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# 1 Feasibility of Using Marine Sediments in SCC Pastes as Supplementary 2 Cementitious Materials

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## 8 **Abstract**

9 In the context of the depletion of natural geomaterials, the increasing amount of dredged  
10 sediments calls for seeking new possibilities for treating and recycling these materials as  
11 cementitious supplementary materials. The aim of this paper is to design ecological SCC pastes,  
12 which require less cement, incorporating treated marine sediments. Despite many approaches  
13 investigating the ultimate formula of self-compacting concrete (SCC), the process remains complex  
14 because it is based on many variables and components. According to the mix design method, twenty  
15 pastes were prepared with cement, superplasticizer, water, and treated marine sediments from the  
16 Dunkirk harbour (France). The pastes' fresh properties were determined by using the mini-slump  
17 cone (workability), and the Marsh cone (fluidity). Rheological properties were determined with a  
18 robust rheometer based on the Bingham model. Cohesiveness and compressive strength were tested  
19 as well. All responses were connected using ternary diagrams, which led to the definition of an  
20 optimal formula. Experimental checking was performed to validate the obtained results.

21 **Keywords:** Sediments; Mix design; Rheology; Recycling; Cohesiveness; SCC pastes

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## 26 **1. Introduction**

27 In a vision of sustainability, and to limit the depletion of natural resources, reduce CO<sub>2</sub>  
28 emissions, and strengthen biodiversity, the adoption of the circular economy as a model of  
29 consumption has become a major necessity. Dredged sediments could be an alternative additive in  
30 concrete, as well as a new source of construction materials. Sediments have been considered as  
31 waste materials for a long time [1]. However, when subjected to particular treatment, they have  
32 been demonstrated to have pozzolanic properties that may allow them to be used with Portland  
33 cement as a reactive or a simple mineral additive in the production of concrete [2,3]. Dredged  
34 sediments can be regarded as a suitable alternative for greener concrete structures.

35 The type of cement, the presence of additives, the water-to-cement ratio, the different types of  
36 admixtures, the dosages, the type and the size of aggregates are some of the factors that determine  
37 the final quality of concrete. However, a suitable mix design is not enough to achieve the expected  
38 properties. Fresh concrete must have rheological properties allowing it to properly fill the space in  
39 the workform between the rebars with the lowest segregation. High workability and good  
40 rheological stability are required for concrete. That is why concrete must meet very strict  
41 performance requirements to ensure the expected service life of any structure, especially for  
42 reinforced concrete elements, where the use of self-compacting concrete (SCC) is increasing.

43 SCC is a very fluid material, homogeneous and stable, that perfectly fits the shapes of the  
44 most complex forms. It was designed by Okamura in Japan for the purpose of facilitating casting in  
45 pieces of complex geometry, or located in inaccessible areas set up under their own weight without  
46 vibration. Different approaches to mix design have been proposed in previous studies [4] such as:

- 47 ■ The empirical design method: Okamura proposed an empirical method based on laboratory  
48 tests by fixing different ranges for each component in the SCC concrete [5]. Khaleel and  
49 Abdul Razak proposed an empirical method for SCC based on metakaolin and coarse  
50 aggregates with different properties. Using this method, they found that metakaolin  
51 enhances the SCC properties [6].

- 52       ▪ The compressive strength method: Kheder and Al Jadiri proposed a method based on  
53       targeting a range of compressive strength by varying the water-to-cement ratio [7]. Dinakar  
54       et al. proposed a method started by fixing the total powder content; based on the required  
55       strength, the other parameters like W/C and fresh properties were then evaluated to meet the  
56       self-compactibility criteria [8].
- 57       ▪ The close aggregate packing method: Kanadasan and Razak proposed to use the particles  
58       packing concept to secure the fresh and the hardened properties of SCC [9]. This method  
59       helped to promote sustainability by incorporating palm oil clinker aggregate. Sebaibi et al.  
60       proposed a method based on optimizing the SCC concrete using RENE LCPC software [10].
- 61       ▪ The mixture design method based on the statistical factorial method: Bouziani developed a  
62       simplex-lattice mixture design with different factors and levels [11]. This study confirms  
63       that this approach is valid for a wide range of mixture proportions. Ozbay et al. used  
64       Taguchi's experiment design by using different factors [12]. This study analyzed the mixture  
65       proportion parameters of a high-strength SCC and it shows that the laboratory tests can be  
66       reproduced in a full-scale production.
- 67       ▪ The mixture design method based on the rheology of the paste model: Ferrara et al.  
68       proposed a method for formulating a steel fibre-reinforced SCC based on developing a  
69       rheological model for pastes [13]. The model proved to be an efficient tool for the  
70       optimization of the SCC mixture.

71       Recently, studies have been focusing on the rheological understanding of this material and it  
72       has been reported that the control of the rheological properties is fundamental to optimize the SCC  
73       [14–16]. The rheological properties depend on many factors such as: the packing density, the  
74       specific surface area, water content, and film thicknesses [17–21]. Other studies show that the main  
75       factors influencing the rheology are the water-to-powder ratio and the superplasticizer dosage  
76       [22,23]. Despite previous studies investigate if those parameters influence individually or jointly the  
77       rheology of cementitious paste, it is still difficult to tell [24].

78 Rheological models are mathematical tools used to characterize the behaviour of a material  
79 during flowing. The choice of a model is determined by the type of material, as well as the rate of  
80 deformation the material is subjected to. Many rheological models have been proposed for the  
81 cementitious matrix since the beginning of the rheology study [25,26], especially for the  
82 cementitious matrix [27,28].

83 The rheology of SCC can be described with the Bingham or Herschel–Bulkley model [22]. In  
84 this paper, the Bingham model is considered to be the most appropriate. It includes three intrinsic  
85 parameters ( $\tau_0$ ,  $k$ ,  $\eta$ ) that describe the steady-state flow of the material in a homogeneous condition  
86 (no particle segregation). The shear stress  $\tau$  (Pa) is then related to the shear rate  $\dot{\gamma}$  ( $s^{-1}$ ) using Eq. 1:

87 
$$\text{Eq. 1} \quad \tau = \tau_0 + \eta \cdot \dot{\gamma}$$

88 where  $\tau_0$  is the shear threshold of the material and  $\eta$  is the viscosity (Pa.s). Although some studies  
89 on SCC used the rheological Herschel–Bulkley model, some others demonstrated that the  
90 rheological model of Bingham is the most adaptable for use in the field of cementitious matrix [29–  
91 31]. The volume of the paste in SCC is much higher than in ordinary concrete, which leads to the  
92 use of more additives and admixtures. The composition of the paste is the main parameter affecting  
93 the self-compactibility properties [32].

94 The purpose of this work is to incorporate treated sediments as a replacement for Portland  
95 cement, and to evaluate their influence on the rheological behaviour of the mixture, which will have  
96 good environmental and economic benefits. This study is a part of a wider project on recycling  
97 sediments in SCC. This experimental study indicated that the volumetric amount of sediments in the  
98 mixtures can reach up to 14%, in other words, up to a sediments-to-cement-ratio of 0.29 in mass.

## 99 **2. Materials and methods**

### 100 **2.1. Test equipment**

101 The morphology of the treated sediments was analyzed using a Scanning Electron  
102 Microscope (SEM) Hitachi S-4700. The thermogravimetric analysis (TGA) was carried out with an

103 apparatus NETZSCH STA 449 using nitrogen gas in a controlled environment with argon flow (75  
104 ml/min) at variable temperatures (105 °C to 1100 °C, ramp rate = 2 °C/min).

105 A mini-slump test developed by Kantro [33], with the proportional dimensions of the standard  
106 slump test cone was used to determine the workability. Strong correlations between this test and the  
107 apparent viscosity [34,35], and, in certain cases, with the yield stress have been reported previously  
108 [36,37]. The fluidity and the consistency were determined with a 10-mm orifice Marsh cone. The  
109 outflow time through the cone was measured to fill 1000 ml.

110 A Vicat apparatus was used to determine the water demand of the sediments. It is equipped  
111 with a consistency probe of 10 mm in diameter, as described in NF EN 196-3 standard [38].

112 For the rheological analysis, a robust Anton Paar Modular Compact Rheometer MCR 102  
113 (Fig. 1) was used with a plane geometry. The shear rate varied from 1 to 100 s<sup>-1</sup>.

