A study on the relationship between the particle size distribution of cement and the development of the compressive strength of the consequent mortar.

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Abstract

The objective of this work resides in the study of the relationship between the PSD of the cement that makes up the mortar and its influence on the development of its compressive strength.
Since the beginning of the use of cement, new and different fields of application have been implemented in the world, thus increasing the demand for cements with different specifications and properties. But producing cement is an extremely expensive and environmentally damaging activity. This study is dedicated to finding an energy efficient (and therefore also economically) way to produce a cement that is competent with the established requirements, focusing on the problem of milling, the most costly and least efficient process in cement manufacturing.
A series of preliminary tests were carried out to determine the most efficient way to reach the target PSD, in which different combinations and variations of the key parameters for the energy efficiency of the milling were tested: milling time, milling body load, pre-milling of the material and non-stick additive. After comparing the percentage of fines (final residue and passing percentage) present in the samples tested, a milling system was established with the most efficient conditions.

With the samples obtained in the grinding, mortar specimens were manufactured that were subsequently cured in a humid chamber. The flexural and compression tests carried out on the specimens showed that their compressive strength is higher as the curing time increases and their proportion of particles between 63 and 125μm is greater.

Key words: cement milling, ball mill, energy efficiency, PSD, compressive strength.

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1. Introduction

Cement is an energy-intensive industry in which the milling circuits consume around 60% of the total electrical energy of the manufacturing process (Figure 1), representing a very high and specific energy consumption of this stage (Genç, 2016). In general terms, cement production consumes 2% of global primary energy and 5% of total global industrial energy.

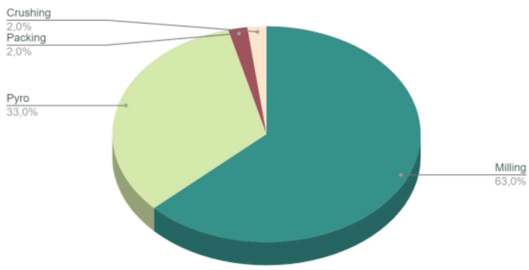

Figure 1: Power consumption in the diverse areas of a cement plant.

Cement has unique physical properties depending on the proportion of main raw materials that make it up, on the amount of secondary raw materials that have been added to the mix, and also on the additives that have been incorporated. In fact, the main function of the addition of other materials or additives to the paste in the global cement industry is small variations in the physical properties of cement, such as compressive strength or workability. The particle size distribution (PSD), which specifies the proportion of fine and coarse particles in the cement, controls the hydration reactions, setting time and water demand, of the mortar created from the cement, as well as how it influences the physical and mechanical properties that the mortar will have. Different types of cements are necessary for different types of functions, and that is why a variation is made in their composition, PSD or production process to achieve a cement that conforms to the required requirements as closely as possible (Ghalandari & Iranmanesh, 2020).

The objective of the study undertook to test the existence of a correlation between the development of the compressive strength and the percentage of fine particles (equal to or less than 45μm) in a mortar specimen, therefore samples with different PSD were chosen to evaluate the variation in compressive strength and the composition of the samples

This study evaluated how the granulometric curve of the cement affects the compressive strength of the mortar, with the intention of obtaining resources to reduce the electrical consumption of cement grinding without losing the properties necessary to obtain a quality mortar that is competent with the evolutionary needs of society.

2. Materials and Method

Laboratory experiments were carried out in order to obtain data on the optimal characteristics of the grinding process of a cement sample and the relationship of the results obtained on the compressive strength of said cement.

2.1 Sample Milling

Many are the variables that affect the milling process and therefore, the PSD of the product. In this experiment these variables were changed in order to find which combination of them was more favorable for the desired PSD. In Table 1 it is shown how the variables changed during the preliminary tests.

Table 1: Variable disturbance during the preliminary tests.

