Analysis of water absorption in industrial waste of superabsorbent polymer and cellulosic fiber

Análise da absorção de água em resíduo industrial de polímero superabsorvente e fibra celulósica

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ABSTRACT

Proper curing of concrete is one of the most important processes in the post-concreting period so that concrete can achieve the desired mechanical strength and durability. However, part of the construction works perform this procedure for inadequate periods or often do not perform it; as a result, surface characteristics are commonly affected, resulting in increased porosity and, consequently, increased permeability, cracking and carbonation. Materials that have water retention capacity and subsequent gradual release may be effective as internal curing agents. Superabsorbent polymers (PSA) are already used and widely studied for this purpose, but usually with commercial origin. The objective of this research is to evaluate different water absorption methods in industrial waste of superabsorbent polymer and cellulosic fiber (PSAR) for later use as an internal curing agent in Portland cement matrices. PSAR was homogenized by a process at two different times. The material in its natural state and homogenized had its absorption capacity evaluated using two methods: ISO 17190:2001 and sieve method. For the sieve method, the influence of hydration time on absorption was analyzed. The ISO 17190:2001 method did not show representativeness in the results due to the high absorption variation. The sieve method was more representative and demonstrated efficiency in the homogenization processes, reducing the coefficient of variation by up to 43%. The material with the highest potential for use was PSAR60, with an absorption value of 68.26 g of water per g of PSAR, since the absorption test showed the highest homogeneity and, from this absorption, no longer had significant variations.

Keywords: Superabsorbent polymer; Internal curing; Absorption; Cellulosic fiber; By product.

1. INTRODUCTION

Population growth has several consequences, such as the need for infrastructure that can meet the needs of population. Whereas in the twentieth century alone there has been a population increase from 1.5 billion to 6 billion people in the world, with a projection of 9.8 billion in 2050, and the main constituent of infrastructure manufacturing is Portland cement, there is a projected increase in cement production between 1990 and 2050 of over 500%. Concrete is the most widely used material in construction: only in 2010, there was a worldwide consumption of approximately 33 billion tons [1, 2]. In the last decades, there was a great advance in construction technologies, with new materials and methods emerging to improve structures and accelerate execution processes. However, the development of techniques that aim to mitigate the occurrence of pathological manifestations that may compromise the useful life of the structure is slowing down in the current demand [3].

Proper curing of concrete is one of the most important processes in the post-concreting period so that concrete can achieve the desired mechanical strength and durability. This procedure basically consists in providing enough moisture for cement hydration, avoiding the evaporation of water necessary for hydration reactions, allowing the material to develop the right properties [3]. However, part of the works performs this procedure for inadequate periods or often do not perform it; therefore, surface characteristics are commonly affected, resulting in increased porosity and, consequently, increasing permeability, cracking and carbonation [4]. According to MEHTA and MONTEIRO [5], without proper curing and keeping the concrete in a dry environment, the compressive strength can decrease by up to 50% at 28 days when compared to the same concrete cured under ideal conditions, according to Figure 1. In order to combat these problems caused by improper curing of concrete, several authors have been studying the use of promising materials as internal curing agents: materials with water retention capacity that, when incorporated into the cementitious matrix, release water and promote hydration from the inside out.

The superabsorbent polymers (PSAs) were developed in the early 1980s, when their first use was in the personal hygiene industry, specifically in disposable diapers, which are still their main application. In contact with water or some kind of aqueous solution, PSA begins to absorb the fluid and becomes a gel. It is absorbed by osmotic pressure [6]. According to FRIEDRICH [7], the fundamental force for the superabsorbent polymer to retain large amounts of water is the osmotic pressure proportional to the concentration of ions in the solution, decreasing this internal force as water absorption occurs and the charge is diluted. These materials are found in two forms on the market, most of which are intended for the toiletries (diaper and pad manufacturing) industry based on cross-linked polyacrylate acids, and those called technical PSAs (landscaping, cable insulation, firefighting and packaging food products) based on acrylamine and acrylic acid [6]. The main difference between them is the way that water is released to the outside, which in toiletries occurs only in higher pH, and in the practice of landscaping more quickly by soil pH [3]. Its absorption depends mainly on the properties of PSA, as well as external factors such as temperature, pressure and environment where it is inserted, and can absorb up to 1500 times its mass weight, as shown in Figure 2 [8]. However, in a cementitious environment, this absorption is reduced to values up to 20 times its weight, a high value when compared to other materials used as internal curing agents [9].

The first use of superabsorbent polymers in the construction sector as an internal water incorporating agent in cementitious materials, focusing on the combat of drying shrinkage, was proposed by JENSEN and HANSEN [10]. With the potential presented by the authors and the growing interest in the use of PSA as an additive for cementitious materials, a "TC SAP-225" technical committee was created to study its use, limitations and solutions to problems identified in the application of this material [6].

