



HAL
open science

Ductility analysis of vegetal-fiber reinforced raw earth concrete by mixture design

S. Imanzadeh, A. Jarno, A. Hibouche, A. Bouarar, S. Taibi

► To cite this version:

S. Imanzadeh, A. Jarno, A. Hibouche, A. Bouarar, S. Taibi. Ductility analysis of vegetal-fiber reinforced raw earth concrete by mixture design. *Construction and Building Materials*, 2020, 239, pp.117829 -. 10.1016/j.conbuildmat.2019.117829 . hal-03488893

HAL Id: hal-03488893

<https://hal.science/hal-03488893>

Submitted on 21 Jul 2022

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial 4.0 International License

24 **1. Introduction**

25 The raw earth-based materials have been the subject of renewed interest in the recent years with
26 the search for alternative building materials, *i.e.* ecological and economical materials. The main
27 advantages of these materials are their low consuming energy for production, their availability
28 in the large quantity, their recyclability, their low-cost construction and their easy in-situ
29 implementation ([1], [2], [3], [4]). Therefore, they are suitable even in impoverished areas.
30 Numerous studies have been dedicated for improvement of the mechanical and hygrothermal
31 natural properties of raw earth material [5]. Therefore, raw earth constitutes a matrix where
32 various components - natural or/and manmade materials - are added to reinforce one particular
33 property. According to the used raw earth construction technique, the compressive strength
34 could be improved in order to be used as a construction and building material. This
35 improvement can be achieved by adding low quantity of binders such as lime and cement and
36 sometimes adding fibers. Recent studies have demonstrated the influence of these binders on
37 the raw earth material properties ([5], [6], [7], [8]). In particular, cement is known to be
38 effective for improving the material strength. For example, Delgado and Guerrero [9], in a
39 review paper on earth construction in Spain, provided the compressive strength range for
40 stabilized and unstabilized raw earth, respectively 1.8 to 8.25 MPa and 0.6 to 2.25 MPa. Then,
41 the challenge is to optimize the amount of cement in the material in an attempt to minimize the
42 non-ecological and expensive component in the raw-earth material [4].

43 However, shrinkage in these raw earth-based materials is one of their weaknesses. It is
44 responsible for the development of cracks. To reduce this effect, their plastic behavior needs to
45 be improved to prevent brittle failures and thus limit cracking [10].

46 The plastic behavior of raw earth-based materials shows a ductile behavior characterized by a
47 compressive strength that increases until it reaches a maximum value and then decreases slowly
48 with deformation. Ductility can be parameterized to characterize the plastic behavior of the
49 materials. Ductility is the ability of the material to deform itself without breaking. For example,

50 it is required for the various building materials in the areas of seismic risks ([11], [12]). The
51 good ductility of the material will prevent the sudden collapse of the structure. Either the
52 material is suitably ductile enough or a constituent, such as biopolymers, plant and animal
53 aggregates or fibers, is added.

54 When focusing on the plasticity of earth material, it can be noticed that the number of studies
55 is limited. Some of the studies pay attention to the determination of fiber properties since they
56 directly impact the plasticity of the composite material. The mechanical characteristics of
57 natural fibers seem to depend on cellulose content [13] and more generally on the environmental
58 conditions of plant growth or on the choice of maturity level ([14], [15]). Moreover, it can be
59 noticed that measuring techniques can partly explain the variability of measured properties ([16], [17]).
60 Vegetal fibers are natural, biodegradable and economical materials. Their addition
61 to materials has several objectives. Their main role is to reduce plastic shrinkage at early age
62 limiting the crack formation occurring during drying. Indeed, fibers allow distribution and
63 dissipation of the tensile stresses due to shrinkage of the clay fraction of the material more
64 effectively ([18], [19]). Vegetal fibers also favor the drying process by draining moisture
65 outwards through the fiber channels and contribute to lighten the building material. However,
66 Segetin et al., [20] showed that the addition of vegetal fibers may also promote shrinkage.

67 Fiber quality, quantity, and its distribution in the material directly affects the benefits and
68 disadvantages on the natural composite material. Therefore, the addition of fibers should be
69 made with care [20].

70 Some experimental studies are limited to the observation of the ductile behavior of raw earth
71 materials. For example Segetin et al., [21] in a laboratory study through the flexural test, used
72 the fiber from the New Zealand flax plant to reinforce soil-cement building materials in an
73 attempt to improve the ductility. They found that adding of 0.6% of fibers content to a soil-
74 cement matrix can significantly enhance the material ductility and avoid the failure pattern

75 displayed by specimens without fiber-reinforcement. Marandi et al., [22] studied the strength
76 and ductility behavior of silty-sand soils reinforced by randomly distributed palm fibers. The
77 composite soils were tested under laboratory conditions and examined for unconfined
78 compressive strength. Their results showed that an increase in the fiber inclusion rate, from
79 0.25 % to 2.5 %, resulted in the soil being more soft and ductile. This behavior led to the failure
80 of the soil specimens at higher axial strains. Krishna Rao et al., [23] investigated the
81 performance of silty-sand soils reinforced with linen fiber using the unconfined compressive
82 test. They pointed out that adding 0.75% of linen fibers has the dual benefit of increasing the
83 stiffness and ductility of the reinforced soil. However, again, the ductile behavior was observed
84 when comparing stress-strain curves for materials with and without fibers but the ductility
85 property was not estimated [23].

86 Because of the difficulty for accurate measurement of the ductility property for porous media,
87 few studies are effectively dedicated to estimate the ductility property of raw earth materials.
88 In the work of Fratini et al. [24], the study of the mineralogical, physical and mechanical
89 characterization of the adobe samples, coming from seven different old buildings in a poor state
90 of conservation, has been carried out together with an analysis of the “local earth” for
91 comparison. Ductility property was quantified by two parameters: the kinematic ductility and
92 the available kinematic ductility. Both ductility parameters for the samples obtained from
93 kneading the earth of the old bricks and the new bricks indicate a “ductile behavior” of all the
94 bricks, with both a fairly good source of resistance after the end of the elastic behavior and a
95 good reserve of resistance after the peak of strength. They concluded that these new bricks can
96 be used for the restoration of the given old buildings. Park [25] performed a series of unconfined
97 compressive tests on specimens of fiber-reinforced cemented sand. He evaluated how the fiber
98 ratio and the cement ratio could influence the measured strength and ductility characteristics of
99 cemented sand. The ductile behavior was quantified by the deformability index, D, which is

100 defined as a ratio of the axial strain at peak strength of fiber-reinforced specimen to that of non-
101 fiber-reinforced specimen. In the case of 1% fiber ratio, the values of D were greater than 4
102 regardless of cement ratios. Triaxial testing was used by Consoli et al., [26] to study
103 experimentally the influence of fiber and cement addition on the behavior of sandy soils. Their
104 findings showed that the inclusion of 3% of fiber on the soil samples containing 1% of cement,
105 reduced from 2.6 to 0.6 the brittleness index I_B . This index is defined as $I_B = (q_f - q_u)/q_u$, where
106 q_f and q_u are respectively the failure and ultimate deviatoric stresses. It demonstrates that the
107 failure behavior becomes increasingly ductile for the fibrous material.

108 Furthermore, numerous experimental studies were carried out to quantify ductility property of
109 reinforced concrete ([27], [28], [29], [30], [31]). Even if reinforced concrete cannot be
110 compared to the raw earth material, the ductility indicators defined for reinforced concrete can
111 be used for earth material.

112 For complex mixtures with several components, the Design Of Experiments (DOE) approach
113 is particularly adapted ([4], [32]). This investigating tool can be used for studying and
114 optimizing the mechanical properties of building materials. It is no longer a question to vary
115 successively the different components of the mixture, and the tests are defined by mathematical
116 criteria to match with the constrained experimental domain [33]. The quality of the
117 experimental design is based on a statistical analysis. Then characterizing and quantifying the
118 influence of each of the components and their interactions on the considered property can be
119 performed.

120 The main objective of this research paper is to quantify the ductility of a raw earth material
121 called Cématerre, with the aim of improving its plastic behavior. Cématerre is a French
122 company settled in Normandy region which developed a new concrete based on raw earth
123 material, in collaboration with the University of Le Havre Normandie. Its originality is its
124 ability to be cast in place such as a traditional concrete ([4], [19]). The choice of using flax

125 fibers is motivated by the important production of flax in Normandy, which is the first flax
126 producer region in France with 55% of total production [34]. Indeed, due to the benefits outlined
127 above, in addition to good mechanical properties, these fibers are considered as a good
128 alternative for construction and building materials.

129 To fulfill this objective, combinations of five-constituent mixtures composed of flax fibers,
130 lime, cement, water and silt were formulated by a D-optimal mixture design to investigate the
131 ductility. D-optimal mixture design is a computer-generated design. It is a class of Design Of
132 Experiments (DOE) which proposes the best set of experiments to be conducted via an
133 optimization process. The optimization is based on the maximization of the determinant of the
134 information matrix [33]. Two ductility indices were evaluated: First, the ductility index used in
135 the literature based on the pre-peak and post-peak regions of the stress-strain curve [35] and the
136 second, a new one estimated in the post-peak region of the stress-strain curve. The definition
137 of these two indices is given in section 2.2.3. The experimental domain was defined according
138 to three constraints that are presented in section 3.1: (i) the fundamental mixing constraint, (ii)
139 an economical and ecological constraint and (iii) a workability constraint.

140 A series of laboratory tests was carried out to study ductility after 90 days of curing time; then
141 the derived model was validated. The effect of each mixture component on the ductility of raw
142 earth concrete was then analyzed through a response trace plot. Thereafter, a normalized
143 representation of stress-strain curve was proposed to study post-peak ductility regardless of
144 material strength.

145

146 **2. Materials and experimental methods**

147 *2.1. Materials*

148 The raw earth concrete is a mixture of several materials as described in the following section.

149 *2.1.1. Soil material*

150 The used building material is natural silt, chosen because it is locally available in abundance.
151 In respect to particle size, the analysis is performed with two different methods (sieving [36],
152 [37]) and sedimentation [38].) based on the particle diameters (Fig. 1). It **should** be noted that
153 no fine-grained clay material ($< 2\mu\text{m}$) was detected as shown in the grading size distribution
154 curve (Fig.1). For this soil, the effective diameter, the Hazen uniformity factor and the curvature
155 factor are respectively $32\ \mu\text{m}$, 4.37 and 0.94. The Atterberg limits are respectively 20% for the
156 liquid limit and 6% for the plasticity index. Based on Atterberg limits and grading size curve,
157 and **also** according to LPC-USCS (ASTM D2487-11) standard [39], this soil is classified as
158 silty sand (SM). [Imanzadeh et al., \[4\]](#) have already described the property of this silty sand in
159 details.

160

161 *2.1.2. Binders*

162 Two binders are used to prepare a raw earth concrete: lime and cement. The used lime comes
163 from the Proviacal® DD range. It is a calcic quicklime CL 90-Q (R5, P3), containing 90.9 %
164 available CaO and reactivity $t_{60} = 3.3$ minutes [40]. The used cement is CEM I 52.5 N, in respect
165 to the NF EN197-1 [41], NF P15-318 [42] and NF EN196-10 [43] standards. More information
166 about these binders' properties is given in details in [Eid 2017 \[18\]](#).

167

168 *2.1.3. Flax fibers*

169 The used flax fibers are extracted locally from the region of Normandy. The choice of flax fibers
170 is motivated by the important production of flax in Normandy, which is the first flax producer
171 in France with 55% of total production [34]. Their physical and mechanical properties (density
172 ρ , fiber diameter d , Young's modulus E , failure stress s_u and failure strain ε_u) are listed in Table
173 1. It is clear that these properties can vary greatly depending on the environmental conditions
174 of plant growth, choice of maturity level and the **measurement methods** ([16], [17], [44]). These

175 mean data show that, in the domain of natural vegetal fibers, flax fibers (Fig. 2) offer good
176 mechanical performances ranking them just after hemp fibers in terms of elastic modulus and
177 tensile strength ([45], [46]). This is due mainly to the high cellulose and low lignin mass
178 contents in flax fibers. Furthermore, there is a link between the cellulose content and the
179 mechanical characteristics of fibers [13].

180 **The used fibers** are natural fibers which have not undergone any chemical surface treatment.
181 **Fibers have diameters ranging between 10 to 15 μm [47] with 50 to 70 mm lengths.** According
182 to their high aspect ratio (length/diameter), of order 5000, flax fibers ensure a good contact
183 surface with the matrix allowing the load transfer to the material. However, their amount in
184 specimen should be limited for different reasons. First, an excess of fibers may result in slipping
185 phenomena between fibers and limiting friction with the mineral matrix. **Furthermore, it should**
186 **be noted that** incorporating fibers without favoring any fiber orientation and with a final
187 homogeneous distribution in the material is a hard task. It will be even more difficult when
188 preparing the material as a traditional concrete on the construction site. It gives another
189 argument in favor of a fiber content limitation. Given all these arguments, fiber content was
190 varied in the range of 0.3% to 0.45% in mass: 0.3% was considered **as a** low level and 0.45%
191 **as a** high level of fiber content in specimens.

