Determination of granulometrical composition of the clinker by grinding in a ball mill to determine the specific consumption of additional energy

Cristian Ciobanu¹, Paula Tudor³, Gabriel-Alexandru Constantin^{2,*} and Gabriel Musuroi²

¹CEPROCIM Bucharest, Romania

²University Politehnica of Bucharest, Faculty of Biotechnical Systems Engineering, Romania ³University Politehnica of Bucharest, Faculty of Entrepreneurship, Business Engineering and Management, Romania

Abstract. This paper presents results regarding the grinding behaviour of two batches of material (clinker) in a ball mill with its own two-step grinding process: the first one with a ball load, the second with a load of cylindrical metal bodies. The first step was performed until the residue R_{009} has reached aprox.35%, and the second stage until Blaine's specific surface was over 3800 cm²/g. Periodically, at 10-minute intervals, the fineness of the clinker and the specific energy consumption were determined. These consumptions are quantified from the first determination and relates to the weight of the material subjected to grinding (20 kg clinker / from a source), determining the grinding ability of the material.

1 Introduction

The cement consists of dispersed solid particles whose particles have a size between 0.1 and 250 μ m. Particle size, particle size distribution, but also the composition of the cement has a special influence on its behaviour during wetting, on the development of microstructures and on the properties of concrete or cement-based materials. It is important that Portland cement particle sizes fall within certain limits so that they are properly moistened and to form a material with a good subsequent behaviour. More, Excessive grinding leads to additional energy consumption (sometimes unjustified), and insufficient grinding can affect the binder properties of the cement particles and their distribution in the prepared material.

Among the properties that have been examined through experiments and computer simulation are: setting time, heat release, capillary porosity percolation, diffusivity, chemical shrinkage, autogenous shrinkage, internal relative humidity evolution, and interfacial transition zone microstructure, [1]. The authors concluded that, for lower water-cement ratio values, coarser cements can be used that can provide equivalent or even superior performance, which would reduce the production costs to the manufacturer.

^{*} Corresponding author: constantin.gabriel.alex@gmail.com

Still, there are researchers who claim that cement particles larger than 45 μ m are difficult to hydrate, and also the largest of 75 μ m cannot be completely hydrated, when analysing the rate of reactivity and the development of stresses in the prepared concrete, [2]. Author of this paper, [2], states, however, that estimates of reactivity rates for similar cement compositions cannot be made without knowing the particle size distribution by sedimentation methods. In any case, there must be a correlation between the setting time and the fineness of the cement.

The standard method for determining particle size and particle size distribution is ASTM C115-96, with a limit of detection of a smaller size of 7.5 μ m [3]. This is based on the turbidimetric method for determining the fineness of the material. The Electrical Zone Sensing (EZS) method, which is based on the Coulter principle, can operate in the particle size range between 0.2 (0.6)–800 μ m, while laser diffraction is used successfully when most particles have dimensions below 1 μ m, [3].

From the four techniques analysed by the authors of the previous paper: laser diffraction (LAS), detection of electrical areas (EZS), X-ray gravitational sedimentation (XRS) and scanning electron microscopy (SEM), laser diffraction (with both wet and dry dispersion), gave the best yields, this being the basis for determining the specific Blaine surface.

For cements, are two systems for determining fineness, namely, specific surface Blaine (air permeability) and turbidimetry. Blaine fineness is an indirect measure of the total surface of each cement sample and can be determined according to ASTM C204-11 "Standard Test Method for Fineness of Hydraulic Cement by Air Permeability Apparatus".

For Portland cement from the same source, the heat of hydration is affected by the fineness of the cement and the particle size distribution. Grinding to a greater fineness Blaine, in correlation with decreasing particle size, leads to an increase in the heat of hydration, experimentally confirmed [4].

The necessary fineness of the final cement is due to the milling means of the ball mills and the size distribution of the feed material. To increase the efficiency of ball mills, for optimum distribution of the final material according to the particle size, it is necessary, therefore, a correlation between the size of the balls and the size of the particles when feeding the mill, [5]. It was concluded that a ternary or at least binary mixture of balls of different sizes (50 + 20 + 10 mm) leads to a final material of optimum size.

