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Influence of the chemical composition of sand and cement on the performance of plasticiser admixture for concrete

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Abstract :

In the current study, the chemical composition of sand and cement was examined in relation to the plasticiser admixture for concrete. Three types of sand and cement were used, on which additives from different origins would be dosed and incorporated into various mortars for evaluation. This study used three additive technologies to determine the admixture quality. The Methylene blue value (MBV) test was performed on five sand samples, and the influence of temperature and rheological parameters was also tested for the selected cement and sand samples. From the results, it was identified that the Concrete and River sands are those with the highest clay content since they have a methylene blue value (MBV) of 10.00 mg/g and 8.50 mg/g, respectively; the mortars designed with coarser sands obtained more excellent resistance, both at 7 and 28 days, regardless of the type of cement and additive.

The Coarse and Utility sands present an MBV of 7.75 mg/g and 7.50 mg/g, respectively, which could also be considered within the range of the maximum permitted limit. When it was observed in the rheological investigations, the ambient temperature can easily vary from 5ºC to 30°C without causing apparent changes in the behaviour of the mortars. The investigated cement had a C3A content that varied between 1.5 and 12.6 and a soluble alkali content between 0.48 and 1.44. The influence of these two parameters can only be compared with different cements or simulated by adding similar materials. It can be concluded that when the sand is very fine (its particles almost reaching the limit size, close to that of clays), it has a larger surface area and can more easily attract the particles to the additive, generating a sand-additive interaction (which does not improve plasticity); avoiding the cement-additive interaction, which will subsequently decrease the plasticity of the mortar since it is precisely this interaction that generates the increase in fluidity and workability.

Keywords: Chemical composition, sand, cement, methylene blue value, rheological investigations.

تأثير التركيب الكيميائي للرمل والإسمنت على أداء خليط الملدنات للخرسانة

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مستخلص:

في هذه الدراسة، تم تحديد التركيب الكيميائي للرمل والإسمنت في خليط الملدنات للخرسانة من خلال أخذ ثلاثة أنواع من الرمل والإسمنت، حيث سيتم إضافة جرعات من هذه المادة من مصادر مختلفة، وستكون جزءًا من أنواع الملاط المختلفة للتقييم. في هذه الدراسة، استُخدمت ثلاث تقنيات إضافية لتحديد جودة الخليط. أُجري اختبار قيمة الميثيلين الأزرق على خمس عينات رمل، كما تم اختبار تأثير درجة الحرارة والمعايير الريولوجية لعينات الإسمنت والرمل المختارة. من النتائج، تبيّن أن الخرسانة ورمل النهر هما الأكثر احتواءً على محتوى الطين، حيث تبلغ قيمة الميثيلين الأزرق (MBV) فيهما 10.00 ملغم/ غم و8.50 ملغم/ غم على التوالي. وقد حصلت الملدنات المصممة برمال أكثر خشونة على مقاومة أفضل، في كل من 7 و28 يومًا، بغض النظر عن نوع الإسمنتقبق والمادة المضافة. فيما يتعلق بالرمال الخشنة والنفعية، فإنها تقدم MBV يبلغ 7.75 مجم / جم و 7.50 مجم / جم على التوالي، والذي يمكن اعتباره أيضًا ضمن نطاق الحد الأقصى المسموح به. ومع ذلك، فإنها تقدم كمية أقل من الطين مقارنة بالسابقة. عندما نلاحظ التحقيقات الريولوجية، يمكن أن تختلف درجة الحرارة المحيطة بسهولة من 5 درجات مئوية إلى 30 درجة مئوية دون التسبب في تغييرات واضحة في سلوك الملاط. كان للأسمنت المدروس محتوى C3A يتراوح بين 1.5 و 12.6 ومحتوى قلوي قابل للذوبان بين 0.48 و 1.44. لا يمكن مقارنة تأثير هذين المعاملين إلا بأسمنت مختلف أو محاكاته عن طريق إضافة مواد مماثلة. يمكن أن نستنتج أنه عندما يكون الرمل ناعمًا جدًا (تصل جزيئاته تقريبًا إلى حجم الحد، بالقرب من حجم الطين)، يكون له مساحة سطح أكبر ويمكنه جذب الجزيئات بسهولة أكبر إلى المادة المضافة، مما يؤدي إلى تفاعل الرمل مع المادة المضافة (الذي لا يحسن اللدونة)؛ تجنب تفاعل الأسمنت مع المواد المضافة، والذي بدوره يقلل من مرونة الملاط، حيث أن هذا التفاعل بالتحديد هو الذي يولد زيادة السيولة وقابلية التشغيل.

1. Introduction

Concrete is a mixture of four basic materials: cement, fine aggregate (sand), coarse aggregate (crushed stone), and water. If poured into a mold and then left, the mixture will harden like a rock. This hardening occurs due to a chemical reaction between water and cement, which happens over time, so the concrete's hardness depends on the mixture's age [1]. As a structural material, it is necessary to know from concrete its properties such as strength, stiffness, water tightness, durability, and weather resistance.

. The compressive strength of the concrete can describe some of these properties, so the quality of the concrete is usually only reviewed based on its compressive strength [2].

The development of various types of additions or admixtures and additives for concrete mixtures, especially water reducers or plasticisers and superplasticisers, has made very rapid progress in concrete technology, successfully producing high and even very high-quality concrete, and which improves almost all concrete performance into a modern high-performance material. The strength of concrete is also inseparable from its constituent materials [3]. It is precisely to manufacture high-performance concretes (resistances above conventional ones), different engineering solutions have been tested; both physical, which involve the use of ultra-fine grinding (with enrichment in mineralogical phases such as Alite and Belite), and the incorporation of active additions (such as Silica Fume, fly ash, Metakaolin, rice husk ash and blast furnace slag); and chemical, which especially involve the use of chemical products (chemical additives),[4] for the optimisation and improvement of the quality of cement and concrete; which will be precisely the subject that this work will detail, since its study and influence, on the properties of cement and sand, are the foundation and purpose of this research.

Finally, it emphasises that this work makes a novel analysis to understand the mechanism through which superplasticisers increase the fluidity of mortars since previous investigations only quantified the adsorption of the additive based on the rheological tests found. Still, this work also analysed the amount of superplasticiser consumed by the cement and the amount retained at the cement-additive interface, which is the only one that can be used in the future to maintain fluidity [5]. This research will seek to identify the influence of the chemistry of the materials (cement and sand) on the performance of concrete additives of different technologies and, thus, find a relationship between the performance of these chemical additives and the properties of the materials that make up the concrete.

2. Materials and Method

2.1 Chemicals and Reagents: Methylene Blue was obtained from SD Fine Chemicals, India, and other chemicals and reagents were obtained from Merck India, with AR quality and 98% purity. All the solutions were prepared with double distilled water with a pH of 7.02. Diluted Methylene Blue, concentration A (For AASHTO specifications): 1.599g (MB)/100g (H2O) and Diluted Methylene Blue, concentration B (For ASTM and IS specifications): 2.500gr (MB)/100g (H2O).