192 193 2.1.4. Incorporation of a superplasticizer additive

194 Since raw earth concrete shrinkage depends on the amount of mixing water, the minimization
195 of shrinkage therefore requires **a limited water content**. However, to preserve the consistency
196 when manufacturing the concrete, the addition of a superplasticizer additive can be an
197 alternative. The additive, referenced SIKA VISCOCRETE TEMPO-10, is a new generation
198 superplasticizer based on acrylic copolymer, according to NF EN 934-2 standard [48]. It
199 contains no chlorides or other substances likely to cause or promote the corrosion of steel, and
200 therefore it can be used without restriction for the construction of reinforced and pre-stressed

201 concrete structures. The super-plasticizer contributes to deflocculating fine grains and to
202 lubrication of the solid surfaces, decreasing the friction stresses between particles ([49], [50]).
203 An amount of 0.3% to 0.5% of cement weight is recommended. It reduces the water content
204 from 15% to 25% in the cement pastes without modifying the consistency. A similar dosing,
205 based on the proportion of cement in the mixings, was chosen for present tests. A constant
206 amount of additive of 5 ml/ m³ has been used for each sample preparation.

207 A potable tap water from the pipe in the laboratory has been used for preparing the raw earth
208 concrete.

209 2.2. Experimental methods

210 2.2.1. Sample preparation

211 The preparation of specimens including fibers and cement requires a particular attention to
212 obtain homogeneous samples with a random distribution of the fibers. The mixing procedure is
213 carried out in a laboratory mixer with a capacity of 4 liters including of two successive phases:
214 in the first phase, a dry mixing during two minutes of soil, binders and fibers, then, in the second
215 phase, water and additive are added for a wet mixing during three minutes. The used silt was
216 first oven-dried at 60 °C for 48 hours in order to control the amount of water in the specimens.
217 Thereafter, the molds of 100 mm of height and 50 mm of diameter are filled by vibration for
218 two minutes with a vibrating table. Then, the specimens were stored in a mold for 90 days of
219 curing-time in controlled laboratory environment (relative humidity RH ≈ 50 % and
220 temperature: T ≈ 22 °C) prior to their testing. After 90 days of curing-time, it can be considered
221 that the majority of the chemical reactions were achieved due to binders.

222 2.2.2. Unconfined Compressive Strength (UCS) test

223 Laboratory-prepared raw earth concrete specimens with different mix proportion were
224 submitted to an axial Unconfined Compressive Strength (UCS) test according to NF P94-420
225 [51], NF P94-425 [52] French standards. The Unconfined Compressive Strength test was

229 preferred to the bending test ([28], [31], [35], [53]) because the studied raw earth concrete is
230 destined to build non-reinforced structural elements only subjected to axial compression ([25],
231 [54], [55], [56]). Therefore, the ductility parameters are estimated from the applied axial stress
232 versus axial strain curve deduced from UCS test.

233 The experimental device includes a press with a maximum load capacity equal to 100 kN and
234 a potentiometric displacement sensor with an accuracy of ± 0.05 mm connected to an
235 acquisition center which is controlled by a computer. The tests have been performed at
236 controlled displacements. A strain rate of 0.1 mm / min was chosen for all the tests. More
237 information about the experimental device was given by Imanzadeh et al. [4].

238 The unconfined compressive strength test is used to plot the material stress-strain curve. Fig.
239 3a presents the stress-strain curve for formulation F9 after 90 days of curing time. This curve
240 is used to illustrate the response curve of the studied raw earth material stabilized with binders
241 and fibers. The stress-strain curve is complex and characteristic of a ductile material with four
242 zones delineating a distinct behavior of the porous material. For low strains (A-zone), the curve
243 is concave, typical of soft porous materials. This is due both to the experiment with the
244 implementation of a homogeneous contact between the specimen and the press and to a physical
245 process: the closure of natural microcracks in the porous material stabilized with binders. As
246 strain increases more, the material already undergoes large deformation. The linear curve
247 portion with maximum slope is detected (B-Zone). At the beginning of C zone, the stress-strain
248 curve exhibits significant nonlinearity. The C zone is described as a plastic zone, usually
249 associated to the non-linear phase due to micro-cracking. The material gains its strength thanks
250 to the strong bonds developed from cementation and internal friction. The slope decreases due
251 to breaking of the cement bonds until the maximum compressive strength is reached. At this
252 point, all the cement bonds at the failure surface have broken. The post-peak zone (D-zone)
253 corresponds to the material behavior after the appearance of a failure plane. The observed

254 reversal of the stress-strain curve is the consequence of the ductile nature of the raw earth
255 material. Flax fibers tend to maintain a relative cohesion of the material that exhibit a ductile
256 failure.

257 258 2.2.3. Tools for ductility analysis

259 To study the ductility of the material, two ductility indices i and i' are proposed and used to
260 characterize the ability of the material to withstand plastic deformation. The first one is
261 representative for the major part of the plastic zone ([35], [57]) and the second one focuses on
262 the post-peak behavior. The i index is an adapted form of the displacement ductility index
263 defined by Cohn and Bartlett [27] for cement concrete. The i index is defined as $i = \varepsilon_3 / \varepsilon_1$ (Eq.
264 1) where ε_3 refers to the axial strain when UCS drops down to 0.85 times of UCS_{max} and ε_1
265 refers to the axial strain when the tangent to the linear section of maximum slope intercepts
266 UCS_{max} line (Fig. 3b).

$$267 \quad i = \frac{\varepsilon_3}{\varepsilon_1} \quad Eq. 1$$

268 The i' ductility index is defined as $i' = \varepsilon_3 / \varepsilon_2$ (Eq. 2) where ε_2 refers to the axial strain for UCS_{max}
269 (Fig. 3b).

$$270 \quad i' = \frac{\varepsilon_3}{\varepsilon_2} \quad Eq. 2$$

271 272 3. Experimental design

273 The considered mixture constraints are described in the following section before explaining the
274 generation of the experimental design.

275 276 3.1. Mixture constraints

277 Three constraints must be considered:

278 - Fundamental constraint where the sum of the ingredients of the mixture is 100 % in weight
279 for all the mixes of the design: Fiber % + Lime % + Cement % + Water % + Silt % = 100 %

280 - Economical and ecological mixture constraints: The raw earth concrete has to be designed to
281 provide the appropriate mechanical and durability properties to be used as a construction
282 building material. In addition, it should be non-energy-intensive in regard to gray energy. Thus,
283 the percentage of added binders (cement and lime) should be limited. For these reasons, the
284 maximum quantities of cement and lime are respectively limited to 16% and 12% [4]. Then,
285 the following condition must be verified: $\text{Cement \%} + \text{Lime \%} < 16 \%$.

286 In agreement with the mentioned constraints above, mixing range chosen for each of the
287 constituents, using fiber, is presented in Table 2.

288 - Workability constraint: workability plays an important role on the mechanical properties. It
289 depends on several properties such as consistency, plasticity and cohesion ([58], [59]).
290 Consistency is, unlike plasticity and cohesion, easy to measure. That is why it is currently used
291 to characterize workability. The classes of consistency were measured on fresh mixtures,
292 according to Abrams cone test (standard NF EN 206-1 [60]). A S3 level of consistency
293 calibrated by standard slump test was chosen to ensure a fluidity similar to a very plastic
294 concrete. A preliminary experimental design [2] was conducted to evaluate a multiple linear
295 regression equation for consistency (Eq. 3). The condition of suitable consistency level (slump
296 S3) sets the boundaries of the equation validity. The condition fitted for mixings with fibers
297 was compared to the equation established for raw earth materials without fibers (Eq. 4, [2], [4])
298 according to a similar experimental protocol.

299 The resolution of workability equations with the condition of fundamental constraint and
300 economical and ecological mixture constraints delineates the regions of acceptable mixtures.

301 The 4- or 5- dimensional spaces defined by Eq. 3 and Eq. 4 were projected in the (cement,
302 water) plane. Fig. 4 depicts the domains of valid workability condition for the proportion of
303 lime fixed to $X_{\text{lime}} = 0.02$. It is representative of what occurs in the whole domain of workability
304 condition validity. It is shown that the two regions are very close with an increase in order of

305 1-2% of water content for mixtures with fibers compared to mixtures without fibers. In spite of
306 the hydrophilic nature of fibers (absorption >100% for flax fibers [14], [61]), it highlights the
307 important water-reducer role of the plasticizer admixture used for tests with fibers.

308

$$309 \quad 2.5 \leq 1.7 - 0.3 \text{ fiber} - 19 \text{ lime} - 6 \text{ cement} + 28 \text{ water} - 7.5 \text{ silt} \leq 2.7 \quad \text{Eq. 3}$$

$$310 \quad 2.5 \leq - 22 \text{ lime} - 9 \text{ cement} + 42 \text{ water} - 9 \text{ silt} \leq 2.7 \quad \text{Eq. 4}$$

311

312 *3.2. Generation of the experimental design*

313 In this research study, experimental region is constrained by three conditions (see section 3.1)
314 to an irregular 3D-polyhedron. Thereafter, D-optimal design based on a computer-aided is
315 adapted to generate the set of experiments. The selection process of the best candidate set of
316 experiments is performed by a D-optimal criterion, applied to maximize the information
317 contained in the different possible data sets and the quadratic model that will be fitted. The G-
318 efficiency criterion is then estimated to choose the best D-optimal data set [62]. The efficiency
319 values of at least 50% are considered acceptable for design purposes [63].

320 In this study, the mixture design was developed through a D-optimal design including three
321 constraints (see section 3.1). A sub-set of 27 formulations was selected from the candidate set
322 proposed by MODDE© software. This choice is validated by a good level of design statistics
323 indicators of efficiency. Indeed, the G-efficiency measure is equal to 66.04 % [64]. The 27
324 formulations (Table 3) were tested in the random run order suggested by the software [65] with
325 two repetitions. Three estimations at the center point of the experimental domain were included.
326 The center point plays an important role. It is used to estimate the variability of the response as
327 well as curvilinear effects. Thereafter, the Partial Least Square projections to latent structures
328 (PLS) adapted to complex experimental design data [66] were used to calculate the model
329 coefficients. For each formulation, three specimens were tested and the mean of the measured
330 values was considered except for the center point for which the three data were entered (F25,

331 F26 and F27, Table 3). The measured values for both ductility indices i and i' are also presented
332 in Table 3. Furthermore, the standard deviation of ductility index was calculated for the
333 different formulations. All the standard deviations (σ) are smaller than 0.1 except for
334 formulation F21 ($\sigma = 0.16$). Due to the small standard deviations, the ductility values can be
335 considered with confidence.

336

337 4. Statistical analysis of regression models

338 Quadratic polynomial model was applied to the five-component mixings issued from the
339 experimental design to make the prediction of the two ductility indices i and i' . This quadratic
340 model includes the different terms in relation to the binary interactions for all possible pairs of
341 ingredients ([67] [68]).

342 The Analysis Of Variance (ANOVA) includes several diagnostic tools that are used to validate
343 models ([64], [68], [67]). The two first diagnostic tools are the regression coefficients (R^2 and
344 R^2_{adj}), informing on the ability of the model to fit the measured data. Furthermore, Q^2 coefficient
345 estimates the model validity *i.e.*, its ability to predict new data. The model validity should also
346 be verified ([69], [67]).

347 The first F-Test is the regression model significance test. It compares regression variance (SS_R)
348 to residual variance (SS_r). The critical Fisher value for a probability at a fixed 95% confidence
349 interval should be smaller than estimated F-value for the model. It reflects the statistical ability
350 of prediction of the model.

351 For the second F-Test (also called the lack of fit test), residual error is decomposed in two parts:
352 lack of fit (LOF) due to imperfection of the model and Pure Error (PE) estimated from replicates
353 data error. The calculated F-value for the model should be smaller than the critical acceptable
354 value at 95% confidence interval.

355

356

357 **5. Results**

358 *5.1. Observations*

359 All the specimens failed in a ductile manner with ductility indices varying between 1.27 and
360 5.12 for i index and between 1.13 and 2 for i' index (Table 3). It can be noted that some data
361 **are missed** in Table 3. Five formulations are concerned (F2, F4, F5, F15 and F19). These
362 specimens show steadily **increases of the strength** with no maximum compressive strength
363 reached. The example of formulation F2 is given in Fig. 5.