Test sample #	Composition	Pre-milling of G	Pre-milling of CK	Grinding aid (drops)	Milling body load	Milling time (mins)
3	93% CK, 7% G	✓			1	60
4	93% CK, 7% G	✓			2	55
5	93% CK, 7% G	✓			2	55
6	93% CK, 7% G	✓			2	120
7	93% CK, 7% G	✓			1	120
8	93% CK, 7% G	✓			1	120
9	93% CK, 7% G	✓	✓	1	1	120
10a	93% CK, 7% G	✓	✓	1	2	120
10b	93% CK, 7% G	✓	✓	1	2	150
11	93% CK, 7% G	✓	✓	1	3	120
12a	93% CK, 7% G	✓	✓	2	1	120
12b	93% CK, 7% G	✓	✓	2	1	180
13	93% CK, 7% G	✓	✓	2	1	120

It should be noted that from sample #13 the variables were always kept constant, adjusting to the criteria shown in this test.

2.2 Ball Charge Pattern

Different ball patterns were used in the grinding of clinker and gypsum (Table 2). In addition, in some tests an additive (i.e. surfactant) was added: MAGA C077 (supplier -MAPEI). The dose used at an industrial level is between 300-500 g/Ton.

Table 2: Specifications of the ball charge patterns.

Ball diameter (mm)	1		2		3	
	Number of balls	Weight (g)	Number of balls	Weight (g)	Number of balls	Weight (g)
48	4	1674,5	7	2791,1	5	2121,8
40	14	3155,1	17	3854,3	15	3492,6
30	28	2747,6	16	1506	22	2226,4
25	4	224,6	7	398,5	6	332,2
23	-	-	9	401,7	15	666,1
19	19	867,9	-	-		
13,5	37	392,6	11	108,1	28	223,9
Total	117	9060,5	67	9059,7		9063

2.3 Granulometric Analysis

Two different tests were carried out to determine the granulometric curve of each initial sample obtained from a preliminary test: screening and Malvern laser test. The reason for the use of this second is the verification and contrast of the data obtained with the screening.

2.4 Constitution of Artificial Samples

Artificial samples were made with the material obtained from the preliminary mills. These samples are made up of different percentages of the same particle size, the particle size range being 45 to 125µm. Between 92-95% of the composition of these artificial samples are particles smaller than or equal to 45µm, the target size for mills.

The procedure was as follows:

I. Study of the grinding conditions of the samples and grouping among the most appropriate.

II. The pertinent calculations were carried out to know how much material of fines (less than 45µm) each artificial sample should contain, putting as a condition values that were between 92 and 95%, the objective value (Table 3). The remaining sizes were randomly designated to fit the new artificial sample closely to an industrial mill production.

Table 3: Composition of the artificial samples.

Artificial Sample	Mixture of samples	Composition (%)	Particle Size Distribution, PSD								Total weight (g)
			Percentage (%)				Weight (g)				
			-45µm	-63 +45µm	-90 +63µm	-125 +90µm	-45µm	-63 +45µm	-90 +63µm	-125 +90µm	
A	#3+#4	93% CK, 7% G	95	3	1,2	0,8	443,3	14	5,6	3,7	466,6
B	#5+#7	93% CK, 7% G	95	2,25	1,75	1	443,3	10,5	8,2	4,7	466,6
C	(#3+#4)+#6+ #8+#10	93% CK, 7% G	93	3,2	2,5	1,3	433,9	14,9	11,7	6,1	466,6
D	(#3+#4)+#6+ #8+#9+#10	93% CK, 7% G	94	2,75	2	1,25	438,6	12,8	9,3	5,8	466,6
E	#11+#12+#14	93% CK, 7% G	92	3,5	2,8	1,7	432,4	16,5	13,2	8	470
F	#11+#12+#14	93% CK, 7% G	93	3,7	1,8	1,5	437,1	17,4	8,5	7,1	470
G	(#3+#4)+#6+ #8+#9+#10+ #13+#15	93% CK, 7% G	94	2	2	2	441,8	9,4	9,4	9,4	470
H	(#3+#4)+#6+ #8+#9+#10+ #13+#15	93% CK, 7% G	92	3	2,5	2,5	432,4	14,1	11,8	11,8	470
I	#16+#17	93% CK, 7% G	93	2,75	2,5	1,75	437,1	12,93	11,75	8,23	470
J	(#3+#4)+#6+ #8+#9+#10+ #13+#15+#1 6+#17	93% CK, 7% G	91	4	2,5	2,5	427,7	18,8	11,75	11,75	470
K	#19+#21	93% CK, 7% G	92	3	2.5	2.5	432.4	14.1	11.75	11.75	470