Three types of cement used in the study are (i) Ordinary Portland Cement (OPC), (ii) Portland Pozzolana Cement (PPC) and (iii) Portland Limestone cement (PLC); five sands used in the study River sand Pit sand, Concrete sand, Utility sand and Coarse sand.

2.2 Equipment: A&D GH-202 model Analytical balance, Mettler Toledo Platform, Remi Model mechanical stirrer with a maximum speed of 500 rpm, Remi 2 Litre Stirrers Magnetic stirrer, with heater 2 MLH, Sunline 45 Litre Aluminium Hot Air Oven, OU-,1001/2with a maximum temperature of 150ºC, Hobart mixer N-50, with a maximum speed of 100 rpm, and Electric Laboratory Sieve (PerkinElmer), with 4” meshes up to 0.075mm

2.3 Description and Characterisation of the Materials to be Used

- Three different additive technologies, namely Naphthalenesulfonate (SNS) Base, Lignosulfonate Base (LS) and Polycarboxylate Base (PC), will be evaluated in the current study.
- The three cements on which the additive will be dosed will have different types and origins. The ce-

ment will be characterised according to ASTM C150 and ASTM C1157 [6].

- The three sands, which will be part of the different mortars for evaluation, will come from various sources seeking variety in their physical characteristics and chemical composition (acceptable material content, clays, silts, etc.).

2.4 Methods

Test to calculate the natural moisture content: The natural moisture of the selected sand samples is determined by the Precisa Moisture Analyser model 330 XM. Finding the natural moisture content of the sands is essential since this value, together with the absorption moisture and the specific weight, constitute the three primary and determining values to build a design table, both for mortars and for the design of concrete;

Test Calculate of the Fineness Modulus (FM): The classification of sands concerning their fineness modulus is a critical parameter to be able to identify them since it allows us to know the average size of the sand particles and FM of the selected samples identified according to the ASTM C136 method.

Test to calculate the percentage of fines that pass the #200 mesh: The classification of the sands, about the "percentage" of fines that pass the #200 mesh, is also essential since it shows a more real indication of the fineness of the sands; and even more importantly, it shows the content of fines in the sands; not wholly, but approximately; since analysing the % of fines that pass the #200 mesh by washing, allows us to see that part of the sand is fine material; and from this, and from applying the other methods (such as MBV), it will be possible to calculate the percentage of clay in the sand, which is what will weaken the concretes and mortars manufactured with it.

It should be noted that this method will calculate the percentage of all fines present in the sands (which includes silts clays, among other mainly bituminous particles), while the methylene blue value (MBV) method only detects clay particles (which are present in the sand samples); and although as mentioned above, both values (% of fines and MBV) generally have the same tendency; for example, a sand with a high content of fines could be found, but with a low content of clays (in these cases, most of the fines would be silts), so this sand would have no problems in forming part of the design of a mortar or a concrete mix. The same analysis could be done in the opposite direction since sands with a low % of fines could have a lousy performance within a mortar.

2.5 Analysis of the chosen Indian cement

As explained above, there are five types of cement available on the world market (I, II, III, IV, V); in addition, there are almost 30 other types of cement, modified from the other 5; however, not all of these types of cement are available in India (as in different countries, some of the cement that is present in India are not present either); therefore, only those produced here will be analysed.

Below, to expand on the above is a detailed analysis of the situation of cement in India, presented by the Portland Cement Association (PCA) [7]:

“India has a wide variety of cement as follows:

- Type I (Normal), with a 7-day strength of 19MPa (190kg/cm2).
- Type II (Moderate resistance to sulphates), with 7-day strength of 17MPa (170kg/cm^2) and maximum C3A of 8%.
- Type III (High initial strength).
- Type IV (Low heat of hydration), with a 28-day strength of 17MPa (170kg/cm^2).
- Type V (High resistance to sulphates) with a 28-day strength of 21MPa (210kg/cm^2) and a maximum C3A of 5%.

The Portland Cement Association mentions that in India, the added cements are:

- Portland pozzolanic cement (IP and P) can have 15 to 40% pozzolan by mass.
- Modified Portland pozzolanic cement – I (PM) have up to 15% of pozzolan.
- Slag Portland cement has 25% to 70% of added blast furnace slag.
- Modified slag Portland cement – I(SM) may have up to 25% slag.
- Composite Portland cement (ICo) may add limestone or inert material of up to 30%, as long as this

material has at least 75% $CaCO_3$.

These cements may have a combination of other properties, such as moderate resistance to sulphates and mild heat of hydration, by adding the suffixes MS and MH, respectively. The 28-day strength requirement for this cement is 25MPa (260kg/cm^2), except for IS(MH) and IP(MH) cement, which must have at least 20MPa (200kg/cm^2), and P cement, which must have at least 21MPa (210kg/cm2) at 28 days.

The IS 4031 standard contains the performance requirements for Portland cement for general and special applications without restrictions on the composition or constituents of the cement. This standard is based on ASTM C1157 [7]. It differentiates modified Portland cement (up to 15% additions) from additive cement (with more than 15% additions) and classifies them according to their properties.

2.6 Experimental Development

Methylene Blue Value (MBV): The methylene blue value test was performed on five sand samples to make the final choice of 3 based on the results. To achieve this, this test was performed using two different methods, first with the AASHTO (American Association of State Highway and Transportation Officials) Methodology and then with the ASTM Methodology; that is, a comparison was made between 2 of the three known and available methods for determining the clay content. The IS Methodology was not performed due to the similarity of results between ASTM and AASHTO. This led us to assume that the results obtained were consistent and sufficient to classify the sands correctly.

Although the two methods were previously described, it is noted that the second method (ASTM Method) was slightly modified by omitting the addition of HCl to the sample since this was not necessary, as the sample was sufficiently reactive and uniform, and could react on its own with methylene blue (it is only because of this small change that the ASTM Method is called the Pasquel Method in this research; because Eng. Enrique Pasquel was the first to base and validate this method without adding HCl.

Mortar and Mini Slump Design: Once the three most efficient cement and 3 grains of sand were chosen, mortars were designed (mixtures of cement, water and sand; only if gravel were added would they be called "concrete"). Nine white mortars were obtained because all possible combinations were made (without additives). When the addition of each of the three additives to be investigated was made, there were 27 combinations representing 27 different types of mortar, which were analysed and studied physically and chemically.

In all cases, the absorption humidity of the three sands was taken every 21 days to make the initial designs, and the natural moisture every 2 days because the environmental conditions can cause this value to vary more quickly, which would have significantly modified the design of the mortars afterwards.

In the case of cement, its specific weight was taken directly from the technical data sheet of each cement provided by each of the three manufacturers. In the case of sand, the particular weight of the three sands was taken at the beginning of the project.

The dosages used were those initially recommended, but they were varied as their plasticising power in the mortars was experimentally measured, trying to reach a reference value required for the future.