364 The classical failure with a well-identified inclined plane is observed when the smallest
365 dimension of the sample is 10 times larger than the size of the biggest particles in the specimen
366 **which guarantees** the mechanics of continuous media hypothesis [70]. For mixtures with fibers,
367 the failure mode can differ from the classical one, as the length of fibers can be as large as the
368 diameter of the specimen. For some formulations, the mechanics of continuous media
369 hypothesis is no longer valid. Therefore, the failure mode becomes random and the failure plane
370 can intersect the top and the bottom of the sample. The friction at the top and at the bottom
371 prevents the development of the failure, and in this case, a continuous increase in strength is
372 observed, without observing a peak of maximum strength. These observations are consistent
373 with the results of the literature on bio-materials based on plant fibers [71].

374 The evaluation of ductility indices cannot be performed for these five specimens and these cases
375 are disregarded for the ductility exploration. Thus, indices were calculated for 22 different
376 formulations. The variation range, by a factor of 4.03 for i and 1.77 for i' is relevant of a strong
377 dependence with the composition of mixtures (Fig. 6).

378 As mentioned earlier, the tests order is random and successive formulations correspond to
379 materials with proportions of the five components **while vary all together**. Ductility analysis
380 cannot be conducted from the direct analysis of Fig. 6 and an approach by DOE is presented in
381 the next section. However, some general remarks can be made. First, it was found that for some

382 specimens (for example F1), i' index is close to unity and i index tends to i' index. A $i=i'=1$
383 condition would correspond to brittle failure of fragile material that hardens with a constant rate
384 and then breaks suddenly when its maximum strength is reached. It is also shown that the two
385 indices clearly do not exhibit the same evolution with a greater amplitude of variation for i
386 index than for i' index. Finally, four formulations (F3, F6, F16 and F17) are noticed **to have**
387 **much greater i index** among the others. As the generation of mixture planification by DOE
388 insures a good repartition of formulations in the constrained experimental domain, the four
389 formulations mentioned above, corresponding to the most ductile specimens tested, need to be
390 examined with attention.

391

392 5.2. Model validation

393 Linear and quadratic polynomial models were first fit to the measured experimental data to
394 identify the most adequate models representing the measured experimental data. The
395 coefficients of the linear and quadratic models for i and i' ductility indices were given in Table
396 4. The values of R^2 , R^2_{adj} and Q^2 were presented in Table 5. As shown in Table 5, for i' ductility
397 index, the two regression coefficients (R^2 , Q^2) noticeably increase when a quadratic model is
398 chosen. Indeed, the interactions between components of the mixture are strong, pointed out by
399 large values of interaction coefficients (Table 4). This indicates a non-linear dependence of the
400 ductility property with the mixture components. However, one can also note a slight decrease
401 in the R^2_{adj} coefficient. Otherwise, for i ductility index, all the three coefficients are smaller than
402 the ones obtained for i' ductility index, for both linear and quadratic models. Furthermore, the
403 statistic indicators of the model validity for i are very low. It can be concluded that a quadratic
404 model can be adapted for i' ductility index but it failed for i ductility index. Then, the statistical
405 model validation procedure for i' ductility index is continued via F-tests of ANOVA.
406 Concerning the first F-Test for i' , the model F-values are higher than the critical Fisher values
407 for a probability at a 95% confidence level (Tables 6 and 7). The P-value is smaller than the

408 significance level, **which shows that** that selected model passed the first F-Test. For the second
409 F-Test, the residual error and the pure error from replication are compared and F-values for the
410 model are estimated. In respect to the criterion, the estimated F-value for the model is
411 significantly smaller than the critical acceptable value at 95% confidence interval with an
412 acceptable P value equal to 0.56 (Tables 6 and 8). Thus, one can reasonably consider that chosen
413 model is valid. Once the best-fit model was selected, an equation for the post-peak ductility
414 index i' was provided for the studied raw earth concrete.

415 One can conclude at the end of this section that it is possible to modelize the ductile behavior
416 of raw earth material stabilized with lime, cement and fibers in the post-peak zone. In the D-
417 zone of stress-strain curve, material undergoes deformation without breaking, tending towards
418 a residual strength, because of internal friction that is the only mechanism that sustains the
419 material. The difficulty to determine a reliable model for i index indicates that characterizing
420 all the plastic domain for this five-component material is a hard task. The pre-peak zone where
421 material exhibits an elasto-plastic behavior cannot be predicted accurately. Indeed, the
422 debonding of the cementitious matrix and friction between the matrix and fibers are
423 mechanisms that contribute in a complex imbricated manner to ductility.

424

425 *5.3. Response trace plot of post-peak ductility index*

426 **The response trace plot is a useful tool to observe the behavior of the predicted values versus**
427 **the change of proportion of each constituent.** Response trace plots were performed; the response
428 of ductility index i' was drawn around a reference mixture. In this research study, the considered
429 reference mixture is the centroid of the constrained experimental domain with the quantities of
430 fiber, lime, cement, water and silt respectively equal to 0.375 %, 3.015 %, 8.402 %, 23.953 %
431 and 64.255 %.

432 Response trace plots of ductility index i' with deviation from reference mixture in proportion
433 for **90 days of** curing time, for the quadratic and linear models, were shown in Fig. 7. This figure

434 points out how each constituent influences on predicted ductility index in respect to the
435 reference mixture. For the reference mixture, the ductility index value is equal to 1.53 (Fig. 7).
436 The ductility index values are very sensitive to changes in cement and silt proportions. The
437 steep slope for silt shows the major influence of this constituent. Figure 7 indicates that an
438 increase of silt proportion in the mixing tends to increase the ductility index. Indeed, since silt
439 exhibits an elasto-plastic behavior and is naturally ductile, it can withstand large strains during
440 loading. Therefore, an increase of silt proportion in the mixture promotes the plasticity of the
441 material, therefore its ductility.

442 Concerning the cement influence, an increase of the proportion of cement in the mixing
443 decreases the ductility index. This negative effect on ductility is due to pozzolanic chemical
444 reactions involving cement that generate a very hard bond between the soil particles. **When the**
445 **load reaches its maximum strength, and even when the cement proportion increases, links can**
446 **break suddenly in a brittle way.**

447 Water and water interaction effect with the other constituents (quadratic model) show a negative
448 effect on the ductility index (Fig. 7 a). In contrast, Fig. 7b shows the positive effect of water in
449 the absence of the water interaction effect with other constituents (linear model). The latter
450 model points out that this negative effect in the quadratic model is due to the water interaction
451 effect with binders. Indeed, when the amount of water increases, the excess water, that is not
452 necessary for the hydration of the binders, evaporates. This evaporation generates a suction
453 (negative pore water pressure) which is at the origin of the shrinkage of soils. **This shrinkage**
454 **induces a densification of the material and consequently decreases its ductility.**

455 The response trace for lime has a negative slope indicating a decrease on the ductility index.
456 Since lime is a binder, its effect is similar to that of cement as explained above.

457 Concerning the fiber effect, the steep slope for fiber indicates the major influence of this
458 component compared with other components. However, its positive effect on the ductility index

459 is very limited because of the small range of variation of fiber proportion in mixings between
460 0.3% and 0.45%. To study the influence of fiber content, it is required to increase the range of
461 its variation.

462

463 **6. Discussion**

464 As mentioned in the introduction, ductility is a desired property but apparently not quantified
465 for raw-earth material. Discussion focuses on a further analysis of the evolution of the two
466 ductility indices concerning fiber content influence and (silt/binders) ratio that emerge as a
467 relevant parameter.

468 *6.1. Normalized representation of stress-strain curve*

469 In order to compare more easily the ductility level of the specimens, a normalized representation
470 of the stress-strain curve is used (Fig. 8). The y-axis designates UCS* defined as UCS/UCS_{max}
471 where UCS_{max} stands for the maximum compressive stress and the x-axis designates $\varepsilon^* = \varepsilon/\varepsilon_{max}$
472 where ε_{max} is the strain associated to UCS_{max} . This representation is particularly adapted to
473 compare ductility of different materials since curves are gathered together at the same scale for
474 the two axes. The stress-strain curves for the two formulations F1 and F22 are plotted in a
475 normalized form (Fig. 8). With this plot, ductility can be characterized regardless of the
476 maximum stress level of the material. The arrow directly gives the i' post-peak ductility index
477 because the strain associated to UCS_{max} is equal to unity for all materials. The length of the pre-
478 peak and post-peak zones can also be compared. A marked dissymmetry of the plastic domain
479 is noted for the two specimens with a greater extension of the post-peak zone compared to the
480 pre-peak zone. Of course, the two domains are more extended for the more ductile material
481 (F22) containing fewer binder. A steeper slope of the linear zone is noted for the more ductile
482 material. It should be noted that the slope does not reflect the strain modulus of the material in
483 this normalized representation.

484

485 6.2. Influence of fibers

486 The normalized stress-strain curves are represented for pairs of formulations almost similar
487 except by the fiber content that can be low (fibers = 0.3%) or high (fibers = 0.45%). This
488 normalized representation is a useful tool to evaluate i' ductility index, as its value can be
489 directly read on the x-axis. Three different comparisons are plotted on figure 9 a, b and c
490 respectively for (F7, F8), (F20, F9) and (F3, F6). While when the ductility is low (Fig. 9a), a
491 weak influence of the variation of fiber content is noted, a clear difference is shown for the
492 more ductile materials (Fig. 9 b and c). The material containing 0.45% fibers is more ductile
493 than the one containing 0.3%. From Fig. 9, one can deduce the ductility index i' and $\varepsilon^* = \varepsilon_l/\varepsilon_{max}$
494 as shown in Table 9. For example, in Fig. 9 b, i' is about 1.29 for 0.35% of fibers and i' is 1.41
495 for 0.45%. It also seems that pre-peak zone is more affected by the level of fiber content than
496 post-peak zone. As shown in this table, for example for Fig. 9 b, the value of ε^* is equal to 0.82
497 for the fibers content of 0.35% and 0.69 for the fibers content of 0.45%.

498
499

500 6.3. Influence of (silt/binder) ratio on ductility indices

501 In the experimental domain studied, the range of (silt/binders) ratio was defined on an extended
502 range from 3.5 to 18. Ductility values for the two indices, issued from the DOE study (see
503 section 5.2, Table 4) and obtained experimentally are shown in Fig 10 as a function of
504 (silt/binders) ratio. For a ratio of 1:18 between silt and binders, binder proportion was reduced
505 to 5.6% compared to silt proportion while for 1:3.5, more than 25% of binders proportion is
506 reached. As expected, the breaking is more brittle when (silt/binders) ratio is decreased. If soil
507 particles are not completely enclosed by binders when the ratio of silt/binders increases, the
508 ductile influence of silt becomes predominant and ductility increases. On the contrary, when
509 silt/binders ratio decreases due to an increase in binder proportion, the cement chains break
510 abruptly and this leads to a decrease in ductility. Furthermore, the two indices increase when

511 the binders proportion **decreases** compared to silt proportion, whatever the fibers content in the
512 range tested. One can also note that i' index seems to saturate when a threshold ratio, around
513 13, is exceeded. This means that when the (silt/binders) ratio exceeds 13, the ductility value i'
514 can converge towards the ductility of the untreated silt (without binders). Since a clear
515 dependence of i' ductility index with (silt/binders) ratio is observed, the search for an
516 experimental law was conducted. In the range of compositions tested, the post-peak ductility
517 index i' can be expressed in the form of a 2nd order polynomial (Eq. 5, Fig. 10) with a regression
518 coefficient of $R^2=0.94$. However, the use of such a law needs to be very careful because of the
519 great dependence on the experimental protocol chosen for the preparation of test specimens.

520

$$521 \quad i' = -0.004 (\text{silt / binders})^2 + 0.14 (\text{silt / binders}) + 0.79 \quad \text{with } R^2=0.94 \quad \text{Eq. 5}$$

522

523 All the formulations were used to find a relation between i' and the silt/binder ratio. The
524 independence of i' with the fiber proportion in Eq. 5 does not mean that fiber does not affect
525 ductility but that no significant change in ductility is noted when the fiber proportion varies in
526 **the testing range** (0.3% to 0.45%).

527 As was already noted when results of DOE were analyzed (see section 5.2), it was not possible
528 to propose a similar model for i index. Indeed, as mentioned above, the four formulations F3,
529 F6, F16 and F17 are characterized by very high i values compared to the other formulations.
530 The observed gap in i index can be supported by the idea that - for formulations with low binder
531 proportion with respect to silt - chains of cementitious bonds between silt particles are short
532 and do not allow the development of a rigid matrix. Thus, during the loading phase, chains
533 simultaneously and brutally break, independently of the silt/binders ratio as long as this ratio is
534 large enough (silt/binders >7-8). Then, only the ductile influence of silt and fibers remains.

