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Application of Mechanical Energy to Activate Pozzolanas

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ABSTRACT

Mechanical energy has been used to improve the reactivity of burnt clay pozzolana from two clay deposits in Ghana, Mankranso and Mankessim, by subjecting the pozzolanas to various types and degrees of milling – hammer milling, ball milling and roll milling. The particle sizes and specific surface areas obtained by these types/degrees of milling were analyzed and their effect on the strength development of Portland pozzolana cement mortar cubes prepared from the pozzolana samples evaluated. Both pozzolana samples were chemically suitable with total $SiO_2+Al_2O_3+Fe_2O_3$ content ≥ 70% as stipulated by the ASTM C 618 standard. Roll milling gave the finest pozzolana powders with characteristic particle sizes, $\bar{x}$ of 6 µm and 10 µm respectively for Mankessim and Mankranso pozzolanas. The respective Blaine indices obtained by roll milling were 1311 m^2/kg and 1395 m^2/kg. Both pozzolana samples showed increase in fineness with increasing duration of continuous ball milling without classification until agglomeration, evidenced by a significant increase in the characteristic particle size, started after 42 h. Compressive strengths for the Portland pozzolana cement mortar cubes prepared from the two pozzolana samples showed that both pozzolanas could be used to replace up to 40% ordinary Portland cement whilst satisfying the ASTM C 595 minimum requirement of 24.1 MPa. Mankessim pozzolana cement mortar cubes gained a 28-day compressive strength of 30.3 MPa after roll milling and 25.6 MPa after 36 h of ball milling whereas Mankranso pozzolana cement mortar gained a 28-day compressive strength of 29.6 MPa after roll milling and 27.7 MPa after 36 h of ball milling—both at 40% ordinary Portland cement replacement.. The 90-day compressive strengths showed that at 50% cement replacement, mortar cubes prepared from Mankranso and Mankessim pozzolanas gained enough strength to satisfy the ASTM C 595 and the EN 197-1 standard requirement. Hammer milling proved to be the least efficient means of activation as compressive strength values obtained at 28 days were below the ASTM C 595 minimum of 24.1 MPa at cement replacement levels above 30%.The activated pozzolanas were found suitable to replace up to 40% of OPC for most ordinary housing construction purposes not requiring high early strength class of cements.

Keywords: Activated pozzolana, ball milling, roll milling, hammer milling, compressive strength and setting time.

I. INTRODUCTION

The over dependence on imported building inputs – especially Portland cement and its derivatives has contributed largely to the high housing construction costs and the high housing deficit in Ghana. Statistics released by the Ministry of Works and Housing in Ghana in the year 2010 showed that about 12.5% of the total Ghanaian population were living in substandard housing structures due to an increase in the population without a corresponding increase in housing provision in Ghana. The statistics further reveals that the annual demand for housing stands between 300,000 - 500,000 units. With the current supply rate of between 25,000 - 40,000 units per annum, there is a current housing deficit of over 1.5 million units (Ministry of Works and Housing, 2007). Attendant environmental and health problems such as the springing up of slums, decrease in productivity, increase in communicable diseases and crime have all compounded the problem of housing.

Pozzolanas have been used with Portland cement to impart several desirable properties to such pozzolana-Portland cement mixes (Lea, 1970). It has been shown that the use of pozzolana to replace 30% of Portland cement results in 25% savings in housing construction costs (Atiemo, 2005). However, 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, 1970). 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 has thus been proposed as a remedy to the slow strength development in Portland pozzolana cements.

II. LITERATURE REVIEW

1.1 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; Palomo *et al.*, 1999; Gaffet *et al.*, 1998; Beke, 1981). High energy milling (mechanical activation) in particular has been widely used to raise the early strength of various pozzolanas.At specific surface areas above 350 m^2/kg, mechanically activated pozzolanas have been produced which when used to replace up to 50%, gave 7-day compressive strengths comparable to that of plain cement (Puri and Srinivsan 1964; Glinicki, 2002; Naceri and Benia, 2006; Uzal and Turanli 2003). 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).

