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Moisture correction in mixtures using innovative thermogravimetric technique to determine water absorption in different Colombian fine aggregates

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Abstract

One aspect that affects the strength of Portland cement mortars is the correction of moisture in the mix. Thus, standard techniques are usually used to obtain the absorption value in fine aggregates; one of them is the standard ASTM C128. However, the ASTM C128 presents variability associated with the identification of the saturated surface dry state in the fine aggregates. This research compares the water absorption of four types of Colombian aggregates obtained using the ASTM C128 and an innovative instrumental and analytical technique with the thermogravimetric balance of halogen light (TBHL), coupled with a software that reads the retained water data in real time. The strength of mortars designed on the basis of moisture correction is also evaluated. The absorption results show that TBHL presents lesser dispersion results (not exceeding 10%) than the standard method ASTM C128, indicating greater precision and less uncertainty. Additionally, resources are optimized through TBHL implementation, requiring 25% less time, 86% less energy consumption, and 98% less fine aggregate for a test. Furthermore, the strength of the mortars varies depending on the method used to determine the absorption, as well as the morphology of the aggregates. The accuracy of TBHL allows better correction of the water–cement ratio in the mortars; thus, leading to higher performance.

Keywords: Halogen light; Water absorption; Fine aggregate; Resources optimization; Mortars; Compressive strength

Corrección de humedad en mezclas mediante innovadora técnica termogravimétrica para determinar la absorción de agua en diferentes agregados finos colombianos

Resumen

Un aspecto que afecta la resistencia de los morteros de cemento Portland es la corrección por humedad de la mezcla. Se suelen utilizar técnicas estandarizadas para obtener el valor de absorción en áridos finos; una de ellas es la norma ASTM C128. Sin embargo, esta norma presenta una variabilidad asociada a la identificación del estado seco superficial saturado en el árido. En esta investigación se compara la absorción de agua de cuatro tipos de agregados colombianos obtenida mediante la norma ASTM C128 y una innovadora técnica instrumental y analítica con la balanza termogravimétrica de luz halógena (TBHL), acoplada a un software que lee los datos de agua retenida en tiempo real. También se evalúa la resistencia de los morteros diseñados a partir de corrección por humedad. Los resultados de absorción muestran que la TBHL presenta resultados con menor dispersión (no superior al 10%) en comparación a la ASTM C128, lo que indica una mayor precisión y menor incertidumbre. Además, se optimizan recursos mediante la aplicación del TBHL, requiriendo un 25% menos de tiempo, un 86% menos de consumo de energía y un 98% menos de áridos finos para un ensayo. La resistencia de los morteros varía en función del método utilizado para determinar la absorción, así como de la morfología de los áridos. La precisión del TBHL permite una mejor corrección de la relación agua-cemento en los morteros, lo que conduce a un mayor rendimiento.

Palabras clave: Luz halógena; Absorción de agua; Agregado fino; Optimización de recursos; Morteros; Resistencia a la compresión

1. Introduction

In the short and long terms, the mechanical performance of concrete made of hydraulic Portland cement depends on the effective hydration of the cement. For this reason, the water absorption of the aggregates is an essential factor in designing mixtures (Djerbi, 2012), which motivates the establishment of a water–cement ratio according to a mix design. In this sense, the measurement of the water retention capacity of the aggregates can be decisive because they account for approximately 60%–75% of the total volume of a mix (Kosmatka &Wilson, 2011). Additionally, the aggregates are the most porous components of the mix, especially fine aggregates, which, regardless of their origin, increase the degree of water retained as their fraction decreases (Gentilini et al., 2015) (Arias, Payá & Ochoa, 2016). To determine the amount of water retained for fine aggregates, the particles must be placed in a saturated surface dry (SSD) state. The particles have no moisture on the surface and no capacity to retain more water internally. Thus, it is guaranteed that the aggregate will not take away or add water to the mix; consequently, it will not alter the water–cement ratio initially established.

