# **Effect of Free Lime and Lime Saturation Factor on Grindability of Cement Clinker**

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Abstract: This research aims to investigate the effect of free lime and lime saturation factor on grindability of cement clinker. Cement is a powdered substance that develops strong adhesive and cohesive properties when mixed with water. Cement production involves a number of processes, these include; Preparation of raw materials, production of clinker, preparation of cement and its packaging. Clinker formed as a result of heating of well blended raw materials that undergo a chemical transformation inside the kiln. The methodology of this research work, are; 10g samples of clinker were collected on the apron conveyor chute with liter weights, (table 2.1), the weighted samples (10g) of clinker were milled and pelletized with the aid of pyridine and binding agent. The pelletized samples were subjected to XRF/XRD machine using thermo fisher analysis. They were done to determine the mineralogical compositions of the clinker samples and their respective elemental oxides. The result of the clinker minerals are; C<sub>3</sub>S (Tri calcium silicate), C<sub>2</sub>S (Di calcium silicate), LSF (Lime Saturation Factor) and other elemental oxide (table 2.2). However each of the sample were subjected to grinding for 40 minute using ball mill after adding 5% gypsum, these was done to determine the energy required to grind the clinker sample, and fineness was determined for each using the Blaine cell and air permeability apparatus (table 2.3). Correlation and regression analysis (statistical method) were applied on these results (table 2.4), the implication of the results shows there increase in energy requirement as FCaO (free lime) and LSF (lime saturation factor) increase in the clinker samples, there is also increase in energy requirement with the increase in Blaine (fineness). Therefore thus a clinker with low free lime would be grinded more easily than with high free lime, and also implies that the high the LSF and hence the harder the clinker to grind.

Keywords: Clinker, Lime saturation factor, Free lime, Belite, Alite Blaine, Correlation, Regression, Energy.

## 1. INTRODUCTION

Cement is a product obtained by the pulverizing clinker from the materials limestone, with cohesive and adhesive properties which make it capable of bonding minerals fragment into a compact whole. Cement is produced by grinding a mixture of a clay and limestone together and heating to a temperature of  $1450^{\circ}$ C, [Mohammed, 2012]. In which the partial fusion occurs and nodules of clinker are formed with the addition of shale, iron ore and gypsum. [Wilcox & Simon, 1995]. Grinding of the raw meal can also be reduced leading to further reduce the energy requirement for grinding. And grinding depends on many parameters and compositions which any slide deviation from these compositions can significantly affect the performance of the instillation and quality of the cement. Clinker is formed from burning or kiln stage, it can be described as nodules, or lumps usually 3 mm to 25 mm in diameter, and it is composition of four minerals:  $C_3S$ ,  $C_2S$ ,  $C_3A$  and  $C_4AF$  and these minerals is as a result of the reaction of oxides in the kiln, which leads to the formation of lime saturation factor (LSF). LSF is the ratio of CaO to SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. Clinker with LSF close or beyond to 1.0 indicate a likelihood of the presence of free lime, this will result to the high burn of clinker and hence difficult in grinding (CCNN-Sokoto, 2013). And it controls the proportion of  $C_3S$  to  $C_2S$ , in clinker, the high the value of it cause free lime(CaO) not to combine with oxide, such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. While free lime is the CaO that didn't combine with those oxides. Excess free lime results in undesirable effect such as volume expansion, increased setting time or reduced strength.

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About 30% of the energy are required to manufacture one ton of cement is used up in grinding. It sums up to a substantial percentage of the over- all cost of cement. The consumption of energy in cement mill varies within, 16.5 to 63.5 KWh/ton, having an average of about 36KWh/ton (M Tokyay, 1998). Several factors affect energy consumption during the process of clinker grinding. Clinker hardness brings about a wide disparity to grindability. About 80% extra power is usually required in processing hard clinker than the soft ones (Nwokedi, 20140.

The fineness of cement samples can be analyzed by a few methods which includes Dry sieve analysis and Blaine air permeability analysis. The dry sieve analysis measures the fineness of cement by means of a standard sieve in which the proportion of cement with grain sizes bigger than the specified mesh size of the sieve is collected and measures as the residue. Blaine air permeability apparatus determines the fineness of Portland cement in terms of surface area in g/cm<sup>2</sup> of cement. It consists of a brass or stainless steel permeability cell and a U tube manometer column. The process is described by an ASTM standard; it is based on the rate of air passage through a porous bed of cement particles under a specific pressure gradient as a function of the surface area. The process consists of a chamber whose volume and cross sectional area is known, filled with a known mass of cement. A specific volume of air is passed through the cement power and the time taken is measured. The surface area is then determined by calculations. [Thomas, Jennings, 2008].

