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Fibre-matrix interface properties in a wood fibre reinforced cement matrix

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ABSTRACT: Wood fibres can be a low cost reinforcement for cementitious materials for structural applications. In order to design a ductile cementitious material reinforced with softwood fibres the fibre-matrix interface properties are studied. Pullout tests have been carried out to determine the bond strength and the influence in the pullout behaviour of the physical, chemical and mechanical properties of both the fibres and the cement matrix. Other fibre-matrix interface characteristics have been evaluated using an Environmental Scanning Electron Microscope (ESEM), a CT-scanner and optical microscopes. Matrix and fibre must be suitably designed in order to allow the fibre to be pulled out without rupture. A controlled mode of telescopic bonding during the pullout will be beneficial to increase the dissipated energy and develop ductility in the composite. Understanding the pullout behaviour in detail will be useful in the development of wood fibre reinforced cementitious materials.

1 INTRODUCTION

In order to turn a brittle cement matrix into a ductile composite different types of man-made fibres are currently in use: stiff fibres such as glass or steel and flexible fibres as polyvinyl alcohol, polypropylene and polyethylene fibres. However, natural fibres can be a low cost replacement for the energy intensive artificial fibres. Compared to these fibres wood fibres are more easily available worldwide as well as friendlier to the environment since they demand less energy to be produced and they are a renewable resource. However, the properties of the wood fibres are not as constant as those of artificial fibres: they shrink and swell in the presence of water and they have a lower tensile strength.

Because of the important roll that the fibre-matrix interface plays in the performance of fibre-reinforced composites, the aim of this paper is to characterize the interface properties in order to maximize the potential of wood bundles as reinforcement in cementitious composites The bonding will depend on: physical and chemical properties of the bundles, water/cement ratio of the matrix and its admixtures, curing conditions and age. Pullout tests have been conducted using seven different cementitious matrices with softwood bundles in order to determine both the bond strength and the pullout behaviour. For all matrices the cement has been partially replaced by other binders such as fly ash, blast furnace slag and silica fume in order to achieve a more sustainable composite. As pointed out by Kosmatka et al (1998) the use of by-products as supplementary cementitious materials will help to reduce both the energy consumption and greenhouse gas releases and additionally produces less waste. An additional benefit of using artificial pozzolans, like fly ash and silica fume, is the decrease of pH, which will make the cementitious matrix a more suitable environment for the wood fibres (Kettunen 2006).

In this study the effects of air curing and water curing in pullout behaviour are evaluated, as well as the influence of the fibre geometry. Beside pullout tests the fibre-matrix interface is also evaluated using optical microscopes, Environmental Scan Electron Microscope (ESEM) and CT-Scan images. The pullout tests results and the ESEM and CT-Scan images are presented and discussed.

2 MATERIALS AND METHODS

2.1 *Wood fibres*

In this paper softwood fibres from spruce are being studied as possible reinforcement for cementitious materials. Softwood fibres have been chosen for their mechanical properties and availability. Compared to hardwood fibres, softwood fibres are more uniform in size throughout the tree and they have a more simple form without vessels. Softwood fibres are also longer (3 to 5 mm) and thicker (up to 45 um). Nevertheless, the length of the single fibres was judged to be too short for the purpose of this study and therefore, instead of single fibres, naturally bonded groups of fibres have been used. These bundles have been cut to a desired length. Additionally, bundles have the advantage over single fibres that they have a rough surface, which may promote a better mechanical bond with the cement matrix. A disadvantage of the bundles is that they will achieve slightly lower tensile strength than single fibres.

In order to prepare the fibre bundles small blocks of spruce lumber pieces have been cooked following a neutral sulphite semi-chemical (NSSC) pulping procedure (Walker 2006) after which the bundles have been manually taken apart. Due to this pulping procedure the bundles' cross-sections vary in dimensions and shape. From now on the bundles will be referred to as fibres.

