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Robustness of cement-based materials:

from dosage variations to yield stress fluctuations

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Abstract

The idea behind the present work is to assess robustness of fresh cement-based materials (*i.e.* their ability to display the same properties when submitted to variations in the way they are produced) from a generic and analytical point of view. First, analytical models from literature relating components proportions and yield stress are combined into a multi-scale and physical approach and compared to our experimental yield stress measurements. In the second part, the derivation of these analytical relationships is used to assess robustness as a function of components proportions, which allows for the most sensitive variations to be mapped onto component dosage from a yield stress point of view. In the case of fluid concretes, this approach showcases the interest of working at slightly higher water content and slightly lower aggregates fraction along with benefits of viscosity modifying agents / plasticizers combination.

30

31 **Keywords:** Fresh Concrete (A); Robustness; Yield stress; Rheology (A); Admixture (D)

32 **1. Introduction**

33 In the construction industry, the term “robustness” captures the ability of a product
34 to display the same properties when submitted to variations in the way it is
35 processed or used. However, when it comes to concrete, robustness most often
36 represents the ability of the material to display the same properties when submitted
37 to either some variations in constituent parts physical and chemical properties or
38 some variations in constituent parts dosage.

39 In most academic or industrial research laboratories, mix design and sample
40 production are carried out using only one unique cement batch. Aggregates are
41 stored in controlled environmental conditions. Dosing and weighting uncertainties
42 are low compared to industrial practice. For a given mixing process, most
43 measurements standard deviations are therefore low. Such low standard deviations
44 allow for the assessment and study of tiny variations in macroscopic properties
45 typical of today’s research. As a consequence, robustness at the laboratory stage is
46 often a fully ignored feature of a given mixture.

47 The large volumes of raw material required for industrial concrete production
48 mandates the use of successive batches of cement and aggregates, increasing the
49 variation in particle size distribution and fine content. In addition, storage in most
50 often uncontrolled environmental conditions exacerbates control issues and
51 variations in aggregate water content in particular. The accuracy in constituent
52 materials weighing is far lower than in the laboratory. All the above results in higher
53 variations in constituent parts proportions. In turn, these larger variations in
54 constituent parts proportions and properties may induce larger variations in the
55 resulting rheological properties of the fresh material. Consequently, formulations
56 that had seemed fully adequate in terms of macroscopic properties in the laboratory
57 may prove difficult to steadily produce on a daily basis at an industrial level. For
58 instance, Ultra-High Performance Concrete (UHPC) [1] and Self-Compacting Concrete
59 (SCC) [2] are recent examples of advanced cement-based materials, the commercial
60 development of which has been hampered by, among others, some robustness

61 issues [3, 4].

62 Robustness of a given cement-based mixture is however an extremely delicate
63 feature to tackle, as it often requires numerous experiments in order to capture
64 variations in macroscopic behavior induced by variations in constituent parts
65 properties and dosages. The most sensitive (or most critical) constituent properties
66 or proportions may vary from one mix design to another. For instance, low W/C ratio
67 concretes are far more sensitive to water content variations than high W/C ratio
68 mixes whereas low slump concretes can be more sensitive to sand/gravel ratio than
69 standard flowable concretes.

70 The idea behind the present work is therefore to make use of the most recent
71 analytical models from literature allowing for the prediction of fresh properties of
72 cement-based materials as a function of constituent parts properties and
73 proportions. The obvious advantage of such relationships lies in their analytical
74 nature, which offers the possibility to derive them and assess robustness from a
75 generic and analytical point of view. This, in turn, may allow for a reduction of the
76 amount of experimental measurements required to assess the robustness of a given
77 cement based-mixture.

78 This above generic idea is illustrated in this work by a fluid concrete example (*i.e.*
79 slump flow higher than 50 cm) although the same work could be carried out for
80 other types of cement-based materials. The approach and the analytical relations
81 used here would be the exact same ones. In this study, it is assumed that all
82 components properties are fixed. The variations, to which the material is submitted,
83 are only, therefore, the respective proportions of its constituent parts. This would
84 correspond in practice to a situation, in which the material is produced from the
85 same batches of constituents. Moreover, in this study, the focus is limited to the yield
86 stress of the resulting mixture and its variations, as this rheological parameter is the
87 most documented in literature and the most important in standard casting processes
88 [5].

89 The objective of the first part of this paper is therefore to show how component
90 proportions variations affect yield stress from the polymer adsorption scale to the

91 concrete scale. Analytical models from literature relating components proportions
92 and yield stress are combined into a multi-scale and physical approach and compared
93 to our experimental yield stress measurements.

94 In the second part of this paper, from the derivation of these analytical relations, we
95 assess robustness as a function of components proportions and map the most
96 sensitive variations in components dosage from a yield stress point of view.

97 Finally, in the specific case of the fluid concrete taken here as an example, we
98 conclude on the potential changes in mix design that may result into a more robust
99 mixture.

100

2. Background and methodology

First, it is important to make a distinction between material parameters that capture the constituent parts properties and are therefore non-dependent on mix proportions and the mixture variables, which result directly from mix proportions.

In this section, from literature, the number and nature of the variables theoretically required to describe one cubic meter are derived in the case of an optimized fluid concrete with a given yield stress and containing five constituents: water, cement, sand, gravel and a super-plasticizer (SP). The proportion of each of these five constituents is obviously a mixture variable by itself. However, when dealing with the proportions required to produce one cubic meter of concrete, it is therefore possible to describe all components proportions with only four mixture variables. Additionally, as the yield stress of the concrete is specified, this provides an additional relation, which leads to reduce the number of independent mixture variables to three. Finally, it is considered in this study that the studied mix has already been optimized. As a consequence, the aggregates size distribution and the relative proportion between sand and gravel have both already been fixed in order to maximize the packing properties (*i.e.* the maximal packing fraction) of the granular skeleton [6-8]. Such a material should therefore be fully described by using only two independent mixture variables. A mineral filler would often be added to such a mixture in order to reduce carbon foot print, to improve workability and to decrease cost, shrinkage and hydration heat according to [9-11] and ASTM C 1797. This would, in turn, increase the number of variables required to describe the mixture to three. For the sake of simplicity, this situation is however out of the scope of the present paper.

In order to identify and select the two previous variables, the present approach is based on several analytical relations from literature, which have all proved, in previous publications from various authors, their ability to predict the related underlying physics. These analytical relations include:

2.1. YODEL model for cement pastes

130 The Yodel is a first principle analysis of yield stress of colloidal particles suspension. It
 131 was shown to successfully predict the main parametric dependencies of cement
 132 suspensions yield stress [12-15]. This model is used here to predict the yield stress
 133 τ_p of the constitutive cement paste of the concrete (*i.e.* the matrix phase). The
 134 model can be written as:

$$\tau_p \cong m \frac{A_0 a^* \varphi_c^2 (\varphi_c - \varphi_{perc})}{d^2 H^2 \varphi_{cm} (\varphi_{cm} - \varphi_c)} \quad (1)$$

135 Where 6 material parameters are:

136 *m*, a pre-factor, which depends on the cement particle size distribution; It
 137 is possible to compute it from the cement particle size distribution but it is
 138 simpler to measure the value of the product $m A_0 a^*$ in Eq. (1) on a
 139 reference cement paste as shown in [14, 15].