114 *Fig. 1. Anton Paar Modular Compact Rheometer MCR 102*

## 115 **2.2. Material properties**

116 The cement used in this study is an Ordinary Portland Cement (OPC) CEM I 52.5 N (standard  
117 NF EN 197-1 [39]). This cement is communally used in studies about recycling sub-products  
118 because it contains no admixtures, in order to measure the real effect of the sediments. The  
119 sediments were dredged from *Grand Port Maritime de Dunkerque* (GPMD), located in the North of  
120 France. The raw sediments were heated at 850°C for 1 hour, after having been crushed and sieved  
121 through 120 µm sieves [3] (Fig. 2). This technique was very efficient to eliminate the high-water  
122 content and organic matter, and to modify the mineralogy and the composition of the raw  
123 sediments. The physical properties of the powders are shown in Fig. 2. Process of treatment (from  
124 left to right: sediment after drying, after grinding, after thermal treatment)

125 Table 1. The distribution of particles is given in Fig. 3. X-ray fluorescence (XRF) analysis  
126 was performed on the cement, and on the raw and treated sediments. The results are provided in  
127 Table 2. One can note that the concentration of most of the oxides increased in the sediments after  
128 the treatment. Fig. 4 shows the mineralogical composition of the raw and treated sediments  
129 obtained from X-ray diffraction (XRD). Quartz (SiO<sub>2</sub>) and calcite (CaCO<sub>3</sub>) were found as the main  
130 phases for the raw sediments. The analysis also indicated some minor phases such as gypsum  
131 (CaSO<sub>4</sub>, 2H<sub>2</sub>O), haematite (Fe<sub>2</sub>O<sub>3</sub>), natrosilite (Na<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>) and halite (NaCl). After treatment, quartz

132 (SiO<sub>2</sub>), gehlenite (several forms), and anhydrite (CaSO<sub>4</sub>) were found as the main phases. The  
133 analysis also indicated some minor phases such as haematite (Fe<sub>2</sub>O<sub>3</sub>), augite, gypsum, and  
134 microcline (KAlSi<sub>3</sub>O<sub>8</sub>).

135 The morphology of treated sediments is shown in Fig. 5. Fig. 5, it was observed that it consist  
136 of fine angular particles. According to Fig. 6, EDX analysis confirms the XRD analysis and it  
137 shows that the mineralogical nature of treated sediments is mainly siliceous.

138 The demand of superplasticizer (S<sub>P</sub>) was measured using two types. The S<sub>P</sub> Chryso Fluid  
139 Optima 206 appears to be the most suitable and the saturation assay starts from 1.5 % of the S<sub>P</sub> dry  
140 extract to the cement mass.

141 The TGA analysis was conducted on the raw sediments. The results are presented in Fig. 7  
142 and it shows that:

- 143     ▪ Between 30 and 120 °C: the free water and some of the adsorbed water escape from the  
144         material. Unbound water is completely removed at 120°C
- 145     ▪ Between 130 and 170 °C: a double endothermic reaction can take place associated with the  
146         decomposition of gypsum (CaSO<sub>4</sub>.2H<sub>2</sub>O)
- 147     ▪ Between 180 and about 300°C: the first stage of dehydration. The heat breaks the particles  
148         and pulls the inter-granular water molecules
- 149     ▪ At about 250 and 370 °C: small endothermic peaks may be produced indicating  
150         decomposition and oxidation effects of metallic elements (haematite)
- 151     ▪ Between 450 and 550 °C: decomposition of the organic matter and organic pollutants
- 152     ▪ Around 570 °C: there is a structural transformation from quartz α to quartz β
- 153     ▪ Between 600 and 700 °C: this is the second step in the decarbonation of calcite.
- 154     ▪ Between 700 and 900 °C: a highly endothermic reaction due to the decomposition of  
155         limestone (CaCO<sub>3</sub>) that releases carbon dioxide (CO<sub>2</sub>) and calcium oxide (CaO)
- 156     ▪ Above 1000 – 1400 °C: the material goes to the mud state

157 The analysis confirms the choice of the calcination temperature, which is above the optimum of  
158 750°C.

159 *Fig. 2. Process of treatment (from left to right: sediment after drying, after grinding, after thermal*  
160 *treatment)*

161 *Table 1. Physical properties of cement and sediments*

162 *Fig. 3. Particle size distribution of cement, raw and treated sediments*

163 *Table 2. Oxide composition of cement, raw and treated sediments using XRF (%)*

164 *Fig. 4. XRD analysis on raw and treated sediments*

165 *Fig. 5. Particle morphology of treated sediments (300  $\mu\text{m}$ )*

166 *Fig. 6. Energy Dispersive X-ray (EDX) analysis of treated sediments*

167 *Fig. 7. TGA analysis on the raw sediments*

### 168 **3. Experimental protocols**

#### 169 **3.1. Packing density and water demand**

170 Water demand consists in determining the mass of water  $M_w$  required to make the  
171 cementitious paste go from the state of pellets to a state of homogeneous paste. Tests were carried  
172 out on 350 g of cement using a standard kneader (described in standard NF EN 196-1 [40]) and the  
173 Vicat apparatus. The optimisation of water demand was performed by using the Réne-LCPC, a  
174 developed software by Sedran and Larrard [41], which is based on a mathematical model in order to  
175 secure a maximum packing density of the skeleton. This model makes it possible to calculate the  
176 theoretical compactness of any granular mixture, and the viscosity of any suspension at a given  
177 concentration of this mixture, based on various material characteristics. Physically speaking, a  
178 higher compactness refers to a minimum percentage of voids between solid grains.



179 Penetration measurements were performed for different water contents. The mass of water  $M_w$   
 180 required to obtain a height  $h = 6$  mm can be determined by successive tests. In order to limit the  
 181 number of tests, it is possible to interpolate  $M_w$  from two points (A & B) framing the normal  
 182 consistency. However, in order to minimize the error, points A and B must fulfill the following  
 183 conditions (Eq. 2):

$$184 \quad \text{Eq. 2} \quad \begin{cases} H_B \geq 2 \text{ mm} \\ 0.25 \leq W/C \leq 0.4 \\ M_{WB} - M_{WA} \leq 5 \text{ g} \end{cases}$$

185 The compactness  $\emptyset$  of the powder is then determined by Eq. 3:

$$186 \quad \text{Eq. 3} \quad \emptyset = \frac{1000}{1000 + \rho_c \frac{M_w}{M_c}}$$

187 where  $\rho_c$  is the density of the cement,  $M_w$  and  $M_c$  are the total mass of water and cement,  
 188 respectively. The theoretical compactness ( $\emptyset$ ) was calculated using the commercial software René-  
 189 LCPC as it shown in Fig. 8. It represents that the amount of sediments up to 70% improve the  
 190 density of the mixture which can be explained by the finesse of the marine sediments and their  
 191 ability to fill into voids between the cement grains. The water demand for treated sediments was  
 192 0.4.

193 *Fig. 8. Theoretical packing density of the mixtures using René-LCPC software*

### 194 **3.2. Experimental design**

195 The design of experiments (DoE) approach is based on a statistical method that can be used to  
 196 optimize experimental tests. It is a well-known approach commonly used for the optimization of  
 197 cementitious mixtures [42–45]. It allows the measurement of multiple responses without extra  
 198 experiments. In our case, DoE allows us to quantify the influence of each mix parameter on the  
 199 fresh properties of the mixtures. Furthermore, the responses depend only on the proportions of the  
 200 compositions in the mixture and not on the mass or the volume.

201 The main characteristic of a random mixture is that the sum of all the components must be  
 202 equal to 1. The experimental plan was based on four factors: cement (C), sediments (S), water (W),  
 203 and superplasticizer (S<sub>p</sub>) taken in volumetric proportion. With a total volume equal to unity, the mix  
 204 design implies that there is an interaction and dependence between the parameters. The  
 205 experimental field was constrained by Eq. 4:

$$206 \quad \text{Eq. 4} \quad \sum_{i=1}^{i=n} x_i = C + S + W + Sp = 1$$

207 However, another important parameter was taken into consideration, namely the solid  
 208 volumetric concentration ( $\Gamma$ ). This is the ratio of the volume of solids to the total volume, which  
 209 comprises the solid particles coming from cement, sediments, and superplasticizer in dry extract.  
 210 This parameter is known to correlate with the yielded value [46]. It also correlates with the viscosity  
 211 [47], which increases with the increase of the volume fraction of solids. This fact is valid only at a  
 212 low shear rate and shear stress and for a  $\Gamma_{\max} = 0.62$  [48]. This limitation was confirmed elsewhere  
 213 [49]. It was recommended to keep the volume ratio below 0.60 to avoid resistance against shear  
 214 stress increase with the shear rate.