After indicating the general conditions of the materials that make up the mortar, the most common process for preparing mortars is described below [8].

Procedure for preparing mortars (Sand Humidity: 9.0 ± 1.0%)

- Choose a manufacturing design for the mortar (Previously prepared table, where the quantities of each material used are specified).
- Modify the volume (m3) of the mortar to be worked, and place the corresponding humidity of the sand on the design sheet.
- Weigh the components of the equivalent mortar, that is, Sand, water, cement, filler (if used) and additive, according to the design sheet obtained previously.
- Weigh the water in the bowl and separate 10% to 15% of the water by weight into a beaker to wash the additive container.
- Turn on the Hobart mixer at speed 1.
- Add the sand to the bowl containing

the remaining 80% to 90% water and mix for 2 minutes.

- After 2 minutes, add the cement and mix for one more minute at speed 1.
- Add the additive after 3 minutes, rinse the bowl with the separated water percentage, and continue mixing for 1 minute.
- After 4 minutes, turn off and scrape the edge and bottom of the bowl for a few seconds to ensure that the mixture remains homogeneous.
- Turn the equipment back on at speed one and continue mixing for 2 minutes.
- Turn off the equipment after 6 minutes.
- Before carrying out the corresponding tests (fluidity, occluded air content, etc.), have the measuring equipment ready and wet, such as the Vernier calliper, glass plate, etc.
- Finally, wash the equipment, glasses, and other equipment used in this test.

Summarising the procedure, the equivalent mortar was prepared, similar to the design of standard concrete, respecting the order of addition defined for such a case. Once the mortar was prepared, the mini-slump test was carried out (necessary to see the physical characteristics of the mixture), which consisted of placing a sample of the mortar in a metal cylinder, which, when lifted, let the mortar flow in a circular shape, whose diameters were recorded.

To finally culminate with this procedure, the mini-slump test was carried out, which consisted of placing a sample of the mortar in a metal cylinder of 4 cm diameter; rodding, first when a third of the volume is placed (1/3), then at two thirds (2/3), and finally when the entire cylinder is filled, levelling off after placing the last layer; then in 3 seconds, lifting the cylinder, immediately measuring with the Vernier Caliper, the largest and smallest diameter of the resulting mortar (whose axis lines must be perpendicular to each other).

The larger the diameter of the mortar, the greater the plasticising power of the additive can be assumed as a priori. However, quantifying the additive's power will also depend on the duration of this plasticising power and not only on the "Plasticizing value or initial fluidity" found with the diameter.

Later, the influence of temperature will be analysed, and to achieve a better understanding of this section, other rheological investigations were cited, which were carried out on cement pastes and mortars, which also used a Howart rotary mixer machine (Figure 5a), finding in all cases that by working the samples at a constant temperature, approximately 20°C, excellent results could be obtained; however, in practice, other investigations have also shown that the ambient temperature can easily vary from 5ºC to 30°C, without causing apparent changes in the behaviour of the mortars.

In this work, the rheological behaviour (flow behaviour) of three superplasticisers (polylignosulfonate, poly naphthalene sulfonate, and polycarboxylate) was intensively studied in combination with various types of cement. To achieve this, the most critical rheological parameters were the amount of C3A, soluble alkalis (Na+, K+), type and number of superplasticisers used, temperature of the mortar or cement and w/c ratio. C3A content varied between 1.5 and 12.6, and a soluble alkali content between 0.48 and 1.44. The influence of these two parameters can only be compared with different cements or simulated by adding similar materials. The impact of the addition of Na_2SO_4 on the behaviour of the mortar could be explained based on a superplasticiser adsorption model.

Mortar cube design and strength test: Mortar cube design and strength test was carried out by Standard ASTM C109 [9] and Indian Standard IS 4031[11].

3. RESULTS

The natural moisture of the fine aggregate was carried out by ASTM C566-13 [11]. Table 1 shows the classification of the five chosen sands according to the natural humidity found:

3.1 Analysis of the Indian cement chosen

It is recalled that the relative content of alkalis is defined as the sum of the relative percentages of the compounds Na_2O and K_2O. However, some sources only consider Na2O to be the factor that influences the behaviour of the mortar. The content of C3A (Tricalcium Aluminate), we remember that its presence is also essential since the more significant the quantity of this compound, the mortar will have a faster and more powerful initial setting.

3.2 Methylene Blue Value (MBV)

AASHTO Method: The equivalence in determining clay content was validated with the Methylene Blue Value (MBV) Method of the following five grains of sand, according to AASHTO TP 57-2000 [12]. Results shown in Table 4 and Figure 1.