2.2 Theoretical considerations on particle size distribution

Two values are used in practice to characterize particle size — 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 characterize the particle-size distribution, three formulae have customarily been adopted - the Gaudin-Schumann, the logarithmic-normal function and the Rosin-Rammler-Bennet equation, given as:

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

where

x = particle size, µm

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

$\bar{x}$ = characteristic particle size, µm

n = the uniformity coefficient of the distribution

The Rosin-Rammler-Bennet equation gives the best approach to the particle-size distribution by mass of ground products. It also allows for an estimation of the specific surface area of the powder by the Anselm equation:

$$S=\frac{368000}{\bar{x}n\rho}$$ (Beke, 1981)

where

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

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

ρ = the density, g/cm^3 and

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

The uniformity coefficient, n, can be calculated as:

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

(Beke, 1981).

The Anselm specific surface area could then be used to verify the more common but rather relative specific areas obtained by practical methods such as the Blaine air permeability method.

III. Materials and Method

Calcined clay pozzolana, produced from two clay deposits located at Mankranso and Mankessim were used for the research.Ordinary Portland cement (OPC) of class 42.5 N, meeting EN 197-1 standards, manufactured by Ghana Cement Company Limited (GHACEM)—a subsidiary of Heidelberg Cement Group was used for the research. Pit sand commonly used for construction was used as fine aggregate for making the mortar cubes. Figure 1 shows the block diagram for the production of the pozzolanas.

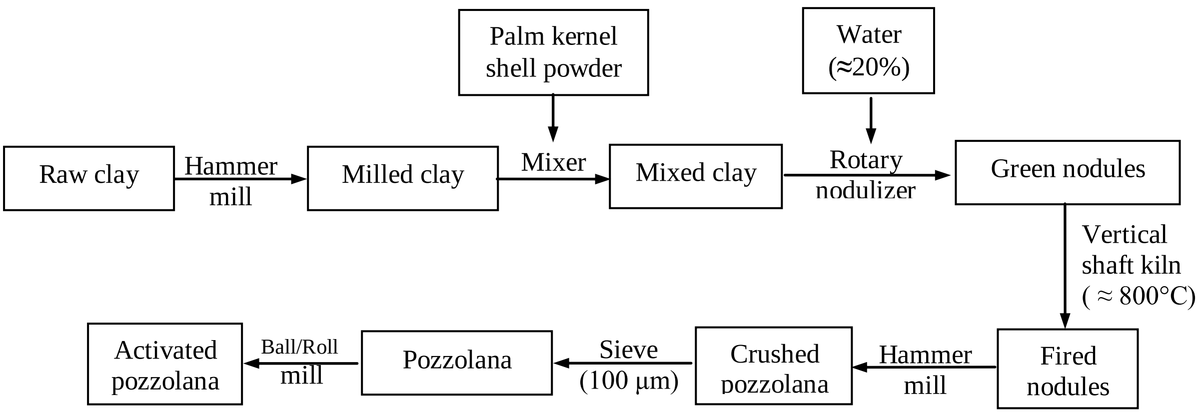


Figure1. Block diagram of activated pozzolana production

3.1 Milling of Pozzolanas

The fired nodules were first crushed with a hammer mill into an average particle size of 80% passing a 150 µm sieve and divided into three portions. One portion was reserved as the hammer mill product. The second 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 pozzolana was analyzed every 2 h until agglomeration was observed. The third portion of crushed pozzolana was milled in a Raymond type ring-roll mill and the particle size distribution of the product also determined.

3.2 Chemical and Physical Evaluation

The milled pozzolana samples were chemically analyzed using a SpectroXlab 2000 X-Ray Fluorescence spectrophotometer. Particle-size distribution and bulk density determinations were done using methods of BS 1377:90. Blaine specific surface area determinations were also performed according to the method of ASTM C 204 whilst the compressive strengths of the blended cements containing 30, 35, 40 and 50% of the pozzolanas were performed according to EN 196-1:2000.

