



# Effects of aggregate coating on the hygral properties of lignocellulosic composites

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## ABSTRACT

Sugar beet pulp may be used as a vegetal aggregate for insulating lightweight lignocellulosic concretes. Yet, sugar beet pulp-based concretes display a number of dimensional variations, as well as problems with set retarding, owing to both the hydrophilic character of the pulp and the organic compound secretion into the cement matrix. The aim of this work consists of conducting physicochemical treatment to minimize the hydrophilic nature of sugar beet pulp so as to avoid the kinds of issues noticed when using untreated pulp. On the whole, materials made with treated pulp show greater water resistance and smaller dimensional variation.

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## 1. Introduction

The industrial sugar production process and, in general, any agro-based industry generates large volumes of waste, effluent and byproducts. In the past, these volumes were often discharged or reused without preliminary treatment as animal feed or fertilizers. Over the last few years however, due to the increasing necessity of incorporating environmental pollution prevention measures as well as for economic reasons, for government regulatory policy compliance and to satisfy energy and new material conservation requirements, new methods and policies for waste handling and treatment have been introduced into the recovery, bioconversion and reutilization of valuable components from food processing waste [1,2].

These byproducts must be treated or recycled in order to obtain useful products of higher value, or even as raw materials for other industries. One such byproduct is sugar beet pulp, which in France represents approximately 1,300,000 tons of dry matter per year. It is currently dried and pelletized for use as cattle feed and, in so doing, yields a small profit.

For a number of years, continuous interest has been shown towards finding new applications for sugar beet pulp and moreover several studies have been undertaken along these lines [3]. Such efforts have involved: making paper pulp from beet pulp [3,4], using beet pectin as a gelling agent in the agribusiness or cosmetic

industries [5], introducing beet pulp as a bioadsorbent for heavy metals removal in industrial water treatment [6], synthesizing vanillin [7], and producing energy in the form of biomass fuel (bio-ethanol, ETBE, etc.) [8,9]. Within this framework, one potential avenue for enhancing beet pulp value would be to use it as a vegetable aggregate in lightweight lignocellulosic concrete.

The principles of Sustainable Development and Green Building have been spreading through the construction industry at an accelerating pace in the last few years. More than ever, the construction industry is now focused on improving the social, economic and environmental indicators of sustainability [10,11]. Due to a continuous reduction in both artificial and mineral aggregate availability, the use of waste materials and byproducts has become a potential alternative within the construction industry. In this respect, a potential tool and strategy for meeting the environmental challenges could include the use of vegetable aggregates as a possible input material or replacement material in construction. Such a strategy offers the double advantage of creating new reuse opportunities for these byproducts while preserving natural resources [10,12].

The use of agricultural byproducts in concrete has led to a lightweight aggregate concrete class called lignocellulosic concrete. The scope of application for this type of concrete includes: cement boards, roof decking, concrete blocks, panels, and highway and road sound barriers.

Throughout the world, research has been undertaken to develop the use of vegetable byproducts in cement composites [13–15]. Some of this work has demonstrated that lightweight lignocellulosic concretes obtained using vegetable aggregates have thermal and acoustic attributes as well [16–18]. Among the already existing

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products on the French market it is particularly advisable to quote the aggregates of Agresta company. These aggregates are elaborated with chips or sawdusts of treated woods, and the concretes elaborated with these loads which have a low bulk density ( $600 < \rho < 800 \text{ kg/m}^3$ ) show a thermal conductivity ( $\lambda$ ) which lies between 0.11 and 0.16 W/m K [18]. Mixed into cementing matrices, beet pulp aggregates make it possible to obtain insulating or bearing insulating concretes. With a same bulk density the performances of the concretes containing beet pulps are competitive ( $0.16 \leq \lambda \leq 0.19 \text{ W/m K}$ ) [19]. Yet it is indeed rare when adding vegetable matter to a cement matrix has not caused problems.

The first difficulty encountered relates to compatibility between the cement matrix and lignocellulosic aggregate. Some of the compounds present in vegetable cell walls exert a set-retarding effect or, depending on their concentration, induce a set-inhibiting action on cement hydration, resulting in a weakening of the ultimate mechanical strength of the composite [20–22]. Another difficulty to be considered pertains to the dimensional variations of composite materials. Lignocellulosic aggregates swell with changing moisture content. This process is reversible and the aggregates shrink as they lose moisture [23,24]. These variations are likely to create pathologies within the composite material at both the macroscopic scale, due to the stresses developed, and microscopic scale, as a result of microcracking that potentially allows aggressive agents to penetrate.

