BIOSYSTEMS ENGINEERING 139 (2015) 87–99

Available online at www.sciencedirect.com

ScienceDirect

journal homepage: www.elsevier.com/locate/issn/15375110

Research Paper

Improved multiple cracking and autogenous healing in cementitious materials by means of chemically-treated natural fibres

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ARTICLE INFO

Article history:
Received 4 May 2015
Received in revised form
13 August 2015
Accepted 17 August 2015
Published online 1 September 2015

Keywords:
Flax
Hemp
Strain-hardening
Mercerisation
Autogenous healing

Natural fibres such as flax and hemp fibres are mainly used in the textile industry, but some have outstanding mechanical properties and have a great potential as reinforcement in cementitious composites, as an alternative to synthetic microfibres. However, due to their hydrophilicity the amount of multiple cracking that occurs in cementitious composites reinforced with natural fibres is reduced. Also, natural fibres may degrade in alkaline environments. Therefore, proper mixtures and multiple chemical treatments are required to improve natural fibre characteristics. The application of flax and hemp fibres in cementitious composites was examined, with a focus on inducing multiple cracking under tensile stresses. The mechanical properties were studied for the natural fibres and the cementitious composites. The degradation of the natural fibres in alkaline environments was also studied. Multiple cracking was achieved and further improvements were made by chemically treating the fibres. Mercerisation with a 2% (m/m) [NaOH] resulted in optimal multiple cracking. This multiple cracking resulted in small cracks widths, which allowed optimal autogenous healing when exposed to wet/dry-cycles. Natural fibres were thus found to be a suitable eco-friendly alternative to synthetic microfibres.

1. Introduction

1.1. Use of synthetic microfibres to induce multiple cracking

Synthetic microfibres have already demonstrated their ability to increase the durability of cementitious materials. As concrete is known to crack under tensile stress, fibres can be used as a reinforcing material. A proper mixture design can produce cementitious composites which exhibit tensile strain-hardening and multiple cracking, while minimising crack width (Li, Lim, & Chan, 1988; Li, Wang, & Wu, 1997; Snoeck, Van Tittelboom, Steuperaert, Dubruel, & De Belie, 2014; Wang, Backer, & Li, 1987; Yang, 2008). These small crack widths improve the durability and promote the autogenous healing of the cracks. This autogenous healing property was studied by Hearn (1998) in water retaining structures, i.e. culverts and pipes. He made a distinction between self-sealing due to blockage and self-healing due to further hydration. This

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http://dx.doi.org/10.1016/j.biosystemseng.2015.08.007

Nomenclature

AA	Acetic acid
AH	Acetylation (Acetic anhydride)
ANOVA	Analysis of variance
BISFA	Bureau International pour la Standardisation des Fibres Artificielles
BP	Benzoylperoxide
(C)	Storage in cement filtrate solution
CF	Cottonised flax
ε_{cu}	The strain at σ_{cu}
ε_{fc}	The strain at σ_{fc}
E	Modulus of elasticity
MC	Multiple cracking
N2, N6, N10	Alkalisation or mercerisation
NH	Non-retted hemp
PVA	Polyvinyl alcohol
ρ	Density
RH	Relative humidity
σ_{cu}	Ultimate bending strength
σ_{fc}	First-cracking-strength
S	Silane primer
SH	Slightly-retted hemp
TF	Technical flax
w	Crack width

is important as possible temporal self-sealing effects may not lead to a full recovery of mechanical properties. Autogenous healing was studied in detail by many other researchers (Edvardsen, 1999; Li, 2008; Li & Li, 2011; Snoeck & De Belie, 2012; Snoeck, Steuperaert, Van Tittelboom, Dubruel, & De Belie, 2012; Snoeck, Van Tittelboom, De Belie, Steuperaert, & Dubruel, 2012; Snoeck et al., 2014; Yang, 2008; Yang, Lepech, Yang, & Li, 2009). Crack healing occurs through the further hydration of non-hydrated cement particles, pozzolanic activity and mainly the precipitation of calcium carbonate crystals, as was shown by Edvardsen (1999).