2.2 *Cementitious matrices*

Initially seven different cement matrices, of which the proportions per weight are shown in Table 1, were tested. In these matrices 55% to 74% of the cement content have been replaced by either pulverized fly ash of blast furnace slag BFS. The cement type is CEM I 42.5 N. In mixes M1 and M3 the sand size ranges from 0.125 –0.25 mm while for mixes M6 and M7 the sand particle size ranges from 0.125 – 2.5 mm. Limestone powder is considered an inert filler material (Zhou et al., in press). Silica Fume SF was added to mixes M6 and M7 to improve the particle size distribution and prevent bleeding. The superplasticiser Cretoplast SL-01 has been used to improve the workability of fresh cementitious material. The last column in the table shows the water-cementitious material ratio, β.

Table 1. Compression strength C.S. (MPa) and mix proportions (weight ratio)

Mix ID	C.S.	Cem	Fly Ash	Sand	BFS	Lim.	SF	β
M1	67	1	1.2	0.8				0.27
M2	21	1	2.8					0.26
M3	58	1		0.8	1.2			0.27
M4	N/A	1			1.2	0.8		0.27
M5	41	1			1.2	2.0		0.26
M6	28	1	2.0	3.0			0.05	0.39
M7	50	1		3.0	2.0		0.05	0.37

2.3 *Pullout tests*

To evaluate the fibre-matrix interface properties single fibre pullout tests have been carried out. A wood fibre with a controlled embedment length is pulled out from a block of cement matrix while the load vs. displacement relation is recorded (Li et al. 2005). The tests have been conducted at a speed of 0.002 mm/s. Two different sets of specimens have been prepared. In the first set the embedment length of the fibres is chose to be 1/10 of the fibre length. The authors consider this to be the minimum embedment length for the fibre to still have contribution to the overall material strength. The set was tested in a MTS 810 testing machine in which the displacement of the test is given by the displacement of the actuator (±80 mm stroke) and the load is measured with a 2 N load cell. In the second set the embedment length of the fibres is 30% of the fibre length. This set has been tested in a micro tension-compression testing device (developed by Kammarath & Weiss) in which the load is measured with a 50 N load cell and the displacement is given by the displacement of the actuator (±6 mm).

In both tests a free fibre length of 1 mm is left between the cement matrix and the plate in which the free fibre end is glued. All specimens have been uncasted after 24 hours and cut prior to testing at an age of 28 days. Some samples have been kept continuously in water while other samples have been air cured at laboratory conditions between 30 and 50 RH and about 22°C.

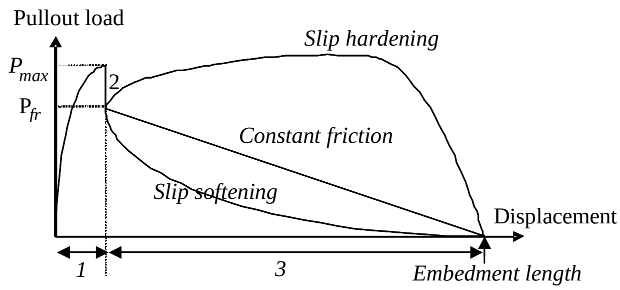

Figure 1. Schematic description of fibre pullout behaviour

A schematic pullout load-displacement curve in which three zones can be clearly distinguished (Redon et al 2001) is shown in Figure 1. In zone 1 a stable de-bonding process takes place between the fibre and the matrix until reaching a maximum pullout resistance P_{max} of the fibre. Then, at zone 2, the load decreases from P_{max} down to the pullout frictional load P_{fr}. The chemical bond between the fibre and the matrix is assumed to be broken if the drop of load is sudden. When the pullout frictional load P_{fr} is reached, fibres with a homogeneous cross section such as polyvinyl alcohol (PVA), polyethylene or steel, are considered fully de-bonded. However, for the spruce fibres with a variable cross section and irregular fibre surface this may not be the case yet. At zone 3 only frictional forces resist the pullout. Depending on the relative hardness of the fibre and the matrix as well as the curvature of the fibre in the cement matrix the fibre undergoes sliding with slip hardening, constant friction or slip softening effect.