Among the studies found, the use of PSA occurred in its commercial form. The first work proposing the use of PSA in its residual form (PSAR) was done by GOMES [11], where the author presents a characterization of this residue from the diaper and pad industry. The second author to present the use of this material was KOPPE [3], where its use was made in mortars as an internal curing agent, obtaining promising results regarding
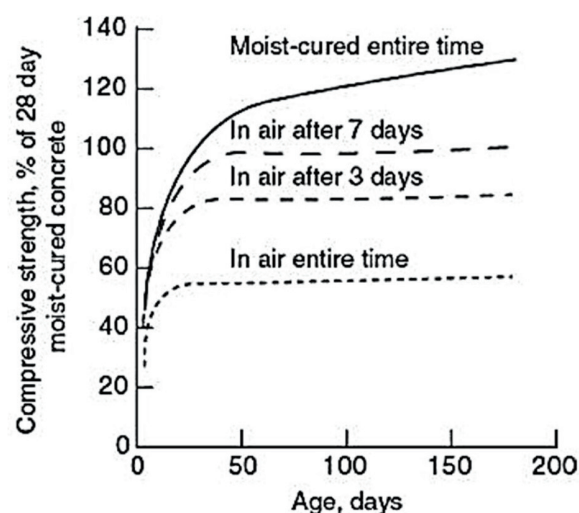

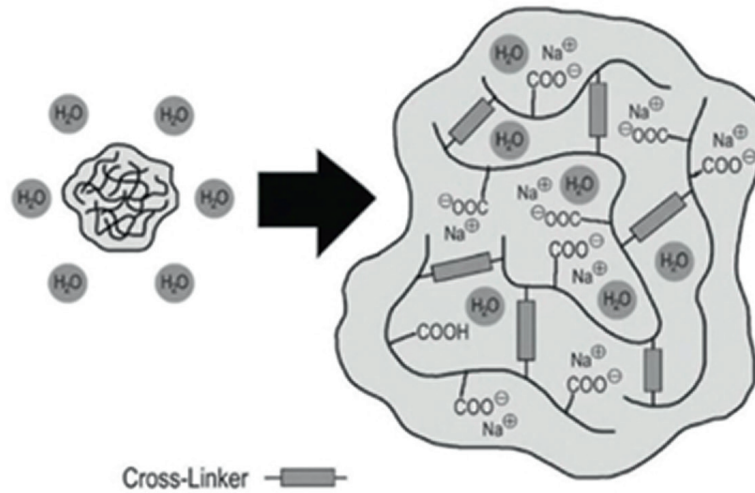**Figure 1:** Resistance variation depending on the cure.

Figure 2: PSA particle based on acid polycrylate, dry and swollen after water absorption.

its use in combating shrinkage and cracking, also contributing to the increase of resistance at older ages. However, no studies were found regarding the use of PSA in its residual form in Portland cement concretes.

For the complete hydration of cement and its compounds, a water / cement ratio of about 0.4 is required. Water not used in chemical reactions remains inside the concrete and forms voids. This water that is not absorbed or that has not been chemically combined will serve as a way for the loading of harmful substances to the structure, directly affecting its useful life. So, for better durability of structures, it is necessary to use lower w / c ratios. However, this is not always possible due to workability issues of this material, using plasticizers or superplasticizers additives. Thus, it is possible to reduce the voids in the hydrated paste as well as the path to these deleterious compounds, directly implying greater durability and strength of the concrete [12].

The authors BENTZ *et al.* [13] proposed a method to calculate the amount of material to provide an internal water reserve in the Portland cement matrix for use as an internal curing agent. The method relates cement consumption, chemical shrinkage, absorption rate of internal curing agent and degree of saturation of internal curing agent to determine the amount of absorbent material to be employed for later, when there is no more free water in the mixture, the internal "reservoir" will hydrate the undrained cement particles.

$$M_{LWA} = \frac{Cf x CS x \alpha \max}{S x \phi_{LWA}} \tag{1}$$

Where:

M_{LWA} - material required for internal cure (kg/m^3)

Cf - cement consumption (kg/m^3)

CS - chemical shrinkage (g/water/g)

αmax - degree of hydration of the internal curing agent

S - degree of saturation of internal curing agent

Φ_{LWA} - absorption of internal curing agent (kg)

It is possible to observe that the absorption of the internal curing agent is fundamentally important for the precise use of the material. If the absorption value used in the calculation is higher than the actual value, more water will be inserted than the necessary to promote internal cure, with a higher amount of kneading water and possibly impairing the durability of the originated matrix. If less than the actual value is used, it will be inserting too much solid material, and the PSA will absorb part of the initial water of the mixture, impairing the workability, which may cause difficulty in filling the voids in the concreting, and may also lead to decreased useful life.