III. Mixing of the samples by particle size. Thus, several samples were joined, for example # 3 and # 4, particles smaller than 45µm on the one hand, particles larger than 45µm and smaller than 63µm on the other hand, and similarly for the rest, the largest size being between 90-125µm.

IV. Following the aforementioned calculations, the artificial samples were produced.

V. 5g of each artificial sample was removed for the subsequent granulometric study at the Malvern, and another 4g of material to be sent to the supplier company. After this extraction the artificial samples were stored.

2.5 Manufacture of Mortar Specimens

According to the standard NP EN 196- 1 “Cement test methods; determination of mechanical resistance”, each mix has to contain 450g of cement, 1350g of CEN standardized sand and 225g of water, resulting in 3 test tubes for each mix produced. The specimens were cured in a humid chamber characterized for having a temperature of approximately 20 ± 2 °C and a relative humidity of 95 ± 5%.

The workability tests carried out to characterize the mortar specimens were made following the UNE-EN 1015-3.

2.6 Bending and Compression Resistance Tests

As there were 3 specimens per sample, each one had a different cure time; one of them healed in the chamber for 2 days, another for 7 days and another for 28 days. After the curing time determined by the standard, tensile tests were performed first and then compression tests on each of the specimens. A bifunctional press was used to perform these tests.

To calculate the tensile and compressive strength after the test two formulas given by the standard were used:

$$R_f = \frac{1,5 \, x \, F_f \, x \, l}{b^3} \qquad (1)$$

Being Rf the flexural strength in megapascals, b is the side of the prism's square section, in millimeters, Ff is the load applied to the center of the prism at break in newtons, and$l$ is the distance between supports in millimeters.

$$R_c = \frac{F_c}{1600} \qquad (2)$$

Where Rc is the compressive strength in megapascals, Fc is the maximum breaking load in newtons and 1600 is the area of the plates or auxiliary plates (40mm x 40mm) in square millimeters.

3. Results

For the evaluation of the results, attention is paid to the values obtained in the two most relevant stages of the experimentation (preliminary tests and resistance tests). These values are compared with each other on the grounds of cause.

3.1 Results observed in the Constitution of the Samples

The results of the initial grindings have been evaluated according to their passing percentage and their final residue. As can be seen in Figure 2, the grinds show different results under the alteration of the grinding variables shown in Table 4.

Sample #6 is the one that stands out the most for its very different values. It can be seen in Figure 2 that as the grinding design advances to obtain a lower PSD, the efficiency of this grinding is improved and the values approach the objective values.

Test #13, with a passing percentage of 77.04% and a final residue of 22.96%, is the one that shows the best results, and therefore its conditions are established as a model to follow for the following production grinds.

Table 4: Parameters according to their condition of constancy or variability in the experiment

Constant parameters	Variable parameters
Speed rotation (rpm)	Milling time
	Body load pattern
	Grinding aid
Feed mass	Pre-milling of clinker
	Pre-milling of

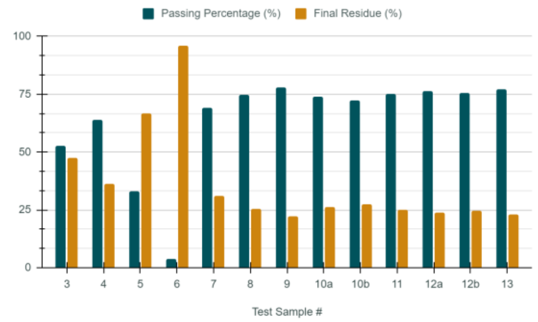

Figure 2: Comparative graph between the passing percentage and the final residue of the initial samples.