It has been challenging to find an efficient and accurate method to determine the absorption value of fine aggregates. The international standard ASTM C128 (NTC 237 in Colombia) has been the conventional method to obtain this value, suggesting a strong dependence on the operator when identifying the SSS state, which varies depending on the morphology, granulometry, and origin of the aggregate (Naël-Redolfi, Keita & Roussel, 2018). This leads to low reliability and reproducibility of the measurement of the water absorption value.

Several new methods have been proposed to obtain the SSS state to reduce the dispersion and uncertainty of the results. For example, Lin and Chuang proposed a method using impedance spectrometry based on the resistivity and conductivity of aggregates with different moisture contents (Lin & Chuang, 2013). (Miller et al.,2014) evaluated the application of a technique based on the centrifugation of prewetted lightweight aggregates, showing that the results are comparable with those obtained using the paper towel method established using the ASTM 1761-13b. Chemical indicators, such as cobalt chloride and fluorescein disodium salt, based on color change have also been investigated as an alternative (Kandhal & Lee, 1970)(Prithvi S. Kandhal; Dah-Yinn Lee, 1970). The aggregate can change from red in the wet state to blue in the dry state using the 5% cobalt chloride solution. It is assumed that the surface is dry, but internally, the aggregate is saturated; however, chemical techniques are not adequate for dark aggregates, besides not having homogeneous drying properties. Therefore, it is necessary to conduct exhaustive tests to establish the reproducibility and limitations.

Automated processes using technological devices have been proposed. For example, (Kandhal & Mallick, 1999) proposed to subject the aggregate to a constant flow of hot air, and thus, measure the temperature gradient of the outgoing air with the relative humidity to establish the state of SSS. Or the SSDetect device proposed by (You, Mills-Beale & Williams, 2009) is based on the use of infrared light with an automatic volumetric mixer, which while injecting water, the light source detects when the pores are completely filled, and the water begins to accumulate on the surface. Meanwhile, the AggPlus (Tran, West & Azari, 2015) consists of a calibrated pycnometer and a vacuum sealing device coupled to a software that allows measuring the density by fully saturating the sample through vacuum sealing and in unsaturated conditions; however, this system is sensitive to the type of aggregates. Additionally, Gilson Company developed the SG-5 aggregate specific gravity and absorption system based on Boyle’s law using dry air to perform several cycles of pressurization and depressurization of the sealed chamber, where the water absorption of the aggregates is automatically calculated. Although the proposed techniques have had considerable improvements with respect to the standard technique, they also have some limitations associated with the complexity of the processes and their reproducibility, the expertise of the operator, and above all, the sensitivity in changing the type of aggregate.

This research proposes an innovative technique based on a thermogravimetric process using a halogen light balance coupled with a software tool to measure the mass loss of particles as a function of time. The SSS state is determined by applying a mathematical concept of the mass loss function over time that reflects a change in slope and inflection point to determine the absorption value of fine aggregates of different origins and morphologies. In this sense, the thermogravimetric balance of halogen light (TBHL) technique was evaluated in different fine aggregates from two Colombian regions to investigate the efficiency of the technique and extend its implementation from the standardization processes required to complement the current standards.

2. Materials and methods

2.1. Fine aggregates

Four natural fine aggregate (sands) types were used: river and quarry sand from two regions in northwest and east of Colombia, Antioquia (region 1) and Orinoquía (region 2), respectively. Quarry sands are obtained from the crushing of both rocks and river aggregates from river sources originating from local companies (Table 1).