140 A_0 , the non-retarded Hamaker constant taken as 1.6×10^{-20} J [12];

141 a^* , the typical size of the surface defects of cement grains of the order of
 142 several hundreds of nm [15];

143 d , the cement particle volume-average diameter d_{50} of the order of 10
 144 μm . It can be measured using laser granulometry (see next section);

145 φ_{cm} , the maximal packing fraction of the cement powder. It can be
 146 measured using the water demand method developed by De Larrard (see
 147 next section) [6].

148 φ_{perc} , the percolation volume fraction; It results from the competition
 149 between Brownian motion and colloidal attractive forces between cement
 150 particles, which, in turn, depend on the SP dosage [15]. This dependency is
 151 however weak. It is therefore considered here as a constant material
 152 parameter. It can be extrapolated from a yield stress measurement on a
 153 reference cement paste as described in [14, 15].

154 And where 3 mixture variables are:

155 τ_p , the yield stress of the constitutive cement paste of the concrete;

156 φ_c , the solid volume fraction of cement particles in the cement paste;

157 H , the average surface-to-surface separating distance between

158 *flocculated cement particles; It depends on the SP dosage as described in*
159 *sub-section 2.2.*

160

161 **2.2. Statistical average separating distance between cement grains as a function** 162 **of admixture surface coverage**

163 The average separating distance between cement grains H [14] can be computed as:

$$\frac{1}{H^2} = \frac{\theta^2}{H_p^2} + \frac{8\theta(1-\theta)}{(H_p + H_0)^2} + \frac{(1-\theta)^2}{H_0^2} \quad (2)$$

164 *Where 2 additional material parameters are:*

165 *H_p , the SP layer thickness at full surface coverage. It could be estimated from*
166 *the molecular structure of the SP [16]. It is however easier in practice and*
167 *when faced with an unknown molecular structure to assess its value from*
168 *yield stress measurements on fresh cements pastes containing a given*
169 *dosage of SP [15];*

170 *H_0 , the average separating distance between cement grains in a system not*
171 *containing any admixtures (of the order of 1-2 nm) [17].*

172 *And where 1 mixture parameter is:*

173 *θ , the so-called “surface coverage” or the ratio between the amount of SP*
174 *adsorbed and the amount of adsorbed SP at surface saturation, which can*
175 *be extracted from an adsorption isotherm (see below).*

176

177 **2.3. Surface coverage as function of SP dosage**

178 It is now accepted that any mathematical relations showing a proportionality at low
179 dosages between SP dosage and SP adsorption and displaying an adsorption plateau
180 at high SP dosages is able to capture the adsorption behavior of a given SP and the
181 surface coverage evolution as function of SP dosage [18, 19]. Langmuir-type relation
182 is an example of this kind of mathematical relations. In this work, for the sake of
183 simplicity, the following analytical relation is used:

$$\theta = \frac{\kappa \cdot D_{rsp}}{1 + \kappa D_{rsp}} \quad (3)$$

184 Where D_{rsp} is an additional mixture parameter, defined as the relative dosage of SP
 185 with $D_{rsp} = D_{sp}/D_{sat}$, where D_{sp} is the SP dosage expressed as a mass
 186 proportion of the cement content per cubic meter and D_{sat} is the so-called
 187 saturation dosage (see below);

188 And where 2 material parameters are:

189 κ , a dimensionless adsorption and desorption equilibrium rate constant; κ
 190 can be assessed from the measurement of the polymer adsorption isotherm;
 191 D_{sat} , the so-called saturation dosage, above which further increase in the
 192 dosage of SP does not increase the surface coverage. It can be assessed
 193 through SP adsorption measurements or yield stress measurements.

194

195 **2.4. Chateau and Ovarlez model for mortar and concrete yield stress**

196 The Chateau and Ovarlez model has proven its ability to predict the yield stress of a
 197 mixture containing rigid inclusions in a yield stress fluid [20, 21]. It was moreover
 198 shown to be able to predict the yield stress of fluid mortars and concretes when
 199 direct frictional contacts between aggregates were neglectable (*i.e.* high matrix
 200 volume content and/or fluid mixtures [7]). It can be expressed as:

$$\tau_M = \tau_p \sqrt{(1 - \varphi)} \left(1 - \frac{\varphi}{\varphi_{div}}\right)^{-1} = \tau_p \sqrt{(1 - \varphi)} \left(1 - \frac{\varphi}{0.8\varphi_{am}}\right)^{-1} \quad (4)$$

201 Where 2 additional mixture parameters are:

202 τ_M , the yield stress of the mortar or concrete mixture;
 203 φ , the aggregates volume fraction at the level of one cubic meter of
 204 concrete;

205 And where one material parameter is:

206 φ_{div} , the aggregates volume fraction for which the yield stress diverges. It
 207 was shown that, for sand, gravels and fibers, $\varphi_{div} = 0.80\varphi_{am}$, where φ_{am}
 208 is the maximal packing fraction of the aggregates [7, 22]. φ_{am} can be
 209 measured using independent compaction measurements carried on the dry

210 aggregates [23].

211

212 **2.5. Integration of all models**

213

214 The yield stress τ_M of a fluid concrete can therefore, in theory, be predicted
215 combining equations ((1) to (4)). Such an approach would involve the 11 material
216 parameters listed above that do capture both the components properties and their
217 interactions. These 11 parameters can all be experimentally and independently
218 assessed. Because of the way the above analytical relations are built, these
219 parameters are only component-dependent and not dosage-dependent (*i.e.* they do
220 not change when varying components proportions).

221 The full description of the mixture from the above equations moreover involves
222 seven mixture variables along with four multi-scale relations. This results into 3
223 independent variables. For these variables, the cement solid volume fraction φ_c at
224 the scale of one cubic meter of the constitutive cementitious matrix (*i.e.* excluding
225 any aggregates), the SP dosage D_{sp} (*i.e.* the mass ratio between SP and cement) and
226 the aggregates volume fraction φ at the scale of one cubic meter of concrete or
227 mortar were selected. As stated previously, only two of these three variables are
228 enough to describe such a system if a target value for its yield stress is first specified.

229

230 **3. Materials and experimental protocols**

231

232 **3.1. Materials properties**

233 A commercial cement equivalent to ASTM Type I cement was used in this study. The
234 chemical composition of the cement is shown in Table 1. The Particle Size
235 Distribution (PSD) was measured by laser diffraction (MALVERN MASTERSIZER S)
236 after dispersion in isopropanol and its volume average diameter d_{50} was around 12
237 micrometers (Cf. **Fig. 1**). The maximum packing fraction of this cement was 0.58. It
238 was measured following the water demand method developed by De Larrard [6]. It

239 consists in finding the minimum water content allowing for the transition during
240 mixing between pasty cement granules and a continuous homogeneous paste.

241

242 The properties of the aggregates used in this study are shown in **Table 2** and the PSD
243 of the aggregates is shown in Fig. 1. The maximal packing fraction of the aggregates
244 was measured using the following protocol. A container with the diameter and
245 height of 16 cm and 32 cm respectively was fixed on a vibration table and was
246 submitted to a 150 Hz vibration. A mass, equivalent to 1 kPa external pressure, was
247 applied above the sample. For each dry batch of aggregates, 7.5 kg of aggregates
248 were poured into the container and, after a 60 s vibration, the height of the sample
249 was measured. Finally, using the mean apparent density of the aggregate mixtures,
250 the maximum packing fraction was computed.

