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Variability of the mechanical properties of hemp concrete

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32 1 Abstract

- 33 The focus of this study is on statistical analysis of hemp concrete properties. The main objective
- 34 is to determine statistically the variability of the three main properties, which are: material
- 35 density, compressive strength and Young's modulus. The analysis is done with respect to four
- 36 main parameters, namely: the testing laboratory equipment and procedure, the hemp shiv type,
- 37 the batch elaboration and finally the specimen size

38 Two types of hemp shiv have been used with two batches for each type. Two cylindrical 39 specimen sizes have been considered: 11x22 cm and 16x32 cm. All the specimens were 40 manufactured and dried in the same laboratory in order to ensure the repeatability and 41 homogeneity of studied material. After 90 days of drying under the same conditions, the 42 specimens were transported to ten different laboratories for compressive testing. Before testing, a 43 drying protocol during 48 hours was applied by all laboratories for all specimens. Then, a unique 44 protocol for compressive testing has been applied using the compressive testing machine in each 45 laboratory. Finally, all data have been collected for statistical analysis. In this study, the results 46 obtained by different laboratories show low variability for compressive strength and dry density; 47 which is not the case for Young's modulus. Three probability distributions, namely: normal, log-48 normal and Weibull, have been proposed to fit the experimental results.

49 2 Introduction

The use of plant origin aggregates is nowadays considered as an essential way in manufacturing environmentally friendly building materials. Many aggregates of this kind exist and are used in the construction industry, either in new structures or renovation of existing buildings, for example, aggregates of sunflower, hemp shiv...[1]–[3]. In contrast to aggreagtes of mineral origin, plant origin aggregates are renewable and carbon neutral materials. They also have other advantages such as good thermal and acoustic insulation properties. However, the major drawback is related to their low mechanical performance [4]–[6].

For more than one decade, the researches on these materials have not ceased to increase. A very recent study was conducted by Binici et al. [7] on the use of sunflower and waste cotton textiles for manufacturing insulation. Other researches have been also conducted on the use of the hemp shiv in insulation [8]. In the framework of the present study, the herein literature review focuses on concrete made from hemp shiv, and particularly on its mechanical behavior. 62 Several parameters influence the mechanical properties of hemp concrete. They include among 63 others, the nature of its constituents such as the aggregate size, the type of binders, and the 64 manufacturing method, such as the compaction energy and the molding method [9], [10].

The density of hemp concrete is related to quality and quantity of constituents, the aggregate size, their porosity and the energy of compaction. Considering all these parameters, different and variable density values are found in the literature. In a study conducted by Cerezo [9], several formulations were tested and specimens between 12 and 29 were manufactured for each formulation. The density distribution of each formulation was homogenous with a coefficient of variation between 1.5% and 3.5%.

For ten different formulations, Cerezo obtained the final average density values ranging from 256 kg/m³ to 782 kg/m³. Although she considers in her analysis that the series have a low dispersion, this is not true at all levels. This is only valid at the intra-formula level, but not for the inter-formula level, because in this latter case, considerable dispersion is observed for both final and initial mean values; which vary in the range of 455 kg/m³ to 1140 kg/m³.

76 In parallel, F. Collet [11] has determined the density of two kinds of hemp concrete (batch A for 77 one hemp and B for another) by using three different methods: weighing and dimension 78 measurement, pycnometer and mercury porosimeter. The first step of its study is to determine 79 the representative elementary volume. The density variation obtained between the samples of 80 5 cm and 20 cm edges was of approximately 4%. Thanks to this low variation in the average 81 density between samples, she concluded that samples of 5 cm edge are representative of hemp 82 concrete. However, the differences in results were observed with respect to the used 83 measurement method. For the pycnometer test, the density for batch A is 390 kg/m³ and 425 kg/m³ for batch B. The test of mercury porosimeter gave a dry density of 609 kg/m³ and 84 664 kg/m³ for batches A and B, respectively. With the method of weighing and measuring 85 dimensions for two different series in batch A, she got 408 kg/m³ and 406 kg/m³ with 6.6% and 86 2.7% of coefficient of variation for the first and second series, respectively. Finally, in batch B, 87

the mean value density for sample of 5cm edge cube is 438 kg/m^3 with a standard deviation equal to 5.7%.

Another study has been conducted by Nguyen [10] on two types of hemp shives: the first with pure shiv particles (CP), while the other one contains fibres (CF). It is shown that there is no difference between the two shives in terms of density. For specimens tested under the same conditions, the observed difference was less than 2%. Results obtained were in the range of 450 kg/m³ to 800 kg/m³ at 90 days. This dispersion is mainly based on three main parameters of formulation and manufacturing process, namely the binder/aggregate ratio; the water/binder ratio and the compaction strength.

97

98 Nguyen [10] also highlighted parameters influencing the compressive strength. Because of the 99 low rigidity of particles, hemp concrete has a very ductile behavior in both compression and 100 tension. Based on test results, he obtained a compressive strength, for a strain equal to 7.5% after 101 28 days, which varies between 0.2 MPa and 3.6 MPa. On her side, Cerezo [9] obtained the 102 compressive strength ranging between 0.25 and 1.15 MPa. For low binder content, the 103 compressive strength is around 0.25 MPa. For intermediate dosage, it varies between 0.4 and 104 0.8 MPa and for high binder content, it is 1.15 MPa. She concluded that mechanically, hemp 105 concrete is characterized by an elastic-plastic behavior, and that this material must be used with a 106 support structure to meet structural requirements.