Laboratory testing procedures established for determining the grindability of clinkers. The generally used procedures for this purpose are; determination of

- The mill energy requirement to produce cement with a definite fineness.
- Revolutions of the mill required to produce cement with definite fineness
- Specific surface area (Blaine) of the ground clinker for a specified energy consumption of the mill

This is mainly based on procedures I and III.

A number of investigations have been carried out to correlate the chemical and mineralogical compositions of clinker with the energy requirement. Table below shows this relationship, the equation drives for determining the energy requirement are;

Energy requirement,  $K = Xe^{BY}$ 

Where B is the Blaine (fineness) of the pulverized clinker simple in  $cm^2$ , X and Y are correlation values and are given below for a wide range of consumption factors.

[Nwokedi, 2014]

Relationships between major chemical parameters of clinker and constants X and Y of the exponential equation are;

Parameter	X	Y		
FCaO	1.2In(1/FCaO)+8.91	0.029In(1/FCaO)+0.5		

# 2. MATERIALS AND METHODS

#### 2.1 Materials

- 10 samples of various weights of clinker
- Water
- Gypsum

#### 2.2 Equipment Instrumentation

• **XRF/XRD Machine:** Is the ideal tool for direct quantification of the mineralogical composition of raw materials, clinker and cement products.

- **Crusher:** Is a machine used to reduce the size of limestone/clinker
- Ball mill: Is a key piece of equipment used for grinding crushed clinker/limestone
- Blaine air permeability apparatus: Is used to determine the Blaine (fineness) of the pulverized samples clinker.
- Weighing balance; It is used to measure the weight of the samples
- **Pyridine:** is a chemical and organic compound used to dissolve samples
- **Spatula:** A thin hand tool used for handling chemicals or other materials, when weighing.

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### **2.3 Experimental Procedure**

• Sample preparation: 10g each of the samples were weighed, milled and pelletized with the aid of pyridine ad binding agent

• The pelletized samples were subjected to XRF/XRD analysis using thermo fisher analysis incorporated with Goniometric. They was done to determine the mineralogical compositions of the clinker samples and their respective elemental oxides. The clinker minerals whose compositions were determined using XRDF/XRD are: C<sub>3</sub>S, C<sub>2</sub>S and C<sub>4</sub>AF, while the elemental oxides compositions determined also are: CaO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and MgO. The silica modulus, alumina modulus, lime saturation factor were found from the elemental oxides compositions using Bogue's equation stored as a program in the XRF/XRD analyzer.

- The free lime were determined using wet chemistry method, with the aid of free lime tester
- Each of the sample were subjected to grinding for 40 minute using ball mill after adding 5% gypsum
- The fineness were determined for each using the Blaine cell and air permeability apparatus

• It is base on the rate of air passage through a porous bed of cement particles under a specific pressure gradient as a function of the surface area.

• The energy requirement, k for grinding is calculated using  $K = Xe^{BY}$ ,

## 3. RESULTS

## 3.1 Results

Table 3.1: Collection of 10 sample of clinker with litre weights (Lt/Wt) as tabulated below

S/N	1	2	3	4	5	6	7	8	9	10
Lt/Wt (g/cm <sup>3</sup> )	1140	1242	1214	1300	1270	1252	1350	1325	1335	1300
Table.3.2: XRF/XRD Analysis of Clinker Samples										
Clinker	CaO	SM	AM	C <sub>3</sub> S	C <sub>2</sub> S	(	C <sub>3</sub> A	C <sub>4</sub> AF	LSF	FCaO
Samples	%			%	%	•	%	%	%	%
1	66.79	2.20	1.47	59.73	15.03	3 9	0.52	11.52	97.21	4.75
2	66.80	2.20	1.47	63.07	11.14	- 9	9.67	11.86	98.56	4.37
3	66.60	2.15	1.48	54.21	17.82	2 9	9.92	12.77	99.33	3.99
4	66.10	2.10	1.32	60.22	12.92	2 8	3.64	12.79	99.51	2.81
5	66.79	2.06	1.34	63.06	11.29	) 8	3.77	12.09	99.65	2.66
6	66.40	2.16	1.37	60.68	13.10	) 8	8.89	11.47	99.72	2.47
7	66.30	2.18	1.50	59.64	13.81	. 9	0.20	11.37	99.88	2.47
8	66.96	2.20	1.48	60.90	11.94	1 7	7.54	10.21	100.33	2.41
9	66.72	2.27	1.63	59.01	15.55	5 8	3.23	9.78	100.52	2.35
10	65.89	2.30	1.81	63.17	25.85	5 8	3.34	9.76	102.65	2.29

Table 3.3: X, Y and Energy requirement, k value for free lime in clinker samples.