535 The ductility of the studied materials described by the i index adapted from the literature is at
536 most 3 times the ductility of a brittle material. Raw earth reinforced with flax fibers could then
537 reach a ductile level, providing an interesting damage tolerance.

538 The ductility index i' reflects only partially the plastic nature of the material. However, if a
539 level of strength is achieved for a material, the knowledge of the post-peak ductility may be
540 sufficient to conclude on its ability to deform. Actually, a high i' indicator is always associated
541 to a high i indicator. Furthermore, a factor of about 2 for the range of variation of i' index is
542 convenient to evaluate significant changes in the material behavior in the post-peak region of
543 the stress-strain curve.

544 The comparison between present ductility indices (i and i') with those from the literature is
545 limited. Indeed, the ductile behavior is generally observed on stress-strain curve in axial loading
546 test or force-displacement curve in bending test but ductility index is rarely estimated and
547 analyzed. Present data for i ductility index were compared to data on reinforced cement concrete
548 from Rakhshanimehr et al., [35]. A similar range of i index is found for the two types of
549 materials: 1.15 to 7 for reinforced cement concrete [35] and 1.27 to 5.12 for the studied raw
550 earth concrete. No data is available in the literature for i' index introduced herein.

551 Finally, the discussion confirms that the relevant parameter for quantifying ductility is the
552 ductility index i' since it is adapted to the study of all the stress-strain behaviors in the range of
553 material tested.

554

555 **7. Conclusion**

556 A building eco-material based on raw earth stabilized with binders and vegetal fibers was
557 produced. The ductile behavior is one of the desired characteristics particularly in the context
558 of an exploitation in seismic zones and the quantification of this mechanical property is
559 necessary. Among the constraints associated to the sample preparation of materials with varying

560 cement and fiber content, the workability condition is essential. The major role of the super-
561 plasticizer admixture is pointed out for the elaboration of workable material with fibers.

562 The axial stress-strain curve provides the basis to study ductility. An alternative ductility index
563 was proposed in order to evaluate post-peak behavior of ductile raw earth material stabilized
564 with fibers. It can be easily evaluated on the basis of a normalized representation of the stress-
565 strain curve of the material. By using this new tool, post-peak ductility of different materials
566 can be directly compared regardless of their compressive stress. Ductility of present specimens
567 was studied with a mixing design. The post-peak plastic domain described by the i' ductility
568 index, where fibers play a key role was studied and modeled successfully with a quadratic
569 model. Strong interactions between the components of the mixing are demonstrated. In contrast,
570 ductility of the plastic domain defined by i ductility index, could not be modeled. It indicates
571 the difficulty to predict ductility of this domain dominated by brittle cement bonds breakings
572 and mobilized friction processes. In an attempt to understand the influence of mixing
573 composition on ductility, silt/binders ratio was calculated and its influence on ductility index
574 was clearly pointed out. **The ability of fibers to reinforce ductility property increases with
575 silt/binders ratio. An increase in the mobilized friction can be linked the silt-fiber interface
576 increases.** Consequently, the collapse of the material is delayed and the ductility is increased.

577 In the light of its ability to predict post-peak ductility of a raw earth material with fibers, the
578 use of i' alternative ductility index could be extended to other raw earth materials. Finally, i'
579 index could be used to define a ductility criterion to meet specific requirements of seismic
580 regions.

581 **The difference in content over the range of testing fibers (0.3 to 0.45% in mass) does not impact
582 significantly the ductility performance of the earthen material. Further investigation with a
583 larger range of fiber content should be undertaken to analyze its influence and to optimize its
584 content.**

585 **Acknowledgement**

586 The authors wish to express their gratitude to the company Cématerre, the CNRS (French
587 National Center for Scientific Research) and the FEDER (European Economic and Social
588 Development Fund) for their support.

589

590

- [1] P. Přikryl, Á. Török, M. Theodoridou, M. Gómez-Heras and K. Miskovsky, "Geomaterials in construction and their sustainability, Sustain. Use Tradit," *Geomaterials Constr. Pract.*, p. 1–22, 2016, doi: 10.1144/SP416.21.
- [2] A. Hibouche, Sols traités aux liants, Performances hydro-mécaniques et hygro-thermiques, Applications en BTP, Dissertation, Normandie Univ., UNIHAVRE, 2013.
- [3] P. Zak, T. Ashour, A. Korjenic, S. Korjenic and W. Wu, "The influence of natural reinforcement fibers, gypsum and cement on compressive strength of earth bricks materials," *Constr. Build. Mater.*, vol. 106, p. 179–188, 2016, doi: 10.1016/j.conbuildmat.2015.12.031.
- [4] S. Imanzadeh, A. Hibouche, A. Jarno and S. Taibi, "Formulating and optimizing the compressive strength of a raw earth concrete by mixture design," *Constr. Build. Mater.*, vol. 163, p. 149–159, 2018, doi:10.1016/j.conbuildmat.2017.12.088.
- [5] T. Ashour, A. Korjenic, S. Korjenic and W. Wu, "Thermal conductivity of unfired earth bricks reinforced by agricultural wastes with cement and gypsum," *Energy Build.*, vol. 104, p. 139–146, 2015, doi:10.1016/j.enbuild.2015.07.016.
- [6] J. M. Kanema, J. Eid and S. Taibi, "Shrinkage of earth concrete amended with recycled aggregates and superplasticizer: Impact on mechanical properties and cracks," *Mater. Des.*, vol. 109, p. 378–389, 2016, doi:10.1016/j.matdes.2016.07.025.
- [7] M. Al-Mukhtar, A. Lasledj and J. F. Alcover, "Lime consumption of different clayey soils," *Appl. Clay Sci.*, vol. 95, p. 133–145, 2014, doi:10.1016/j.clay.2014.03.024.
- [8] A. Lasledj, Traitement des sols argileux à la chaux : processus physico-chimique et propriétés géotechniques, PhD Thesis. University of Orleans, France, 2009, p. 361.
- [9] M. Carmen Jimenez Delgado and I. Canas Guerrero, "Earth building in Spain," *Constr. Build. Mater.*, vol. 20, p. 679–690, 2006, doi:10.1016/j.conbuildmat.2005.02.006.
- [10] E. Araldi, E. Vincens, A. Fabbri and J. P. Plassiard, "Identification of the mechanical behavior of rammed earth including water content influence," *Mater. Struct.*, pp. 51-88, 2018, doi:10.1617/s11527-018-1203-2.
- [11] F. Loccarini, Behaviour of rammed earth structures: Sustainable materials and strengthening techniques, PhD dissertation, Civil Engineering and Environmental Sciences, University of Braunschweig – Institute of Technology and Department of Civil and Environmental Engineering, University of Florence, Italy, 2017.
- [12] P. H. Joen and R. Park, "Flexural strength and ductility analysis of spirally reinforced prestressed concrete piles," *PCI JOURNAL*, pp. 64-83, 1990.
- [13] Y. Millogo, J. C. Morel, J. E. Aubert and K. Ghavami, "Experimental analysis of Pressed Adobe Blocks reinforced with Hibiscus cannabinus fibers," *Constr. Build. Mater.*, vol. 52, p. 71–78, 2014, doi:10.1016/j.conbuildmat.2013.10.094.

- [14] K. Charlet, Contribution à l'étude de composites unidirectionnels renforcés par des fibres de lin : relation entre la microstructure de la fibre et ses propriétés mécaniques, Dissertation, Université de Caen, 2015.
- [15] S. H. Hulleman, J. M. Van Hazendonk and J. E. Van Dam, "Determination of crystallinity in native cellulose from higher plants with diffuse reflectance Fourier transform infrared spectroscopy," *Carbohydrate Research*, vol. 261, p. 163–172, 1994.
- [16] S. M. Hejazi, M. Sheikhzadeh, S. M. Abtahi and A. Zadhoush, "A simple review of soil reinforcement by using natural and synthetic fibers," *Constr. Build. Mater.*, vol. 30, p. 100–116, 2012, doi:10.1016/j.conbuildmat.2011.11.045.
- [17] H. L. Bos, M. J. Van den Oever and O. C. Peters, "Tensile and compressive properties of flax fibres for natural fibre reinforced composites," *Journal of Materials Science*, vol. 37, no. 8, pp. 1683-1692, 2002.
- [18] J. Eid, "New construction material based on raw earth: cracking mechanisms, corrosion phenomena and physico-chemical interactions," *Eur. J. Environ. Civ. Eng.*, vol. 8189, p. 1–16, 2017, doi:10.1080/19648189.2017.1373707.
- [19] J. Eid, S. Taibi, J. M. Fleureau and M. Hattab, "Drying, cracks and shrinkage evolution of a natural silt intended for a new earth building material. Impact of reinforcement," *Constr. Build. Mater.*, vol. 86, p. 120–132, 2015, doi:10.1016/j.conbuildmat.2015.03.115.
- [20] H. Khelifi, T. Lecompte, A. Perrot and G. Ausias, "Mechanical enhancement of cement-stabilized soil by flax fibre reinforcement and extrusion processing," *Mater. Struct. Constr.*, vol. 49, p. 1143–1156, 2016, doi:10.1617/s11527-015-0564-z.
- [21] M. Segetin, K. Jayaraman and X. Xu, "Harakeke reinforcement of soil-cement building materials: Manufacturability and properties," *Build. Environ.*, vol. 42, p. 3066–3079, 2007, doi:10.1016/j.buildenv.2006.07.033.
- [22] S. M. Marandi, M. H. Bagheripour, R. Rahgozar and H. Zare, "Strength and Ductility of Randomly Distributed Palm Fibers Reinforced Silty-Sand Soils," *Am. J. Applied Sci.*, vol. 5, no. 3, p. 209–220, 2008,.
- [23] S. V. Krishna Rao and A. M. Nasr, "Laboratory study on the relative performance of silty-sand soils reinforced with linen fiber," *Geotech. Geol. Eng.*, vol. 30, p. 63–74, 2012, doi:10.1007/s10706-011-9449-2.
- [24] F. Fratini, E. Pecchioni, L. Rovero and U. Toniatti, "The earth in the architecture of the historical centre of Lamezia Terme (Italy): Characterization for restoration," *Applied Clay Science*, vol. 53, p. 509–516, 2011, doi:10.1016/j.clay.2010.11.007.
- [25] S. S. Park, "Unconfined compressive strength and ductility of fiber-reinforced cemented sand," *Constr. Build. Mater.*, vol. 25, p. 1134–1138, 2011, doi:10.1016/j.conbuildmat.2010.07.017.
- [26] N. C. Consoli, P. D. Prietto and L. A. Ulbrich, "Influence of fiber and cement addition on behavior of sandy soil," *J. Geotech. Geoenvironmental Eng.*, vol. 124, p. 1211–1214, 1998.

- [27] M. Z. Cohn and M. Bartlett, "Computer-simulated flexural tests of partially pre-stressed concrete sections," *ASCE Journal of Structural Division*, vol. 108(ST12), p. 2747–2765, 1982, .
- [28] A. Azizinamini, R. Pavel, E. Hatfield and S. K. Gosh, "Behavior of lap-spliced reinforcing bars embedded in high strength concrete," *ACI Structural Journal*, vol. 96, no. 5, p. 826–836, 1999, doi:10.14359/737 .
- [29] M. R. Esfahani and M. R. Kianoush, "Development/splice length of reinforcing bars," *ACI Structural Journal*, vol. 102, no. 1, p. 22–30, 2005, doi:10.14359/13527.
- [30] A. Perrot, D. Rangeard, F. Menasria and S. Guihéneuf, "Strategies for optimizing the mechanical strengths of raw earth-based mortars," *Constr. Build. Mater.*, vol. 167, p. 496–504, 2018, doi:10.1016/j.conbuildmat.2018.02.055.
- [31] J. Claramunt, H. Ventura, L. Fernández-Carrasco and M. Ardanuy, "Tensile and flexural properties of cement composites reinforced with flax nonwoven fabrics," *Materials*, vol. 10, no. 2, p. 215, 2017, 215. doi:10.3390/ma10020215.
- [32] R. H. Myers, D. C. Montgomery and C. M. Anderson-Cook, *Response surface methodology: process and product optimization using designed experiments*, 4th ed., New York: Wiley, 2016, p. 856 .
- [33] L. Eriksson, E. Johansson, N. Kettaneh-Wold, C. Wikström and S. Wold, *Design of Experiments: Principles and Applications*, Learnways AB, Umeå, 2000, pp. 425, ISBN 91-973730-4-4.
- [34] B. Abbar, A. Alem, S. Marcotte, A. Pantet, N. D. Ahfir, L. Bizet and D. Duriatti, "Experimental investigation on removal of heavy metals (Cu 2+ , Pb 2+ , and Zn 2+) from aqueous solution by flax fibres," *Process Safety and Environmental Protection*, vol. 109, p. 639–647, 2017, <http://dx.doi.org/10.1016/j.psep.2017.05.012>.
- [35] M. Rakhshanimehr, M. R. Esfahani, M. R. Kianoush, B. A. Mohammadzadeh and S. R. Mousavi, "Flexural ductility of reinforced concrete beams with lap-spliced bars," *Can. J. Civ. Eng.*, vol. 41, p. 594–604, 2014, doi:10.1139/cjce-2013-0074.
- [36] Standard AFNOR: NF P94-056, *Sols: reconnaissance et essais-Analyse granulométrique-Méthode par tamisage à sec après lavage*, 1996.
- [37] Standard AFNOR: XP P94-041, *Sols: reconnaissance et essais-Identification granulométrique-Méthode de tamisage par voie humide*, 1995.
- [38] Standard AFNOR: NF P94-057, *Sols: reconnaissance et essais-Analyse granulométrique des sols-Méthode par sédimentation*, 1992.
- [39] ASTM D2487-11: *Standard classification of soils for engineering purposes (Unified Soil Classification System)*, 2012.
- [40] Standard AFNOR: NF EN 459-1, *Chaux de construction - Partie 1: définitions, spécifications et critères de conformité*, 2012.

