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# The effect of fibre chemical treatment on the steel fibre/cementitious matrix interface

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## HIGHLIGHTS

- ▶ Enhanced bonding was achieved via treatment of steel fibres with ZnPh conversion.
- ▶ ZnPh crystals precipitation resulted into a rough surface of the steel fibres.
- ▶ ZnPh treatment led to significant increase in pullout energy.
- ▶ ZnPh treatment has a prominent effect, when mechanical interlocking is negligible.
- ▶ Optimum fibre configuration can be chosen based on the fibre surface contact area.

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## ABSTRACT

This paper aims to study the interfacial bonding between steel fibres and cement-based matrix. The fibres were treated chemically using a zinc phosphate conversion to achieve enhanced bonding with the mortar matrix. In order to gain a better perspective on the effect of chemical treatment different fibre parameters were considered in this research, such as the fibres shape, length and diameter. In this respect, single-sided fibre pullout tests were conducted on treated as well as on as received (untreated) fibres of hooked-end, straight and undulated shape. The analysis of the experimental results revealed that the fibre shape is of great importance since it contributes to mechanical interlocking that prevail during the pullout of the fibres. Chemical treatment was also shown to play an important role on the fibre–matrix interface especially when mechanical interlocking is absent. Treated fibres exhibited a modified surface with a rough topology caused by precipitation of ZnPh crystals on the fibre. Optimum fibre configuration for maximum pullout performance can be chosen based on the fibre surface contact area, since the pullout load and pullout energy is directly related to this property.



## 1. Introduction

Fibres are being introduced in a wide range of engineering materials in order to reinforce the matrix and enhance material's performance. As it is known, concrete without reinforcement has low tensile strength and low strain capacity at fracture. Therefore, fibres are added to concrete in order to enhance its mechanical properties. Considerable research work worldwide is being conducted on fibre reinforced concrete [1] to determine the influence of the fibre inclusion on the compressive, flexural, fatigue properties, etc. [2–8].

The macroscopic performance of a composite system is dependent on the fibre and matrix properties, fibre volume fraction, fibre orientation, etc. [9]. Much research work is being very recently published on the effect of wavy or undulated microstructures on the overall behaviour of composites [10]. However, not only the aforementioned parameters, but also the composite's interfacial behaviour is of key importance. Therefore, the determination of the fibre–matrix interfacial properties and the understanding of its failure mechanisms are considered essential [9]. The two main ways to improve adhesion in cement based composites are through altering the mechanical bonding or the chemical bonding between the matrix and the fibre. Most research work done in the area takes into account the use of fibres with different geometries which results in different mechanical bonding, while the chemical bonding has been achieved via matrix modification or fibre treatment [11–15]. The determination of the fibre/matrix adhesion is commonly identified using the single fibre pullout test [9] alone or in concurrence with non-destructive methods, such as acoustic emission [16].

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The effect of various fibre shapes, diameters, embedment lengths on the pullout behaviour of steel fibre reinforced concrete has been of special interest in the past decades [12–14,17,18]. Regarding the fibre shape, it has been suggested that deformed and hooked fibres present higher resistance to pullout than smooth ones due to the mechanical contribution [14]. Next to that bonding enhancement has been documented with an increase in the embedment length, but this influence was more evident on smooth fibres [13]. Some researchers have studied the effect of fibre inclination angle on the pullout behaviour of steel fibres [12,17,18]. It has been observed that the peak load increased up to an angle of 30° and decreased for an inclination of 60° [12]. The research interest on the pullout behaviour is not limited in steel fibres since other types of fibres or bars were under investigation in the past [19–24].

It has been well documented that a way to enhance the adhesion between fibre and matrix is by using high strength matrices and/or modifying the matrix [11,14,17,25–28]. In general, bonding between fibre and matrix raises when the matrix strength is increased. A comprehensive study on the effect of embedment length, fibre orientation and w/c ratio on the pullout behaviour concluded that the w/c ratio, i.e. matrix strength plays a significant role on the maximum value reached at the peak load, while it has a secondary role during the slipping process [17]. According to the authors, debonding followed by slipping is the most frequent mode of failure. During the slipping process, the maximum load is being reached after the fibre starts to slip and not necessarily at the time when it is totally debonded [17].

