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Quality Control of Hydrated Limes Marketed in the Metropolitan Region of Recife, Pernambuco, Brazil: A Case Study

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ABSTRACT

In the midst of an extensive and diversified lime market, it is a great challenge to choose a product that follows the standards to meet the buyer's need. The lack of control and quality of a lime is reflected in its use. The interest in its hydration and its retention power are factors linked to the ideal condition of a good lime. This study aimed to evaluate the quality of commercialized hydrated limes, aiming at the improvement and durability of the product, as well as safety for the consumer. It was determined in eight different samples of lime, the unit specific mass, water retention, fineness, silica (SiO2) plus insoluble solids, iron and aluminum oxides and humidity, which served to prove the lack of quality in some samples, specifically C2, which were out of bounds established by the existing Brazilian Technical Standards for limes.

Keywords: *Mortars, Hydrated lime, Quality control.*

INTRODUCTION

According to NBR 7175 [1] hydrated lime is a dry powder obtained from the hydration of quicklime, consisting essentially of calcium hydroxide and/or magnesium hydroxide. Virgin lime, according to NBR 6453 [2], is a product obtained from the calcination of calcium and magnesium carbonates, or even a mixture of calcium oxide, magnesium oxide and calcium hydroxide. Hydrated lime is used, among other applications, in the steel, paper, construction industries and in environmental control [3].

Aiming at the products to be obtained from the limestone, it is necessary, in a first stage, to carry out physical-chemical analysis in order to characterize it according to its constitution. The various brands of lime in the trade are controlled through the quality seal provided by the Brazilian Association of Lime Producers [4].

Based on the characterization and use, it is necessary to evaluate which processes can be used for the production of hydrated lime. For the production of lime, there are several types of furnaces, technologies, and fuels, although the chemical process is the same [5].

The lime manufacturing process, regardless of the type of oven used, consists of the following steps, according to[6]: Extraction of raw material and crushing; Selection of the optimum size range and transport to the oven; Calcination and its control; Grinding suitable for the type of hydrator; Hydration and grinding; Bagging and distribution for commercialization.

According to[7] the calcination process is the thermal decomposition reaction of calcium carbonate to form calcium oxide, as shown in Equation 1.

$$CaCO_3 \rightarrow CaO + CO_2 \qquad \text{(Eq. 1)}$$

The main factors that influence the quality of virgin lime are related to temperature and residence time in the calcination oven.

From the stoichiometric point of view, hydrated lime, considering only the formation of calcium hydroxide from calcium oxide, is formed from Equation 2.

$$CaO + H_2O \rightarrow Ca(OH)_2 \qquad \text{(Eq. 2)}$$

According to Eq.2, to hydrate 1 mol of calcium oxide (56.08 g/mol), 18.016 g/mol of water are needed to produce 74.096 g/mol of calcium hydroxide, a ratio of 321.26 liters of water per

ton of oxide of calcium. A significant property of this reaction is the release of 15,288.0 kcal/kgmol of energy to produce the stoichio metric amount of calcium hydroxide. This amount of energy generated will increase the reaction temperature above 100ºC, unless excess water is added [8].

In relation to the virgin lime hydration process, there are basically two methods: dry hydration of calcium oxide or production of a hydrated lime suspension, commonly called lime milk. The method to be chosen will depend on the desired surface area and its application [9, 10].

Hydrated lime must comply with NBR 7175 [1]. This standard specifies the requirements for receiving hydrated lime to be used in mortars for civil construction. The values established by the Brazilian Association of Technical Standards are maximum and minimum and include physical and chemical characteristics, as shown in Chart 1.

