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Attempt to The Valorization of Brown Algae *Sargassum sp.* in Cement Mortar Composites for Housing or Building Purposes

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ABSTRACT: In this study, material composites composed of cement and normalized sand as matrix and brown algae *Sargassum sp.* as reinforcement and/or filler were prepared. When used as reinforcement, algae size was comprised between 0.4 and 1 mm at various amounts (0%, 1% and 2% wt) whereas when used as filler, algae size was lower than 0.4 mm and the content was 0 or 3 wt %. The aim of this study was to evaluate the potentiality of those materials as building materials; consequently, they have been tested after 7 and 28 days of curing by helium pycnometer, thermal conductivity and setting temperature, mechanical measurements and thermogravimetry.
Results show that using brown algae *Sargassum sp.* as reinforcement of cement decreases by up to 23% compressive performance of matrix, it has a negative influence on thermal conductivity and almost no influence on density. Fillers seem to act as set retarder of cement and to have a synergistic effect (when combined with 2% wt of on the decrease of thermal conductivity.

KEYWORDS: cement composites, compression strength, flexural strength, *Sargassum sp.*, thermal conductivity.

1. INTRODUCTION

For decades, some members of COVACHIM-M2E research team, located at Université des Antilles in Guadeloupe (French West Indies), develop cement-based materials for housing and construction [1]. Guadeloupe is a Caribbean Island (tropical climate) located between the Caribbean Sea and Atlantic Ocean in a strongly seismic region that is prone to major hurricanes [2-3]. For these reasons, modern building and housing in Guadeloupe are mainly made of concrete [4].
The materials elaborated by COVACHIM-M2E research team are non-conventional composites because in those materials, cement matrix is reinforced with vegetal resources, readily available in Guadeloupe, such as fibers of sugarcane bagasse, banana, coconut coir, … [1, 3].
Due to its geographical location, Guadeloupe does not produce any conventional energy and must therefore import the energy from the United States of America and South America [2]. Thus, the non-conventional materials elaborated by COVACHIM-M2E team provide an opportunity to solve the problem of over-consumption of air conditioning in concrete-based infrastructures because vegetal fibers cement composites are recognized to have thermal insulation properties [1].

For the past fifteen years, the beaches of Guadeloupe island have been victims of regular strandings of more than 20 million tons (cumulative) of brown algae *Sargassum sp.* [5-7]. In addition to the negative visual impact that constrains tourism and fishing activities [5], these strandings cause environmental degradation such as emission of H_2S (and CO_2, NH_3 and CH_4) during the decomposition algae which can cause health problems and alteration of marine wildlife near the shores [7]. Moreover, these same studies reveals that Caribbean zone is concerned with strandings of *Sargassum natans* (morphotypes I and VIII) and *Sargassum fluitans* (morphotype III) [5, 7].

According to the literature [8], there are "some examples exploring the use of brown seaweed (macroalgae) in building materials such as:

- Use of an alginate extracted from kelp to make algae slabs which are reported to be much stronger than concrete;
- Biopolymers extracted from brown seaweed have shown to be useful as additives in unfired clay bricks to increase particle bonding".

To this date, in Guadeloupe, a very few promising studies regarding the valuation of brown algae *Sargassum sp.* has emerged as biochar [6, 9] or as fertilizer [10] or as stabilizer in sustainable earth-based bricks [11] but there is no established value chain of the tons of these stranded algae.

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As some members of COVACHIM-M2E research team develop composites materials made of cement reinforced by vegetal fibers, in this study, in order to turn this problem of massive strandings into opportunities in a tropical area such as Guadeloupe island, an evaluation of the potentiality of using *Sargassum natans* and *fluitans* fibers as reinforcement of cement matrix for housing and building purposes is proposed. To our knowledge, no study including *Sargassum sp.* fibers as reinforcement of cement was published but according to authors, *Sargassum sp.* ash should not be used as pozzolanic material, but the high presence of minerals may suggest the application as filler [7]. By the way, in order to improve performance of cementitious matrix, we decide to use *Sargassum sp*. algae as reinforcement (fibers) and as filler in composites. The aim of this investigation is to obtain insulating materials having density and mechanical properties at least equal to those of conventional construction materials.
For this purpose, we elaborated mortar samples and characterized them by several methods: specific density, thermal properties (setting temperature, thermal degradation, thermal conductivity) and mechanical testing. Two ages of samples were studied: 7 and 28 days.

2. MATERIALS AND METHODS

2.1. *Sargassum sp.* fibers and filler

Raw brown sargassum algae (a mix of *Sargassum natans* and *Sargassum fluitans*) have been collected during their stranding (April, 14th 2021) on the beach of Bois-Jolan, Sainte-Anne (16°13'60'' north, 61°22'60'' west) in Guadeloupe while still in water to avoid the beginning of decomposition and to limit the presence of sand [10].
Previous study [10] has established that a random mix of *Sargassum* sp. is composed by 33 wt% of *Sargassum fluitans* (morphotype III) and 67 wt% of *Sargassum natans* (morphotypes I and VIII). As shown by Figure 1, the pelagic Sargassum species and/or morphotypes have morphological differences.

