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### THE EFFECT OF HYDRAULIC CEMENTS ON THE FLEXURAL BEHAVIOR OF WOOL REINFORCED MORTARS

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#### Abstract

It is known that natural wool is a good thermal insulating material, but recent results suggest another application: the use of wool as a fiber-reinforcement in mortars and concretes. Indeed, the mechanical properties of wool filaments are comparable to those of some synthetic polymeric fibers (e.g., made with polypropylene). However, wool can dissolve in alkaline environments and, therefore, the performances of reinforced cement-based matrixes cannot be guaranteed for a long time. Accordingly, three series of reinforced mortar beams have been made with low alkali, high alkali, and sulfoaluminate cements. To investigate the chemical compatibility, and the subsequent effects on the mechanical performances, the beams have been tested in three point bending. As a result, the lower the alkalinity of the cement paste, the better the post-cracking capability of wool fibers to arrest the growth of cracks.

#### Keywords:

Wool reinforcement, low alkali cement, high alkali cement, sulfoaluminate cement.

#### **1 INTRODUCTION**

The use of natural fibers as a reinforcement of cementbased composites can increase the toughness of concretes and mortars, and represents a sustainable option to the traditional industrial fibers as well. Indeed, such fibres can bridge the surfaces of the cracks in the post-cracking stages and reduce the environmental impact of the construction industry [Fantilli 2017a].

Nevertheless, some limitations in the use of natural fiber have recently arisen, mainly due to durability reasons. Because of the alkalinity of the cementitious matrixes, both vegetal and animal fibers can be completely destroyed with the time [Tolêdo Filho 2000, Fantilli 2017b].

In the case of vegetal fibers, the reduced durability is associated to a reduced pullout strength, not only because the alkaline corrosion reduces the tensile of the fibers. In fact, the change of volume produced by water absorption and the migration of hydration products, especially calcium hydroxide, to the fiber lumen, walls and voids, drastically reduce the bond between fibers and matrixes [Tolêdo Filho 2000].

However, the principal mechanisms that produce the degradation of vegetal fibers are summarized in the following points:

• Peeling off phenomenon produced by alkaline water contained in the micro-pores of the cementitious matrix. This water can dissolve the lignin and hemicellulose existing in the middle lamellae of the

fibers, and makes the link between the cellules of the fibers weaker [Tolêdo Filho 2000].

 Alkaline hydrolysis of the cellulose molecules, which produces a degradation of the molecular chains and a reduction of both the degree of polymerization and tensile strength [Wei 2014].

To mitigate the degradation of the vegetal fibers used to reinforce cement-based mortars and concretes, either a special treatment of the fibers or the modification of the cementitious matrixes can be adopted. Chemical or physical methods are frequently used to improve the performance of the fibers. Wei and Meyer [Wei 2014] showed that both the acrylic emulsion and alkylalkoxysilane surface treatments of natural fiber provided a better durability properties and higher bending strengths of the fiber–reinforced composites.

On the other hands, the effectiveness of the vegetal fiber-reinforcement in the cementitious matrixes can be improved by adding supplementary cementitious materials to the common concrete and mortar components. In some cases, the partial substitution of the Portland cement with fly ash or silica fume can reduce the alkalinity of the cementitious matrix and, consequently, the degradation of the vegetal fibers. Positive results can be obtained by mixing sisal or coconut fibers with silica fume before adding them to the cementitious matrix [Tolêdo Filho 2000].

Although a wide research activity is devoted to the use of vegetal fibers as a reinforcement of cement-based composites, studies on the compatibility between animal fibers and cementitious matrixes are very scares in the literature. As wool fibers pre-treated with atmospheric plasma do not remarkably improve the performance of the mortars [Fantilli 2017a], new tests were performed with the aim of modifying the cementbased matrix. Specifically, the flexural behaviour of wool reinforced mortars made with different Portland cements was investigated and described in the following sections.

#### 2 DESCRIPTION OF THE TESTS

The experimental research, regarding some cementitious mortars reinforced with wool fibers, has been carried out in accordance with the rules reported by EN 196-1 [EN 196-1 2005].

#### 2.1 Materials

The main components of the mortars herein investigated are:

- Tap water
- CEN Standard sand, consisting of siliceous rounded particles, whose size distribution lies within the limits given by EN rules.
- Wool fibers with a density of 1.0 g/cm<sup>3</sup> (Fig.1). The mechanical and geometrical properties of the wool reinforcement have been already described in previous papers [Fantilli 2017a, Jóźwiak-Niedźwiedzka 2017].



Fig. 1: Sheep wool used to reinforce mortars.