251 In addition to the independent assessment of sand and gravel, the packing properties
252 of dry mixtures of sand and gravel with varying proportions were also measured (Cf.
253 **Fig. 2**). The sand mass proportion was then chosen in order to optimize the packing
254 properties of the granular skeleton and is therefore equal to 45%.

255 The SP used in this study is a non-blended commercial polycarboxylate provided
256 under liquid form with a solid content of 29.5%. The recommended highest dosage of
257 this SP is 3.0% by mass of cement.

258

259 **3.2. Mixing protocols**

260 Cement pastes with different dosages of SP ranging from 0% to 3.5% were prepared.
261 The SP was first added into the mixing water (with controlled temperature of around
262 $22 \pm 2^\circ\text{C}$) prior to the contact with cement. For each dosage of SP, cement pastes
263 with
264 W/C mass ratio ranging from 0.275 to 0.5 were prepared. For each sample, a volume
265 of around 200 ml of cement paste was mixed for 3 min using a Turbo test Rayneri
266 VMI mixer at the speed of 840 rpm. A total of 53 different cement pastes was
267 prepared.

268 The mortars were produced from cement pastes prepared using the same protocol
269 as above. Then, the sand was introduced into the cement paste and stirred for 2 min
270 by hand in order to decrease the influence of mixing in the presence of rigid particles
271 on the rheological properties of the interstitial constitutive cement paste [14]. The
272 sand content was varied from 0 to 52.5% volume fraction (*i.e.* around 475 liters of
273 paste per cubic meter) while the W/C ratio and SP mass dosage were varied between
274 0.275 to 0.50 and between 0 and 3% respectively in order to produce 41 different
275 mortars.

276 In the case of concrete mixing, a Zyklos-type concrete mixer from Schwelm was used.
277 The corresponding cement paste was first mixed alone for 3 min then the aggregates
278 were introduced and dispersed for another 2 min. We checked that the cement
279 pastes prepared in this mixer had a similar yield stress as the ones prepared with the
280 Rayneri mixer above (results not shown here). Around 10 liters of concrete were
281 produced for each concrete mixture. The total aggregate content was varied from
282 zero to around 62% volume fraction (*i.e.* around 380 liters of paste per cubic meter of
283 concrete) over 5 different concretes mixes.

284 All experiments were carried out at a temperature of $22 \pm 2^\circ\text{C}$ and at a relative
285 humidity of around $50\% \pm 5\%$.

286 All mix designs are provided as supplementary materials along with measured yield
287 stress values.

288

289 **3.3. Yield stress measurements**

290 In this study, the values of the yield stress of cement pastes, mortars and concrete
291 mixtures were computed from simple flow tests [7, 24].

292 At the scale of cement paste and mortar, volumes of around 200 ml and around 500
293 ml were used respectively to measure flow spread. Each mixture was slowly poured
294 from the mixing cup on a horizontal steel plate. The spread diameter was assessed
295 from the average of two measured diameters. The yield stress value was then
296 computed using the following equation [24, 25]:

$$\tau_0 = \frac{225\rho g\Omega^2}{128\pi^2 R^5} \quad (5)$$

297 where τ_0 is the yield stress (Pa); ρ is the bulk density of the mixture (kg/m^3); Ω is
 298 the volume of the mixture (m^3); R is the spread radius (m).

299 At the scale of concrete, 6 l of material were slowly poured from a bucket into one
 300 end of the LCPC box and the spread length was measured [26]. The yield stress of the
 301 concrete mixtures was computed from the existing analytical correlation between
 302 the measured spread length in the LCPC box and the yield stress of the tested
 303 material [26, 27] (Cf. Eq. (6) and (7)).

$$V = \frac{l_0^3}{4A} [LN(1 + U_0)] + \frac{U_0(U_0 - 2)}{2} \quad (6)$$

$$L = \frac{h_0}{A} + \frac{l_0}{2A} LN\left(\frac{l_0}{l_0 + 2h_0}\right) \quad (7)$$

304 where V is the tested concrete volume (L); l_0 the width of the LCPC box (20 cm); L
 305 the measured spread length of concrete in the box (cm); h_0 the height of the
 306 sample at the point where concrete is poured at the end of the test (cm); $A =$
 307 $2\tau_0/\rho g l_0$, $U_0 = 2h_0/l_0$, where τ_0 is the yield stress of the concrete (Pa), ρ the
 308 apparent density of the concrete ($\text{kg}\cdot\text{m}^{-3}$), g the gravity ($\text{m}\cdot\text{s}^{-2}$). Solving the above
 309 equations simultaneously provides the value of the concrete yield stress. An abacus
 310 is also available in [26].

311

312 **3.4. Polymer adsorption measurements**

313

314 A total organic carbon (TOC) analyzer manufactured by Shimadzu was used in this
 315 work. Cement pastes with W/C equal to 0.4 and dosages of SP ranging from 0 % to
 316 3.5% by mass of cement were mixed as described above. The cement pastes were
 317 then centrifuged and the extracted interstitial fluid was diluted and analyzed. By
 318 comparing the TOC values of the extracted interstitial fluid with a reference polymer
 319 solution, the amount of polymer adsorbed on cement grains was computed and the
 320 surface coverage (*i.e.* the ratio between adsorbed polymer and adsorbed polymer at
 321 saturation) as a function of the polymer relative dosage (*i.e.* ratio between dosage

322 and saturation dosage) is plotted in **Fig. 3**. The amount of organic carbon in the
323 interstitial fluid of a reference cement paste without polymer was also measured to
324 account for the presence in the cement powder of organic compounds such as
325 grinding agents.

326

327 **4. Experimental results and analysis of results**

328 **4.1. Yield stress of cement paste**

329 The yield stress of cement paste as a function of cement volume fraction for various
330 SP dosages is plotted in **Fig. 4**. Only non-bleeding cement pastes (visual observation)
331 are shown in **Fig. 4**. The values of the measured yield stress range from around 1 Pa
332 to around 100 Pa. It should be noted that the higher the SP dosage, the higher the
333 minimum cement volume fraction required to obtain a non-bleeding mixture.

334 The prediction of the yodel model is also plotted in **Fig. 4** (continuous lines). It is
335 worth noting that the only parameter changing from one SP dosage to another in the
336 Yodel is the average surface-to-surface separation distance (H) between cement
337 particles (not shown here). This distance is varied from 2 nm (reference paste) to 9
338 nm (full surface coverage). As already shown in previous papers [12, 13], the YODEL
339 analytical model is able to capture the evolutions of a cement paste yield stress as a
340 function of solid volume fraction and inter-particle distance over more than two
341 orders of magnitude.

342

343 **4.2. Yield stress of mortar**

344 **Fig. 5** shows the relative yield stress of the tested mortars (*i.e.* the ratio between the
345 mortar yield stress and the yield stress of the corresponding constitutive cement
346 paste) as a function of sand volume fraction for sand volume fractions varying from 0%
347 to around 50%. It can be seen that the values of the relative yield stress range from 1
348 to around 50 times the yield stress of the corresponding constitutive cement pastes.
349 Interestingly but as already shown in [23] and as predicted by the Chateau and
350 Ovarlez model, the relative yield stresses of the studied mortars does not depend on

351 the yield stress of the constitutive cement pastes. The amplification of the
352 constitutive paste behavior by the rigid sand particles is well predicted in **Fig. 5** by
353 the Chateau and Orvarlez model without any need for fitting nor corrections.

