

Microstructure and mechanical performance of modified mortar using hemp fibres and carbon nanotubes

Rabah Hamzaoui

R. Hamzaoui, S. Guessasma, B. Mecheri, A. M. Eshtiaghi, Abdelkrim Bennabi, “Microstructure and mechanical performance of modified mortar using hemp fibres and carbon nanotubes », Materials and Design 56 (2014) 60–68.

**ESTP 28, avenue du Président Wilson- 94234
Cachan, France**

Presentation plan

I- Introduction
Generalities

II- Materials and Methods
and
III-Results and discussions

IV- Conclusion and Perspectives



Introuction
of nanomaterials
in Construction Materials

I- INTRODUCTION (Generalities)

Nanomaterials



Nanomaterials (nanostructured materials) = nano-size $< 100 \text{ nm}$ + specific properties [1,2]



Processes

Rapid solidification, PVD , CVD, crystallization of amorphous, Inert gaz condensation, electrodeposition, mechanical alloying ...[3]



Properties

Mechanical performance, magnetic properties, hardness, physicochemical properties..., [4-7]



Contribution

Obtaining smart materials

[1] E. Gaffet, G. Le Caër, *Encyclopedia of Nanoscience and Nanotechnology*, 1-39 (2004)

[2] C. Suryanarayana, , *Progress in Materials Science* 46 (2001) 1±184

[3] M. S. El-Eskandarany, *Mechanical alloying for fabrication of advanced engineering materials*, William Andrew Publishing, Norwich, New York, U.S.A., (2001)

[4] **R. Hamzaoui**, O. Elkedim, N. Fenineche, E. Gaffet and J. Craven, “*Structure and magnetic properties of nanocrystalline mechanically alloyed Fe-10%Ni and Fe-20%Ni*” *Materials Sciences and Engineering A* 360 (2003) 299-305

[5] Tria, O. Elkedim, **R. Hamzaoui**, X. Guo, F. Bernard, N. Millot and O. Rapaud “*Deposition and characterization of cold sprayed nanocrystalline NiTi*” *Powder Technology* Vol210, 181-188 (2011)

[6]] L.W. Huang , O. Elkedim , **R. Hamzaoui** “First principles investigation of the substitutional doping of Mn in Mg_2Ni phase and the electronic structure of Mg_3MnNi_2 phase”, *J. Alloy. Comp.*, V 509, 328-333(2011)

[7] **R. Hamzaoui** , O. Elkedim, Magnetic properties of nanocrystalline Fe-10%Ni alloy obtained by planetary ball mills *J. Alloy. Comp.*, 573, 157-162, 2013

I- INTRODUCTION (Generalities)

Mix proportion of the specimens

Mixture no	W/b	Mix proportion of the specimens (per 768 cm ³)						
		Water (ml)	Cement (g)	Sand (g)	Nano-SiO ₂ (g)	Nano-Fe ₂ O ₃ (g)	UNF (g)	Silica fume (g)
A	0.5	225	450.0	1350	–	–	–	–
B ₁	0.5	225	436.5	1350	–	13.5	3.4	–
B ₂	0.5	225	427.5	1350	–	22.5	6.5	–
B ₃	0.5	225	405.0	1350	–	45.0	11.2	–
C ₁	0.5	225	436.5	1350	13.5	–	6.8	–
C ₂	0.5	225	427.5	1350	22.5	–	11.2	–
C ₃	0.5	225	405.0	1350	45.0	–	22.5	–
D	0.5	225	427.5	1350	9.0	13.5	7.9	–
E ₁	0.5	225	427.5	1350	–	9.0	3.0	13.5
E ₂	0.5	225	405.0	1350	–	18.0	6.0	27.0
F	0.5	225	382.5	1350	–	–	3.4	67.5

Compressive strength of mixtures

Mixture no	Compressive strength at the 7th day		Compressive strength at the 28th day	
	Target (MPa)	Enhanced extent (%)	Target (MPa)	Enhanced extent (%)
A	17.6	0	28.9 (29.7)	0
B ₁	21.4	22.7	36.4	26.0
B ₂	20.6	16.7	33.1	14.5
B ₃	21.1	20.0	30.0	3.7
C ₁	18.6	5.7	32.9 (33.4)	13.8 (12.6)
C ₂	21.3	20.1	33.8 (34.1)	17.0 (14.7)
C ₃	21.3	20.1	36.4	26.0
D	22.4	27.0	35.4 (34.8)	22.0 (17.1)
E ₁	19.4	10.0	29.8	3.0
E ₂	23.2	32.0	34.3	18.6
F	18.9	7.4	31.8	10.0

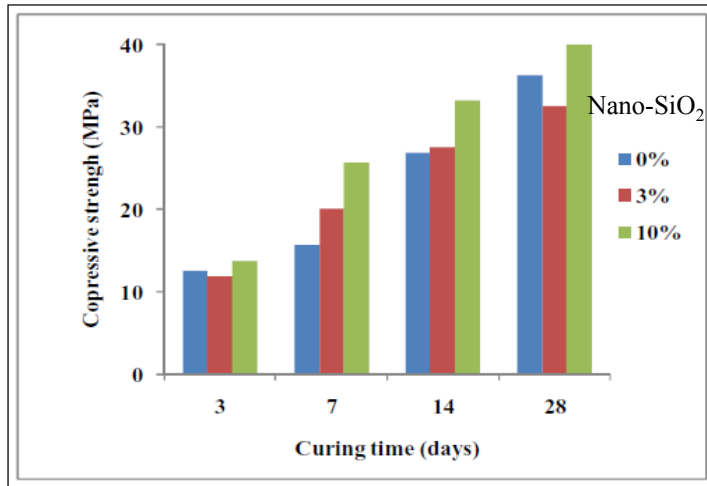


Fig: Relationships between compressive strengths and curing time of mortars [3]

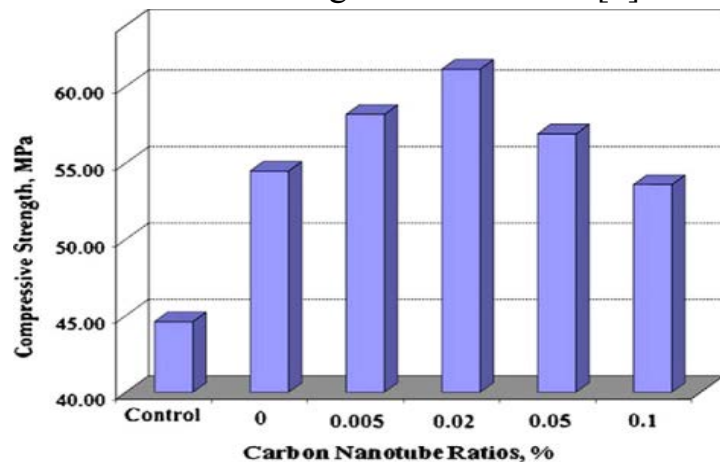


Fig Compressive strength of blended cement mortar containing exfoliated 6% NMK versus CNTs ratios at 28 days of hydration [4]

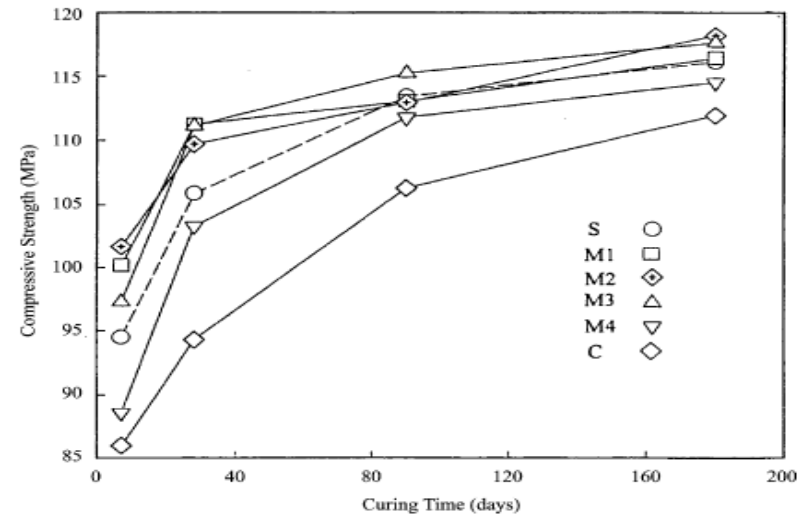


Fig Compressive strength versus curing time in Mortar : C : control, S Silica fume, (M1, M2, M3, M4) different % of MK [1].