215 The mathematical model for this mix design converges toward a 2<sup>nd</sup> degree polynomial (Eq. 5),  
 216 which can be written in a matrix form (Eq. 7) as the following:

$$217 \quad \text{Eq. 5} \quad Y = \sum_{i=j}^k \beta_i x_j + \sum_{i<j} \sum_j \beta_{ij} x_i x_j + \varepsilon$$

$$218 \quad \text{Eq. 6} \quad [Y] = [X].[\beta] + [\varepsilon]$$

219 where Y is the response,  $x_i$  and  $x_j$  correspond to the volumetric proportions of the mix factors, and  
 220  $\beta_i$  and  $\beta_{ij}$  are the regression coefficients. This model is known as the Scheffé canonical polynomials,  
 221 widely used in mixture experiment applications [50,51]. This equation can be expanded to (Eq. 7):

$$222 \quad \text{Eq. 7} \quad Y = \beta_1.C + \beta_2.S + \beta_3.W + \beta_4.SP + \beta_{12}.C.S + \beta_{13}.C.W + \beta_{23}.S.W +$$

$$223 \quad \beta_{14}.C.SP + \beta_{24}.C.Sp + \beta_{34}.W.Sp$$

224 where  $[X]$  is the experimental matrix,  $[\beta]$  is the vector of the model coefficients, and  $[\varepsilon]$  is the vector  
225 of the experimental error. After some preliminary tests in the laboratory, the range of variation of  
226 each component was as follows (Eq. 8):

$$227 \quad \text{Eq. 8} \quad \begin{cases} 0 \% \leq S \leq 30\% \\ 0 \% \leq S_p \leq 2\% \\ 0.25 \leq W/C \leq 0.4 \\ 0.57 \leq \Gamma \leq 0.59 \end{cases}$$

228 Table 3 represents the range of variation of the different components.

229 *Table 3. Implicit constraints*

230 The field of study was a space of four dimensions, since there were four factors. The  
231 experimental matrix generated a hyper polyhedral. Numeric analysis was performed using a  
232 commercial software (Design Expert) dedicated for experimental mixture plans. The volume  
233 proportion of each component was calculated based on the given criteria.

### 234 **3.3. Parametric study**

235 The optimal experimental matrix was defined to achieve reliable and effective results. The  
236 dedicated software gives the mix proportions of a total of 20 points for modelling. Table 4 presents  
237 the determined arrangements of the 20 runs and their corresponding responses. Six mixtures were  
238 removed from the model design because they did not meet the criteria of self-compactibility (having  
239 a low workability and fluidity).

240 The water demand of the sediments was not considered in this arrangement. It was added  
241 separately for each mixture depending on the mass of sediments. The significance of the model  
242 (linear, quadratic, cubic, two-factor interaction, etc.), was investigated using robust software  
243 (Design-Expert and SPSS) and using the statistical model ANOVA (analysis of variances).

244 *Table 4. Mix proportions and fresh properties of the mixtures*

### 3.4. Preparation of specimens and Test methods

Mixture preparation was made according to NF EN 196-1 [40] procedure. Tests were conducted on fresh paste first. Using the mini slump flow diameter, the spreading out diameter ( $D_{\text{flow}}$ ) was measured on a horizontal glass plate after 1 and 5 min to determine the workability. The fluidity and the consistency were determined using the Marsh cone. The outflow time ( $T_{\text{flow}}$ ) was taken for 500 ml according to EN 445 standard [52]. The rheological analysis was performed between 5 and 7 min after starting the mixing operation.

The fresh pastes were poured in three 50-mm steel cubes, according to ASTM C 139 [53], and allowed to fill under its own weight. The specimens were removed from the moulds one day after casting, and were placed in curing water at a temperature of  $20 \pm 2$  °C. At 28 days, the compressive strength was measured in accordance with standard ASTM C109 [54].

Up to now there is no standard test method for evaluating the cohesiveness for a cementitious paste. However, Kwan has developed a protocol to measure this parameter using a mini version of the sieve segregation test [24,55]. The standard test consists in evaluating the sieve segregation index (SSI) by quantifying the portion of fresh SCC sample ( $4.8 \pm 0.2$  kg) passing through a 5 mm sieve, from a height of  $50 \pm 5$  cm. Kwan suggested instead, to pour about 400 g of paste onto a 0.3 mm sieve from a height of 300 mm. In this study this protocol was used to measure the cohesiveness of the different pastes.

## 4. Results and discussion

The results of all the tests are presented in Table 5.

*Table 5. Results of all the conducted tests*

### 4.1. Workability

The results of the ANOVA analysis on the  $D_{\text{flow}}$  are provided in Table 6. This model is characterized by a  $R^2$  of 0.923 and a predicted  $R^2$  (0.844) in this case is in fair agreement with the adjusted  $R^2$  (0.900). The Model F-value suggests that the model is significant. There is only a

270 0.01% chance that such F-value could occur due to noise. A p-value below 0.05 indicates that the  
271 model terms are significant. Values greater than 0.1 indicate that the model terms are not  
272 significant. The "Lack of Fit F-value" of 12.95 implies that the Lack of Fit is significant. There is  
273 only a 0.69% chance that such "Lack of Fit F-value" could occur due to noise.

274 Fig. 9 shows the slump flow responses in a ternary graph while fixing the cement proportion.  
275 For the same proportion of sediments (e.g. 0.112), the spread flow changes quickly from 140 to 185  
276 mm, which means that the sediments have no effect on this parameter. The spread flow was not  
277 affected by the water content either; 180 mm could be obtained for a water range of variation  
278 between 0.41 and 0.43. The orientation of hatching confirms this fact. Again, the  $S_p$  content appears  
279 to be the dominant parameter in this ternary combination. According to Gomes [56] the value  
280 considered satisfactory for the flowing is  $180 \pm 10$  mm.

281 *Table 6. ANOVA analyses of results for the  $D_{flow}$*

282 *Fig. 9. The counter plots for slump flow (mm): 2D and 3D plot*

## 283 **4.2. Flowability**

284 The linear model was a match for the flowability. The results of the ANOVA analysis on the  
285  $T_{flow}$  are presented in Table 7. This model is characterized by a  $R^2$  of 0.888 and a predicted  $R^2$   
286 (0.734) in this case is in fair agreement with the adjusted  $R^2$  (0.854). The Model F-value suggests  
287 that the model is significant. There is only a 0.01 % chance that such F-value could occur due to  
288 noise. A p-value below 0.05 indicates that the model terms are significant. The "Lack of Fit F-  
289 value" of 4.72 implies a 5.69 % chance that a "Lack of Fit F-value" could occur due to noise, which  
290 is very low.

291 Fig. 10 shows the Marsh flow responses in a ternary graph while fixing the cement proportion  
292 (four components generate a cubic form that is hard to analyze). While all three variables affect the  
293 fluidity, the  $S_p$  content appears to be the dominant parameter in a ternary combination.

294 According to previous studies that used mini-slump and Marsh cones [57–60], good paste  
295 properties correspond to a  $D_{\text{flow}} \geq 165$  mm and a  $T_{\text{flow}} \leq 40$ s, depending on the ratios of the  
296 concentration of solids to the total volume  $V_s/V_T$ , and of the volume of water to the total volume  
297  $V_w/V_T$ .

298 *Table 7. ANOVA analyses of results for the  $T_{\text{flow}}$*

299 *Fig. 10. The counter plots for Marsh flow [s]: 2D and 3D plot*

### 300 **4.3. Rheological properties**

301 The paste rheology was characterized using a rheometer and a plane geometry with variation  
302 of the shear rate ( $\dot{\gamma}$ ) from 1 to 100  $\text{s}^{-1}$ . The following properties were determined: yield stress ( $\tau_0$ ),  
303 shear stress ( $\tau$ ), and viscosity ( $\eta$ ). The temperature was kept constant at  $20 \pm 0.1$  °C. The results fitted  
304 perfectly the Bingham model (Table 5) with a coefficient of determination  $R^2 \geq 0.95$  as depicted in  
305 Fig. 11. This mixture can be characterized by a yield stress of 2.990 Pa and a viscosity of 1.163  
306 Pa.s.

307 A correlation analysis was performed using the commercial software SPSS 24. Spearman's  
308 correlation coefficient ( $\rho_{\text{Spearman}}$ ) was used to investigate the correlation between variables. The  
309 Spearman correlation is a non-parametric test that does not involve any assumptions about the  
310 distribution of the data.

311 Fig. 12 shows a moderate correlation between the  $V_w/V_C$  ratio and both viscosity and shear  
312 stress at a constant shear rate of 100  $\text{s}^{-1}$ . The shear rate decreases with the increase of  $V_w/V_C$  ratio  
313 ( $\rho_{\text{Spearman}} = -0.908$ ), and the viscosity decreases with the increase of  $V_w/V_C$  ratio ( $\rho_{\text{Spearman}} = -0.904$ ).

314 A correlation analysis between both Marsh flow and yield point with the mini-slump flow is  
315 shown in Fig. 13. There is a strong relationship between both properties  $T_{\text{flow}}$  and  $D_{\text{flow}}$  ( $\rho_{\text{Spearman}} = -$   
316 0.913), a low flowability generates a high workability and vice-versa. There is also a strong  
317 correlation between  $D_{\text{flow}}$  and the yield point ( $\rho_{\text{Spearman}} = -0.911$ ).

318 Fig. 14 presents a correlation between both  $D_{\text{flow}}$  and  $T_{\text{flow}}$  with the  $V_W/V_C$  ratio. There is a  
319 strong relationship between the  $V_W/V_C$  ratio and the fresh properties.  $D_{\text{flow}}$  increases along with the  
320  $V_W/V_C$  ratio ( $\rho_{\text{Spearman}}= 0.915$ ), and the  $T_{\text{flow}}$  decreases while increasing the  $V_W/V_C$  ratio ( $\rho_{\text{Spearman}}= -$   
321  $0.943$ ). It is found that the good results (low  $T_{\text{flow}}$  & high  $D_{\text{flow}}$ ) are associated with a  $V_W/V_C$  ratio  
322 greater than 1.