3.2 Granulometry Analysis of the Milled Product

The analysis carried out on the artificial samples in the laser granulometry was performed to have some comparison values with the values obtained in the screening for the percentage of particles -45µm. Figure 3 shows a comparison between the 3 measured values in each analysis and the average values obtained. The variation between the values obtained for this particle size was studied using the standard deviation formula.

Figure 3: Comparative graph between the values obtained in the individual measurements and the mean of these values.

3.3 Results of the Compression Tests

It was found that sample I presents a resistance much higher than the others from the first test (Figure 4), with a value of 39.25MPa after 2 days of cure, and a value of 56.31MPa in the test at 28 days. On the other side is the specimen with the worst results, A, with 30.38MPa at 2 days of curing and 41.22MPa at 28 days. The difference between these specimens is almost 9MPa in the case of the first test and more than 15MPa in the third. But both specimens follow the same behavior regardless of whether they are more or less resistant: both have proven in all tests to be the best and the worst specimen.

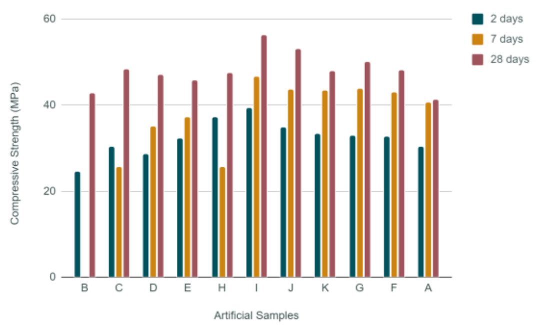

Figure 4: Compressive strength obtained in the tests with different percentages of fine particles below 45μm and different curing times.

4. Discussion

4.1 Discussion about the Constitution of the Samples

It is known that ball pattern, milling method and feed materials directly influence the PSD obtained in the product after milling.

In 2006 Binici et al. confirmed through their experiment the influence of one of these variables: the milling method used. After interpreting the data from their experimentation, they conclude that milling efficiency (greater production of fine particles) is higher in a milling method where the materials that make up the compound are ground separately and then brought together than when the materials are ground all together.

In the case of this study, a variable to take into account to achieve a lower PSD could have been the performance of tests where the clinker was ground on one side and the gypsum on the other.

Regarding the pattern of balls used, it is Ghalandari and Iranmanesh who, in 2020, carry out an experiment in which, as in this experiment, they vary the sequence of the loading of milling bodies. This action confirms that increasing the surface area of the ball pattern improves the performance of the mill and is permissible as long as this increase does not lead to a reduction in the crushing force; in the event that this parameter is reduced excessively, the efficiency of the mill would decrease.

Extrapolating this question to this study, it can be seen that the same premise is fulfilled; the most efficient ball pattern is 1, and it is also the one with the highest contact surface area (Table 5). Although the difference between the standards is small, it would have been an option to increase the difference in surface area of the standards to a greater extent to be able to see if it was a beneficial change in order to obtain the desired PSD.

Table 5: Surface area of the body load patterns.

Ball diameter (m)	Surface area per ball (m^2)	1		2		3	
		Number of balls	Surface area per ball size (m^2)	Number of balls	Surface area per ball size (m^2)	Number of balls	Surface area per ball size (m^2)
0,048	0,0072	4	0,0288	7	0,0504	5	0,036
0,04	0,005	14	0,07	17	0,085	15	0,075
0,03	0,0028	28	0,0784	16	0,0448	22	0,0616
0,025	0,002	4	0,008	7	0,014	6	0,012
0,023	0,0017	-	-	9	0,0153	15	0,0255
0,019	0,0011	19	0,0209	-	-	-	-
0,0135	0,00057	37	0,02109	11	0,00627	28	0,01596
Total surface area (m^2)			0,22719		0,21577		0,22606
Total weight (g)			9060,51		9059,7		9063
Total surface area per ton (m^2/ton)			**25,07**		**23,81**		**24,94**