Researchers	Nanocomposites	Amount of CNTs (wt.% of cement)	Flexural strength increase (%)
Li (2005) [8]	Mortars	0.50	25
Cwirzen (2008) [12]	Cement paste	0.042	10
Konsta-Gdoutos et al. [present work]	Cement paste	0.08 and 0.048	25

Tab: Comparison of flexural strength increase in CM reinforced with CNTs [2].

- [1] B.B. Sabir et al, Cement and Concrete Research 23 (2001) 441-454
- [2] M.S. Konsta-Gdoutos et al. / Cement and Concrete Research 40 (2010) 1052–1059
- [3] M. Ltfi et al, Procedia Engineering 10 (2011) 900–905
- [4] M.S. Morsy et al, Construction and Building Materials 25 (2011) 145–149

HEMP FIBERS AND MECHANICAL PROPERTIES

I- INTRODUCTION (Generalities)

Mix code series	Aggregate size (mm)	Fibre content (%)	Fibre length (mm)	Fibre factors
20R000L00	20	0	0	0.00
14R000L00	14	0	0	0.00
07R000L00	07	0	0	0.00
20W018L10	20	0.18	10	0.78
14W018L10	14	0.18	10	0.78
07W018L10	07	0.18	10	0.78
20W018L20	20	0.18	20	1.57
14W018L20	14	0.18	20	1.57
07W018L20	07	0.18	20	1.57
20W036L20	20	0.36	20	3.13
14W036L20	14	0.36	20	3.13
07W036L20	07	0.36	20	3.13
20W060L20	20	0.60	20	5.22
14W060L20	14	0.60	20	5.22
07W060L20	07	0.60	20	5.22
20W106L30	20	1.06	30	13.83
14W084L30	14	0.84	30	10.96
07W084L30	07	0.84	30	10.96
20D036L10	20	0.36	10	1.57
20D054L20	20	0.54	20	4.70
20D072L30	20	0.72	30	9.39
14D036L20	14	0.36	20	3.13
14D054L30	14	0.54	30	7.04
14D072L10	14	0.72	10	3.13
07D036L30	7	0.36	30	4.70
07D054L10	7	0.54	10	2.35
07D072L20	7	0.72	20	6.26
20D060L10	20	0.60	10	2.61
14D060L10	14	0.60	10	2.61
07D060L10	7	0.60	10	2.61

Tab: Experiment details (W: wet fibers, D: dry fibers)

Mix code series	Compressive stress (MPa)	Flexural stress (MPa)
20R000L00	30.81 ± 0.35	4.77 ± 0.26
14R000L00	33.45 ± 2.45	5.09 ± 0.09
07R000L00	30.57 ± 2.91	5.08 ± 0.24
20W018L10	23.82 ± 0.23	4.52 ± 0.24
14W018L10	35.22 ± 1.06	4.78 ± 0.12
07W018L10	32.73 ± 5.89	5.04 ± 0.36
20W018L20	32.65 ± 2.09	4.62 ± 0.50
14W018L20	34.30 ± 3.55	5.10 ± 0.36
07W018L20	24.70 ± 2.80	4.69 ± 0.20
20W036L20	32.09 ± 0.82	5.18 ± 0.24
14W036L20	26.41 ± 2.44	4.96 ± 0.25
07W036L20	24.23 ± 3.40	4.56 ± 0.32
20W060L20	26.76 ± 0.29	4.11 ± 0.24
14W060L20	25.52 ± 1.93	4.41 ± 0.22
07W060L20	20.08 ± 3.71	4.49 ± 0.13
20W106L30	13.88 ± 0.44	3.10 ± 0.08
14W084L30	21.73 ± 1.18	4.07 ± 0.14
07W084L30	20.11 ± 0.58	4.10 ± 0.05
20D036L10	30.81 ± 0.35	4.77 ± 0.26
20D054L20	33.45 ± 2.45	5.09 ± 0.09
20D072L30	30.57 ± 2.91	5.08 ± 0.24
14D036L20	25.41 ± 1.58	4.43 ± 0.04
14D054L30	25.31 ± 0.65	4.20 ± 0.16
14D072L10	20.72 ± 1.20	3.72 ± 0.15
07D036L30	25.93 ± 2.11	4.59 ± 0.13
07D054L10	23.69 ± 1.42	3.88 ± 0.04
07D072L20	18.49 ± 1.20	3.71 ± 0.11
20D060L10	22.94 ± 2.11	4.02 ± 0.22
14D060L10	21.59 ± 0.85	4.31 ± 0.25
07D060L10	15.40 ± 1.12	4.69 ± 0.27

Tab: Mechanical and physical properties of HFRC

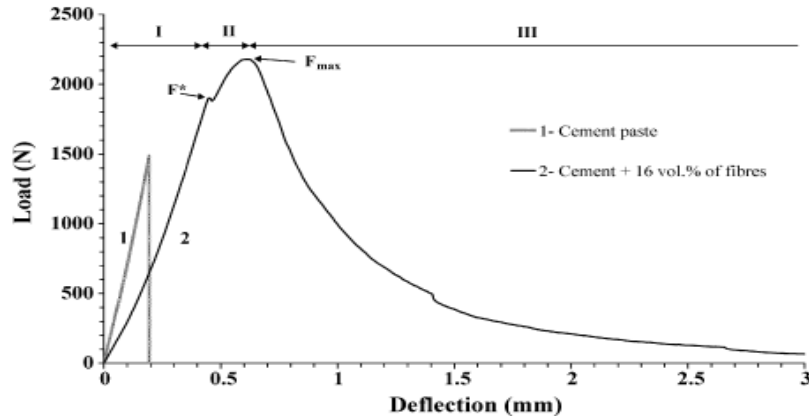


Fig. Example of flexural load–deflection curves for cement paste sample (1) and hemp cement based composite (2) [1]

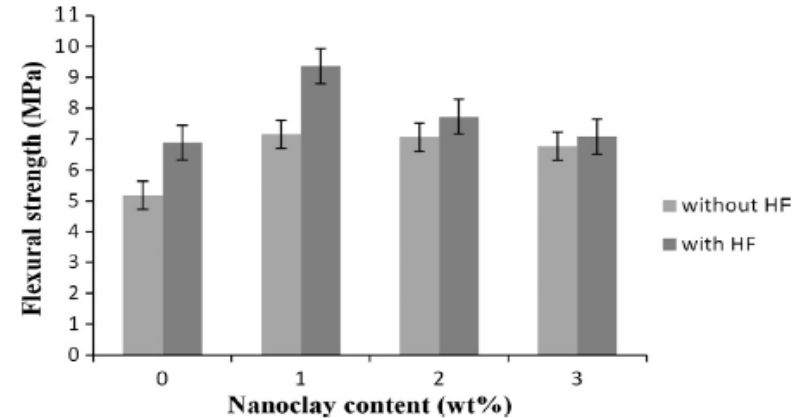


Fig. Flexural strength as a function of nanoclay content for control cement paste and its nanocomposites with and without HF [2]

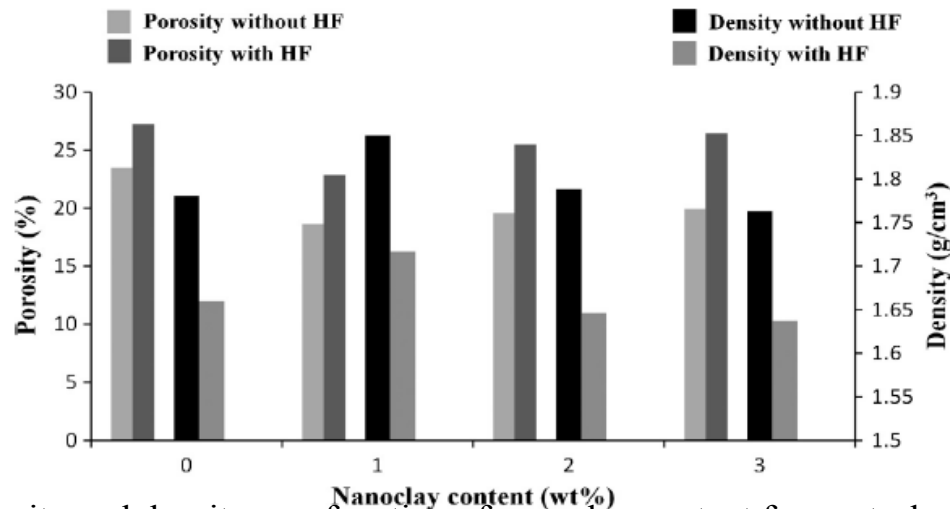


Fig. Porosity and density as a function of nanoclay content for control cement and its nanocomposites with and without fibers [2]

I- INTRODUCTION (Generalities)

PROBLEM

Hemp fibers in Mortar or Concrete
Concerning mechanical properties

COMPRESSIVE STRENGTH

PROPOSITION

NANOMATERIALS

In our case carbon nanotubes

Presentation plan

I- Introduction
Generalities

**II- Materials and Methods
and
III-Results and discussions**

IV- Conclusion and Perspectives

$S/C=3$, $W/C=0.50$

MORTAR

European Standard EN 196-1 (ISO 679).