323 *Fig. 11. The data from the 2<sup>nd</sup> mix fitted perfectly the Bingham model*

324 *Fig. 12. Range of the variation of viscosity and shear stress corresponding to  $V_W/V_C$  variation*

325 *Fig. 13. Range of the variation of flowability and yield point corresponding to  $D_{\text{flow}}$*

326 *Fig. 14. Range of the properties of the pastes corresponding to  $V_W/V_C$  variation*

#### 327 **4.4. Cohesiveness**

328 The sieve segregation index (SSI) for all the mixtures is presented in Table 5. A low SSI  
329 refers to a high cohesiveness and vice-versa. SSI were lower than 10 % as recommended [55],  
330 which ensure that the SCC pastes have a good stability as a coherent mass.

#### 331 **4.5. Paste strength**

332 Results of the compressive strength tests conducted on the pastes specimens are shown in  
333 Table 5 and Fig. 15. The strength values are the average of three test specimens at the age of 28  
334 days. As it is seen, the compressive strength varies from 78 to 101 MPa and by fixing cement  
335 percentage to 0.442%, strength varies from 83 to 87 MPa. Treated sediments generated a low  
336 reduction in compressive strength. However, from the orientation of hatching, the  $S_p$  content  
337 appears to be the dominant parameter in this ternary combination. The compressive strength  
338 increases with the increase of  $S_p$  content. Compared to the reference paste, a compressive strength  
339 of 85 MPa is required.

340 *Fig. 15. The counter plots for compressive strength (MPa): 2D and 3D plot*

## 341 5. Mixture optimization and validation of the model

342 The multi-criteria technique in Design-Expert allows us to set an optimization based on the  
343 overlay plots. The objective of this study is to maximize the mass of incorporated sediments while  
344 maintaining a good property of the pastes performances. Derringer and Suich [61] developed a  
345 global desirability function, represented in Eq. 9.

$$346 \quad \text{Eq. 9} \quad D = (d_1^{r_1} \times d_2^{r_2} \times d_3^{r_3} \times \dots \times d_n^{r_n})^{\frac{1}{\sum r_i}}$$

347 The combination of the different criteria is optimum for a D value close to 1. In Eq. 9,  $r_i$  is the  
348 relative importance assigned to the response  $i$ . It is a comparative scale of weighing each of the  
349 resulting  $d_i$  varying from 1 to 5. Goals can be assigned to the variables as to the responses and can  
350 be one of the following: maximize, minimize, target, or in the range. Table 8 provides the  
351 characterisation of the optimization function.

352 Fig. 16 shows the zone of desirability according to the overlay plot method. It suggests that  
353 the volumetric amount of sediments in the mixture can reach up to 14 %. Table 9 represents the  
354 optimal paste proportions according to both functions.

355 To assess the representability, repeatability, reproducibility, rapidity, and sensitivity of the  
356 model, a series of experiments were conducted with the optimal mixture proportions provided in  
357 Table 9. Table 10 shows the results with their absolute relative deviation (ARD) [42,51]. ARD is a  
358 parameter expressed in %, as in Eq. 10. It measures the predictability of the model and should not  
359 exceed 10%.

$$360 \quad \text{Eq. 10} \quad \text{ARD (\%)} = \frac{\text{Experimental} - \text{Model}}{\text{Experimental}} \times 100$$

361 According to Table 10, the results of the ARD are in the desirable range (less than 10 %). The  
362 model is validated by the small difference between the theoretical and the experimental values. This  
363 experimental study indicated that the volumetric amount of sediments in the mixtures can reach up



364 to 14 %, in other words, sediments can replaces cement up to 0.29 as a sediments-to-cement-ratio,  
365 0.30 as a water-to-cement-ratio, and 1.5 % of  $S_p$  dosage in the extract sec of cement mass.

366 *Table 8. Characterization of the desirability function*

367 *Fig. 16 . Overlay plot optimization following the proposed criteria*

368 *Table 9. Composition of the optimal pastes*

369 *Table 10. Absolute relative deviation of the predicted responses*

## 370 **6. Conclusion**

371 The production of cement is a major concern to the industrial and scientific community. The  
372 process of producing 1 ton of cement releases around 0.6 to 1.4 tons of  $CO_2$  depending on the  
373 process in each country [62–66]. The replacement of a part of the cement by treated sediments has a  
374 major economic and environmental impact. Treated sediments can play a relevant role in  
375 sustainability.

376 This paper focuses on the study of the effect of treated sediments on the rheological, fresh,  
377 and hardened properties of SCC pastes. The results demonstrated that sediments can be used as a  
378 supplementary cementitious material, with a volumetric amount up to 14 % ( $S/C = 0.29$ ). At the  
379 economical level, this result is very encouraging especially that the used sediments are naturally  
380 available in large quantities and require less energy in the treatment comparing to the cement.

381 A mix design method based on the statistical mixture design approach was used in order to  
382 evaluate the influence of each component of the mix. A numerical optimization efficiently  
383 accomplished made it possible to design an SCC paste based on treated sediments. The statistical  
384 analysis of the results highlighted the real relevance of the measured effects on the various factors,  
385 as well as their interactions on the responses. The effects of the factors and their interactions were  
386 more significant than the uncertainties.

387 The treated sediments improve the packing density of the cementitious materials. The  
388 empirical tests on fresh properties performed on the pastes correlate perfectly with the rheological  
389 parameters of the SCC pastes, namely the shear stress and the viscosity. The rheological tests  
390 performed less than 7 min after the start of mixing confirmed that the rheological behaviour of the  
391 pastes evolves from young ages.

392 The developed mini version of sieve segregation test is very promoting protocol that can be  
393 used as a foundation for a standardized test. Further research in this direction is suggested.

394 The use of different superplasticizers and viscosity agents may affect the repeatability of the  
395 results.

396 An extensive research is going on at our laboratories in order to evaluate the effect of  
397 entrained air and viscosity agent on a SCC mixture incorporating treated sediments.

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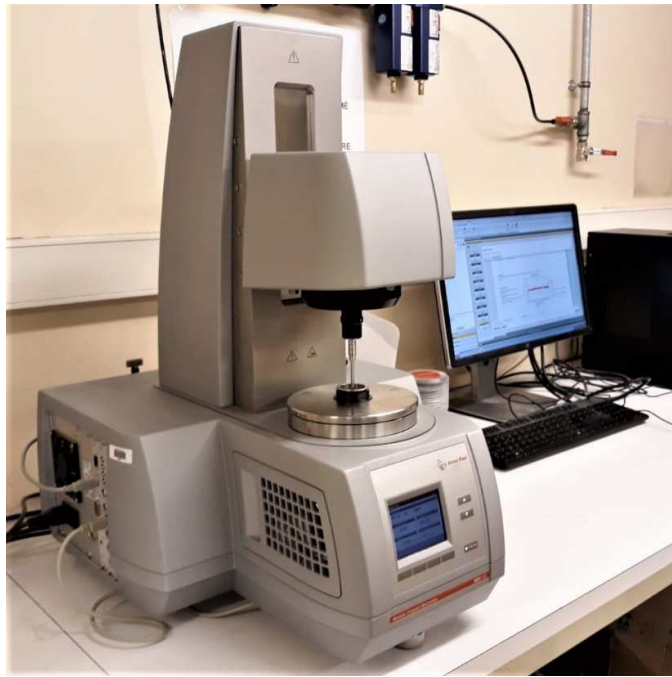
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*Fig. 1. Anton Paar Modular Compact Rheometer MCR 102*



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*Fig. 2. Process of treatment (from left to right: sediment after drying, after grinding, after thermal treatment)*