4.2 Relationships found between the PSD observed in the Samples and the Compressive Strength obtained in the Tests

Ghalandari and Iranmanesh (2020) established that the component particles of cement that have a size greater than 60μm provide a "filling effect", so they do not really contribute to the development of compressive strength. On the other hand, particles smaller than 3μm "can cause higher early compressive strengths, but also can lead to some problems during the setting time, like unfavorable volume variations and decline in rheological properties".

Following the results of Ghalandari and Iranmanesh, the quantity of particles smaller and larger than 60μm present in the test tubes was analyzed. Taking a look at Table 3, it can be seen that within the group of the best specimens (G, I and J, Figure 4) and compared to the rest there is no notable difference or pattern in terms of the percentage of particles -63 + 45μm. Comparing the best result of test piece I with the worst result of test piece A (in terms of resistance to compression), it can be seen that in the particle size between -90 + 63μm, it is I together with the samples that follow it in best values, the one that have the greatest amount of particles in this size, and is A, the specimen that by far has the least amount of these particles. If we look at the following size range, -125 + 90μm, it can be seen that the same dynamics occurs.

5. Conclusions and Future Work

Bearing in mind that the objective of this experimentation is to test experimentally the relationship that the PSD of the ground cement has with the compressive strength shown by the manufactured mortar specimens, always from an energy efficiency point of view in the mixing phase, some statements are concluded with the results and their analysis established:

1. The energy efficiency of the milling process depends on several parameters; grinding time, ball loading pattern, additives for the reduction of caked material on the mill walls and particle size in the input feed. By altering these parameters, a more efficient and functional system can be achieved for a specific objective.
2. Variable time shows no significant improvement in grinding efficiency after a certain number of minutes (120 minutes). Likewise, it has been shown that the ball loading pattern that has worked best has been the one with the largest surface area (pattern 1). In addition, it has been seen in the results that a pre-grinding of the feed material has a positive influence on the PSD obtained, that is, if the feed has a smaller maximum size, the reduction of particle size in the grinding increases.
3. In the laboratory conditions followed, it has not been possible to reduce more than 77.04% of the total feed to a size smaller than 45μm, so new experiments with different variables or industrial conditions are necessary in this regard.
4. The tested specimens follow the same variation regardless of whether their compressive strength is high or low: they evolve towards greater compressive strength as the curing time passes, but they remain stable in terms of their position, that is, the specimen that has shown better results from the beginning always keeps the best and vice versa with the worst.
5. The rapidity of the increase in compressive strength with curing time, so that it is confirmed that the presence of fine particles in the cement improves the speed with which a mortar specimen increases its compressive strength.
6. The specimens that have turned out to be the most resistant to compression are those that, in proportion to all the others, have a higher percentage of particles that varies between 63 and 125μm. On the contrary, the worst result is presented by the specimen with the least percentage of particles in this range.

Several research fields are left open that could open the doors to new information regarding the subject of this study and that have not been included:

1. The influence of the size of the mill chamber on the impact force of the grinding bodies on the feed. Due to the laboratory nature of this experimentation, it has not been tested in real industrial conditions, which could modify, or even improve, the efficiency in reducing the size of the milling phase.

2. In relation to the article written by Ghalandari and Iranmanesh in 2020 that states that cement particles with a size greater than 60μm only cause a “filling effect”, it would be interesting to study the compressive strength in test tubes that are manufactured with a cement whose PSD maximum is 60μm. In this experimentation, the maximum PSD has been 125μm, following a "random" laboratory formulation (in order to simulate a non-artificial grinding) in the mixture of this size with the target size of 45μm.

Due to the good results obtained regarding the pre-milling of the feed material, an investigation is suggested on a circuit in which the pre-milling and milling are put sequentially.

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