The addition of silica fume [11,26,29] and high reactivity metakaolin (HRM) [11,29] has been discussed in respect to the near surface characteristics of concrete as well as the bond-slip behaviour. It has been suggested that HRM and silica fume were both very effective in improving the bond-slip behaviour. The combination of HRM and silica fume however, led to excessive bond improvement, which in turn resulted in undesirable fibre fractures [11].

An efficient alternative to modify the fibre–matrix bonding in cementitious composites is by treating the fibres. Sugama et al. [15] and Sun et al. [30] studied the interface between zinc phosphate (ZnPh)-deposited steel fibres and cementitious matrix. They reported that the bond strength of treated fibres improved by 40–50% over that of the untreated ones. Moreover, the ZnPh conversion provided protection on the steel fibre from corrosion. Sugama et al. [15] claimed that ZnPh not only reduced the corrosion rate, but also resulted in an open surface topography that contributed to the strong mechanical interlocking bonds formed with the adhesive material. The influence of fibre chemical treatment on the macroscopical fracture behaviour of the final composite (steel fibre reinforced concrete) has been evaluated previously [31] by means of acoustic emission.

As aforementioned, few studies are available on the effect of fibre treatment on the fibre–matrix bonding. Next to that the interrelated effect of fibre treatment with that of fibre geometry has not been thoroughly investigated. The scope of this paper is, to further investigate this interface by modifying the fibre while the composition of the mortar matrix was kept constant. A further aim is to study the influence of the fibre shape and geometric characteristics on the fibre–matrix interface. The purpose is therefore to address how chemical bonding through ZhPh treatment affects the overall pullout behaviour compared to geometrical parameters that influence mechanical interlocking. In order to isolate the effect of fibre treatment from that of coarse aggregates all experiments have been conducted on mortar matrix. The steel fibres were treated with a ZnPh conversion. The effect of this treatment in relation to fibre diameter, fibre length (i.e. embedment length) and fibre shape was investigated.

## 2. Experimental study

### *2.1. Materials and specimens preparation*

The single-sided fibre pullout tests were conducted on six treated and six as received different types of fibres, including hooked-end, straight and undulated ones (Fig. 1). The geometric properties of fibres can be seen in Table 1, based on the data provided by the supplier. The tensile strength of the fibres was 1100 MPa.

The as received fibres were treated with a ZnPh conversion with formulation of 0.46 wt% zinc orthophosphate dihydrate, 0.91 wt% 85% $H_3PO_4$ and 98.63 wt% water, and prepared as proposed by Sugama et al. [15]. Particularly, the fibres were immersed for 5 min in the ZnPh conversion at 90 °C and then were rinsed with water and dried for 10 min in an oven at 150 °C. Based on the experimental procedure the ZnPh conversion treatment does not require any special equipment while ZnPh conversion is easily applicable. This results in an overall straightforward and cost effective procedure. The chemical treatment alters the surface of the fibres. A valid concern relates to the effect of such treatment on the overall mechanical performance of the steel fibres. Since the bulk of the fibre is decisive for its tensile properties, the alteration of the surface is not expected to have a significant impact on the fibre's tensile strength.

The specimens consisted of a cement-based mortar matrix with w/c ratio of 0.5. The specific gravity and the water absorption of the sand were 2.50 kg/dm$^3$ and 2.44% respectively. The proportions of the mixture and its air content according to ASTM C231-03 [32] can be found in Table 2. The specimens were cured in water saturated with calcium hydroxide at 23 ± 2 °C.

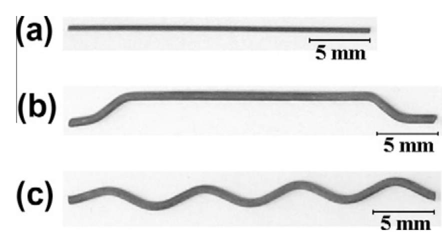


**Fig. 1.** (a) 25 mm long straight fibre. (b) 30 mm long hooked-end fibre. (c) 30 mm long undulated fibre.

**Table 1**
Sample's designation and fibre properties.

| Mixture code | Geometry | Condition | $l^a$ (mm) | $d^a$ (mm) | $l/d^a$ ratio |
|---|---|---|---|---|---|
| S04.25 | Straight | As received | 25 | 0.4 | 62.5 |
| cS04.25 | Straight | Treated | 25 | 0.4 | 62.5 |
| H07.30 | Hooked ends | As received | 30 | 0.7 | 42.9 |
| cH07.30 | Hooked ends | Treated | 30 | 0.7 | 42.9 |
| H06.30 | Hooked ends | As received | 30 | 0.6 | 50.0 |
| cH06.30 | Hooked ends | Treated | 30 | 0.6 | 50.0 |
| H06.25 | Hooked ends | As received | 25 | 0.6 | 41.7 |
| cH06.25 | Hooked ends | Treated | 25 | 0.6 | 41.7 |
| U07.30 | Undulated | As received | 30 | 0.7 | 42.9 |
| cU07.30 | Undulated | Treated | 30 | 0.7 | 42.9 |
| U05.30 | Undulated | As received | 30 | 0.5 | 60.0 |
| cU05.30 | Undulated | Treated | 30 | 0.5 | 60.0 |

