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M. Benzerara, Simon Guiheneuf, R. Belouettar, A. Perrot. Combined and synergic effect of algerian natural fibres and biopolymers on the reinforcement of extruded raw earth. Construction and Building Materials, 2021, 289, pp.123211. 10.1016/j.conbuildmat.2021.123211. hal-04506790

HAL Id: hal-04506790 https://hal.science/hal-04506790v1

Submitted on 22 Jul 2024

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1 Combined and synergic effect of algerian natural fibres and biopolymers on

- 2 the reinforcement of extruded raw earth
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- 13 14 * corresponding author 15
- 16 Abstract

- 17 This paper deals with the development of sustainable building earth-based materials. More 18 precisely, it addresses the study of the reinforcement of raw earth with natural fibres 19 originated from Algeria (diss and date palm tree fibres) and their stabilisation with xanthan 20 gum. The aim of this study is the design of extruded earth-based building blocks with 21 improved mechanical properties such as compressive strength and ductility. The effects of 22 separate and simultaneous addition of date palm trees or diss fibres (with length varying 23 between 5 and 15 mm at dosages of 1.5 and 3% in volume) and stabiliser (xanthan gum and 24 HMP at 1 and 2% of the dry earth mass) on the flexural and compressive strength of the 25 stabilised and unstabilised extruded materials are compared. Results show that adding only 26 fibers decreases the compressive strength of the earth in comparison with the unreinforced 27 sample. It is also shown that diss fibres provides better reinforcing efficiency than date palm 28 tree fibres and that a combined addition of xanthan gum and natural fibres create a synergic 29 effect that greatly improved the material mechanical behaviour: higher compressive and 30 tensile strengths and better ductile properties. These results are fully supported by 31 microscopic observations and pull-out tests carried out on single fibres.
- 32 **Keywords:** Biopolymer, natural fibre, raw earth, extrusion, pull-out tests.

1. Introduction

Climate change is the major challenge that currently faces humankind. Anthropic carbon dioxide emissions weaken societies especially in less advanced and developing countries [1]. Building materials such as concrete are responsible of a significant part of this human carbon footprint [2] and it is required to find efficient alternative to conventional building materials in order to decrease drastically the building industry's environmental impact [3–5]. It is also worth noting that world's population is still growing and that affordable and sustainable housing solutions have to be found.

One promising solution lies on the valorisation of natural resources or inert by-products of the construction industry such as extracted and raw earths [6–8]. For instance, according to Hamard et al. [7], at the scale of the French Brittany region (27 200 km²), earthwork induced 0.7 Mt / year of non-valued excavated earth which can cover a large part of the material needs for local housing. Since about twenty years, many works have been carried out to reinvent vernacular construction techniques and makes it compatible with conventional construction techniques in terms of productivity and cost. However, raw earth material is a variable resource which is very sensitive to water and may present limited resistance to seismic loading which prevents its use in compliance with structural design standards in many countries [9,10].

It is therefore required to provide guaranteed mechanical properties and improve the durability of earth material in order to make it compatible with modern building design and standards requirements. One solution is the use of hydraulic binders that are known to efficiently increase the water resistance of raw earth while increasing their compressive strength. This solution has been used for different techniques such as compressed earth blocks [11,12], rammed earth [13–15] or extruded blocks[16,17]. But, even at a limited dosage, cement addition can make earth materials having a higher carbon footprint than concrete blocks [18]. Moreover, according to Van Damme and Houben [19], the ratio between carbon dioxide emission and compressive strength (carbon intensity ratio) is higher for cement-stabilised raw earth than for other construction materials such as concrete or unstabilised raw earth. Another drawback of this stabilisation technique is that cement or lime stabilised materials require to undergo a humid cure in order to allow for the binder hydration as shown by [16].

Another promising method of earth stabilisation consists in the addition of biopolymers that have been used for centuries worldwide in vernacular constructions methods [20]. Many studies have dealt with the stabilising effects of biopolymers of earth-based materials since the early 2010's. In 2012, Chang and Cho [21] first studied the effect of β 1,3/1,6 glucan biopolymer on the compressive strength of residual Korean soil. They show that the addition of a small amount (less than 5g/kg) of the studied biopolymer is able to triple the compressive strength while the addition of 10% of cement only leads to double the mechanical strength. The β 1,3/1,6 glucan biopolymer induces the formation of crosslinked hydrogen bonding with the clay particles that allows for the soil strengthening. It is worth noting that, according to the authors, this stabilisation solution is less expensive and has less environmental impact than the cement stabilisation.

- Since, different biopolymers have been tested including cow-dung [22], alginate salt [23–27],
- natural gum such as guar [28] or xanthan [29,30], linseed oil [24], chitosan [31], ...
 Each type of biopolymer allows for an improvement of the material compressive strength and
- 83 can present additional beneficial effects such as rapid strengthening (alginate soil) [26,27] and
- 84 water resistance. For instance, recent studies have shown that biopolymers such as xanthan
- gum and linseed oil are efficient to increase both compressive strength and water resistance
- 86 even at low dosage [30,32]. For example, xanthan gum is able to form a gel that can be bond
- 87 with clay particles through hydrogen and ionic bonds [28,30,33,34] while keeping earth

88 recyclable [35].

Nevertheless, even with cement or biopolymers stabilisation, earth remains a fragile mineral material that displays low tensile strength and can be subjected to shrinkage during drying and to fractures occurrence. The addition of fibres is a solution to reduce these weaknesses. Natural fibres such as hemp or flax fibres can be found in large amounts and are already valorised in order to make high performance composites materials. In Algeria, large amounts of fibres from diss and date palm tree remain unevaluated and can be used in order to reinforce earth-based building materials. Moreover, it is interesting to note that the use of extrusion process in order to make building blocks is able to enhance the reinforcement effect of natural fibres (improved cracking resistance and tensile strength) by creating an extrusion pressure-induced better interface as shown by recent studies carried out with flax fibres [36,37]. Like other modern processes including self-compacting earth developed at ETH Zurich [8,38–41], hypercompaction [42] or vibrocompaction [29], use of extrusion is also

very interesting in order to meet the lowest porosity and the highest mechanical strength [36,43].