 Hydraulic cements, which are classified on base of strength, type and the alkali content. In particular, the alkalinity was measured through the equivalent percentage of sodium oxide (Na<sub>2</sub>O)<sub>eq</sub>. Three different types of cement were used herein:

H – high alkali cement, which is a CEM I 42.5R having  $(Na_2O)_{eq} = 1,1\%$ ;

L – Low alkali cement, which is a CEM I 42.5R having  $(Na_2O)_{eq} = 0.4\%$ ;

S – 42.5 R sulfoaluminate cement with  $(Na_2O)_{eq} = 0,65\%$ . Previous studies [Li 2018] demonstrated that the bond strength between polymeric fibers and sulfoaluminate cement matrix are relatively better than that between the counterparts made with Portland cements. As wool is a polymeric fiber, it was expected that the use of such a type of cement could improve the performance of wool reinforced mortars.

#### 2.2 Test setup

By using the materials previously described, three series of mortars have been tailored. They are classified in Tab.1 on the base of the type of cement. Each series includes four different batches, depending on the curing conditions. As with a single batch three prismatic specimens 40x40x160mm have been moulded, a total of 36 specimens have been prepared for bending test. They remained in the moulds for 1 day in normal conditions (20°C, RH = 50%). After demoulding, and before the tests, the specimens of the series H1, L1, S1, H2, L2 and S2 were cured in the same normal conditions for 27 days. On the contrary, those of the series H3, L3 e S3 were left in water for 27 days in water, and those of the series H4, L4 and S4 remained in water only 3 days. The temperature of water was constant, 20°C.

All the specimens were tested in three point bending (see Fig.2, [EN 196-1 2005]) 28 days after casting, with the exception of specimens H4, L4 and S4, tested 4 days after casting. The external load P was applied through a MTS loading machine, having a load capacity of 100 kN. Tests were performed by driving the displacement of the loading cell, whose stroke moved at a velocity of 0.05 mm per minute.

Tab.	1:	The	composition	and and	curing	condition	s of	the
			analys	ed m	ortars.			

Series	Type of	Wool content	Curing				
	cement	(g)					
H_1	Н	-	27 days in normal conditions				
			(20°C, RH = 50%)				
H_2	н	10	27 days in normal conditions				
			(20°C, RH = 50%)				
H_3	Н	10	27 days in water				
			(20°C)				
H_4	Н	10	3 days in water (20°C)				
L_1	L	-	27 days in normal conditions				
			(20°C, RH = 50%)				
L_2	L	10	27 days in normal conditions				
			(20°C, RH = 50%)				
L_3	L	10	27 days in water (20°C)				
L_4	L	10	3 days in water (20°C)				
S_1	S	-	27 days in normal conditions				
			(20°C, RH = 50%)				
S_2	S	10	27 days in normal conditions				
			(20°C, RH = 50%)				
S 3	S	10	27 days in water				
	-		(20°C)				
S_4	S	10	3 days in water (20°C)				



Fig. 2: Three point bending tests for cementitious mortars [EN 196-1 2005].

#### **3 TEST RESULTS**

The three point bending tests provide the load-midspan deflection diagram shown in Fig. 3a. From the *P*- $\delta$  curves important information can be obtained. In particular, as illustrated in Fig.3a, both the flexural strength,  $P_{\text{max}}$ , and the corresponding displacement,  $\delta_{\text{p}}$ , are measured for the 36 tests, and their values are reported in Tab.2.

As the fiber-reinforcement works in the post-cracking stage, a new approach was proposed by Fantilli et al. to quantify the effect of the fiber-reinforcement [Fantilli 2017a]. Starting from the *P*- $\delta$  curves depicted in Fig.3a, the new post-peak diagrams reported in Fig.3b can be introduced. The values of the normalised load ( $y = P / P_{max}$ ) are reported on the ordinate. Conversely, the difference x between the post-peak deflection and  $\delta_p$  is on the abscissa of such diagrams. All the post-peak diagrams are limited to the value x = 0.2 mm. In correspondence of this deflection, the residual load detected in plain mortars is nearly zero. The ductility of the mortars herein investigated can also be quantified by calculating the area  $A_F$  delimited by the post-peak curves (see Fig.3b).