[a] *l*: Fibre length. *d*: Diameter. *l*/*d*: Fibre aspect ratio.

**Table 2**
Mix proportions and properties of mortar mixture.

| Material | Quantities |
|---|---|
| Cement II 42.5 N (kg/m$^3$) | 440 |
| Water (kg/m$^3$) | 220 |
| Sand (kg/m$^3$) | 1591 |
| Superplasticizer (kg/m$^3$) | 3.6 |
| Compressive strength (MPa) | 42.9 |
| STDEV[a](MPa) | 3.87 |
| Air content (%) | 0.45–0.6 |

[a] STDEV corresponds to standard deviation.

*2.2. Compressive and pullout testing*

The compressive tests were conducted on 150 × 150 × 150 mm specimens at a rate of 0.5 MPa/s according to BS EN 12390-3:2002 [33] (see Table 2).

Cubic specimens were used for the pullout testing with dimensions of 100 × 100 × 100 mm. Every specimen included two fibres (see Fig. 2). For the whole range of samples described in Table 1, eight to sixteen pullout tests were performed depending on the way failure occurred. In the case of fibre failure in the gripping area the test was disregarded and repeated. A frame was designed and used to appropriately place the fibre into the fresh mortar specimens. A predefined embedment length was selected and the fibres were immersed into the fresh mortar. The frame was in place during the first 24 h in order to avoid any fibre dislocation. The embedment length was half of the fibre's length.

The tests were conducted after 28 days of curing on a servo-hydraulic Instron 8801 machine. The experimental setup is presented in Fig. 3. As can be seen, the mortar specimens were placed on a proper frame that held the specimens stable during the test and was restrained on the bottom end of the testing machine. The fibres were gripped on the upper end of the machine at an average gripping length of 3–5 mm depending on the fibre's geometry.The displacement rate during the pullout test was 0.6 mm/min.

Images of the fibres before and after the pullout test were made using a high resolution digital camera. In order to verify the effect of chemical treatment on the surface of the fibres untested treated and as received fibres were examined using a Leica DM-4000M optical microscope at 200 magnification.

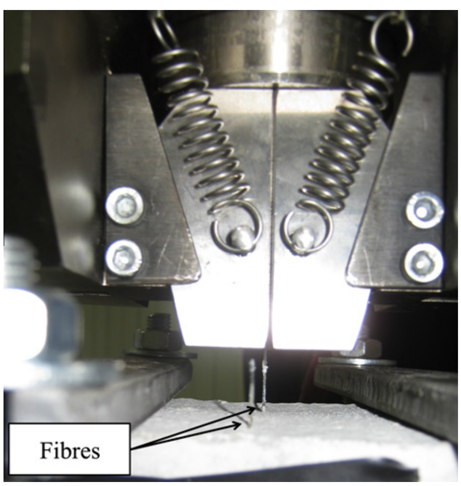


**Fig. 2.** Pullout test setup.

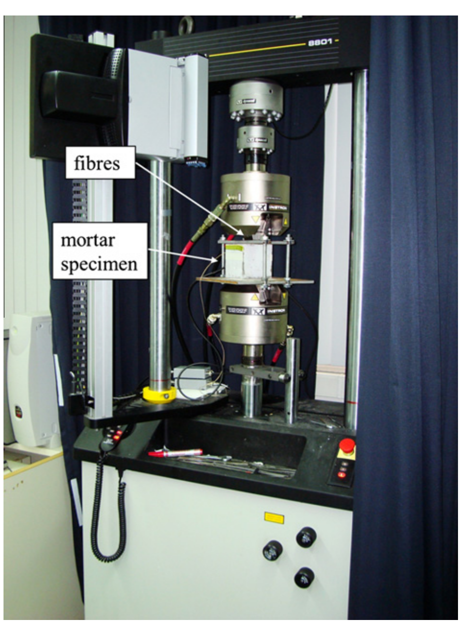


**Fig. 3.** Experimental setup.

## 3. Results and discussion

The results of the pullout tests for the specimens with as received and treated fibres are presented in Figs. 4–10.