It is also important to note that the combined addition of natural fibres and xanthan gum is efficient in order to reinforce sand for geotechnical applications [44] and can be a good way for the mechanical improvement of earthen construction material.

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This study deals with the stabilisation of extruded earth-based materials with xanthan gum and their reinforcement with diss and date palm tree fibres from Algeria to provide tensile strength and ductility. Compressive and flexural strength measurements are performed and show the effect of biopolymer and natural fibres on the mechanical properties of the material and results are supported by observations and pull-out tests. It is, to the authors knowledge, the first measurements of pull-out strength of natural fibres in a biopolymer stabilised matrix. It provides new insights on the bonding improvement between fibres and matrix, brought by the biopolymer. It is finally shown that the combined addition of xanthan gum and natural fibres (especially diss with its rough surface) is able to provide ductile properties with significantly improved mechanical strength (from 3.3 MPa for the reference sample to 4.5 MPa for the compressive strength and from 3.6 MPa to 8.4 MPa for the tensile strength. The maximum values are obtained with a Diss fibre volume content of 1.5% and a xanthan gum dosage of 1% of the earth weight ratio). It is worth noting that this study is limited to two types of natural fibres and on a specific earth. These results will need to be generalised to other earth mineralogy and particles size distribution in further studies. Moreover, the economic and environmental pertinence of the proposed solutions have to be discussed considering the local resources and context.

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2. Methods and materials

2.1 Methods

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Samples are fabricated using extrusion. This fabrication method requires some rheological requirements in terms of earth material consistency and stability [36,45]. A minimal shear yield stress is required for the extrudates handling [46]. Toutou et al., for cement-based materials, targets a shear yield stress of 20 kPa [46]. In order to check that the materials have

the required consistency, cone penetrometry has been performed using the fall cone conventionally used for the determination of the consistency limits of the soil (tip angle 30°, cone weight 80g). The ASTM standard D4318 is used for the test protocol [47]. Penetration test is a fast and sufficiently accurate method that can be used for the consistency evaluation and also for an estimation of the material's yield stress [48,49]. The penetration of the cone is performed using a controlab cone penetration device. The cone tip is placed on the sample surface (cylindrical cup, 50 mm high and 50 mm in diameter) and is then released for 5 seconds. The penetration depth is measured using a digital length measurement device which has a 0.01 mm accuracy. Preliminary tests have shown that tested materials can be extruded when the penetration depth ranges between 3 and 5 mm.

For each extrusion, a material's mass of 2.5 kg was prepared. Dry materials (dried earth, fibres, additives) are first hand-mixed together during one minute. Then, water is added and the samples is mixed using a Hobart mixer at low speed during one minute and at high speed during 4 minutes. Then, the bowl is scrapped and the material is mixed during 2 minutes at high speed.

Penetration test is then carried out and if the penetration depth ranges between 3 and 5 mm, extrusion is performed. Otherwise, a water correction is performed (step by step addition of 20 g of water). The used extruder is a VARS 50A/2-type from ECT. Screw rotational speed was 15 rpm. A vacuum system is fitted on the extruder in order to reduce porosity. The mixed earth-based material is fed into the extruder chamber (50mm inner diameter barrel) of the single screw extruder via the hopper. The cylindrical shaped extruder die extrudates with a diameter of 35 mm. Note that for unreinforced earth, cylindrical samples of the same diameter were cast in order to provide additional value of compressive strength of the samples at lower densities (no vacuum system, lower processing energy).

2.1.2 Mechanical characterization

The mechanical characterizations were performed on an Instron 4505 load machine equipped with a 100 kN load sensor (accuracy of 0.1 kN). Compressive and tensile (using 3-points bending test) strengths are tested on 35 mm diameter cylindrical samples with lengths of 35 mm and 140 mm respectively, as shown in figure 1a and 1b. Samples are loaded using a controlled displacement rate of 6 mm/min with an accuracy of 0.5%. It is an experimental test

protocol because there is no specific standard published with such geometry. For each tested extrudates composition, five samples were tested in compression and in bending. Before testing, samples are cut at the targeted length and are stored at controlled temperature of 40°C with 50% of relative humidity until mass stabilisation, which occurs after approximatively one week. Tests are carried out 14 days after the extrusion.

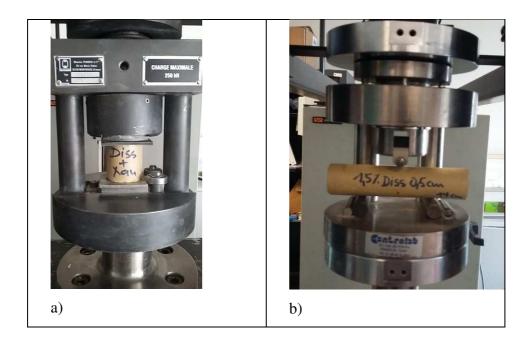


Figure 1: Settings of strength measurement tests. a) compression configuration -b) 3-point bending configuration

2.1.3 Microstructural investigations

The transversal section of the fibres was measured using a CCD camera mounted on a LEICA MZ16 trinocular magnifying glass. This device enabled to take pictures that were magnified by 10 and allows for a length measurement with an accuracy of 1 µm.

Microscopic observations are carried out using a scanning electron microscope (SEM) to characterise the morphology of Diss and Date Palm fibres. Fibres were cut transversely and longitudinally, taking care not to alter their original state. Fibres and broken samples were observed with a JEOL JSM-6460LV SEM at an acceleration voltage of 20 kV using backscattered electron signals (BES).

2.1.4 Fibres tensile strength and Pull-out tests

For the determination of the tensile strength of fibres, the used method is applicable for both single ultimate fibre or bundles of fibres as in many works related to the topic [50–53].

