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## Effect of the Use of Autoclave on Mechanical Behavior of the Bio-Construction Materials

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### Abstract

The paper discusses the use of autoclave as a method for increasing the compressive strength of bio-construction materials. Hemp fibers were mixed with three different binders, lime and silica fume mix ( $\text{CaO/SiO}_2 = 1$ ), cement and silica fume mix ( $\text{CaO/SiO}_2 = 0.96$ ) and cement, silica fume and lime mix ( $\text{CaO/SiO}_2 = 0.95$ ). Three autoclaved temperatures were used ( $110^\circ\text{C}$ ,  $130^\circ\text{C}$  and  $180^\circ\text{C}$ ).

The results showed a significant increase of the compressive strength for autoclaved samples. The highest strength obtained for autoclaved mix was at  $110^\circ\text{C}$ . The compressive strength of lime mix was multiplied with 9 (0.5MPa to 4.4MPa). While the cement and silica fume have been increased from 0.8MPa to 1.5MPa once the samples were autoclaved at  $110^\circ\text{C}$ .

### Keywords:

Autoclave, Cement, Compressive Strength, Hemp, Lime, Silica Fume.

### 1. Introduction:

Concrete is among the least expensive materials we use around the world. However, the production of one ton of cement emits about 700-900 kg of  $\text{CO}_2$  and other greenhouse gases (GHGs), which menace the stability of the planet.

In 2008, cement manufacturers from World Business Council for Sustainable Development recognized the urgency to reduce  $\text{CO}_2$  emissions from cement industries and requested the International Energy Agency (IEA) to evaluate long term options to decrease the environmental impact. For that purpose, improving energy efficiency, the use of alternative fuels and material to produce clinker and the capture of  $\text{CO}_2$  emissions were the main strategies considered. Since the

main source of CO<sub>2</sub> in cement production comes from the decomposition of limestone and emissions. Therefore, hemp concrete can represent a sustainable solution which respects the environment demands.

The compressive strength of hemp concrete is very low, it is less than 2 MPa (1, 2 and 8-14), which is considered as a negative property for this type of concrete. Many factors play a role in the compressive strength of hemp concrete. The density is considered as a determinant factor in the increase of the compressive strength, higher density mix has higher compressive strength, mix with density 1.2g/cm<sup>3</sup> has in the compressive strength of 1.8MPa (1) while the density of 0.8g/cm<sup>3</sup> gives 0.8MPa (2). The mix composition represents a second factor where the presence of cement in mix increases the compressive strength (5). An increase of cement from 29 to 50% (weight) doubled the compressive strength (4). The relative humidity (RH) is third factor where the mix with 50% of RH gives the strength of 0.35MPa (7).

In order to ameliorate the compressive strength, the use of autoclaved cycle can be used. However, the choice of curing temperature is governed by the change of the binder microstructure. Changes can occur from CSH and CH to tobermorite (CSH) crystalline structure, which denser thus it has a higher compressive strength than both structures (CSH and CH) (16). The use of autoclave increases the compressive strength of mix; the highest strength was given for hemp mix with quartz powder, which is less than 2MPa at 190°C autoclaved temperature (15).

In other hand, the hemp concrete has an advantage concerned its low thermal conductivity, its absorption of sound thus it used as thermal and acoustic isolation purposes. Florence et al. have shown that hemp concrete has a good thermal behavior which is related to its low thermal conductivity (3). Samri states that hemp concrete presents good hygric properties in comparison to other types of concrete and has a real economical heat comfort (16).

The aim of this paper is to overcome the lower compressive strength of hemp concrete by the use of autoclave cycle at different temperature (110, 130 and 180°C).

## **2. Experimental and Testing Program**

### **2.1. Materials**

Hemp from Urika (French supplier for hemp) was used with three type of binder:

- 1- Lime Tradical 98 (commercial name),
- 2- Cement CEMI 52.4,
- 3- Silica fume type S95 DS from Condensil,
- 4- Superplasticizer Adefor 2003 LOM was used; the solid concentration is 22%.