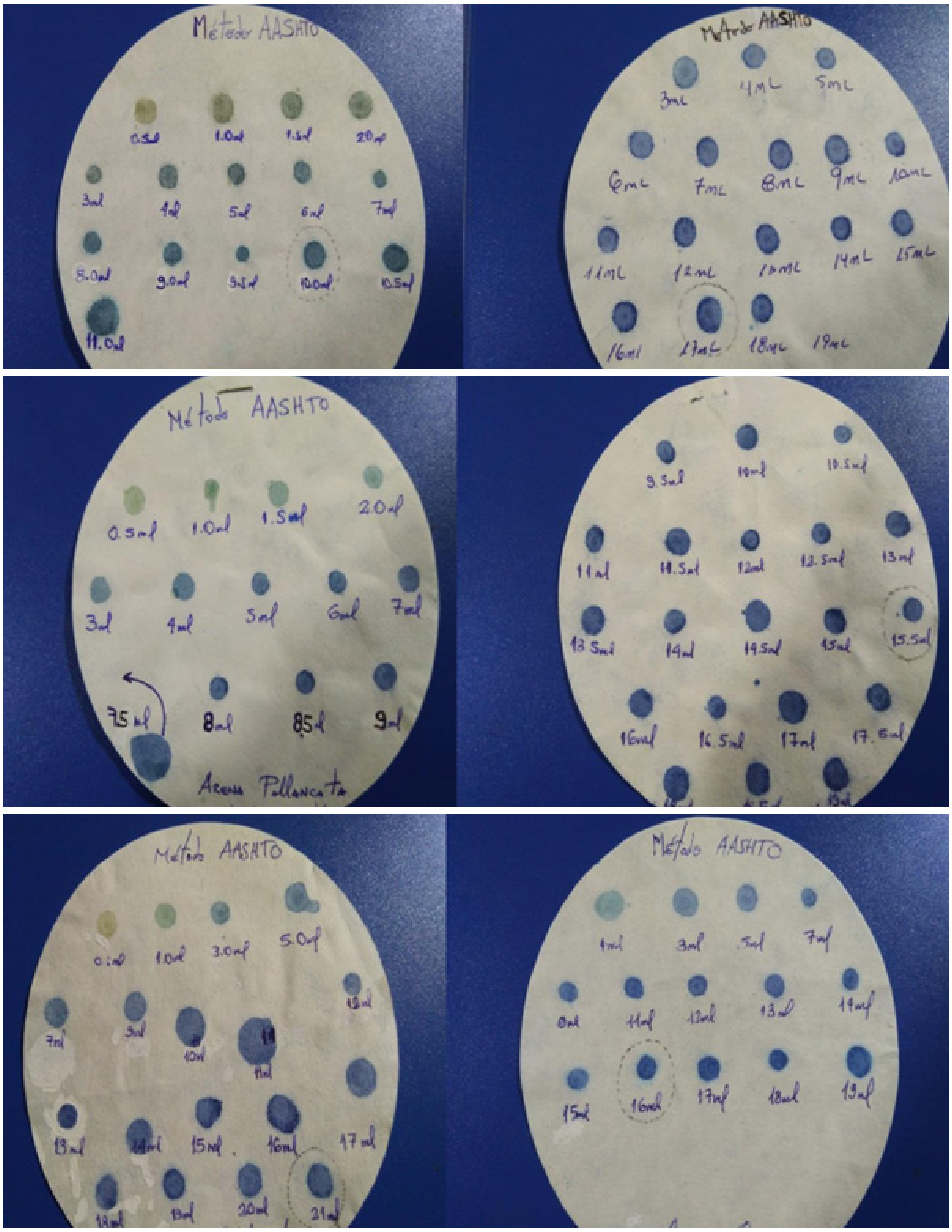

Figure 1: Images with the Watman Filter Papers, given to calculate the Methylene Blue Value (AASHTO):

ASTM Method: The equivalence in determining clay content was validated with the Methylene Blue Value (MBV) Method of the following five sands, according to ASTM C03 - 2003 [13]. The results are shown in Table 5 and Figure 2. As a source of additional information, it is noted that by comparing these two tables (designed with two different methods), it was possible to analyse, for the last time, the five available arenas in a more detailed way, results that served for a correct final choice of the three sands.

3.3 Mortar Design and Mini Slump Testing

In each case, the diameter of the mortar is measured once the metal cylinder is lifted for five different dosages and five different moments; the table with the results obtained and a Diameter vs Time graph are presented for each particular case. In addition, three graphs are presented, which show how the diameter of the mortar varies as the dosage of each additive increases, taking as a reference the initial instant (at 7min); additionally, tabulations are presented to see what the dosages of additive necessary, to obtain specific generic diameters; which has as its goal, provide a more significant amount of data, which can be used in the future, for new research.

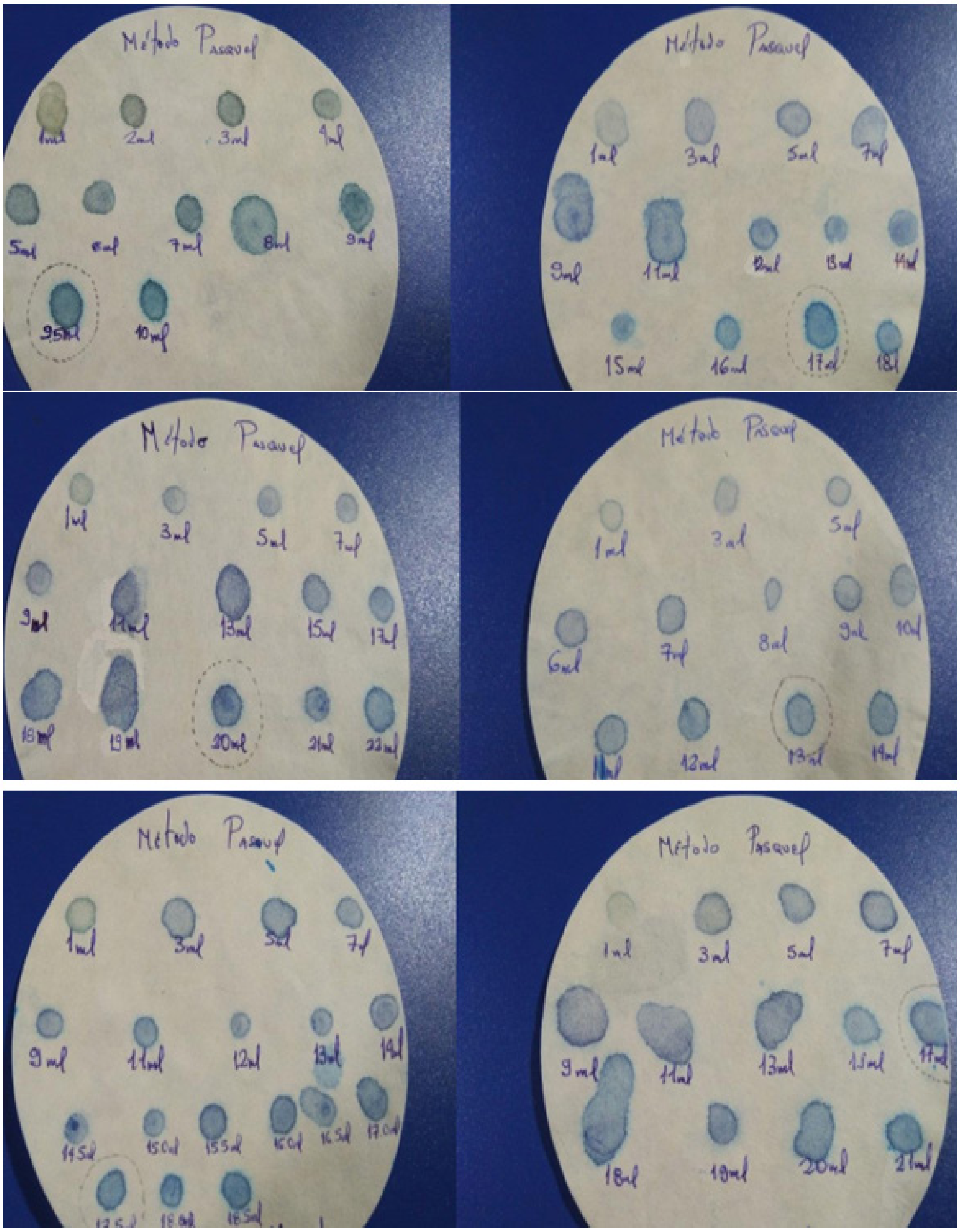

Figure 2: Images with the Watman Filter Papers,
used to calculate the Methylene Blue Value (ASTM):

3.3.1 Percentage of fines that pass the #200 mesh: Table 3 shows the classification of the five sands chosen, according to the % of penalties that pass the #200 mesh that was found

5.1.2 Classification of sands, according to the Fineness Module: Table 2 shows the classification of the five sands chosen according to the fineness modulus (FM) found.