Based on the above, the objective of this research is to evaluate different methods of absorbing superabsorbent polymer and cellulosic fiber industrial waste for later use as an internal curing agent in Portland cement matrices.

2. MATERIALS AND METHODS

2.1. Materials

The material used in this research is a waste generated in a personal hygiene company, composed of disposable diapers and pads, resulting from waste generated in quality control and errors during the company's production process, where the material is collected and goes through a process of separation and sorting. The plastic is separated from the residual superabsorbent polymer and cellulosic fiber (PSAR) and recycled, and the cellulosic fiber containing the PSAR is pressed and stored in a moisture free place. This entire process of deconstructing the waste is done by a company in the Vale do Caí region, and it is estimated that this recycling company receives a volume of 40 to 50 tons per month [11].

PSAR when dry looks a lot like cotton (Figure 3a); however, it is mainly composed of cellulose fibers. When water is added to the material, PSAR absorbs the solution and resembles a gel (Figure 3b), increasing its volume, so-called hydrogel material [3].

2.2. Methods

2.2.1. Homogenization and mechanical dispersion

To grind and homogenize the material, a horizontal ceramic ball mill with alumina balls was used, with 537 g of 40 mm diameter balls, 217.90 g of 20 mm balls and 511.98 g of 10 mm balls, with a can volume of 7,5 dm^3, a rotation speed of 50 rpm and 150 g of dry PSAR. Two grind times were applied: 15 min (PSAR15) and 60 min (PSAR60).

2.2.2. Water absorption

In order to obtain PSAR water absorption, two methods were used, one adapted and one empirical: ISO 17190-6:2001 [14] and the sieve method.

2.2.2.1. Adapted method ISO 17190:2001

The method according to ISO 17190-6:2001 determines the absorption of water from polymers in a solution that simulates urine for the personal care industry, but in the research, it was used water from the supply network (SEMAE) instead of solution with the normative pH. The procedures have been adapted to follow the standards. The centrifuge equipment used for the test was a centrifuge of the brand HANIL LTDA, model Combi – 514R.

For the test, mate pump filters of approximately 5 cm diameter each were used. To increase their resistance to centrifugal force and not risk tearing, they were re-forced with a seam line so that the original would not sag, resulting in material loss. Initially, the filters were weighed, having the dry mass of each reference filter (m_{b1} and m_{b2}) which served as tare, as well as the other two filters to which 0.2 g of PSAR was added. Then, the sachets were closed and immersed in solution for 30 min. After the standard hydration time described, the filters were removed from the Beaker and placed in test tubes so that trapped water would remain inside the tube, so as not to damage the centrifuge and facilitate the test. The bags were arranged so that they were suspended within the test tube, so that the excess water after the test was left in the bottom, separated from the PSAR, being careful
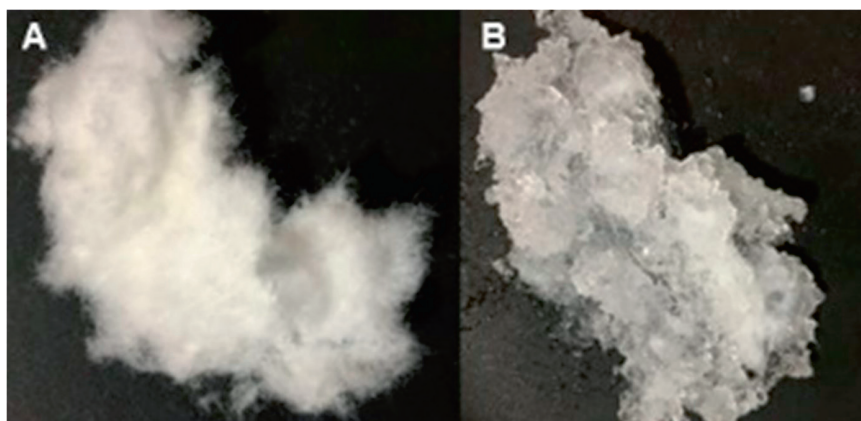