Other parameters may also influence the mechanical behavior of hemp concrete such as drying conditions, the age of hemp concrete and the size of hemp particles [12]. Taking into account these parameters, Arnaud and Gourlay [12] obtained compressive strength, which varies between 0.35 MPa and 0.85 MPa for the age of 21 days to 24 months. Increasing the energy of compaction during the manufacturing process may enhance the maximum compressive strength. However it has been proven that the compressive strength is limited to 3 MPa for a compaction pressure between 0.6 MPa and 1 MPa [13]. Nguyen [10] obtained a compressive strength beyond

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3.5 MPa at 28 days by using a compaction stress maintained during 48 hours before demouldingthe hemp concrete fresh paste.

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117 Young's modulus values found in the literature have also high variability and the methods used 118 for its calculation are also different. According to Cerezo [9], the Young's modulus is defined as 119 the slope at the origin of the strength-strain curve by considering the validity of the small strain 120 assumption. Young's modulus varies from 1 to 3 MPa for low binder content; 32 to 95 MPa for 121 intermediate dosages and 100 to 160 MPa for high dosage. For various formulations, 122 Nguyen [10] obtained, at 90 days, the Young's modulus between 25 MPa and 176 MPa; using 123 pure hemp particles. According to this study, the Young's modulus of a given specimen is 124 calculated based on the strongest increase in the ratio strength/strain recorded at the beginning of 125 the loading stage.

126 The results in the literature show that the values for properties of hemp concrete have a great 127 variability and are sensitive to many factors. The literature shows also that there is a lack on 128 consideration of the accuracy of testing instruments used and the variability of results due to 129 experimentations. For example on one hand, Mounanga et al. [14] studied the influence of the 130 composition and method of implementation on the development of mechanical properties of 131 hemp concrete. On the other hand an analysis of the variability on the self-compacting concrete 132 was led by Almeida Filho et al. [15]. In this last study, in order to reduce the impact of statistical 133 errors, they used results from 10 to 24 specimens for each type of formulation.

As other materials, the variability performance of hemp concrete has two origins: intrinsic variability of the studied material itself and uncertainty caused by insufficient information with respect to these mechanical performance [16]. It is of course fundamental, even though not necessarily easy, to distinguish between these two sources through appropriate statistical modeling. For this reason, a statistical study is required to assess the certainty and variability ofthe results for the mechanical properties of hemp concrete.

In the present study, a statistical analysis of the results taking into account two types of hemp shives, four types of batches, and two specimen sizes, is carried out in order to define the probability distributions fitting the experimental results. The considered characteristics are: density, maximum compressive strength and Young's modulus. The mechanical compression tests were conducted in ten different laboratories, which allow analyzing the impact of the laboratory on the estimation of material characteristics.

146 **3 Material and methods**

The specimens in this study, were manufactured using two hemp shives with the same binder, prompt natural cement (PNC) and citric acid. The characterization results for bulk density, water absorption and particle size distribution, are given for both shives in section 3.1. The protocols and methods related to manufacturing, mixing process and compressive testing are given in section 3.2. The compressive tests have been made using different machines under the same protocol, and the experimental results were collected for statistical analysis. The considered parameters during the mixing and manufacturing process are provided in Table 1.

Spe	ecimen sizes	Speci	men 11	cmX2	2cm	Speci	men 16	cmX3	2cm	Total per
Тур	Type of batch		II	III	IV	Ι	Π	III	IV	laboratoire
	Institut Pascal (A)		3		3		3			9
	Belfast (B)		2	2						4
le	Trinity (C)	2			2					4
nam	LMDC Toulouse (D)	2		2						4
L N	Bath univ (E)		3		3					6
rato	LGCGM Rennes (F)		3		3				3	9
abo	Vicat (G)	3	1	6				3		13
L	IFSTTAR (H)		3		4					7
	LiMATB Lorient (I)	3		3		3				9
	Lhoist (J)	2		2						4

Total per batch	12	15	15	15	3	3	3	3	69
Total per specimen size		57	7			12	2		

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Table 1: Summary datas for tested specimens

155 3.1 Raw material characterization

156 **3.1.1** Shives

The shives used in this study are from the same producer, but they were stored in two separate places. One bag with the reference 13 0173 KANABAT at the ENTPE laboratory, noted S1 shiv, and the other one at Vicat laboratory with the reference 13 0174 KANABAT, noted S2 shiv. Samples, of about 1 kg each, have been taken and characterization tests were conducted according to the protocol proposed in [17], [18]. The characterization tests were made in laboratories A and G; they include among others: bulk density, water absorption and particle size distribution by two methods: mechanical sieving and image analysis.

164 **3.1.1.1** Bulk density (kg/m³)

Tests of bulk density were conducted according to the protocol in [17]. The results obtained for the two types of hemp shives show that there is slight difference, with 143.6 kg/m³ for S1 and 147.5 kg/m³ for S2. Observed differences may be due to errors in manual handling or to the accuracy of the used method. Whatever, these differences are acceptable as they are below 2.7%.

169 3.1.1.2 Water absorption

Tests of water absorption were conducted according to the protocol in [17]. The water absorption capacity of these aggregates is determined gravimetrically by applying the expression: $W(t) = \frac{M(t)-M_0}{M_0} \times 100$, where W(t) is the water absorption ratio at time t, M(t) the soaked hemp shive aggregate mass at time t, and M_0 is the initial oven-dried aggregate mass. The water absorption W is calculated after soaking for 48 hours using the expression: $W = IRA + K_1 \times Log(t)$, where K_1 is a kind of diffusion rate in shiv cells. IRA represents the characteristic factor of the 176 external water adsorption on the shiv surface, and is related to the first minute measurement. The



177 results are shown in Figure 1.

Figure 1: Water absorption curves (experimental and analytical) for S1 and S2, immersion time in log scale
For comparison purpose, the test was conducted in two separate laboratories: A and G. The

results are almost identical for both laboratories especially for initial mass water absorption but with a slight difference in the case of S2. In terms of initial water absorption, they are different, with initial mass water absorption around 150% and 200% for S2 and S1, respectively.