Table 2: Classification of the sands, according to their fineness modulus

Origin of the Sand	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Average	Sand Classification	
Coarse	3.03	3.06	3.04	3.09	**	**	3.06	Medium Sand	FM< 3.1 > 2.4
Utility	3.22	3.19	3.17	3.16	**	**	3.19	Coarse Sand	FM > 3.1
River	3.52	3.49	3.49	3.52	**	**	3.51	Coarse Sand	FM > 3.1
Pit	3.82	3.82	3.87	3.9	**	**	3.85	Coarse Sand	FM > 3.1
Concrete	3.82	3.91	3.76	3.89	3.85	3.94	3.86	Coarse Sand	FM > 3.1

Table 1: Classification of sands, according to the natural moisture found

Origin of the Sand	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Average (%)	Sand Rating Approximate Humidity) (Range
Coarse	11.84	11.42	11.55	11.67	11.36	11.29	11.522	H < 12%% > 10%
Utility	6.16	6.15	6.27	6.22	6.24	6.18	6.203	H < 6.5%% > 5.5%
River	5.16	5.49	4.56	4.96	NA	NA	5.043	H < 5.5%% > 4.5%
Pit	1.67	1.69	1.73	1.71	1.65	1.67	1.687	H < 2.5%% > 1.5%
Concrete	0.9	0.92	0.84	0.96	NA	NA	0.905	H < 1.5%% > 0.5%

Table 3: Classification of the sands, according to the % that pass the #200 mesh

Origin of the Sand	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Average (%)	Sand Classification	
Coarse	2.49	2.53	2.52	2.46	2.48	2.4	2.48	Acceptable fine content	Pass Mesh # 200% < 6%
Utility	3.53	3.21	3.58	3.31	3.51	3.33	3.412	Acceptable fine content	Pass Mesh # 200% < 6%
River	4.27	4.34	4.64	4.48	4.43	4.3	4.41	Acceptable fine content	Pass Mesh # 200% < 6%
Pit	5.05	5.62	5.26	5.39	**	**	5.33	Acceptable fine content	Pass Mesh # 200% < 6%
Concrete	6.78	7.17	6.58	7.05	**	**	6.895	Excess fine content	Pass Mesh # 200% > 6%

Table 4: Classification of the sands, according to the AASHTO Method, to find the MBV

Origin of the Sand	Sample 1	Sample 2	Promedio ((mg/g	Sand Rating	
Utility	7.5	NA	7.5	Margi. Acceptable clay content	6mg/g < Clay Content < 12mg/g
Pit	5.75	NA	5.75	Excellent low clay content	Clay Content < 6mg/g
Coarse	7.75	NA	7.75	Margi. Acceptable clay content	6mg/g < Clay Content < 12mg/g
River	8.5	NA	8.5	Margi. Acceptable clay content	6mg/g < Clay Content < 12mg/g
Concrete	10	NA	10	Margi. Acceptable clay content	6mg/g < Clay Content < 12mg/g

3.3.2 Mortar cubes design and strength test: With Naphthalenesulfonate Technology Additive (Additive 1)

Table 6: Mortar with Minimum Dosage - After 7 days and Maximum Dosage - After 7 days of curing of the Mortar Cube - Additive 1

	Minimum Dosage - After 7 days			Maximum Dosage - After 7 days		
Type of Sand	Cement Type OPC	Cement Type PPC	Cement Type PLC	Cement Type OPC	Cement Type PPC	Cement Type PLC
Type of Sand	252.54	243.09	237.33	261.67	254.99	247.16
Pit Sand	247.76	240.39	233.43	253.85	243.27	237.52
River Sand	241.21	237.27	225.86	249.78	239.35	228.82

Table 5: Classification of sands, according to the ASTM Method, to find the MBV

Origin of the Sand	Sample 1	Sample 2	Average (%)	Sand Rating	
Utility	1088	**	1088	Tolerable clay content	Clay Content < 1.2% > 0.6%
Pit	0.576	**	0.576	Excellent low clay content	Clay content < 0.6%
Coarse	0.768	**	0.768	Tolerable clay content	Clay Content < 1.2% > 0.6%
River	0.833	**	0.833	Tolerable clay content	Clay Content < 1.2% > 0.6%
Concrete	1344	**	1344	Possible failure due to clay content	Clay Content < 2% > 1.2%

Table 7: Mortar with Minimum Dosage - After 28 days and Maximum Dosage - After 28 days of curing of the Mortar Cube - Additive 1

	Minimum Dosage - After 28 days			Maximum Dosage - After 28 days		
Type of Sand	Cement Type OPC	Cement Type PPC	Cement Type PLC	Cement Type OPC	Cement Type PPC	Cement Type PLC
Type of Sand	320.46	327.79	345.09	326.24	339.13	347.83
Pit Sand	313.54	323.19	332.54	322.49	330.8	338.87
River Sand	296.96	312.46	325.69	306.36	319.59	329.06

5.2.3.2. With Polycarboxylate Technology Additive (Additive 2)

Table 8: Mortar with Minimum Dosage - After 7 days and Maximum Dosage - After 7 days of curing of the Mortar Cube - Additive 2

	Minimum Dosage - After 7 days			Maximum Dosage - After 7 days		
Type of Sand	Cement Type OPC	Cement Type PPC	Cement Type PLC	Cement Type OPC	Cement Type PPC	Cement Type PLC
Type of Sand	241.56	229.45	224.32	243.48	237.39	228.37
Pit Sand	232.78	226.23	219.29	237.21	231.56	225.74
River Sand	227.98	218.72	207.87	230.34	223.71	214.45

3.4 With Lignosulfonate Technology Additive (Additive 3)

Table 10: Mortar with Minimum Dosage - After 7 days and Maximum Dosage - After 7 days of curing of the Mortar Cube - Additive 3

	Minimum Dosage - After 7 days			Maximum Dosage - After 7 days		
Type of Sand	Cement Type OPC	Cement Type PPC	Cement Type PLC	Cement Type OPC	Cement Type PPC	Cement Type PLC
Type of Sand	233.73	229.59	221.28	236.17	232.05	225.33
Pit Sand	227.91	222.84	214.29	231.39	226.14	220.58
River Sand	219.63	215.93	210.64	223.48	218.73	215.06

Table 9: Mortar with Minimum Dosage - After 28 days and Maximum Dosage - After 28 days of curing of the Mortar Cube - Additive 2

	Minimum Dosage - After 28 days			Maximum Dosage - After 28 days		
Type of Sand	Cement Type OPC	Cement Type PPC	Cement Type PLC	Cement Type OPC	Cement Type PPC	Cement Type PLC
Type of Sand	339.04	347.27	360.77	353.09	364.27	373.81
Pit Sand	333.47	343.41	353.94	339.31	347.53	362.64
River Sand	322.66	338.96	344.59	326.89	341.93	356.83

4. DISCUSSION

Moisture Content: Here, the classification of sands is analysed according to the natural moisture found. Remember that the results shown in Table 1, referring to the moisture content, are for reference since these will depend on the environmental conditions in which the sand was taken. The study was carried out to eliminate the natural moisture variable; it sought to bring all the sands to a natural moisture of 91%. Therefore, to improve the control of the mortar design process, all the sands were brought to a humidity between 8% and 10% to try to homogenise these five sands as much as possible before mixing them.