Chart1. *Chemical and physical characteristics of limes*

Chemical Characteristics				
Compounds		**Limits**		
		CH-I	CH-II	CH-III
Carbonic anhydride (CO_2)	In the factory	≤ 5%	≤ 5%	≤ 13%
	In the warehouse	≤ 7%	≤ 7%	≤ 15%
Calculated unhydrated calcium and magnesium oxide (CaO + MgO)		≤ 10%	≤ 15%	≤ 15%
Total oxides in the non-volatile base (CaO_T + MgO_T)		≥ 90%	≥ 88%	≥ 88%
Physical Characteristics				
Fineness (cumulative percent retained)	(F_{30})Sieve0.600mm	≤ 0.5%	≤ 0.5%	≤ 0.5%
	(F_{200}) Sieve 0.075mm	≤ 10%	≤ 15%	≤ 15%
Water retention		≥ 75%	≥ 75%	≥ 70%
Incorporation of sand		≥ 3.0%	≥ 2.5%	≥ 2.2%
Stability		Absence of cavities or protuberances		
Plasticity		≥ 110	≥ 110	≥ 110

CH-I, CH-II, CH-III = types of limes.

***Source**: NBR 6473 [11].*

Thus, this study aimed to evaluate the quality of hydrated lime used in commerce, aiming at the improvement and durability of the product and consumer safety, according to the existing Brazilian Standards for the product.

MATERIALS AND METHODS

The experiment was installed at the Analytical Chemistry Laboratory, 8thfloor of block D of the Science and Technology Center at Catholic University of Pernambuco, Recife, Pernambuco, Brazil.

Eight samples of lime were purchased, from different manufacturers in the Metropolitan Region of Recife, whose brands correspond to the most sought after in the trade, identified as C1, C2, C3, C4, C5, C6, C7 and C8.

Due to the great variations in the quality of the limes and the quality control for the sale, the determinations indicated by NBR 6473[11] and modified by [12] were carried out, with three repetitions: unit specific mass, to verify the specific mass of the limes; water retention, an important property of mortar; fineness, to indicate the amount of lime retained in the 0.600mm and 0.075mm sieves; silica (SiO_2) + insoluble solids, to quantify impurities in lime; amount of iron and aluminum oxides, to identify the present amount of both oxides; and humidity, to indicate the amount of water incorporated in the sample.

Unit Specific Mass

To predict the incorporation of water by lime, the following procedure was proposed: 50.0g of each lime sample was transferred to a beaker with a capacity of 100.0ml and the volumes occupied by the samplesC1, C2, C3, C4, C5, C6, C7 and C8 were determined. Equation 3 calculated the results of the unit specific mass.

Unit specific mass (kg / m^3) = (M / V) * 1000(Eq. 3)

Where, M =Initial sample mass (g);V =Cylinder volume (ml); 1000 = unit transformation factor.

Water Retention

Using a non-standardized method, 50.0 g of each sample of lime were weighed and transferred to a beaker with a capacity of 250.0 ml. 100.0 g of distilled water was added and homogenized with a glass stick. The volume (zero time) of the lime samples C1, C2, C3, C4, C5, C6, C7 and C8 was verified and, after that, it was left to rest for 150 minutes, determining the volumes not decanted, as a result of rest time presented by [12]. Equation 4 calculated the results of water retention.

Water retention (%) = [(A – 125) / (B – 125)] * 100 (Eq. 4)

Where, A =Initial sample mass (g); B = Cylinder volume (ml); 125 = Initial total volume (ml).

Fineness

For the determination of fineness, the NBR 9289 procedure [13] was used, namely: in two coupled sieves [one 0.600mm (F_{30}) and the other 0.075mm (F_{200})], 50.0g of each sample of lime C1, C2, C3, C4, C5, C6, C7 and C8 was added washing with distilled water jets. Soon after, the residues were transferred to porcelain crucibles already tared (P1) and dried in an oven at 105°C, until constant weight (P2). Equation 5 calculates fineness.

Fineness (%) = (P2 – P1) * 100 (Eq.5)

Where, P1 =Empty crucible weight (g);P2 =Crucible weight+lime, after oven (g).