- [41] Standard AFNOR: NF EN197-1, Cement Part 1: composition, specifications, and conformity criteria for common cements, 2001.
- [42] Standard AFNOR: NF P 15-318, liants hydrauliques ciment à faible chaleur d'hydratation initiale et à teneur en sulfures limitée, 1991.
- [43] Standard AFNOR: NF EN196-10, Méthodes d'essais des ciments-Partie 10: Détermination de la teneur en chrome (VI) soluble dans l'eau des ciments, 2015.
- [44] R. Girault, F. Bert, C. Rihouey, A. Jauneau, C. Morvan and M. Jarvis, "Galactans and cellulose in flax fibres : putative contributions to tensile strength," *Int J Bio Macromol*, vol. 21, pp. 179-188, 1997.
- [45] A. Laborel-Préneron, J. E. Aubert, C. Magniont, C. Tribout and A. Bertron, "Plant aggregates and fibers in earth construction: A review," *Constr. Build. Mater.*, vol. 111, p. 719–734, 2016, doi:10.1016/j.conbuildmat.2016.02.119.
- [46] N. H. Dhakal, Z. Y. Zhang and M. O. Richardson, "Effect of water absorption on the mechanical properties of hemp fibre reinforced unsaturated polyester composites," *Compos. Sci. Technol.*, vol. 67, p. 1674–1683, 2007, doi:10.1016/j.compscitech.2006.06.019.
- [47] B. Abbar, A. Alem, A. Pantet, S. Marcotte, N. D. Ahfir and D. Duriatti, "Experimental investigation on removal of suspended particles from water using flax fibre geotextiles," *Environmental Technology, Taylor & Francis Group*, pp. 1-15, 2017, <http://dx.doi.org/10.1080/09593330.2017.1284270>.
- [48] Standard AFNOR: NF EN 934-2, Adjuvants pour béton, mortier et coulis - Partie 2: adjuvants pour bétons. Définitions et exigences, 1998.
- [49] A. Pierre, R. Mercier, A. Foissy and J. M. Lamarche, "The adsorption of cement superplasticizers on to mineral dispersions," *Adsorption Science & Technology*, vol. 6, no. 4, pp. 219-231, 1989.
- [50] P. C. Aitcin, C. Jolicoeur and J. G. MacGregor, "Superplasticizers: how they work and why they occasionally don't," *Concrete International*, vol. 16, no. 5, p. 45–52, 1994.
- [51] Standard AFNOR: NF P94-420, Détermination de la résistance à la compression uniaxiale, 2000.
- [52] Standard AFNOR: NF P94-425, Méthodes d'essai pour roches - Détermination du module d'Young et du coefficient de Poisson, 2002.
- [53] A. P. Fantilli, B. Chiaia and A. Gorino, "Fiber volume fraction and ductility index of concrete beams," *Cem. Concr. Compos.*, vol. 65, p. 139–149, 2016, doi:10.1016/j.cemconcomp.2015.10.019.
- [54] M. H. Maher and Y. C. Ho, "Mechanical properties of Kaolinite/fiber soil composite," *J. Geotech. Eng.*, vol. 120, no. 8, p. 1381–93, 1994, [https://doi.org/10.1061/\(ASCE\)0733-9410\(1994\)120:8\(1381\)](https://doi.org/10.1061/(ASCE)0733-9410(1994)120:8(1381)).
- [55] A. B. Groeneveld, T. M. Ahlborn, C. K. Crane, C. A. Burchfiel and E. N. Landis, "Dynamic strength and ductility of ultra-high performance concrete with flow-induced fiber alignment,"

International Journal of Impact Engineering, vol. 111, pp. 37-45, 2017,
<http://dx.doi.org/10.1016/j.ijimpeng.2017.08.009>.

- [56] N. C. Consoli, J. P. Montardo, M. Donato and P. D. Prietto, "Effect of material properties on the behaviour of sand–cement–fibre composites," *Ground Improv*, vol. 8, no. 2, p. 77–90, 2004, <https://doi.org/10.1680/grim.2004.8.2.77>.
- [57] C. K. Ma, A. Z. Awang and W. Omar, "Flexural ductility design of confined high-strength concrete columns: Theoretical modelling," *Measurement*, vol. 78, p. 42–48, 2016, <http://dx.doi.org/10.1016/j.measurement.2015.09.039>.
- [58] W. C. Panarese, S. H. Kosmatka and F. A. Randall, *Concrete masonry handbook for architects, engineers, builders*, 5th ed ed., Skokie, Ill. : Portland Cement Association, Libraries Australia, 1991.
- [59] Q. B. Bui, J. C. Morel, S. Hans and P. Walker, "Effect of moisture content on the mechanical characteristics of rammed earth," *Constr. Build. Mater.*, vol. 54, p. 163–169, 2014, doi:10.1016/j.conbuildmat.2013.12.067.
- [60] Standard AFNOR: NF EN 206-1/CN, Béton–Partie 1: Spécification, performances, production et conformité (in French), 2012.
- [61] S. Chafei, F. Khadraoui, M. Boutouil and M. Gomina, "Optimizing the formulation of flax fiber-reinforced cement composites," *Constr. Build. Mater.*, vol. 54, p. 659–664, 2014, doi:10.1016/j.conbuildmat.2013.12.038.
- [62] P. F. De Aguiar, B. Bourguignon, M. S. Khots, D. L. Massart and R. Phan-Thau-Luu, "D-optimal designs," *Chemom. Intell. Lab. Syst.*, vol. 30, p. 199–210, 1995, doi:10.1016/0169-7439(94)00076-X.
- [63] P. Krajnik, J. Kopac and A. Sluga, "Design of grinding factors based on response surface methodology," *J. Mater. Process. Technol.*, vol. 162–163, p. 629–636, 2005, doi:10.1016/j.jmatprotec.2005.02.187.
- [64] G. E. Box, J. Stuart Hunter and W. G. Hunter, *Statistics for experimenters: design, innovation, and discovery*, John Wiley and sons, 2005, ISBN-13: 978-0471718130 , p. 629.
- [65] Software for design of experiments and optimization, Umetrics: <http://www.umetrics.com>, 2016.
- [66] B. Li, A. J. Morris and E. B. Martin, "Generalized partial least squares regression based on the penalized minimum norm projection," *Chemom. Intell. Lab. Syst.*, vol. 72, p. 21–26, 2004, doi:10.1016/j.chemolab.2004.01.026.
- [67] S. Bouzalakos, A. W. Dudeney and B. K. Chan, "Formulating and optimising the compressive strength of controlled low-strength materials containing mine tailings by mixture design and response surface methods," *Miner. Eng.*, vol. 53, p. 48–56, 2013, doi:10.1016/j.mineng.2013.07.007.
- [68] J. Goupy, *Plans d'expériences : les mélanges*, Paris: DUNOD, 2001, p. 285.

- [69] R. A. Fisher, *The design of experiments*, New York, Hafner: Libraries Australia, 1971, p. 248.
- [70] J. Biarez and P. Y. Hicher, *Elementary mechanics of soil behaviour: saturated remoulded soils*, A.A. Balkema, Amsterdam, Netherlands, 1994, ISBN 90-5410-156-3 (rel.). - ISBN 90-5410-157-1 (br.).
- [71] Y. Brouard, N. Belayachi, D. Hoxha, N. Ranganathan and S. Méo, "Mechanical and hygrothermal behavior of clay – Sunflower (*Helianthus annuus*) and rape straw (*Brassica napus*) plaster bio-composites for building insulation," *Constr. Build. Mater.*, vol. 161, p. 196–207, 2018, doi:10.1016/j.conbuildmat.2017.11.140.
- [72] A. Beukers, *Lightness. The inevitable renaissance of minimum energy structure*, Rotterdam: v. Hinte, 1998.
- [73] R. A. Fisher, *Statistical methods, experimental design, and scientific inference: a re-issue of statistical methods for research workers, the design of experiments, and statistical methods and scientific inference*, New York: Hafner publishing company, 1995, p. 323.
- [74] D. Gallipoli, A. W. Bruno, C. Perlot and J. Mendes, "A geotechnical perspective of raw earth building," *Acta Geotech.*, vol. 12, p. 463–478, 2017, doi:10.1007/s11440-016-0521-1.

593

594

595 **Figure captions**

596 Fig.1: Silt grading size distribution curve

597 Fig.2: Vegetal flax fibers

598 Fig. 3: a) UCS stress-strain curve for formulation F9 after 90 days of curing time with the
599 identification of four zones, b) Ductility indices definitions ($i = \frac{\epsilon_3}{\epsilon_1}$, $i' = \frac{\epsilon_3}{\epsilon_2}$)

600 Fig. 4: Projection in the (X_{cement} , X_{water}) plane of the experimental region validating the
601 workability condition (dashed regions) for mixtures without fibers (Eq. 4, [4]) and with fibers
602 (Eq. 3). X_{cement} and X_{water} are respectively the proportions of cement and water. The proportion
603 of lime is fixed to $X_{\text{lime}} = 0.02$. Dashed lines: Isovalues of X_{silt} proportion.

604 Fig. 5: Unconfined Compressive Stress (UCS) as a function of the axial strain for formulation F2

605 Fig. 6: Ductility indices versus formulation number

606 Fig. 7: Response trace plots of ductility index i' a) quadratic model, b) linear model

607 Fig. 8: Normalized stress-strain curve for the two formulations F1 and F22

608 Fig. 9: Normalized stress-strain curve for formulations almost similar except by the fiber
609 content

610 Fig. 10: Ductility indices as a function of the silt/binders ratio

611 **Table captions:**

612 Table 1: Mechanical and physical properties of vegetal flax fibers ([47], [72])

613 Table 2: Mixing range

614 Table 3: Experimental design of the D-optimal design for a raw earth concrete

615 Table 4: The coefficients of the linear and quadratic models for i and i' ductility indices for 90
616 days of curing time