The Fineness Modulus (F.M.): The results shown in Table 2 show us that the Utility and Coarse Sands are the coarsest since they present an F.M. of 3.86 and 3.85, respectively; this is a value much higher than that recommended by the standard (3.10) and is even higher than the maximum limit allowed for use as construction sand (3.45). The Pit and River sands have a value of 3.50 and 3.19, respectively,

Table 11: Mortar with Minimum Dosage - After 28 days and Maximum Dosage - After 28 days of curing of the Mortar Cube - Additive 3

	Minimum Dosage - After 28 days			Maximum Dosage - After 28 days		
Type of Sand	Cement Type OPC	Cement Type PPC	Cement Type PLC	Cement Type OPC	Cement Type PPC	Cement Type PLC
Type of Sand	316.11	327.99	333.9	321.9	331.5	337.39
Pit Sand	306.13	318.34	325.59	315.11	323.06	330.56
River Sand	300.91	308.47	313.76	307.23	312.47	319.26

which could be considered within the range of the maximum permitted limit. Still, they are coarse sands compared to those used in construction. Finally, the Concrete sand, with an M.F. of 3.05, could be considered the only one among the five sands which is not coarse but intermediate (M.F. between 2.40 and 3.10), so a priori, one could think that it is an ideal sand for construction; but this would have to be verified with other physical and even chemical tests, which could show a more detailed and revealing picture.

The percentage of fines that pass the 200-Mesh: The results shown in Table 3 show that the Utility and Pit sands are those that present the lowest amount of fines since they present a % Passing the 200 Mesh of 2.48% and 3.41%, respectively; this is a value much lower than that recommended by the standard (6.00%) and is even lower than the maximum limit allowed for use as construction sand (4.50%). The Coarse and River sands present a value of 4.41% and 5.33%, respectively, which could be considered within the range of the limit recommended by the standard. Still, they already seem to have very high fines compared to those used in construction. Finally, the Concrete sand, with a %Pass the # 200 mesh of 6.89%, could be considered the only one among the five sands presenting a confirmed excessive quantity of fines. Still, again, this would have to be verified with the other physical and chemical tests and even with subsequent mechanical tests on the mortar cubes, which will show a more accurate picture.

Analysis of the chosen Indian cement: Analysing the properties of all the cement described above and comparing their physical-chemical characteristics, the following three types of cement were chosen a priori to design the mortars because they have the most differentiated features (they are at the extremes of the characteristics of different properties, such as, for example, regarding the alkali content, pozzolan content, resistance to sulphates, etc.). Still, the parameter on which the choice of the cement that is the subject of this study was the alkali content since it is a factor whose content dramatically influences the behaviour of the future mortar designed with this cement. For example, cement with high alkali contents will result in mortars with greater plasticising sensitivity, and cement with low alkali contents will result in mortars with more excellent resistance to fluidisation [15]. Three types of cement were chosen: Portland Pozzolana Cement (PPC) (with a relative alkali content equal to 1.44), Type V cement (with a relative alkali content equal to 0.48), and finally Ordinary Portland Cement (OPC) (with a relative alkali content equal to 0.80), necessary because being an intermediate cement in alkali content, it can be used to make a comparative analysis between the three cements.

Remember that cement particles are refined grains of irregular shapes with positive and negative charges, which are located within the layers of crystal that are being formed; so, when cement is mixed with water, the cement particles flocculate due to the electrostatic attraction between its particles with positive and negative charges; it is worth remembering that within the flocculated particles of hydrated cement, there are voids that trap part of the mixing water.

At this point, it is essential to make a pertinent clarification about the content of “alkalis” in cement; chemical analysis usually reports the content of alkalis as the sum of Na_2O and K_2O; however, as previously analysed, many alkalis come in the form of Na_2SO_4 and K_2SO_4; and it is with this structure that the alkalis can be quantified in their majority since this analysis gives us an excellent reference to know how much efficiency there will be in the cement-additive union; As mentioned above, cement with a low alkali content (also called cement with a low sulphate content) will have a high consumption of superplasticiser, but it will be unnecessarily lost in the co-precipitation zone. For this reason, at the same additive dose, cement with a high alkali content will provide better fluidity and workability results; this is because less additive will be lost within the co-precipitation zone (however, as explained later, a high alkali content will also cause mortars designed with this cement to accelerate their strength gain, so much so that at 7 days, mixtures designed with high alkalis have the highest strength, and at 28 days they have the lowest).

Alkalis are also closely related to the reactivity of cement. A cement with a high alkali content has less chemical reactivity and vice versa (because the alkalis internally balance the ionic zones of the cement, and it is also for this reason that all cement is bare in nature). Thus, the more reactive the cement, the greater the amount of additive it will absorb unnecessarily, decreasing its fluidising power in the long term and preventing the mixture from continuing to disperse over time, reaching the setting faster.

However, it should be noted that these effects are much more noticeable in superplasticisers with an electrical nature (Naphthalene and Lignosulfonates) and not in those with a steric nature, such as Polycarboxylates, where there was practically no difference when mixing the additive with all types of cement (with low and high alkali contents); this perhaps due to its nature, which does not involve many electrostatic ionic forces, which are the primary source of action of alkalis; For the same reason and consequently, it is assumed that there would not be a significant change when adding this type of additive, directly or delayed, to the mixture (unlike other kinds of superplasticisers, where it is assumed that there will be a relatively significant impact).

According to Rao, et al [14], the fluidity of the cement paste shows a good correlation with the slump of the concrete; therefore, in this case, the slump of the concrete can be considered to determine the fluidity of the paste. At this point, it is worth clarifying that obtaining a good performance in a paste, with a specific dosage, does not imply that the same good performance will be obtained in a mortar or in the same concrete designed with the same characteristics (this is because the chemical and physical properties of the aggregates can affect the rheological behaviour of the mortar and the concrete); However, the opposite is true, that is, if a poorly designed paste is obtained, with an incorrect dosage, the mortar designed with that dosage, and the subsequent concrete that may also be created, will also have poor rheological behaviour.

Below is the role played by each cement component in the rheological behaviour of the different types of pastes, mortars and concretes.