To avoid these harmful effects, various types of treatments may be applied to either the cement matrix or the vegetable aggregate [18]. The treatments of vegetable aggregates are: thermal processes with high temperature and pressure [25], chemical processes [26] and mineral or organic coating processes [27]. Since the first treatments are energy consuming and the second treatments use is not very environmentally friendly, reagents coating process has been chosen to be applied to beet pulp. The two kinds of coating used were: mineral (cement) and organic (linseed oil). The purpose of this paper is to assess the impact of coating treatments introduced, with the aim of minimizing the influence due to the hydrophilic nature of beet pulp on the hydric performance of lignocellulosic composites containing beet pulp.

## 2. Materials and methods

### 2.1. Materials

#### 2.1.1. Beet pulp (BP)

The beet pulp supply (Fig. 1) has been provided by the USICA Beet Pulp Processing Trade Association in dried shreds of up to 20 mm in size.



Fig. 1. Beet pulp.

#### 2.1.2. Cement

A CPA CEM I 52.5 Portland cement, compliant with the NF EN 197-1 Standard [28], was used for treating beet pulp and manufacturing all samples.

#### 2.1.3. Water

Water absorption was measured by means of immersion in distilled water. Drinking water from the local water supply network was used in the composite manufacturing process. The calcium content of water introduced had already been measured at the LTI Laboratory [29], recorded at rates of  $100 \pm 2 \text{ mg/L}$ . The water is thus classified as very hard, according to the NF EN 1008 Standard [30].

### 2.2. Beet pulp treatment

Treatments applied to the beet pulp involved a surface coating, for purposes of: isolating the aggregates, making them inert with respect to external agents, and attenuating their strong affinity with water. This surface coating step was carried out with a hydraulic binder (cement) and linseed oil, which is a siccative oil, that hardens quickly by oxidation with air to form a solid film [31].

Several preliminary tests were conducted in order to obtain the most complete and homogeneous coating of the external surface of these beet pulp shreds. At the conclusion of these tests, the water-to-cement (W/C) mass ratio was set at 1, and samples with beet pulp-to-cement (BP/C) mass ratios of 0.8, 1.0 and 1.2 were produced. Application of the coating was followed by a curing period of 2, 7, 14 or 28 days (a step necessary to ensure hardening of the binder on treated pulp), in a climatic chamber at 20 °C and 98% relative humidity. For the linseed oil treatment, beet pulp-to-linseed oil (BP/LO) mass ratios of 1–3 were used. This treatment continued with a polymerization time for the linseed oil film extending 7, 14 and 21 days under environmental conditions and at a temperature of 50 °C.

### 2.3. Beet pulp characterization

#### 2.3.1. Microstructural morphology

The microstructural morphology of these coating layers was studied by means of scanning electron microscopy (SEM) using a PHILIPS FEG XL 30 microscope.

#### 2.3.2. Water absorption capacity

The water absorption capacity of these aggregates was determined gravimetrically by applying the following expression:

$$W_t (\%) = 100 \times \frac{M_t - M_0}{M_0}$$

where  $W_t (\%)$  is the water absorption ratio at time  $t$ ,  $M_t$  the soaked pulp mass at time  $t$ , and  $M_0$  is the initial oven-dried pulp mass.

#### 2.3.3. Swelling

The formulation of concretes for a specific use is often based on the volumic proportion of its components. More, in the case of lignocellulosic aggregates, a pre-wetting is made to avoid the absorption of mixing water by the vegetable material. The result is generally a swelling of the lignocellulosic aggregates which disappear during the concrete drying. So, to preserve a good volumic proportion of the different phases in the hardened concrete, it is necessary to know the swelling rate  $S(\%)$ . This parameter can be calculated and presented as a relative percentage of dry apparent volume, based on the following expression:

$$S(\%) = 100 \times \frac{V_f - V_0}{V_0}$$

where  $S(\%)$  is the swelling,  $V_0$  the oven-dried apparent beet pulp volume,  $V_f$  is the water-saturated apparent beet pulp volume.