The acceptable failure modes in the pullout experiment are fibre debonding, followed by fibre slippage and finally pullout from the mortar or fibre breakage within the matrix before pullout occurs. All specimens failed by fibre/matrix debonding.The peak pullout load ($P_{max}$), the interfacial shear strength ($\tau_{max}$) and the pullout energy ($G$) are presented. The pullout energy is calculated as the area under the load–displacement curve. The interfacial shear strength is given by the equation:

$$\tau_{max} = \frac{P_{max}}{l_e \pi d} \quad (1)$$

where $\tau_{max}$ is the interfacial shear strength (MPa), $P_{max}$ is the maximum load ($N$), $l_e$ is the embedment length (mm) and $d$ is the fibre diameter (mm).

In Fig. 4 the peak load of all specimens tested (with as received and treated fibres) is presented. The straight fibres (S04.25) present the minimum peak load value for both treated and untreated fibre conditions. This can be attributed to the fact that this fibre has the lowest diameter (0.4 mm) and short embedment length (12.5 mm). Next to that, its shape (straight fibre) does not promote any mechanical interlocking mechanisms with the cement matrix. On the contrary, the hooked-end and undulated shapes contribute to an improved anchorage with the matrix and result in higher peak load values.

This can be also seen in Fig. 5a–c, where the load–displacement curves of each shape of treated and as received fibres are illustrated. It can be noticed that the shape of the curves are dependent on the fibre shape. In the case of straight fibres, due to the lack of anchorage after the peak load a sudden drop is observed, while in the case of hooked and undulated fibres more than one peak is present in the graph. When comparing fibres with the same diameter and embedment length, such as the undulated fibres U07.30 and the hooked-end H07.30, it can be concluded that the undulated shape is better in terms of mechanical interlocking (Fig. 4). Regarding the effect of ZnPh conversion treatment on the fibres, it can be noticed that all treated fibres have increased peak load compared to as received ones. The increase of the peak load ranges between 8% and 390% for the treated fibres. The most significant improvement is found in the case of the straight fibres. This was expected, since in straight fibres the improvement of the chemical treatment is of key importance, due to the absence of any mechanical interlocking. On the other hand when mechanical interlocking