Table 1. Microphotographs of fine aggregates (sands)

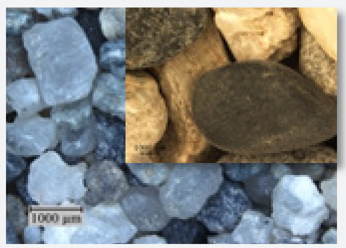
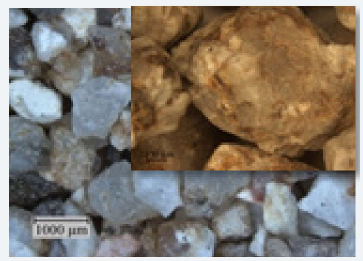
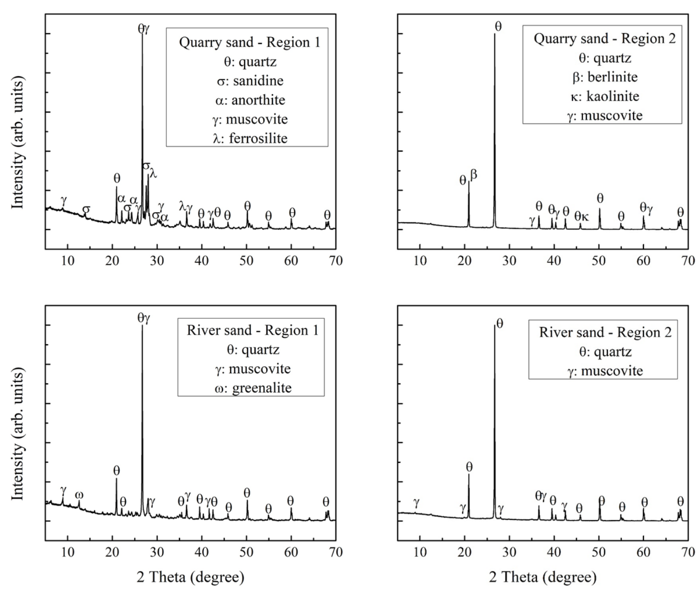
Type	River	Quarry
Antioquia		
Orinoquía		

Table 2 shows the chemical composition of the aggregates determined using X-ray fluorescence using a Phillips PW 2400 XRF spectrometer. Mineralogy analysis was performed using X-ray diffraction on a PANalytical X'Pert MPD PRO between 5° and 60° for 120 min with a $CuK_{\alpha1}$ (λ = 1.54059 Å) source. The mineral composition of the four fine aggregates is shown in Figure 1.

Table 2. Chemical composition of fine aggregates (sands)

	Quarry		River	
	Region 1	*Region 2*	*Region 1*	*Region 2*
Oxides	**Composition (%)**			
SiO_2	70.592	92.251	70.589	85.476
Al_2O_3	16.513	4.871	15.635	9.251
Fe_2O_3	2.450	0.829	4.866	2.440
CaO	0.505	0.153	2.278	0.221
MgO	1.450	0.706	3.094	0.084
MnO	0.035	-	0.074	0.070
SO_3	0.141	0.128	0.182	0.187
P_2O_5	0.447	0.439	0.512	0.495
Cl	0.197	0.160	0.193	0.197
K_2O	5.168	0.280	2.039	1.157
TiO_2	0.471	0.182	0.494	0.360
ZnO	0.006	-	0.007	0.006
SrO	0.024	-	0.007	-
Y_2O_3	0.010	-	0.016	-
ZrO_2	0.031	-	0.013	0.056

Figure 1. Mineralogical composition of fine aggregates (sands)