184 3.1.1.3 Particle size distribution

The particle size distributions are analyzed by using two methods: mechanical sieving and image analysis. The first method is the reference technique when dealing with characterization of mineral aggregates [19]. For the analysis of vegetal origin aggregates, square mesh sieves are of limited interest because they do not take into account the elongation of aggregates [20].

By the sieving method, Nozahic [17] made a comparative study on hemp shiv and sunflower aggregates and realized that the two types are almost similar in size. He concluded that mechanical sieving technique is not yet suitable neither for determining the size of a lignocellulosic particle aggregate, nor for comparison of two different kinds of aggregates. His conclusion is in accordance with the results of mechanical sieving obtained in the present study and shown in Figure 2 where both S1 and S2 are almost identical.

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Figure 2: Grading curve by mechanical and image analysis methods for S1 and S2.

However, the second method of image analysis brings clearly richer information than the
previous method. This latter has been used and published for the first time in 1996 [21]. In our
study, we have used a similar approach to the work in [10], [12], [18] but with the ImageJ
software [22] and a sample of 3g has been considered for each hemp shiv. The comparison of
both S1 and S2, illustrated in Figure 2, reveals, in contrast to sieving method, significant
differences between both axes. The obtained specific surface areas are 13187mm² and
13913mm² for S2 and S1, respectively.

204 3.2 Preparation of compression test specimens

205 3.2.1 Mix proportioning

In construction, hemp concrete has several applications, such as: filling wooden frame walls, roofing insulation, etc. To each application correspond a given number of specifications such as minimum compressive strength and Young's modulus [23], which can be met by specific formulations. For the purpose of the herein study, it has been decided to use the formulation for wall application [23]; as the objective is not to analyse the formulation, any other one may have been used. The quantities in kilograms per batch of 80 liters are detailed in Table 2.

	Shiv (kg)	PNC (kg)	Citric Acid	Water (kg)	Ratio Water/PNC	Ratio Shiv/PNC
	8	20	0.06	19,2	0.96	0.4
212			Table 2: Tested form	nula for wall app	lication per batch	

213 **3.2.2** Mixing of hemp concrete

Each constituent is weighed in buckets. The shiv is put in the mixer, then the PNC with Citric Acid is introduced; they are then mixed with 40% of the mixing speed for few minutes. Water is added and the mixing retaining. The mixing speed is increased to 50% then kept until homogeneous mixture is obtained. Finally, for the use, the mixer is emptied into a wheelbarrow.

218 **3.2.2.1** Casting method for specimens

The mold is filled by 5 or 6 layers; two consecutive layers must be compacted using a suitable tool. For the last layer, the upper surface is kept smooth and the specimen is weighed. A cover is put and the specimen is kept returned for a period of at least 72 hours after which the cover and the bottom are removed. The specimen is then kept at 20°C and 55% of relative himidity for 90 days. To ensure that the tested specimens are identical, they were manufactured the same day and were dried for 90 days under the same conditions at the laboratory G. After this drying period, samples were transported to ten different laboratories for compression testing.

226 3.2.2.2 Protocol of the compressive test

Tests were done under the same conditions, the detailed below protocol, was carefully followed by all laboratories. Specimens were dried under an oven at 50°C for 48 hours before the compressive test.

- Weighing the specimen with the mold; then remove the mold using a cutter: remove the
 sample ends then cut just the surface of the mold; and mark it with the same reference on
 the mold;
- weighing the specimen without the mold; then put it in an oven at 50°C until a stabilization of weight equal to +/-2%; and left it in a sealed plastic bag until the test day;
 before the test, measure three diameters (at top, bottom and middle) and the height every 120°;

- 4. no surfacing of the sample and a perfect parallel plates is made before the starting of thetest;
- 5. The test must be displacement controlled at the rate of 3mm/min for loading stage. Theunloading stage should be 6mm/min or free if it is not possible to control it;
- 241 6. Applying three load cycles depending on specimen size:
- 2421st cycle: loading is done from 0 to 1% of relative deformation and unloading until zero243load or zero displacement; 2nd and 3rd cycles are the same as the 1st, the strain is always244increased by 1% for each cycle. The final loading: from 0 until the total failure load of245the specimen (maximum of 20% of strain) and unloading until zero load (when possible)246or zero displacement.
- Voluntarily for some specimens, in the case of lab C: I-11-7; I-11-8; IV-11-11; IV-11-12; and I
 lab: I-11-2; III-11-11; I-16-1; the compressive tests were done with a monotonic loading.

249 3.3 Mechanical analysis of hemp concrete properties

250 3.3.1 Young's modulus (Floating modulus on loading stage)

251 For the hemp concrete, the Young's modulus is not constant because of strong nonlinear 252 behavior even in the elastic domain. As applied in soil mechanics [24], the hemp concrete may 253 have also four different types of modulii, which can be calculated as shown in Figure 3. The initial tangent modulus Eini corresponds to the slope at the beginning of loading in the stress-254 255 strain curve. The various loading levels of the curve may be described by a "secant" modulus 256 E_{sec} , defined by the slope of the line connecting the origin at the current point and a "tangent" 257 modulus E_{tan} , may be determined by the slope of the curve in the neighborhood of a given point. 258 In cyclic loading, the modulus E_{CYC} may be determined by the slope of the line connecting the 259 two points reversing the strain direction. In opposite to initial Young's modulus, which might 260 have errors due to small strains, the tangent modulus calculated on loading phase with higher strain values, which allows reducing the errors. In the current study, the modulus is calculatedaccording to the procedure named "floating modulus".

263 **3.3.1.1** Young's modulus calculation method (floating modulus).