617 Table 5: Values of R^2 , R^2_{adj} and Q^2

618 Table 6: ANOVA Table for i' ductility index

619 Table 7: First F-Test (model significance test: (F-value)_{critical} < (F-value)_{model})

620 Table 8: Second F-Test ((F-value)_{critical} > (F-value)_{LOF})

621 Table 9: Deduced values of i' and $\epsilon_1/\epsilon_{max}$ from Fig. 9

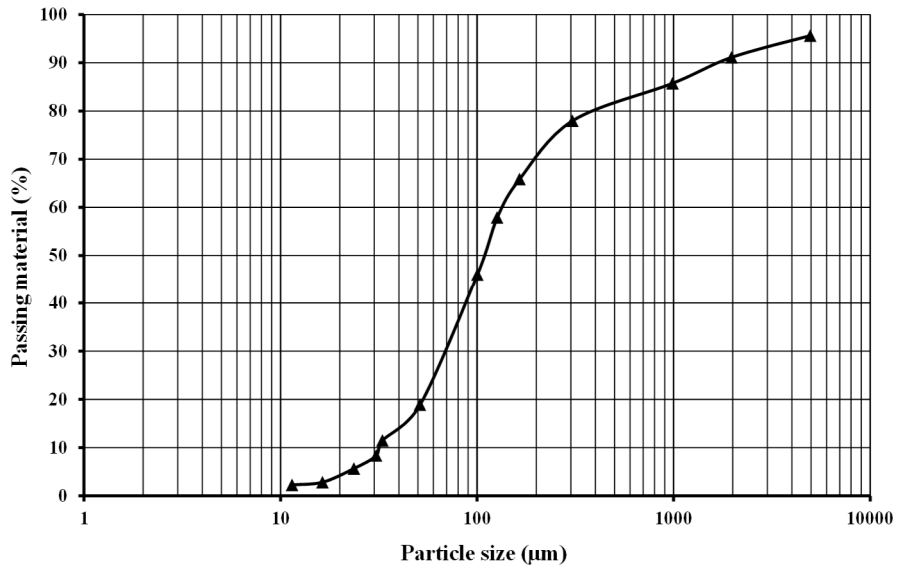
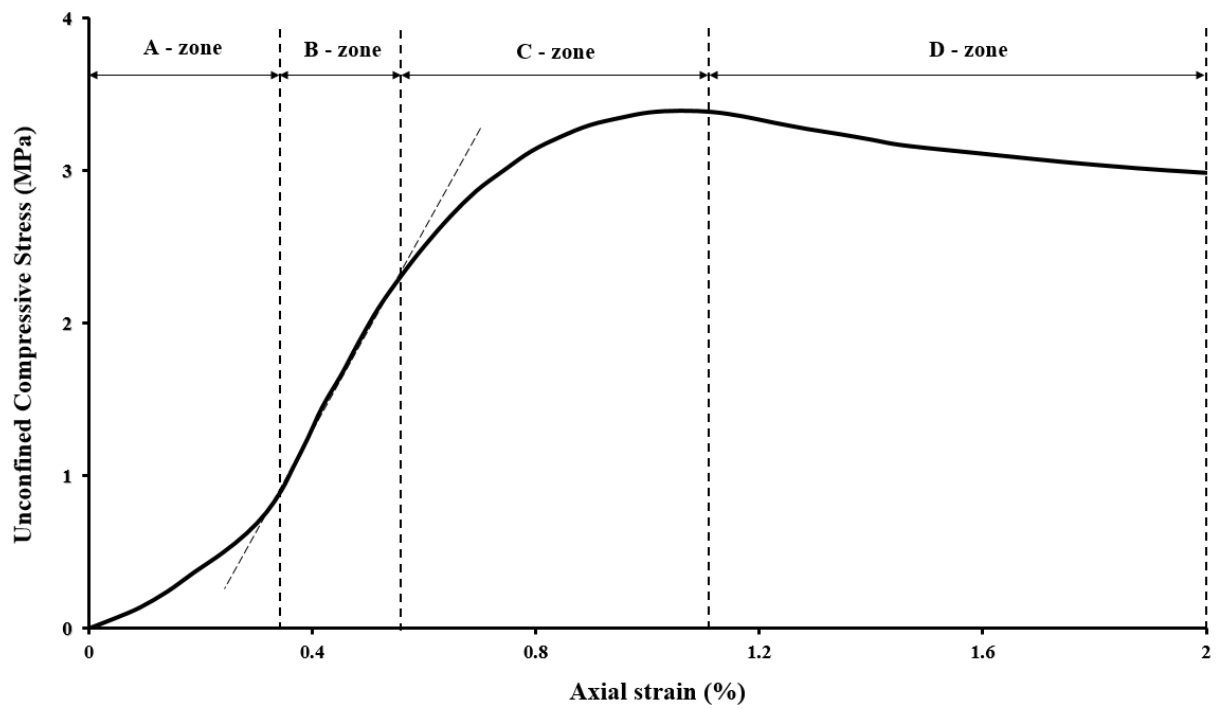


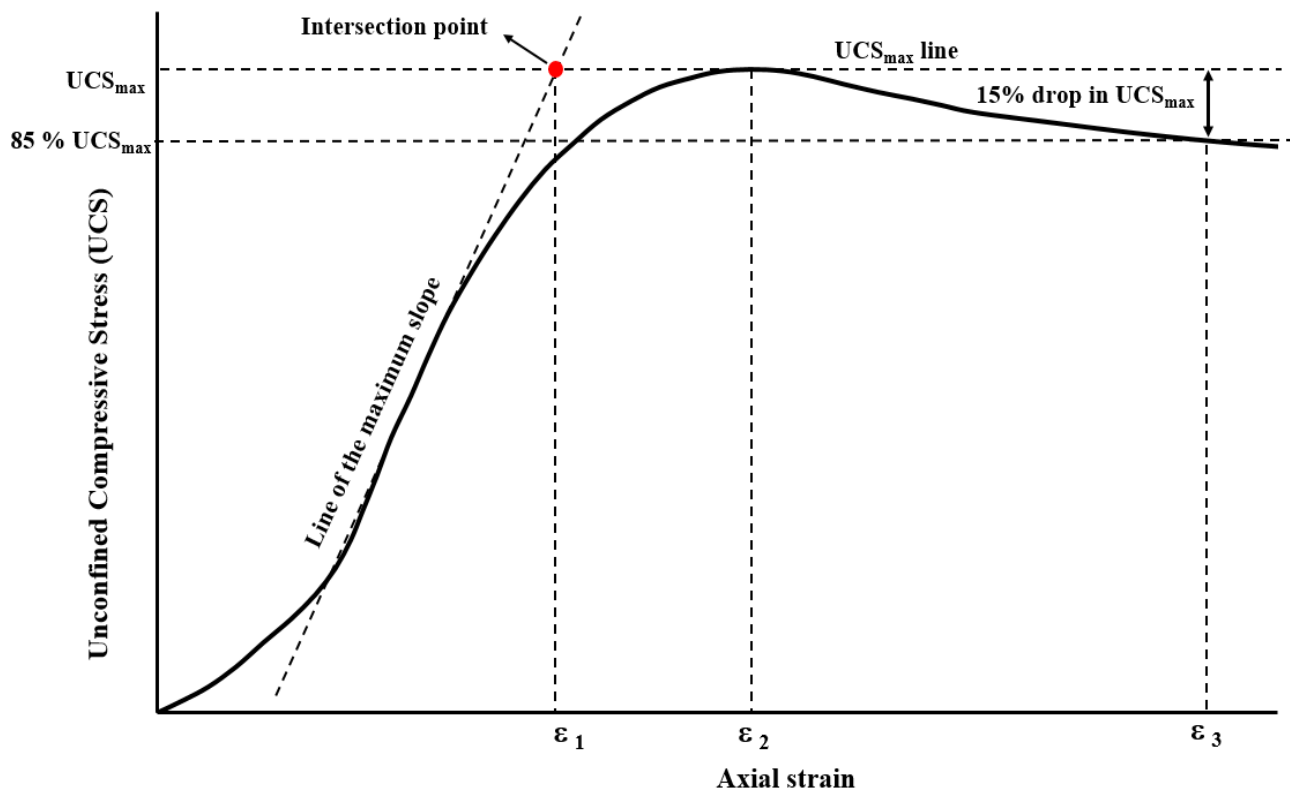
Fig.1: Silt grading size distribution curve



Fig.2: Vegetal flax fibers



a)



b)

Fig. 3: a) UCS stress-strain curve for formulation F9 after 90-days of curing time with the identification of four zones, b) Ductility indices definitions ($i = \frac{\epsilon_3}{\epsilon_1}$, $i' = \frac{\epsilon_3}{\epsilon_2}$)

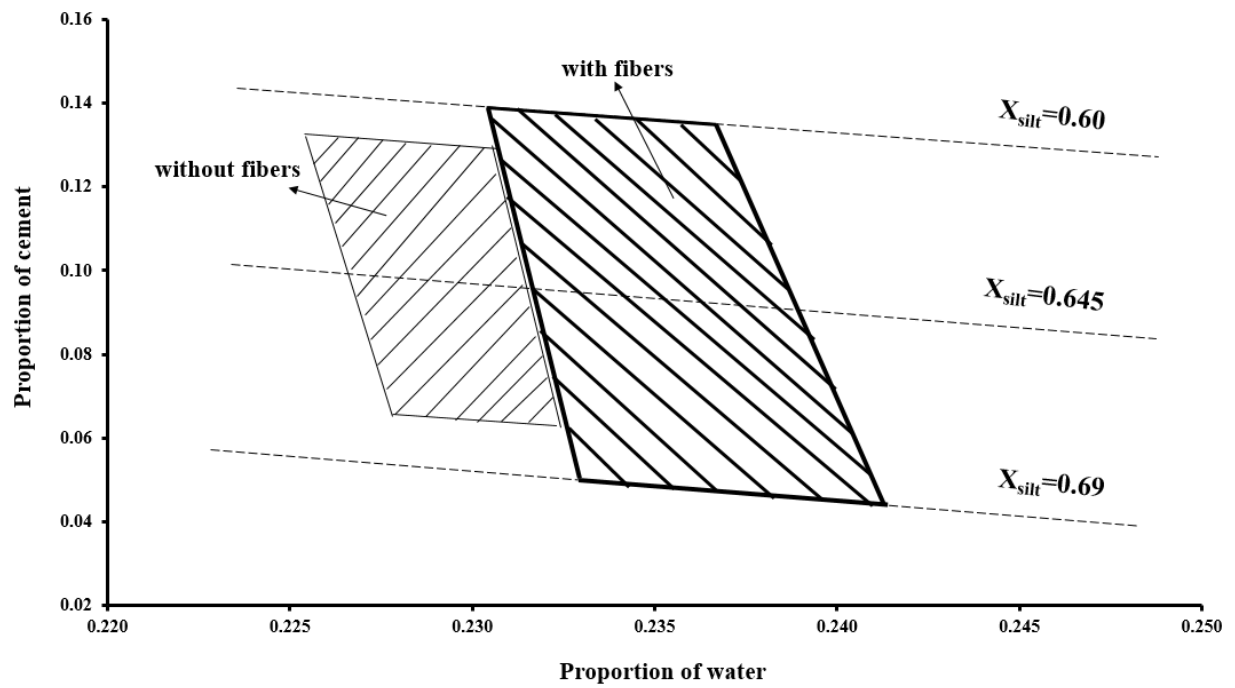


Fig. 4: Projection in the $(X_{\text{cement}}, X_{\text{water}})$ plane of the experimental region validating the workability condition (dashed regions) for mixtures without fibers (Eq. 4, [4]) and with fibers (Eq. 3). X_{cement} and X_{water} are respectively the proportions of cement and water. The proportion of lime is fixed to $X_{\text{lime}}=0.02$. Dashed lines: Isovalues of X_{silt} proportion.

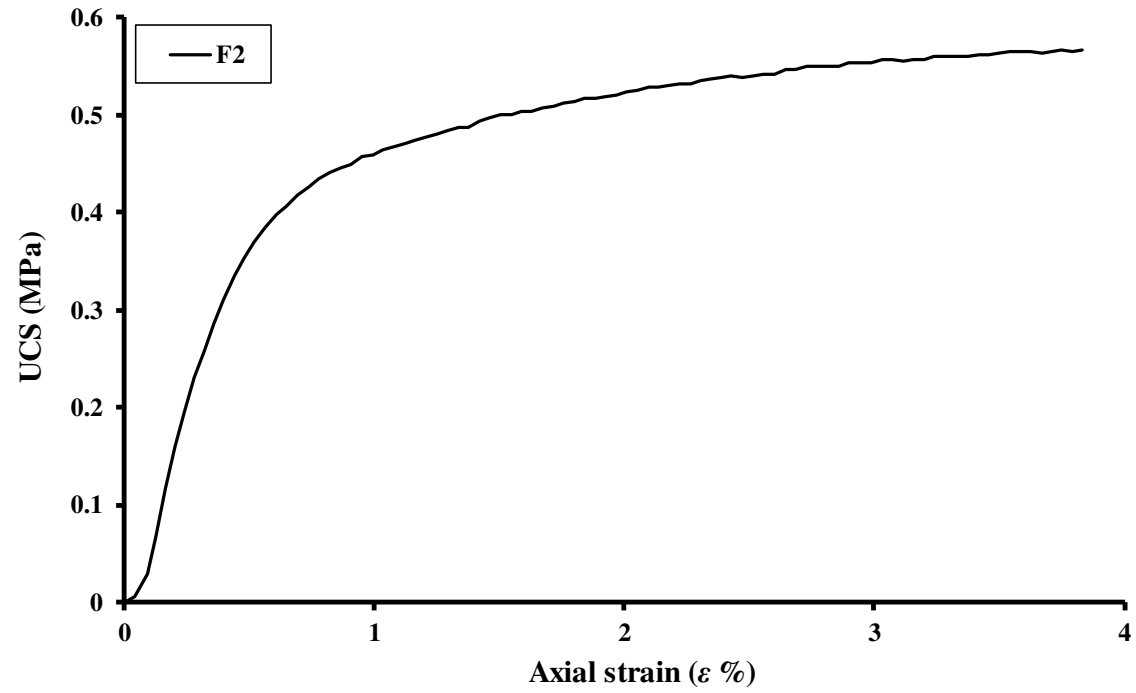


Fig. 5: Unconfined Compressive Stress (UCS) as a function of the axial strain for formulation F2