- C3A content in cement is high, and the availability of sulphate is low; mortars with or without additives experience high fluidity loss ratios; this is why cement containing moderate to high C3A (>9%) shows increases in fluidity losses compared to reference control mortar; however, to show one of the benefits of C3A, we can state that when there is less C3A available in the mix, superplasticisers tend to adsorb high amounts of C3S and C2S, resulting in a reduction in the future strength development of the mortar; despite this, many special Portland cement is currently used, which contain less than 10% aluminates in their interstitial phase (on average, 3.6% C3A and 6.9% C4AF.
- There is an excellent interaction of the calcium cation Ca^{+2} with the additives, especially in the internal structure of Naphthalenesulfonate, without having a significant influence on the technologies of the other two types of additives; however, it has been reported that adding small amounts of diluted sodium sulphate (Na_2SO_4) (0.2%) to the additive, this additive became much more effective, interacting with all types of cement, even with cement with low alkali content; achieving an even greater fluidity than that achieved by mixing cement with a high alkali content, with an additive without sodium sulphate [16]. It was found that the presence of this type of additive can decrease the consumption of the additive (it reduces the absorption of the additive by the cement), achieving an improvement in workability since there is a freer additive in the liquid interface, active to continue acting, keeping the paste or mortar fluid. Similar results were found in the literature [14-17], indicating that the Lignosulfonates (LS) form complex salts with Ca^{+2} more quickly than Naphthalenesulfonates.
- Alkalis in cement are essential to accelerate the hydration of C3S; however, an excess of alkalis could also cause adverse effects, one of them being the existence of secondary reactions that will form alkali aggregates.

- The problem with cement with low alkali contents can be compensated by adding an optimal content of soluble alkalis, mainly as metasilicate or in some other sulphated form. It was found that maximum fluidity can be obtained with a content of 0.4 to 0.5% of soluble alkali. At the same time, the long-term loss of workability of the concrete can be reduced by optimising its efficiency [18].
- Regarding cement fineness, it is well known that the finer the cement, the larger the specific surface area, and consequently, the water demand to obtain certain workability will be higher. Obviously, and as will be analysed later, the dosage and type of additive have the most significant effects on the cement and, therefore, on the rheological behaviour of the mixture. In addition, it is also known that almost all superplasticisers increase the duration of the inactivity period and slow down the cement hydration process. This process occurs mainly because high-range superplasticisers slow down the dissolution of Ca^{+2} and inhibit the crystallisation of Ettringite (which is what allows the initial setting).
- Regarding the temperature factor, it was found that cements containing low C3A content (or low C3A/SO3 ratio) were more sensitive to temperature variation in fluidity retention. Comparing with the characteristics of cement with high C3A content (or high C3A/SO_3 ratio) and in the opposite sense, cement with high equivalent alkali contents generated mixtures whose fluidity was more sensitive to temperature variations, this concerning mortars with low alkali contents. Also, similar investigations [22] show that the SO_3 content in cement clinker is highly correlated with the content of soluble alkalis, and it can also be deduced that the SO_3 content in clinker is mainly derived from solid and soluble alcohols; it also seems to be noted that during the first 30 minutes of mixing, the soluble alkalis dissolved almost wholly, for w/c ratios of approximately 0.50; so it can be assumed that up to this point, only some of the cement has been hydrated; To achieve a very high dissolution rate, the sulphate alkalis have

to be preferably as fine as possible, so that the chemical reactions preferentially occur on the surface of the cement particles (the rheological results corroborate these interactions).

This analysis of cement and superplasticiser interactions at ambient temperatures is the basis of rheology. It will help to understand the implications of the chemical composition of each phase in cementitious systems [19].

(**Methylene Blue Value** (MBV **4.1**

AASHTO Method: The results shown in Table 4 show that Concrete and River sands are those with the highest clay content since they have an MBV of 10.00 mg/g and 8.50 mg/g, respectively; this is a value much higher than that recommended by the standard, to have an excellent low clay content (6.00 mg/g), but it is lower than the maximum limit allowed, to be used as sand for construction (12.00 mg/g). The Coarse and Utility sands present an MBV of 7.75 mg/g and 7.50 mg/g, respectively, which could also be considered within the range of the maximum permitted limit. Still, they even present a lower amount of clay than the previous ones, although they need to reach the value of the excellent low content. Finally, the Pit sand, with an MBV of 5.75 mg/g, could be considered as the only one among the five sands which presents an excellent low clay content (less than 6.00 mg/g), so a priori, one could think that it is an ideal sand for construction; but as with all approximate results, this would have to be verified with the other tests, which will show a more detailed picture, such as the physical and mechanical tests.

ASTM Method: The results reported in Table 5 show that the Concrete and Utility sands are those with the highest clay content since they have an MBV of 1.344% and 1.088% respectively; this is a value much higher than that recommended by the standard, to have an excellent low clay content (0.6%); and in the case of the Concrete sand, its value is even higher than the tolerable limit of clay content (1.2%), which would prove its excessive content of fines, present in its structure. However, in the case of utility sand, its MBV is lower than the tolerable limit allowed to be used as sand for construction (1.2%). The River and Coarse sands present an MBV of 0.833% and 0.768%, respectively, so they could also be considered within the range of the permitted tolerable limit. Still, they present fewer clays than the Utility sand, although this value cannot be regarded as low content.

Finally, the Pit sand, with an MBV of 0.576%, could again be considered as the only one among the five sands which presents an excellent low clay content (less than 0.6%), so a priori, it could be thought again that it is an ideal sand for construction; but as in the previous case, being an approximate result, this would have to be verified with the other tests that will show a more detailed picture, such as the physical and mechanical tests, which will reinforce the results.

Finally, from both tests, comparing their results, the following three sands have been chosen a priori for having the most differentiated characteristics (they are at the ends of the table):

- Concrete Sand (It has, on average, the highest methylene blue value (MBV), which means that this sand has the highest amount of clay compared to the other 4).
- River Sand (It has, on average, an intermediate MBV value, which means that this sand has an intermediate amount of clay compared to the other 4).
- Pit Sand (It has, on average, the lowest methylene blue value (MBV), which means that this sand has the lowest amount of clay compared to the other 4).

4.2 Mortar and Mini Slump Design

Below is an analysis of all possible combinations of additives, cement and sand: 3 types of additives*5 additive dosages*3 types of cement*3 types of sand *2 samples for each combination; this makes a total of 270 kinds of mortars with different designs, which were analysed immediately after their preparation, to measure their fluidity and see their initial plasticity; subsequently, measurements were taken after 30, 60, 90 and 120 minutes, to verify how much they can delay their setting, that is, it was observed how long they could maintain their fluidity and manageability.

- A loss of fluidity (plasticising power) can be observed, depending on the additive content; the more significant the additive volume, the more fluid the mortar will be.

- A loss of fluidity (plasticising power) can also be observed, depending on the type of additive; that is, depending on the additive technology (Lignosulfonate, Naphthalenesulfonate or Polycarboxylate), the mortar will have greater or lesser fluidity, both in the short and long term.

- It can be observed preliminarily, from the tables shown above (with simple inspection), that the most potent additive, at least initially (at 7 min), is additive with Naphthalenesulfonate technology because it has the most excellent plasticising power in the short term.

- In addition, it can be observed preliminarily, from the tables shown above (with simple inspection) , that the additive that appears to be the most durable with Polycarboxylate technology, as it maintains its plasticising power for a longer time.