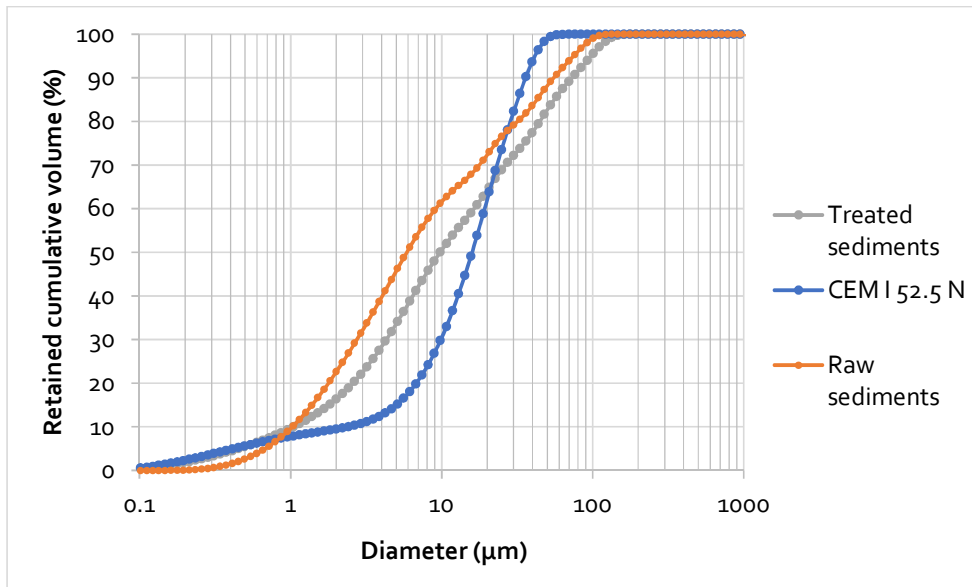
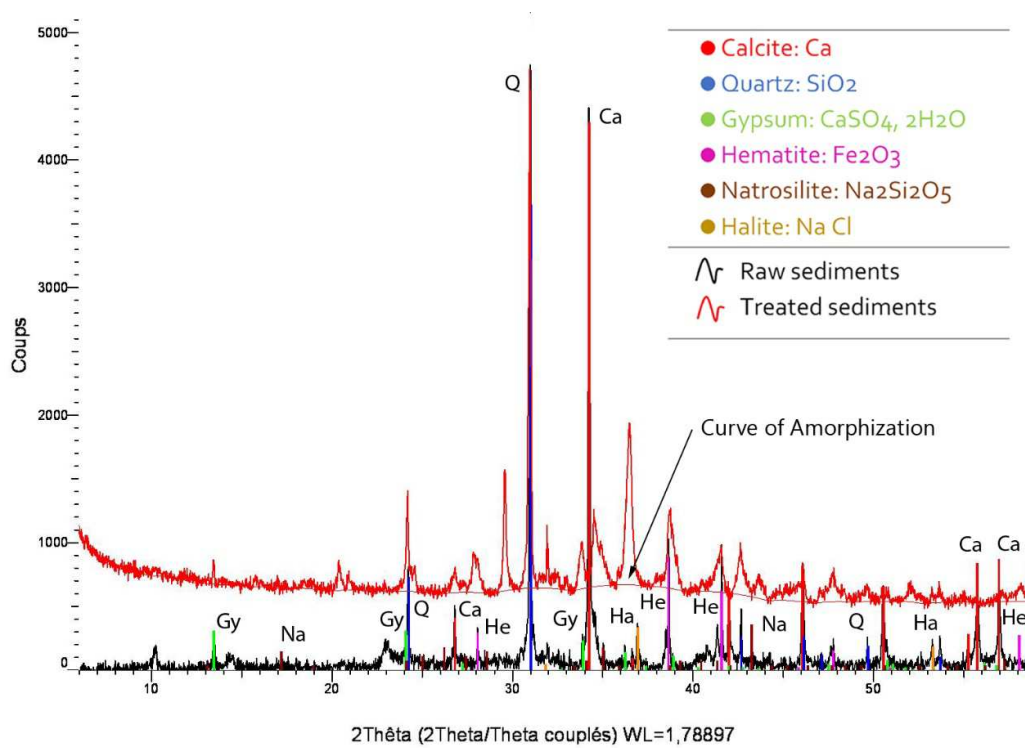


Fig. 3. Particle size distribution of cement, raw and treated sediments

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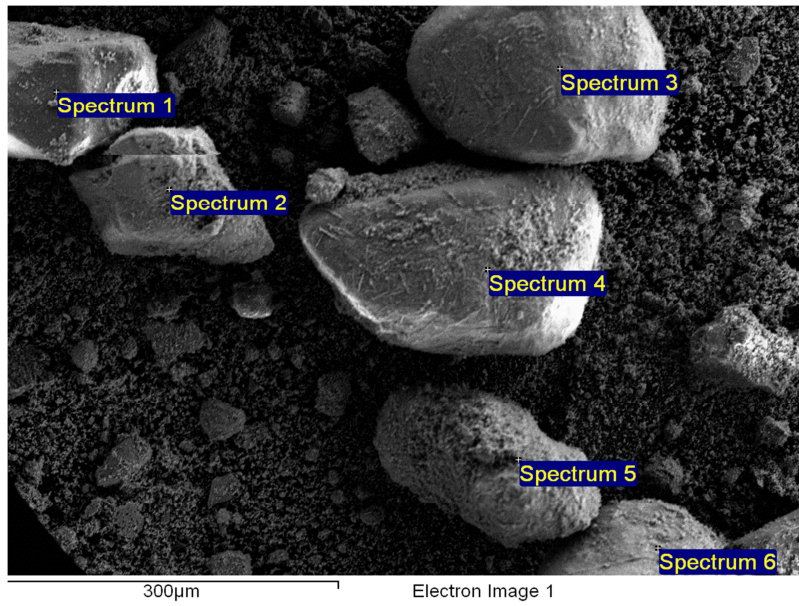


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*Fig. 4. XRD analysis on raw and treated sediments*

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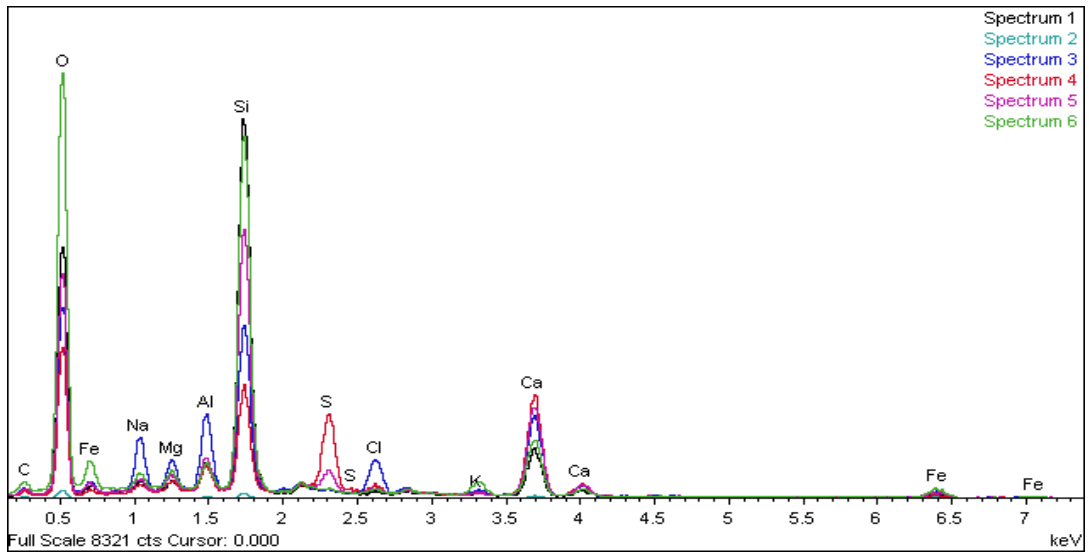


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*Fig. 5. Particle morphology of treated sediments (300 µm)*

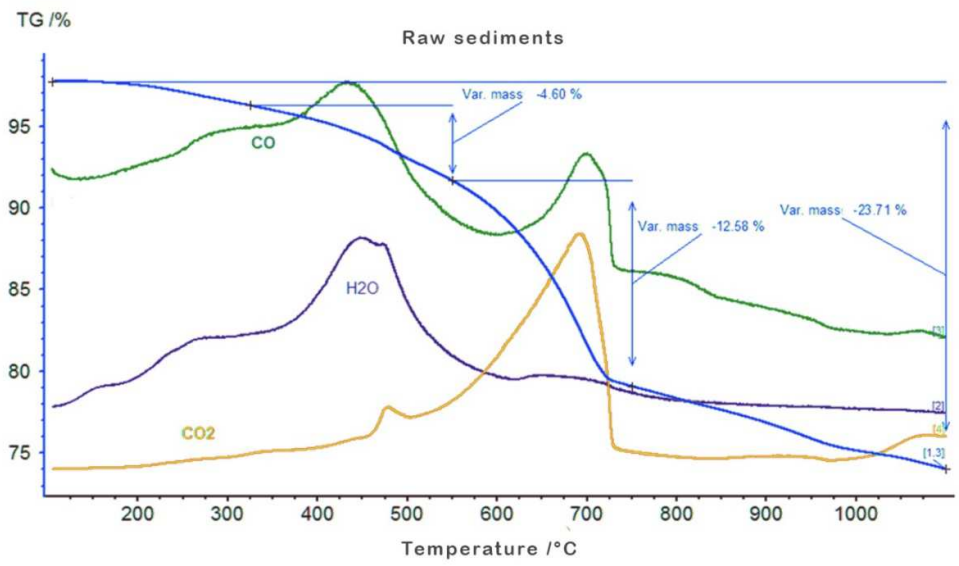


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*Fig. 6. Energy Dispersive X-ray (EDX) analysis of treated sediments*