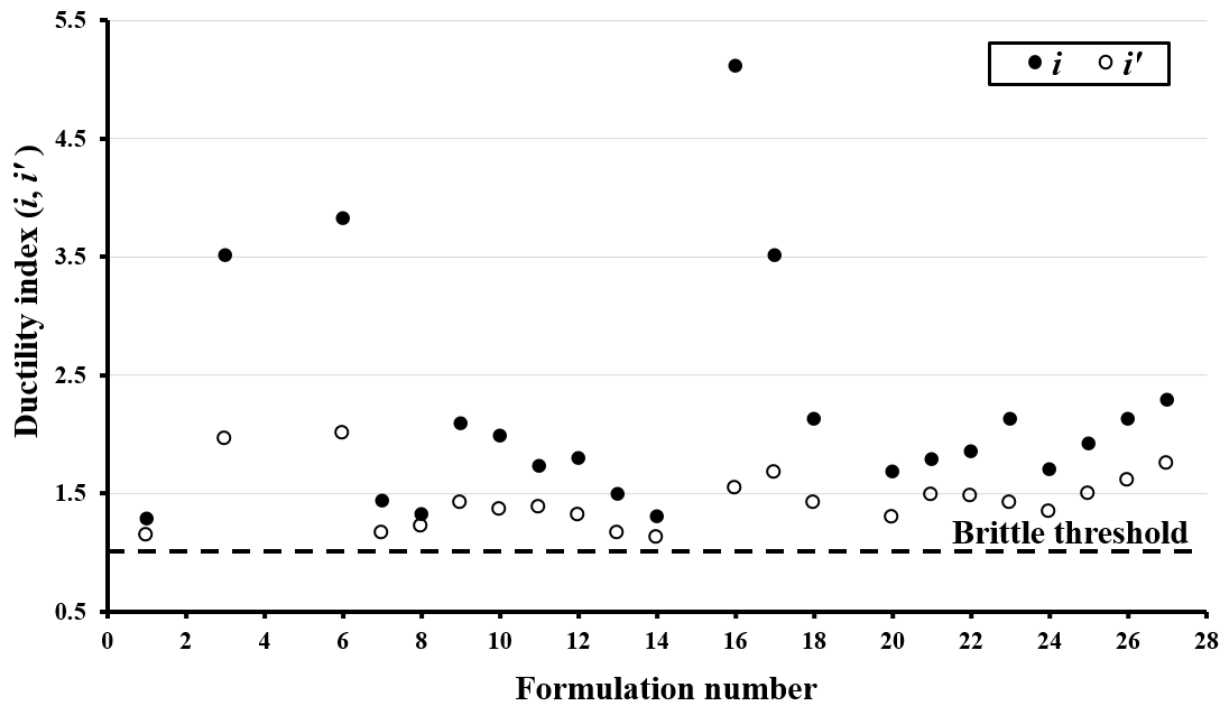
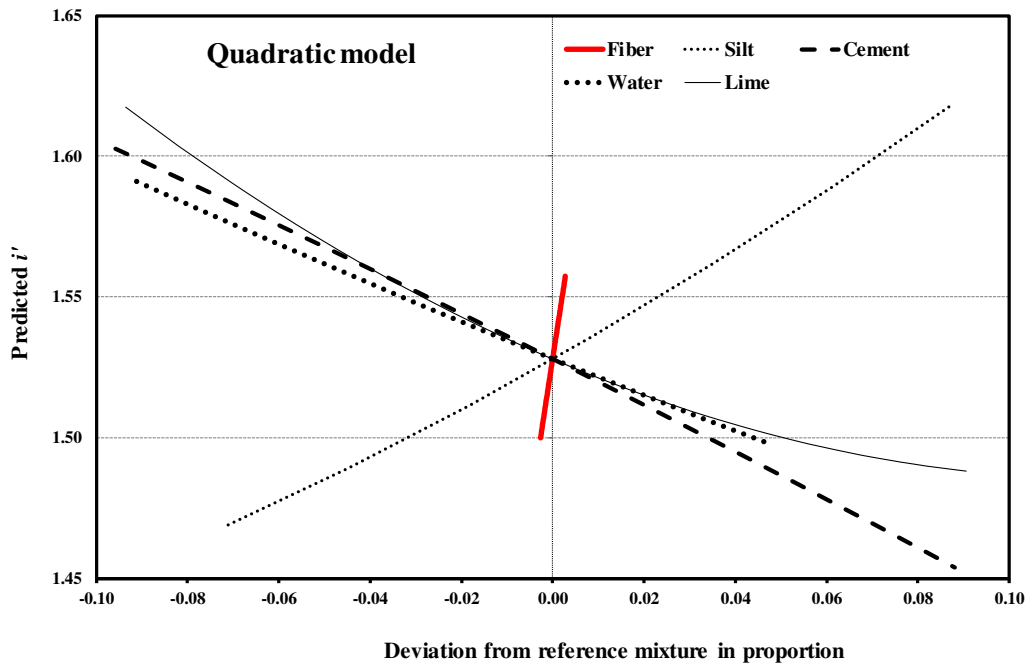
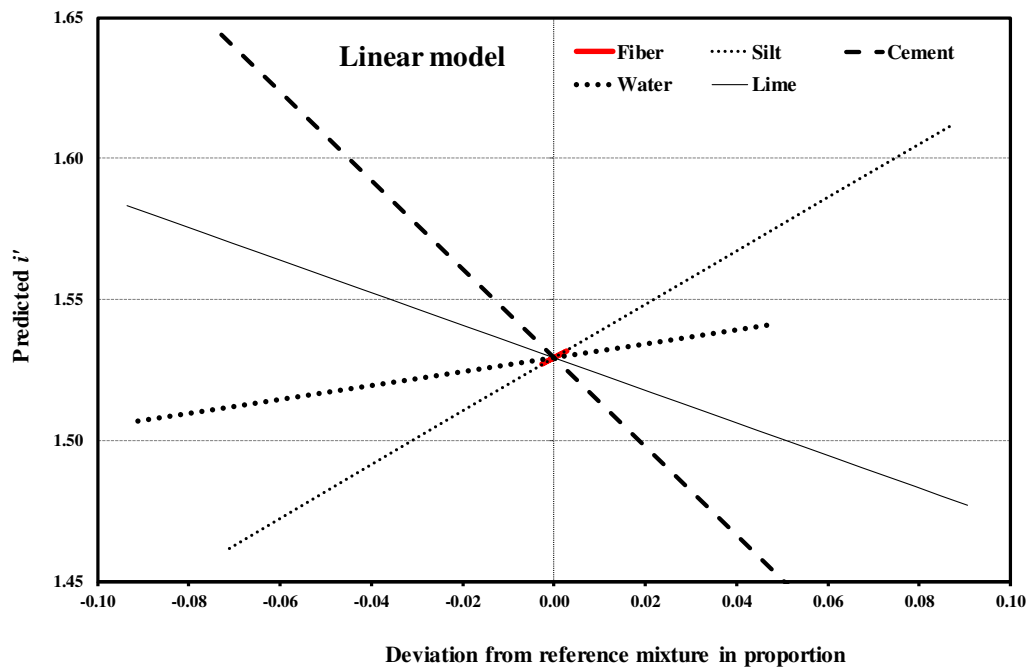


Fig. 6: Ductility indices versus formulation number



a)



b)

Fig. 7: Response trace plots of ductility index i' a) quadratic model, b) linear model

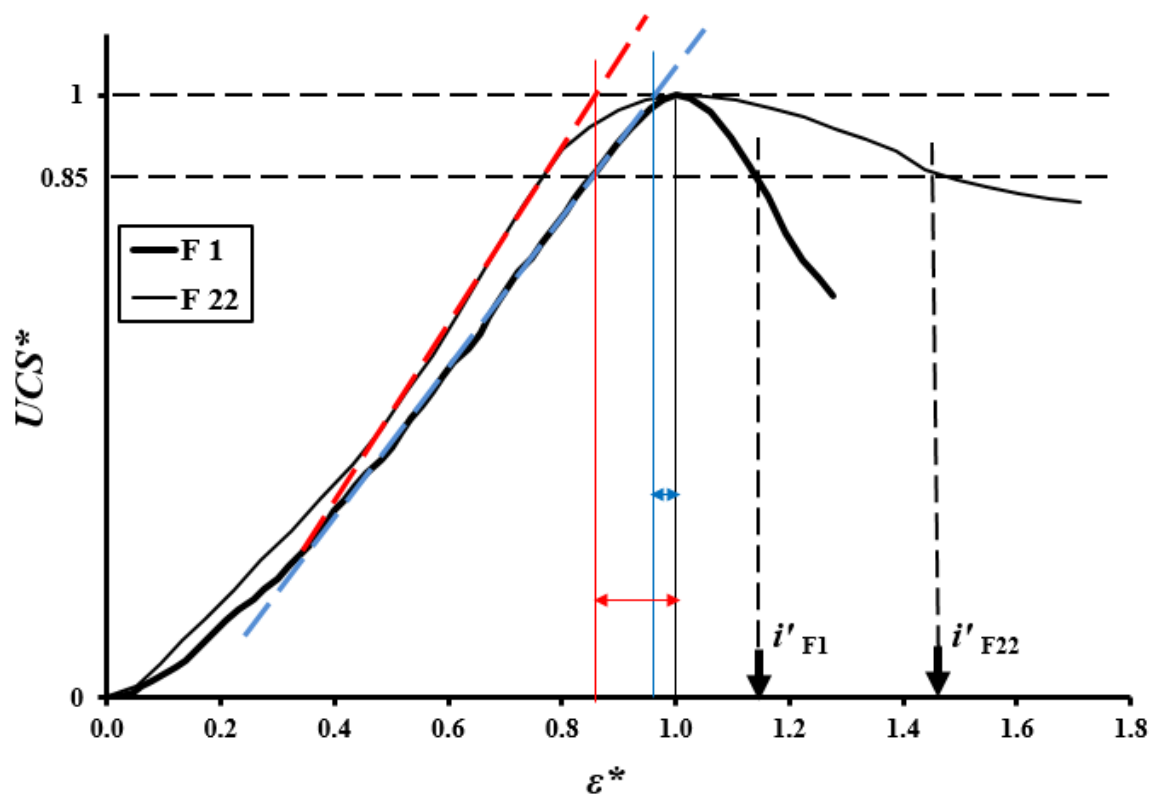
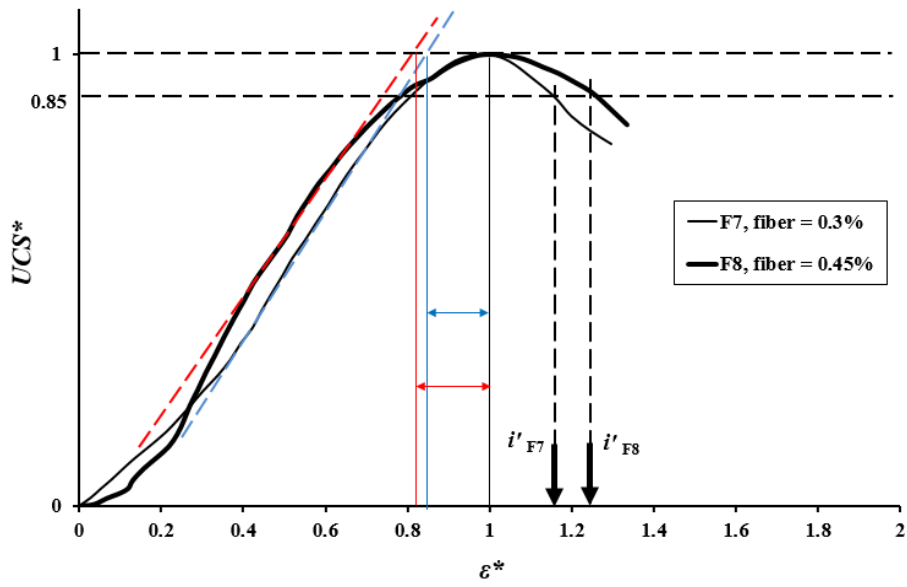
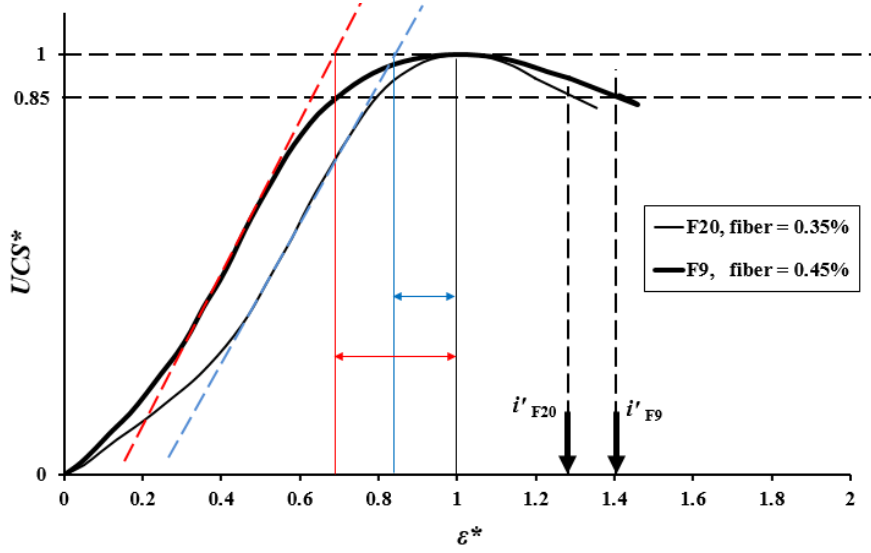