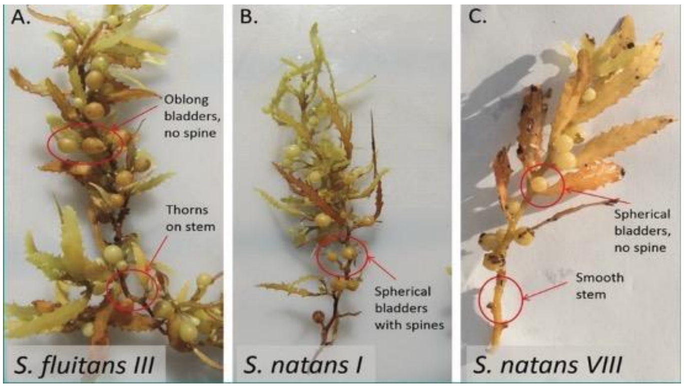

Figure 1: Morphological differences between pelagic Sargassum species and/or morphotypes [8]

As we have a simplified and economic valuation approach, we decide to use mixes of *Sargassum sp.* algae without differentiating the two species or different morphotypes.

At the laboratory, the following process of preparation of brown algae was applied:

(1) Primary rinsing under running tap water in order to eliminate impurities/small marine species, other types of algae and remove mostly the sea water;
(2) Drying at room temperature (28°C) and 80% of relative humidity (RH) for one month until completely dry;
(3) Storage in a cardboard box (28°C, RH = 80%);
(4) Coarse scissor cutting of the mix;
(5) Grinding using a cutter mill (Retsch GmbH, Germany) at 1500 rpm;
(6) Sieving: fibers with diameters ranging from 0.4 to 1 mm were selected as reinforcement of cement matrix whereas particles with diameters lower than 63 mm were used as filler.

2.2. Binder

The cement used was CEM II/B-ll 32,5 R marketed by Lafarge® under the name "Le Classic".
Some of its chemical, physico-chemical and mechanical characteristics are presented in Table 1.

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Cementitious matrix is a mortar made of a mix of cement, normalized sand (marketed by E2ME, France; granulometry ranges from 0.08 to 2 mm), algae filler and water. The sand grains are isometric and rounded in shape.

Table 1: Characteristics of cement

Composition (% wt)	Clinker Portland 72% Limestone 27% Gypsum 3,5%
Chemical composition	C3S 58% C2S 15% C3A 11% C4AF 9% MgO 1,5%
Physico-chemical properties	Specific density = 3 g/cm^3 Specific surface 4100 cm^2/g Setting 180 min
Mechanical strength	2 days compressive strength 18 MPa 28 days compressive strength 38 MPa

Elaboration of binder:
Binder was made of cement:normalized sand with the proportion of 1:3 and the proportion of algae filler was 3 wt %. Water/cement ratio was 0.5.
Table 2 reports the formulations of binders whose were elaborated according to NF EN 197.1 [12] normalized protocol.

Table 2: Formulation of binder.

Binder name	Mass of cement (g)	Mass of normalized sand (g)	Mass of *Sargassum sp.* filler (g)	Mass of water (g)	$^{water}/_{cement}$ ratio
CP	450.0	1350.0	0.0	225.0	0.5
CFA	450.0	1350.0	13.5	225.0	0.5

CP: Pur Cement; CFA: Algae Filler Cement

2.3. Elaboration of mortar composites

Mortar composites materials were elaborated according to NF EN 196.1 normalized protocol [13] using an industrial cement mixer (reference 10031.5 Controlab, France). *Sargassum sp*. fibers were used as reinforcement of binder.
In order to replicate measurements and to evaluate standard deviation, six standard parallelepiped specimens (4x4x16 cm^3) have been elaborated thanks to normalized molds.

Table 3. presents their formulations and associated nomenclatures.

Nomenclature	**Cement mass (g)**	**Sand mass (g)**	**Filler content (%wt)**	**Fiber content (%wt)**	**Water content (g)**	**Water/Cement Ratio**	**Number of elaborated specimens**
Cement (reference) CP	450	1350	0	0	225	0.5	6
CFAAB0	450	1350	3	0	225	0.5	6
CFAAB2	450	1350	3	2	261	0.59	6
CAB1	450	1350	0	1	243	0,545	6
CAB2	450	1350	0	2	261	0,59	6

Table 3: formulations and nomenclatures of elaborated specimens

C is for cement; P is for pure; FA is for algae filler; AB is for raw algae
For example, CFAAB2 is for a material made of (cement + algae filler) reinforced with 2%wt of raw algae.