Studied fibres have been attached to a hollow support made of paper as shown in figure 2a. The hole has an opening of 60 mm which corresponds to the length submitted to tension. The fibres are glued with araldite at their extremities in order to avoid any slippage at the device fixation. Once prepared with their paper support, the fibres are attached on an MTS CMT4000 universal testing machine (figure 2b) used in a displacement-controlled mode at a constant velocity of 0.1 mm/min with an accuracy of 0.1%. The load sensor has a capacity of 50 N with a sensitivity of 1 N. It is an experimental test protocol because there is no specific standard published.

In order to account for the natural variability of the fibres, tensile test measurements have been carried out on 40 fibres which have been submitted to measurements of their transversal section's dimensions. This allows for the computation of both, average tensile strength and apparent Young modulus and the standard deviation of these results.





211 a) b)

Figure 2: Tensile strength measurements -a) Sample preparation with hollow support made of paper and b) tensile loading machine.

In order to investigate the quality of the bonds between stabilised earth and fibres, pull-out tests are performed. The assessment of these interfacial properties is expected to help to

understand the reinforcement mechanisms and their potential efficiency for improving the tensile strength and the ductility of the extruded earth. The sample fabrication is based on the study of Lecompte et al. [37] who have developed a device that is intended to mimic the pressure effect induced by the extrusion process. As shown in figure 3a, the device consists of two drilled PVC plates, the lower plate is the bottom mould with a 1 mm diameter hole where the fibres can pass through. The top plate, with a 5 mm diameter hole, constitutes the cell mould into which the paste can be poured and pressed. An aluminium punch is used to apply a constant load on the poured earth. The pressure of 250 kPa is applied on the sample via the punch during 5 minutes. After the sample fabrication, it is removed from the plates 48 hours later. Then the samples are stored in a similar manner than the extruded samples until mass stabilisation before the mechanical characterisation.

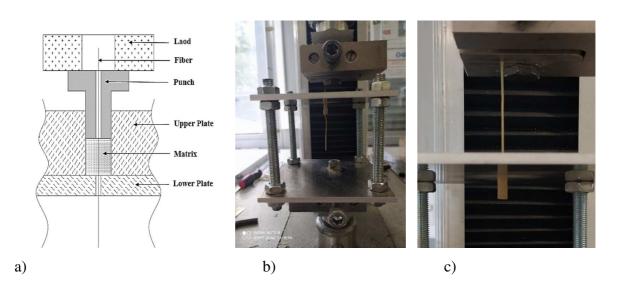


Figure 3: Pull out tests -a) sketch of the sample fabrication b) testing device c) sample mounted on the testing device.

Pull-out tests are performed using the same testing machine than the one used for the tensile strength of the fibres at a same constant controlled displacement velocity of 1 mm/s. A drilled dedicated plate is used to block the earth matrix while putting the fibre extension. It creates tension within the fibre and the shearing of the interface between the matrix and the fibre as shown in figures 3b and 3c. At least six samples were prepared for each type of fibres and matrix. During the test, the displacement and the tensile force are recorded. It is worth noting that no slippage occurred between the machine fixations and the fibre. The interfacial strength (pull-out strength) is considered to be the average interfacial shear stress which is computed

from the ratio of the maximum recorded force to the fibre section (measured using the protocol provided in section 2.1.3).

2.2 Materials

249 2.2.1 Earth

The studied raw earth originates from the north of Rennes, France. It is a fine-grained clayey soil, which displays a particle size distribution with 70% (in mass) of the particles having a diameter under 20 μ m (Figure 4). The particle size distribution is obtained through sieving and laser diffraction technique for the particle having a diameter lower than 80 μ m and the average particle size is close to 10 μ m. This earth has been used historically for cob construction and the clayey part is composed of quartz, kaolinite, illite, chlorite and vermiculite [25,32,54]. The earth plasticity index is 16.5 with a liquid limit of 46.2% and a plastic limit of 29.7. The water content at the optimum proctor is 20.6% and the methylene blue value of the soil is 1.03.

2.2.2 Natural fibres

In the frame of this study, natural fibres are used to reinforce earth-based materials by providing them ductility and tensile strength. Diss and palm date tree fibres are tested in this work.

Diss (Ampelodesmos mauritanicus, Poaceae) is an herbaceous plant of the Gramineae family with an average height of around one meter and containing extremely robust and rough fibres. It is widespread over southern Africa, western Asia, south-western North America and throughout the Mediterranean basin. Diss is abundantly available throughout the year [55,56]. Diss fibres were historically vaporized as ropes, hats, bags and in construction of diss fibres reinforced renders and hut roofs are reported taking advantages and the good mechanical and thermal insulation properties. Recently, diss ability to reinforce matrix has been assessed in order to develop cementitious or plastic composite materials [57–60]. The specific mass of the diss is 1250 kg/m³. In this study, the used fibres were collected in the region of Annaba (north-eastern Algeria). According to the study of Bourahli et al [61], diss

fibres contain about 44% of cellulose, 27% of hemi-cellulose, 17% of lignin, 9% of extractives and 3% of ashes.

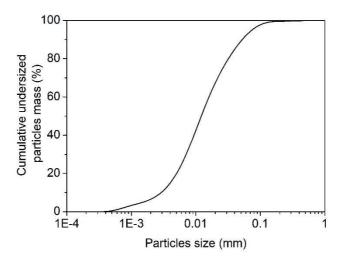
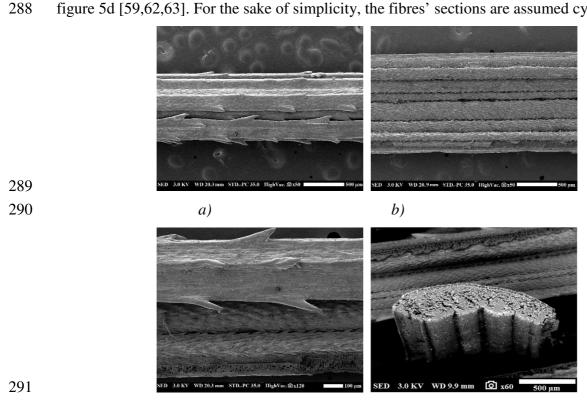


Figure 4: Particle size distribution of the studied earth

Fibres are cut in 5, 10 or 15 mm long pieces and present an average diameter of 0.94 mm (measured using a trinocular magnifying glass). SEM observations of the Diss fibres (figure 5) show that outer surface of the fibres presents $100 \mu m$ long thorns, five times longer than the inner ones (figure 5a to c). Thorns are distributed homogeneously all along the inner surface. The fibres wall present a mixed tubular - cavernous structure with closed cavities showing that they do not transport sap as shown by the cross-sectional view displayed in figure 5d [59,62,63]. For the sake of simplicity, the fibres' sections are assumed cylindrical.