2.4 *Microstructure analysis*

Optical microscopy (Leica M26), electronic scanning electron microscopy (ESEM, XL30 FEI) and computer tomography (CT-Scan Phoenix) have been used to analyse the fibre-matrix interface. With the aid of these devices it is possible to study different microstructure features such as the alignment of the fibre inside the cement matrix, the matrix penetration into the fibre and the gaps between fibre and matrix due to the different curing conditions. The microstructure characteristics have been correlated with the mechanical properties of the samples measured by the pullout tests.

3 TEST RESULTS AND DISCUSSION

3.1 *Cement matrix*

When comparing the overall results for the seven cement matrices, fibres embedded in M3 show the highest pullout stress for both water cured and air cured samples (Fig. 2). The air cured samples embedded in the cement matrix M2, achieved the lowest average values of pullout stress, while for the water cured sample the maximum pullout stress couldn't even be measured. Though M2 has the lowest compression strength, there seems to be no clear relationship between the pullout stress and the compression strength apart from just a tendency to improve the pullout stress if the compression strength increases.

In water cured conditions a higher pullout stress was reached with a cement matrix with 55% cement replacement by slag than with the same percentage replaced by fly ash.

Beside the pullout stress values the performed tests also give important information about the behaviour in the interface when the fibre is pulled out. A higher amount of energy is released if the fibre undergoes sliding with slip hardening during the pullout (Fig. 1). As explained by Sierra et al. (2008) a material that shows a strain-hardening behaviour with multiple micro cracks prior to failure develops enhanced ductility. The pullout of the fibre rather than its rupture will favour the development of ductility in the fibre-reinforced composites (Lin & Li 1997), and slip hardening behaviour will do so even more. In this sense the best pullout behaviour has been achieved with matrices M1 and M4. Even though some fibres embedded in M1 broke during testing the samples with this matrix have the higher percentage of slip hardening behaviour (29%). Some pullout curves with different behaviour are plotted in Figure 3. Of all the M4 samples 44% show an interface bond governed by friction (Fig. 4). There is no sudden drop of the load after reaching P_{max} and the area under the pullout curve suggests a higher amount of energy needed to pull out the fibre. On the other hand, though the tests with M3 reach higher pullout stress values the samples cured in water have the highest percentage of slip softening behaviour and the air cured samples had the highest percentage of broken fibres which is both unfavourable.