The samples were unmolded after 24 hours then placed in a climatic chamber (25°C, 50% RH) before tests at 7 and 28 days.
To respect the aging times of cementitious matrix composite materials, we stopped the hydration of the cement with an organic acetone/methanol mixture with a ratio of 1:1. The ratio between the quantity of composite and the organic mixture is 1:100. Samples are soaked (until tests) and dried at 80°C for 1 hour according to [11].

2.4. Chemical structure of raw algae

Characterization of the chemical structure of the surface constituents of raw materials was carried out by Fourier Transform InfraRed (FTIR) spectroscopy using Attenuated Total Reflection (ATR) mode. The spectra were recorded on a transportable Fourier Transform InfraRed spectrometer marketed as Spectrum Two FT-IT spectrometer, Universal ATR Single Reflection Diamond (Perkin Elmer, France). For each sample, one acquisition analyzes was conducted between 4000 and 450cm^{-1}, with a resolution of 2 cm^{-1} at 7 and 28 days.

2.5. Thermal characteristics of mortar composite materials

Determination of setting time:
Hydration monitoring was carried out according to the method used by Bilba et al. [8]. For mortar samples, the highest temperature reached during hydratation is called "setting temperature" and the corresponding time to this highest temperature is called "setting time".

Thermal degradation :
Thermogravimetric analysis (TGA) was applied to our specimens. TGA is a thermal analysis method which consists of measuring variation the mass of a sample according duration and temperature. The equipment is a TG-ATD STA 6000 simultaneous thermal analyzer (Perkin Elmer, France). Thermal degradation of the samples was monitored from room temperature (30°C) to 900°C (10°/min). The thermograms were collected using Pyris series 6000 software at 7 and 28 days.
Thermal conductivity measurements :
Thermal tests were carried out under controlled laboratory conditions (temperature = 298 K and 70–80% relative humidity) on 7- and 28-days old specimens. The apparatus used was a thermal conductimeter ''CT-mètre" with a thermal probe (Controlab, France). The principle of the method is described by [3]. At least six measurements per formulation were conducted in order to evaluate the mean and the standard deviation of the results.

2.6. Specific density

Specific density of the materials was determined using a helium pycnometer "Pycnomatic" (Thermo Electron, France). This equipment allows to determine the specific volume of a solid sample of known mass and its specific density. Five measurements were conducted for each sample at 298 K and 70-80% of relative humidity.

2.7. Mechanical characteristics

Three-points bending and compression tests of the cementitious matrix composites materials (4x4x16 cm^3) were carried out according to standard EN 196.1 [13] with a standardized testing machine (3R, France).
In order to evaluate standard deviations, flexural tests have been repeated six times and compressive tests have been conducted twelve times at 7 and 28 days.

3. RESULTS AND DISCUSSIONS

3.1. Raw materials

Chemical structure:
According to literature, the main constituents of *Sargassum sp.* are cellulose, hemicellulose, lignin, lipids and protein [8]. More precisely, [14, 15], brown algae *Sargassum sp.* are composed of 20.35% of cellulose and 25.73% of hemicelluloses. these contents are significantly lower compared to those of plant fibers commonly used in the field of construction and housing [16].
Other authors do not agree with these contents and state the botanical composition presented by Figure 2.

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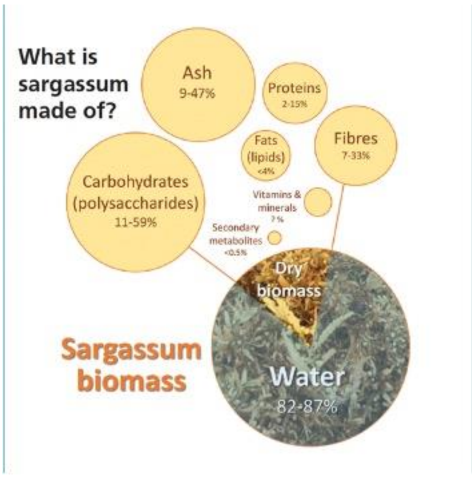

Figure 2: Main constituents of Sargassum biomass [9]

FTIR analysis, described by Figure 3, shows that *Sargassum sp.* algae contain O-H groups at ~2900 cm^{-1} (broad band of high intensity) and O-H groups at ~3600 cm^{-1} (fine low intensity band) [17]. The numerous bands between 1000 and 1300 cm^{-1} indicate the presence of C-O groups [17].
The observed chemical groups are in agreement with the presence of cellulose and hemicelluloses [14, 15].