Fig Technocem 32.5 R - CEM II – Multifunctional gray cement - CEMENTS CALCIA)

Fig Normalised Sand

Fig Mixer for mortar

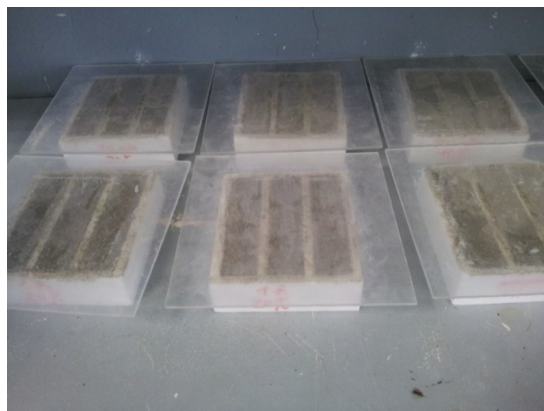


Fig shaking table for mortar

Fig Confection and conservation of samples mortar (20°C in 100% H)

CONCRETE



Fig. Sand bucket



Fig. Aggregates bucket

Constituents	Aggregates Percentage	Formulation (en kg/m ³)
0/4 (Sand)	38 %	704.90
4/8	17 %	315.35
8/16	40 %	742.00
16/20	5 %	92.75
Total Aggregates	100 %	1855
Cement		310
Water		1600

Tab Modified Concrete formulations used with CNT.



Fig Mixer of concrete



Fig Surfacing Machine



Fig shaking table for mortar



Fig Confection and conservation of samples mortar (20°C in 100% H)

Carbon Nanotubes (Graphistrength CL1-020) is delivered by Arkema Company

Properties	Specifies
Electric conductivity	$10^2 - 10^4$ s/cm
PH	11
Density	1000-1100 kg/m ³
Concentration liquid solution	2%wt of CNT
Liquid solution color at 20°C	Black
Liquid solution odor	Slight
Liquid solution	Solubility in other solvents

CNT concentrations:

0.01%, 0.02% , 0.03, 0.04%
and 0.06% for mortar
0.003%, 0.006% and 0.01%
for concrete

Tab: Properties of carbon nanotubes (CNT).

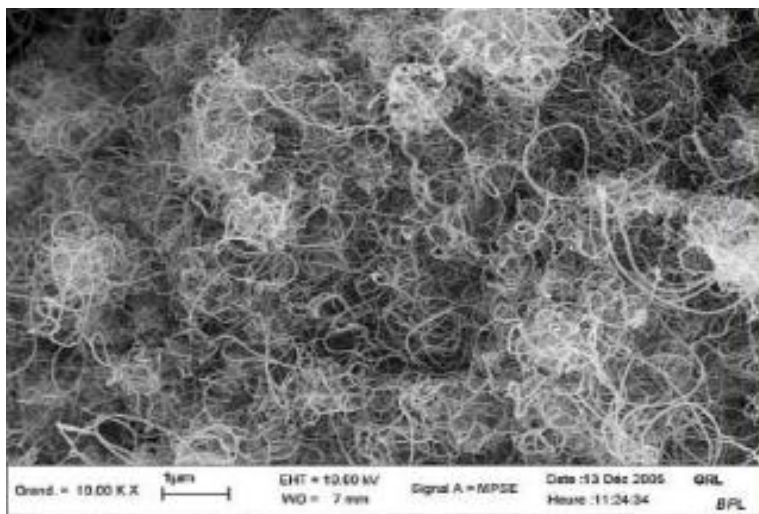


Fig. SEM CNT : ARKEMA – Graphistrength source

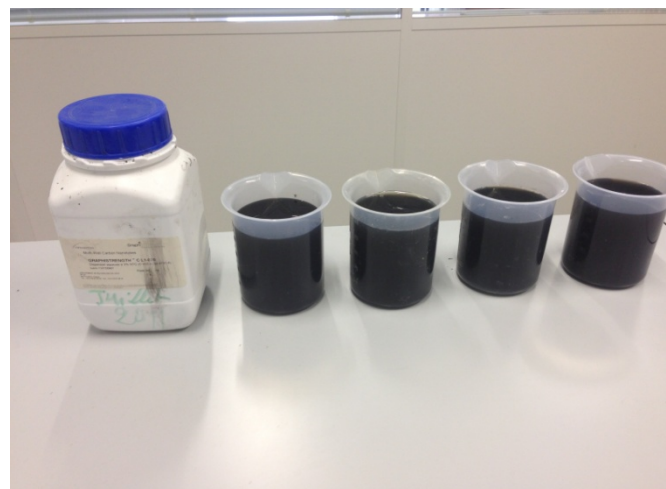


Fig: Liquid solution of CNT for different concentrations

Optimal CNT percentage

Mechanical properties

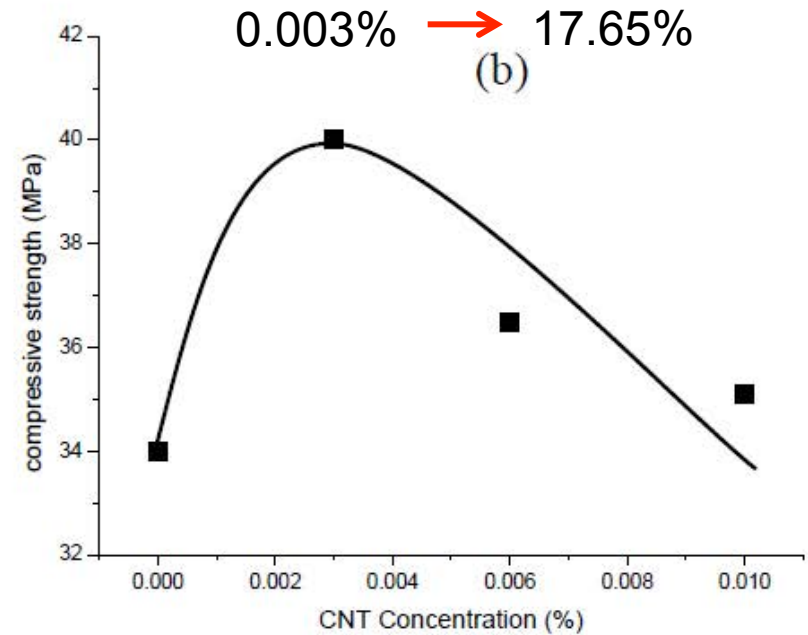
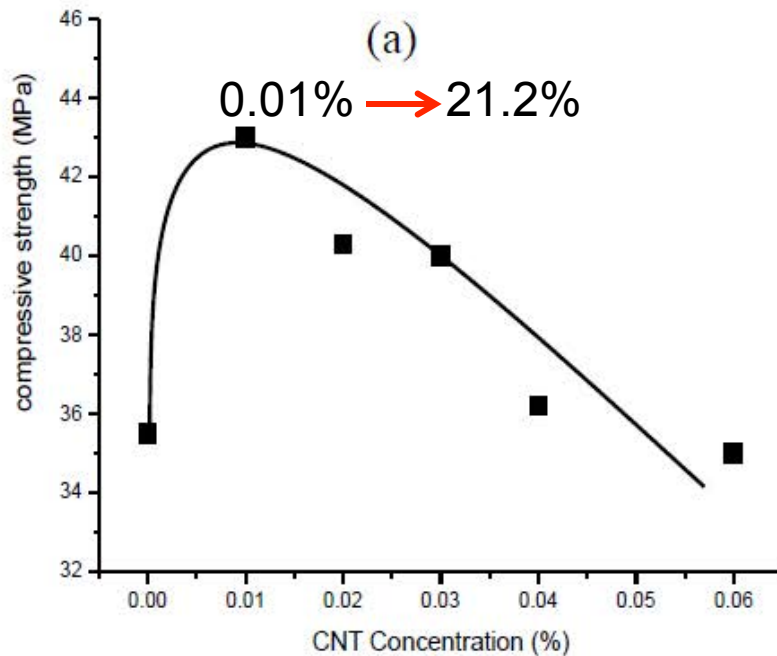