292 d) c)293 Figure 5: SEM observation of Diss fibres. a) outer surface, b) inner surface, c) mixed view of 294 inner and outer surfaces, d) cross sectional view of diss fibres. 295 296 Tensile test measurements show that Diss fibres presents an average tensile strength of 82 297 MPa (standard deviation of 26 MPa) and a Young modulus of 5.8 GPa (standard deviation of 298 1.8 GPa). 299 300 The date palm (Phoenix dactylifera) is a hardy tree with a crown of leaves (the palms) and is 301 well adapted to desert and arid regions around the world [64,65]. After date harvests, it is 302 estimated that one and a half million tonnes per year (1.5 106 t/year) of palms in the world 303 remain unevaluated and become waste [51,66]. These residues are one of the renewable 304 sources which can be recovered in the construction sector and even in the reinforcement of 305 composite materials [64,67–71]. This study focuses only on fibres extracted from palm 306 leaflets and produced using a Retsch-type grinder. Fibres are cut in 5, 10 and 15 mm long 307 pieces and present a flat-shaped cross-sectional area with an average width of 1.41 mm and 308 their specific mass is 1350 kg.m³. The tested date palm tree fibres originates from Biskra area 309 (Algeria). According to the review of Jonoobi et al., the leaflets fibres contain between 4 and 310 8% of extractives, 40 and 54% of cellulose, 15 and 32% of lignin and 13 and 20% of hemi-311 cellulose [72]. 312 313 The SEM morphological study shows that the date palm fibre has a heterogeneous structure 314 and has a flat shape with rigid grooves [52,63,73]. The fibre structure is porous as shown in 315 figure 6. The cross-section of a leaflet shows that it consists of bundles of ultimate fibres 316 (fibril assembly) and grooves composed of conductive libero-linear vessels. The diameter of 317 the ultimate fibres is of the order of 10 µm and the lumen has a diameter of 1 to 2 µm 318 [63,74,75]. 319 320 Tensile tests measurement shows that palm date tree fibres presents an average tensile 321 strength of 164 MPa (standard deviation of 25 MPa) and a Young modulus of 8.7 GPa 322 (standard deviation of 1.8 GPa). 323

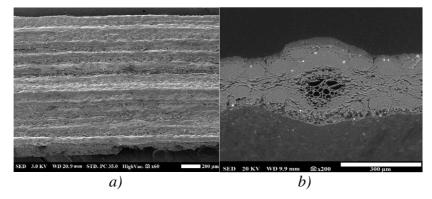


Figure 6: SEM observation of palm date tree fibres. a) longitudinal view – b) cross-sectional view.

Water absorption of fibres was measured for diss and date palm tree fibres at 5 minutes and 24 h using the protocol described in Lecompte et al. [37]. At 5 minutes, the water absorption coefficient is 70% for the diss while it is only 65% for the date palm tree fibres. At 24 hours, these values increase at 140% for the diss and 130% for the date palm trees fibres.

2.2.3 Additives

In the frame of this study, xanthan gum has been used as an efficient earth-based materials stabiliser [24,32]. Xanthan gum is a-highly stable branched heteropolysaccharide produced by fermentation of sugars by the "Xanthomonas campestris" bacterium. This type of gum is mostly used as a consistency modifier in cosmetics or food industry (Additive E415). Recent studies showed that xanthan gum (Xan) could also increase strongly the mechanical properties of soils and earthen materials. These long macromolecules are likely to adsorb onto the surface of clay particles. Xanthan gum increases the viscosity of the freshly mixed wet material [34] and improves the water resistance of earth-based material because of the formation of a gel within the clay particles porosity [76,77]. Two xanthan gum's dosages (provided by "wake-up-easy sportfood) have been tested: 0.5% and 1% of the mass of the dry earth as proposed by Guihéneuf et al. [32].

In order to compensate for the thickening effect of the xanthan gum, sodium hexametaphosphate was used as a fluidity agent in order to limit the initial water content of the earth material. Hexametaphosphate (HMP) when added at a small concentration, can be an efficient dispersant for clay particles [8,26,38,48]. The dosage chosen here is 0.3% in mass of the dry earth material mass.

2.2.4 Mix design of materials

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356 In order to meet the extrusion's consistency requirements for these earth-based materials, a 357 water content between 23 and 27% is needed for the earth without fibres and additives. 358 Additional mixes at close water contents 22 and 28% are cast in order to obtain additional 359 samples with lower densities. In the first part of the results section, the influence of additions 360 of natural fibres are tested on unstabilised earth. Two dosages of 1.5% and 3% volume 361 content are tested for 3 different fibre lengths of 5, 10 and 15 mm. This first study allows for 362 assessing the best dosage and length of fibres; i.e. fibres dosage and length that provide good 363 reinforcement with sufficient workability for the extrusion process. 364 The same materials were tested with additions of dosages of Xanthan gum of 0.5%; 1% and 365 2% and a dosage of HMP of 0.3%. A referencing system was chosen for the mix-design 366 denomination. A series of 5 letters, each followed by a number is used in order to identify the 367 samples mix-design. The first letter W indicates the water content (between 23 and 28), the 368 second and third letter L and C refers to the fibres length (in mm) and content (volume ratio) 369 and the fourth and fifth letters H and X refers to the stabilisation strategy. H is linked to the 370 HMP addition and can be followed by 0 (no HMP) and 1 (HMP added). X is the dosages of 371 xanthan gum and ranges between 0 and 2%. For example, the mix-design W25L10C3H1X1 372 has a water content of 25%, addition of 10 mm long fibres at a volume content of 3% and 373 contains 0.3% of HMP and 1% of xanthan gum (weight ratio). When natural fibres are added, 374 the name of the mix-design is followed by the fibre type (diss or date palm). 375 In the last part of the results section, combined additions of natural fibres and stabilising 376

additions are tested for the best fibre length and content. It allows studying the combined effect of natural fibres and studied additives additions.