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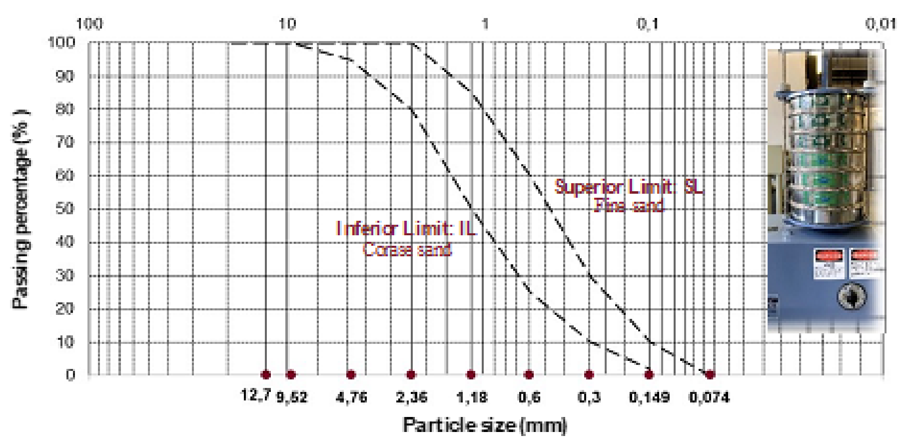
The aggregates were graded according to ASTM C33 (NTC 174 in Colombia) to establish the specifications of fine aggregates for concrete. According to the standard specifications, the fine aggregates were standardized using several sieves with diameters between 0.075 and 4.75 mm to adjust each aggregate to two types of granulometric curves called inferior limit (IL) and superior limit (SL), as shown in Figure 2.

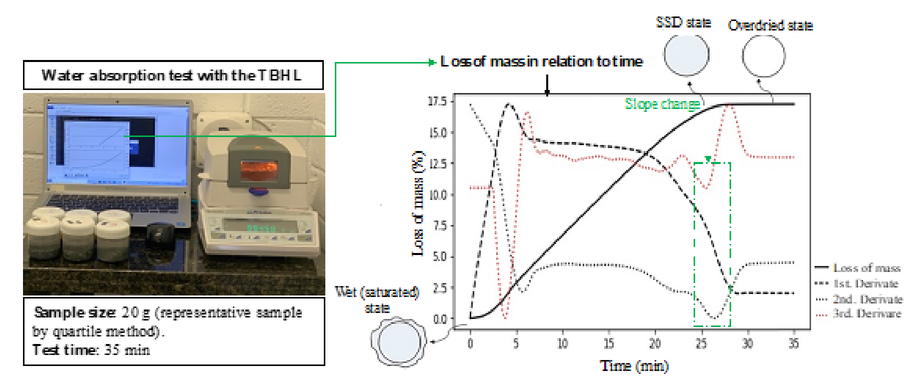
Figure 2. Standardization of aggregate particle size



2.2. Innovative technique TBHL

Aggregates are porous structures, so they have different states depending on their moisture content. The innovative TBHL technique starts from the saturated state to find the SSD state by a thermogravimetric process using a halogen light balance. By using the halogen moisture analyzer, continuous measurements of water loss can be made, which can be associated with adsorbed and absorbed water in a fine aggregate. The SSD state is considered the most suitable, since the particles in this state are not adsorbed, so the properties of the concrete are not affected (Arias, Payá & Ochoa, 2016). The TBHL is coupled to a software tool to measure the mass (water) loss of the fully saturated aggregate sample as a function of time, which ensures a soft heating of the sample up to a maximum temperature of 85°C+-1°C. The SSD state is determined by a derivative process based on the change in the slope of the water-loss/time curve and the inflection point (2^{nd}. and 3^{rd}. derivative) associated with that function, which under isothermal conditions, can be representative of water absorption, see Figure 3. In this sense, the initial moisture loss rate will be higher, and will decrease as the particles go from losing moisture at the surface (adsorption water) to losing water inside the pores (adsorption water).

Figure 3. Loss of mass over time to determine absorption with TBH**L**



The software used is called ABSORPTION INNOVATION. It has registration No. 13-87-33 in Dirección Nacional de Derecho de Autor, Ministerio del Interior, Colombia. Its validity lies in its ability to read the data from the halogen light balance to construct the mass loss curve as a function of time, perform the calculations, and provide the final absorption report in real time. Determining the percentage absorption in natural fine aggregates (sands) using the TBHL technique has been shown a resources optimization, ensuring reduced deviation and improved precision of the results compared with those using the standard method (Gómez et al., 2021).