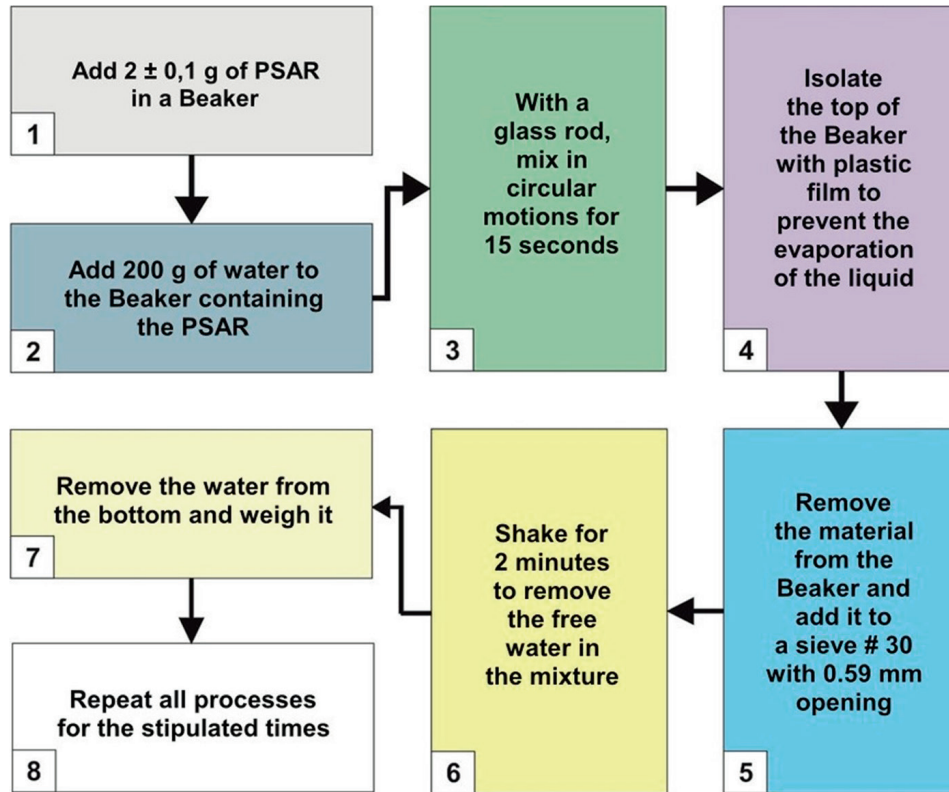**Figure 3:** Dry PSAR (a) and hydrated PSAR (b).

Figure 4: Sieving absorption process steps.

not to touch the excess water. The test tubes were arranged in pairs of the same mass so that centrifugal desta-bilization did not occur. The rotational force used was the same as described in the standard: 250× the force of gravity. The centrifuge was then set to gradually increase its rotation for 5 seconds until it reached the stipulated force until the end of the test at 3 minutes. Equation 2 is used to obtain material absorption (wi).

$$wi = \frac{\left(m_{wi} - \left(\frac{m_{b1} + m_{b2}}{2} \right) \right) - m_{si}}{m_{si}} \qquad (2)$$

The variables m_{wi} and m_{si} refer, respectively, to the mass of the tea bag containing the hydrated PSAR after 3 minutes under the action of centrifugal force and the initial dry sample of 0.2 g PSAR; m_{b1} and m_{b2} are the samples used as a reference for PSAR-free filters, as a reference for absorbing tea bags.

2.2.2.2. Sieve method

The empirical method of sieving absorption is the removal of free water after polymer saturation. The polymer was hydrated at time intervals of 1, 2, 3, 4, 5, 10, 15, 20, 25, 30, 60, 120 and 1440 minutes, with enough water to saturate it. Five trials were performed for each time analyzed. 2 ± 0.1 g PSAR was placed in 250 ml volume beaker, added 200 g water and mixed by glass rod for 15 seconds. Then, it was kept in a room of controlled temperature and humidity (23ºC and 60% U.R.), isolating the top with plastic film to prevent the evaporation of the liquid. After hydration time, the set was subjected to sieve # 30 with 0.59 mm opening and shaken for 2 minutes with a metal spoon to remove free water in the mixture. Figure 4 presents the steps of the sieving absorption process.

Excess water was collected and weighed. The absorption was obtained through Equation 3.

$$Abs = \frac{200 - passing\ water\ (g)}{PSAR\ (g)} \qquad (3)$$

3. ANALYSIS AND DISCUSSION OF RESULTS

3.1. Homogenization and mechanical dispersion

The homogenization process was evaluated at two grind times: 15 and 60 minutes. The times were arbitrated so that in the first half there was little energy employed in the beneficiation process, while in the second half one sought to evaluate potential modifications.

In Figure 5 it is possible to see the difference of the materials after going through the homogenization process, where the PSAR nat (A) has an appearance of a larger fiber conglomerate, resembling a cotton, formed by larger compacted parts. After 15 minutes of homogenization, the material was decompressed according to the mill's use time.

As expected, the 60-minute time had a greater impact on material homogeneity, where larger cellulosic fiber and PSA conglomerates became significantly shorter. By unpacking the material, the surface area may have been increased; in this way, water percolates and surrounds all the material, while in the natural PSAR the water moisturizes the often-compacted surface layers, so that all the material can be hydrated. Reaction velocity is known to be closely linked to the surface area, where a larger contact area leads to higher hydration velocities [15].

3.2. Absorption

3.2.1. Adapted method ISO 17190:2001

Due to irregularities in the masses of the tea bags, it was adopted the average absorption as reference for the absorption calculation to reduce the final absorption of the PSAR. The results are presented in Tables 1 and 2.