Table 1 shows the chemical composition of each material according to the main composition CaO and SiO<sub>2</sub>.

Tab. 1: Chemical composition of Lime (Tradical 98), silica fume and cement.

Mix	CaO %	SiO <sub>2</sub> %	Miner %
<b>Lime</b>	92.5	0	7.5
<b>Silica fume</b>	0	95	5
<b>Cement</b>	64	29.4	6.6

## 2.2. : Mix proportions

Three mixes were studied; they were prepared in order to have a CaO/SiO<sub>2</sub> ratio around 1. Hemp fibers were mixed with three different binders. The CaO/SiO<sub>2</sub> ratio is given in below for each mix:

- 1- Lime and silica fume mix (CaO/SiO<sub>2</sub> = 1),
- 2- Cement and silica fume mix (CaO/SiO<sub>2</sub> = 0.96)
- 3- Cement, silica fume and lime mix (CaO/SiO<sub>2</sub> = 0.95).

Three curing temperature (110°C, 130°C and 180°C) were used. Table 2 shows the composition of mixes used in the study, the quantities were calculated for 1M<sup>3</sup>. The water content was optimized in order to have a good workable mix. The quantity of superplasticizer is 2% of the binder; the water was recalculated and a mixer of 20 liter was used.

Table 2: Composition of mixes for 1M<sup>3</sup>.

Mix	Hemp (Kg)	Lime (Kg)	Cement (Kg)	Silica fume (Kg)
<b>Cement</b>	116	0	286	130
<b>Lime</b>	116	286	0	290
<b>Lime + cement</b>	116	100	140	170

### 2.2.2. Mix proportions and concrete mix design

A special mixing method for hemp concrete was used and not according to ASTM, samples were casted in cylindrical mold 11\*22cm, each sample was casted using 6 increments, using 50N for each compacted one. Samples were conserved at 25°C and 50% of RH for 24h and then samples placed in the autoclave for 24h at 30°C, then the autoclaved cycle was realized at desired temperature.

### 2.2.3 Autoclave cycle

Three autoclaved temperatures were used in the study, 110°C, 130°C and 180°C, the choice of the three autoclaved temperatures (100°C, 130°C and 180°C) was done in order to study the effect of the change on the microstructure on the compressive strength. It supposes that the increase on the autoclaved temperature (higher than 110°C) will tend the microstructure to lose the bonded water thus decrease the compressive strength.

The speed used for heating is 60°C per hour, and then the temperature is stabilized for 6h at the desired temperature in order to have a homogenous temperature in all parts of the samples. Fig. 1 shows the temperature and the pressure cycle used in the autoclave cycle at 180°C. The figure below shows two curves, the temperature was noted on the left and the pressure on the right as a function of time. At the end of the autoclave cycle, the samples were conserved at 23°C and 50% RH. The mass was measured each day until stabilization was achieved, where the mass loss is inferior to 1%.

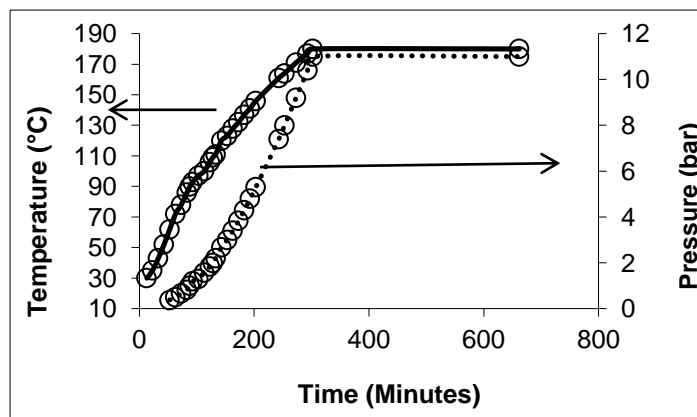


Figure 1: Temperature and Pressure cycle used for autoclave at 180°C

Fig. 2 shows images of hemp before and after autoclaved. The hemp became darker after it had been autoclaved at higher temperature (more than 100°C).