More energy efficient grinding technologies, applied in the grinding of cement, are described comparatively in the paper [6], from the point of view of operating principles, grinding efficiency, specific energy consumption, the production capacity and the quality of the cement obtained. Other papers addressing the theme of milling process with balls dedicated to grinding cement, including its control are [7-11]

The present paper presents the results regarding the grinding behaviour of two batches of material (clinker) in a ball mill with its own two-step grinding process: the first one with a ball load, the second with a load of cylindrical metal bodies.

2 Material and method

Energy consumption between the moments when the fineness of the material is determined (in our case the consumption was read directly on the electricity meter) was determined using a wattmeter. These consumptions have accumulated since the beginning of the determination and have been related to the mass of the batch (20 kg clinker/factory from 2 factories), calculating the specific energy consumption w_{1i} . The aptitude index for milling is the specific energy consumption w_1 corresponding to a reference fineness and w - the specific energy consumption of the industrial mill, [12].



a): ball mill diagram; 1- motor, gear reducers and ring and pinion gear; 2- cylinder; 3- brick lining; 4- discharge hatch; 5- bearings; *b)*: working process of ball mill

To determine the specific surface (SSB) of the grinded material we chose the method of the Blaine permeability meter. The Blaine procedure is applicable for all cements defined in the standard EN 196-6 :2018, [14].



Fig.2 Permeabilimeter Blaine, [14,15]: *a)General aspect; b)Manometer: 1- conical socket; 2-manometer; 3- stopcock; 4- rubber bulb; 5-8- etched lines; c)Permeability cell: 3- cell; 4- cement bed compacted by the plunger; 5- paper filters; 6- perforated disc; d)Plunger piston: 1- piston body; 2-flat*

The Blaine permeability meter also includes: stopwatch; analytical balance; manometric liquid; apparatus for determining the density of cement (Le Chatelier 100 ml quoted balloon).

The specific surface S was calculated accordingly SR EN 196-6:2018 [16] and is conventionally expressed in $\text{cm}^2 \cdot \text{g}^{-1}$, as it is:

$$S = \frac{K}{\rho} \cdot \frac{\sqrt{e^3}}{(1-e)} \cdot \frac{\sqrt{t}}{\sqrt{10 \ \eta}} \tag{2}$$

where: K - the constant of the device, e - porosity of the layer; t - time measured (s); ρ – bulk density of the material, (g·cm⁻³), η - the viscosity of the air at the test temperature, according to table no.1, (Pa·s)

With the specified porosity e = 0.500 and the test temperature $20 \pm 2^{\circ}C$:

$$S = \frac{52.43 \cdot K \sqrt{t}}{\rho} \tag{3}$$

where: *K* thus determined:

$$K = 1.414 \cdot S_o \cdot \rho_o \frac{\sqrt{10 \eta_o}}{\sqrt{t_o}} \tag{4}$$

 S_0 - the specific surface of the reference cement, $(cm^2 \cdot g^{-1})$, ρ_0 - the bulk density of the reference cement, $(g \cdot cm^{-3})$, t_0 - the average of three timed values of time, (s), η_0 - the viscosity of the air corresponding to the average of three temperatures, (Pa \cdot s) and is given according to table 1.

Temperature, º ºC	Bulk density of Hg, ρ _{Hg} [g·cm ⁻³]	Viscosity of the air η_0 , Pa·s	$\sqrt{10 imes \eta_0}$
16	13.560	0.00001800	0.013416
17	13.560	0.00001805	0.013435
18	13.550	0.00001810	0.013454
19	13.550	0.00001815	0.013472
20	13.550	0.00001819	0.013487
21	13.540	0.00001824	0.013506
22	13.540	0.00001829	0.013524
23	13.540	0.00001834	0.013543
24	13.540	0.00001839	0.013561
	Intermediate values must be obtain	ed by linear interpolation	

Table 1. Bulk density of Hg, viscosity of the air $\eta_{0 \text{ and }} \sqrt{10 \eta_0}$ function of temperature, [16]

For the determination of the laser particle size analysis for Clincher A and F samples, the Mastersizer 2000E (MALVERN, UK) was used, [16].