According to the frequency of data acquisition (nearly 10Hz or 10 values per second, Figure 4): the loading steps are identified then the floating modulus is calculated in each step using: $E = \frac{\Delta \sigma}{\Delta \epsilon}$; where: E is the modulus around a given point, $\Delta \sigma$ and $\Delta \epsilon$ are strength and strain respectively considered between -5 and +5 seconds around the considered point. The maximum of modulus is identified for each step. The floating Young's modulus value is therefore, the mean value of maximum values obtained at the 2nd; 3rd and 4th loading steps.

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Figure 3: Modulus definitions [24]

Figure 4: Identification of the loading phases to calculate the floating modulus

271 **3.3.2** Characteristic values and coefficient of variation (COV)

The characteristic value of a quantity measured experimentally corresponds to the representative value to be included in the computation procedure for a purpose of design, maintenance or rehabilitation or any other decision process. For example, the compressive strength of concrete is defined as the resistance below which there are only 5% of test results [25]. Under the assumption of normality, it is proposed to calculate the characteristic value of concrete compressive strength as follows: $f_{ck} = f_{cm} - 1.645\sigma_{fc}$ (1); where f_{ck} is the characteristic value, f_{cm} is the average value of all the test results and σ_{fc} is the standard deviation of test results; the coefficient 1.645 corresponds to a 5% quantile of the normal gaussain distribution. It is to note that all experimental results were subjected to the test of normality and the test was not rejected.

Then formula in (1) is used in the current study with the probability level of 5%.

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The coefficient of variation (COV) indicates the dispersion of the experimental results; it is calculated by the ratio between the standard deviation and the mean value, in (%). Table 3 gives accepted limits of standard deviation and coefficient of variation for concrete [15].

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Quality control	Accepted limits for the coefficient of variation (%)
A (excellent)	10
B (average)	15
C (poor)	20

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 Table 3: Accepted limits of variability of concrete compressive as a function of the quality control [15]

288 3.3.3 Statistical tests

289 According to the samples used, they can be classifid in eight populations, four batches for both 290 hemp shives and two specimen sizes in each batch. Using statistical tests, like Student test [26] 291 by comparing samples two by two or ANOVA [27], one can determine whether the samples 292 originate from the same population or not. ANOVA (ANalysis Of Variance) is a generalization 293 of the mean comparison with K subpopulations or samples. K equal to 2 correspons to Student 294 test. For ANOVA test, if the null hypothesis H0 is rejected, it is not possible to know the 295 subpopulation that doesn't belong to the population. It is then necessary to use Student test by 296 comparing subpopulations two by two. The main objective of these statistical tests is to provide 297 the best synthetic information about the characteristics of the population. For a given population, 298 one can determine the probabilistic distribution that fits better the observed data. The parametric 299 Student test [26] has bee used by comparing the mean values and a significance level $\varepsilon=0.05$ 300 hasbeen considered. The tests were carried out using the software XLSTAT [28].

301 3.3.3.1 Identification of mean values

For a Student test, two conditions are necessary: the two compared samples must satisfy the Gaussian distribution, this condition is very often verified, and the second condition is that the two samples must have the same variance [26]. In this study, both conditions have been satisfied and the Student test has been performed. It leads to rejection results in some cases (Table 4).

			Test results for :	
Specimen size	Test between batches	Density	Maximum compressive strength	Young's modulus
11x22cm	I and II	not reject	reject	not reject
	III and IV	reject	reject	reject
16x32cm	I and II	not reject	not reject	not reject
	III and IV	reject	reject	not reject

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307

Table 4: Student test for batches in both specimen sizes

308 Consequently it is not possible to combine samples into one population for statistically 309 meaningful size (i.e. statistical analysis requires a minimum number of samples to get acceptable 310 error; the required sample size depends on the statistical property or test to be applied, e.g. mean, 311 standard deviation, density function fitting, etc.). In order to assess the dispersion of data it is 312 proposed to adjust the sample mean values with respect to the reference mean; this leads to shift 313 the whole probability distributions, such that their mean values become centered on the same 314 reference point. It is important to note that this adjustment is only applied to characterize the 315 sample standard deviation and distribution type, but not to determine the mean values. By 316 applying this approach, Student's test results for all samples are not rejected. For more clarity, 317 the approach is detailed below.

318 Consider two given samples $X = \{x_1; x_2; ...; x_i\}$ and $Y = \{y_1; y_2; ...; y_i\}$; their respective mean 319 values \overline{X} and \overline{Y} ; if one wants to adjust the mean value of Y to the mean value of X, then one has 320 to proceed as following: calculate the adjusted mean value $\overline{Y'}$ by using the formula:

$$\overline{Y'} = \frac{1}{n} \sum_{i=1}^{n} [y_i + (\overline{X} - \overline{Y})]$$
(3)

321 If the above formula in (3) is developed, it comes that: $\overline{Y'} = \overline{Y} + \overline{X} - \overline{Y}$ and finally gives: 322 $\overline{Y'} = \overline{X}$, but for this time with variables of $Y' = \{y'_1; y'_2; ...; y; y'_n\}$ which are different of those 323 of $X = \{x_1; x_2; ...; x_m\}$ and $Y = \{y_1; y_2; ...; y_n\}$ respectively. This approach leads to two different 324 samples having the same mean value; hence it allows combining both samples for scatter and 325 goodness-of-fit analyses.

326 4 Results and Discussions

327 By considering the testing laboratory, the batch, the hemp shiv type and the specimen size, the 328 studied properties are: density, compressive strength and Young's modulus. In order to simplify 329 the notations, the following abbreviations are used: MV for the Mean Value, SD for the Standard 330 Deviation, COV for the Coefficient Of Variation and CV for the Characteristic Value.