It can be seen that the amount (0.2 g) proposed by ISO 17190-6:2001 for the test does not represent the whole material, as the amount of PSAR added into the tea bag may vary between the proportions of cellulosic fiber and PSA, leading to a high disparity between the absorption values found - it is observed by the high values of the coefficient of variation, where in PSAR nat it reached 76.56%, as well as in PSAR15 to 16.34% and PSAR60 to 41.26%, making it difficult to use these results.
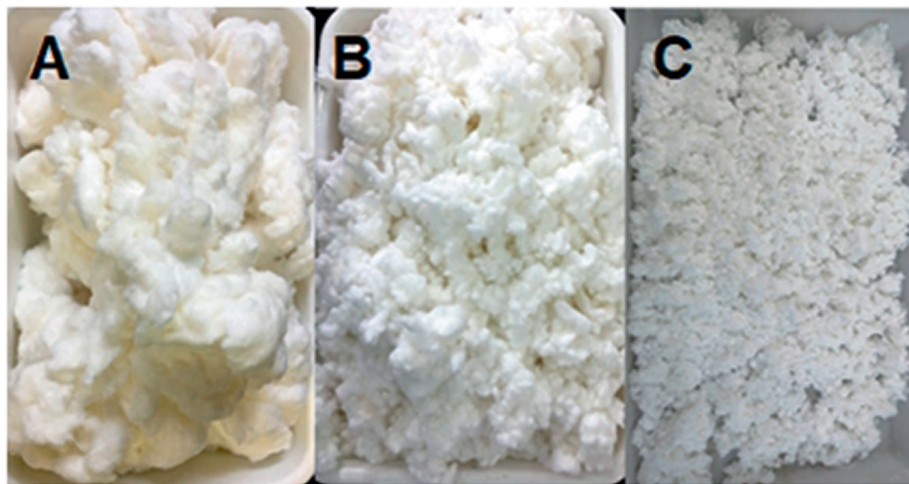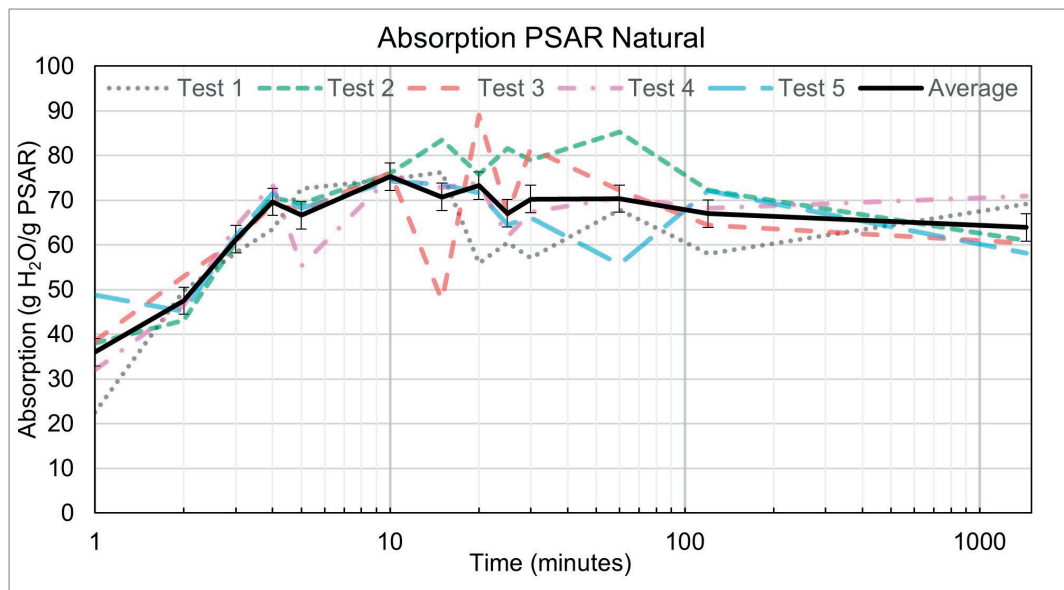

Figure 5: PSAR nat (A), PSAR15 (B) and PSAR60 (C) after homogenization.

Table 1: Absorption of reference tea bags.

		[DRY] BAG (g)	[HYDRATED] BAG (g)	ABSORPTION (g)	AVERAGE (g)
PSAR nat	**Ref 1**	0.402	0.477	0.075	0.064
	Ref 2	0.397	0.450	0.053	
PSAR15	**Ref 1**	0.414	0.452	0.038	0.060
	Ref 2	0.399	0.481	0.082	
PSAR60	**Ref 1**	0.406	0.458	0.052	0.063
	Ref 2	0.397	0.472	0.075	

Table 2: Absorption test adapted to ISO 17190-6:2001.