- It can also be observed preliminarily, from the tables shown above (by simple inspection), that the additive that appears to be the least powerful and the least durable is the additive with Lignosulfonate technology, as it has the lowest plasticising power; however, it is also observed that the behaviour of this additive is the one that varies the most, depending on the type of cement and sand used.

4.3 Mortar cubes design and strength test

After designing the cubes, they were broken by compression at 7 and 28 days, obtaining the results that were shown in Table 6 to Table 11; and now we will analyse how the additive behaved inside the mortar (according to the characteristics of each cement and sand); but no longer in its plastic state, but in its final state (after setting and hardening), so the strength that each mortar cube reaches has been measured.

Regarding the content or dosage of the Additives: At this point, as expected, the strength at both 7 and 28 days increased when the additive dose was increased (for all types of cement and sand), although only slightly. However, it was noted that this slight increase is seen with greater intensity in the polycarboxylates at 28 days (which can be explained by analysing the nature of this additive, which has a late action tendency; and that is why, precisely at 28 days, its effect will be seen, in terms of increasing resistance, in a more noticeable way).

Regarding the type of Additive: From the tables indicated above, it can be observed that the additive based on Naphthalenesulfonate is the one that presents more excellent resistance at 7 days (for all types of sand and cement), followed by the additive based on Polycarboxylate, and finally by the additive based on Lignosulfonate; However, this trend seems to be reversed when the analysis is done at 28 days, where the Polycarboxylate is the one that obtains the highest resistances, followed by the Naphthalenesulfonate, always leaving the mortars with Lignosulfonate at last (The explanation could be similar to the one given in the previous analysis, that is, due to the late effect of the additives based on Polycarboxylate, and the weaker effect that the Lignosulfonate additives have).

Regarding the time of the test: At this point, it was observed without exception, that in all cases at 28 days, there was an increase in the resistance in all the cement-sand-additive combinations; however, according to what has explained above, this increase varies according to each combination, reaching the mortars to obtain at 7 days, resistances of between 60 and 80% for the final resistances at 28 days; although in this case, the increase in resistance depends to a greater extent on the type of cement and sand; since even in mortars without additives, similar percentages of early resistance are observed (which is generally around 70%).

Regarding the type of sand: Observing the tables already indicated, it can be stated that in most cases, the mortars designed with coarser sands obtained more excellent resistance, both at 7 and 28 days, regardless of the type of cement and additive (this is due to the significant damage that the fine sands do, especially the clays, in the future crystallisation and setting of the mortar).

That is why the Pit sand is the one that obtains more excellent resistance at 7 and 28 days, followed by the River sand, and finally by the Concrete sand (the finest sand) since even if the best additive in the world is used, with the best type of cement; The clay quantity factor causes the mortar cube to have more significant imperfections, such as the presence of fissures, the presence of a more substantial number of internal and external pores (more permeable cube), etc., which weakens the cube, significantly reducing its resistance.

Regarding the type of cement: Finally, on the kind of cement, it can be stated by observing the tables specified initially that the cement with a lower amount of alkalis and C3A generated mortars with more excellent resistance, especially at 28 days, but this trend was opposite at 7 days; this is because a higher alkali content will develop a more excellent absorption of the additive at early ages (especially in the case of additives with Naphthalenesulfonate technology, since essentially, alkalis are chemical compounds based on sulphates and a more significant interaction is generated with them, even concerning Lignosulfonates).

Additionally, a higher C3A content generates early hydration of the cement, which produces a rapid gain in resistance, but which then becomes balanced until at 28 days, all the C3A has been consumed; instead, cements with a lower amount of this compound (but with a higher number of other components) will generate mortars with higher resistance (called "late" by some research), both at 28 days and at later dates.

That is why, at 7 days, the PPC is the one that presented the highest compressive strength, followed by the Ordinary Portland Cement (OPC), and then by the type V cement, although as explained above, this trend changes at 28 days; always leaving the Ordinary Portland Cement (OPC) (which is the most commonly used) in the intermediate zone, and now obtaining the highest resistance the Type V cement, which is why it is widely known as the "strongest" cement in the Indian market; noting that, in contrast to the case of PPC, the additive that improves the mortar behaviour the most is the additive in the form of Polycarboxylate (as the scarcity of alkalis in this type of cement positively influences the behaviour of this type of additive, which does not have sulphates in its structure).

All this final analysis was supported by multiple investigations related to this section, especially by Ramezananpor et al., who analysed in detail the effect that superplasticisers have on the future compressive strengths of cementitious mixtures [20]. In the following chapter, conclusions and recommendations of great importance will be provided, and perspectives on future topics that could be investigated will be shared.

5. Conclusions

Based on all the results obtained, the following conclusions are drawn, ranging from immediate to others requiring a higher level of analysis.

- From the current analysis results, a relationship between the intensity of fluidity (plasticising power) was observed, and the greater the volume of additive, the greater the fluidity of the mortar, and the additive content was observed. Order of the three additives found as additive 01<additive 02<additive 03.
- the gradient of fall will depend on the type of additive; that is, depending on the technology of each additive, the mortars will have a greater or lesser loss of fluidity.
- it can also be observed from the results that the most durable additive is additive 02, which maintains its plasticising power for a longer time, from which it can be concluded that this additive is the one that holds the most significant reserves in the aqueous interfaces of the mortars.
- on average, the least powerful and least durable additive is additive 3, which has the lowest plasticising power.
- the behaviour of additives is the most variable since it is very dependent on the type of cement and sand.
- the clay content in the sand dramatically influences the behaviour of the mortar since the finer the sand, the greater the amount of water it absorbs, and it generates drier mortars (working with the same humidity.
- From a chemical point of view, it can be concluded that when the sand is very fine (its particles almost reaching the limit size, close to that of clays), it has a larger surface area and can more easily attract the particles to the additive, generating a sand-ad-

ditive interaction (which does not improve plasticity); avoiding the cement-additive interaction, which will subsequently decrease the plasticity of the mortar, since it is precisely this interaction that generates the increase in fluidity and workability.

- Regarding the variation in the type of cement in mortars, it can be concluded that the more alkali and tricalcium aluminate (C3A) the cement has, the drier mortars will be obtained in the long term since the alkalis and C3A will cause good absorption of water on the surface and a faster setting (at two hours); except for the mixtures with Concrete sand, where it seems that the mortars acquire additional fluidity, due to the chemical characteristics of each constituent element; and also after 7 and 28 days, the mortars designed with these cement will obtain the lowest compressive strengths, compared to the other mortars.
- Although a higher content of alkalis and C3A generates greater initial fluidity and workability.

Finally, regarding the mechanical properties that the mortars will gain after 7 and 28 days, we can conclude that even observed in the corresponding tables, the incorporation of plasticisers or superplasticisers does not significantly affect the mechanical resistance of the mortar (as long as the water-cement ratio is maintained); according to Barabanshchikov et al [21], the essential mechanical benefit will lie in the improvement of the impermeability, and in the consequent increase in durability, of the mortar or equivalent concrete, in the long term.

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