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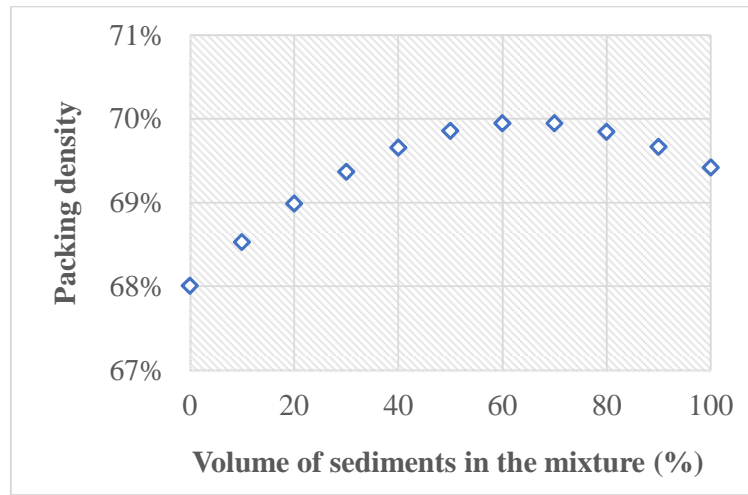


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Fig. 7. TGA analysis on the raw sediments



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*Fig. 8. Theoretical packing density of the mixtures using René-LCPC software*

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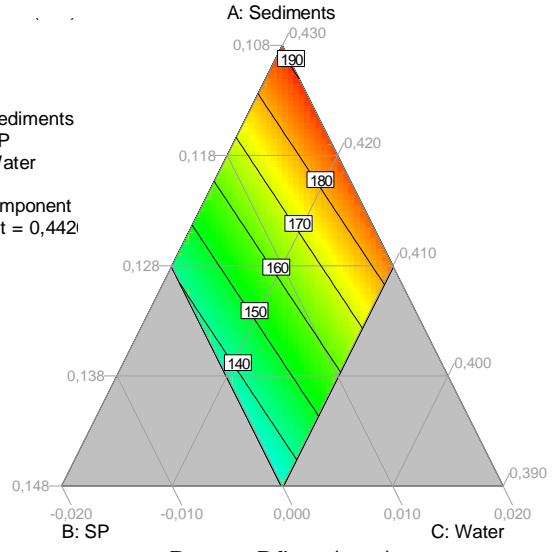
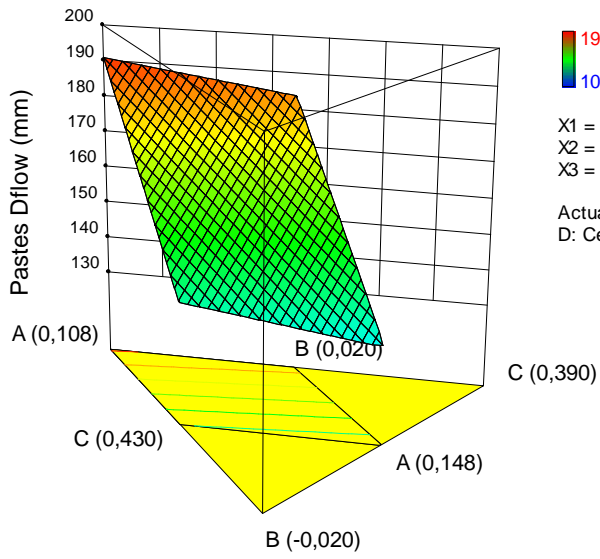
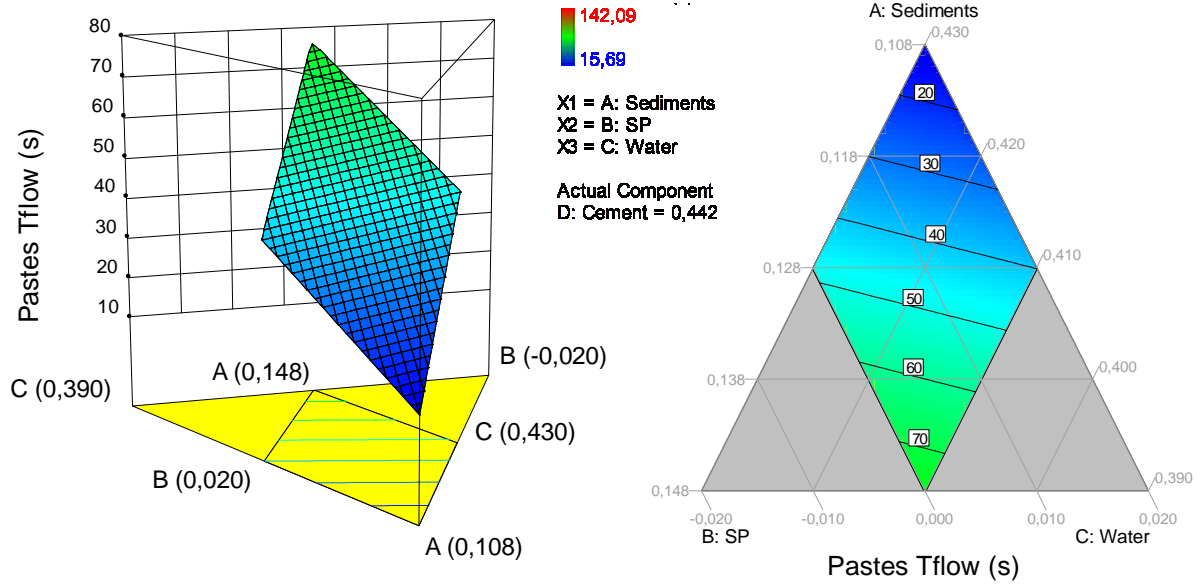


Fig. 9. The counter plots for slump flow (mm): 2D and 3D plot

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*Fig. 10. The counter plots for Marsh flow [s]: 2D and 3D plot*

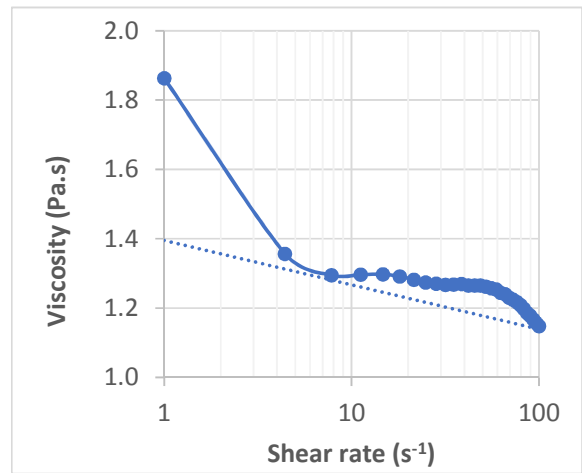
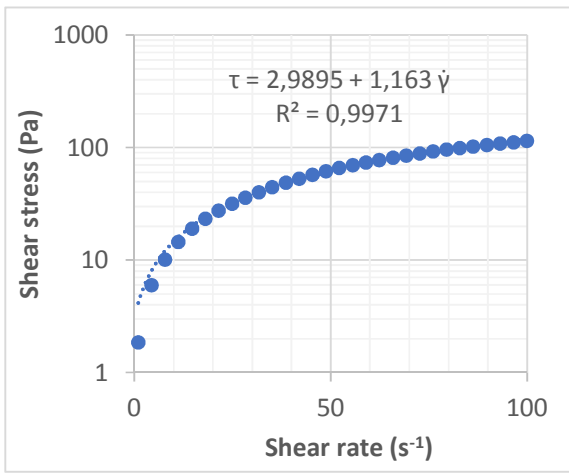
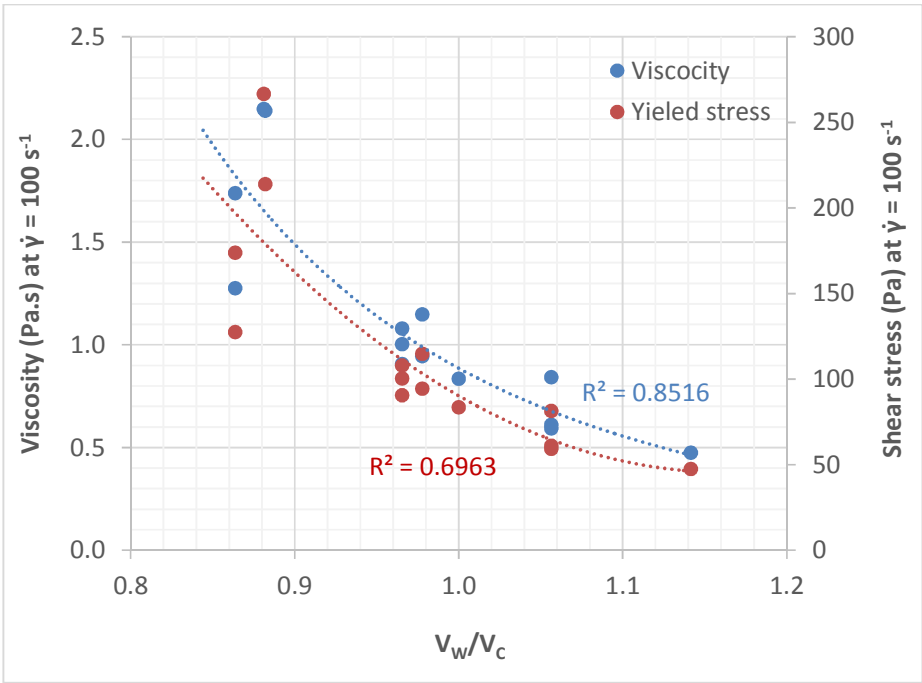