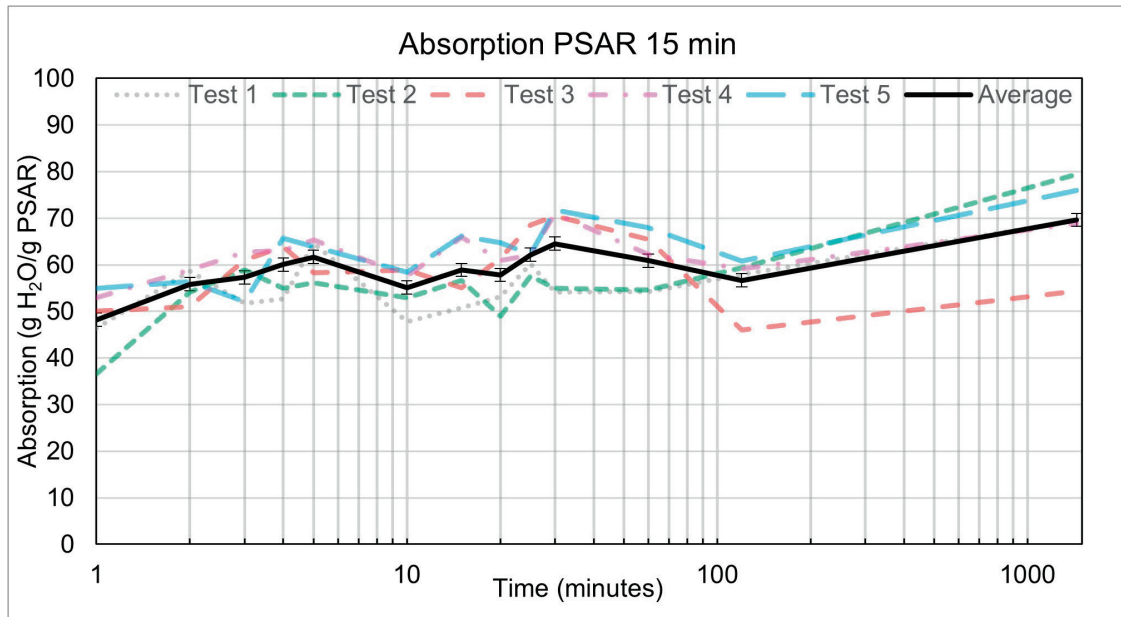|		[DRY] BAG (g)	PSAR (g)	[HYDRATED] BAG + PSAR (g)	ABSORPTION (g/g PSAR)	AVERAGE (g/g PSAR)	STANDARD DEVIATION (g/g PSAR)	COEFFICIENT OF VARIATION
PSAR nat	1	0.432	0.2	10.316	49.36			
	2	0.460	0.2	10.526	50.27			
	3	0.407	0.2	1.944	7.62	33.09	25.33	76.56%
	4	0.397	0.2	1.077	3.34			
	5	0.428	0.2	11.411	54.85			
PSAR15	1	0.448	0.2	8.251	38.95			
	2	0.463	0.2	7.920	37.22			
	3	0.398	0.2	8.075	38.32	35.63	5.82	16.34%
	4	0.461	0.2	5.528	25.27			
	5	0.436	0.2	8.120	38.36			
PSAR60	1	0.423	0.2	5.337	24.51			
	2	0.443	0.2	6.060	28.02			
	3	0.409	0.2	8.134	38.56	24.41	10.07	41.26%
	4	0.443	0.2	4.354	19.49			
	5	0.393	0.2	2.695	11.45			

Graph 1: Natural PSAR absorption by sieve method.

Based on the coefficients of variation found, only with this method, it is not possible to confirm that the homogenization process was effective, because the small amount of material used for the test (0.2 g) may not have been able to contain concentration variations of cellulosic fiber and superabsorbent polymer that are representative to the batch.

3.2.2. Sieve method

The sieve method was used because the centrifuge method proved impractical to perform, given the boundaries imposed by the ISO 17190-6:2001 method and the amount of residue tested, that may not have been enough to represent the whole residue. Furthermore, the method proposed by ISO 17190-6:2001 is restricted to composite material exclusively of superabsorbent polymers, not including composite materials, which is the case of the PSAR under analysis.

