7.985295	45.54639
150	98.6	1.4	10.2	5.010635	25.10647	51.10848	256.086
75	88.4	11.6	18.2	4.317488	18.6407	78.57828	339.2608
44.6	70.2	29.8	6.2	3.797061	14.41767	23.54178	89.38957
38.0	64	36	5.4	3.638112	13.23586	19.64581	71.47365
32.6	58.6	41.4	8	3.484312	12.14043	27.8745	97.12346
23.9	50.6	49.4	7.8	3.173878	10.0735	24.75625	78.57333
17.4	42.8	57.2	4.1	2.85647	8.159422	11.71153	33.45363
14.2	38.7	61.3	7.2	2.650421	7.024732	19.08303	50.57807
9.5	31.5	68.5	6	2.251292	5.068315	13.50775	30.40989
6.8	25.5	74.5	5.9	1.916923	3.674592	11.30984	21.68009
4.9	19.6	80.4	6.4	1.589235	2.525669	10.17111	16.16428
3.4	13.2	86.8	12.6	1.223775	1.497626	15.41957	18.87009
1.5	0.6	99.4	0.6	0.405465	0.164402	0.243279	0.098641

From Table A2-5, $\sum \ln x \Delta R = 3.149365$, $(\sum \ln x \Delta R)^2 = 9.9185$ and $\sum \ln^2 x \Delta R = 11.48708$.

Thus the uniformity coefficient, n is:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

$n = 1.023611$ and Anselm's formula is used to calculate the specific surface area as:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 364.1 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 35 \mu\text{m}; \delta = 2.82 \text{ g/cm}^3.$$

Appendix 3

Calculation of Anselm's specific surface for Mankranso pozzolana

Table A3-1: Calculation of the uniformity coefficient, n for the 36 h milling product of Mankranso pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnxΔR	ln ² xΔR
75	100	0	4	4.317488	18.6407	17.26995	74.56281
44.8	96	4	4	3.801538	14.45169	15.20615	57.80677
34	92	8	4.1	3.525183	12.42692	14.45325	50.95036
28	87.6	12.4	9.04	3.332205	11.10359	30.12313	100.3764
18.6	76.86	23.14	9.26	2.922086	8.538585	27.05851	79.0673
15	69.6	30.4	6.92	2.70805	7.333536	18.73971	50.74807
12	62.68	37.32	6.6	2.484907	6.174761	16.40038	40.75342
9.5	56.08	43.92	10.48	2.254445	5.082521	23.62658	53.26482
6.7	45.6	54.4	3.4	1.899118	3.606649	6.457001	12.26261
5.5	42.2	57.8	11	1.704748	2.906166	18.75223	31.96783
3	31.2	68.8	11.7	1.098612	1.206949	12.85376	14.1213
1.3	19.5	80.5	19.5	0.270027	0.072915	5.265529	1.421836

From the Table A3-1:

$\Sigma \ln x \Delta R = 2.062062$, $(\Sigma \ln x \Delta R)^2 = 4.252099$ and $\Sigma \ln^2 x \Delta R = 5.673036$. Thus the uniformity coefficient, n is:

$$\frac{1}{n} = 0.781 \sqrt{\Sigma \ln^2 x \Delta R - (\Sigma \ln x \Delta R)^2}$$

n = 1.073798.

From Anselm's formula, the specific surface area, S is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 943.2 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 12 \text{ μm}; \delta = 2.56 \text{ g/cm}^3.$$

Table A3-2: Calculation of the uniformity coefficient, n for the roll mill product of Mankranso pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnx ΔR	ln ² x ΔR
75	100	0	1.4	4.317488	18.6407	6.044483	26.09699
52.8	98.6	1.4	8	3.965753	15.7272	31.72603	125.8176
37.8	90.6	9.4	17.8	3.63178	13.18982	64.64568	234.7789
29.8	72.8	27.2	6.9	3.395179	11.52724	23.42674	79.53797
23.1	65.9	34.1	7.3	3.140698	9.863984	22.9271	72.00708
21.2	58.6	41.4	8	3.053057	9.321159	24.42446	74.56927
16.9	50.6	49.4	5.9	2.827314	7.993702	16.68115	47.16284
12.9	44.7	55.3	6	2.553344	6.519565	15.32006	39.11739
8.3	38.7	61.3	5.4	2.116256	4.478537	11.42778	24.1841
5.2	33.3	66.7	6.8	1.644805	2.705384	11.18467	18.39661
3.6	26.5	73.5	26.5	1.283708	1.647906	34.01826	43.6695