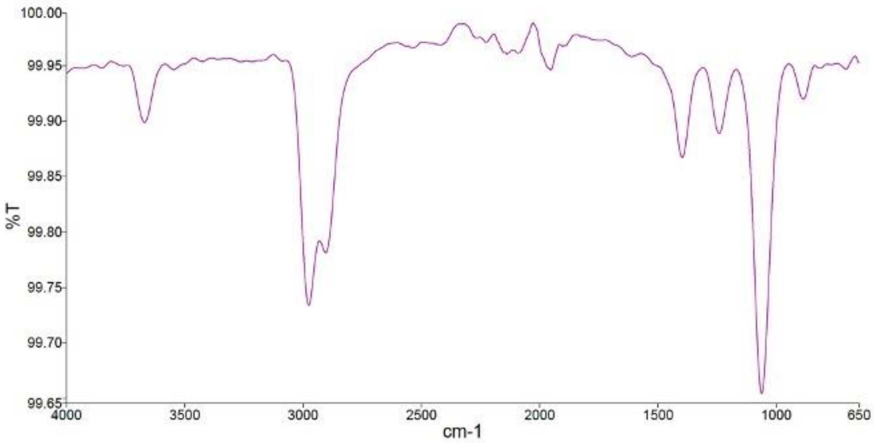

Figure 3 : FTIR spectrum of raw *Sargassum sp.* algae

We presume that the presence of hydroxyl groups will promote the affinity of algae for water molecules and by the way make the algae vulnerable to the basic pH of the cement matrix.

Specific density
According to the dealer, specific density of cement is 3 g/cm^3.
As shown by Figure 4, taking into account the standard deviations, specific density of algae (filler or fiber) is lower than cement one.
Indeed, specific density of fillers is (1.998 ± 0.008) g/cm^3 and that of fibers is (1.662 ± 0.06) g/cm^3.

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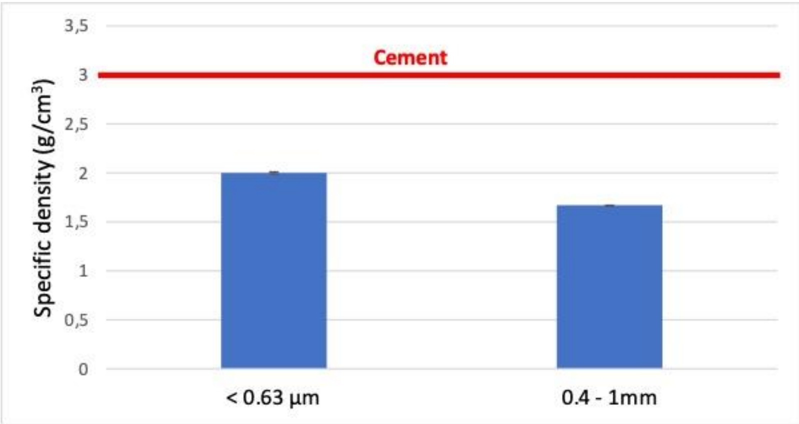

Figure 4: Specific density of *Sargassum sp*. algae according their diameters

Thermal degradation

Figure 5 shows raw *Sargassum sp.* algae thermogravimetric curve that is the same than the one observed by [8].

Analysis of this curve (Figure 5) reveals:

• A first region where there is a peak around 80°C corresponding to the evaporation of free water. The relative mass loss is 12%, value which is close to that observed by others [8, 18] who studied the same *Sargassum sp*. morphotypes;

• The second aera is located between 200 and 400°C. The most intense peak (200-300°C) is attributed to the enthalpic decomposition of carbohydrates and the second peak (320-400°C) is identified as the degradation of proteins [10]. It corresponds to 8% of relative mass loss;

• The third region is observed above 400°C where the relative mass loss is around 3%. The peak between 620 and 800°C is due to, according to authors [8, 15, 18], the decomposition of bioplymer or lignin-like compounds reported in various algae in response to stress condition as ultraviolet B radiation.

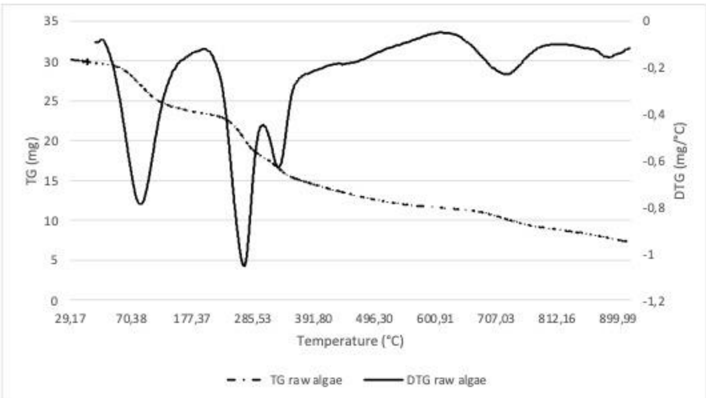

Figure 5: Thermal degradation of raw *Sargassum sp.* algae

3.2. Thermal characteristics of mortar composite materials

Determination of setting parameters.