354 As predicted by Eq. (4), when the relative solid volume fraction (*i.e.* the ratio
355 between the sand volume fraction and the maximum packing fraction of the sand as
356 measured in section 3.1) approaches $0.80\varphi_{am}$ (dotted vertical line in **Fig. 5**), the
357 yield stress of the tested mortars shows a sharp increase of a couple orders of
358 magnitude [7, 22]. This feature indicates that frictional contacts between sand grains
359 start to percolate and dominate the macroscopic rheology of the mixtures [20, 21,
360 23].

361

362 **4.3. Yield stress of concrete**

363 **Fig. 6** represents the relative yield stress of the concrete (*i.e.* the ratio between the
364 yield stress of the concrete and the yield stress of the constitutive cement paste) as a
365 function of the total aggregate volume fraction (*i.e.* sand and gravel). The relative
366 yield stress predicted by the Chateau and Ovarlez model is also plotted in **Fig. 6**. As
367 expected from the previous section, the model captures the experimental data and
368 shows a divergence as the aggregates volume fraction reaches the divergence
369 volume fraction around 62% (vertical dotted line in **Fig. 6**).

370

371 **5. Robustness assessment**

372 **5.1. Robustness index definition**

373 In order to assess the robustness of a given mixture, we define here a robustness
374 index R_τ for a component dosage variation. As yield stress is the parameter of
375 interest in this study, such an index can be expressed as follows:

$$R_\tau = \frac{d\tau_m}{dx} \cdot \frac{\Delta x_{max}}{\tau_m} \quad (8)$$

376 where R_τ is the robustness index from the yield stress point of view (dimensionless).

377 τ_m is the yield stress of the mixture (Pa); x stands for the dosage of one of the
378 mixture components while Δx_{max} is the maximum variation of this dosage in

379 industrial practice. In order to define Δx_{max} , the average variations of components
380 dosage in concrete plants from various technical recommendations and studies [28,
381 29] are considered (Cf. **Table 3**). It has to be noted that the derivation in Eq. (8)
382 includes the fact that, if the volume of one component is increasing or decreasing,
383 then the volume of all other components has to be decreased or increased
384 respectively in order to cover for the total volume change at the level of the
385 considered one cubic meter of concrete. This effect is neglectable for SP and cement
386 dosage variations but plays a non-neglectable role for aggregates and water dosages
387 variations. Finally, it has to be kept in mind that, in the following, the higher the
388 robustness index, the less robust is the mix.

389 It can first be noted that the more restrictive values from the ACI standards [28]
390 seem to be closer to real industrial practice when compared to the
391 industry-representative values reported in literature [30-32]. We choose therefore to
392 consider these limits in this study.

393 However, the water dispenser accuracy from the ACI standard [28] in **Table 3** does
394 not take into account some potential uncertainties on the moisture content of the
395 sand [4, 33]. These are reported to be measured with a 0.5% accuracy for most
396 existing sensors as reported in technical data sheets for this kind of sensors. Some
397 more accurate water-moisture online assessment technologies do exist but we do
398 not consider that they are representative of the common practice.

399 Moreover, we choose here to turn these relative uncertainties requirement for
400 standard concrete production into absolute uncertainties in order to extrapolate the
401 consequences on robustness of these variations for non-standard component
402 dosages. In order to do so, we consider that the previous uncertainties are given for
403 one cubic meter of standard concrete that is considered here to contain 300 kg of
404 cement, 150 kg of water, 800 kg of sand, 1200 kg of gravel and 3 kg of
405 super-plasticizer. The absolute variation for water dosage is then computed as the
406 square root of the sum of the water dispenser accuracy (4.5 kg) at the power two
407 and the uncertainty of the sand moisture sensor (4 kg) at the power two resulting in
408 an overall absolute uncertainty of 6 kg. Due to the fact that the accuracy of adding SP

409 might be difficult to guarantee during the opening and closing of the SP feed port, a
410 higher absolute variation for SP dosage (i.e. 150 g) is considered according to the EHE
411 limits [29].

412 Finally, we choose here to ignore any potential variations in the sand-to-gravel ratio
413 and only consider variations in the total aggregate content. The reason behind is that
414 aggregates influence on yield stress is mostly driven by total solid fraction and
415 maximum packing fraction of the sand-gravel mixture. However, variations in sand to
416 gravel ratio for an optimized mixture of aggregates do not induce strong variations in
417 maximum packing fraction. Indeed, around the optimal sand-gravel proportions,
418 variations in maximum packing properties of the resulting granular skeleton are
419 neglectable as illustrated in this work in **Fig. 2**. Similar to water dosage variations, the
420 overall absolute uncertainty on the total aggregate content is then computed from
421 the combined variations in sand and gravel content and reported in **Table 3**.

422

423 **5.2. Robustness maps**

424 **5.2.1. SP dosage variations**

425 **Fig. 7** represents the robustness index for SP dosage variations. It is plotted for the
426 150g SP dosage variation from **Table 3** for W/C between 0.3 and 0.55 and relative
427 aggregates content (i.e. the ratio between aggregates volume fraction and maximum
428 packing fraction) between 74 and 79%. This W/C range is typical of most fluid
429 concretes as it covers high strength materials (excluding ultra-high-performance
430 material) for civil engineering application along with standard concretes for housing
431 applications. Obviously, lower W/C shall lead to better mechanical and transfer
432 properties while higher aggregates volume fraction shall result in cheaper mixes as
433 they lead to lower matrix and cement content. Two features bound the range
434 covered here by the aggregates relative fraction variation. On one hand, for the
435 highest aggregates relative fraction, as illustrated in **Fig. 6**, it is not possible to mix
436 design a stable fluid material as direct frictional contacts between aggregates
437 dominate [7]. No matter the amount of water or SP, the material shall either display a
438 high yield stress or be unstable [34]. On the other hand, for lowest aggregates

439 relative fraction, the matrix volume becomes high inducing potential shrinkage,
440 hydration heat and/or creep issues [35].

441 For a SP saturation dosage taken as 2% of the cement mass, **Fig. 7** shows that the
442 highest robustness index is around 20% (*i.e.* 10 Pa variation) and occurs for the
443 highest W/C and lowest relative aggregates volume fraction. It is indeed in this mix
444 design range that the dosage of SP is the lowest. As a consequence, the relative
445 variation in SP dosage for a given dosage accuracy is the highest. This feature is
446 amplified by the shape of the adsorption isotherm in **Fig. 3**. Indeed, as the dosage of
447 SP gets closer to the saturation dosage, the sensitivity of surface coverage to SP
448 dosage decreases and the system becomes more robust from a SP dosage point of
449 view. This is one of the reasons why admixtures manufacturers often recommend a
450 minimum SP dosage. This minimum dosage ensures that the resulting mix is not too
451 sensitive to SP dosage variation in industrial production. As a consequence, at high
452 W/C, it is obviously safer to use a less efficient SP that would require a higher dosage
453 to get a similar yield stress. For instance, the robustness index values around 20% in
454 **Fig. 7** become of the order of 15% if the saturation dosage of the SP used increases
455 from 2% to 3% (*i.e.* less effective SP).

456

457 **5.2.2. Cement dosage variations**

458 **Fig. 8** represents the robustness index for cement dosage variations. It is plotted for
459 the 3 kg cement dosage accuracy from **Table 3**. Firstly, it can be noted that the
460 mixture is far less sensitive to standard cement variations than it is to standard SP
461 variations. Moreover, it can be noted that the trend here is more complex with
462 different regimes clearly appearing, namely for low and high W/C and for high
463 relative aggregates volume fractions.