331 4.1 Repeatability of the results between testing laboratory

As seen, the density, the compressive strength and the Young's modulus may vary according to many parameters such as: compaction energy [9], [10], measuring method [11] and hemp shiv type [12]. In this section, analyses for results in Table 5 and Table 6 focus on the impact of testing laboratories.

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]	Density	(kg/m ³)	Со	mpressive	strength (N	MPa)	Young	Young's modulus (MPa)			Compressive strength (MPa) Young's modulus (MPa)				
Lab name	MV	SD	COV	CV	MV	SD	COV	CV	MV	SE) CO	V CV					
All lab	471.22	28.28	6.00	424.84	0.45	0.05	10.69	0.37	36.86	5 7.0	8 19.22	2 25.24					
Α	496.88	31.25	6.29	445.63	0.49	0.03	5.46	0.45	33.82	4.5	8 13.5	5 26.31					
В	476.93	17.64	3.70	448.00	0.48	0.04	8.45	0.41	40.72	2 5.0	1 12.29	32.51					
С	471.44	29.14	6.18	423.65	0.44	0.03	6.73	0.39									
D	465.95	18.47	3.96	435.65	0.42	0.06	13.87	0.32	34.16	3.4	3 10.05	5 28.53					
Ε	468.44	29.35	6.27	420.31	0.49	0.02	4.47	0.45	40.87	8.6	3 21.13	3 26.71					
F	465.20	26.58	5.71	421.61	0.49	0.04	8.36	0.42	35.27	3.1	8 9.02	30.05					
G	453.72	12.96	2.86	432.47	0.41	0.04	9.42	0.34	35.33	5.1	1 14.4	5 26.96					
Н	472.48	35.16	7.44	414.82	0.46	0.05	10.56	0.38	44.01	9.8	9 22.47	7 27.79					
Ι	452.71	12.64	2.79	431.99	0.45	0.05	10.78	0.37	36.01	5.2	3 14.53	3 27.43					
J	514.62	15.53	3.02	489.16	0.43	0.04	8.30	0.37	28.81	4.7	5 16.49	9 21.02					
344 т 345	able 5: De	ensity, n	naximu	m compr	essive str	ength and 11x22	Young's mo cm	odulus val	ues per	labora	tory, spe	cimens					
346																	
347]	Density	(kg/m ³)	Cor	npressive	strength (N	(IPa)	You	ıng's n	nodulus ((MPa)					
Lab name	MV	SD	COV	CV	MV	SD	COV	CV	MV	SD	COV	CV					
All lab	443.53	29.70	6.70	394.81	0.38	0.06	16.77	0.28	35.58	4.46	12.54	28.26					
Α	423.41	1.96	0.46	420.20	0.32	0.02	5.07	0.29	30.97	2.28	7.35	27.24					
F	495.45	8.56	1.73	481.42	0.48	0.02	4.47	0.44	39.13	3.04	7.77	34.14					
G	445.47	4.10	0.92	438.74	0.39	0.00	1.17	0.39	32.77	2.95	9.02	27.93					
Ι	420.38	2.10	0.50	416.93	0.32	0.01	4.15	0.29	39.06	3.42	8.76	33.45					

348 349

Table 6: Density, maximum compressive strength and Young's modulus values per laboratory, specimens16x32cm

350 **4.1.1 Density**

The analysis of results obtained by different labs shows small variability for a given specimen size; with a COV of 6.0% and 6.7% for all labs in both cases small and large specimens respectively, as shown in Table 5 and Table 6. The observed difference in the characteristic values of the density, while comparing both specimen sizes will be discussed in section 4.4.

355 Within each category of a specimen size, the observed results have excellent quality with 356 reference to the accepted limits in Table 3.

357 4.1.2 Maximum compressive strength

For small specimens 11x22cm, the compressive strength results show values ranging from 0.32 MPa for lab D to 0.45 MPa for labs A and E, as shown in Table 5. In general, there is no considerable variability in the obtained results. The method and machines used give similar results for the characteristic strength with 10.69% of COV for all labs. In case of large specimen size, the COV is 16.77% for all labs, as shown in Table 6; this high variability leading to poor quality of the strength. For small specimen size, the quality is excellent with average COV close to the accepted limits as given in Table 3.

365 4.1.3 Young's modulus

366 Results taking into account the impact of testing laboratory on the evaluation of Young's 367 modulus show mean values ranging from 28.81 MPa to 44.01 MPa. In fact, there are two classes 368 of values, one in the interval from 33 MPa to 38 MPa, and the other in the interval from 40 MPa 369 to 45 MPa, the value of lab J looks like an isolated case. For larger specimen size, the results 370 seem to be homogeneous with a maximum COV equal to 9.02%. These results must be analysed 371 carefully as the number of specimens are not statistically large. Two laboratories have high COV 372 values of 22.47% and 21.13%, leading to a COV for all laboratories equal to 19.22%, (Table 5). 373 With such COV, the results are of poor quality compared to the limits in Table 3. There is a 374 significant impact of the testing laboratory on the Young's modulus where the obtained results 375 have poor quality, although the obtained results have excellent quality for the compressive 376 strength. This has to be considered carefully, since it is known that there is a strong correlation 377 between the Young's modulus and the compressive strength. The main explanation to this 378 observation is the nonlinear behavior of strength-strain curve, because the maximum strength 379 was calculated beyond the linear phase of the curve, as detailed in section 4.5.