#### 2.4. Concrete sample characterization in the hardened state

##### 2.4.1. Concrete preparation

An aggregate pre-wetting time is required for the production of concretes using beet pulp. This time was set at 2 h for untreated pulp [32] and for pulp treated with linseed oil. A pre-wetting time of 5 min was adopted for pulp coated with cement, as discussed in Section 3.1.2 below.

After this pre-wetting period, cement was added and the batch was mixed at low speed until homogenization of the mixture. The remaining water (targeting a water-to-cement (W/C) mass ratio = 0.3) was then added. The  $40 \times 40 \times 160 \text{ mm}^3$  prismatic samples were unmolded after 24 h and preserved in a wet chamber (98% RH, 20 °C) for 28 days. They were ultimately dried at 50 °C until reaching constant mass. Drying the samples made it possible to eliminate free and weakly-bound water, thus resulting in maximum shrinkage of the beet pulp concrete samples.

##### 2.4.2. Dimensional variations (DV)

The dimensional variations that accompany climatic variations are indeed reversible. They are due to exchanges with the external medium [33] and the presence of cracks furthers the propagation of water in concretes and can modify the exchanges on the surface.

The material can undergo repeated cycles of swelling and shrinking due to the hydrous gradients existing between the material and the external medium. These deformations can generate cracking, which in turn affects material durability.

A determination of the extreme dimensional variations (EDV) between the dry and water-saturated states could subsequently be carried out. These testing conditions correspond to the transition from a dry climate to accidental contact with water. In practice, such tests are performed by measuring the kinetics of dimensional variations of concrete specimens sized  $40 \times 40 \times 160 \text{ mm}^3$  using a Controlab®, NF P 15-433 [34] and NF P 18-427 [35] retractometer, which offers a variation accuracy of  $10^{-3} \text{ mm}$ . Results are expressed in terms of millimeters per meter (mm/m).

##### 2.4.3. Weight variations (WV)

The weight variation kinetics of soaked samples can be calculated by applying the gravimetric method in conjunction with DV measurements. These values must then be incorporated into the building structure design.

### 3. Experimental results and discussion

#### 3.1. The effect of treatment on the hydrous properties of beet pulp

SEM observations indicate that untreated beet pulp shreds have a rough surface (Fig. 2a), which should allow for good adherence between the aggregates and the cement matrix. The enlarged SEM images (Fig. 2b) reveal the conducting vessels formed by secondary xylem of the beet root. These vascular bundles provide the aggregate with its distinct heat and acoustic insulation.

SEM observations of treated pulp also indicate that the coating layer does not completely cover the aggregate surface (Fig. 3a), hence treatment should not completely control the hydrophilic nature of the pulp. Fig. 3b shows that the treatment matter does not penetrate into the pulp conducting vessels. These treatments thus maintain the intraparticle air that gives rise to the insulating characteristic of pulp aggregates.

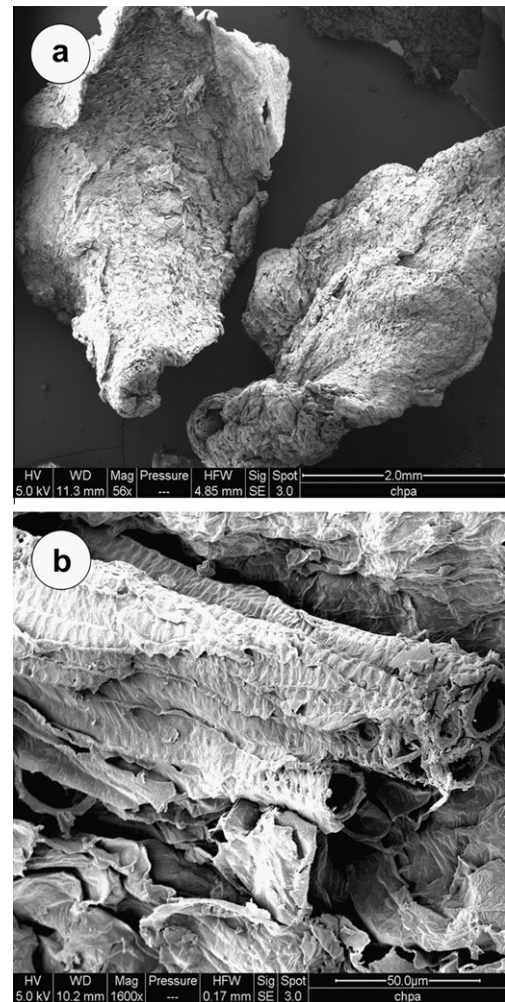


Fig. 2. SEM micrographs of untreated beet pulp.