Fig. 2. Images of Hemp before and after autoclaved.

Fig. 3 shows the change on the microstructure of the ratio of C/S as a function of autoclaved temperature. The 200°C autoclaved temperature represents a limit between the changes of tobermorite crystalline to truscotite. This change is coming from the decomposition of the tobermorite, which is caused by the loss of the water binder of the microstructure and the mix will shrink when dried. The truscotite microstructure represents a crystal in form of fibers which is less denser than the tobermorite thus a decrease on the compressive strength. The density of tobermorite is higher than CSH which will give higher compressive strength to the mix.

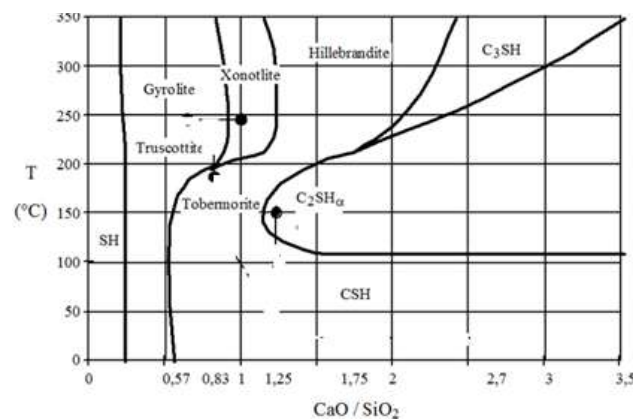


Fig. 3. Changes occur on the composition of CSH due to the increase of the temperature (17).

### 2.3.2 Mechanical test

MTS machine was used for mechanical test, with 100N/sec speed. Fig. 4 shows the hemp sample during the compressive strength test.



Fig. 4. Hemp sample during the compressive strength test.

### 3. Results and Discussion

#### 3.1 Density

The initial density of mixes (after demolded) is reported in table 3. The lime mix showed the highest density then the cement and lime and the lowest density was obtained for the cement mix. The highest density of lime mix can be attributed to the presence of more quantity of silica fume than in cement (see table 2).

Table 3

Density of mixes.

Mix	Density (g/cm <sup>3</sup> )
Cement + Silca Fume	0.90 ± 0.02
Cement + Lime + Silcia Fume	1.10 ± 0.05
Lime + Silca Fume	1.18 ± 0.02

Fig. 5 shows the mass loss as a function of conservation time at 50% of RH and 23°C for autoclaved mixes at 180°C. The curves show a stabilization of the mass after 11 days for both autoclaved mixes. The notation of Lime mix was done for the lime and silica fume mix while the notation of Cement mix was done for the cement and silica fume mix in the paper

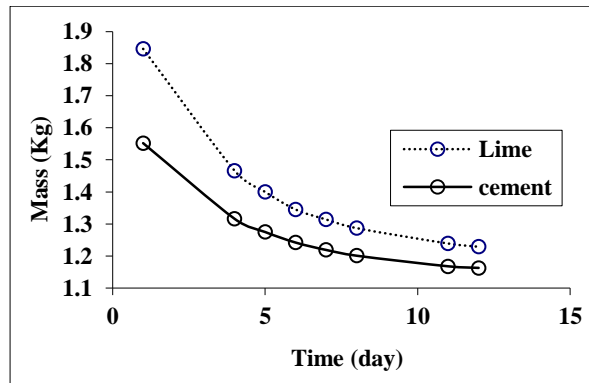


Figure 5: Mass loss as a function of mix and time

### 3.2. Mechanical tests

#### 3.2.1 Non autoclaved mixes

Fig. 6 shows the stress strain curves for the non-autoclaved mixes, cement, cement + lime, and lime. Cement mix produced the highest compressive strength (0.8MPa). The lime mix gave the lowest compressive strength (0.5MPa) while the lime + cement mix gave a compressive strength between them (0.7MPa).