380 4.2 Repeatability of the results between batches

381 Although the batch type is not yet studied in the literature to our knowledge, but this parameter

	Com	pressive st	trength (M	Yo	ung's moo	lulus (MPa	a)	
Batch type	MV	SD	COV	CV	MV	SD	COV	CV
I. (11x22cm)	0.41	0.04	9.78	0.35	30.46	3.81	12.52	24.21
II. (11x22cm)	0.47	0.04	8.18	0.41	33.35	3.85	11.53	27.04
III. (11x22cm)	0.44	0.05	10.93	0.36	38.04	4.54	11.94	30.59
IV. (11x22cm)	0.48	0.04	8.16	0.42	44.07	7.46	16.93	31.84
I. (16x32cm)	0.32	0.01	4.15	0.29	39.06	3.42	8.76	33.45
II. (16x32cm)	0.32	0.02	5.07	0.29	30.97	2.28	7.35	27.24
III. (16x32cm)	0.39	0.00	1.17	0.39	32.77	2.95	9.02	27.93
IV. (16x32cm)	0.47	0.02	4.51	0.44	39.13	3.04	7.77	34.14

Table 7: Maximum compressive strength and Young's modulus values per batch, specimes 11x22cm and16x32cm

382 may influence the results as shown in Table 7.

383 384

385

386 4.2.1 Maximum compressive strength

The results for compressive strength show that the values for batch IV are higher for both specimen sizes than is the case for Young's modulus. Batches from S2 seem to have high values as shown in Table 7. This trend is analyzed in section 4.3 where the impact for both shives is studied. As it will be discussed in the next section for the Young's modulus, the compressive strength shows also some variability for different batches, therefore the mixture in different batches must be carefully performed.

393 4.2.2 Young's modulus

Mean values for Young's modulus increase from Batch I with 30.46 MPa to Batch IV with 44.07 MPa as given, Table 7. There is no explanation for this observed trend. However, even with this trend, it is clear that batches from the same shiv have comparable results. In batch IV, the COV equal to 16.93% is greater than other batches, as this one had been manufactured the 398 lastest, maybe the operators did not maintain the same conditions (e.g. compaction energy...)
399 since the beginning up to the end. As this trend is not the same case for large specimens, the
400 justification given above is not necessarily true. For both cases (small and large specimen sizes),
401 an average quality is observed, with respect to limits in Table 3. This means that the bach does
402 not have a great impact on the results, but sometime it may cause variability, as it is the case of
403 batch IV. Therefore, it is necessary to be careful when mixture is done in different batches.

404 4.3 Repeatability of the results for different hemp shiv types

Arnaud and Gourlay [12] studied the impact of hemp shiv; they concluded that the use of smaller shiv results in concretes whose higher mechanical properties at long term. Nguyen [10] compared two shives one pure another containg fibers; as conclusion to its study there was no big difference on their mechanical properties. In our study, some differences have been observed, according to the results given in Table 8.

	Density (kg/m ³)						Compressive strength				dulua (I	
							(MPa)				Young's modulus (MPA)	
Hemp shiv	MV	SD	COV	CV	MV	SD	COV	CV	MV	SD	COV	CV
S2 (11x22cm)	488.88	23.11	4.73	450.9	0.46	0.05	10.38	0.38	43.45	6.72	15.47	32.43
S1 (11x22cm)	451.61	19.17	4.25	420.1	0.44	0.05	10.79	0.37	31.86	3.14	9.87	26.71
S2 (16x32cm)	469.78	27.08	5.76	425.3	0.43	0.05	10.58	0.36	32.77	2.95	9.02	27.93
S1 (16x32cm)	422.10	2.51	0.60	417.9	0.32	0.01	4.00	0.30	30.97	2.28	7.35	27.24

410

 Table 8: Density, maximum compressive strength and Young modulus values per hemp shiv

411 **4.3.1** Density

When comparing both hemp shives in terms of density, slight differences are observed between the obtained densities, even with the specimen size. 420.16kg/m³ and 450.97kg/m³ with 417.98 kg/m³ and 425.38kg/m³ characteristic values for S1 and S2 in both small and large specimen sizes, respectively are obtained as shown in Table 8. Large values have been observed for S2, which is consistent with the drying kinetics.

417



Figure 5: Drying kinetics per hemp shiv and specimen size

Figure 6: Drying kinetics per specimen size

418 According to the drying kinetics in Figure 5, it appears that the drying is only affected by the 419 specimen size, which seems normal, because they dry faster since they have a greater specific 420 area than larger specimens. On the other hand, a difference in fresh density is also observed 421 depending on both specimen sizes and hemp shiv types. Small specimens have a higher fresh 422 density than the large specimen, which could be explained by a greater compaction (same 423 "compaction energy" applied by the operator on a smaller area). The specimens made from S2 424 have a higher fresh density than those from S1, which means that, they were more compacted. 425 The initial water contents measured are 10,18% and 11,12% for S1 and S2 respectively. This 426 difference in initial water content between S1 and S2 confirm our results. As the water content of 427 the S2 was more important than in S1, the initial absorption of water was reduced (which is the 428 case according to results in Figure 1) and S2 was more easily compacted which explains the 429 high value for fresh density.

430 4.3.2 Maximum compressive strength

The observed compressive strength results are 0.38 MPa and 0.37 MPa for small specimen size; with 0.36 MPa and 0.30 MPa for large specimen size both for S2 and S1, respectively. The maximum strength values for S2 are greater than for S1; this trend is the same for Young's modulus.

435 4.3.3 Young's modulus

For both specimen sizes, results show that, Young's modulus values for S2 are greater than for
S1 values. Observed results show also a high variability for S2 with a COV equal to 15.47%.
This is probably due to the fact that batch IV is for S2 and as shown in the previous section,
there is a high variability within this batch.

440 With respect to the type of shiv, in both cases of Young's modulus and compressive strength: 441 these differences can be explained by the fact that, since S2 has a small specific area 13187mm², versus 13913mm² for S1, the hemp particles are better coated by the binder during the mixing 442 443 process of the concrete, which may explain this better mechanical properties of the hemp 444 concretes made from S2. This remark is similar to the results obtained by Arnaud [12] where he 445 remarked that after 4 months, the finer hemp particles gave better mechanical properties than 446 longer hemp particles. This difference may be also justified by the fact that the initial water 447 absorption of S2 is 146% and for S1 is 212%. This means that S1 absorbs a lot of mixing water 448 and this results in a dry mixture, leading to poor mechanical properties. To avoid this problem, 449 shiv particles may be wetted before the mixing process.