##### 3.1.1. Saturation time in water immersion

An evaluation of treatment effectiveness was based on comparing the results of water absorption measurements, after 2 h of immersion, on both treated and untreated samples (Fig. 4). For each studied mix formulation, the water absorption values obtained correspond to the average of three tests.

For pulp samples treated with linseed oil, the water absorption value decreases as the BP/LO ratio decreases, whether at 50 °C or at room temperature. As of the third week, a greater drop in absorption at 50 °C than at room temperature starts to appear. A significant temperature influence on the polymerization reaction can also be observed as of the third week. Polymerization is accelerated by temperature: this finding is very valuable since during mixing or during immersion in water for absorption measurements it leads to fewer rejections of oil in water as well as to a gummier and non-oily pulp, which should improve the compatibility between pulp resulting from this treatment and the cement matrix during materials development. The oxidation of linseed oil is indeed faster at higher temperature, yet the reaction is exothermic and can trigger spontaneous combustion. It is therefore recommended not to use this oil at temperatures above 60 °C [31].

Similarly, the reduction in water absorption capacity from beet pulp is improved by the cement coating treatment; this reduction increases with a higher proportion of binder. Storage conditions appear to exert an influence, albeit a weak one. On the other hand, the length of sample storage in the curing chamber has a signifi-



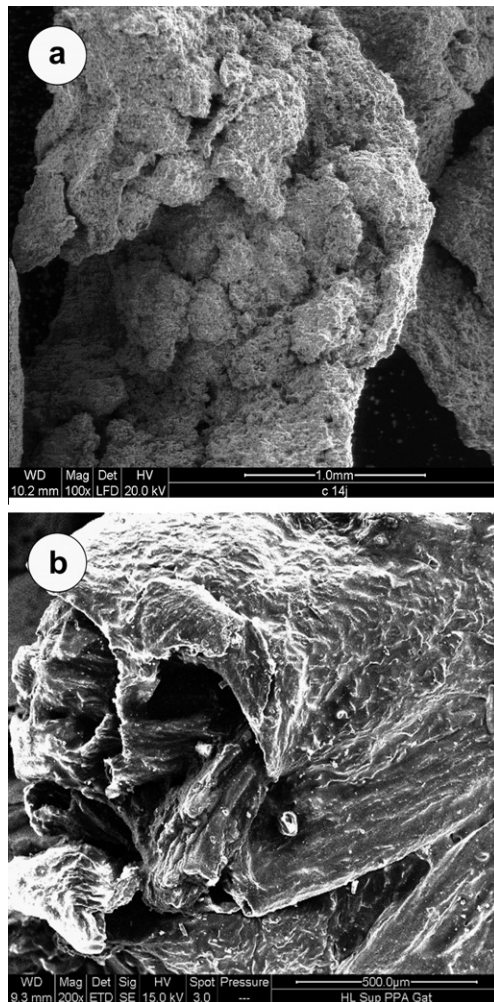


Fig. 3. SEM micrographs of: (a) cement-coated beet pulp (100× magnification), and (b) linseed oil-coated beet pulp (200×).

cant impact on water absorption, as shown in Fig. 5; this impact might be explained by the gradual development of hydrates.

These two treatments offer similar effectiveness with regard to controlling water absorption: they are able to divide by three the beet pulp water absorption capacity. Values close to 80% by weight of dry matter are obtained for the optimal formulations, which consist of:

- linseed oil coating: BP/LO = 1; polymerization time = 21 days at 50 °C,
- cement coating: BP/C = 0.8; W/C = 1; curing in a climatic chamber for 28 days.

### 3.1.2. Kinetics of water absorption by immersion of the treated pulp

The evolution in beet pulp saturation rate of absorption after treatment has made it possible to establish a measure of the effectiveness of each treatment applied. This task has been supplemented by a study of the kinetics of treated pulp water absorption. The curves representing the water absorption kinetics for pulp resulting from the various treatments applied have been displayed in Fig. 6.