IV. RESULTS AND DISCUSSIONS

4.1 Chemical Composition

Table 1: Chemical composition of pozzolana samples and OPC

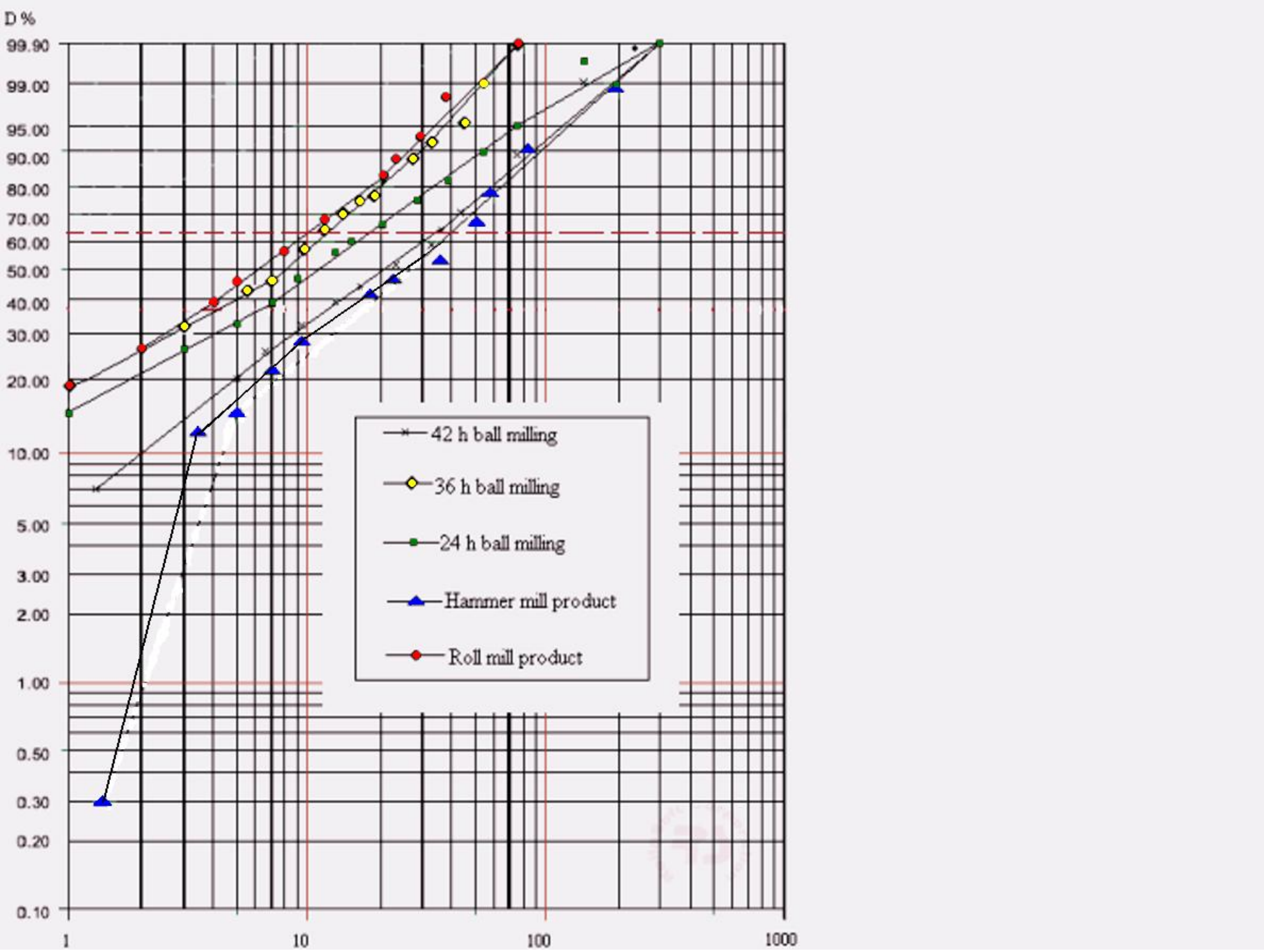
Constituent,%	Mankranso Pozzolana	Mankessim Pozzolana	OPC
SiO_2	62.03	65.85	19.70
Al_2O_3	16.66	18.34	5.00
Fe_2O_3	12.98	9.48	3.16
CaO	0.21	0.72	63.03
MgO	1.75	0.13	1.75
SO_3	0.16	0.04	2.80
LOI	2.97	3.82	2.58
Others*	3.24	1.62	1.98
Mineralogy			
C_3S			61
C_2S			11
C_3A			8
C_4AF			10

Table 1 shows the chemical composition of the pozzolana samples and OPC. The SiO_2 content of all the pozzolanas exceeded the minimum limit of 25.0% as prescribed by EN197-1:2000. The sum of SiO_2, Al_2O_3 and Fe_2O_3 also exceeded the minimum of 70% prescribed by ASTM C 618 for 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.

4.2 Particle size characterization

Figures 3 and 4 show the Rosin-Rammler-Bennet distribution curves for the pozzolana samples after hammer milling, 24, 36 and 42 hours of ball milling and roll milling. The Blaine index, Anselm specific surface area, characteristic particle size, uniformity coefficient and bulk density are shown in Table 2 for Mankranso (A) and Mankessim (B) samples.