464 It is first obvious that the system is more sensitive to cement dosage variation when
465 the W/C is low. Indeed, at high cement volume fractions (*i.e.* close to the cement
466 powder maximum packing fraction), as shown in **Fig. 4**, despite the log scale, yield
467 stress shows an extremely high sensitivity to solid volume fraction.

468 At high W/C, on the other hand, the dosage of SP and the resulting surface coverage

469 is low. A variation in cement content result therefore in a variation in surface
470 coverage, which, in turn, induces variations in yield stress as discussed above for **Fig.**
471 **7**.

472 Finally, at high relative aggregates volume fractions, our analysis suggests that the
473 higher sensitivity of the mixture finds its origin in the fact that, at the level of one
474 cubic meter, a decrease in cement dosage results in a relative increase in aggregates
475 content. Such systems are however close to the divergence aggregates volume
476 fraction illustrated in **Fig. 6** and are therefore very sensitive to small fluctuations in
477 aggregate content.

478 Overall, however, it will be seen further that fluctuations in yield stress induced by
479 cement dosage variations are neglectable compared to the other sources of
480 fluctuations.

481

482 **5.2.3. Water dosage variations**

483 **Fig. 9** represents the robustness index for water dosage variations. It is plotted for
484 the 6 kg water dosage accuracy from **Table 3**. The high sensitivity of the system to
485 water dosage compared to SP and cement can first be noted. Moreover, as W/C is
486 decreasing, the sensitivity of the mixture to water variations increases. This finds its
487 origin in the robustness of the cement paste itself that decreases at low W/C as
488 shown in **Fig. 4** where the yield stress, for high solid fractions, shows an extremely
489 high sensitivity to solid volume fraction.

490 We moreover note that, when the relative aggregate volume fraction increases, the
491 sensitivity of the mixture to water increases. This unexpected trend finds its origin in
492 the indirect consequence of a decrease in water dosage at the scale of one cubic
493 meter. If the water content decreases, the proportions of all other components
494 increase. A 6 liters variation in water is indeed able to induce a 15 kg variation in
495 aggregate content or around 20 kg in cement dosage. As shown previously, cement
496 content variations only had minor consequences. This is not the case for aggregates
497 content, especially at high aggregate fractions when yield stress becomes extremely
498 sensitive to aggregate content as shown in **Fig. 6**.

499

500 **5.2.4. Aggregate dosage variations**

501 **Fig. 10** shows the robustness index for aggregate dosage variations. It is plotted for
502 the 29 kg total aggregate dosage accuracy from **Table 3**. Firstly, it can be noted that
503 this graph shows the second highest sensitivity after water variations (Cf. **Fig. 10**).
504 The robustness index does not depend on the W/C ratio but only on the relative
505 aggregates solid volume fraction. For high aggregate fractions, yield stress becomes
506 extremely sensitive to aggregate content as shown in **Fig. 6**.

507

508 **5.2.5. Practical consequences on the mix design of robust fluid concretes**

509 **Fig. 11** shows the overall sensitivity map of the fluid concrete taken as an example
510 here by computing the quadratic average of all the above robustness indexes.
511 Unsurprisingly, the overall answer is dominated by the influence of water variations
512 and aggregates dosage variations. As a consequence, although the most economical
513 mixtures shall be obtained for aggregates relative solid fraction close to 80%, **Fig. 11**
514 suggests that mixtures with acceptable robustness are obtained for lower aggregates
515 dosages than the economical optimum.

516 Moreover, **Fig. 11** suggest that more robust mix could be produced by moving to
517 higher W/C ratio (when obviously mechanical strength and durability are not an
518 issue). This could reach a limit at high W/C ratio combined with lower aggregates
519 dosage as it would lead to a decrease in the SP dosage required and bring the system
520 to the darkest zone of Fig. 7 where the mixture becomes very sensitive to SP dosage.
521 One solution would then be to combine the SP with a viscosity modifying agent
522 (VMA). The resulting adsorption competition between these molecules [36, 37]
523 would increase the required SP dosage and decrease therefore the sensitivity of the
524 mixture to SP dosage. It should be noted that this solution seems to be adopted in
525 many countries where Self-compacting Concretes are designed with VMAs. Our
526 results suggest, however, that VMAs do not increase robustness directly as
527 sometimes suggested [38] but, instead, allow for the design of mixtures with higher
528 water content or higher SP dosages, which are, in turn, more robust.

529 Finally, as both aggregates content and water dosage are strongly correlated to
530 aggregates water content and aggregates water content measurement, it can be
531 extrapolated that, from a concrete production point of view, it is on its ability to
532 measure aggregates water content that the concrete plant technology performance
533 has the strongest influence on a fluid concrete robustness.

534

535 **5 Conclusions**

536 The idea behind the present work was to assess robustness of fresh cement-based
537 materials from a generic and analytical point of view.

538 In a first part, analytical models from literature relating components proportions and
539 yield stress were combined into a multi-scale and physical approach and compared
540 to our experimental yield stress measurements.

541 In the second part of this paper, from the derivation of these analytical relations, we
542 assessed robustness as a function of constituent parts proportions and mapped the
543 most sensitive variations in constituent parts proportions from a yield stress point of
544 view.

545 The results in this investigation demonstrated the dominant sensitivity of the mixture
546 to water and aggregates dosages and, indirectly, to aggregates moisture variations.
547 Although these results are known in practice, the work presented here allows, for the
548 first time, for the quantitative prediction of concrete robustness.

549 Moreover, the results, obtained here in the case of the fluid concrete taken as an
550 example of the methodology, provide some physical explanations for the positive
551 effect on robustness of some trends in SCC mix design. For instance, working with
552 slightly lower fractions of aggregates than the economical *optimum* brings the
553 system further from the aggregates contact percolation packing fraction where the
554 sensitivity of the system to aggregates dosage is the highest. Similarly, increasing
555 slightly the water content brings the system further from the cement powder
556 maximum packing fraction where the sensitivity to water dosage is the highest.
557 However, increasing water dosage and decreasing aggregate content both induce a

558 decrease in the required plasticizer dosage. This, in turn, increases the sensitivity of
559 the mixture to plasticizer dosage. Combining viscosity modifying admixtures with
560 plasticizers, because of their competitive adsorption, is a potential way to go back to
561 standard plasticizer dosages and improve robustness.

562

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566

567 **Conflict of interest**

568 The authors declare that they have no conflict of interest.

569

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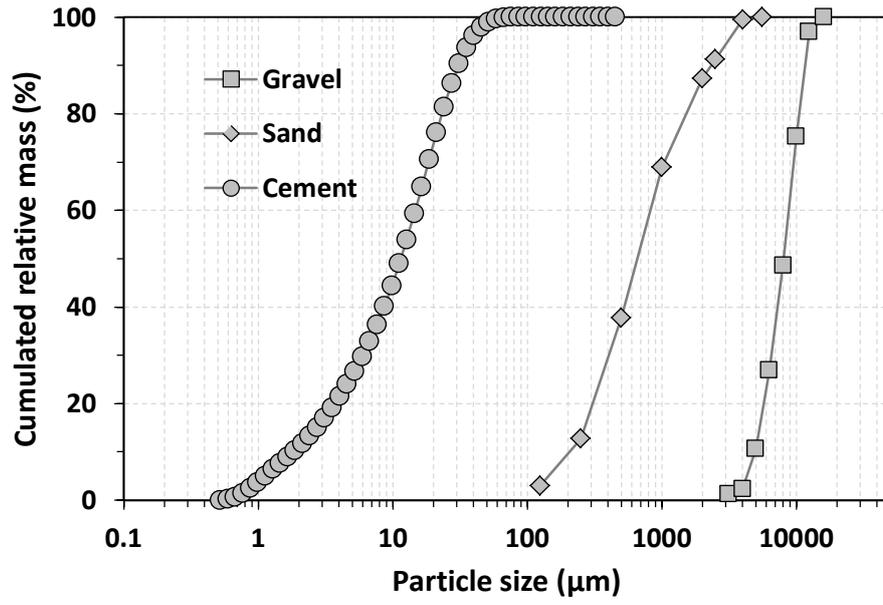


Figure 1. Particle size distribution of the cement and sand used in this study.