Another factor that implied the choice of a new method for the absorption test was to facilitate its execution by the industry, being possible to work with composite materials and not only the PSA in its pure form, applying a larger volume of material in the method and increasing the reliability of the results.
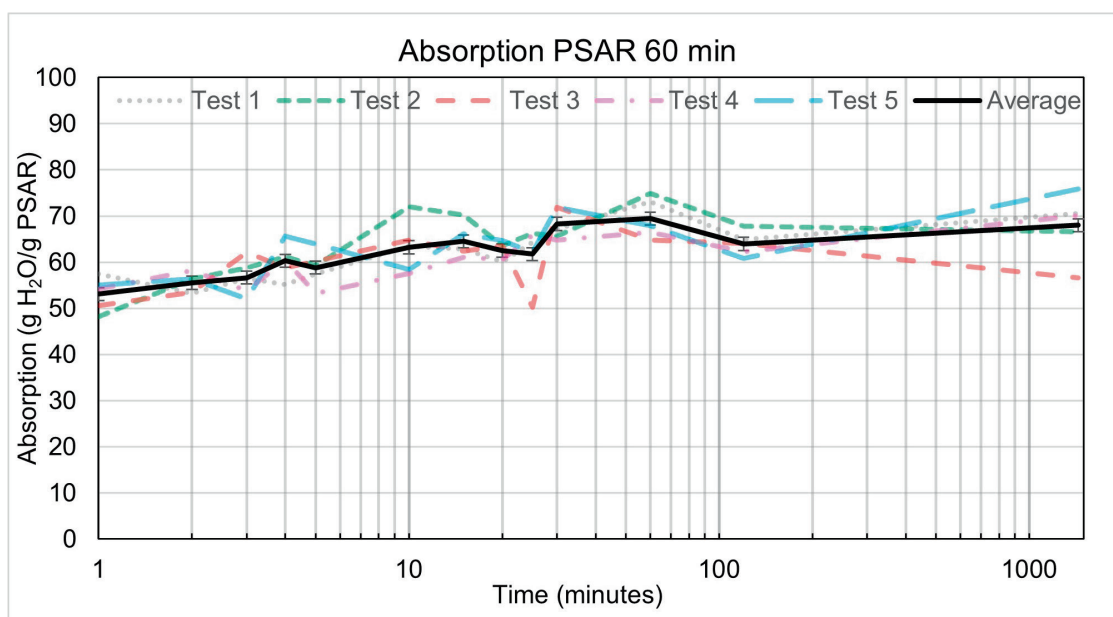
Graph 2: PSAR15 absorption by sieve method.

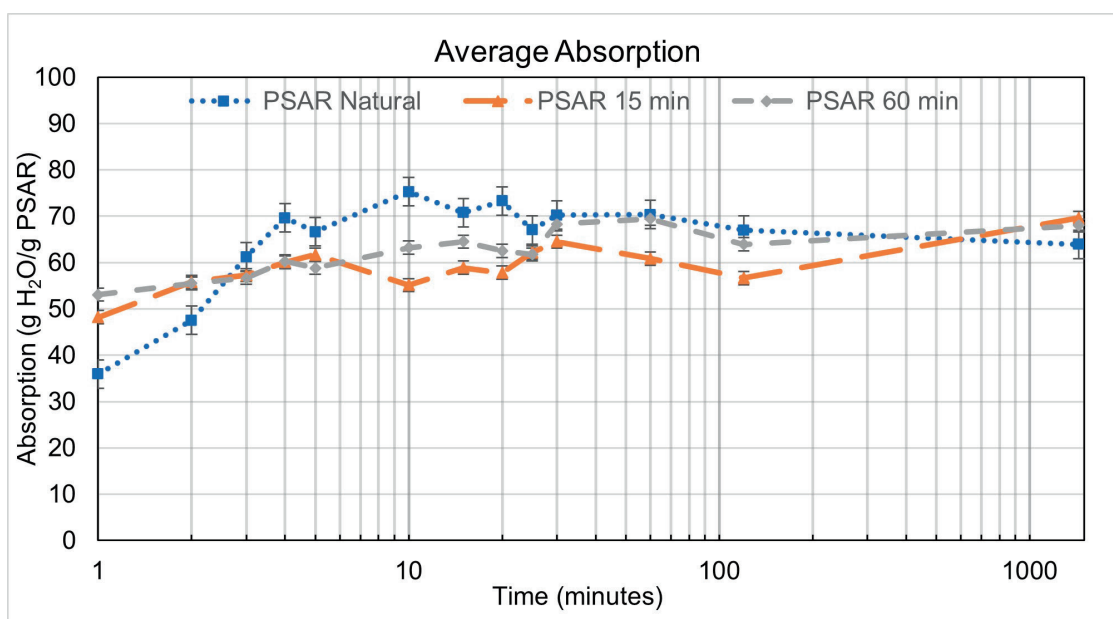**Table 3:** Absorption by sieve method.