Mankranso pozzolana attained a Blaine index of 1025 m^2/kg after 36 h ball milling. Agglomeration in the ball mill reduced the specific surface area and increased the characteristic particle size from 12 µm to 38 µm after 42 h of ball milling. Roll milling was the most efficient, producing a characteristic particle size of 10 µm and a specific surface area determined by the Blaine method of 1395 m^2/kg (Anselm fineness = 1123 m^2/kg). Hammer milling produced a specific surface area of 349 m^2/kg as determined by the Blaine method. The calculated Anselm fineness was also 349 m^2/kg.



Particle size, µm

Figure 2: Rosin-Rammler distribution curves for Mankranso pozzolana

The finest particle size for ball milling Mankessim pozzolana was obtained after 36 h miling, 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 (Anselm fineness = 374 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 (Anselm fineness = 1072 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.

As expected, there were discrepancies between the calculated Anselm's fineness and the Blaine fineness due to the scatter of the particle size distribution and the relative nature of the Blaine fineness (calibrated relative to Portland cement).

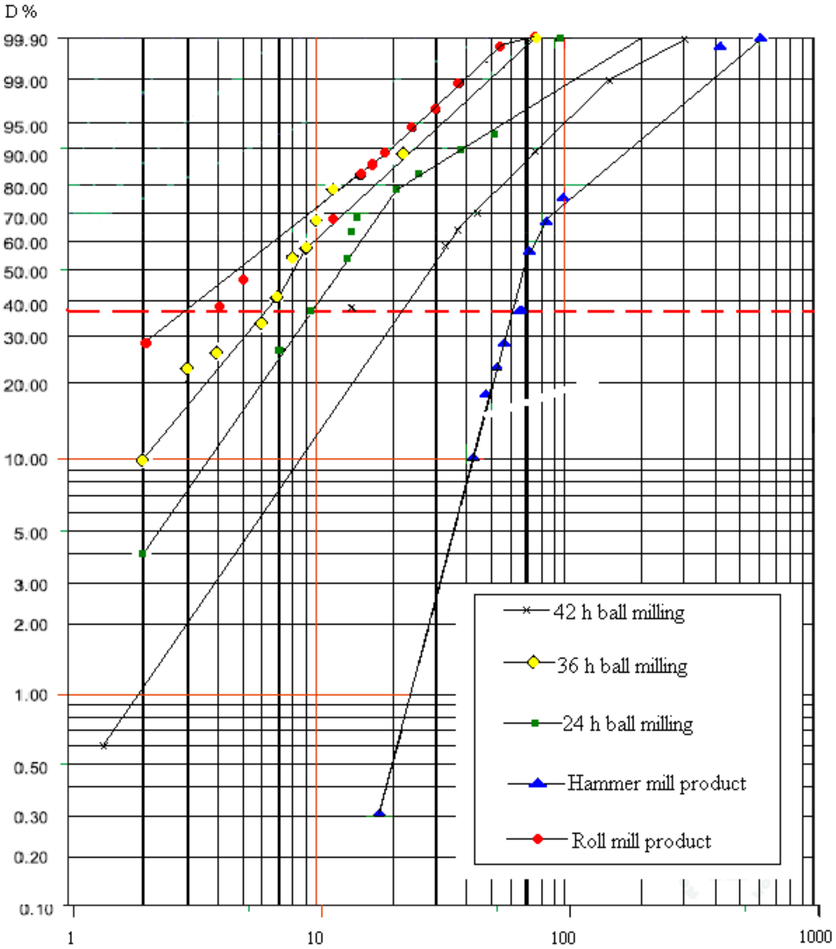


Figure 3: Rosin-Rammler-Bennet distribution curves for Mankessim pozzolana

Table 2: Particle size distribution, bulk density, specific surface area, uniformity coefficient and characteristic particle size for Mankranso (A) and Mankessim (B) Pozzolanas

Property	Hammer Milling		Roll Milling		Ball Milling 24 h		Ball Milling 36 h		Ball Milling 42 h	
	A	B	A	B	A	B	A	B	A	B
Bulk Density, kg/m^3	1052	1043	425	384	953	864	685	528	984	920
Blaine Index, m^2/kg	394	96	1395	1311	892	798	1025	1128	743	696
Anselm's Fineness, m^2/kg	349	59	1123	1072	814	584	943	1062	371	374
Characteristic particle size ($\bar{x}$), µm	18	80	10	6	19	17	12	11	38	38
Uniformity Coefficient (n)	1.0	3.1	1.1	1.3	0.9	1.5	1.1	1.2	1.0	1.0

4.3 Compressive Strength

Table 3 shows the compressive strengths in MPa at various ages of curing and for 30% to 50% pozzolana replacement of the cement for Mankranso pozzolana.