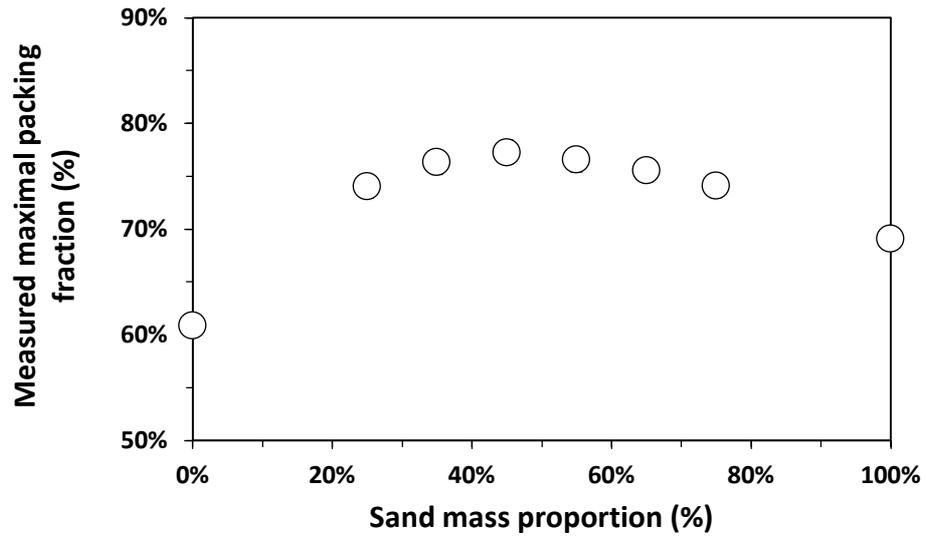


Figure 2. Maximal packing fraction of aggregate mixtures as a function of sand mass proportion at the level of the granular skeleton.

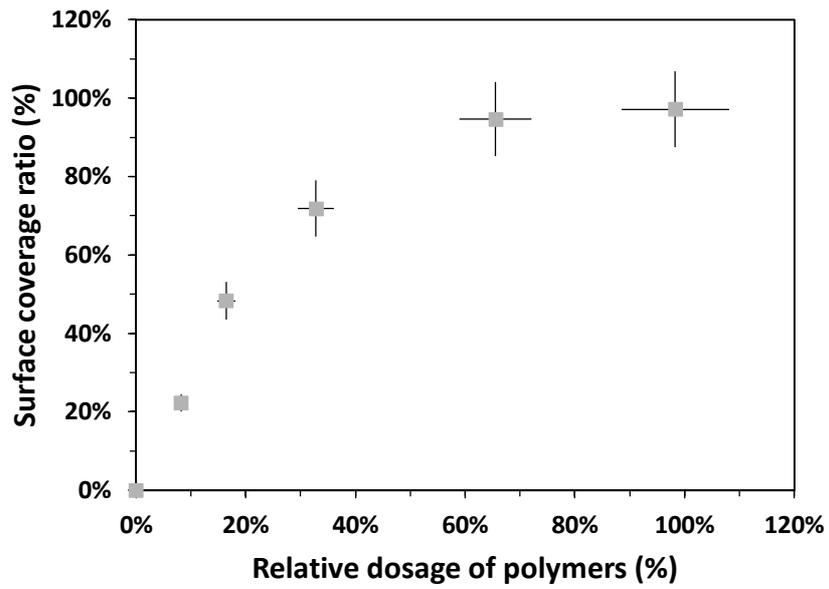


Figure 3. Surface coverage ratio (i.e. ratio between adsorbed polymer and adsorbed polymer at saturation) as a function of D_{rsp} , the polymer relative dosage (i.e. ratio between dosage and saturation dosage).

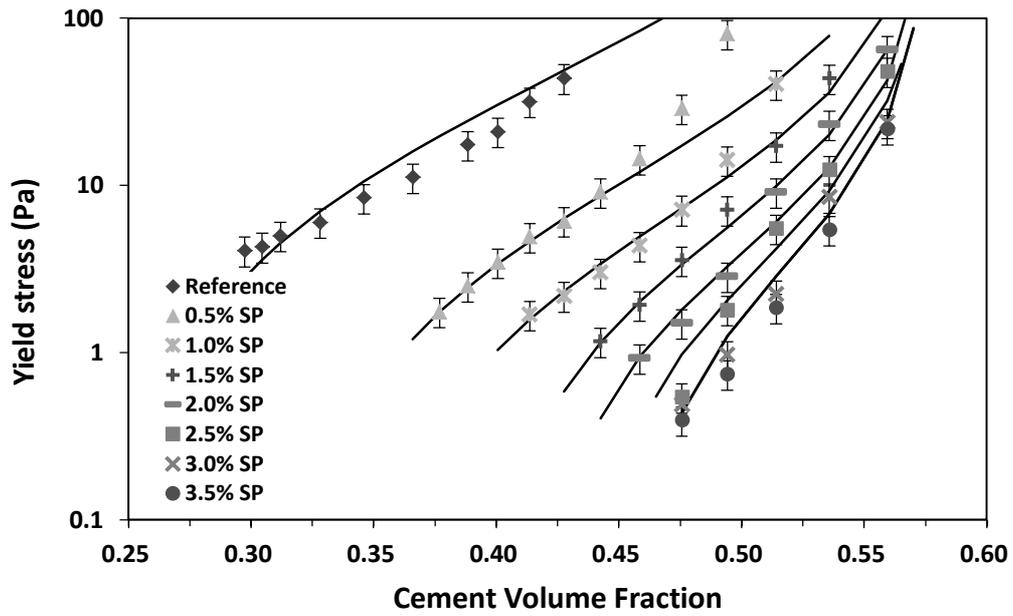


Figure 4. Yield stress of cement paste as a function of cement volume fraction for various SP dosages for the two types of the cement. The solid lines are the computed predictions of the YODEL.