From Table A3-2:

$\Sigma \ln x \Delta R = 2.062062$, $(\Sigma \ln x \Delta R)^2 = 4.252099$ and $\Sigma \ln^2 x \Delta R = 5.673036$. The uniformity coefficient, n is:

$$\frac{1}{n} = 0.781 \sqrt{\Sigma \ln^2 x \Delta R - (\Sigma \ln x \Delta R)^2} ; \quad n = 1.073798.$$

The specific surface area, S from Anselm's formula is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 1123.1 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 10 \mu\text{m}; \quad \delta = 2.56 \text{ g/cm}^3.$$

Table A3-3: Calculation of the uniformity coefficient, n for the 24 h mill product of Mankranso pozzolana

x μm	D %	R %	ΔR %	ln x	$\ln^2 x$	lnx ΔR	$\ln^2 x \Delta R$
300	100	0	0.4	5.703782	32.53313	2.281513	13.01325
150	99.6	0.4	4.4	5.010635	25.10647	22.0468	110.4685
75	95.2	4.8	5.2	4.317488	18.6407	22.45094	96.93166
53.5	90	10	7.7	3.979682	15.83787	30.64355	121.9516
39	82.3	17.7	8.2	3.663562	13.42168	30.04121	110.0578
28.6	74.1	25.9	9.2	3.353407	11.24534	30.85134	103.4571
21	64.9	35.1	4.6	3.044522	9.269117	14.0048	42.63794
17.2	60.3	39.7	4.6	2.843746	8.086891	13.08123	37.1997
13.9	55.7	44.3	9.5	2.634045	6.938192	25.02343	65.91282
9.4	46.2	53.8	7.7	2.24071	5.02078	17.25346	38.66001
6.2	38.5	61.5	5.9	1.824549	3.32898	10.76484	19.64098
4.5	32.6	67.4	5.6	1.504077	2.262249	8.422833	12.66859
3.2	27	73	10.7	1.163151	1.35292	12.44571	14.47624
1.4	16.3	83.7	16.3	0.336472	0.113214	5.484497	1.845381

Therefore:

$$\sum \ln x \Delta R = 2.447962, (\sum \ln x \Delta R)^2 = 5.992516 \text{ and } \sum \ln^2 x \Delta R = 7.889215.$$

Hence n, the uniformity coefficient is:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

$$n = 0.930869.$$

From Anselm's formula, the specific surface area, S is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 813.5 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 19 \mu\text{m}; \delta = 2.56 \text{ g/cm}.$$

Table A3-4: Calculation of the uniformity coefficient, n for the 42 h mill product of Mankranso pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnx ΔR	ln ² x ΔR
300	100	0	0.2	5.703782	32.53313	1.140756	6.506627
232.3	99.8	0.2	1.8	5.4479	29.67962	9.806221	53.42332
195	98	2	9.4	5.272948	27.80398	49.56571	261.3574
81.9	88.6	11.4	13	4.404888	19.40304	57.26355	252.2395
57	75.6	24.4	8.8	4.043402	16.3491	35.58194	143.8721
51.3	66.8	33.2	11.9	3.937496	15.50387	46.8562	184.4961
37.4	54.9	45.1	9.8	3.621938	13.11844	35.49499	128.5607
23.3	45.1	54.9	4.5	3.145445	9.893821	14.1545	44.5222
18.5	40.6	59.4	13	2.91723	8.510231	37.92399	110.633
9.5	27.6	72.4	6.8	2.251292	5.068315	15.30878	34.46454
6.8	20.8	79.2	4.8	1.916923	3.674592	9.201229	17.63804
4.9	16	84	5.7	1.589235	2.525669	9.058641	14.39631
3.4	10.3	89.7	10	1.223775	1.497626	12.23775	14.97626
1.5	0.3	99.7	0.3	0.405465	0.164402	0.12164	0.049321