Fig.3 Mastersizer 2000E (MALVERN, UK) for laser particle size distribution, [16]

Laser particle size distribution was performed under the following measurement conditions: Wet dispersion (dispersion liquid: Ethanol \approx 850ml); Refractive index dispersion liquid: 1.360; Ultrasound duration: 60 sec; Ultrasound level: 10 µm; Stirring pump speed: 2300 rpm; Laser diffraction apparatus: Mastersizer 2000E; Focal distance of the lenses:2.35; dimensional range: 0.100 µm -1000.00 µm; Optical concentration:18.45 %; Light dispersion model applied : MieAbsorbtie theory: 0.1.

3 Results and discussions

Following the determinations were recorded the following energy consumption and specific surface Blaine (SSB) for two types of clinker A and F from the 2 cement factories (Table 2).

Grinding time, [min]	No. on counter [Kwh], clinker A	No. on counter [Kwh], clinker F	Difference on counter, [Kwh], clinker A	Difference on counter, [Kwh], clinker F	Energy consumption [kwh/t] clinker A	Energy consumption [kwh/t], clinker F	Correlation coefficient c ₁ clinker A	Correlation coefficient c ₁ clinker F	SSB [cm ² /g]-bi-cones Residue [%]-balls clinker A	SSB [cm ² /g]-bi-cones Residue [%]-balls clinker F	Grinding bodies
0	7350421	7356265	0	0	-	-	-	-	-	-	Grinding balls
10	7350661	7356504	240	239	-	-	-	-	51.68 %	53.4 %	Grinding balls
20	7350882	7356729	221	225	-	-	-	-	33.6%	34.5 %	Grinding balls
30	7351096	7356946	214	217	10.7	10.85	3.08	3.03	2250	2190	Bi-cones
40	7351314	7357167	218	221	10.9	11.05	3.02	2.98	2650	2640	Bi-cones
50	7351526	7357387	212	220	10.6	11	3.11	2.99	2830	2960	Bi-cones
60	7351746	7357617	220	230	11	11.5	2.99	2.86	3180	3130	Bi-cones
70	7351968	7357837	222	220	11.1	11	2.97	2.99	3520	3290	Bi-cones
80	7352196	7358069	228	232	11.4	11.6	2.89	2.84	3590	3580	Bi-cones
90	7352417	7358290	221	221	11.05	11.05	2.98	2.98	3700	3870	Bi-cones
100	7352642	-	225	-	11.25	-	2.93	-	3870	-	Bi-cones

 Table 2. Specific consumption and Blaire surface area for the two types of clinker

Total energy consumption clinker A $=\frac{7352642-7350421}{20} = 111.05$ kwh/t (5) Total energy consumption clinker F $=\frac{7358290-7356265}{20} = 101.25$ kwh/t (6)

W_{industrial mill consumption}=32.92 kWh/t according to the value calculated by the cement factory. Regarding energy consumption, Atmaca and Kanoglu found that this consumption is 24.75 kWh/ton farine, depending on the temperature and humidity of the material, [17].

Results regarding laser chemical and particle size distribution of the material subjected to grinding (clinker A and F) are presented in Table 3.

Determination	TIM	Test code	Test code			
Determination	U.M.	Clinker A	Clinker F			
Loss on calcination – L.C.	%	0.53	0.96			
SiO ₂	%	21.20	20.88			
Al ₂ O ₃	%	5.66	5.04			
Fe ₂ O ₃	%	2.99	3.80			
CaO	%	66.34	66.78			

Table 3. Chemical analysis results clinker A and F

MgO	%	1.20	0.80
SO ₃	%	0.83	0.74
Na ₂ O	%	0.21	0.12
K ₂ O	%	0.50	0.34
CaO free	%	0.31	0.92
HCl-Na ₂ CO ₃	%	0.15	0.17

Following the measurements the following charts were obtained.



Fig.4 Grinding aptitude index clinker A



Fig.5 Grinding aptitude index clinker F

Based on the equilibrium diagrams, the basic composition of the clinkers was calculated. The calculation was made in the hypothesis of solidification at thermodynamic equilibrium and at thermodynamic nonequilibrium. In case of solidification at full equilibrium, the glass phase is completely absent. In reality, no thermodynamic balance is achieved at solidification and the mineralogical composition calculated from such a hypothesis can be considered as a potential composition, that is, possible, understanding by this that the respective chemical composition is not equal to the composition of the real minerals. Assuming the clinker as being placed in the thermal equilibrium subsystem $C_3S-C_2S-C_3A-C_4AF$ (MA_i > 0.64), Bogue has established the following calculation formulas for mineralogical constituents, [12]:

% $C_4AF = 3.04$ % Fe_2O_3 % $C_3A = 2.65$ % $Al_2O_3 - 1.69$ % Fe_2O_3 $\% \ C_3S$ =4.07 % CaO - 7.60 % SiO_2 - 6.72 % Al_2O_3 - 1.42 % Fe_2O_3 -

% $C_2S = 8.60$ % $SiO_2 + 5.06$ % $Al_2O_3 + 1.07$ % $Fe_2O_3 - 3.05$ % CaO.