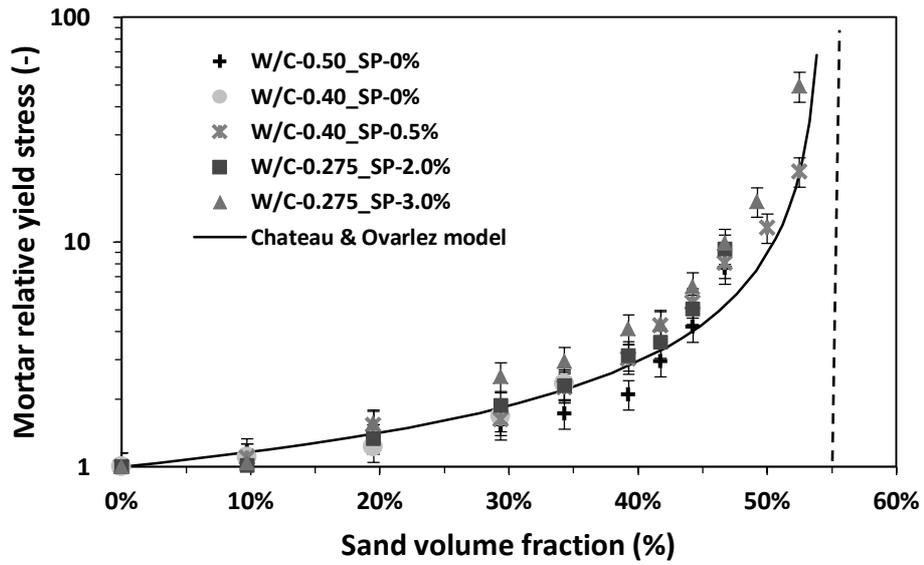


Figure 5. Mortar relative yield stress (i.e. ratio between the mortar yield stress and the constitutive cement paste yield stress) as a function of the sand volume fraction. The dotted vertical line corresponds to the theoretical divergence volume fraction (see text).

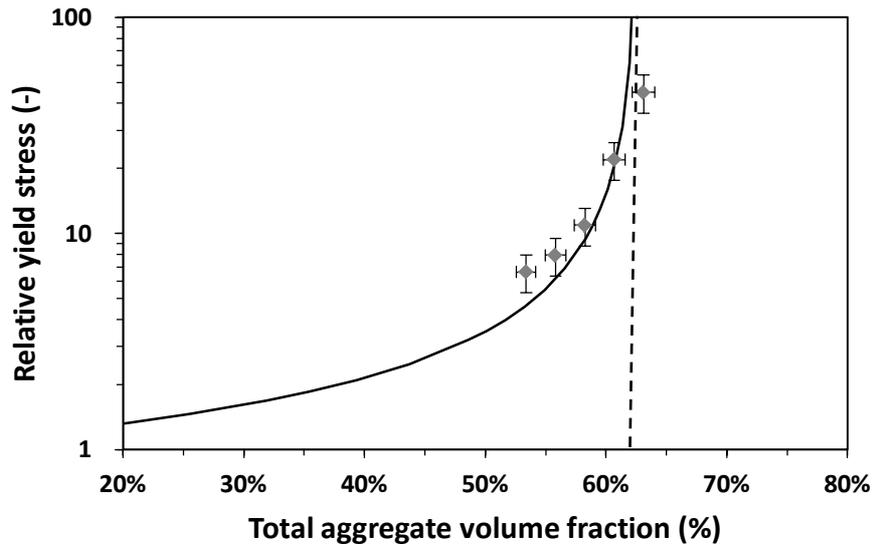


Figure 6. Relative yield stress of concrete as a function of aggregate volume fraction. Continuous line corresponds to the fitting of the Chateau and Ovarlez model (see text).

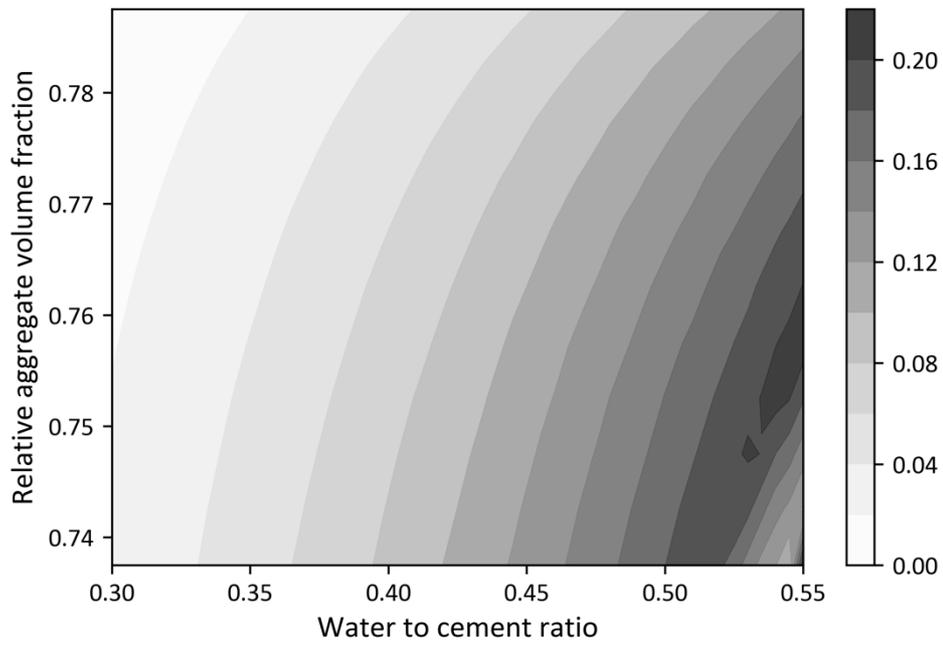


Figure 7. Robustness index as a function of W/C ratio and relative aggregates volume fraction for standard SP variations in industrial production. SP saturation dosage taken as 2% of the cement mass.

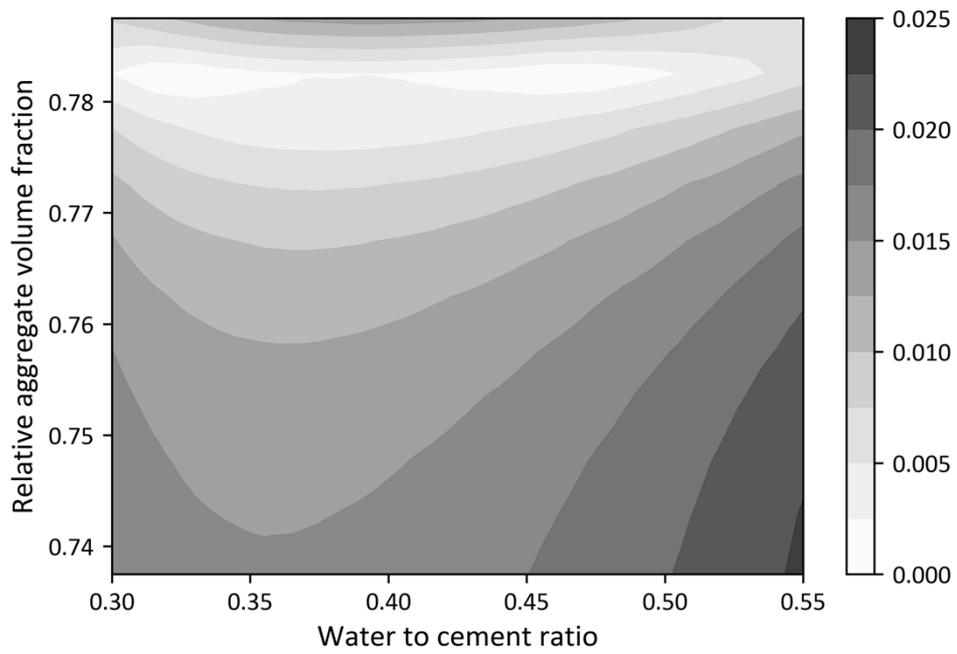


Figure 8. Robustness index as a function of W/C ratio and relative aggregates volume fraction for standard cement variations in industrial production.