From Table A3-4:

$\Sigma \ln x \Delta R = 3.337159$, $(\Sigma \ln x \Delta R)^2 = 11.13663$ and $\Sigma \ln^2 x \Delta R = 12.67135$. The uniformity coefficient is therefore:

$$\frac{1}{n} = 0.781 \sqrt{\Sigma \ln^2 x \Delta R - (\Sigma \ln x \Delta R)^2}$$

$n = 1.034839$ and the specific surface area from Anselm's formula is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 370.8 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 38 \mu\text{m}; \delta = 2.56 \text{ g/cm}^3.$$

Table A3-5: Calculation of the uniformity coefficient, n for the hammer mill product of Mankranso pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnx ΔR	ln ² x ΔR
300	100	0	0.2	5.703782	32.53313	1.140756	6.506627
232.3	99.8	0.2	1.8	5.4479	29.67962	9.806221	53.42332
195	98	2	9.4	5.272948	27.80398	49.56571	261.3574
81.9	88.6	11.4	13	4.404888	19.40304	57.26355	252.2395
57.02	75.6	24.4	8.8	4.043402	16.3491	35.58194	143.8721
51.3	66.8	33.2	11.9	3.937496	15.50387	46.8562	184.4961
37.4	54.9	45.1	9.8	3.621938	13.11844	35.49499	128.5607
23.2	45.1	54.9	4.5	3.145445	9.893821	14.1545	44.5222
18.5	40.6	59.4	13	2.91723	8.510231	37.92399	110.633
9.5	27.6	72.4	6.8	2.251292	5.068315	15.30878	34.46454
6.8	20.8	79.2	4.8	1.916923	3.674592	9.201229	17.63804
4.9	16	84	5.7	1.589235	2.525669	9.058641	14.39631
3.4	10.3	89.7	10	1.223775	1.497626	12.23775	14.97626
1.5	0.3	99.7	0.3	0.405465	0.164402	0.12164	0.049321

Thus:

$\sum \ln x \Delta R = 3.337159$, $(\sum \ln x \Delta R)^2 = 11.13663$ and $\sum \ln^2 x \Delta R = 12.67135$. The uniformity coefficient of the distribution, n is:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

n = 1.034839.

From Anselm's formula, the specific surface area, S is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 348.9 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 18.0 \mu\text{m}; \delta = 2.56 \text{ g/cm}^3.$$

Appendix 4

Calculation of Anselm's specific surface for Mfensi pozzolana

Table A4-1: Calculation of the uniformity coefficient, n for the 36 h ball milling product of Mfensi pozzolana

x μm	D %	R %	ΔR %	$\ln x$	$\ln^2 x$	$\ln x \Delta R$	$\ln^2 x \Delta R$
75	100	0	0.1	4.317488	18.6407	0.431749	1.86407
54	99.9	0.1	3	3.988984	15.91199	11.96695	47.73598
39	96.9	3.1	7.9	3.663562	13.42168	28.94214	106.0313
28	89	11	6	3.332205	11.10359	19.99323	66.62152
21	83	17	10.5	3.044522	9.269117	31.96749	97.32573
15	72.5	27.5	6	2.70805	7.333536	16.2483	44.00122
12	66.5	33.5	8.42	2.484907	6.174761	20.92291	51.99149
9	58.08	41.92	9.84	2.197225	4.827796	21.62069	47.50551
6	48.24	51.76	5.64	1.791759	3.210402	10.10552	18.10667
5	42.6	57.4	10.5	1.609438	2.59029	16.8991	27.19805
3	32.1	67.9	11.5	1.098612	1.206949	12.63404	13.87991
1.3	20.6	79.4	20.6	0.271705	0.073824	5.597124	1.520767

From Table A4-1:

$\sum \ln x \Delta R = 3.337159$, $(\sum \ln x \Delta R)^2 = 11.13663$ and $\sum \ln^2 x \Delta R = 12.67135$. For this distribution, the uniformity coefficient, n is calculated as:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

$n = 1.034839$ and the specific surface area, S, from Anselm's formula is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 1079.2 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 12 \mu\text{m}; \delta = 2.56 \text{ g/cm}^3.$$

Table A4-2: Calculation of the uniformity coefficient, n for the roll mill product of Mfensi pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnxΔR	ln ² xΔR
75	100	0	0.2	4.317488	18.6407	0.863498	3.728141
52.76	99.8	0.2	2.9	3.965753	15.7272	11.50068	45.60888
37.78	96.9	3.1	3.5	3.63178	13.18982	12.71123	46.16439
29.82	93.4	6.6	4.9	3.395179	11.52724	16.63638	56.48349
24.23	88.5	11.5	4.2	3.187592	10.16074	13.38788	42.67511
19.26	84.3	15.7	5.5	2.95803	8.749944	16.26917	48.12469
14.57	78.8	21.2	2.8	2.678965	7.176851	7.501101	20.09518
13.18	76	24	13.5	2.578701	6.649696	34.81246	89.7709
8.3	62.5	37.5	10.5	2.116256	4.478537	22.22068	47.02464
5.18	52	48	7.6	1.644805	2.705384	12.50052	20.56092
3.61	44.4	55.6	44.4	1.283708	1.647906	56.99663	73.16701

Hence:

$\Sigma \ln x \Delta R = 2.054002$, $(\Sigma \ln x \Delta R)^2 = 4.218925$ and $\Sigma \ln^2 x \Delta R = 4.934033$. The uniformity coefficient, n for the distribution is:

$$\frac{1}{n} = 0.781 \sqrt{\Sigma \ln^2 x \Delta R - (\Sigma \ln x \Delta R)^2}$$

n = 1.51601.

From Anselm's formula, the specific surface area, S is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 1316.9 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 7.2 \text{ μm}; \delta = 2.56 \text{ g/cm}^3.$$

Table A4-3: Calculation of the uniformity coefficient, n for the 24 h ball milling product of Mfensi pozzolana

x μm	D %	R %	ΔR %	$\ln x$	$\ln^2 x$	$\ln x \Delta R$	$\ln^2 x \Delta R$
75	100	0	1.1	4.317488	18.6407	4.749237	20.50477
53.5	98.9	1.1	3.3	3.979682	15.83787	13.13295	52.26496
39	95.6	4.4	9.1	3.663562	13.42168	33.33841	122.1373
28.6	86.5	13.5	7.9	3.353407	11.24534	26.49191	88.83816
21	78.6	21.4	10.1	3.044522	9.269117	30.74968	93.61808
15.3	68.5	31.5	8.3	2.727853	7.441181	22.64118	61.7618
11.5	60.2	39.8	7.6	2.442347	5.965059	18.56184	45.33445
8.5	52.6	47.4	10	2.140066	4.579883	21.40066	45.79883
6.2	42.6	57.4	6.5	1.824549	3.32898	11.85957	21.63837
4.5	36.1	63.9	8.1	1.504077	2.262249	12.18303	18.32422
3.2	28	72	8	1.163151	1.35292	9.305206	10.82336
1.4	20	80	20	0.336472	0.113214	6.729445	2.264271

Therefore:

$\sum \ln x \Delta R = 2.111431$, $(\sum \ln x \Delta R)^2 = 4.458141$ and $\sum \ln^2 x \Delta R = 5.833086$. The uniformity coefficient of the distribution is:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

$n = 1.093315$.