Fig Evolution of the compressive strength as function CNT percentages for 90 days for (a) mortar, (b) concrete

Microstructures

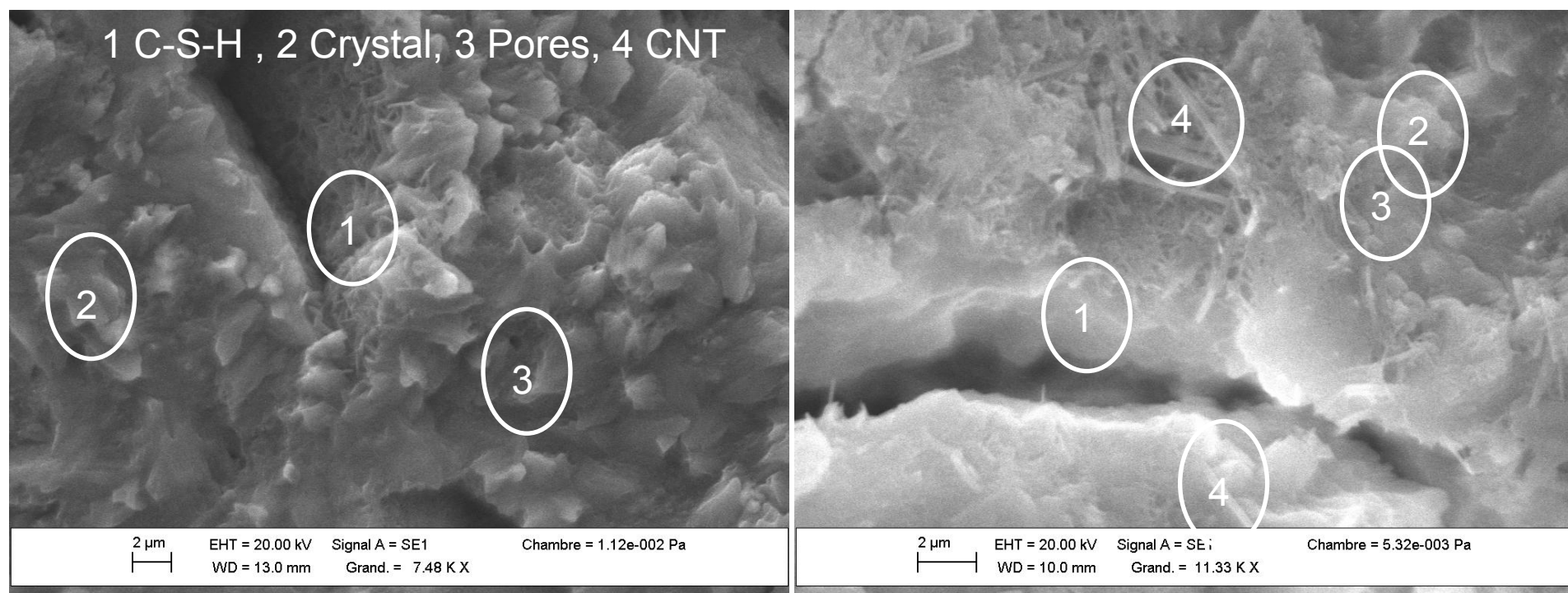


Fig. Morphology analysis by scanning electronic microscopy is performed for concrete samples aged of 90 days: (a) reference concrete, (b) concrete + 0.003% CNT

HEMP FIBERS + CNT

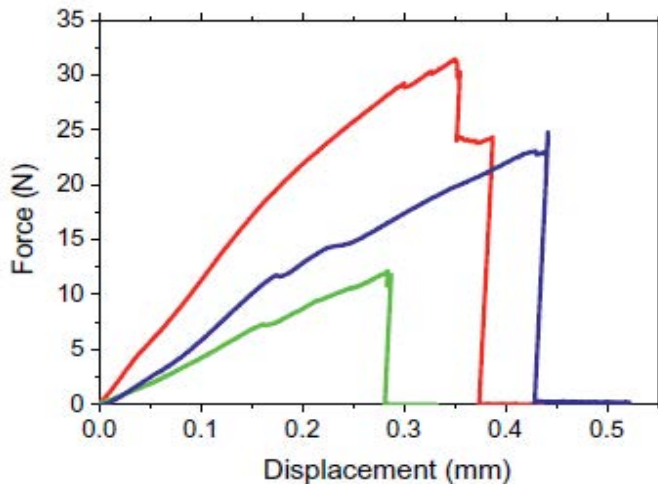
HEMP FIBERS + CNT



Fig X ray - micro-tomography skyscan1174 compact micro-CT.



(a)



(b)

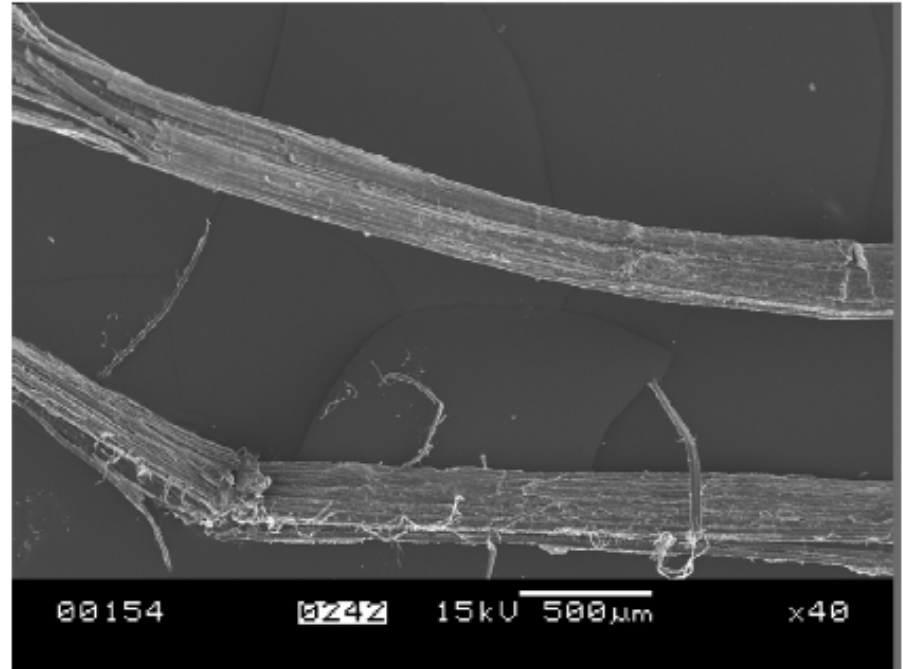
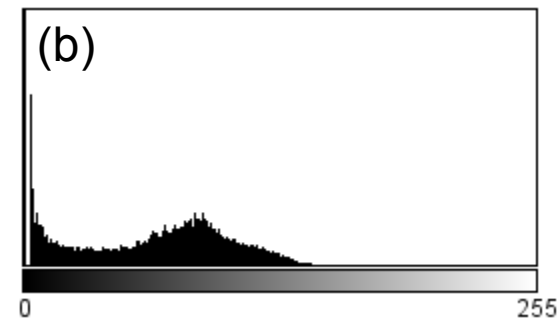
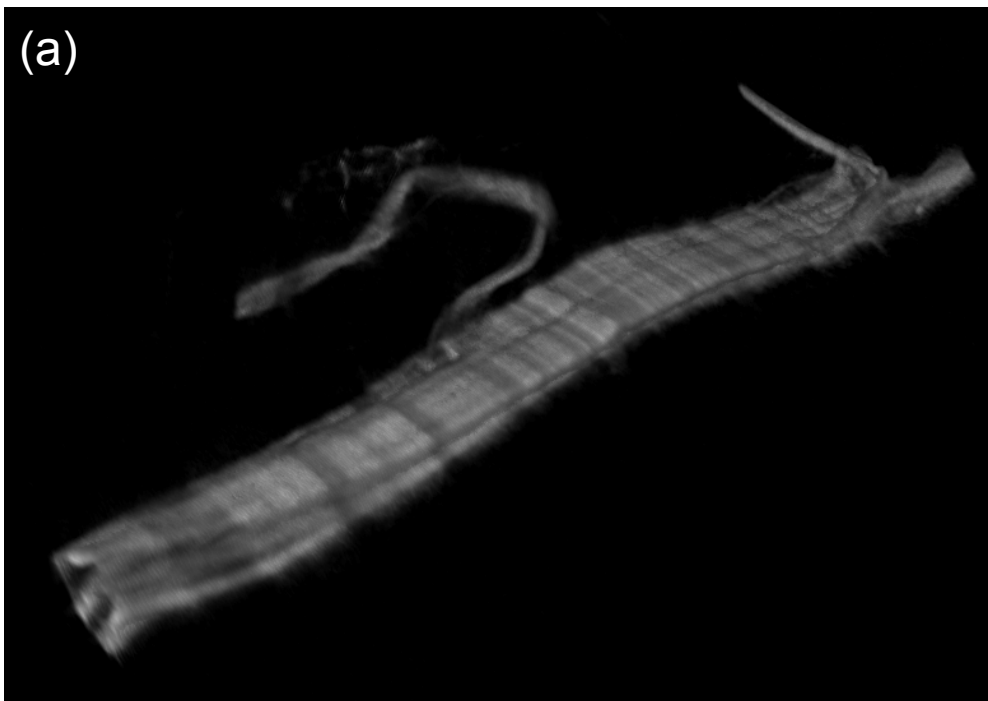