450 **4.4** Repeatability of the results with respect to specimen sizes

451 **4.4.1 Density**

Although the results for each specimen size are not varying too much, density characteristic values obtained for both sizes are 424.84 kg/m³ and 394.81 kg/m³ for small and big size, respectively (Table 5 and Table 6). Unlike to what is observed in the case of the maximum compressive strength, there is no difference for the COV values, as discussed in 4.3.1, there are always great values for small specimen size.

457 4.4.2 Maximum compressive strength

458 Considering the results obtained for the characteristic values 0.37 MPa and 0.28 MPa for small 459 and big specimens, respectively (Table 5 and Table 6); the specimen size does not have exactly 460 the same trend for the compressive strength as for Young's modulus. Since there is no big 461 difference for minimum, maximum and mean compressive strength values, then the observed 462 difference for characteristic values is related to the COV values.

463 4.4.3 Young's modulus

Results on the impact of specimen size in the case of Young's modulus show comparable values
for the mean and characteristic values, (Table 5 and Table 6). A significant difference is
observed for the maximum values with a factor equal to 1.32.

467 4.5 Correlation between mechanical characteristics

468 The representation given in (Figure 7) between cubic root of compressive strength and Young's 469 modulus shows that these two mechanical properties are correlated. According to Hooke's law, 470 the Young's modulus corresponds to the slope calculated in linear stage on the curve strength/ 471 strain. For a given homogeneous material; different samples should give almost the same values 472 of stress and Young's modulus for a given strain. Hence the graph strength/Young's modulus 473 may correspond to a concentrated cloud of points. As in the current study, the maximum 474 strengths are obtained around 5% of strain, the Figure 7 was expected to be a concentrated cloud 475 of points but it is not the case.



476

Figure 7: Correlation for cubic root of maximum strength and Young modulus for all specimens The trend observed in Figure 7 shows to what extend the mechanical properties of hemp concrete are sensitive to studied parameters. As explained in section 4.1.3, this may also due to the fact that the maximum compressive strength is calculated beyond the linear phase of the curve. Compressive strength varies from 0.3 MPa to 0.52 MPa. A great amount of values is located between 30 MPa and 40 MPa for Young's modulus. Nevertheless, the cubic root of compressive strength is in general increasing with Young's modulus.

484 4.6 Summary of observations

At one hand, there are more or less considerable variabilities for hemp concrete properties related to the type of parameters considered. The results, seen so far and performed analyses, have significant scatter that is difficult to estimate or to measure. The main source of these dispersions is the interference of different parameters on the observed results.

Moreover, according to the literature review, it has been shown the inadequacy of the accuracy and the confidence level to be given to the results in literature. Regarding the characteristic values of this study, they are up to now given with respect to different parameters. A study taking into account all parameters for a unique characteristic value is necessary. This study is

23

493 proposed in the next section, with the goal of computing the characteristic values for the three494 properties.

495 4.7 Probability distributions

496 In addition to statistical values (mean, standard deviation...) computed in the previous sections 497 of this paper, it is now required to specify the probability distribution that fits properly the 498 experimental data. As a first step, a preliminary statistical analysis has been carried out to test a 499 large number of probability density functions, in order to select the most appropriate candidates. 500 For each batch, and also for grouped batches, the goodness-of-fit tests have been performed with 501 various distributions, in order to determine which distribution fits better the data. Although 502 various types of probability distributions have been considered to fit the experimental results, 503 three distributions are recommended for practical engineering, namely: normal, log-normal and 504 Weibull distributions, which are commonly used in reliability analysis of civil engineering 505 structures [29]. For Weibull distribution it is commonly used in mechanical engineering to 506 describe statistical variation of failure strength of a material [30]; maybe it is not suitable for 507 hemp concrete, in the current study, it is proposed for information, others studies are required to 508 confirm its use. This goodness-of-fit test is conducted separately for the density, the compressive 509 stress and the Young's modulus. However, before performing these tests, it is mandatory to 510 check whether the dispersion is due to the scatter of the population, or due to mixing different 511 populations with different mean values. For this reason, a test has been conducted to verify that 512 the batches belong to only one consistent population, as the undeneath populations have similar 513 mean values; otherwise the goodness-of-fit test results will be insignificant and the batches 514 should be splitted into two or more populations.

515 4.7.1 Density

As explained in section 3.3.3, the statistical tests have been done on different batches. The results given in Table 4 show that it is only possible to combine batches II and I. This leads to have three populations: batches (I&II), batch III and batch IV.

519 As a matter of fact, although batches III and IV come from the same type of hemp shiv S2, the 520 statistical tests showed that they do not belong to the same population, as this hypothesis has 521 been rejected! In order to explore the reasons for this statistical test result, it has been assumed 522 that there could be a laboratory, which disturbs the results in one or both batches. To detect this 523 laboratory, ANOVA test may have been used, but specimens in each lab are not enough (Table 524 1) to perform a significant test. As result, comparison tests have been conducted for both batches 525 by eliminating lab results, one after another. Unfortunately, the test results remained negative. 526 Furthermore, it has been noticed that by eliminating the batch III results for lab G, the test 527 showed that both batches III and IV belong to the same population, with the risk of rejecting the null hypothesis H0, equal to 9.05%. This result let believe that the impact of lab G is related to 528 529 the fact that it is the only laboratory which tested 6 specimens of batch III, while the others had 530 tested 2 or 3 specimens each. For this reason, the statistical test has been conducted by keeping 531 only 5, 4, 3 and 2 specimens among those of lab G, but in vain. As a conclusion both batches are 532 considered as two separate populations.