To facilitate the analysis of the results, a further simplification is herein introduced. In general, the postpeak curve depicted in Fig. 3b can be, approximated by a bilinear relationship (see Fig.3c). Thus, the most relevant data from pre-peak stage is  $P_{max}$  (i.e., the strength of the mortar) and the corresponding deflection  $\delta_p$ , whereas the residual stress of the post-cracking stage can be evidenced by A<sub>F</sub>, and the coordinates [x<sub>1</sub>, y<sub>1</sub>] and [0.2 mm, y<sub>2</sub>]. Hence, the values of  $A_{\rm F}$ , which represents the overall capacity of the fiber to bridge the crack surfaces, can be computed through the following formula:

$$A_F = \frac{1+y_1}{2} \cdot x_1 + \frac{y_1+y_2}{2} \cdot (0.2 - x_1) \tag{1}$$

where,  $y_1$  = percentage of the residual load just after the cracking;  $y_2$  = percentage of residual load in presence of large cracks; and  $x_1$  = abscissa (in mm) in correspondence of  $y_1$ .

Obviously, the larger the benefit of the fiber the larger  $y_1$ . Whereas, the capacity of maintaining the residual stress increases with  $A_F$  (and  $y_2$ ). The values of these parameters, as measured in the tests, are reported in Tab.2.

#### **4 ANALYSIS OF RESULTS**

The average values of  $P_{max}$ ,  $y_1$  and  $A_F$  are reported in Figs.5-7, respectively. In all the diagrams, the values measured in unreinforced series are herein assumed to be the reference. It is possible to observe that in normal curing conditions (27 days at 20°C and RH =50%), the lowest flexural strength is measured in presence of L cements, with and without the fiber-reinforcement (see Fig.5a). The highest flexural strength has been measured in the case of S cement, especially in presence of wool fibers. In accordance with the tests performed on industrial polymeric fibers [Li 2018], also in the case of wool fibers, the flexural strength increases when a sulfoaluminate cement matrix is used.

As shown in Fig.5b, in the fiber-reinforced specimens stored 27 days in water at 20°C, the higher the alkalinity the higher the flexural strength. Nevertheless, as the (Na<sub>2</sub>O)<sub>eq</sub> increases, the strength reduces with respect to plain and fiber-reinforced mortars cured for 27 days at 20°C and RH =50%. This reduction of strength can be ascribed to the larger degradation of the wool fibers, which seems to affect also the cementitious matrix. Moreover, in presence of water, the beneficial effect produced by sulfoaluminate cements vanishes.

The same trend can also be observed in Fig.5c, where the data related to the wool reinforced specimens left only 3 days in water (at  $20^{\circ}$ C) are reported. Obviously, the flexural strength of mortars containing fibers is lower than that of plain mortar cured for 28 days at  $20^{\circ}$ C and RH =50%.



Fig. 3: The results of the three point bending tests: (a) the whole  $P-\delta$  diagram; (b) the post-peak behaviour; (c) the bilinear relationship used to quantify the effect of the fibers.

		L_1			L_2			L_3			L_4	
	а	b	с	а	b	с	а	b	с	а	b	С
P <sub>max</sub> (kN)	2.59	2.31	2.58	1.90	1.76	1.69	2.35	2.19	2.35	1.44	1.60	1.43
δ <sub>p</sub> (mm)	0.56	0.43	0.48	0.52	0.78	0.65	0.51	0.39	0.74	0.35	0.41	0.38
<b>x₁ (mm)</b>	0.02	0.02	0.02	0.03	0.02	0.09	0.02	0.02	0.03	0.02	0.02	0.02
<b>y</b> 1	0.02	0.05	0.04	0.28	0.30	0.42	0.05	0.05	0.05	0.12	0.15	0.17
<b>y</b> 2	0.01	0.01	0.01	0.12	0.12	0.20	0.02	0.01	0.02	0.07	0.11	0.10
A <sub>F</sub> (mm)	0.01	0.02	0.01	0.05	0.05	0.10	0.02	0.02	0.02	0.03	0.03	0.03
		H_1			H_2			H_3			H_4	
	а	b	с	а	b	с	а	b	с	а	b	с
P <sub>max</sub> (kN)	3.34	3.38	3.58	3.25	3.42	3.33	2.83	2.80	2.96	2.20	2.46	2.53
δ <sub>p</sub> (mm)	0.56	0.33	0.45	0.41	0.47	0.52	1.11	0.50	0.56	0.45	0.32	0.44
<b>x₁ (mm)</b>	0.01	0.02	0.03	0.03	0.03	0.03	0.02	0.03	0.03	0.03	0.02	0.02
<b>y</b> 1	0.01	0.01	0.01	0.03	0.05	0.04	0.03	0.03	0.02	0.03	0.06	0.06
<b>y</b> 2	0.00	0.00	0.00	0.02	0.03	0.02	0.00	0.01	0.01	0.01	0.02	0.02
A⊧ (mm)	0.01	0.01	0.01	0.02	0.02	0.02	0.01	0.02	0.02	0.02	0.02	0.02
		S_1			S_2			S_3			S_4	
	а	b	С	а	b	С	а	b	С	а	b	С
P <sub>max</sub> (kN)	3.36	4.08	3.53	4.38	4.42	4.17	2.82	2.43	2.69	2.16	2.23	2.03
δ <sub>p</sub> (mm)	0.42	0.60	0.37	0.43	0.35	0.47	0.36	0.30	0.77	1.21	0.81	0.78
<b>x₁ (mm)</b>	0.03	0.03	0.03	0.03	0.03	0.03	0.01	0.02	0.03	0.02	0.02	0.02
<b>y</b> 1	0.01	0.01	0.01	0.01	0.02	0.01	0.04	0.05	0.04	0.10	0.08	0.10
<b>y</b> 2	0.01	0.00	0.00	0.00	0.02	0.01	0.01	0.02	0.01	0.05	0.03	0.04
A⊧ (mm)	0.01	0.01	0.02	0.02	0.02	0.02	0.01	0.02	0.02	0.02	0.02	0.02