Figures 4 to 7 shows the early age and ultimate compressive strengths of Mankessim pozzolana cement mortars.

Table 3: 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

Generally, the compressive strength of Mankranso pozzolana cement mortar cubes increased faster after 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 levels of replacement. The highest compressive strengths were obtained for the roll milled pozzolana, followed closely by the 36 h ball milled pozzolana. The least strengths were obtained with hammer milling. For Mankranso pozzolana, with hammer milling as a basis at 28 days, roll milling resulted in 23-29% strength increase at 30 and 35% cement replacement, 49% strength increment at 40% cement replacement and 37% increment in strength at 50% cement replacement. The 90-day compressive strengths were 40-50% higher for roll milling than for hammer milling. As the pozzolana content increased, the compressive strength of Mankranso pozzolana cement mortar cubes decreased. Except for hammer milling, all Mankranso pozzolana cement mortar cubes 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.

The 28-day compressive strength of all Mankranso pozzolana cement samples satisfied the European standard EN 197-1 up to 30% cement replacement, except for the hammer milled 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.

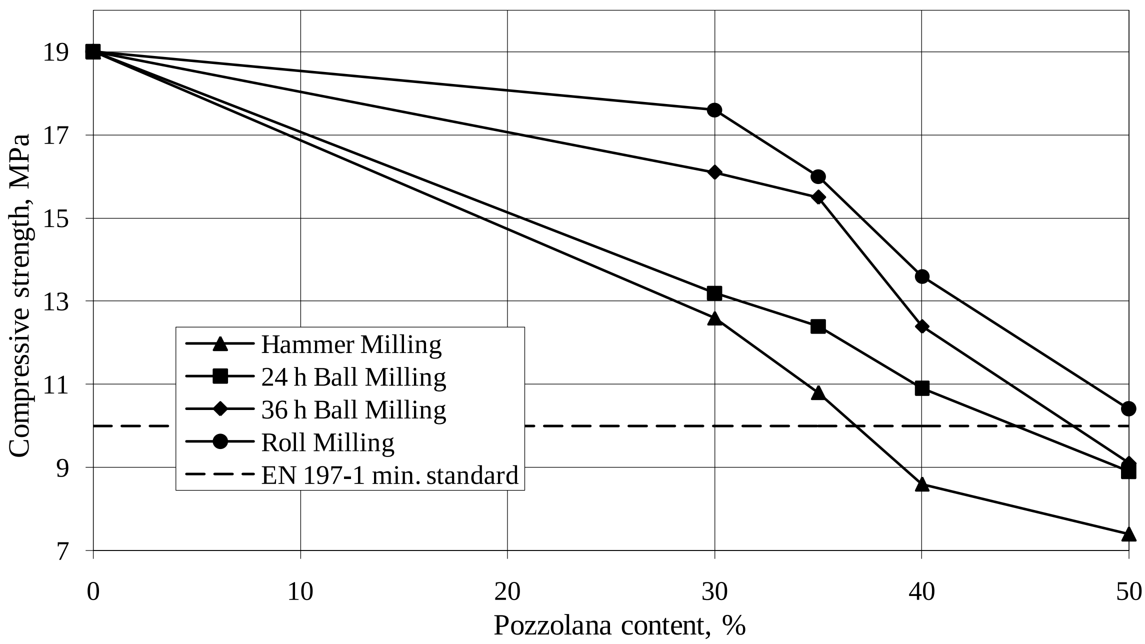


Figure 4: 2 - day compressive strength of Mankessim pozzolana cement

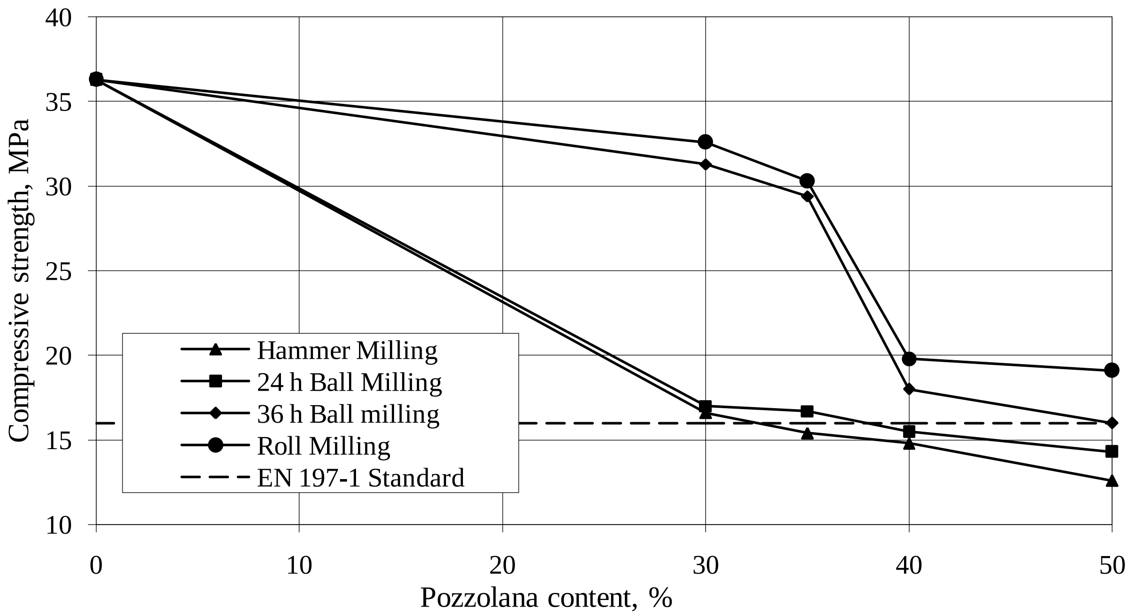


Figure 5: 7 - day compressive strength of Mankessim pozzolana cement

At 2 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 5, Mankessim 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 Mankessim pozzolana cement mortar cubes 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.

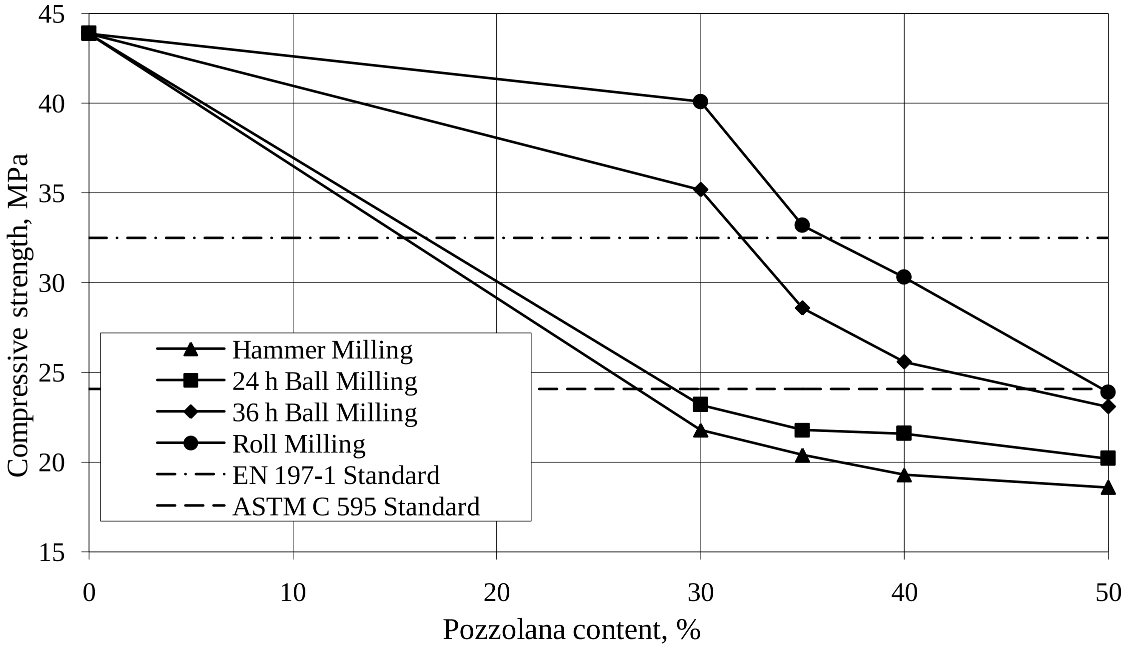


Figure 6: 28-day compressive strength of Mankessim pozzolana

The 36 h ball milled and roll milled Mankessim pozzolana cement mortar cubes attained strengths satisfying the EN 197-1 minimum standard of 32.5 MPa at 28 days for 30% cement replacement. Beyond 30% replacement of OPC, the 28-day 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 at all replacement levels, both the 36 h ball milled and roll milled Mankessim pozzolana cement mortar cubes attained 28-day 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 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.