Clinker Samples	Х	Y	K(kWh/ton)	FCaO (%)	Blaine (cm <sup>2</sup> /g)
1	7.04	0.443	45.25	4.75	4200
2	7.14	0.445	40.85	4.37	3920
3	7.25	0.447	41.07	3.99	3880
4	7.67	0.458	45.14	2.81	3870
5	7.74	0.459	37.19	2.66	3420
6	7.82	0.462	36.08	2.47	3310
7	7.82	0.462	32.90	2.47	3110
8	7.85	0.462	31.54	2.41	3010
9	7.88	0.463	29.62	2.35	2860
10	7.92	0.464	26.84	2.29	2630

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RELATIONSHIP	CORRELATION (r)	REGRESSION
BLAINE(X) VS ENERGY REQUIREMENT(Y)	0.978	Y = 0.012x - 4.404
FREE LIME(X) VS ENERGY REQUIREMENT(Y)	0.745	Y = 5.12x + 20.996
FREE LIME(X) VS BLLAINE(Y)	0.8606	Y = 483.56x + 1942.8
LSF(X) VS BLAINE(Y)	-0.8962	Y = 338.283x - 30317.99

 Table.3.4: Represent the regression and correlation result of the analysis.

#### **3.2 Discussion of results**

**Table.3.2;** The results Shows the minerals composition of 10 samples of clinker subjected to XRF/XRD analysis, the samples were collected at random because of the varying amount of lime (CaO) and the calcium oxides. The composition factors AM, SM and LSF are factors relating the proportions of SiO<sub>2</sub>, CaO, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> in the clinker samples. It was observed that, the free lime (FCaO) present in each samples of clinker decreasing, the LSF increased. This indicated that the more burning of clinker, the higher the composition of belite (C<sub>2</sub>S) and hence less free lime is available for the formation of alite (C<sub>3</sub>S) after the burning zone of the kiln. However, it is a known fact that stable glazy crystal structure of belite are very hard to grind and hence clinker with very low free lime is not healthy to Ball mill during grinding.

**Table 3.3;** Contain X, Y and Energy requirement (K) values for free lime in clinker samples, where X and Y are the correlation factors and are drives from the below empirical equation;

X = 1.2In (1/FCaO) + 8.91

Y = 0.029In (1/FCaO) + 0.488 and the Energy Requirement K calculated from this below Equation;  $K = Xe^{BY}$ . Where B = B laine (fineness)

It was also observed that there increases in the energy requirement as the free lime increases.

Table 3.4; Represent the entire calculated results of the regression and correlation analysis.

The calculation was done using the statistical equations below;

 $r(correlation) = \frac{\sum dx dy}{\sqrt{\sum dx 2 dy^2}}$ 

This regression equation: (y = bx + a) was also used to find the best fit line between the two variables

Where  $b = \frac{\sum dx dy}{\sum dx^2}$  and  $a = \overline{y} - b\overline{x}$ 

Where  $\overline{y}$  and  $\overline{x}$  are the mean of the respect variables

However in the relationship between free lime and energy requirement, the correlation shows positive, which implies increase in energy requirement as the free lime increase. And also indicated increase in energy requirement to achieve fineness as the LSF or lime saturation factor increased. However there is negative correlation in the relationship between LSF and Blaine (fineness), which implies decrease in Blaine (fineness) of the clinker as the LSF increase. This confirms that the clinker is completely burnt in the kiln, the high the LSF and hence the harder the clinker to grind.

# 4. CONCLUSION AND RECOMMENDATIONS

#### 4.1 Conclusion

Finally it has been concluded that, free lime and LSF (lime saturation factor) compositions have considerable effects on cement clinker grindability. As the results indicated there increases in energy requirement as free lime & LSF increases in the clinker samples. Thus a, clinker with low free lime would be grinded more easily than with high free lime. Also indicate an increase in energy requirement with increase in Blaine. Thus in achieving finer particles, more energy is required in grinding. The negative results indicate a decrease in Blaine of the clinker with increase of LSF. This confirms that, the clinker contain a higher LSF in the kiln, the high the LSF and hence the harder the clinker to grind.