Tab. 2: The main results of the three point bending tests.

In normal curing condition (27 days at 20°C and RH =50%), the residual strength after cracking is nearly zero without any fiber-reinforcement. As shown in Fig.6a, for all the types of cement, the reduction of strength in plain mortar is larger than 95%. Whereas, in presence of fiber, a remarkable residual strength is clearly evident. Nevertheless,  $y_1$  decreases as the alkalinity of cement increases. Specifically, in low alkali cement the contribution of wool fibers cannot be neglected, as the relative residual stress  $y_1$ = 0.3. If the alkalinity increases, the wool is progressively less effective, and  $y_1$  reduces and vanishes in the case of S cement (and it is less than 0.05 for H cement). This is due to fact that wool is corroded by the environmental alkalinity of cement.

For specimens stored 27 days in water (at 20°C), the values of residual strength in mortars containing fibers is more or less the same for all the types of cements (Fig.6b). These values are close to those obtained in normal curing conditions (27 days at 20°C and RH =50%).

The same trend (i.e., the higher the alkalinity the lower  $y_1$ ) can also be observed in Fig.6c, where the values of  $y_1$  measured in the fiber-reinforced specimens left only 3 days in water at 20°C are reported. In all the cases, the values of  $y_1$  are higher than those depicted in Fig.6b for the same type of cement. In other words, the larger the pH and time of curing in water, the higher the degree of wool fibers degradation.

In the SEM micrographs, the effect of curing in water is visible. Mortar specimens made with wool fibres and cement H, which were stored for one day in water, are characterized by a partial degradation of the fibres. Whereas, in those stored for 27 days in water, empty gaps after dissolved fibres are present (see Fig.4).

Finally, all the observations previously made for the relative residual strength  $y_1$  can be extended to the area  $A_F$  (see Fig. 7).



Fig. 4: SEM microphotograph taken on the specimen H3, arrows indicate similar sizes empty spaces after wool fibers, scale bar =20  $\mu$ m.





Fig. 5: The flexural strength P<sub>max</sub> measured in the three point bending tests: (a) specimens cured for 27 days in normal conditions (20°C, RH = 50%); (b) specimens cured for 27 days in water (20°C); (c) specimens cured for 3 days in water (20°C).

Fig. 6: The residual stress y₁ measured in the three point bending tests: (a) specimens cured for 27 days in normal conditions (20°C, RH = 50%); (b) specimens cured for 27 days in water (20°C); (c) specimens cured for 3 days in water (20°C).



Fig. 7: The area A<sub>F</sub> measured in the three point bending tests: (a) specimens cured for 27 days in normal conditions (20°C, RH = 50%); (b) specimens cured for 27 days in water (20°C); (c) specimens cured for 3 days in water (20°C).

#### **5 SUMMARY**

From the results of the experimental analyses performed on mortars made with different types of cements and reinforced with wool fibers, the following conclusions can be drawn:

- The flexural strength increases with the equivalent percentage of sodium oxide (Na<sub>2</sub>O)<sub>eq</sub>. The highest strength is detected in the sulfoaluminate cement matrixes reinforced with wool and cured in normal conditions (20°C, RH = 50%).
- On the contrary, the capability of the fibers to bridge the crack surfaces, and guarantee the presence of a residual tensile strength in the post-cracking stage, reduces in high alkali cements and in presence of water. This is particularly highlighted by the values of the residual strength y<sub>1</sub> and the area A<sub>F</sub> of the post-peak diagrams.

Future researches will be devoted to compare the residual strength measured in low alkali cements mortars reinforced with wool with those containing polypropylene fibers.

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