The water absorption kinetics of pulp coated with cement is very fast as of the beginning of immersion. After just 2.5 min, 80% of the saturation rate is reached, with saturation (at 82%) occurring after approximately 30 min of immersion.

The pulp treated with linseed oil exhibits a much slower rate of water absorption. After 5 min of immersion, the absorption rate equals only 40% of the maximum rate. Stabilization starts to occur 2 h after immersion and saturation (96%) requires several hours.

The differences obtained in beet pulp water absorption kinetics following these treatments must be taken into account when studying concrete mix design, especially as regards introducing pre-wetting of aggregates prior to the materials development stage. The pre-wetting time for pulp has been set at 5 min for cement-coated samples and at 2 h for the linseed oil treatment.

### 3.1.3. Swelling

The absorption of water by pulp treated under the optimal conditions listed in Section 3.1.1 involves an increase in pulp particle volume, which significantly affects the level of dimensional variations in the eventual hardened concrete materials. Table 1 presents the apparent volumetric swelling of treated pulp, compared with the dry pulp volume as defined in Section 2.3.2

These treatments strongly decrease the amount of beet pulp swelling. The difference between apparent volumetric swelling values for water-saturated beet pulp, with respect to the treatment selected, corresponds roughly to the difference between the water absorption rates for treated beet pulp.

## 3.2. Effects on the hydrous properties of composites

### 3.2.1. Extreme dimensional variations (EDV)

Previous work [36] has shown that the formulations leading to optimal mechanical properties of concrete produced with treated pulp call for volumetric ratios of  $BP_{\text{saturated}}/C$  that equal 3 for cement coating and 2 for linseed oil coating, along with a W/C ratio of 0.3.

The evolution over time of dimensional variations (DV), starting from the dry state for composites prepared with these formulations, is displayed in Fig. 7 (and expressed in terms of millimeters of elongation per meter of material, mm/m). The final value of this evolution represents the extreme dimensional variation.

The materials prepared using untreated pulp ( $\rho = 570 \text{ kg/m}^3$ ) do not resist immersion in water. This low water resistance threshold can be explained by the high rate of absorption and swelling for this kind of pulp, and this might also be responsible for mechanical disorders.

The materials developed with both cement ( $\rho = 770 \text{ kg/m}^3$ ) and linseed oil ( $\rho = 750 \text{ kg/m}^3$ ) coated pulp exhibit prolonged resistance over time. The evolution in material DV presents a different behavior depending on the type of beet pulp treatment performed.

The composites made with cement-coated beet pulp reveal dimensional variations that stabilize at around 5.5 mm/m after just 1 h of immersion. This kind of concrete however demonstrates a remarkable improvement compared to those prepared using untreated pulp, which exhibit dimensional variations of up to 6.5 mm/m and which are only able to withstand 1 h of immersion prior to crumbling. Both types of composites show a similar evolution during the initial moments of immersion, but beyond the first 30 s, the materials derived with cement-coated pulp reached 50% of the EDV value and began to stabilize.

The materials prepared with linseed oil-coated pulp yielded a lower EDV value (1.3 mm/m) at a slower rate of evolution (i.e. stabilization did not start until 4 h after the beginning of immersion).

A significant difference has been found in the EDV values of composites produced using the various types of treated pulp, whereas water absorption capacities remain largely the same for pulp resulting from both treatments; moreover, the swelling of beet pulp coated with linseed oil is only slightly higher. This finding may be explained by the difference in pulp stiffness resulting from the two treatments; cement coating confers a certain rigidity