This result is correlated to the composition of each mix, see table 2. The highest compressive strength was obtained for the mix with the highest quantity of cement, thus the cement mix proved to have higher strength than the cement lime mix. This can be explained by the amount of the hydrate products of cement and lime. The hydration process of cement produced the majority of CSH and minor quantity of CH while lime produced the opposite. Accordingly, the compressive strength CSH is higher than CH.

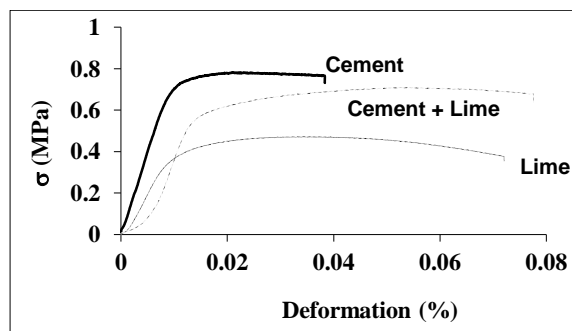


Figure 6: Stress Strain curve of non-autoclaved samples

Table 4 shows the compressive strength and the modulus of elasticity for the non-autoclaved mixes. The modulus of elasticity for cement mix is highest, which was generated from the highest amount of CSH in cement mix.

Table 4

Compressive strength and modulus of elasticity of non-autoclaved mixes.

Mix	Compressive strength (MPa)
Cement	0.78
Cement + Lime	0.71
Lime	0.47

### 3.2.2 Autoclaved mixes at 110°C

Fig. 7 shows the stress strain curves of autoclaved mixes at 110°C, the highest compressive strength was obtained for the lime mix with 4.4MPa where lime and cement mix comes next with 2.8MPa and the lowest strength is obtained for cement mix with 1.5MPa. It supposes that the highest strength of lime mix is emerging from the reaction between CH and SiO<sub>2</sub> to create tobermorite which has a higher strength than CH and CSH. Regarding figure 3, due to the increase of the temperature the structure has the tendency to form tobermorite at elevated temperature (more than 100°C).

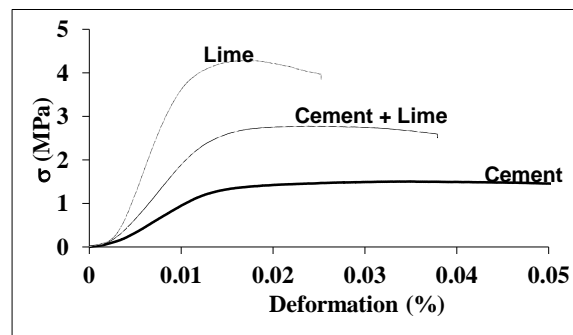


Figure 7: Stress Strain curves of autoclaved mixes at 110 °C

Table 5 shows the compressive strength and the modulus of elasticity of autoclaved mixes at 110°C. It shows that the strength values of autoclaved mixes are higher than non-autoclaved mixes.

The strength for autoclaved cement mix is twice higher than the non-autoclaved one, but autoclaved lime mix is nine times higher than the non-autoclaved one. The modulus of elasticity

of lime mixes is higher for autoclaved mixes than non-autoclaved mixes while it shows absent significant change for cement mix.

Table 5: Compressive strength and modulus of elasticity of autoclaved mixes at 110°C.

Mix	Compressive strength (MPa)	Modulus of elasticity (MPa)
Cement	1.47 ± 0.04	100.5 ± 10.8
Cement + Lime	2.80 ± 0.03	241.3 ± 22.6
Lime	4.35 ± 0.27	509.4 ± 99.1

As a result, it is evident that autoclave stand as good tools for increasing the strength of mixes where lime is present.

### 3.2.2 Autoclaved mixes at 130°C

Fig. 8 shows stress strain curves of autoclaved cement and lime mixes at 130°C. The lime curve shows the highest ductility and the lowest compressive strength. Table 7 shows the compressive strength and the modulus of elasticity of autoclaved cement and lime mixes.