A summary of all tested mix-design is provided in table 1. A water content of 25% allows to obtain required penetration depth for all tested fibres addition.

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Table 1: Summary of tested mix design. The table provided the water content W in function of the added fibres (length L and content C) and the stabilization strategy (addition of HMP H and xanthan gum dosage X). H0X0 means that the mix doesn't contain stabiliser and L0C0 that the mix doesn't contain any fibre.

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| | H0X0 | | H1X0 | | H1X0 | 5 | H1X1 | | H1X2 | |
|---------|------|-----|------|-----|------|-----|------|-----|------|-----|
| L0C0 | W22 | W23 | W22 | W23 | W23 | W24 | W23 | W24 | W22 | W23 |
| | W24 | W25 | W24 | W25 | W25 | | W25 | | W24 | W25 |
| | W26 | W27 | W26 | | W26 | | | | W26 | |
| | W28 | | | | | | | | | |
| L5C1.5 | W25 | | - | | - | | - | | - | |
| L10C1.5 | W25 | | W23 | | - | | W23 | | - | |
| L15C1.5 | W25 | | - | | - | | - | | - | |
| L5C3 | W25 | | - | | - | | - | | - | |
| L10C3 | W25 | | - | | - | | - | | - | |
| L15C3 | W25 | | - | | - | | - | | - | |

3. Results and discussions

3.1 Effects of fibres addition

3.1.1 Compressive strength

The compressive strength of bricks made of earth-based material has to reach a minimal value given by local standards. For instance, the French Standard XP P 13-901 [78] defines three classes of bricks depending on their compressive strength at 2, 4 and 6 MPa. The German standard also defines classes of bricks up to 6 MPa [79].

It has been shown that the compressive strength of a specific earth-based material is governed by its density [25,29,32,39,42]. In this work, the obtained results confirm this statement: as shown in figure 7, which displays the evolution of the compressive strength of the earth-based samples in function of their densities, it can be seen that all results follow the same trend whatever the type or dosage of fibres. For unreinforced sample (L0C0), the density variation is linked to the initial water content of the earth.

It is also observed from figure 7 that the addition of fibres induces a decrease of the sample dry density. Natural fibres are less dense that mineral materials and it is observed that the sample density decreases with their fibres content.

Moreover, for a same dosage, the density decreases with the fibre length. A decrease of the workability of cement-based materials was already reported in the literature with fibre

addition. Martinie et al. showed that this workability loss increases with the fibre aspect ratio (with the fibre length at a constant fibre diameter [80–82]). The effect of fibre length observed in this study is in line with the results of Martinie et al.

It is worth noting that for a same dosage and length, the reduction in dry density is higher for the diss fibres than for the date palm fibres. This result can be explained by the fact that date palm fibres exhibit less flexural rigidity than the diss fibres due to their flat shape. This lower rigidity allows for higher fibre deformation that alters less the material rigidity [83].

It can be interesting to find solutions that allows reaching higher dry density for fibre reinforced earth-based materials. The ability of xanthan gum and HMP to allow for a more efficient densification of the earth-based matrix will be tested later in this work.

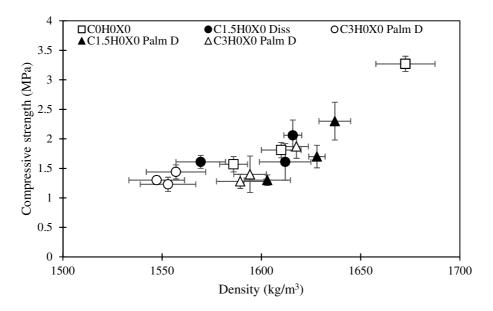


Figure 7: Evolution of the compressive strength of fibre reinforced earth-based materials as a function of sample dry density for different dosage of fibre type (Diss and Date palm), dosage and length.

3.1.2 Tensile strength

Figure 8 plots the evolution of flexural strength as a function of the dry density of the earth-based samples for different types and dosages of fibres. As observed for the compressive strength, the addition of fibres decreases the dry density of the samples. However, for a given dry density of a sample, it is observed that material reinforced with diss fibre presents the highest flexural strength, followed by the date palm fibre reinforced samples. Unreinforced

samples exhibit the lowest flexural strength. These observations confirm the reinforcing effect of natural fibres that increases earth-based material flexural strength [36]. It is interesting to note that diss fibres display a more efficient reinforcing effect than the date palm tree fibres. This enhanced effect is attributed to the diss fibre surface that presents thorns as shown in SEM observations displayed in figure 5 and is supported by the highest pull-out strength measured for diss fibres: the average interfacial shear strength measured for the diss is equal to 242 kPa while it is only of 150 kPa for date palm fibres.

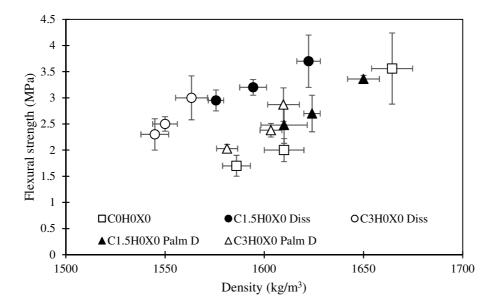


Figure 8: Evolution of the flexural strength of fibre reinforced earth-based materials as a function of sample dry density for different dosage of fibre type (Diss and Date palm), dosage and length.