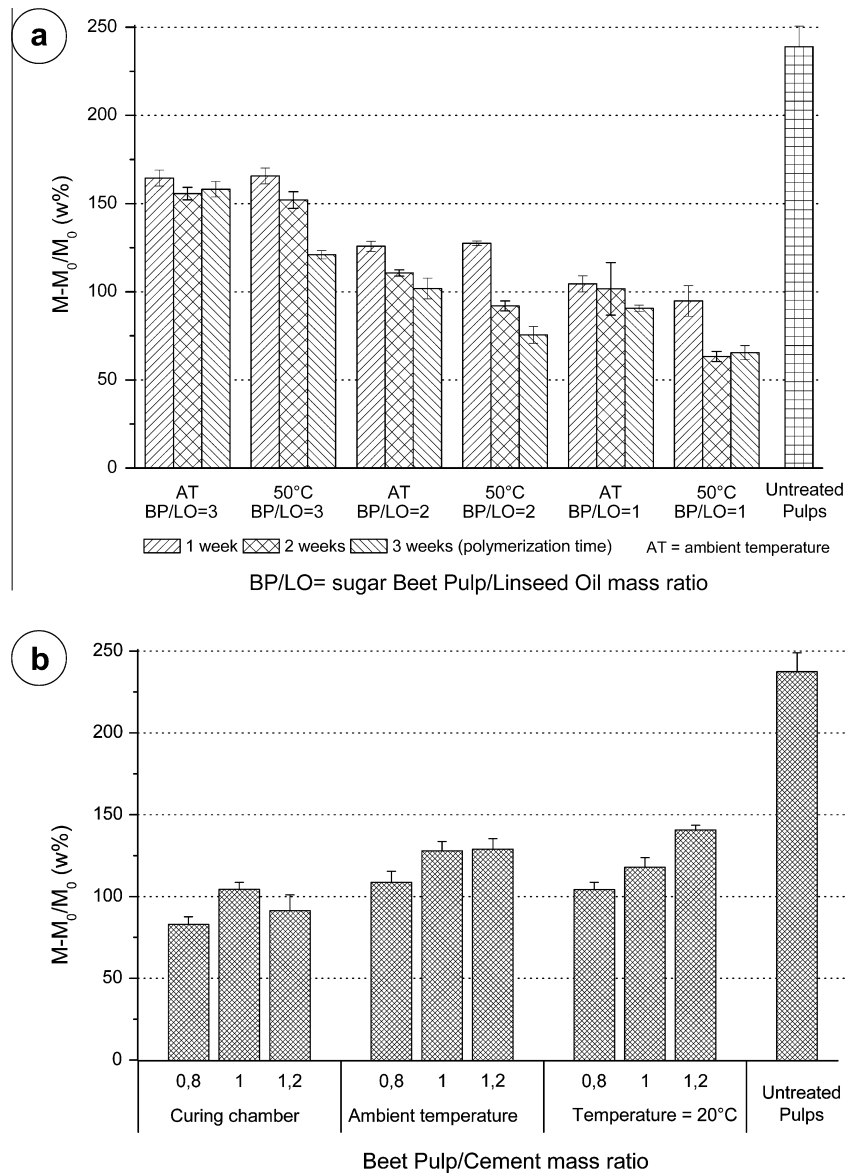


Fig. 4. Beet pulp water absorption after coating treatments using: (a) linseed oil, and (b) cement.

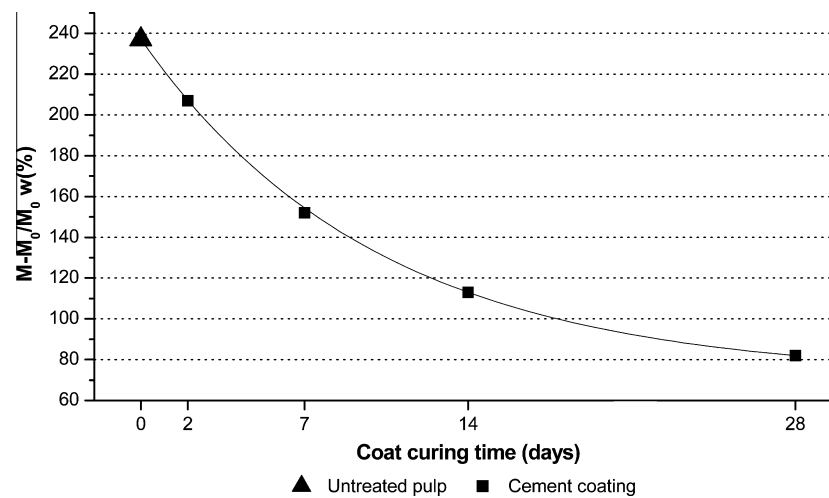


Fig. 5. Beet pulp water absorption capacity vs. cement coating curing duration.