Histogram for all specimens







Figure 8: Probabilistic law for density

Distribution	Nor	mal	Log-n	ormal	Weibull (3)			
Parameters	μ	σ	μ	Σ	к	λ	η	
	450.992 22.348		6.11 0.049		1.917	39.806	415.679	
Statistical moments (µ;	(450.992;		(450.995;		(45	1 207 [.] 22	236)	
σ)	22.3	48)	21.	99)	(431.207, 22.230)			



Table 9: Distribution parameters and statistical moments for material density

536

537 As there are not a large number of specimens in each one of the three populations, the 538 characterization of the coefficient of variation can be carried out by scaling the experimental 539 result of each specimen using the mean value of the population corresponding to batch I. The 540 approach described in section 3.3.3.1 is therefore applied to scale the mean values, in order to get 541 appropriate representation of the dispersion. The obtained population is thus shown to follow 542 properly normal, log-normal and three-parameter Weibull distibutions, while logistic and GEV 543 provide also good fitting. For the considered distributions, Figure 8 shows how the density 544 functions fit the experimental data and Table 9 indicates their statistical moments.

545 4.7.2 Maximum compressive strength

Regarding the maximum compressive strength, the statistical tests have been performed in order to determine whether it can be considered only one population or not. All test results were negative for both specimen sizes and batch parameters, except batch I and II with large specimen dimension, as shown in Table 4. In other words, the Student's tests have led to 7 different populations, which should then be fitted by normal, log-normal and logistic distributions.



551

552

Figure 9: Probabilistic law distributions for maximum strength

Distributions	Nor	mal	Log-n	ormal	Weibull (3)			
Parameters	μ	σ	μ	Σ	к	λ	η	
	0.397	0.052	-0.93	0.135	2.3	0.115	0.295	
Statistical moments (μ; σ)	(0.397; 0.055)		(0.397;	0.055)	(0.397; 0.045)			



Table 10: Distribution parameters and statistical moments for the maximum compressive strength

554

555 In order to analyze the scatter of experimental results, the scaling procedure described in section

556 3.3.3.1 is applied to get a unique scaled population. The experimental results can then be fitted to

normal, lognormal and Weibull distributions, Figure 9, with the statistical moments given in Table10.

559 4.7.3 Young's modulus

The Young's modulus is calculated by the method explained in section 3.3.1. The experimental results obtained by this method are subjected to Student's test, and the results are given in Table 4. As the tests are rejected in the case of small specimen for batches III and IV, we used the approach described in section 3.3.3.1 to scale the results. The fitting of probability distributions is shown in **Figure 10** with their statistical moments in Table 11.

The trend of the experimental results indicates the existence of two sub-populations: a first subpopulation is located at the mean value of 27.75MPa and a second subpopulation has a mean of 35.75MPa, as shown in **Figure 10**. This trend may be due to the fact that we have two specimen sizes. The same trend was also observed in 4.4.3 where a significant difference is observed for the maximum values with a factor equal to 1.32.

Histogram for all specimens







- 571 572
- 573

Distributions	Normal		Log-n	ormal	Weibull (3)			
parameters	μ	σ	μ	Σ	к	λ	η	
	31.874	5.669	3.445	0.185	2.76	15.868	17.751	
Statistical moments (μ ; σ)	(31.874; 5.669)		(31.898; 5.943)		(3)	94)		

574

Table 11: Distribution parameters and statistical moments for Young's modulus

575

576 4.8 Proposed characteristic values for studied properties

As discussed above, it is not possible to consider each parameter separately. Further probabilistic studies could be required to take into account the interaction of all parameters, but this is beyond the scope of the present work. As the marginal probability distribution for each parameter is determined, one can compute the characteristic values with the formula given in eq (1) with the probability level equal to 5%. Using the obtained normal distributions, the characteristic values of the three parameters are computed as: **22.5 MPa** for the Young's modulus, **0.30 MPa** for the compressive strength and **415 kg/m³** for the density.

584 5 Conclusion

585 The statistical analysis has been performed for three material properties, namely the density, the 586 compressive strength and the Young's modulus, by taking into account four parameters: testing 587 laboratory, batch type, hemp shiv type and specimen size. The results obtained by different 588 laboratories show that there is an accurate repeatability for compressive strength and dry density. 589 However, the results for Young's modulus are of a large variability, with results varying from 590 excellent to poor quality. The results also showed that there is some variability between different 591 batches, and therefore the mixing procedure must be done with an utmost care. The impact of 592 initial water content on the density has been also highlighted. More initial water content is, less

will be the density of the corresponding hemp concrete. It has also been noticed that the hempwith small particle sizes leads to better mechanical properties of hemp concrete.

According to the obtained results, plausible evidence for specimen size effect was observed. However, further investigations should be undertaken in the future on larger number of specimens with different sizes, in order to provide full understanding of the effect of specimen size.

Regarding the statistical analysis, the mean values and standard deviations of the considered batches have been computed and provide consistent results. A statistical procedure has been proposed to assess the scatter and the distribution type of the combined batches. The goodnessof-fit test has shown that the experimental results are in good agreement with the probability distributions: normal, log-normal and Weibull. According to usual recommendations in civil engineering, especially in Eurocodes, the log-normal distribution may be suggested to model the considered properties.

This study will be enhanced by ongoing works on separating the statistical contributions of each basic paremeter (batches, hemp shiv...), through the development of Bayesian network approaches. This Bayesian network study may bring useful informations to answer to the remaining questions.

For future works, on one hand, the acoustical and thermal properties for hemp concrete material should be also analysed. On the other hand the impact of fabrication method such as vibration damping on the properties performance of hemp concrete material should also be investigated.

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