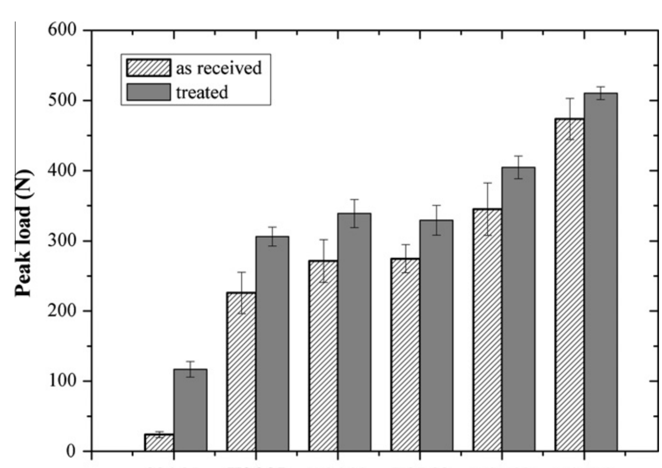


**Fig. 4.** Peak load during pullout tests of mortar with treated and as received fibres.

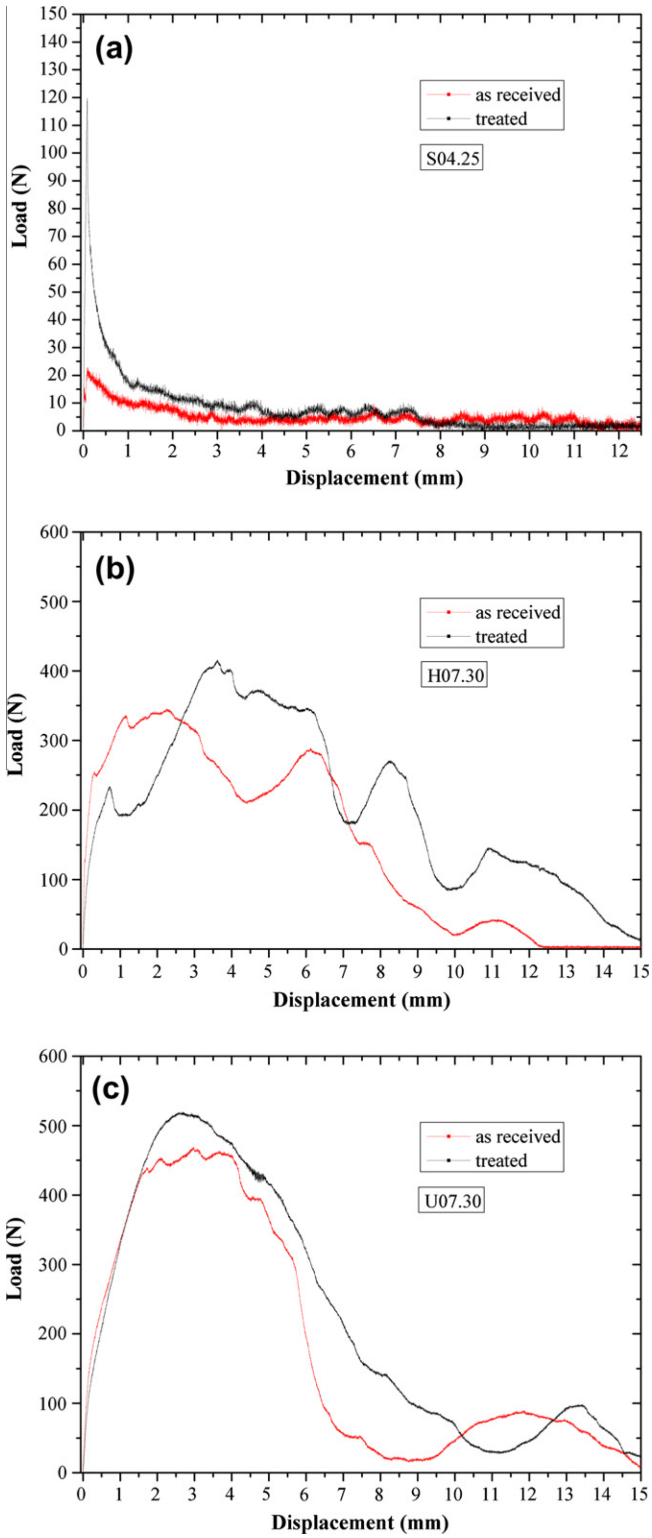


**Fig. 5.** Load–displacement curves of (a) straight fibres, (b) hooked-end fibres, (c) undulated fibres, for both fibre conditions.

is present (see undulated fibres) the contribution of the chemical treatment seems to be marginal. For better understanding, Fig. 6 presents the increase of the peak load of the treated fibres as a function of the peak load of the same fibre in the untreated (as received) state. It can be concluded that, the weaker the mechanical interlocking between the fibre and the matrix, the higher the significance of the chemical treatment.