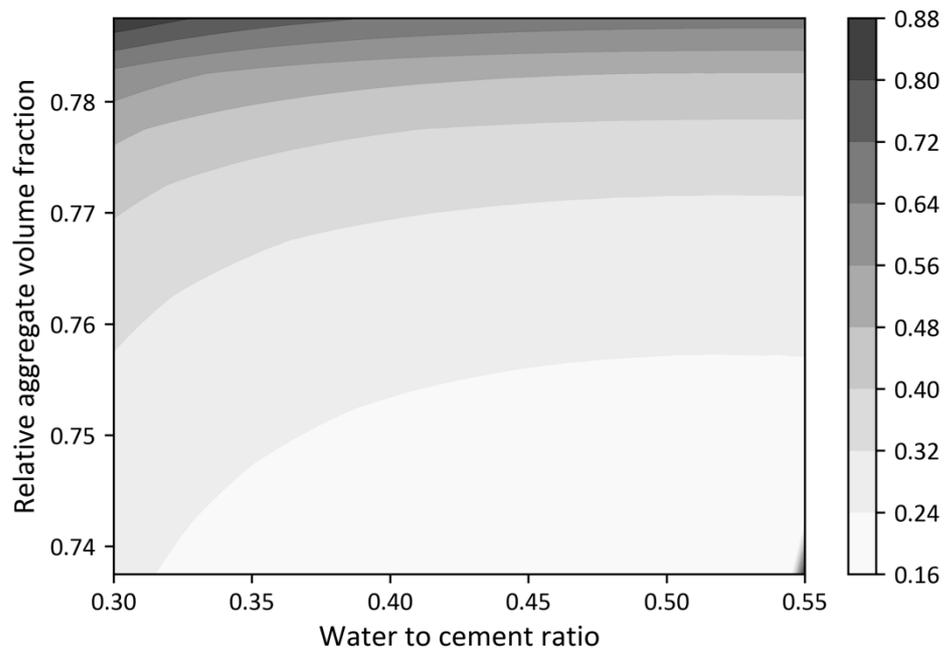


Figure 9. Robustness index as a function of W/C ratio and relative aggregates volume fraction for standard water dosage variations in industrial production.

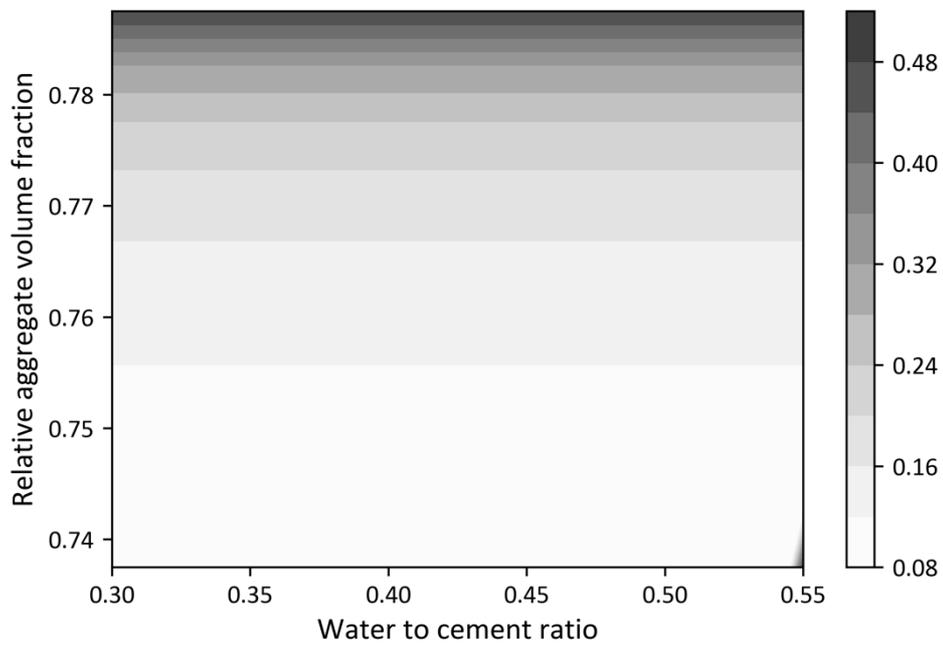


Figure 10. Robustness index as a function of W/C ratio and relative aggregates volume fraction for standard aggregates dosage variations in industrial production.

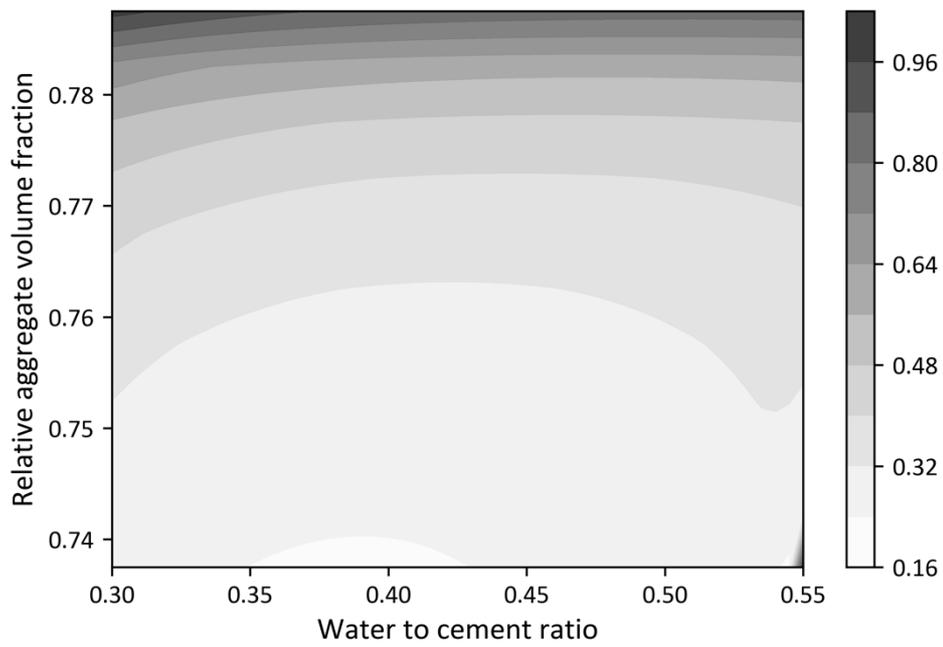


Figure 11. Overall robustness index as a function of W/C ratio and relative aggregates volume fraction for standard dosage variations in industrial production.

Table 1 Chemical composition of the cement.

Composition	%Weight
SiO ₂	21.81%
Al ₂ O ₃	3.67%
Fe ₂ O ₃	5.54%
CaO	64.10%
MgO	1.31%
SO ₃	2.41%
Cl	0.04%

Table 2 Aggregates properties.

Aggregates	Origin	Size range/mm	Specific density	Water absorption ratio/%	Maximum packing fraction/%
Sand	Palvadeau	0 – 4	2.64	0.6±0.05	68.3
Gravel	Seine	4 – 12	2.56	< 0.5	60.9

Table 3 Minimum required dispenser accuracy for concrete components in a ready-mix concrete plant from existing standards.

Components	ACI limits [28]	EHE limits [29]	Absolute variation (kg/m ³)
Cement	1.0%	3.0%	3
Sand	2.0%	3.0%	16
Gravel	2.0%	3.0%	24
Total aggregate content	-	-	29
Water	3.0%	3.0%	6
Admixture	3.0%	5.0%	0.15