These measurements have been conducted in order to determine the influence of the presence of algae as fillers on setting of the cementitious matrices.

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Table 4 presents the main results of setting.

	CP	CFAAB0	CFAAB2
Setting temperatures (°C)	44,44	44,43	40,36
Setting time (h)	13	17	18

When comparing CP and CFAAB0 data, the presence of fillers does not influence the setting temperature of cement but it has a setting retarder effect because there is an increase in setting time. Whereas, in presence of reinforcement, there is a decrease of setting temperature of cement and the synergy between filler and reinforcement still has a setting retarder effect.
It appears that *Sargassum sp.* filler seems to be a set retarder. This fact has been established by [19]. According to the same authors, hydroxyl groups found in algae (see 3.1.) have been commonly found to retard cement hydration.

Thermal degradation
According to the literature [20-23], during the hydration of cement, two major products are formed: hydrated calcium silicates (C-S-H), responsible for the enhancement of mechanical properties of cementitious matrix and portlandite ($Ca(OH)_2$).

As the observations at 7 and 28 days are similar, we choose to only present the results obtained at 28 days. Thermogravimetric analysis of composite materials (Figure 6) highlights three main zones:

- Zone 1 between 30 and 220°C, attributed to the departure of water and the decomposition of hydrated calcium silicates (C-S-H) [22];
- Zone 2 between 400 and 500°C, attributed to the decomposition of portlandite ($Ca(OH)_2$) [22];
- Zone 3 between 550 and 750°C, attributed to the decomposition of calcium carbonate ($CaCO_3$) [22].

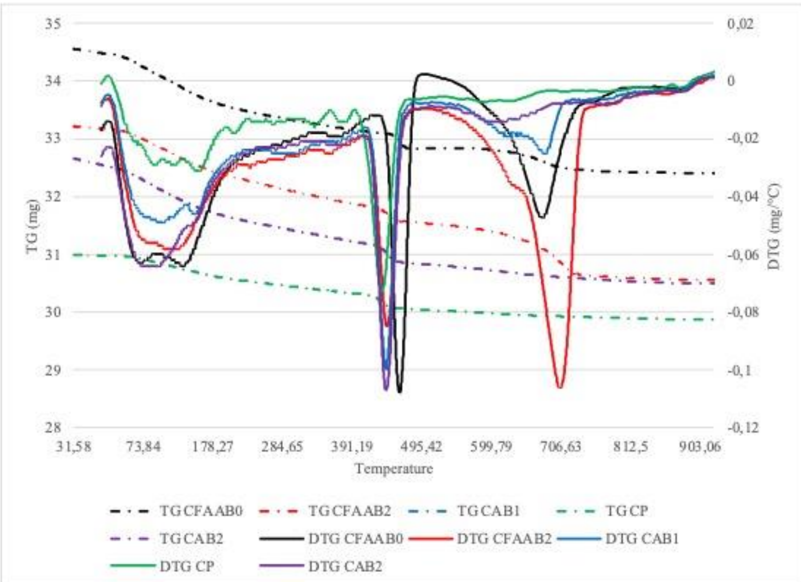

Figure 6: Thermal degradation of cement (CP) and composites

We assume that the peaks normalized areas are proportional to the magnitude of the mass loss. So, the peak areas were normalized, taking into account the mass of sample introduced, and the results of normalization for each peak temperature (minimum of the peak) are reported in Table 5.

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Table 5: Normalized peak areas (%) of cementitious matrix composite materials at 28 days

Material	Zone 1 30-220°C	Zone 2 400-500°C	Zone 3 550-750°C
CP	~100°C 0.9%	~441°C 0.7%	
CFAAB0	~80°C 1.6%	~468°C 0.7%	~680°C 0.8%
CFAAB2	~85°C 1.4%	~449°C 0.5%	~708°C 1.6%
CAB1	~84°C 1.2%	~447°C 0.7%	~685°C 0.2%
CAB2	~100°C 1.4%	~447°C 0.7%	~614°C 0.7%

Comparison of the surfaces of the different zones of samples CP, CAB1, CAB2 shows the influence of the presence of algae as reinforcement on the degradation of materials because there is an increase in the weight loss. The same fact is observed when algae are used as filler (comparison of CP, CFAAB0 and CFAAB2).

More precisely, we can affirm that the addition of algae (filler or reinforcement):

- has an influence on the content of C-S-H formed (zone 1);
- induces no change on the content of Portlandite (zone 2).

In the issue of building and construction materials, this means that *Sargassum sp*. algae, although generating more C-S-H, would not contribute to an improvement in the mechanical performance of cement.