### 4.2 Recommendations

In order to eliminate the problem of high free lime, during pre-calcination of kiln feed in the precalciner and sintering zone of the raw meal in the kiln, the correlation between the kiln feed and clinker LSF should be carefully care. Therefore the kiln operators should place more attention on the LSF target of the clinker/kiln chemistry by increasing burning and hence, free lime of the kiln output will be relatively low. This therefore will guarantee cement quality, suitable and profitable fuel and energy requirement at the cement mill to grind the clinker to a specified fineness and time, since the production time can also affect the output and profit.

The balanced feed rate to fuel flow rate in the kiln should pay attention by the cement plant process engineer. This is to improve the proper burning of the sintering raw material in the kiln, so that the resulting clinker has relatively low free lime and suitable target of lime saturation factor and hence, suitable/profitable energy requirement for grinding the clinker at the cement mill.

However, the grinding aid will increase the product fineness, reduce the power consumption and reduce ball coating. Therefore the mill operators should ensure proper used of grinding aid.

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#### REFERENCES

- [1] Hills.M.L. (2007). Solid Fuel Grindability. www.cement.org. @Portland Cement
- [2] Gavel. V. (2013) Grinding theoretical and technological investigation to establish the production of environmental friendly cement with reduced clinker content. Unpublished Ph.D, Thesis.
- [3] Cement Manufacturers Association of Nigeria, Optimizing clinker production in Nigeria industrial Standard, Standard of Cement pages 9, A publication of Standard Organization of Nigeria, Wuse Zone 7, Abuja Nigeria
- [4] Cement Company of Northern Nigeria PLC Sokoto (2004), Cement Grinding Training Book page 11-13
- [5] Cement Company of Northern Nigeria Plc Sokoto, (2003) Cement Production Course, Milling Technology, page 6-16
- [6] (Nwokedi, 2014). Investigation of the effect of Free Lime and Lime Saturation Factor on Grindability of Cement Clinker. Research work. Federal University of Technology, Minna. Niger State, Nigeria. Unpublished
- [7] Hashim. S, Hussin. Azizli. K.A, Palaniandy, S. (2008) Study of Clinker Characteristics and Grindability during Cement Production. Final Report of short term Research project, University of Malaysia.
- [8] Ted Krapkat, 2015. Note on effect of Free Lime and LSF on Burning of Clinker
- [9] Tokyay. M. (1999). Effect of Chemical Composition of Clinker on Grinding Energy Requirement. Civil Engineering Department, Middle East Technical University, 06531 Ankara, Turkey. Cement and Concrete Resarch (29) 531-535.
- [10] Winter N.B (2005). Understanding Cement. 2005-2014 WHD Microanalysis Consultancy LTD. Pg 75.
- [11] Gavel.V. (2003). Grinding theoretical and technological investigation to establish the Production of Environmental Friendly and Cement with Reduced Clinker Content. Unpublished Ph.D. Thesis. University of Miskolc Faculty of Earth Science & Engineering Institute of Raw Materials Preparation and Environmental Processing.
- [12] Svinning. K. (2011). Design and Manufacture of Portland Cement Application of statistical Analysis. Ph.D. Thesis. Faculty of Natural Science and Technology.
- [13] Soumaya. I, N jib. B, Khaoula. M and Mohammed. B. (2011). Comparative Study for Grinding of Two Cement Clinkers, International Journal of Concrete Structure and Materials. Vol.5, No.2, pp.113-117.

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- [14] Gebhardt, 1988, Research on Free Lime Determination in Clinker
- [15] Effect of Chemical Composition of Clinker on Grinding Requirement, [M. Tokyay, 1998] Where is the iron? Clinker Microanalysis with XRD Rietveld, Optical Microscopy, Alison Crumie, 2006.
- [16] Kebede, M.A (2010). Investigation of calcite and Volcanic ash for their Utilization as Cement Filling and additive Materials. Unpublished M.Sc. Thesis Addis Ababa University School of Graduate Studies.
- [17] Christian J. E. (2008). Quality Improvers in Cement making State of the art COIN Project report 2. Retrieved from www.sintef.no/upload/.../coin-no2.pdf
- [18] Mohammed, T.A. (2012). Composition and phase mineral Variation of Portland Cement in Mass Factory Sulaimani-Kurdistan Region NE – Iraq, International Journal of Basic & Applied Sciences, 12(06), pg 116 – 117.
- [19] Cement Company of northern Nigeria plc Sokoto, September 2003, Milling Training Book.