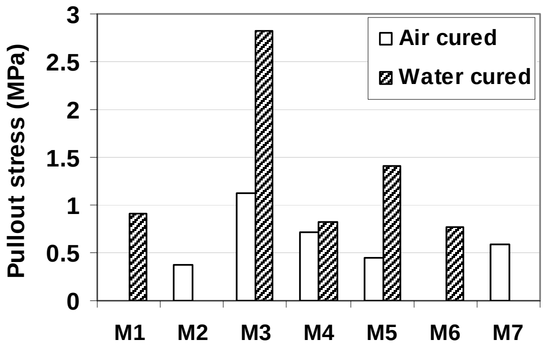

Figure 2. Pullout stress under different curing conditions

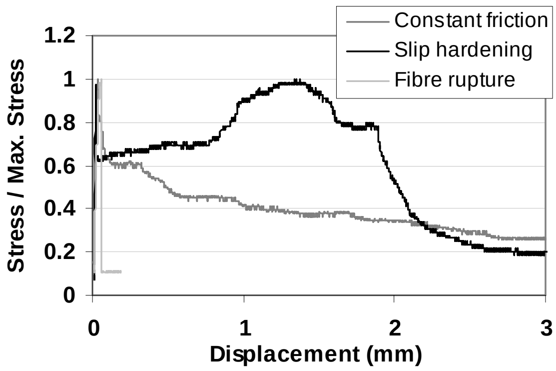

Figure 3. Pullout curves of M1 air cured samples

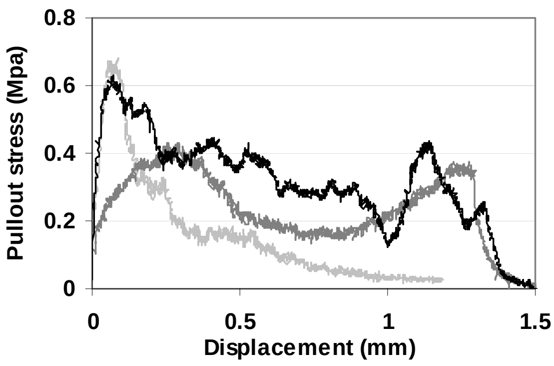

Figure 4. Pullout curves of M4, different curing conditions

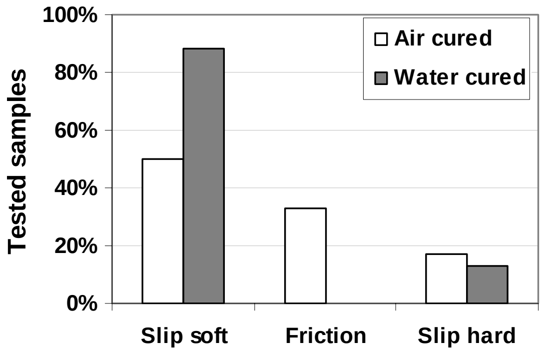

Figure 5. Pullout behaviour of M5, different curing conditions

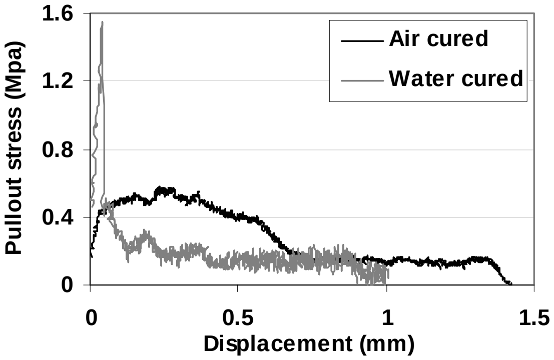

Figure 6. Pullout curves of M5, different curing conditions

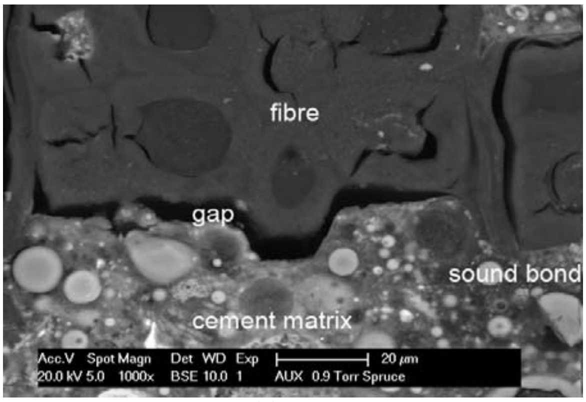

Figure 7. ESEM image of fibre embedded in M6, air cured

3.2 *Curing conditions*

The curing conditions have a strong influence on the pullout behaviour. The samples from matrices M3, M4 and M5 that have been water cured have higher pullout stress values than those that were air cured (Fig 2). The most observed pullout behaviour is slip softening for both curing conditions but the percentage is higher for water cured samples as shown in Figure 5 for M5. For this matrix 50% of the air cured samples and 88% of the water cured samples have a slip softening behaviour while one third of the air cured samples has a behaviour governed by friction. An example of these two different behaviours can be seen in Figure 6.