2.3. Experimental program and mortar mix proportions

The effectiveness of the innovative technique was evaluated by randomly determining and comparing the percentage absorption of the four fine aggregates using the TBHL method and the standard method (ASTM C128), with three replicates per type of aggregate. In this case, the type and grain size distribution of aggregates with different levels are considered (Table 3). In the case of river fine aggregate from the Orinoquia region, only the size LS.

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Table 3. Factors for determining water absorption

Type of fine aggregate (sand)		DTP	Code
River	Antioquia: 1	LI	R1-LI
		LS	R1-LS
	Orinoquia: 2	LS -	R2-LS -
Quarry	Antioquia: 1	LI	Q1-LI
		LS	Q1-LS
	Orinoquia: 2	LI	Q2-LI
		LS	Q2-LS

The mix design for target strength of 30 MPa is shown in Table 4. In the mixing process, UG Portland was used; the cement–sand ratio of the mortar was 1:2 by mass (300 and 600 g of cement and aggregate, respectively), and the water–cement ratio was constant at 0.48. All the mixtures were corrected for moisture, considering the absorption value determined with both the innovative technique TBHL and the standard ASTM C128, from which the amount of additional water required by each mixture is known. The flow of the mixtures was evaluated in accordance with ASTM C1437, and the compressive strength was tested using 50 × 50 × 50 mm cubes at 7 days after curing. Three replicate cubes were tested according to the type of aggregate included in the mix, the objective of which is to know the variance of the data by means of the standard deviation and to compare the effect of the moisture correction of the mixes from the two methods, ASTM and BTHL.

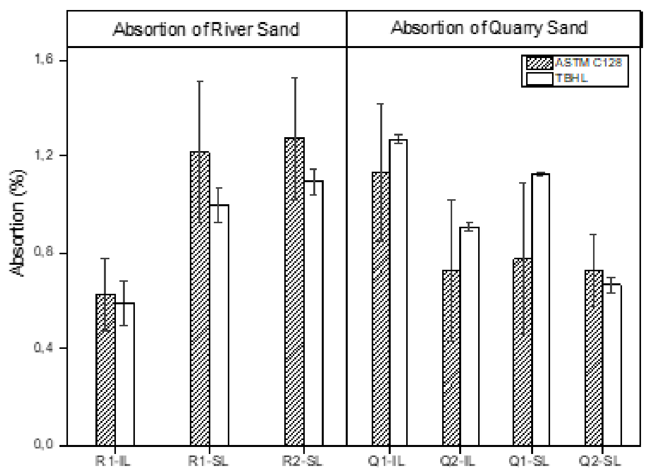
Table 4. Mortar mix proportions and moisture correlation

Code	Cement (g)	Fine aggregate (g)	Total water (g)		Additional water (%)		D_{WATER} (%)	D_{WATER}/ ASTM 128 (%)
			TBHL	*ASTM 238*	*TBHL*	*ASTM 238*		
R1-LI	300	600	147,50	151,50	2,5	5,2	−2,7	−52,0
R1-LS			150,00	150,90	4,1	4,8	−0,7	−15,0
R2-LS			150,60	148,80	4,6	3,3	1,3	39,0
Q1-LI			151,60	148,40	5,3	3,0	2,3	77,0
Q1-LS			150,80	149,40	4,7	3,8	0,9	24,0
Q2-LI			149,40	148,90	3,8	3,4	0,4	12,0
Q2-LS			148,00	148,40	2,8	3,0	−0,2	7,0