Fig SEM micrograph showing surface defects and overall aspect of hemp fibers.

Fig. (a) Initial and final tensile loading stages of a hemp fibre (gage length = 10 mm) and (b) typical force–displacement response for three samples.

Reinforced to modified X ray - micro-tomography



Circularity (C_c)	0.52 ± 0.14
Aspect ratio (C_a)	3.05 ± 0.67
Roundness (C_r)	0.37 ± 0.18
Solidity (C_s)	0.87 ± 0.10
Bounding area ratio (C_b)	0.50 ± 0.15

Tab Summary of the hemp fibre shape descriptors.

Fig. (a) Perspective view showing hemp fiber bundle morphology using X-ray micro-tomography, (b) Grey level histogram showing Gaussian distribution profile centred around 86 that corresponds to the imaged material.

Optimal percentage of Hemp fibers



Fig. Lengths of hemp fibers about 2–3 cm.



Fig Dry hemp fibers



Fig Wet hemp fibers for 15 minutes



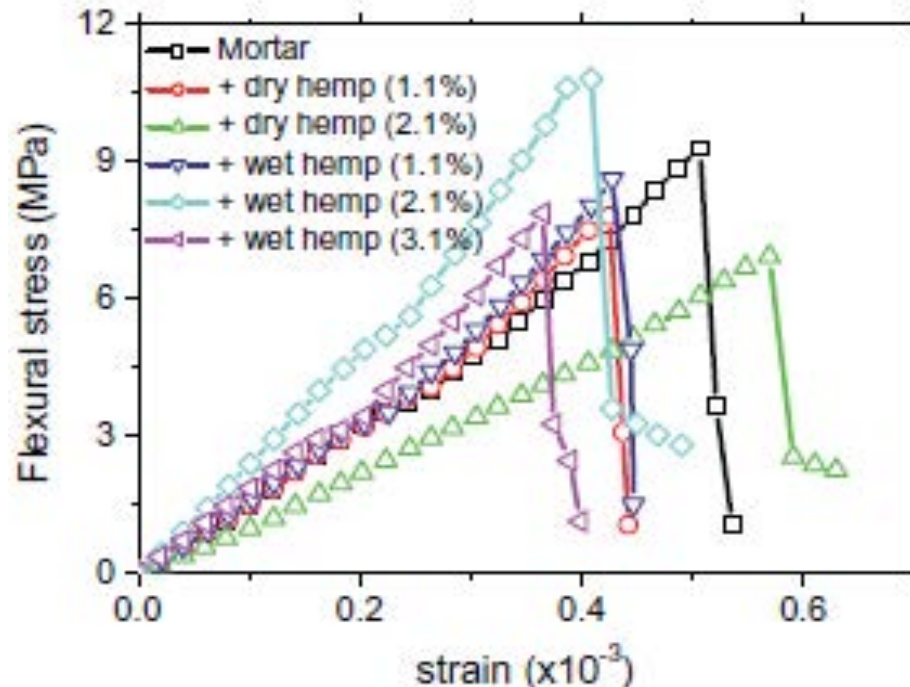
(a)



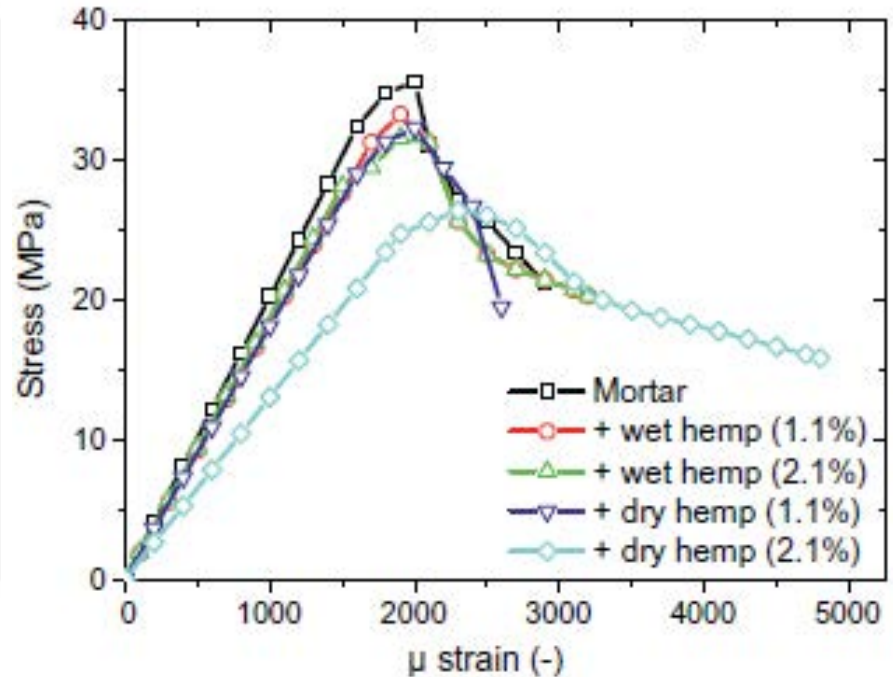
(b)

Fig. MTS equipment for (a) 3-point bending and (b) compression tests

Hemp fibers are added with different percentages (1.1 wt%, 2.1 wt% and 3.1 wt%) to the cement paste. The addition of fibers implies the reduction of the cement content by the same percentage.



(a)



(b)

Fig. (a) Flexural response of mortar-hemp composites for an increasing content of dry and wet hemp fibres. **(b)** Compressive response of mortar-hemp composites as function of dry and wet hemp contents.

Flexural stress : WHF in 2.1% HF we win 13%, DHF loss 38%

Compressive strength loss: WHF is 11%, DHF is 26%.