In order to understand the behaviour depending on the curing conditions untested samples have been analysed with the ESEM. Wood fibres have the characteristic to undergo volume changes in the presence or absence of water. The same phenomenon is observed in the fibres embedded in cement matrices: fibres swell in the fresh paste stage and while later they shrink during the hydration of cement. However, the phenomenon is not uniform in all the fibres since the volumetric changes in the fibres will vary according to their cross section shape, their chemical composition, the water/cementitious materials ratio, the pH of the cementitious composite and the temperature and relative humidity during curing. As can be seen in Figure 7 the swelling is not uniform in every direction of the cross section of this fibre embedded in air cured M6 material. In the central segment the fibre that once was attached to the cement matrix has shrank and there is a gap between the cement matrix and the fibre. On the right side of the image the fibre-matrix interface remains sound. This behaviour will explain the tendency of the tested fibres to undergo a slip softening behaviour. During the pullout test, once the chemical bond in the sound segments is broken, the fibre is loose inside the cement matrix and it is easy to pull out, therefore the pullout load decreases rapidly. On the other hand, if the fibre shrinkage is uniform along the fibre contour so that the fibre matrix chemical bond is lost but the fibre remains in contact with the cement matrix due to its irregular shape, rough surface or curvature along its length, then the fibre will show a behaviour governed by friction during the pullout test, as seen for instance with fibres embedded in M4 (Fig. 4).

The internal cracking in the fibre shown in Figure 7 was due to the vacuum condition inside the ESEM. The cracks developed during the observations with this equipment.

3.3 *Fibre treatment*

In order to reduce the volumetric changes of the spruce fibres in presence of water the fibres have been treated. The selected treatment reduces the hygroscopicity of the fibres. The effect of this treatment in the fibre-matrix interface has been studied with the aid of the ESEM (Fig. 8) and through pullout tests.

As can be seen in Figure 8 the contact between the fibre and matrix remains sound after 73 days cured in laboratory conditions (30 -50 RH). The cross section of the fibre has not changed and there is no gap between the fibre contour and the matrix.

From pullout tests with treated and untreated fibres embedded in matrix M7 the maximum pullout stress for treated fibres and untreated fibres is similar (0.55 and 0.59 MPa). Additionally, the pullout behaviour of the treated fibres is mainly slip hardening. This is an interesting improvement compared to the slip softening and constant friction behaviour of the untreated fibres. The pullout curves plotted in Figure 9 illustrate this difference. A higher amount of energy is released when pulling out the treated fibres.

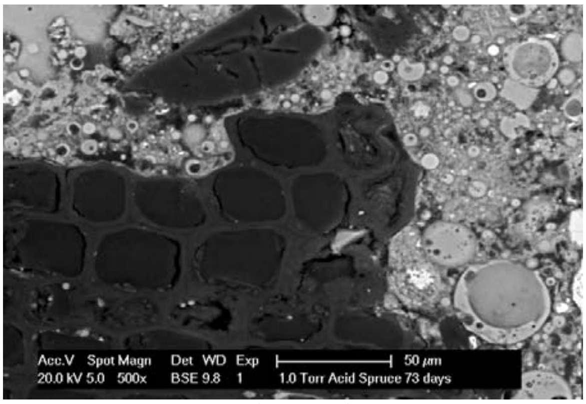

Figure 8. ESEM image of treated fibre embedded in M7

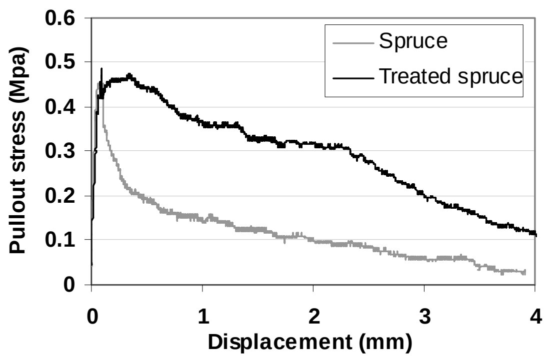

Figure 9. Pullout curves of treated and untreated fibre in M6

3.4 *Interface geometry*

As explained in previous sections, a rough surface of the bundle and irregular shapes in the cross section help to increase a mechanical bond with the cement matrix (Fig. 7 and 8). Along the length of the fibre the mechanical bond may also be increased due to the tendency of the fibres to curve. The degree of curvature also changes due to the presence or absence of water. If the fibre is curved inside the cement matrix there is an additional bending moment needed to be able to pullout the fibre. Therefore the curving of the fibre will increase the mechanical bond up to a degree where the fibre strength is exceeded by the bending moment, which will lead to the fibre to rupture.