1.2. *Natural fibres and their advantages and disadvantages*

Natural fibres may also be used to receive multiple cracking and autogenous healing. The use of natural fibres is very attractive for two main reasons. Firstly, the production of natural fibres can come at low cost and with a limited ecological impact. The concrete industry in particular is prone to produce high carbon dioxide emissions and it would therefore benefit from the use of renewable, eco-friendly natural materials. Secondly, some natural fibres have particularly good mechanical properties, as is especially the case for hemp and flax fibres. The use of these natural fibres in concrete compared to synthetic microfibres is therefore of great interest and is the subject of this study. A review of different types of available (natural) fibres can be found in Snoeck and De Belie (2015a).

Unfortunately natural fibres also have a few disadvantages, such as their high water absorption (Boghossian & Wegner, 2008) and their susceptibility to degradation (Agopyan, Savastano Jr., John, & Cincotto, 2005; Pacheco-Torgal & Jalali, 2011). This degradation can occur in several ways. Exposure to moisture can cause biodegradation after only three days, and, more importantly, cellulosic fibres can deteriorate in alkaline environments (Bentur & Akers, 1989; Gram, 1983; Stamboulis, Baillie, & Peijs, 2001) such as when incorporated in cementitious materials. The main polymers in natural fibres are cellulose, hemicellulose, lignin and pectin (Baley, 2002b; Bledzki & Gassan, 1999). The hydrophilic polymer hemicellulose is mostly responsible for water absorption (Davies & Bruce, 1998) and for thermal and biodegradation (Aziz, Paramasivam, & Lee, 1981; Sahed & Jog, 1999). Lignin and pectin are thermally stable, but they are responsible for UV-degradation (Sedan, Pagnoux, Smith, & Chotard, 2008). Also, the hollow section of the natural fibre, the lumen, may harden in contact with cement products producing so-called petrification (Savastano Jr. and Agopyan, 1999).

1.3. *Chemical treatment of natural fibres*

To overcome these disadvantages, the fibres may be chemically treated. Chemical treatment may reduce the amount of hemicellulose, lignin, pectin and natural oils covering the surface of the fibre, therefore changing their behaviour and roughness (Li, Tabil, & Panigrahi, 2007; Mwaikambo & Ansell, 2002). By chemically treating the natural fibres, the fibre surface is modified, the fibres are cleaned and most of the impurities are removed. This changes the synergetic interaction between the natural fibre and the cementitious matrix. A treatment of the hydrophilic fibres (due to their hydroxylic groups) to obtain hydrophobic properties, leads to a reduction in water absorption by the fibres and reduced biological deterioration in humid environments. Physical treatments, such as cottonisation may partly remove hemicellulose and pectin (Pickering, Beckermann, Alam, & Foreman, 2007; Snoeck & De Belie, 2012), leading to a reduction of the degradation in an alkaline environment. Some treatments may improve the mechanical properties and the interaction with the cementitious matrix due to the change in surface and roughness properties (Bledzki & Gassan, 1999). This may possibly lead to an increase in ductile behaviour and the extent of multiple cracking.

1.4. *Focus of the paper*

Two typical natural fibres were studied: flax and hemp. Flax is mainly used for linen, but it is also one of the strongest and most durable of natural fibres (Boghossian & Wegner, 2008). That is why it is so attractive for use as a reinforcing fibre in composite materials. Technical flax fibre has a fibre diameter of 50–100 μm but is composed out of elementary fibres with diameters in the range 10–20 μm (Bos, H., Van den Oever, M., & Peters, O., 2002). Elementary fibres are glued together by pectin and hemicellulose (Baley, 2002a). Cottonisation may partly remove pectin and hemicellulose, improving the overall susceptibility to degradation (Snoeck & De Belie, 2012).

Hemp fibre is strong and stiff and is already commonly used as a reinforcing material in the car industry (Pickering et al., 2007). The structure and architecture of hemp fibre is