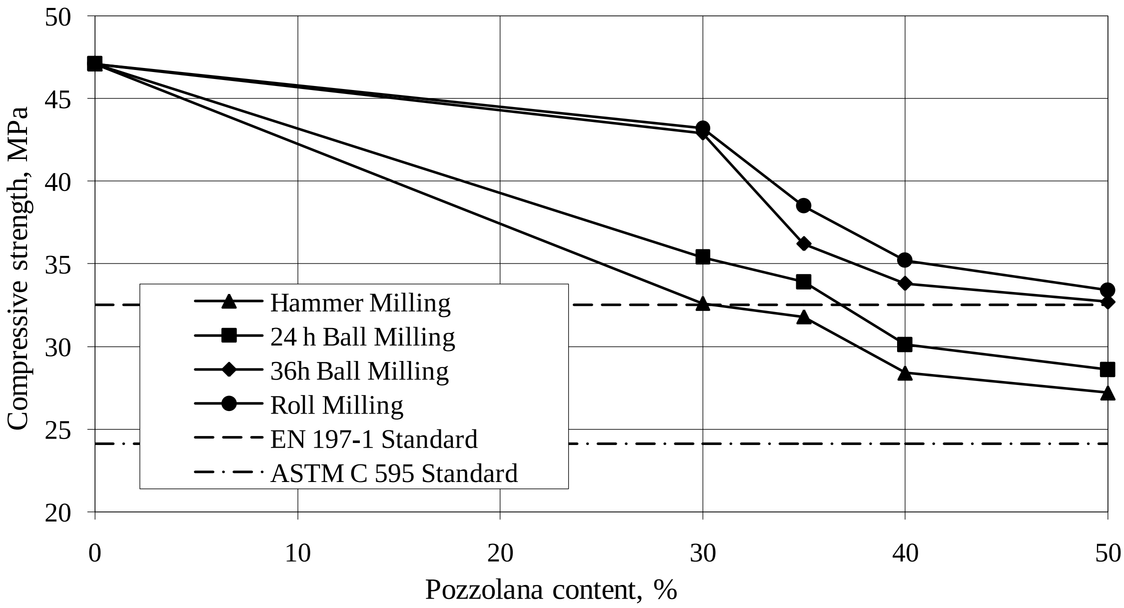


Figure 7: 90-day compressive strength of Mankessim pozzolana

After 90 days, all 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.

V. CONCLUSIONS

The pozzolana samples studied were activated by roll milling and after 36 h of ball milling beyond which agglomeration prevented activation. Roll milling with classification offered the most effective means of activation of the pozzolanas. Particle sizes attained were typically in the micrometer range with Mankessim pozzolana having a characteristic particle size of 6 µm after roll milling.Hammer milling without classification was inappropriate for mechanical activation of the burnt clays pozzolanas studied, as specific surface areas attained with the pozzolana samples were too low to effect any significant changes in the physical properties of the pozzolana cement samples. Roll milling gave the highest 28-day compressive strengths of all the types of milling studied with Mankessim pozzolana gaining a compressive strength of 30.3 MPa whilst Mfensi attained 29.6 MPa at 40% cement replacement. These strengths both passed the ASTM C 595 minimum strength of 24.1 MPa. After 36 h ball milling, the activated pozzolanas could be used to replace 30-40% OPC with Mankranso pozzolana cement attaining the highest strength of 27.7 MPa whilst Mankessim pozzolana showed a compressive strength of 25.6 MPa at 40% cement replacement at 28 days. The activated pozzolanas could thus be used successfully to replace up to 40% OPC for most non-load bearing housing construction purposes. The 90-day compressive strengths of the pozzolana cement mortar cubes for both Mankranso and Mankessim pozzolanas satisfied the ASTM C 595 and EN 197-1 standards.

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