Cement & Concrete Composites 55 (2015) 112–121

Contents lists available at ScienceDirect

Cement & Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

# Impact of hemp shiv on cement setting and hardening: Influence of the extracted components from the aggregates and study of the interfaces with the inorganic matrix

Youen Diquélou [a,b], Etienne Gourlay [c,1], Laurent Arnaud [c,2], Bernard Kurek [a,b,*]

[a] INRA, UMR614 Fractionnement des AgroRessources et Environnement, F-51100 Reims, France
[b] Université de Reims Champagne-Ardenne, UMR614 Fractionnement des AgroRessources et Environnement, F-51100 Reims, France
[c] Ecole Nationale des Travaux Publics de l'Etat – Université de Lyon, Département Génie Civil et Bâtiment, 3, rue Maurice Audin, F-69518 Vaulx-en-Velin Cedex, France

ARTICLE INFO

*Article history:*
Received 22 January 2014
Accepted 11 September 2014
Available online 20 September 2014

*Keywords:*
Hemp
Hydration
Retardation
Mechanical properties
Interface
Cement

ABSTRACT

The need for efficient building insulation with low environmental impact has led to a growing market for hemp lightweight concretes. However, a deeper understanding of the interactions between hemp particles and binders during the setting and hardening phase is needed to meet required industrial specifications. In this paper, the effects of three types of shiv and their corresponding water extracts on cement setting are compared. Various analyses show that these extracts delay hydration, thereby negatively affecting the mechanical properties of the cement. A comparison of the chemical composition of shiv water extracts highlights the importance of plant quality and the variability of their effects on cement. The development of a new methodology, combined with the use of $^{13}C$ labeled plant material, made it possible to closely monitor the physiochemical phenomena occurring at the plant aggregate and cement matrix interface.



## 1. Introduction

Shiv (also called woody hemp core) refers to fragments of the central section of dried hemp stalk (*Cannabis sativa*) (Fig. 1a). It is generally considered as a byproduct of long fiber and seed production and until now has been used mostly as animal bedding or mulch [1,2]. While the chemical composition of shiv is comparable to that of wood, it has a much lower density due to its porous microstructure (Fig. 1b). Because it has such low bulk density (110 kg/m$^3$), shiv is used as a natural aggregate to produce hemp lightweight concretes (HLC) (Fig. 1c) [2]. HLCs are made with shiv, water and a binder, with densities ranging from 200 to 650 kg/m$^3$ depending on the mix formulation (binder content). They are used for various applications in building construction such as walls and roofing, among others [3].

Various types of binders are used in HLCs, such as lime, hydraulic or pozzolanic binders (e.g., cement or pozzolan). These materials have small carbon footprints thanks to plant particle $CO_2$ storage [4]. It has also been shown that HLCs offer various interesting thermal and acoustic properties [5–9] depending on their mix formulations.

However, HLCs do have some drawbacks compared to conventional hydraulic concrete. In particular, their measured mechanical properties are low [9] and slow or weak setting is often observed onsite. These problems are generally worse when HLC is produced using a pure hydraulic binder like cement. HLCs are comparable to wood cement concretes but have lower densities (700–1200 kg/m$^3$ for wood cement concrete). Various studies have reported similar issues to those above when cement is mixed with wood [10–12] or other lignocellulosic residues like wheat straw [13], oil palm [14] or bagasse [15]. Based on setting monitoring of cement mixed with different species of wood, some researchers have suggested indexes to assess or predict wood/cement compatibility [16,17]. Predictive indexes take into account several parameters that are mainly related to the extractives (e.g., pH, extractives content and buffering capacity). In fact, extractives which correspond to easily solubilized molecules (e.g., monosaccharides, fatty acids or phenolic compounds) are thought to play a major role in the setting and hardening process [18,19]. Moreover, the high alkalinity of cement can cause lignocellulosic compounds to degrade and release byproducts, thereby impacting setting even more than simple extractable compounds do alone [20]. The setting impairment caused by these molecules

* Corresponding author at: INRA, UMR614 Fractionnement des AgroRessources et Environnement, F-51100 Reims, France. Tel.: +33 3 2677 3593; fax: +33 3 2677 3599.
*E-mail address:* bernard.kurek@reims.inra.fr (B. Kurek).
[1] Present address: CEREMA, BP9, F-67035 Strasbourg, France.
[2] Present address: ENSAM, F-71250 Cluny, France.

http://dx.doi.org/10.1016/j.cemconcomp.2014.09.004
0958-9465/

(extracts and/or degradation byproducts) could be explained by various phenomena like adsorption on hydrated or non-hydrated cement particles, ionic complexation or the formation of a thin barrier around cement grains by precipitation [21].