Silica (SiO2) + Insoluble Solids

To determine the amount of waste that is not lime and that is mixed to provide volume to the product, reducing its quality, the NBR 6473 [11] standards were followed. 0.7g (M1) of each sample of lime C1, C2, C3, C4, C5, C6, C7 and C8 was weighed and transferred to a beaker with a capacity of 250.0ml.10.0ml of perchloric acid was slowly moistened and added, stirred slowly and covered with watch glass. It was heated on a hot plate until white lumps were released, for approximately 10 minutes. It was cooled to room temperature. The watch glass was washed with 50.0 ml of hot water, collecting the washing water in the beaker, and 10.0 ml of the 50% hydrochloric acid solution was added. The set was again heated on the hot plate, with stirring, for two minutes. It was hot filtered by collecting the filtrate in a 250.0ml volumetric flask with 10 drops of concentrated nitric acid, until all silica was removed, and all chloride was eliminated (silver nitrate test).The volumetric flask was checked and reserved. The filter paper containing the silica and insoluble solids was transferred to a previously weighed porcelain crucible (M2).It was calcined in a muffle furnace at 1,000°C for 45 minutes and weighed (M3). Equation 6 calculated the content of silica + insoluble solids.

Silica + insoluble solids (%) = [(M3 – M2) / (M1)] * 100 (Eq. 6)

Where, M1 = Initial sample mass (g); M2 = Empty crucible weight (g); M3 = Crucible weight with residue after muffle (g).

Iron and Aluminum Oxides

To determine the content of iron and aluminum oxides in each sample of lime C1, C2, C3, C4, C5, C6, C7 and C8, the NBR 6473 [11] standard was used. 50.0ml of the reserved filtrate was removed from the determination of silica + insoluble solids and transferred to a beaker with a capacity of 250.0ml.The pH was adjusted to 2 to 2.5 with 50% ammonium acetate solution. Three drops of 5% sulfosalicylic acid solution were added and the iron was titrated with standardized 0.025 M EDTA solution until turning from brown to pale yellow or colorless (V1).After titrating the iron, 5.0 ml of 0.025 M EDTA was added, boiled for 5 minutes, cooled and 1.5 g of sodium acetate was added. The pH was adjusted to 5.5 using drops of 3N NaOH solution or drops of 1 + 3 HCl solution. The orange xylenol indicator was added, and the aluminum was titrated with a standardized Zinc Sulphate solution 0.005 M until turning from orange to reddish violet (V2).

The content of iron and aluminum oxide (R_2O_3) was calculated according to Equation 7.

R_2O_3 (%) = [(V2 – V1) / (M1)* f] * 100 (Eq. 7)

Where, M1 = Initial mass of the sample (g); V1 = Volume spent for Iron (ml); V2 = Volume spent for Aluminum (ml); f = iron and aluminum transformation factor for FeO and AlO.

Humidity

The percentage of moisture in the lime samples C1, C2, C3, C4, C5, C6, C7 and C8 is related to the amount of water available in it. It is possible

to determine the amount of water that has been removed or added to the limes when it is known about its initial humidity and the new (end) humidity, after changing its state.

To determine the humidity, NBR 6473 [11] was used with the following procedure: a porcelain crucible preheated in an oven at 105°C was weighed until constant weight (M2).1g of lime samples (M1) was added and the set was taken to the oven at 105°C until constant weight (M3). The humidity percentage was determined using Equation 8.

Humidity (%) = [(M2 – M3) / (M1)] * 100 (Eq. 8)

Where, M1 = Initial mass of the sample (g); M2 = Empty crucible weight (g); M3 = Weight of the crucible with sample, after oven (g).

RESULTS AND DISCUSSION

The results obtained for the unit specific mass, water retention, fineness, silica (SiO_2) + insoluble solids, iron and aluminum oxides and moisture in the lime samples C1, C2, C3, C4, C5, C6, C7 and C8 are in Chart 2, as well as the values allowed according to the NBR 7175 [1].