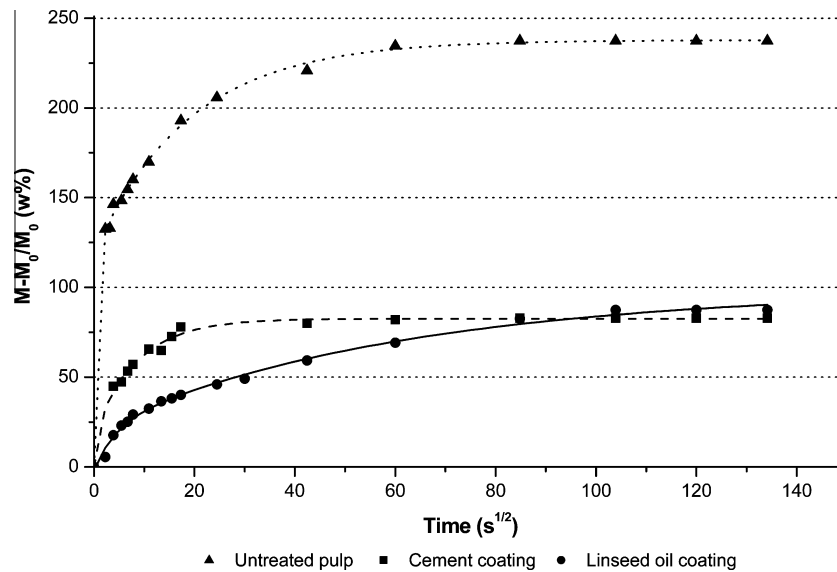


Fig. 6. Water absorption kinetics for the various treated beet pulp samples.

**Table 1**  
Apparent volumetric swelling of water-saturated beet pulp.

Treatment	Apparent volumetric swelling (%)
Untreated pulp	310 ± 8
Cement-coated pulp	72 ± 5
Linseed oil-coated pulp	87 ± 4

to the pulp. In contrast, pulp coated with linseed oil maintains its soft and internal porous structure. When the matrix is swelling due to water absorption porous and soft aggregates can buckle and partially absorb the generated mechanical stresses. Thus the level of composite deformation decreases.

### 3.2.2. Water absorption capacity and extreme weight variations (EWV)

Gravimetric monitoring of sample mass evolution during immersion has made it possible to study the water absorption kinetics of materials, in addition to calculating extreme weight variations (EWV).

The evolution in water absorption by materials, expressed as a percentage of the initial dry specimen mass, is depicted in Fig. 8. The type of treatment performed on the pulp serves to influence the water absorption kinetics of concretes prepared using such treated pulp.

It was not possible to measure a final value of the water absorption capacity for composites prepared with untreated pulp since the samples crumbled before reaching saturation. Crumbling occurred after one hour of immersion, when the specimen absorption rate had reached approx. 70%.

The EWV (extreme weight variation) of composites prepared using pulp treated with cement or linseed oil are both very close to 45%, although the time required for sample stabilization differed: 5 min for the cement-coated case vs. 2 h for pulp coated with linseed oil.

The beet pulp concretes studied herein are considered no-fines concrete (Fig. 9), meaning that they feature high porosity. These materials are well-known to be very good heat insulators and acoustic absorbents [18]. Observed behavior suggests that for the

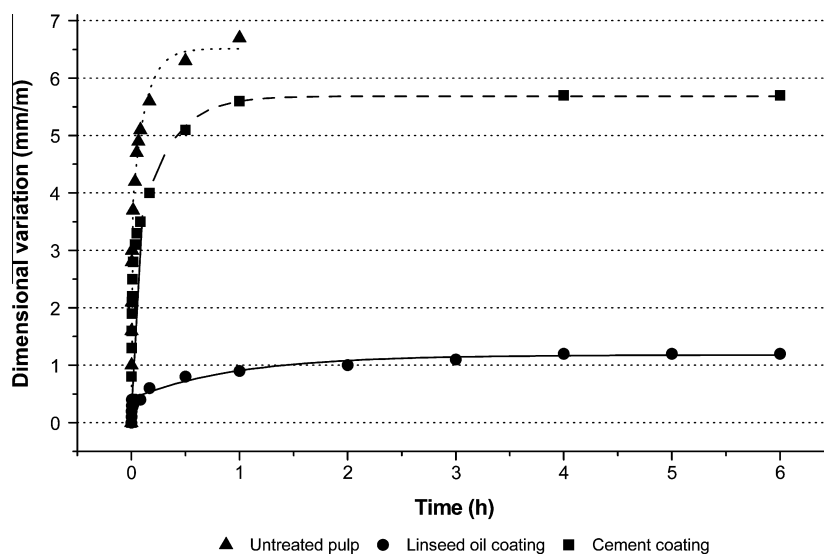


Fig. 7. Evolution in the dimensional variation of beet pulp concretes vs. immersion time, depending on the type of treatment applied to beet pulp aggregates.