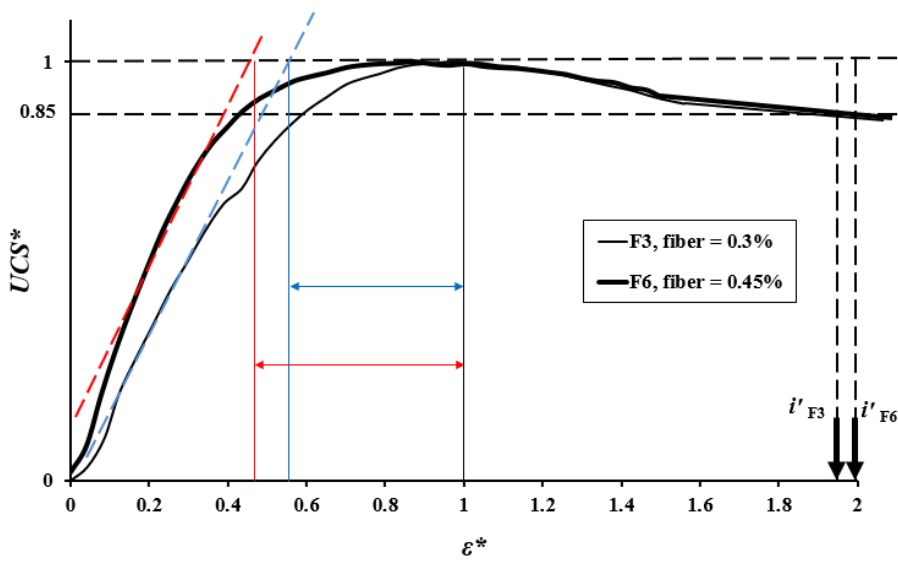
Fig. 8: Normalized stress-strain curve for the two formulations F1 and F22



a)



b)



c)

Fig. 9: Normalized stress-strain curve for formulations almost similar except by the fiber content

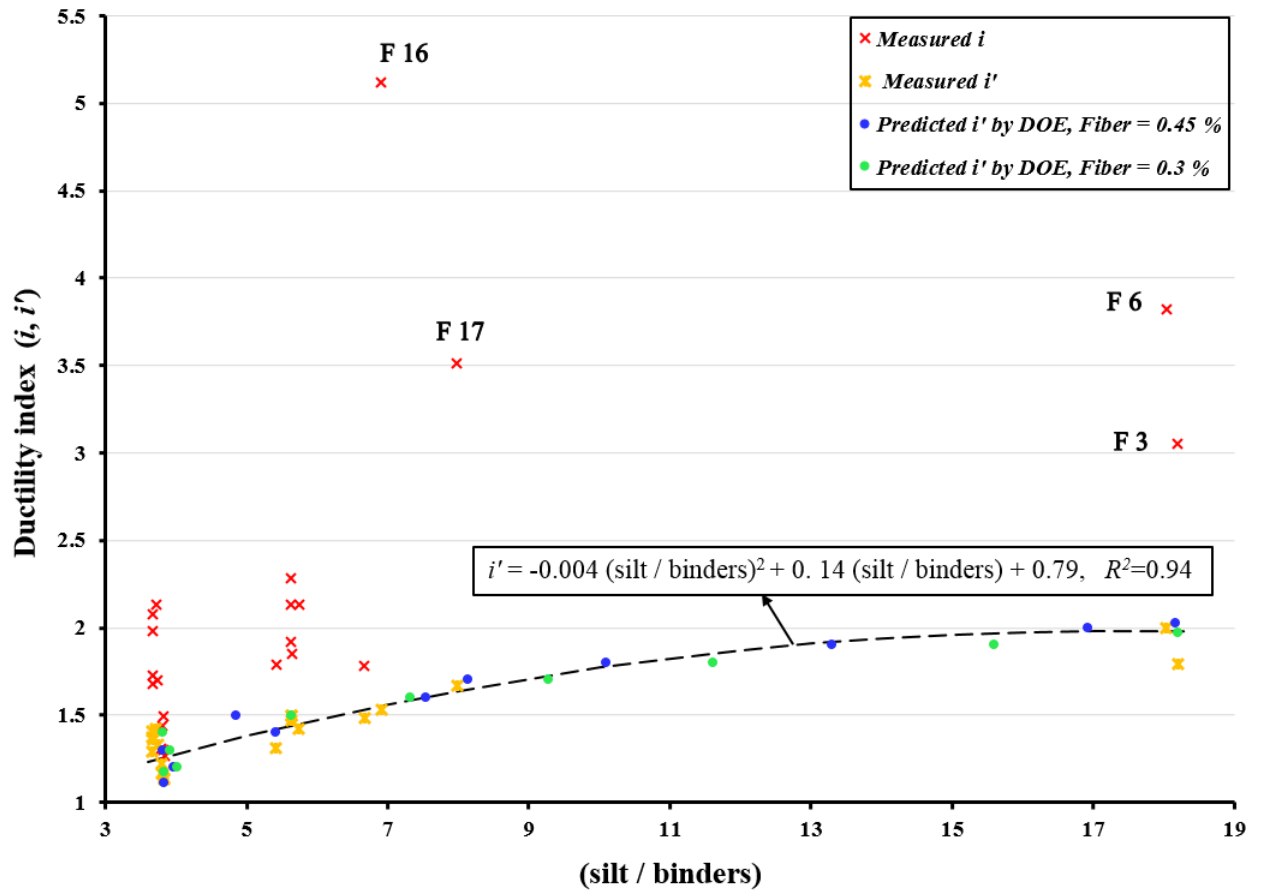


Fig. 10: Ductility indices as a function of the silt/binders ratio

Table 1: Mechanical and physical properties of vegetal flax fibers ([47], [72])

Parameters	
Density $\rho_{\text{fiber}}/\rho_{\text{water}}$	1.4
Fiber diameter d (μm)	10-15
Young's modulus E (GPa)	40-85
Failure stress σ_u (MPa)	800-2000
Failure strain ε_u (%)	2.4-3.3

Table 2: Mixing range

x_i	Lower limit (%)	Upper limit (%)
x_1 : Fiber	0.3	0.45
x_2 : Lime	0	12
x_3 : Cement	4	16
x_4 : Water	20	25
x_5 : Silt	47	75

Table 3: Experimental design of the D-optimal design for a raw earth concrete

Formulation	Fiber (x_1)	Lime (x_2)	Cement (x_3)	Water (x_4)	Silt (x_5)	Ductility index i	Ductility index i'
1	0.0030	0.0000	0.1600	0.2239	0.6131	1.27	1.14
2	0.0030	0.0647	0.0400	0.2500	0.6423	-	-
3	0.0030	0.0000	0.0400	0.2290	0.7280	3.51	1.96
4	0.0045	0.0000	0.0400	0.2287	0.7268	-	-
5	0.0030	0.0000	0.0400	0.2345	0.7225	-	-
6	0.0045	0.0000	0.0400	0.2342	0.7213	3.82	2.00
7	0.0030	0.0000	0.1600	0.2295	0.6075	1.44	1.17
8	0.0045	0.0000	0.1600	0.2292	0.6063	1.31	1.22
9	0.0045	0.0719	0.0881	0.2500	0.5855	2.08	1.41
10	0.0030	0.0560	0.1040	0.2500	0.5870	1.98	1.36
11	0.0045	0.0568	0.1032	0.2500	0.5855	1.73	1.37
12	0.0045	0.0000	0.1200	0.2253	0.6502	1.79	1.31
13	0.0045	0.0000	0.1600	0.2255	0.6100	1.49	1.16
14	0.0040	0.0000	0.1600	0.2237	0.6123	1.30	1.13
15	0.0030	0.0237	0.1363	0.2326	0.6044	-	-
16	0.0045	0.0543	0.0400	0.2500	0.6512	5.12	1.53
17	0.0045	0.0438	0.0400	0.2429	0.6688	3.51	1.67
18	0.0045	0.0479	0.1121	0.2412	0.5943	2.13	1.42
19	0.0035	0.0480	0.0400	0.2500	0.6585	-	-
20	0.0035	0.0713	0.0887	0.2500	0.5865	1.68	1.29
21	0.0038	0.0000	0.1000	0.2291	0.6672	1.78	1.48
22	0.0030	0.0299	0.0841	0.2396	0.6433	1.85	1.47
23	0.0038	0.0261	0.0859	0.2409	0.6433	2.13	1.42
24	0.0038	0.0320	0.1280	0.2383	0.5980	1.70	1.33
25	0.0038	0.0301	0.0840	0.2395	0.6426	1.92	1.50
26	0.0038	0.0301	0.0840	0.2395	0.6426	2.13	1.60
27	0.0038	0.0301	0.0840	0.2395	0.6426	2.28	1.50

Table 4: The coefficients of the linear and quadratic models for i and i' ductility indices for 90-days of curing time

Coefficients	Quadratic model, i	Quadratic model, i'	Linear model, i	Linear model, i'
a_0	35.00	0.41	2.48	-0.71
a_1	-5321.80	-504.08	254.05	4.60
a_2	-2.71	54.77	-7.95	-0.14
a_3	151.13	-5.74	-20.78	-2.98
a_4	-174.68	-6.21	4.52	2.39
a_5	-43.99	5.38	-0.08	2.97
a_{12}	4507.81	321.78	-	-
a_{13}	-5379.10	-369.63	-	-
a_{14}	17838.80	1900.83	-	-
a_{15}	2862.35	168.07	-	-
a_{23}	-2.69	42.04	-	-
a_{24}	-87.11	-218.54	-	-
a_{25}	15.34	-13.30	-	-
a_{34}	-198.25	92.14	-	-
a_{35}	-146.26	-27.75	-	-
a_{45}	228.84	-5.10	-	-

Table 5: Values of R^2 , R^2_{adj} and Q^2

Ductility index	Linear model	Quadratic model
i	$R^2=0.74, R^2_{adj}=0.68, Q^2=0.60$	$R^2=0.84, R^2_{adj}=0.43, Q^2=0.35$
i'	$R^2=0.86, R^2_{adj}=0.81, Q^2=0.77$	$R^2=0.92, R^2_{adj}=0.73, Q^2=0.84$

Table 6: ANOVA Table for i' ductility index

Source of variation	Degrees of Freedom (DF)	Sum of Squares (SS)	Mean Square (MS)	F-value	P-value
<i>Quadratic model</i>					
Regression	15	1.131	0.075	4.688	0.032
Residual error	6	0.094	0.016		
Lack-of-fit	4	0.063	0.016	0.99	0.558
Pure error	2	0.032	0.016		
Total	21	1.225	0.058		

Table 7: First F-Test (model significance test: $(F\text{-value})_{\text{critical}} < (F\text{-value})_{\text{model}}$)

Model	F-value (critical)	F-value (model)
<i>Quadratic model for i' ductility index</i>	3.940	4.688

Table 8: Second F-Test ((F-value)_{critical} > (F-value)_{LOF})

Model	F-value (critical)	F-value (LOF)
<i>Quadratic model for i' ductility index</i>	19.2	0.99

Table 9: Deduced values of i' and $\varepsilon_1/\varepsilon_{max}$ from Fig. 9

Fig. 9	0.35% fibers		0.45% fibers	
	i'	$\varepsilon_1/\varepsilon_{max}$	i'	$\varepsilon_1/\varepsilon_{max}$
Fig. 9 a	1.17	0.83	1.22	0.82
Fig. 9 b	1.29	0.82	1.41	0.69
Fig. 9 c	1.96	0.55	2	0.49