Fig. 11. The data from the 2<sup>nd</sup> mix fitted perfectly the Bingham model

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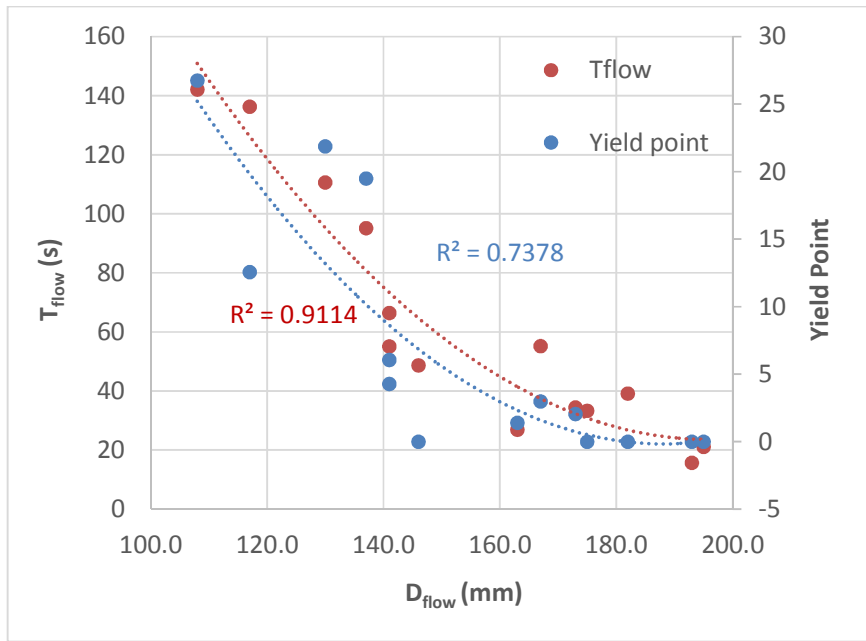


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*Fig. 12. Range of the variation of viscosity and shear stress corresponding to  $V_w/V_c$  variation*

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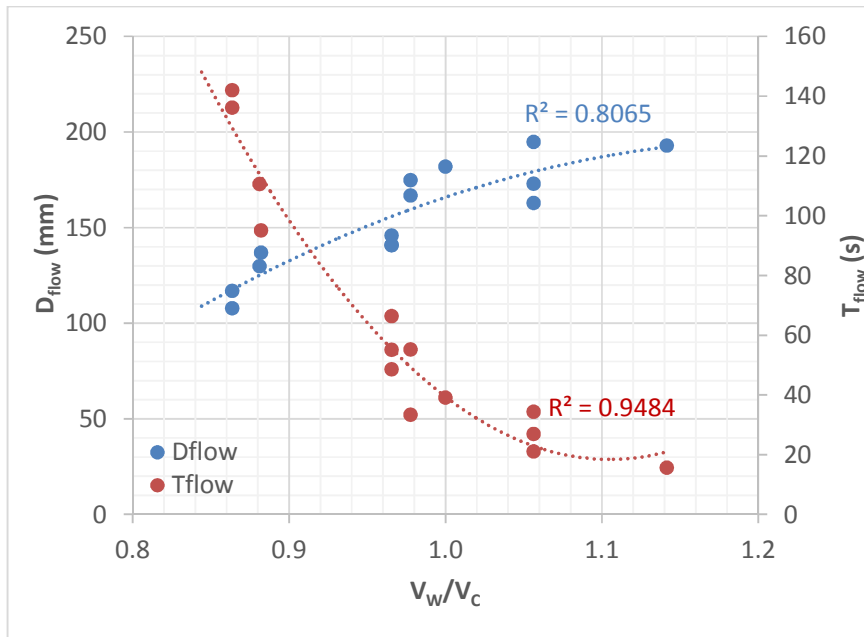


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Fig. 13. Range of the variation of flowability and yield point corresponding to  $D_{flow}$

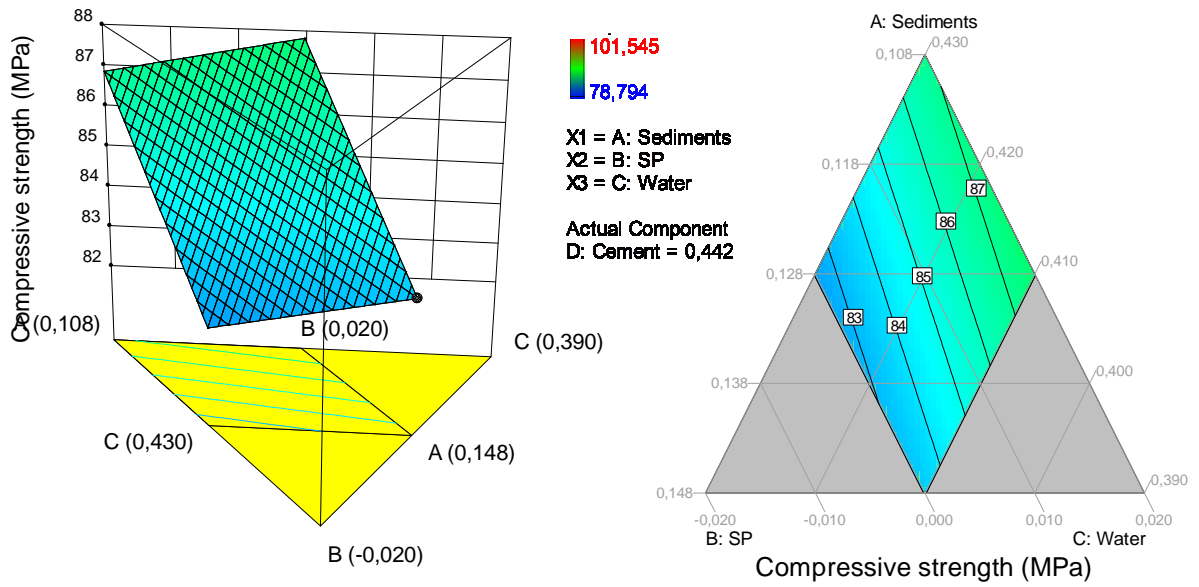


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Fig. 14. Range of the properties of the pastes corresponding to  $V_w/V_c$  variation

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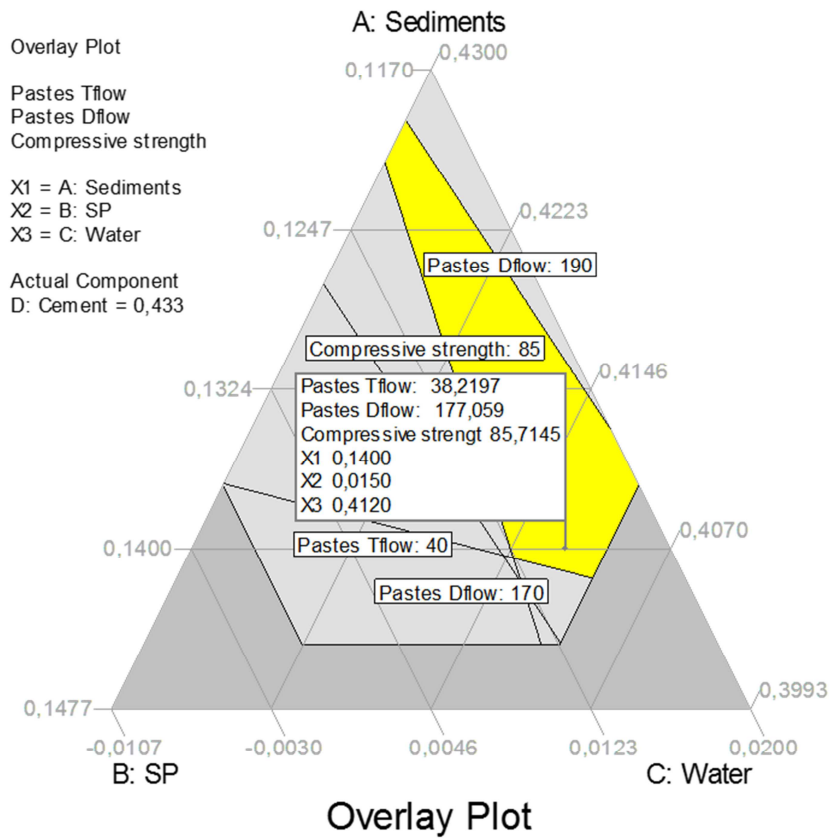