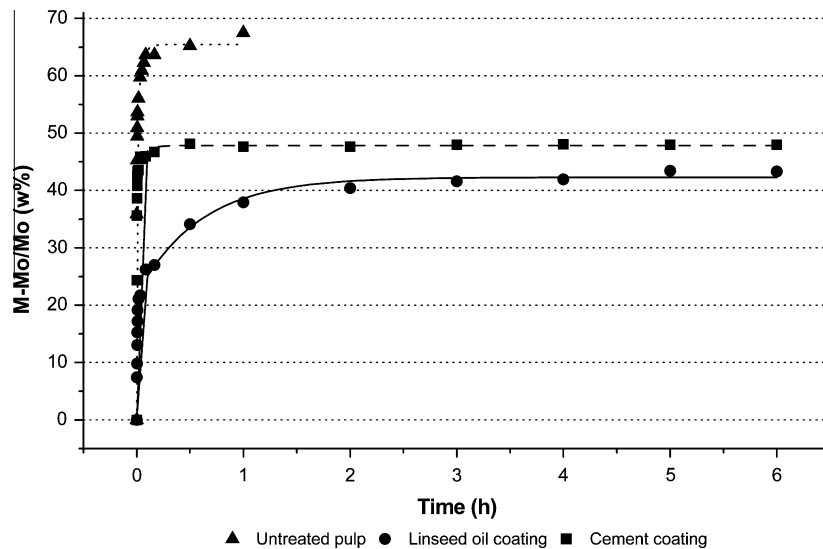


Fig. 8. Water absorption kinetics of concretes vs. type of treatment applied to beet pulp aggregates.

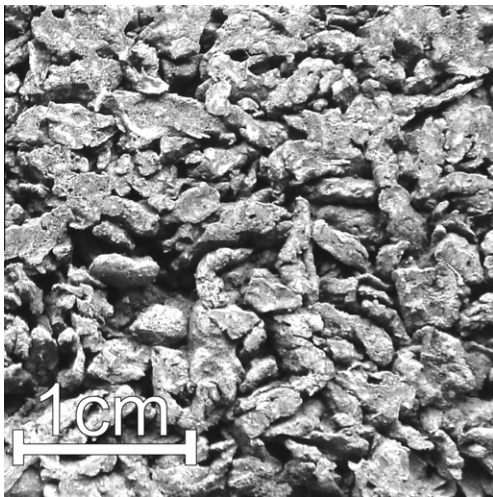


Fig. 9. The no-fines concrete appearance of cement-coated, sugar beet pulp concrete.

analyzed formulations, this high porosity enables rapid water entry into the material. Since the pores are easily accessible, they fill quickly with water, and the various types of coating layers then control the absorption rate until stabilization is reached. The saturation time of concrete made with cement-coated and linseed oil-coated pulp is actually very similar to what was measured in Section 3.1.2 for the pulp subjected to these same treatments.

#### 4. Conclusion

This work has been aimed at improving the hydrous properties of lightweight lignocellulosic concretes containing beet pulp as aggregates, by means of applying different treatments to the aggregates.

Moreover let us recall that the concretes of beet pulps were already mechanically studied [36]. The results showed that treated pulp enables producing materials with densities of less than 800 kg/m<sup>3</sup>, i.e. classified as LC 1.0 according to the EN 206-1 Standard [37], and with a mechanical compressive strength in the neighborhood of 2 MPa.

The materials made using treated pulp, in comparison with those containing untreated pulp, lead to significantly better hydrous performance. The reduction in both water absorption and swelling values allows for a greater incorporation of pulp into the materials development. The resulting materials display smaller extreme dimensional variations, especially in the case of concretes made with linseed oil-coated pulp, as well as better resistance to water over time.

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