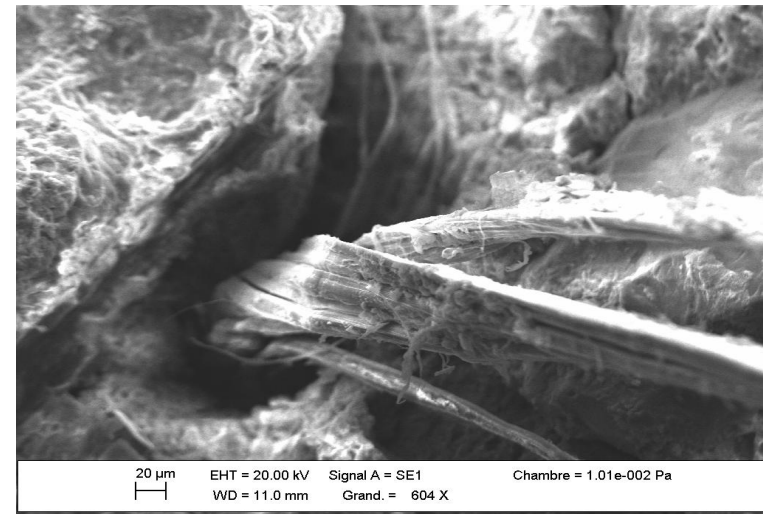
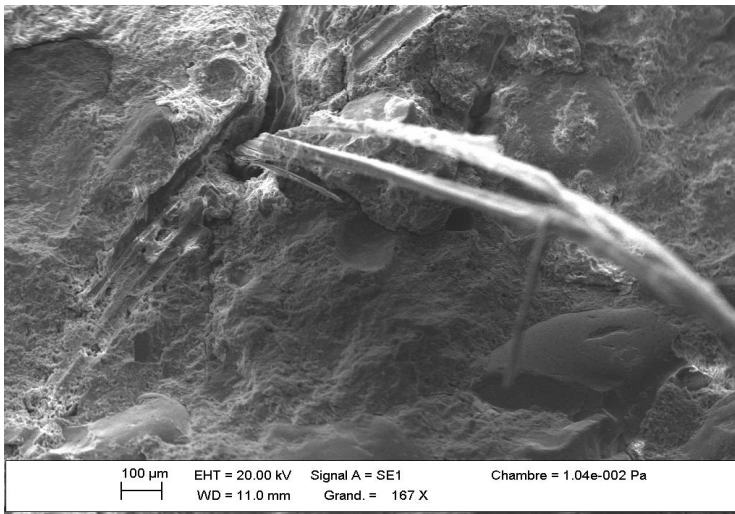
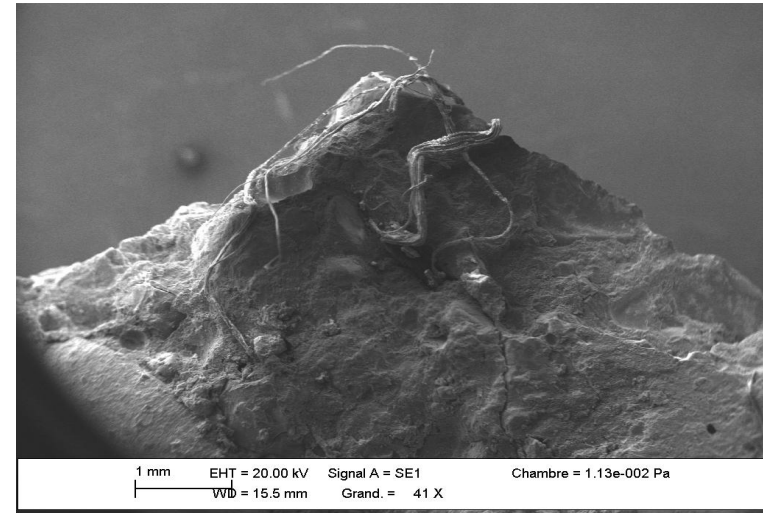
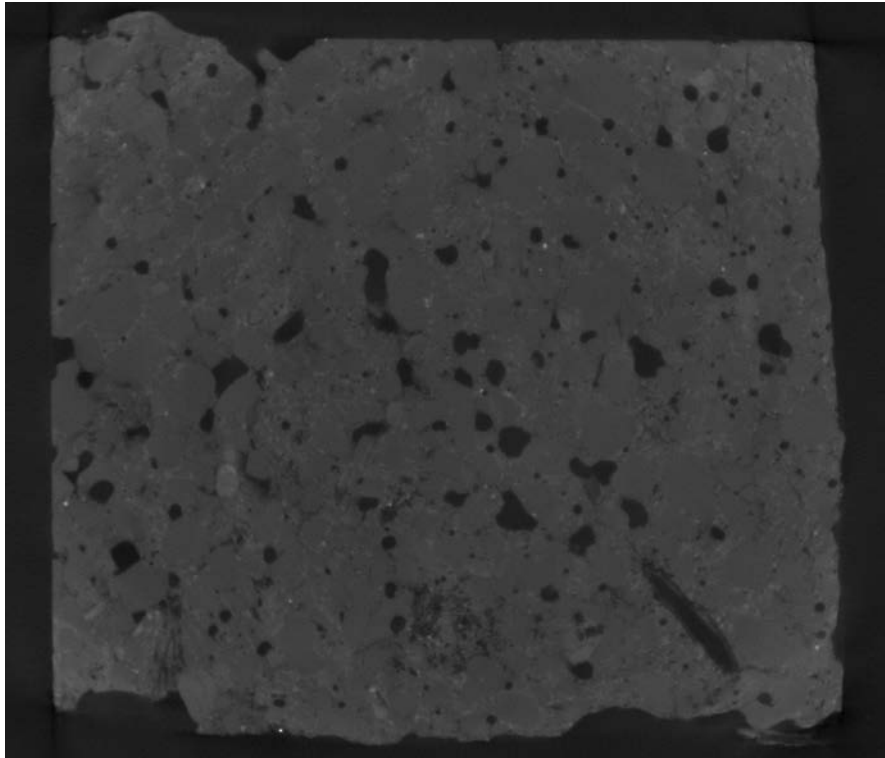
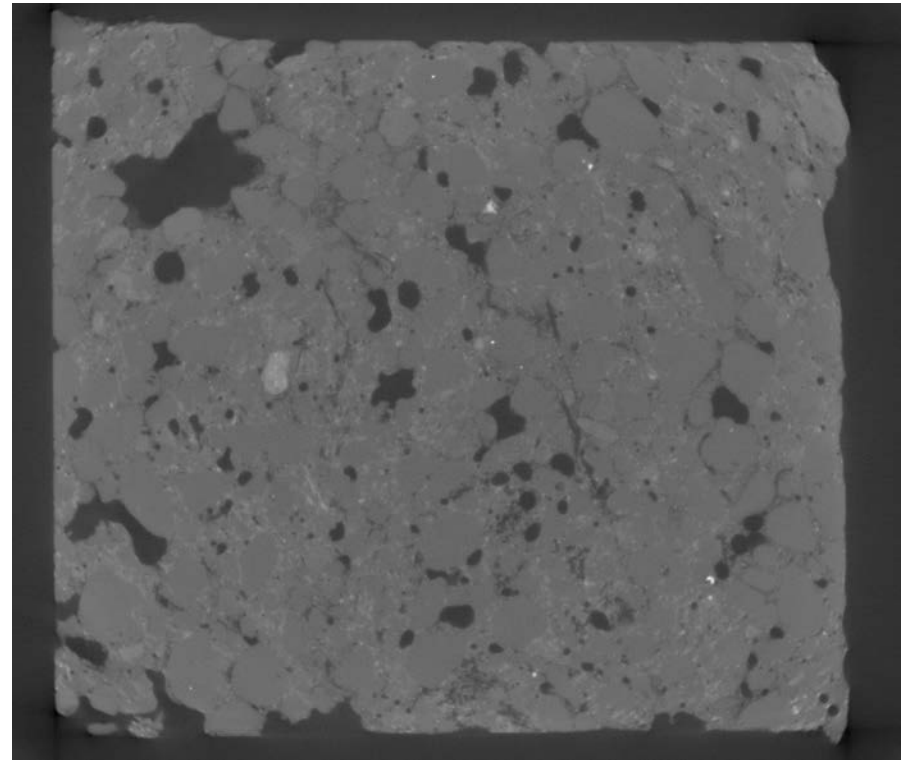


Fig Photo and SEM micrograph showing good adherence with fiber, twisting and tortuosity