3. Results and discussion

Figure 4 shows the water absorption results for river and quarry fine aggregates (sand), demonstrating that the innovative TBHL technique is suitable for any fine aggregate size below 4.75 mm. The results obtained using TBHL allows identifying that water absorption values can be evaluated in a shorter time (25% less), with lower energy consumption (86% less), and can maintain significantly lower sample mass (98% less). However, as evidenced by previous studies, the traditional practice with the standard ASTM C128 depends on individual skill and judgment to determine the SSD status of fine aggregates, thus, increasing its variability (Lin & Chuang, 2013). On the other hand, as evidenced in previous studies, traditional practice with ASTM C128 depends on individual skill and judgment to determine the SSD status of fine aggregates, thus increasing its variability (Lin & Chuang, 2013). In this case, unlike the ASTM C128 results, the TBHL technique has lower scatter and higher accuracy of absorption values for all types of aggregates evaluated, see Figure 4.

Figure 4. Water absorption of aggregates by TBHL and the standard ASTM C128



The absorption values obtained with TBHL did not exceed 1.5%, which is consistent with the absorption rate range for sand between 0.4% and 3.8% reported by (Klein et al., 2014). It can also be observed that for quarry and river fine aggregates, in all cases of LI (coarse) size, there is a greater difference between the absorption results when comparing both techniques.

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In terms of morphology, the TBHL technique underestimates the absorption result for rounded smooth aggregates compared to the ASTM C128, which may be due to the fact that rounded smooth particles have less friction between them when stacked. In addition, due to rolling or sliding effects between particles, gravity allows rounded aggregates to have greater mobility towards crumbling. Thus, the ASTM C128 absorption test requires greater amounts of water to generate a layer on the surface of the particles allowing adhesion between them to achieve the suggested conical shape, i.e. the SSD condition (see Figure 5). On the contrary, LI (coarse) particles with more edges, such as quarry particles, have higher inter-particle adhesion and friction and require less water for the ASTM C128 test, which is ratified by what was reported by (Kasemchaisiri & Tangtermsirikul, 2007).

Figure 5. Cone shape for determining SSD (ASTM C128)

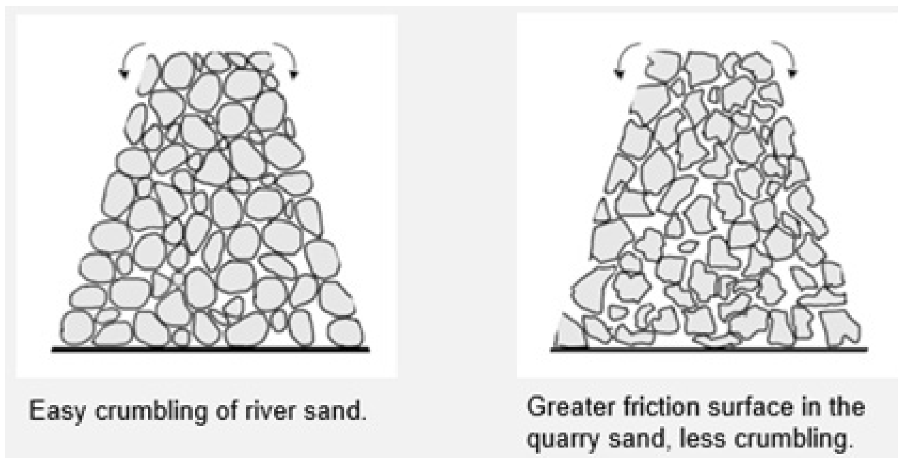

Table 5 and Figure 6 show the results of the flowability and compressive strength of the five mortar mixes produced from the moisture correction. It can be seen that the predicted compressive strength of 30 MPa was achieved in all cases.