Thus, from the mineralogical composition according to Bogue's calculation, the values presented in table 4 resulted.

Clinker A	Clinker F
$C_3S = 66.48$	$C_3S = 71.39$
$C_2S = 11.48$	$C_2 S = 6.8$
$C_3A = 9.95$	$C_3A = 6.93$
$C_4AF = 9.09$	$C_4AF = 11.55$

Table 4. Mineralogical composition according to Bogue's calculation for two clinkers

It is observed that C_2S clinker $A > C_2S$ clinker F (11.48 > 6.8) which results in clinker A requiring additional energy consumption in the mill because it has a much higher content of C_2S .

The results of the determinations of the laser particle size distribution for clinker A are presented in Table 5.

Granul ometric class	Cumula tive passing	Granulo metric class	Cumulativ e passing (% vol)	Granul ometric class	Cumula tive passing		Granul ometric class	Cumula tive passing	Granul ometric class	Cumula tive passing
0.10	0.00	3.00	12.26	13.00	42.36	-	<u>(µm)</u> 64.00	93.99	175.00	100.00
0.20	0.00	3.15	12.99	14.00	44.32		70.00	95.79	200.00	100.00
0.30	0.00	3.50	14.66	15.00	46.21		75.00	96.96	225.00	100.00
0.40	0.00	4.00	16.95	16.00	48.05		80.00	97.87	250.00	100.00
0.50	0.35	4.50	19.13	18.00	51.58		85.00	98.59	280.00	100.00
0.60	0.95	5.00	21.16	20.00	54.92		90.00	99.14	315.00	100.00
0.70	1.60	5.50	23.06	22.00	58.10		95.00	99.52	355.00	100.00
0.80	2.21	6.00	24.83	24.00	61.12		96.00	99.58	400.00	100.00
0.90	2.77	6.30	25.84	25.00	62.58		100.00	99.78	450.00	100.00
1.00	3.28	6.50	26.49	28.00	66.71		106.00	99.94	500.00	100.00
1.20	4.16	7.00	28.06	31.50	71.10		110.00	99.99	560.00	100.00
1.40	4.98	7.50	29.53	32.00	71.69		115.00	100.00	600.00	100.00
1.50	5.38	8.00	30.93	36.00	76.10		120.00	100.00	650.00	100.00
1.60	5.79	8.50	32.26	40.00	79.95		125.00	100.00	700.00	100.00
1.80	6.63	9.00	33.53	45.00	84.06		128.00	100.00	750.00	100.00
2.00	7.51	9.50	34.76	48.00	86.17		130.00	100.00	800.00	100.00
2.20	8.42	10.00	35.94	50.00	87.45		140.00	100.00	850.00	100.00
2.50	9.84	11.00	38.19	55.00	90.24		150.00	100.00	900.00	100.00
2.60	10.33	12.00	40.32	60.00	92.51		160.00	100.00	950.00	100.00
2.80	11.29	12.50	41.35	63.00	93.65		170.00	100.00	1000.0	100.00

Table 5. Particle size distribution for clinker A

The sample subjected to the laser particle size analysis was the material passed through the sieve of 1000 μ m. Residue value on sieve of 1000 μ m it was of R₁₀₀₀ = 0.0 %. Following the results obtained, the chart of the cumulative passage of material was drawn (% vol) depending on the granulometric class (μ m) from 0.10 μ m to 1000 μ m (see fig. 6).



Fig.6 Chart clinker A

The results of the determinations of the laser particle size distribution for clinker F are presented in Table 6.