To circumvent setting problems when plant aggregates are mixed in cement paste, different strategies have been devised [22]. For example, lignocellulosic residues can be pretreated by solvent extraction or a coating process. Enhanced hydration and carbonation through the addition of lime and $CO_2$ injection has been shown to accelerate hardening; similarly, set accelerators can also be added to cement. Concerning HLCs, some improvements have also been made. Good practices have been compiled based on empirical evidence [3] and systematic mechanical tests have made it possible to define acceptable pairs of binder/shiv types that reach good performance thresholds and hence show relatively good compatibility. Finally, a projection process has been designed in order to improve the characteristics of HLCs through increased compaction and better water addition control [23]. However, despite all these advances, further progress is necessary for HLCs to meet the specifications required by industrial and final customers. For instance, HLCs must:

- Take into account the natural variability of hemp quality.
- Preserve their low density and insulation capacity.
- Be inexpensive and directly applicable onsite.
- Offer reasonably good and reliable mechanical properties.

To achieve these objectives, the physicochemical influence of shiv on the binder needs to be better understood. This study made use of two complementary approaches. First, the impact of water extracts on setting time, hydration rates and mechanical performances was investigated; second, the interface between shiv and cement paste was studied because it constitutes a key factor in the mechanical performances of concrete.

In Europe, and more specifically in France, most HLC binders used by building professionals are a mix of lime and hydraulic binder. This study focuses solely on cement because it is a good model for hydration reactions that are responsible for early strength. Additional studies currently underway on cement and lime mixes will later provide greater insight into the full setting phenomenon of classic HLC binders.

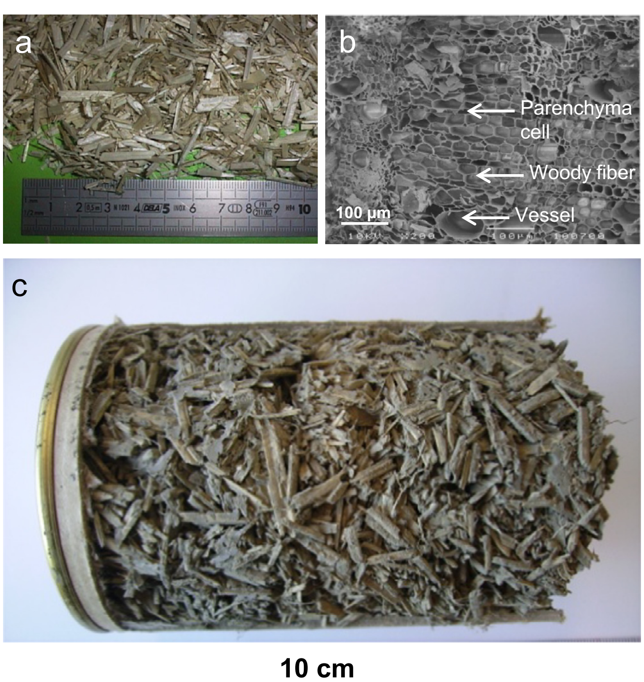


**Fig. 1.** Photograph of raw shiv particles (a); SEM micrograph showing shiv microporosity due to the organization of the different types of plant cells (b); photograph of a hemp cement concrete sample (formulated for wall application) (c).

## 2. Materials and methods

### 2.1. Raw materials

#### 2.1.1. Cement

The cement used in this study was Portland cement CEM I 42.5 R-HS (Schwenk). The mineral composition of the cement was determined by X-ray diffraction. Potential phases were calculated using the Rietveld method (Table 1).

#### 2.1.2. Shiv

This study used three kinds of shiv issued from three long fiber production chains. They differ by the cultivar, date and method of harvest, and defibration process used.

- *CA Shiv*: Hemp grown in France. Plants were harvested at maturity and processed via hammer mill.
- *CB Shiv*: German hemp, dried onsite without cutting and harvested via combine harvester and processed via hammer mill.
- *CC Shiv*: Hemp grown in the UK. Plants were harvested before maturity, dew-retted, and processed via hammer mill.

These shiv types were chemically analyzed according to the techniques described in Section 2.2.1; results are shown in Table 2. Only minor differences in initial composition were observed.

### 2.2. Chemical analysis

#### 2.2.1. Shiv chemical analysis

Prior to analysis, the shiv was extracted in a Soxhlet apparatus with three successive solvents. The extraction time was 6 h with a liquid to material ratio of 26.6 ml $g^{-1}$. The three solvents used

**Table 1**
Potential phase composition as determined using the Rietveld method [24].

| Phases | % in mass |
|---|---|
| $C_3S$ | 43.40 |
| $C_2S$ | 25.80 |
| $C_3A$ | 0.50 |
| $C_4AF$ | 19.80 |
| CaO | 0.30 |
| Gypsum | 0.30 |
| $Ca(OH)_2$ | 0.00 |
| $CaCO_3$ | 2.40 |
| $SiO_2$ | 0.10 |
| Anhydrite | 3.70 |
| Bassanite | 1.60 |

**Table 2**
Chemical composition of the three types of shiv (in percent of dry weight). Ash can be included in two component classes: soxhlet extracts and others.

| Components (% of dry weight mass) | CA | CB | CC |
|---|---|---|---|
| Cellulose | 47.3 | 45.6 | 49.2 |
| Hemicelluloses | 18.3 | 17.8 | 21 |
| Klason lignin | 21.8 | 23.3 | 21.9 |
| Soxhlet extracts | 6 | 5.1 | 6.2 |
| Others | 6.6 | 8.1 | 1.6 |
| Ash content | 3.7 | 2.6 | 3.5 |