X ray - micro-tomography



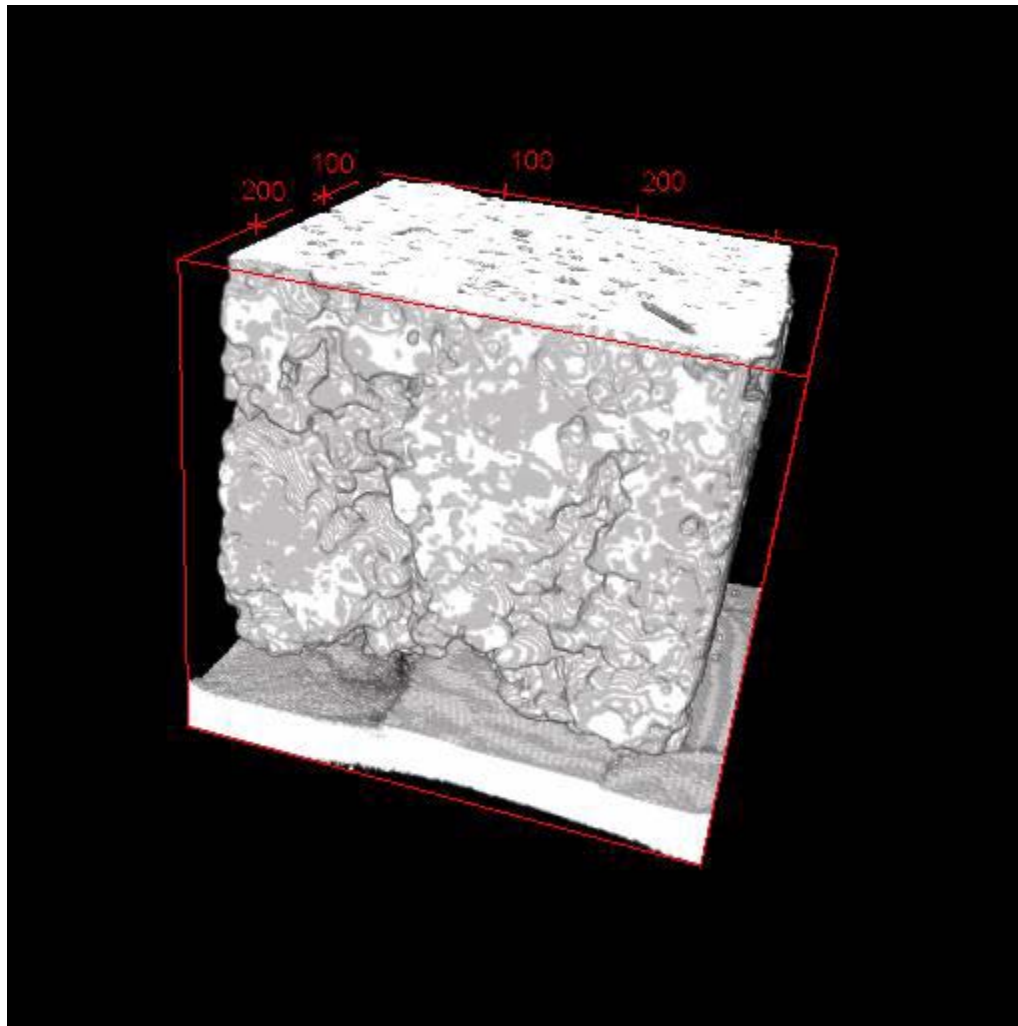
Mortar volume
1377x1117x1260 voxels
230 KV, Number of projection 1440



Slice 1 (porosity in black,
matrix in dark grey, gravel in
light grey)

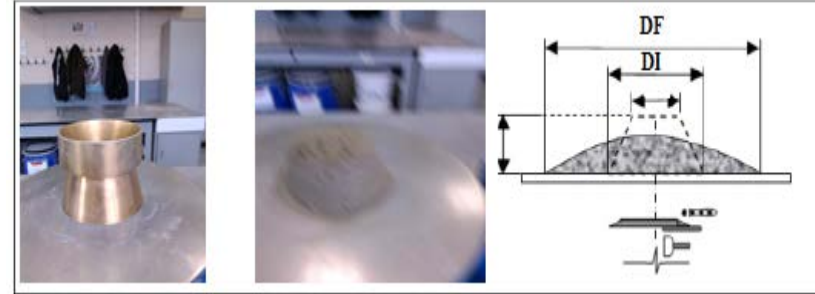
Slice 236 hemp in white

HEMP FIBERS + CNT



3D

Workability with Fresh Hemp fibers mortar



$$E(\%) = \frac{Df - Di}{Df} * 100$$

Fig LCPC equipment

Fibers %	Reference	1.1%	2.1%	3.1%
Time (s) wet fibers	4.1	8	12	18
Time (s) dry fibers	4.1	15	30	
E(%) wet fibers	40.1	35	30	21
E(%) dry fibers	40.1	27	7	

Tab: Workability of wet hemp fibers modified mortar and dry hemp fibers modified mortar