Granu	Cumul	Granu	Cumul	Ĺ	Granu	Cumula	Granu	Cumul	Granu	Cumul
lometri	ative	lometri	ative		lometri	tive	lometri	ative	lometri	ative
c class	passing	c class	passing	Ĺ	c class	passing	c class	passing	c class	passing
(µm)	(% vol)	(µm)	(% vol)	L	(µm)	(% vol)	(µm)	(% vol)	(µm)	(% vol)
0.10	0.00	3.00	10.79		13.00	41.44	64.00	93.24	175.00	100.00
0.20	0.00	3.15	11.48		14.00	43.47	70.00	95.04	200.00	100.00
0.30	0.00	3.50	13.08		15.00	45.43	75.00	96.24	225.00	100.00
0.40	0.00	4.00	15.32		16.00	47.33	80.00	97.21	250.00	100.00
0.50	0.30	4.50	17.49		18.00	50.97	85.00	97.99	280.00	100.00
0.60	0.87	5.00	19.54		20.00	54.41	90.00	98.61	315.00	100.00
0.70	1.49	5.50	21.47		22.00	57.66	95.00	99.10	355.00	100.00
0.80	2.08	6.00	23.29		24.00	60.75	96.00	99.19	400.00	100.00
0.90	2.59	6.30	24.32		25.00	62.22	100.00	99.50	450.00	100.00
1.00	3.04	6.50	24.99		28.00	66.40	106.00	99.84	500.00	100.00
1.20	3.79	7.00	26.60		31.50	70.80	110.00	99.97	560.00	100.00
1.40	4.45	7.50	28.13		32.00	71.39	115.00	100.00	600.00	100.00
1.50	4.77	8.00	29.58		36.00	75.76	120.00	100.00	650.00	100.00
1.60	5.10	8.50	30.96		40.00	79.55	125.00	100.00	700.00	100.00
1.80	5.79	9.00	32.28		45.00	83.55	128.00	100.00	750.00	100.00
2.00	6.52	9.50	33.55		48.00	85.61	130.00	100.00	800.00	100.00
2.20	7.31	10.00	34.77		50.00	86.85	140.00	100.00	850.00	100.00
2.50	8.56	11.00	37.11		55.00	89.56	150.00	100.00	900.00	100.00
2.60	9.00	12.00	39.32		60.00	91.77	160.00	100.00	950.00	100.00
2.80	9.89	12.50	40.39		63.00	92.89	170.00	100.00	1000.0	100.00

Table 6. Particle size distribution for clinker F

The sample subjected to the laser particle size analysis was the material passed through the sieve of 1000 μ m. Residue value on sieve of 1000 μ m it was of R₁₀₀₀ = 0.0 %. Following the results obtained, the chart of the cumulative passage of material was drawn (% vol) depending on the granulometric class (μ m) from 0.10 μ m to 1000 μ m (see fig.7).



Fig.7 Chart Clinker F

4 Conclusions

Cement production requires large amounts of thermal and electrical energy. Energetic efficiency, defined as the total thermal energy consumed per ton of clinker produced and, respectively as the total electricity consumed per ton of cement produced, depends almost entirely on the technology applied in the production process.

According to the laboratory analyses it is observed that the mineralogical composition according to Bogue's calculation C_2S clinker $A > C_2S$ clinker F (11.48 > 6.8) which results in clinker A requiring additional energy consumption in the mill because it has a much higher content of C_2S (calcium silicate) fact shown by the energy consumption to clinker A 111.05 kwh/t > clinker F 101.25 kwh/t.

Results for laser particle size distribution for clinker A and for clinker F they showed that both clinkers at granulometric class of 115 μ m, has a cumulative passing (%material volume) is integral (100%). Besides reducing production costs, increasing energy efficiency contributes positively to the conservation of natural resources (fuels) as well as reducing greenhouse gas emissions (CO₂) generated both by combustion of fuels in the clinker oven (direct emissions) or in power plants for the production of electricity consumed in the cement factory (indirect emissions)

A direction of increasing energy efficiency is represented by the use of alternative fuels from waste, method with significant potential for reducing conventional fuel consumption.

Reduction of electricity consumption per tonne of product contributes to the refurbishments made in recent years at cement plants in Romania, which include:

- · Replacing air compressors with some efficient ones
- · Replacement of old pneumatic cement transport installations
- Resizing the ball mill load and installing systems to optimize their operation
- Installation of a high energy efficiency separator and a state-of-the-art filter
- Optimization of control of the machine flows through the Process Control System.

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