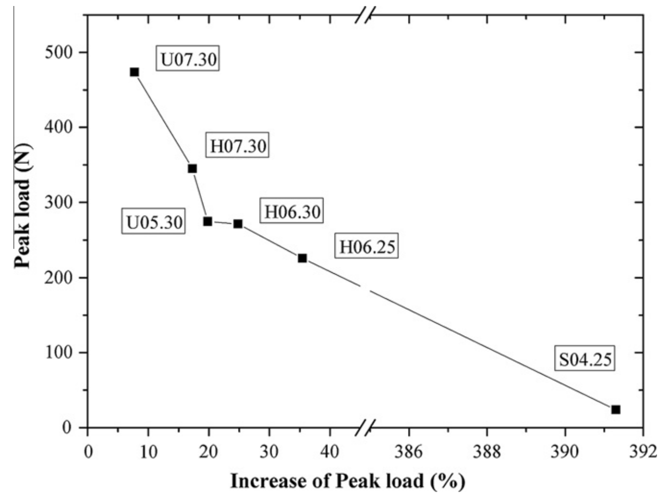


**Fig. 6.** Increase of peak load due to treatment in relation to peak load of as received fibres.

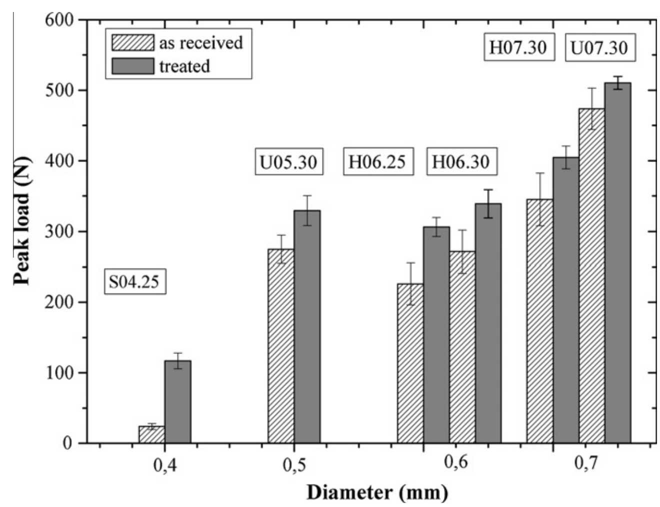


**Fig. 7.** Peak load in relation to diameter for as received and treated fibres.

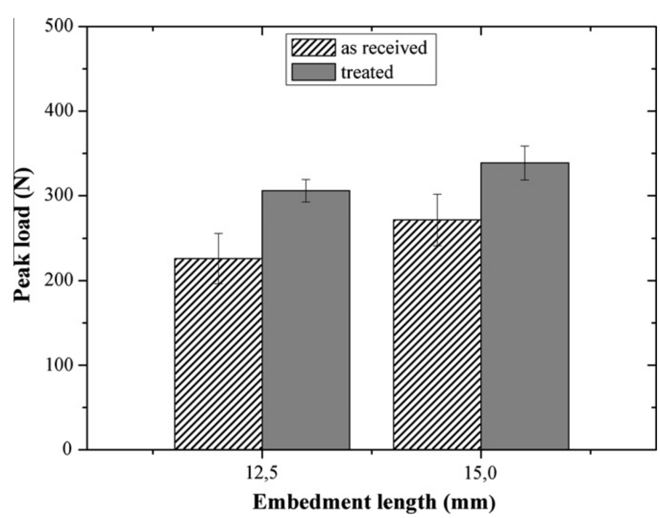


**Fig. 8.** Variation of peak load for the different embedment length of fibres with 0.6 mm diameter.

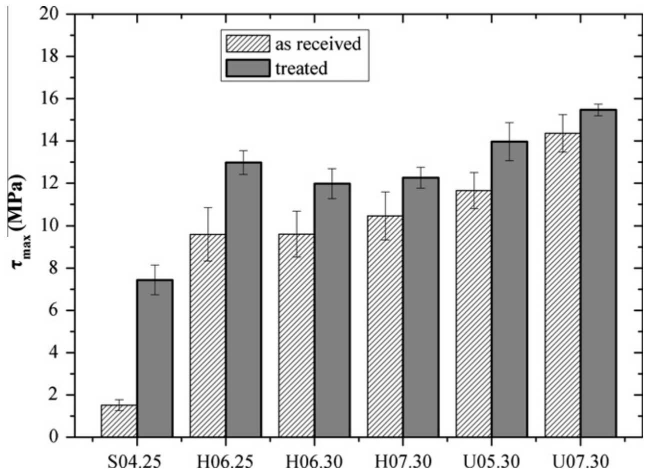


**Fig. 9.** Interfacial shear strength for both fibre conditions.

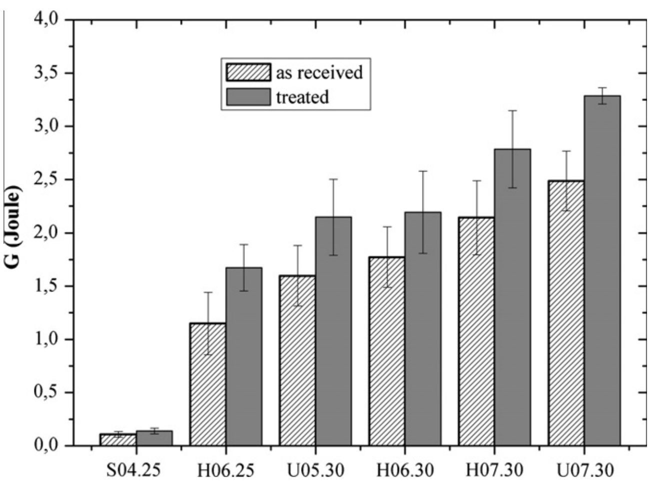


**Fig. 10.** Pullout energy for as received and treated fibres.

The effect of the fibre diameter on the peak load values can be found in Fig. 7. The first observation that can be made is that increasing the diameter of fibres with the same shape, an increase in the peak load is obtained. A 0.1 mm increase in hooked-end fibres results in 19–27% increase in the peak load (treated and untreated fibres, respectively). In undulated fibres an increase of 0.2 mm results in a peak load enhancement of 55–72%. For both fibre shapes, the diameter effect is more obvious in the case of the untreated fibres. This is due to the fact that, where chemical treatment is absent, there is a greater need for substantial embedded fibre surface. Undulated fibres offer greater enhancement, since their contact surface is greater than in the case of hooked-end fibres.

The effect of the embedment length on the peak load of fibres with 0.6 mm diameter is exhibited in Fig. 8. Higher embedment length leads to higher peak load. Particularly, an increase of 2.5 mm in the embedment length leads to an increment of 20% in the peak load to the as received and 11% to the treated fibres. Similarly to the aforementioned effect of the diameter, the effect of the embedment length is more obvious in the case of the as-received fibres.