Chart 2. *Results of the determinations made on the samples of lime*

Determination	Unit	Lime samples								Limits[1]		
		C1	C2	C3	C4	C5	C6	C7	C8	CH-I	CH-II	CH-III
Specific mass	kg /m^3	957	1064	1020	962	524	926	1030.9	515	-	-	-
Water retention	%	88	96	106	104	120	100	105	103	≥ 75%	≥ 75%	≥ 70%
Fineness (F_{30})	%	4.36	2.55	4.00	0.28	0.01	0.01	18.68	0.02	≤ 0.5%	≤ 0.5%	≤ 0.5%
Fineness (F_{200})	%	6.54	11.92	9.24	0.28	0.01	0.01	3.79	0.01	≤ 10%	≤ 15%	≤ 15%
Silica (SIO_2) + insoluble solids	%	5.60	44.0	86.20	9.40	3.40	5.40	87.40	3.4	-	-	-
Ironand aluminum oxides	%	1.80	4.40	2.40	5.80	24.00	12.80	1.38	0.38	-	-	-
Humidity	%	0.46	5.72	3.12	0.70	0.74	0.52	1.38	0.38	-	-	-

Where: *[1]according to ABNT NBR 7175 [1];(F_{30}) = 0.600mm aperture sieve; (F_{200}) = 0.075mm aperture sieve.*

After determining the unit specific mass, it can be seen from Chart 2 that the samples of limes presented different volumes for the same mass (50.0g), namely: C1 (52.25ml), C2 (46.99ml), C3 (49.02ml), C4 (51.97ml), C5 (95.42ml), C6 (53.99ml), C7 48.50ml) and C8 (97.09ml), being in sample C2 (46.99ml) the smallest volume and in sample C8 (97.09ml) the largest volume, albeit with values lower than the volumes found in the experiments by[12].

According to Chart1, plasticity (≥ 110), which is an indication of good quality lime, favored the C5 sample (120%) over the other lime samples. In determining water retention (Chart 2), the lowest quality were samples C1 (88%) and C2 (96%), although all samples of limes (C1, C2, C3, C4, C5, C6, C7 and C8) presented values higher than those stipulated by NBR 7175[1].

By Chart 2, samples C4, C5, C6 and C8 presented results within the NBR 7175 standards [1] for Fineness (F_{30}) and also for Fineness (F_{200}), indicating adequate degree of purity. Samples C1, C2, C3 and C7 presented values according to NBR 7175 [1] only for Fineness (F_{200}) and among them, C2 (11.92%) presented a value that did not fit the CH-I limit (≤ 10%).

Analyzing the values for Silica + Insoluble Solids in Chart2, it is possible to notice the lack of quality control between the limes, due to the high variation of results between them, indicating a reduction in the quality of the lime and its useful life, especially in the C2 samples (44.0%), C3 (86.20%) and C7 (87.40%), which do not obey the legislation to be sold.

It can be seen in Chart 2 that the levels of iron and aluminum oxides, with the exception of samples C5 (24.00%) and C6 (12.80%), are acceptable, even with variations. Because, within the limits, the amount of iron oxide in the limestone samples improves viscosity.

The humidity among the lime samples (Chart 2) showed very varied values due to the impurities present in the samples. The greater the result, the greater the amount of lime impurity; therefore, samples C2 (5.72%), C3 (3.12%) and C7 (1.38) are considered less pure than samples C1, C4, C5, C6 and C8.

CONCLUSION

The determinations carried out on the eight samples of limes acquired as representative of trade in the Metropolitan Region of Recife, Pernambuco, proved the lack of quality in some

of them, specifically C2, which were far from the limits established by the Brazilian Technical Standards for the product. Therefore, it is necessary that they be reformulated in their production process so that they conform to the Standards, benefiting the functionality and safety of consumers.

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