TIME (MIN)	AVERAGE ABSORPTION (g/g)			STANDARD DEVIATION (g/g)			COEFFICIENT OF VARIATION		
	PSAR	PSAR15	PSAR60	PSAR	PSAR15	PSAR60	PSAR	PSAR15	PSAR60
1	35.94	48.17	53.06	8.60	6.51	3.29	23.9%	13.5%	6.2%
2	47.49	55.79	55.50	3.48	2.91	1.92	7.3%	5.2%	3.5%
3	61.23	57.27	56.65	1.90	4.55	3.58	3.1%	7.9%	6.3%
4	69.61	60.04	60.27	3.38	5.21	3.40	4.9%	8.7%	5.6%
5	66.61	61.65	58.80	5.89	3.71	3.50	8.8%	6.0%	5.9%
10	75.27	55.11	63.20	0.75	4.24	5.20	1.0%	7.7%	8.2%
15	70.73	58.88	64.50	12.18	6.12	3.30	17.2%	10.4%	5.1%
20	73.25	57.80	62.49	10.58	5.83	1.81	14.4%	10.1%	2.9%
25	67.04	62.13	61.75	7.60	3.64	5.96	11.3%	5.9%	9.7%
30	70.23	64.49	68.26	8.97	8.15	3.04	12.8%	12.6%	4.4%
60	70.32	60.85	69.41	9.46	5.60	3.88	13.4%	9.2%	5.6%
120	66.95	56.62	63.93	5.34	5.42	2.38	8.0%	9.6%	3.7%
1440	63.89	69.61	67.97	5.10	8.60	6.40	8.0%	12.3%	9.4%
Average	64.50	59.11	61.98	6.40	5.42	3.66	10.3%	9.2%	5.9%

Results were evaluated in the three forms of PSAR: natural (Graph 1), PSAR15 (Graph 2) and PSAR60 (Graph 3). Table 3 presents the average absorption, standard deviation and coefficient of variation, with illustration of the results in Graph 4.

It is possible to notice a variation in the absorption in each proposed time. However, this variation tends to be lower when compared to the adapted method of ISO 17190:2001. Probably because the amount of PSAR used in the assay is 10× higher. This variation in material absorption can be explained by the inhomogeneity of the sample: as the company manufactures several hygiene products. Some of them - such as diapers - contain a higher amount of PSA, resulting in a batch of material with greater absorption, in the case of female pads, where the amount of PSA is lower. It results in a lot of material with lower absorption.

Graph 3: PSAR60 absorption by sieve method.

Graph 4: PSAR average absorption.

The homogeneity of the material was related to its standard deviation, where the reduction of this values tends to show that a better dispersion of the superabsorbent polymers may have occurred. Reducing the variation of the absorption values found. Looking at these values, it is possible to notice a significant improvement in the standard deviation of the absorptions at each time in the 3 residue variations, where PSAR15 homogenized for 15 minutes had a 15% reduction in standard deviation when compared to PSAR nat. This reduction is more evident in PSAR60, with a longer homogenization time and a 43% reduction in its variation compared to PSAR nat. Therefore, the significant improvement in material homogeneity as a function of mill utilization time is evident.

The values that went through the homogenization process obtained higher absorption values in the initial times when compared to PSAR nat, and the final values change little among themselves. Thus, the biggest difference may be related to the material's hydration rate, since there was no chemical alteration of the material,

causing the PSAR hydration reactions to happen more sharply because the homogenization process resulted in a decompression of material.

It was observed that, after 30 minutes of hydration, no significant variations were obtained. Therefore, it is recommended that for the application of the prehydrated material as an internal curing agent in Portland cement matrices. Hydration times of less than 30 minutes should not be applied – as well as not applying the material with its hydration potential. It may still end up absorbing the kneading water from the mixture. Impairing workability and altering the availability of water for hydration reactions. Based on the results obtained, due to homogenization and absorption potential, resulting in lower amounts of solid PSAR to be increased in cement mixtures by the method of BENTZ *et al.* [13]. The result with higher application was PSAR60 with hydration time of 30 minutes, with its hydration value of 68.26 g/g.

4. CONCLUSIONS

Through the method proposed by ISO 17190-6:2001. It was not possible to efficiently determine the absorption of PSAR, because the amount used in each assay (0.2 g) did not demonstrate a real representativeness of the batch.

The proposed method for homogenization was effective in reducing the material absorption variation provided using the mill, where the reduction in the coefficient of variation between the tests performed on PSAR15 and PSAR60, when compared to PSAR nat, were significantly smaller than the ones used as longer mill times were applied with variation values reduced by 15% for mill use for 15 min and 43% for 60 min.

It is possible to notice that, in the averages of the presented variations, there was a stabilization in the hydration in the 30 minutes point, and in the subsequent times. Little significant variations were noticed. Therefore, at 30 minutes potential hydration, values have already been obtained.

To the use of the material as an internal curing agent in Portland cement matrices. The material with the highest application potential was PSAR60 with hydration time of at least 30 minutes and absorption of 68.26 g/g. After 30 minutes of hydration, no significant variations in absorption were obtained. The homogenization method that originated the PSAR60 presented the greatest reduction in the variation of the results, making the batch more homogeneous.

5. ACKNOWLEDGEMENT

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