Anselm's formula gives the specific surface area as:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 879.2 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 15 \mu\text{m}; \delta = 2.56 \text{ g/cm}^3.$$

Table A4-4: Calculation of the uniformity coefficient, n for the hammer mill product of Mfensi pozzolana

x μm	D %	R %	ΔR %	ln x	ln ² x	lnxΔR	ln ² xΔR
300	100	0	0.2	5.703782	32.53313	1.140756	6.506627
150	99.8	0.2	11.4	5.010635	25.10647	57.12124	286.2137
75	88.4	11.6	12.3	4.317488	18.6407	53.1051	229.2807
60.8	76.1	23.9	13.3	4.10759	16.87229	54.63094	224.4015
44.5	62.8	37.2	6.1	3.795489	14.40574	23.15248	87.875
38.2	56.7	43.3	10.5	3.642836	13.27025	38.24977	139.3376
23.9	46.2	53.8	5.8	3.173878	10.0735	18.4085	58.42633
17.4	40.4	59.6	4.4	2.85647	8.159422	12.56847	35.90146
13.77	36	64	6.6	2.622492	6.877466	17.30845	45.39128
9.5	29.4	70.6	6.5	2.251292	5.068315	14.6334	32.94405
6.8	22.9	77.1	4.9	1.916923	3.674592	9.392921	18.0055
4.9	18	82	4.8	1.589235	2.525669	7.628329	12.12321
3.4	13.2	86.8	12.6	1.223775	1.497626	15.41957	18.87009
1.5	0.6	99.4	0.6	0.405465	0.164402	0.243279	0.098641

From Table A4-4:

$\Sigma \ln x \Delta R = 2.111431$, $(\Sigma \ln x \Delta R)^2 = 4.458141$ and $\Sigma \ln^2 x \Delta R = 5.833086$. The uniformity coefficient, n is:

$$\frac{1}{n} = 0.781 \sqrt{\Sigma \ln^2 x \Delta R - (\Sigma \ln x \Delta R)^2}$$

n = 1.093315.

Anselm's formula is used to calculate the specific surface area as:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 293.1 \text{ m}^2/\text{kg}$$

for

$$\bar{x} = 45.0 \text{ μm}; \delta = 2.56 \text{ g/cm}^3.$$

Table A4-5: Calculation of the uniformity coefficient, n for the 42h ball milling product of Mfensi pozzolana

	D %	R %	ΔR %	ln x	ln ² x	lnxΔR	ln ² xΔR
300	100	0	1.4	5.703782	32.53313	7.985295	45.54639
150	98.6	1.4	8	5.010635	25.10647	40.08508	200.8517
75	90.6	9.4	17.8	4.317488	18.6407	76.85129	331.8045
44.57	72.8	27.2	6.9	3.797061	14.41767	26.19972	99.48194
38.02	65.9	34.1	7.3	3.638112	13.23586	26.55822	96.62179
32.6	58.6	41.4	8	3.484312	12.14043	27.8745	97.12346
23.9	50.6	49.4	5.9	3.173878	10.0735	18.72588	59.43368
17.4	44.7	55.3	6	2.85647	8.159422	17.13882	48.95653
13	38.7	61.3	5.4	2.564949	6.578965	13.85073	35.52641
9.5	33.3	66.7	6.8	2.251292	5.068315	15.30878	34.46454
6.8	26.5	73.5	6.8	1.916923	3.674592	13.03507	24.98723
4.9	19.7	80.3	6.5	1.589235	2.525669	10.33003	16.41685
3.4	13.2	86.8	12.6	1.223775	1.497626	15.41957	18.87009
1.5	0.6	99.4	0.6	0.405465	0.164402	0.243279	0.098641

For this distribution,

$$\sum \ln x \Delta R = 2.111431, (\sum \ln x \Delta R)^2 = 4.458141 \text{ and } \sum \ln^2 x \Delta R = 5.833086.$$

The uniformity coefficient, n is:

$$\frac{1}{n} = 0.781 \sqrt{\sum \ln^2 x \Delta R - (\sum \ln x \Delta R)^2}$$

$$n = 1.093315.$$

From Anselm's formula, S, the specific surface area is:

$$S = \frac{36800}{xn\delta}, \text{ m}^2/\text{kg} = 363.7 \text{ m}^2/\text{kg}$$

where

$$\bar{x} = 38 \mu\text{m}; \delta = 2.56 \text{ g/cm}^3.$$