The decrease of the dry density affects the mechanical strength and can be attributed to both low density of the fibres and workability loss. As shown in figure 9 and as predicted by recent literature of the rheology of fibre-reinforced suspensions, the effect of fibre addition can be described by a spatial congestion parameter, which is the product of the fibre dosage, by the fibre aspect ratio (fibre length L to square root of fibre cross-section $A^{0.5}$) and can be written $C/(L/A^{0.5})$ [80–83]. For a given type of fibres, flexural strength of the samples versus congestion parameter follows a same trend. It can also be seen that for a given value of spatial congestion parameter, the flexural strength is higher for diss fibre than for date palm tree fibre. This observation is in line with conclusions stated from figure 8.

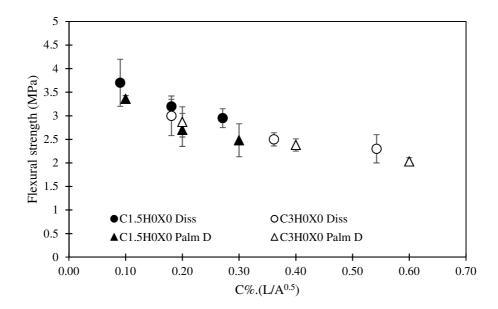
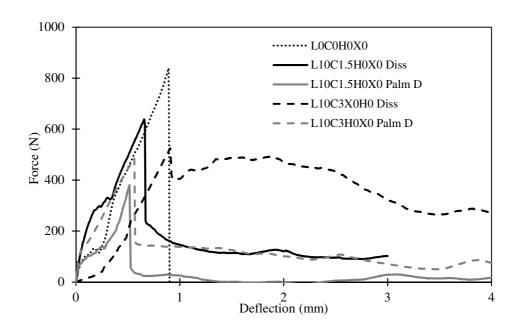


Figure 9: Evolution of the flexural strength of fibre reinforced earth-based materials as a function of the spatial congestion parameter for different dosage of fibre type (Diss and Date palm), dosage and length.

3.1.3 Ductility

Flexural test data shows that unreinforced earth samples display a fragile behaviour and after the force peak, the recorded force directly falls to 0, as shown in figure 10.



468 Figure 10: Flexural strength measurements test for fibre-reinforced earth-based materials.

Force vs. deflection tests for different fibre dosage (1.5% and 3%) and fibre type (diss and

470 date palm tree).

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However, when fibres are added, a post-peak behaviour is observed with a residual force value that increases with the fibre content. It is also worth mentioning that the residual force is higher for the diss fibre-reinforced samples, probably due to the surface morphological characteristics of these fibres. Such ductile behaviour properties can be very useful to prevent dramatic collapse of structures made of earth.

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3.2 Effects of HMP and Xanthan gum

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The figure 11 shows the effect of HMP and xanthan gum on the compressive strength of unreinforced extruded earth-based samples. Many conclusions can be drawn from the observation of this figure but we first have to take into consideration that it was not possible to extrude earth sample with only xanthan gum and no HMP: the material appeared either too viscous or too dry to be extruded at various water contents. Earth with only xanthan displays a granular-like behaviour under a water content of 28%. Above this value of water content, the material becomes pasty and shows a cone penetration between 3 and 5 mm but the material viscosity is too high to allow for the material extrusion. It seems that the water demand of xanthan gum is high and has to be compensate using a dispersing agent such as HMP. Addition of HMP allows increasing the earth sample dry density leading to higher compressive strength. HMP changes the organization of the clay particles at the microscale as demonstrated by the SEM observations displayed in figure 12. It can be seen that the addition of HMP changes the particles network from a house-of-cards network to a more dispersed network where all clay platelets particles are parallel and superimposed. This observation is consistent with common results observed for kaolinite clay particles [48,84]. This change in particles' assembly leads to a decrease in the materials shear yield stress at the fresh state [8,38,85] and an increase in the dry state compressive strength [25].

Xanthan gum is known as an efficient stabiliser for earth-based material in order to increase both compressive strength and water resistance [32,86,87]. In this study, it can be seen that a combined used of HMP and xanthan gum allows obtaining materials with increased compressive strength and similar dry densities than for the unstabilised and reference earth samples. Observation= of figure 11 seems to indicate that the optimal xanthan gum dosage is

1% as this xanthan gum content provides the highest compressive strength. This result seems to be confirmed by the SEM observation of samples stabilised with 1% and 2% of xanthan gum along with HMP depicted in figure 13a and 13b respectively. It can be seen on these observations that a 1% dosage provides a homogeneous mix of clay particles covered and bonded by xanthan gum gel but that with a 2% dosage, a heterogeneous mix of uncovered clay particles and localized xanthan gum gel is encountered.

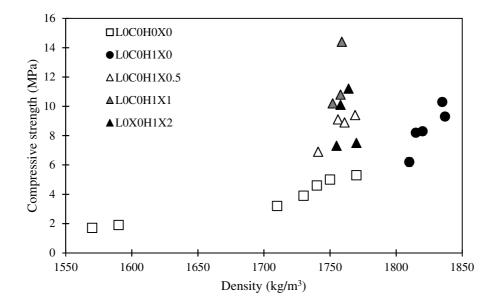


Figure 11: Compressive strength versus dry density for stabilised unreinforced earth-based materials containing HMP and HMP + varying dosage of xanthan gum.

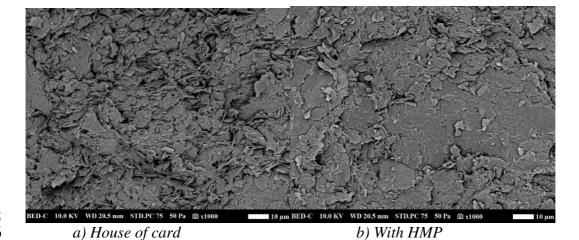
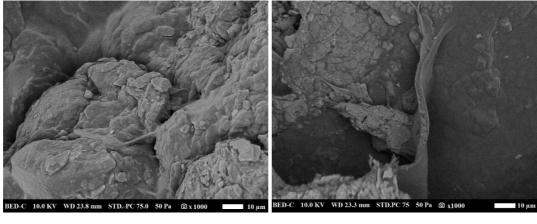


Figure 12: SEM observation of the microstructural organisation of clay particles without and with HMP.