Interfacial shear strength values of all fibre types can be found in Fig. 9. The undulated fibre shape (U05.30, U07.30) is the most effective in terms of adhesion compared to the hooked-end and straight fibres. The straight fibres present overall the lowest values, even though the chemical treatment increased their interfacial shear strength significantly. This graph supports all previous observations, i.e. that an increase in the diameter leads to an increase of the bond strength for both untreated and treated fibres, which is more obvious in the undulated fibres.

Fig. 10 illustrates the pullout energy values for all fibre types. The variation in the pullout energy values follows in most cases the variation in the peak load values. As aforementioned, undulated fibres are more effective than hooked-end ones (H07.30, U07.30), while an increase in the diameter and the embedment length increases the energy. The main conclusion that can be drawn from this graph is that chemical treatment leads to a pronounced increase in the energy required to debond the fibre from the cement matrix.

In order to elaborate the role of various parameters on the pullout behaviour of fibre/mortar the fibre contact surface area has been selected as a representative geometrical parameter. The fibre contact surface area integrates the embedded length, shape, diameter and aspect ratio ($l/d$) into one parameter and represents the area of the fibre that is embedded in the mortar matrix. Fig. 11a and b illustrate the pullout load and pullout energy versus the contact surface area of the fibres, respectively. As can be observed in Fig. 11a and b, an increase in the contact surface area results in a respective increase of the pullout load as well as the pullout energy. This confirms that, independently to the fibre shape, higher contact surface area requires higher load/energy to pullout the