When algae are used as filler, in the case of CFAAB2, there is a larger influence on the $CaCO_3$ content in comparison with CFAAB0. It would mean that the presence of 2% wt. of *Sargassum sp.* filler would help improve the rheology of the mortar.

Thermal conductivity

Figure 7 presents, at 7 and 28 days, evolution of thermal conductivity of composite materials according of content of *Sargassum sp.* filler and/or %wt content of *Sargassum sp*. reinforcement.

(a) 7 days (b) 28 days

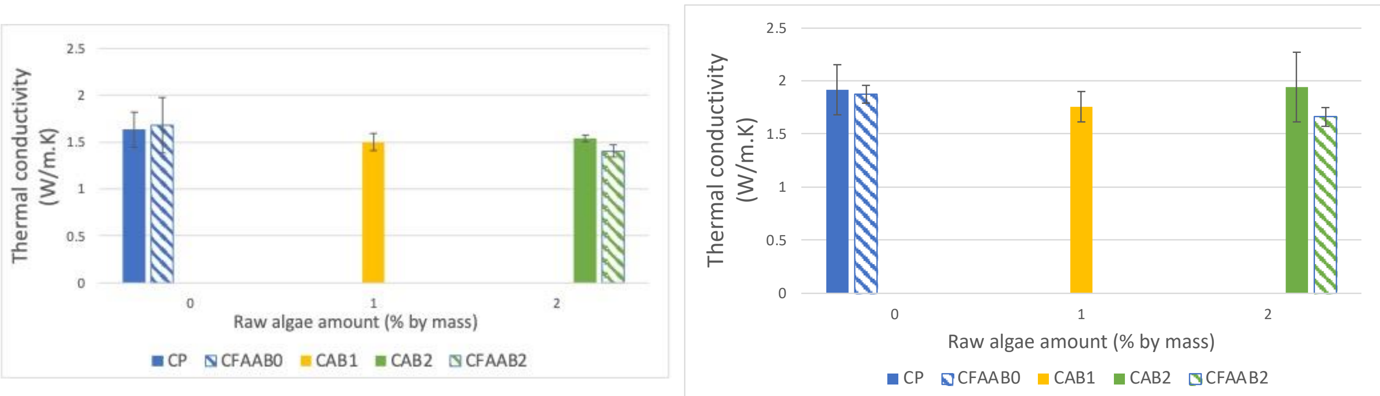

Figure 7: Evolution of thermal conductivity according to the presence of Sargassum sp. as filler and/or reinforcement at 7 days (a) and 28 days (b)

In this study, thermal conductivity of pure cement mortar is approximately l = 1,63 W/m.K. This value is in the range of those reported by Khushefati et al., [24] i.e. for mortars, thermal conductivity ranges between 0.53 and 2.5 W/mK.

Other authors [25] established that thermal conductivity of dehydrated *Sargassum fluitans* and *Sargassum natans* is l = 0.346 W/mK.

Figure 7 shows that no content of raw algae (filler and fibers) has any insulating effect on cement paste, according the standard deviation; not even a simple law of mixtures is followed by thermal conductivity of composites.

When adding raw algae to cement, the authors expected, like the plant fibers reinforcement, more insulating composites with a purpose to apply them as insulating element in housing.

Furthermore, there is an increase of thermal conductivity values between 7 and 28 days which means that *Sargassum sp.* fibers/filler are not thermal insulating components.

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According to Khedari et al. statement [26], "the thermal conductivity of porous medium (such as cement) is inversely proportional to the voids in the specimen". Thus, *Sargassum sp.* algae would not generate pores in cement paste allowing air to circulate and therefore ultimately reducing the thermal conductivity of the composite materials [3].
Asadi et al. [27] confirm that the voids inside concrete and the type of admixture have an important effect on the thermal property of concrete.
In the issue of building and construction materials, Sargassum sp. raw algae don't contribute to obtaining insulating materials.

Specific density
Specific density of cement paste and composites is presented by Figure 8.
We remind that specific density of filler and reinforcement are 2 and 1.5 g/cm^3, respectively (see Figure 4).
According standard deviation, when *Sargassum sp.* are used as reinforcement and filler, the algae have no influence on specific density of cement paste because this specific density is the same with or without algae.

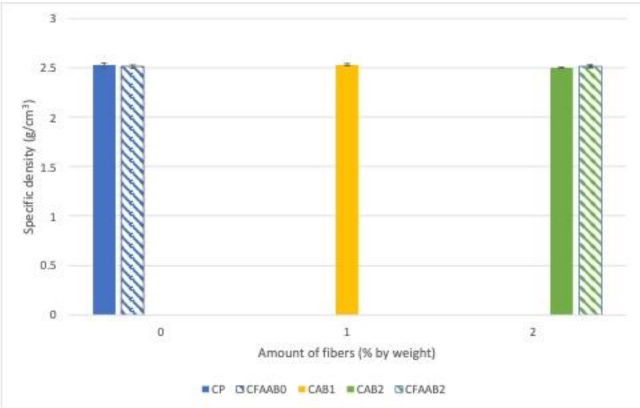

Figure 8: Specific density of composites materials (28 days)

These data are in agreement with poor thermal conductivity results (see Figure 7). If *Sargassum sp.* algae behaved like plant fibers in cementitious matrix, we would observe a decrease in specific density.
This expected decrease would be linked to the appearance of pores or air voids [28].