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520 a) Xan 1% b) Xan 2%

Figure 13: SEM observation of the microstructural organisation of clay particles with HMP and 1% and 2% xanthan gum.

3.3 Combined effects of stabilisers and fibres

3.3.1 Compressive strength

In this section, the effect of a combined addition of fibres (10 mm long at 1.5% dosage) and stabiliser (HMP and xanthan gum) on the material's mechanical properties is investigated. Materials are compared to reference materials without any fibres and stabilisers and with materials with fibres but without stabiliser. This choice of length and dosage is justified because these parameters allow for the best compromise between reinforcing ability and extrudability (limited workability loss).

In figure 14, the compressive strength of the samples is plotted as a function of the dry density of the samples. It is worth noting that addition of stabiliser allows increasing both compressive strength and dry density of the earthen materials. This effect is even more pronounced when xanthan gum is added along with HMP.

It is worth noting that for a given stabilisation strategy, diss fibres reinforced samples provide slightly higher compressive strength than samples reinforced with date palm tree fibres. This can also be attributed to the morphology of the surface of the fibres that exhibits thorns.

The simultaneous addition of stabiliser along with natural fibres seems to compensate for the workability loss induced by the natural fibres' presence. It allows reaching relatively high density (higher than the reference mixes, unreinforced and unstabilised) with materials having high compressive strength.

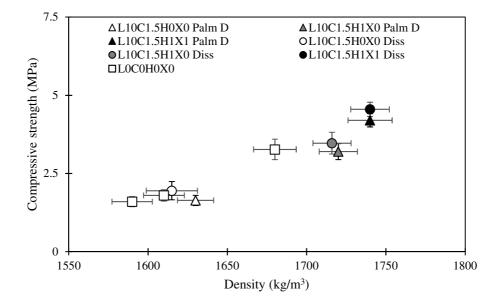


Figure 14: Compressive strength of earth-based materials as a function of sample's dry density for earth-based materials reinforced with natural fibres (10 mm long at 1.5%) and stabilised with HMP and HMP+Xanthan gum. For the sake of comparison, data obtained from reference and unstabilised samples are plotted.

3.3.2 Tensile strength

Combined additions of natural fibres and stabilisers induce an increase in both tensile strength (measured under bending) and dry density. As shown in figure 15, contrarily to unstabilised materials, the fibres addition does not seem to weaken the earth-based materials. The simultaneous addition of stabiliser with natural fibres seems to compensate for the workability loss induced by the natural fibres' presence. It allows reaching relatively high density (higher than the reference mixes, unreinforced and unstabilised) with materials having high tensile strength.

It is worth noticing that for a given stabilisation strategy, diss fibres reinforced samples provide slightly higher tensile strengths than samples reinforced with date palm tree fibres. Also, all reinforced and stabilised samples show higher tensile performance than the reference mixes while it is not the case without stabiliser (figure 8).

The only addition of HMP seems to be sufficient to improve the materials extrudability and allows for an efficient reinforcement of the earth-based material but the combination of HMP and xanthan gum as stabiliser provides the best results. SEM observation (figure 16) confirms the role of xanthan gum in the bonding between earth matrix and reinforcing fibres as a film

of xanthan gum can be observed on the surface of both diss and date palm tree fibres. This film is expected to enhance the interfacial strength between clay matrix and natural fibres.

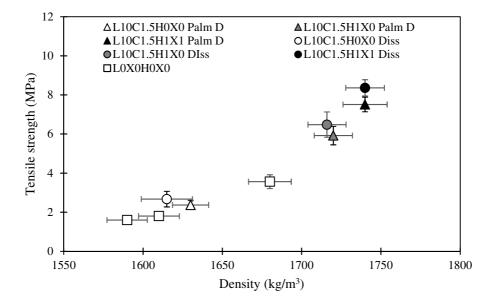


Figure 15: Flexural strength of earth-based materials as a function of sample's dry density for earth based materials reinforced with natural fibres (10 mm long at 1.5%) and stabilised with HMP and HMP+Xanthan gum. For the sake of comparison, data obtained from reference and unstabilised samples are plotted.

Moreover, it is interesting to note that measured pull-out strengths provided in table 2 (called interfacial shear strength IFSS) are in line with improvement in tensile properties measured using 3-point bending tests. Highest IFSS values are obtained with HMP and xanthan gum stabilised mixes and then for HMP stabilised samples. For each type of stabilisation strategy, diss fibres shows the most resistant interface because of its irregular surface with thorns.

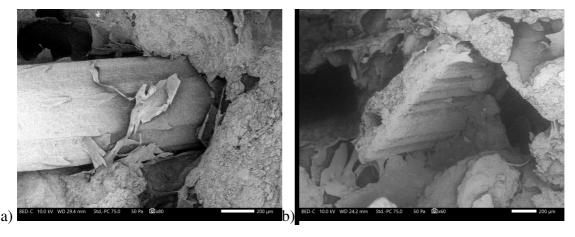


Figure 16: SEM observation of fractures samples of earth-based materials reinforced with natural fibres (10 mm long at 1.5%) and stabilised with HMP+Xanthan gum. a) diss reinforced sample - b) date palm tree reinforced sample.

Table 2: Pull-out test results – average interfacial shear stress and standard deviation.

| | | Diss | | Date palm | | | |
|--------------------------------|------|------|------|-----------|------|------|--|
| | H0X0 | H1X0 | H1X1 | H0X0 | H1X0 | H1X1 | |
| IFSS (kPa) | 242 | 269 | 368 | 150 | 237 | 340 | |
| Standard deviation (kPa) | 42 | 48 | 45 | 16 | 22 | 34 | |

3.3.3 Ductility

Figure 17 shows that during bending, diss fibre reinforced behaviour exhibit a post peak behaviour with a residual force value that increases when HMP or HMP and xanthan gum is added. On the contrary, figure 18 shows that even with the addition of HMP or xanthan gum, the residual force after the peak remains low for date palm tree fibre reinforced samples. This results clearly show the role of the fibre morphology on the stress transfer and the overall ductile behaviour of the earth composites. Diss fibres present surface roughness that allows to obtain a residual stress after the peak.