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Fig. 15. The counter plots for compressive strength (MPa): 2D and 3D plot



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Fig. 16 . Overlay plot optimization following the proposed criteria



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*Table 1. Physical properties of cement and sediments*

<b><i>components</i></b>	<b><i>Density (kg/m<sup>3</sup>)</i></b>	<b><i>Specific area BET (m<sup>2</sup>/kg)</i></b>	<b><i>Average diameter D<sub>50</sub> (μm)</i></b>
<i>Cement</i>	3176	914	15.9
<i>Raw sediments</i>	2520	3652	5.90
<i>Treated sediments</i>	2851	2335	9.80

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Table 2. Oxide composition of cement, raw and treated sediments using XRF (%)

<b><i>Oxides composition</i></b>	<b><i>Raw sediments</i></b>	<b><i>Treated sediments</i></b>	<b><i>CEM I 52.5 N</i></b>
<i>SiO<sub>2</sub></i>	38.8	36.4	17.3
<i>Al<sub>2</sub>O<sub>3</sub></i>	11.0	10.1	5.41
<i>MgO</i>	2.69	2.62	1.23
<i>Fe<sub>2</sub>O<sub>3</sub></i>	13.3	12.7	4.09
<i>CaO</i>	23.3	26.0	62.8
<i>Na<sub>2</sub>O</i>	2.08	2.31	0.71
<i>K<sub>2</sub>O</i>	2.08	1.65	0.76
<i>P<sub>2</sub>O<sub>5</sub></i>	0.51	0.47	0.49
<i>SO<sub>3</sub></i>	4.37	5.43	4.49
<i>TiO<sub>2</sub></i>	0.54	0.51	0.35
<i>MnO</i>	0.17	0.18	Traces
<i>ZnO</i>	0.20	0.17	0.13

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*Table 3. Implicit constraints*

<b><i>Component volume</i></b>	<b><i>S</i></b>	<b><i>S<sub>P</sub></i></b>	<b><i>E</i></b>	<b><i>C</i></b>
<i>Coded</i>	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>
<i>Lower constraints</i>	0.009	0.000	0.410	0.412
<i>Higher constraints</i>	0.148	0.020	0.430	0.541

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Table 4. Mix proportions and fresh properties of the mixtures

Run	Percentage of mixture volume				Relevant parameters				
	Sediments	$S_P$	Water	Cement	W/C	S/C	$V_W/V_P$	$S_P/C\%$	$V_W/V_C$
1	0.09	0.02	0.42	0.47	0.24	0.16	0.79	2.10	0.93
2	0.09	0.02	0.42	0.47	0.24	0.16	0.79	2.10	0.93
3	0.06	0.01	0.41	0.52	0.23	0.11	0.72	1.05	0.81
4	0.05	0.00	0.42	0.53	0.23	0.09	0.72	0.00	0.79
5	0.15	0.00	0.41	0.44	0.22	0.30	0.69	0.00	0.93
6	0.08	0.01	0.43	0.48	0.25	0.15	0.79	1.05	0.92
7	0.12	0.02	0.42	0.45	0.23	0.23	0.75	1.58	0.95
8	0.14	0.01	0.42	0.43	0.24	0.30	0.75	1.05	1.01
9	0.08	0.01	0.43	0.48	0.25	0.15	0.79	1.05	0.92
10	0.03	0.02	0.42	0.53	0.24	0.05	0.79	2.10	0.83
11	0.03	0.00	0.43	0.54	0.24	0.05	0.75	0.00	0.79
12	0.14	0.01	0.42	0.43	0.24	0.30	0.75	1.05	1.01
13	0.08	0.01	0.42	0.50	0.23	0.15	0.72	0.53	0.84
14	0.01	0.02	0.43	0.54	0.25	0.01	0.82	2.10	0.83
15	0.14	0.02	0.43	0.41	0.25	0.30	0.82	2.10	1.09
16	0.06	0.01	0.41	0.52	0.23	0.11	0.72	1.05	0.81
17	0.08	0.01	0.43	0.48	0.25	0.15	0.79	1.05	0.92
18	0.14	0.00	0.43	0.43	0.24	0.30	0.75	0.00	1.01
19	0.11	0.00	0.41	0.48	0.22	0.21	0.69	0.00	0.86
20	0.14	0.02	0.41	0.43	0.23	0.30	0.75	2.10	1.01

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Table 5. Results of all the conducted tests

Mix N <sup>o</sup>	Mini-slump diameter (mm)		March cone flow (s)	Yield point $\tau_0$ (Pa)	Viscosity $\eta$ (Pa.s)	ISS (%)	Dry weight (kg/m <sup>3</sup> )	Compressive strength (MPa) 28d
	After 1 min	After 5 min						
1	175	182	33.38	0.007	0.964	4.4	2110	97.6
2	167	170	55.28	2.990	1.163	5.9	2116	94.8
3	117	118	136.3	12.57	1.183	4.4	2150	95.3
<del>4</del>	-	-	-	-	-	-	-	-
<del>5</del>	-	-	-	-	-	-	-	-
6	141	143	66.47	6.051	0.853	2.0	2118	96.8
7	182	185	39.13	0.003	0.839	5.9	2083	90.0
8	173	173	34.47	2.052	0.600	4.5	2092	83.1
9	141	142	55.16	4.285	0.973	1.2	2125	82.3
10	137	138	95.16	19.50	2.186	1.2	2170	82.4
<del>11</del>	-	-	-	-	-	-	-	-
12	163	164	26.94	1.395	0.586	0.8	2103	80.4
<del>13</del>	-	-	-	-	-	-	-	-
14	130	130	110.7	21.89	2.448	1.3	2187	99.9
15	193	195	15.69	0.007	0.460	9.8	2062	78.8
16	108	108	142.1	26.76	1.519	1.2	2106	90.5
17	146	146	48.68	0.007	1.052	1.8	2117	88.9
<del>18</del>	-	-	-	-	-	-	-	-
<del>19</del>	-	-	-	-	-	-	-	-
20	195	196	21.16	0.004	0.856	8.1	2085	87.5

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Table 6. ANOVA analyses of results for the  $D_{flow}$

<i>Source</i>	<i>Sum of Squares</i>	<i>df</i>	<i>Mean Square</i>	<i>F- Value</i>	<i>p-value Prob &gt; F</i>
<i>Model</i>	8847	3	2949	40.06	< 0.0001
<i>Linear Mixture</i>	8847	3	2949	40.06	< 0.0001
<i>Residual</i>	736.2	10	73.62		
<i>Lack of Fit</i>	683.4	5	136.7	12.95	0.0069
<i>Pure Error</i>	52.79	5	10.56		

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Table 7. ANOVA analyses of results for the  $T_{flow}$ 

<i>Source</i>	<i>Sum of Squares</i>	<i>df</i>	<i>Mean Square</i>	<i>F-value</i>	<i>p-value Prob &gt; F</i>
<i>Model</i>	20262	3	6754	26.41	< 0.0001
<i>Linear Mixture</i>	20262	3	6754	26.41	< 0.0001
<i>Residual</i>	2556	10	255.7		
<i>Lack of Fit</i>	2110	5	422.0	4.72	0.0569
<i>Pure Error</i>	447.2	5	89.43		

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*Table 8. Characterization of the desirability function*

<b><i>Response and variables</i></b>	<b><i>lower</i></b>	<b><i>Upper</i></b>	<b><i>Criteria</i></b>
<i>Workability (mm)</i>	170	190	In range
<i>Flowability (s)</i>	15.6	40.0	In range
<i>Cohesiveness (ISS %)</i>	0.00	10.0	In range
<i>Compressive strength (MPa)</i>	85.0	99.9	In range

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Table 9. Composition of the optimal pastes

<i>Mixes</i>	<i>Component</i>				<i>Relevant parameters</i>			
	<i>C</i>	<i>S</i>	<i>S<sub>P</sub><sup>1</sup></i>	<i>W<sup>2</sup></i>	<i>W/C</i>	<i>S/C</i>	<i>V<sub>w</sub>/V<sub>P</sub></i>	<i>V<sub>w</sub>/V<sub>C</sub></i>
<i>Volume proportions (%)</i>	0.433	0.140	0.015	0.412	0.30	0.29	0.75	1.00
<i>Dosage (kg/m<sup>3</sup>)</i>	1375	399.2	15.00	412.0				

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<sup>1</sup> In dry extract

<sup>2</sup> Plus the water demand of the sediments

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*Table 10. Absolute relative deviation of the predicted responses*

<b><i>Response and variables</i></b>	<b><i>Theoretical values</i></b>	<b><i>Experimental values</i></b>	<b><i>ARD (%)</i></b>
<i>Workability (mm)</i>	177	174	4.1
<i>Flowability (s)</i>	38.2	34.7	9.5
<i>Cohesiveness (ISS %)</i>	7.55	7.12	6.0
<i>Compressive strength (MPa)</i>	85.7	81.2	5.5

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