Authors established the curve presented by Figure 9. It is the representation of thermal conductivity according to specific density for CP, CAB1 et CAB2 specimen (in order to evaluate the fiber content).

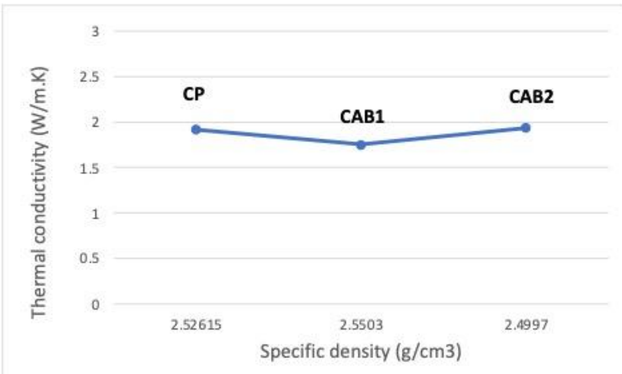

Figure 9: Experimental thermal conductivity according specific density of composites CP, CAB1 and CAB2.

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The linear correlation between the thermal conductivity k and the specific density r is:

$$k = 0.0112r + 1.848 \ (R^2 = 0.0122) \qquad \text{Equation (1)}$$

This linear correlation (Equation 1) is not consistent with classical equations applied to evaluate thermal conductivity of insulating materials used in the field of construction [3]

In the same way, Figure 10 was established. It seems that filler and 2% wt of fibers have a synergistic effect on thermal conductivity.

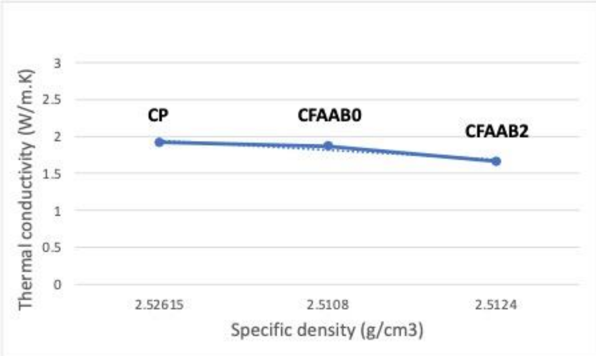


Figure 10: Experimental thermal conductivity according specific density of composites CP, CFAAB0 and CFAAB2.

In this latter case, the linear correlation between the thermal conductivity k and the specific density r is:

$$k = -0.1283r + 2.0731 \ (R^2 = 0.8779) \qquad \text{Equation (2)}$$

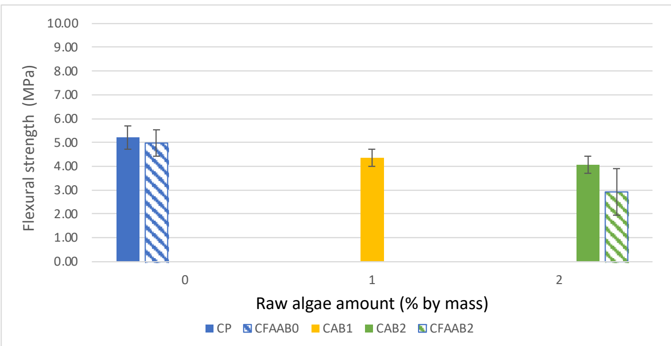
Equation (2) is in good agreement with equation of mortar containing silica fume, fly ash and blast furnace [27].

Mechanical properties: flexural and compressive strengths.

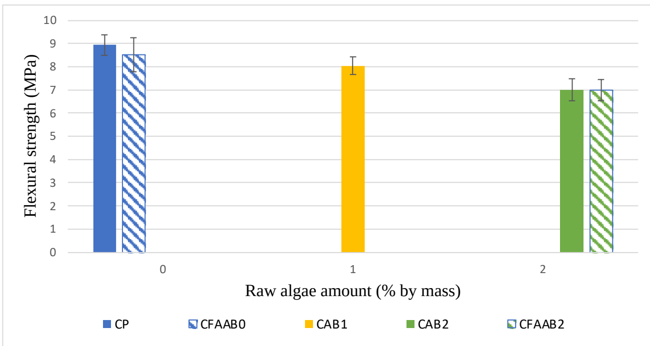
Figure 11 presents flexural strength of composites materials after 7 (a) and 28 (b) days of curing. Figure 12 presents compressive strength of composites materials after 7 (a) and 28 (b) days of curing.