Table 5. Flow of mortars

Type of fine aggregate	ASTM C128		TBHL	
	Flow (%)	***Standard deviation***	***Flow (%)***	***Standard deviation***
R1-LI	151.0	*0.000*	150.0	*0.000*
R1-LS	113.3	*0.053*	107.9	*0.010*
R2-LS	113.4	*0.003*	115.5	*0.003*
Q1-LI	115.0	*0.028*	115.6	*0.010*
Q1-LS	33.2	*0.019*	49.8	*0.016*
Q2-LI	124.9	*0.017*	124.8	*0.000*
Q2-LS	93.8	*0.058*	95.9	*0.003*

Figure 6. Compressive strength of mortars

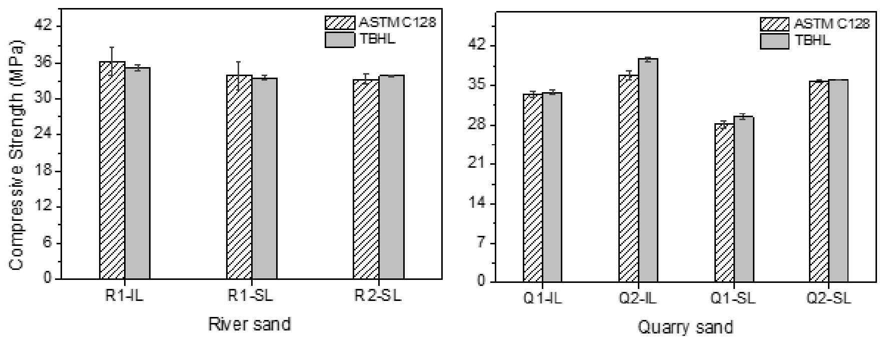

Figure 6 shows that the mechanical performance of mortars with different aggregates varied as a function of the moisture correction technique. Thus, the higher the water demand in the mix, the lower the compressive strength for mortars cured in 7 days. These results are consistent with those of (Cortas et al., 2014), where the behavior of the concrete and the development of its properties at an early age depend to a large extent on the water saturation of the aggregates. This may be because the effect of the capillary pores of the aggregates by the excess water is more noticeable at longer ages, 28 or 90 days of curing. In this sense, the evaluation of longer curing ages will allow us to observe in detail the efficiency of TBHL because the water demand from the moisture correction in most cases was lower for each mix.

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In early curing ages, a higher amount of water in the mix (ASTM case) guarantees a better mechanical performance; however, no significant increases are observed, which indicates that both techniques are comparable, and that according to the results found, their difference is that the BTHL technique has demonstrated to be more efficient in terms of resource optimization and precision of the results.

Although there are differences in moisture content (Table 4), ranging from 7% to 77% for quarry aggregates and 15% to 39% for river aggregates. Figure 6 shows that there is no significant effect on the flowability of the mixes. However, in terms of the precision of the compressive strength results, there is still a significant difference between the mortars with moisture correction using the TBHL and standard techniques. This situation confirms the importance of decreasing the dispersion of water absorption values for the moisture adjustment in the mix design.

4. Conclusions

The following conclusions are obtained from the experimental results and discussion.

- In contrast to ASTM C128, the TBHL technique requires 25%, 86% and 98% less time, energy and material, respectively, when determining water absorption. Thus, the standard ASTM technique requires more resources and depends on individual skills to determine the SSD state of fine aggregates (sands), making it evident that the innovative TBHL technique proves to be more efficient.
- The morphologies of the two types of fine aggregates (sands) evaluated have an influence on the degree of water absorption. Unlike the river aggregates (redounds), the quarry aggregates (angular) presented higher absorption values. For all cases, the water absorption results determined using the ASTM C128 technique show greater dispersion than those using the TBHL technique, where the deviation of the results does not exceed 10%. This indicates higher precision and lower uncertainty in the values.
- The expected compressive strength of 30 MPa was achieved, even exceeded in many cases. The mechanical performance for mortars with different aggregates varied depending on the technique under which the moisture correction was performed. The higher the water demand in the mix, the lower the compressive strength for mortars cured in 7 days.

- Because the effect of excessive capillary pore water may be more visible at prolonged curing ages, 28 or 90 days, it is recommended to evaluate prolonged curing ages to further observe the effectiveness of TBHL.

5. Acknowledgments

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