Optimal percentage of
Hemp fibers
+
Optimal CNT
0.01% CNT + 2.1%wt
WHF

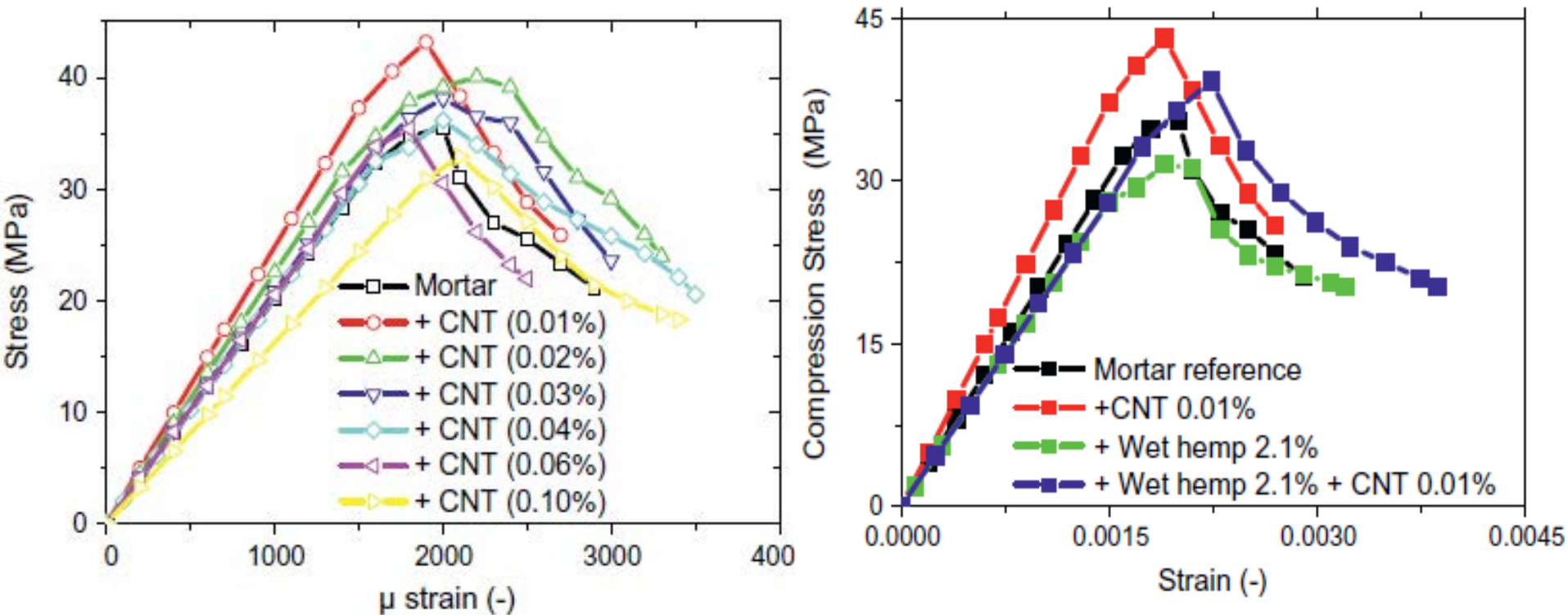


Fig Compressive response for CNT-mortar and hemp-CNT-mortar

Compressive strength

Mortar+CNT 21%

Mortar + Hemp fibers + CNT 17%

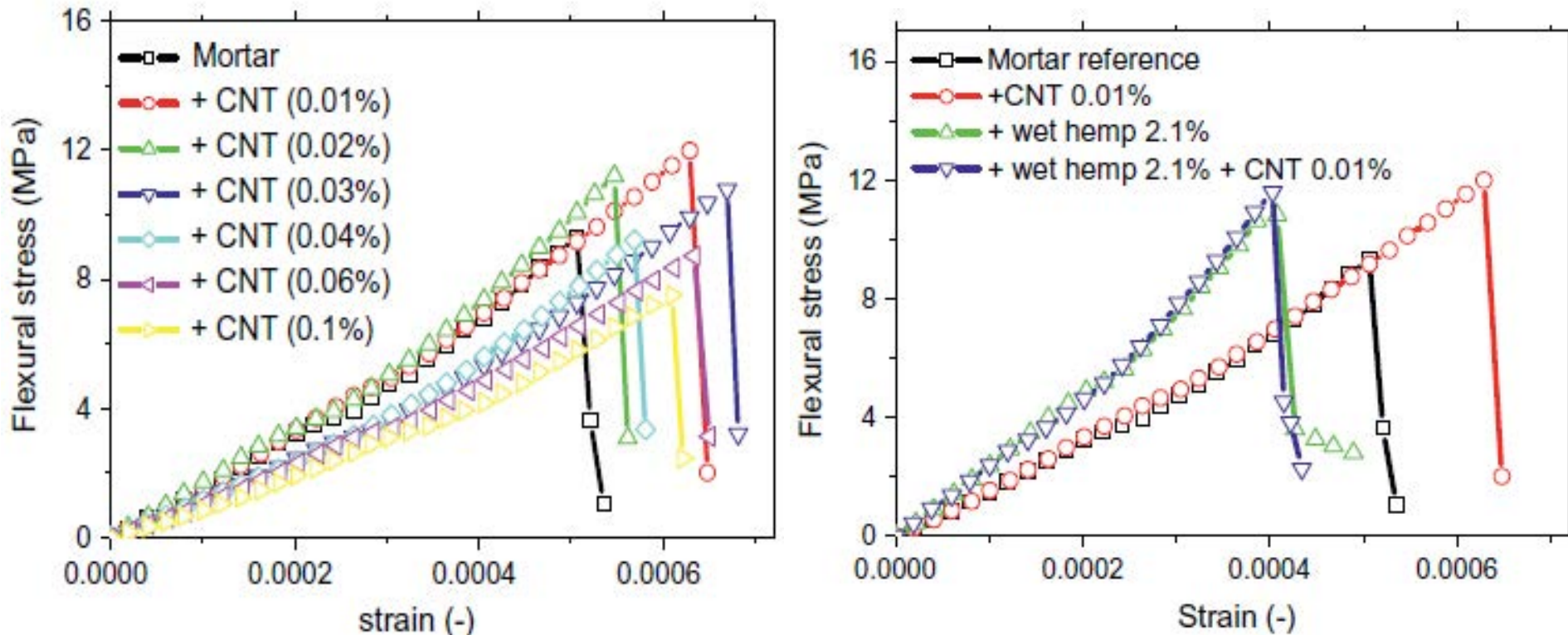


Fig Flexural response for CNT-mortar and hemp-CNT-mortar

- Optimal CNT percentage is 29% higher than the reference mortar.
- Mortar +CNT+Hemp Fibers:14.5%

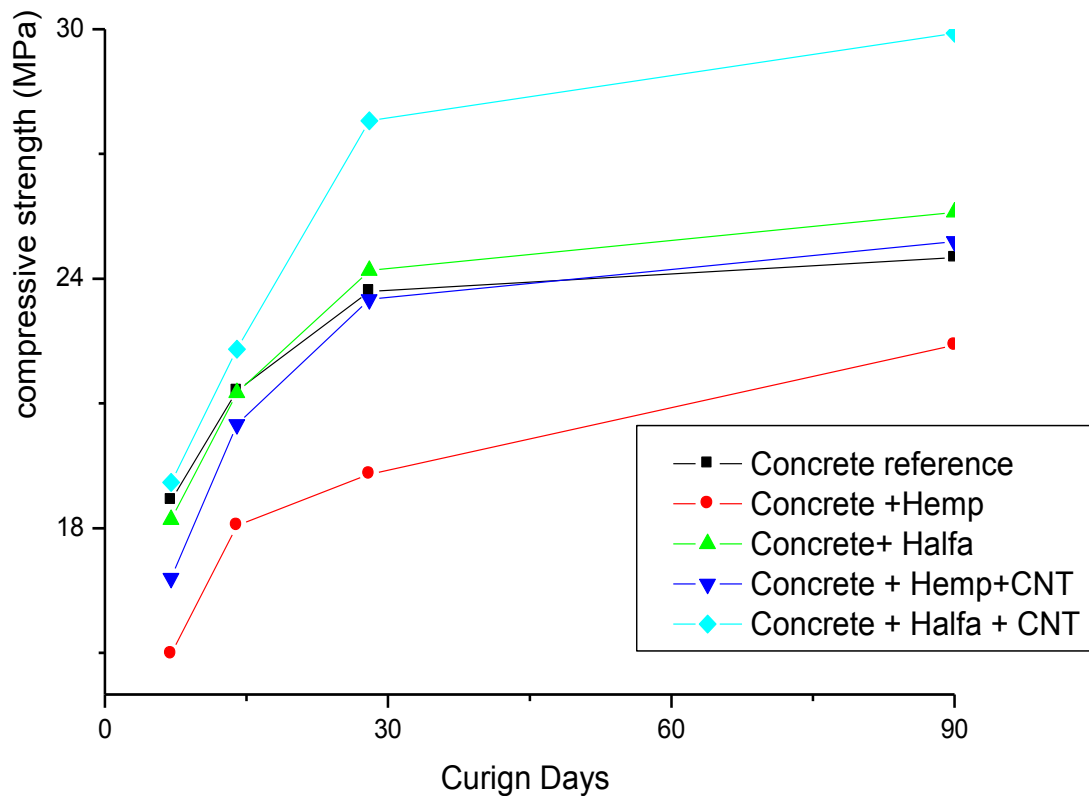


Fig Compression Strength versus curing days for different concretes mixtures (Hemp and Halfa).

Presentation plan

I- Introduction
Generalities

II- Materials and Methods
and

III-Results and discussions

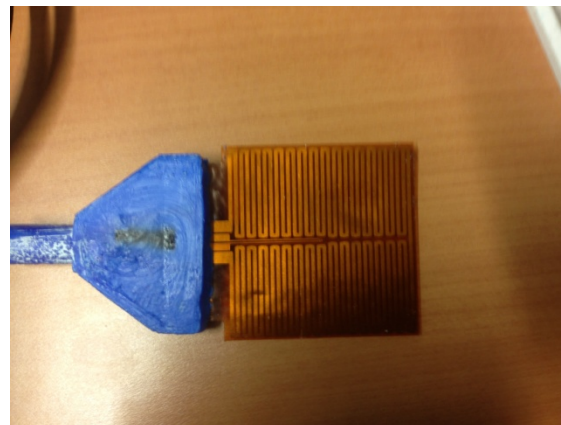
IV- Conclusion and Perspectives

Conclusion

- 1) Optimal concentrations of CNT which have given the maximum percentages improvement in compressive strength are 0.01%wt and 0.003%wt for mortar and concrete respectively.
- 2) The improving percentage of compressive and flexural strength reaches about 21.2% and 29% than reference mortar for 0.01%wt CNT percentage in mortar respectively. The improving percentage of compressive strength reaches about 17.65% than reference concrete for 0.003%wt CNT percentage in concrete.
- 3) The addition of small volume content of hemp fibers (<10%) increases linearly the porosity content in mortar. The rate of porosity decrease is larger if wet hemp is used. Optimal percentage of hemp fiber is 2.1% wt.
- 4) The improving percentage of compressive strength reaches in Hemp and about 17% and 9% than reference mortar 0.01%CNT and reference concrete 0.003% CNT respectively.

Perspectives

1) Depth study in thermal behavior of HFMM (HFRM) and HFMC



Not adapted equipment for
HFMM or HFMC

Perspectives

2) Spraying Hemp fibers



Spray machine of glass fibers

3) Obtaining smart materials of construction with nanopozolanic materials using natural materials (kaolinite, illite, zeolites, ...) and co-product materials (slag, fly ash, nano-SiO₂) with high ball milling energy and mechanical alloying technique.

4) Obtaining smart materials of construction with nanomaterials and natural fibers (Hemp, Halfa Fibers,.....)

THANK YOU FOR YOUR ATTENTION