"Tensile strength is an important property for cementations materials because it is their ability to resist deformation under load and the compressive strength is very important for building materials because it refers to the strength of the pillar to resist from fracture" according to [28].

In our study, whatever the use and the amount of *Sargassum sp.* raw algae (fillers or fibers), there is an increase of the mechanical properties. This behavior was expected because the hydration of cement produces C-S-H, C-A-H or C-A-S-H, compounds that improve the bonding strength between particles in cement paste and consequently improve the mechanical properties of the cement paste [29]. In our case, the production of C-S-H is confirmed by thermal degradation data.



(a) 7 days



(b) 28 days

Figure 11: Flexural strength of composites materials at (a) 7 and (b) 28 days

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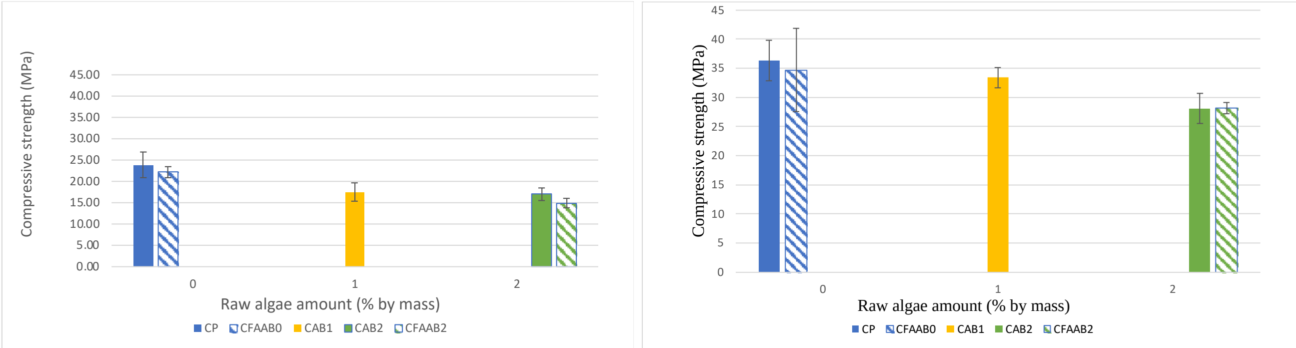

(a) 7 days (b) 28 days

Figure 12: Compressive strength of composites materials at (a) 7 and (b) 28 days

But, at 28 days, it appears that the addition of raw algae filler or fibers decreases both flexural and compressive strengths of composites materials according standard deviation. This behavior is contradictory with the use of these composites in the building sector.

These unexpected data could be explained by (1) the fact that there is a loss of adhesion in fiber/matrix interface [28] or (2) the fact that there is a lack of distribution/poor orientation of *Sargassum sp.* raw algae fibers or (3) that the amount by weight of fibers is not sufficient or (4) the increase of voids in the samples due to the air entrainment [28]. This last hypothesis is ruled out since according to the Figure 8, the specific density of the composite materials hardly varies.

4. CONCLUSIONS AND PERSPECTIVES

The purpose of this study an attempt to valorize *Sargassum sp.* raw algae as reinforcements (fibers) or additives (fillers) to cementitious matrices in order to apply those composite materials to the building & housing sector. Therefore, influence of different amounts of *Sargassum sp.* raw algae (fillers or fibers) on cement mortar physico-chemical, thermal and mechanical properties were investigated.

We remind that we expect more insulating materials than cement paste with, at a minimum equal density and mechanical properties. The main points may be concluded from the results obtained in this work as follows:

(1) Adding Sargassum sp. raw algae (fillers or fibers) does not contribute to lightweight composites;

(2) From a thermal point of view, *Sargassum sp.* filler are set retarder, fact that could be useful on construction sites. As shown by Figure 10, fillers combined to 2% wt of fibers may be promising in the construction field. When adding algae as fibers, there is an increase in thermal conductivity leading to less insulating materials;

(3) When correlating thermal conductivity to specific density of composite materials, the linear Equation 1 is not consistent with the application of these materials in the building sector;

(4) Tensile and compressive tests reveal that *Sargassum sp.* raw algae presence degrades mechanical properties in comparison with cement paste.

In conclusion, the authors argue that algae (as used in this study) is not promising as additive or reinforcement of building composite materials.

As perspectives,

- based on the study of Cengiz et al. [30], we could envisage the use of nanofibers derived from *Sargassum sp.* raw algae as reinforcement of cement;
- we have to determine Sargassum sp. raw algae fibers mechanical characteristics. If they prove promising, it would be interesting to increase fiber content.

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