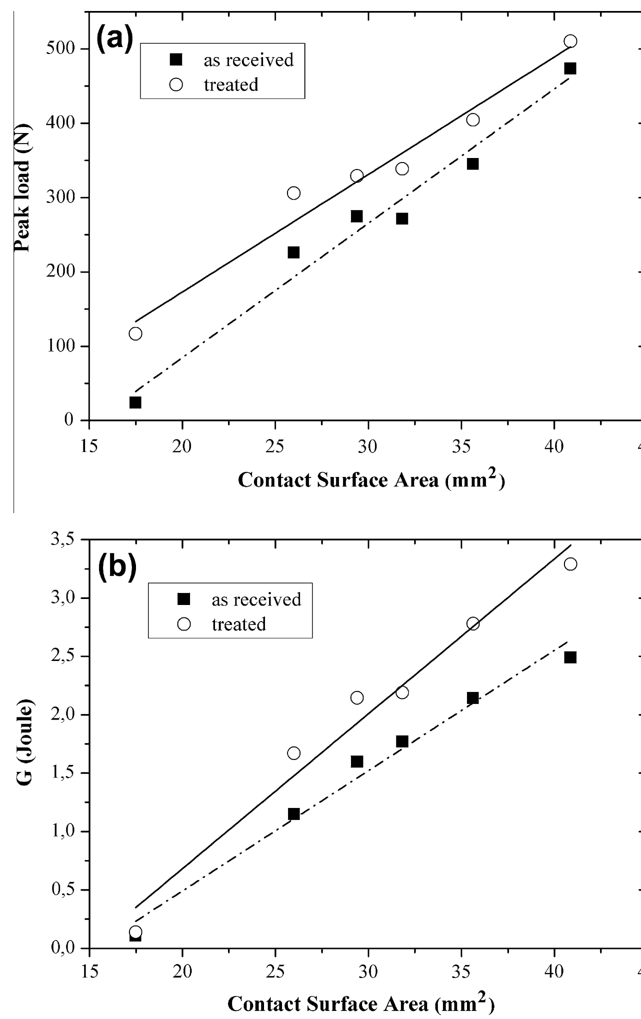


**Fig. 11.** (a) Pullout load vs. fibres contact surface area. (b) Pullout energy vs. fibres contact surface area.


**Fig. 12.** (a) straight fibres, (b) hooked-end fibres, (c) undulated fibres, after pullout test.

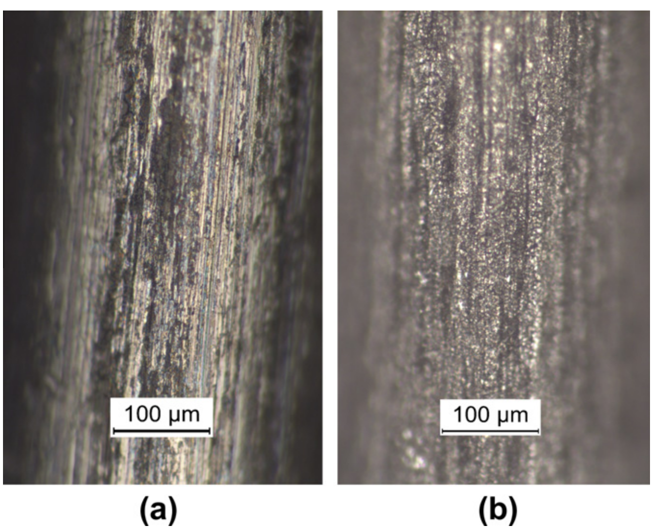


**Fig. 13.** (a) hooked-end fibre as received (×200), (b) hooked-end fibre treated (×200).

embedded fibre. These graphs suggest that the fibre should be selected on the basis of the contact area for optimizing the pullout performance.

Fig. 12a–c show fibres of different shape after they have been pulled out of the cementitious matrix. As can be observed, the deformed fibres have lost their initial shapes and have been more straightened. The extent of damage imposed by the different fibre shapes is related to the geometrical characteristics of the fibres and mainly to the diameter and the waviness of the fibres. The higher the damage area, the more energy would be absorbed during the fracture process. This is supported by the results presented in Fig. 10, where the total pullout energy for the U07.30 fibres (highest waviness and diameter) is the highest for both untreated and treated conditions.

Images from the untested treated and as received fibres from the optical microscope can be seen in Fig. 13a and b. As it can be clearly seen in these figures, the as received fibres present grooves originating from the fibre manufacturing process and smoother surfaces than the treated fibres. On the contrary, the presence of a crystalline structure on the surface of treated fibres becomes evident, while the grooves disappear. This is due to the presence of a zinc phosphate conversion coating which is an inorganic crystalline coating. Apparently, the original smooth surface of the steel fibres was modified into a rough topographical feature by precipitating ZnPh crystals on the fibre. This contributed to improved adhesion properties between the carbon steel substrates and the cementitious matrix, leading to the formation of interfacial chemical bonding between treated fibres and matrix. These observations are in agreement with previous observations where the surface of the treated fibres were examined in details using SEM [15].

## 4. Conclusions

Pullout tests have been carried out in this study in order to investigate the steel fibre/mortar interface mechanical property. For this purpose, fibres with different geometries, both treated with a ZnPh conversion and as received, have been tested.

It has been shown that the peak load and pullout energy for both fibre conditions, i.e. treated and untreated, increases with an increase in the fibre diameter and embedment length. However, in terms of interfacial shear strength, only a relationship with the fibre diameter could be confirmed for both untreated and treated fibres. In addition, the undulated fibre shape proved to be the most efficient compared to the hooked-end and straight fibres.

ZnPh treatment was proven to be effective in modifying the fibre/matrix interface, especially when mechanical interlocking was absent (straight fibres). Precipitation of ZnPh crystals resulted in the formation of rough topographical morphology of the fibre surface. This was confirmed by optical microscopy. This treatment led to a significant increase in the peak pullout load, the interfacial shear strength, as well as the energy required for fibre debonding.

Optimum fibre configuration for maximum pullout performance can be chosen based on the fibre surface contact area, since the pullout load and pullout energy is directly related to this property.

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