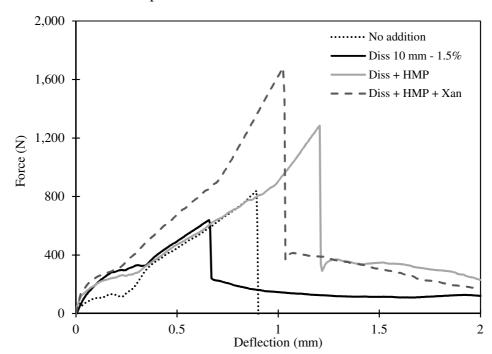


Figure 17: Bending force as a function of sample's deflection for earth-based materials reference sample, reinforced with diss fibres (1.5%, 10 mm long), reinforced with diss fibres and stabilised with HMP and reinforced with diss fibres and stabilised with HMP+xanthan gum.

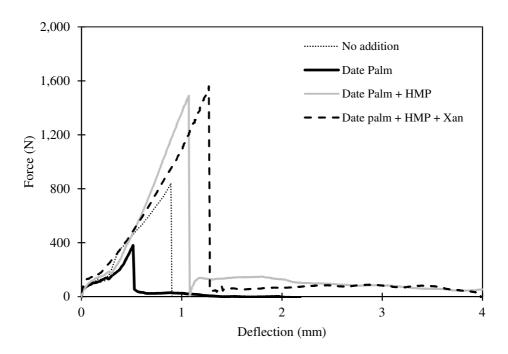


Figure 18: Bending force as function of sample's deflection for earth-based materials reference sample, reinforced with date palm tree fibres (1.5%, 10 mm long), reinforced with date palm tree fibres and stabilised with HMP and reinforced with date palm tree fibres and stabilised with HMP+xanthan gum.

This result is confirmed by the pull-out test data. Pull-out of diss fibres provides higher residual stress after the debonding peak in comparison with pull-out of data palm tree fibres whatever the stabilisation strategy as shown by figures 19 and 20. It is also worth mentioning, as shown in table 1, that the maximum pull-out force is higher for diss than for date palm tree whatever if the samples is stabilised or not. Moreover, the residual force is kept for longer pull-out displacement showing the prominent role of the diss fibre morphology on the stress transfer between natural fibre and matrix.

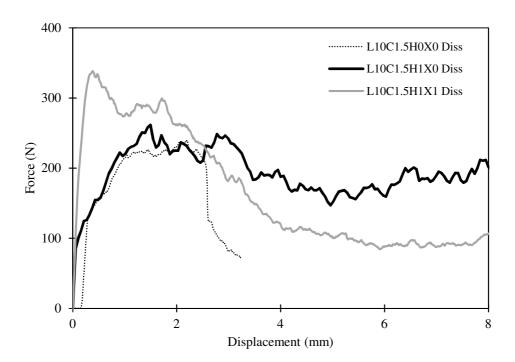


Figure 19: Pull-out force for diss fibres in unstabilised matrix, matrix stabilised with HMP and matrix stabilised with HMP+xanthan gum.

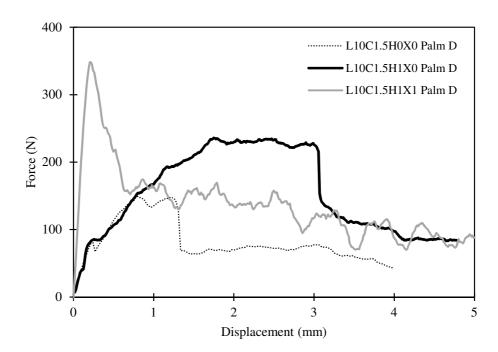


Figure 20: Pull-out force for date palm tree fibres in unstabilised matrix, matrix stabilised with HMP and matrix stabilised with HMP+xanthan gum.

4. Conclusions

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632 This paper highlights the beneficial effect of simultaneous natural fibres additions and earth 633 stabilisation by biopolymer (by adding HMP and xanthan gum) in order to obtain a structural 634 material displaying better compressive strength, good tensile strength and a ductile behaviour. 635 It is first shown that the addition of natural fibres alone is detrimental for the extrusion and 636 leads to less dense material with lower compressive strength ranging from 1.2 to 2.3 MPa 637 (instead of 3.3 MPa for the unreinforced sample) and similar tensile strength (around 3 MPa), 638 even if reinforced materials have ductile properties. This is attributed to a problem of 639 workability loss induced by the fibres. It is shown that the workability loss is induced by the 640 spatial congestion of the fibres network. 641 Then, in agreement with already-known results, it is shown that the stabilisation of the studied 642 earth with Na-HMP (0.25% of the earth weight) and xanthan gum leads to improve both 643 density and mechanical strength (up to 14 MPa at a dry density of 1759 kg/m³, xanthan gum 644 dosage of 1% of the dry earth weight). It is concluded that xanthan gum and HMP allow 645 compensating for the workability issue and thus allowing for higher compressive strength at a 646 given dry density. 647 Therefore, simultaneous addition of both natural fibre and stabilisation permits to obtain 648 earth-based materials with enhanced mechanical properties (in compression with a maximal 649 compressive strength of 4.5 MPa and in tension with a maximal tensile strength of 8 MPa for 650 10 mm long diss fibres at 1.5 %volume and xanthan gum at 1%weight) combined with 651 improved ductile properties. This synergic effect is even more obvious for diss fibres due to 652 the presence of a rough surface displaying thorns that allows for an efficient stress transfer 653 between the fibres and the matrix (as shown by pull-out tests). 654

- 655 Author Contributions: Conceptualization, M. B., A. P.; methodology, A. P, S. G; .;
- 656 validation, M. B., A. P.; formal analysis, S. G., A.P., M. B.; investigation, M. B..; resources,
- 657 A. Pe.; writing—original draft preparation, A. P., M. B.; writing—review and editing, S.G.,
- 658 R. B. supervision, R. B., A. P; project administration, A.P., R. B.. All authors have read and
- 659 agreed to the published version of the manuscript.

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