From CT-scans of samples with different pullout behaviour it is clear that the fibres that rupture have a sharper curvature inside the cement matrix. For example, the CT-scan images in Figures 9 and 10 show the difference in degree of curvature for fibres embedded in M5. In Figure 10 the trace of the fibre that was pulled out with constant friction behaviour had an angle of about 12°. The fibre in Figure 11 broke during the test and had a curvature angle of about 30°.

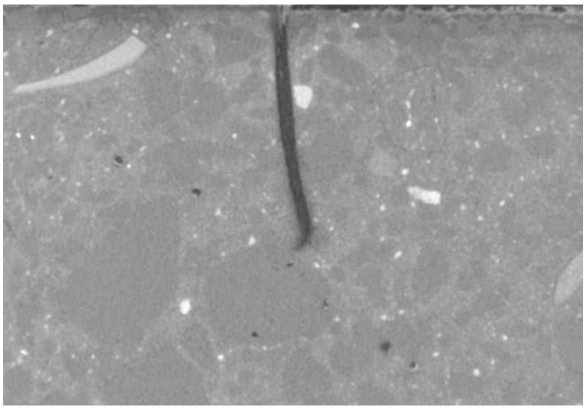

Figure 10. CT-Scan of fibre trace in M5, constant friction

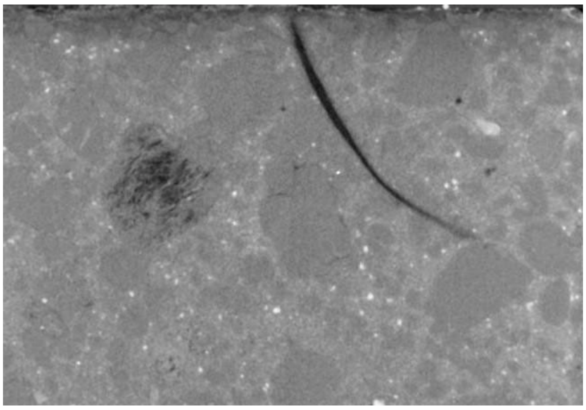

Figure 11. CT-Scan of fibre trace in M5, fibre rupture

4 CONCLUSIONS

This study explores the effect of the cement matrix composition, the curing conditions and fibre geometry on the interface behaviour between softwood fibres and cement matrices. Among these factors it is necessary to highlight the loss of interface bond due to the volume changes of the fibres in the presence of water. To reverse this effect the spruce fibres have been treated successfully to reduce the shrinkage and swelling after which a constant and sound interface bond was observed between the fibre and the cementitious matrix. Additionally, the treated fibres show improved pullout behaviour. Furthermore, the natural curving from the fibres increases the mechanical bond. However, it is necessary to take into account that excessive curving will lead to fibre rupture when the fibre is being pulled out.

Spruce fibres tend to show slip softening behaviour when pulled out. The pullout stress achieved by the fibres embedded in a cement matrix with slag replacement is twice the value of the pullout stress from fibres embedded in a cement matrix with fly ash. But since the stress is not the only criteria, the best pullout behaviour is observed in samples made with M1 and M4. M1 is a cement matrix with fly ash and sand while M4 includes slag and limestone as filler.

Future work will include further study of the interface with the use of IR-spectroscopy and nano identation to investigate a possible organic bond between the wood fibres and the cement matrix as well as the strength of the bond.

5 ACKNOWLEDGEMENTS

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