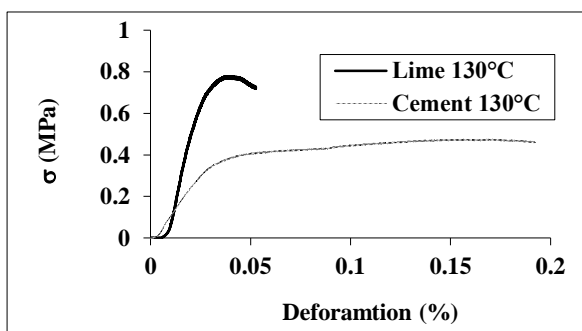


Fig. 8. Stress Strain curves of autoclaved mixes at 130°C.

Table 6 shows the compressive strength and the modulus of elasticity of both autoclaved mixes at 130°C. The lime mix shows a higher modulus of elasticity than the cement mix and lower compressive strength than cement mix.

Table 6: Compressive strength and modulus of elasticity of autoclaved mixes at 130°C.

Mix	Compressive strength (MPa)	Modulus of elasticity (MPa)
Cement	0.96 ± 0.13	20.9 ± 5.0
Lime	0.79 ± 0.40	37.8 ± 13.4

### 3.2.3 Autoclaved mixes at 180 °C

Fig. 9 shows the stress strain curves of autoclaved cement and lime mixes at 180°C, both curves show a similar behavior.

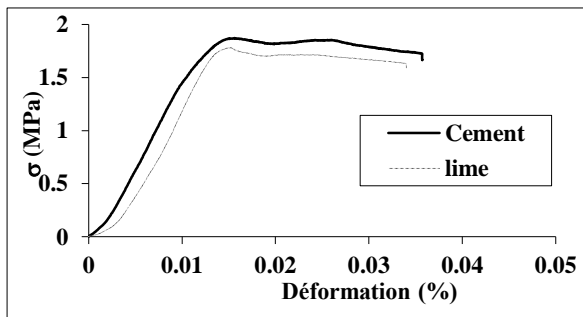


Figure 9: Stress Strain curves of autoclaved mixes at 180 °C

Table 7 shows the compressive strength and modulus of elasticity of autoclaved cement and lime mixes at 180°C. Cement mix show doubled increase in compressive strength (from 0.72 MPa to 1.8 MPa) while lime mix shows a five-time increase in compressive strength (from 0.33 MPa to 1.7 MPa). Both mixes show a similar compressive strength and modulus of elasticity.

Table 7: Compressive strength and modulus of elasticity of autoclaved mixes at 180°C.

Mix	Compressive strength (MPa)	Modulus of elasticity (MPa)
Cement	1.80 ± 0.15	167.73 ± 18.7
Lime	1.68 ± 0.11	171.85 ± 35.5

## 4. Conclusion

Fig. 10 shows a summary of autoclaved mixes results of cement and lime mixes at 110°C, 130°C and 180°C. Autoclaved lime mix at 110°C shows the highest compressive strength while autoclaved mixes at 130°C show the lowest compressive strength. The highest strength can be the result of the formation of more tobermorite, while the decrease of the compressive strength for the autoclaved mixes at 180°C erupts from the decomposition of the hydrate products and the shrinkage after turn dry.

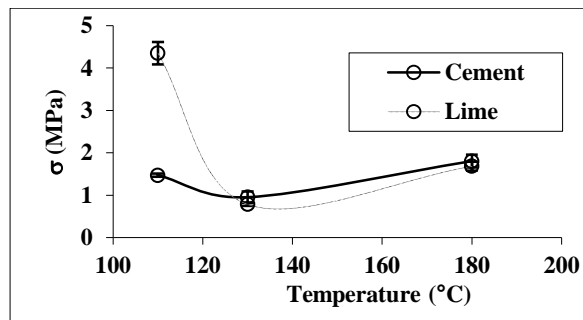


Figure 10: Summary of autoclaved result for cement and lime mixes

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