Investigation of the interfacial bonding between flax/wool twine and various cementitious matrices in mortar composites

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HIGHLIGHTS

• Improvement of interfacial bonding between natural fibres and cement paste.
• Crack arrest of resin coated NFR cementitious composites.
• Enhanced mechanical performance of resin coated NFR cementitious composites.
• GGBS and FA addition improved the impermeability of NFR cementitious composites.

ABSTRACT

This study investigates the interfacial bonding of natural fibre reinforced (NFR) cementitious composites by exploring the incorporation of 20–30 mm strands of uncoated and resin coated flax/wool twine into various cementitious matrices. Cementitious matrices consisting of pulverised fly-ash (FA), ground granulated blast furnace slag (GGBS) and ordinary Portland cement (OPC) in various ratios were tested with the addition of flax/wool twine (1% volume ratio). The mechanical properties of these samples were assessed at 7 and 28 days. The results showed a reduction in the flexural and compressive strength of uncoated NFR samples compared to unreinforced (UNR) counterparts due to weak interfacial bonding between the uncoated fibres and the cementitious paste, therefore, formation of voids. Epoxy (EP) and polyurethane (PU) resin were then used to coat the flax/wool twine prior to their inclusion in various cementitious pastes. The results revealed improvements in both flexural and compressive strengths exhibited at 7 and 28 days compared to UNR and uncoated NFR samples. The greatest improvement of flexural strength, 61% compared to UNR, was achieved by the mix consisting of 50% OPC and 50% FA matrix with EP resin coated flax/wool twine at 28 days. While PU coated samples exhibited an increase of 31% in flexural strength at 28 days for the same cementitious mix ratio. The morphology of the resin coated NFR samples showed an intimate interfacial bond to surrounding cementitious paste, which explains the increased mechanical performance.

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1. Introduction

Affordable housing and energy conservation are among the current issues in the construction industry. The utilisation of indigenous renewable resources in construction materials development are vital for the construction industry which worldwide is responsible for more than 40% of global energy consumption and as much as 33% of carbon dioxide equivalent emissions (CO2e) [1]. Efforts to work with sustainable and renewable construction materials has gained a momentum in recent years [2–4]. A potential saving can come from substituting synthetic fibres by natural fibres having advantages, such as: (1) abundance, (2) renewability, (3) minimal health hazards, (4) low density, (5) desirable fibre aspect ratio, and (6) relatively high tensile and flexural modulus [5]. Studies have shown that the incorporation of plant-based natural fibres for making construction materials and products reduces material handling costs and heat transfer in buildings [6]. Aggarwal [7] showed that the mechanical and durability performance of cementitious composites reinforced with bagasse met the requirements of ISO: 8335-1987 and BS: 5669: Part 4: 1989 standards. Hence, they could be used as internal and external building components.
Khedari et al. [8] presented in their work that coir fibre reinforced soil cement blocks were lighter in weight and had reduced thermal conductivity compared to unreinforced samples. New applications such as thin cement bonded natural fibre reinforced (NFR) composites is explored in sound and thermal insulation materials in buildings. Increased energy efficiency of buildings will reduce energy demands, heating costs and the environmental impacts linked with energy production [6]. Moreover, NFR cementitious composites, are used in thin sheet components that must stand locally high loads or deformation and for components in which fibres are added to primarily control cracking such as slabs and pavements.

In many developing countries, where natural fibres of various origins are in abundance, adaptation of innovative approaches could significantly influence the utilisation of natural fibres in a cost-effective approach to produce good quality fibre reinforced cementitious composites for low-rise buildings. Natural fibres are generally categorised into four groups according to their origin: stem/bast, leaf, fruit/surface and wood/cellulose.

When it comes to the workability of natural fibre reinforced cementitious composites the key factors that contribute to loss of workability are fibre aspect ratio and volume fraction in mixtures. It was shown that the incorporation of flax fibres in concrete significantly reduced its fresh state workability as the result of high-water absorbability of flax fibre with high specific surface area [9]. Mansur and Aziz [10] observed, as the length and content of jute fibre increased the workability of mortar decreased.

Currently, the concrete industry faces challenges to meet the growing demand of Portland cement due to limited reserves of limestone, slow manufacturing growth and increasing carbon taxes. The aim is to reduce the environmental impact of construction and use greater proportion of waste pozzolan, whilst improving concrete performance. High alumina cement, gypsum, and a variety of special low carbon and low energy supplementary cementitious materials have been used to produce natural fibre reinforced cementitious composites, which may improve the durability of the composites, and/or reduce chemical interactions between fibres and cementitious matrix [11–13]. Commonly, the addition of synthetic fibres to cement composites enhances toughness, ductility and impact resistance properties [14,15]. Similar results have been reported for some cement composites containing plant-based natural fibres. Most importantly, the durability of cementitious composites is a very important consideration; studies have shown that plant-based natural fibre reinforced cementitious composites are vulnerable to deterioration in cement matrices due to absorbed water and high alkaline pore solution weakening of natural fibres. The issues are associated with weakening of the fibres by alkali attack, fibre mineralisation due to migration of hydration products to lumens and volume variation in natural fibres due to their high water absorption [11,16,17]. By reducing the soluble alkali content of cement and the reduction of portlandite (Ca(OH)₂) through pozzolanic reaction, partial substitution of cement with options such as FA, silica fume and GGBS reduces the alkalinity of cementitious mixtures. Study by Tolédo Filho et al. [17] has shown that early carbonation and reduced alkalinity using partial replacement of cement by un-densified silica fume was effective in preventing the deterioration of natural fibres in cementitious composites. For interfacial bonding improvements, Lecompte et al. [18] found out that higher consolidation pressure leads to better interfacial bond strength between the cementitious paste and natural fibres in an extrusion-based manufacturing. However, in this study, the main objective is to develop composites with well-balanced properties that achieve a homogeneous dispersion of the fibres in the matrix with strong interfacial bonding between the cement matrix and the fibres. Different pozzolanic matrices are investigated (i.e. GGBS and FA) with varying mix ratios by weight. Moreover, the epoxy and polyurethane resin coated flax/wool twine is used to assess the mechanical and morphological properties of mortars with respect to unreinforced (UNR) and uncoated samples.

2. Experimental methodology

2.1. Raw materials

Fine, sharp river sand with 2 mm nominal maximum grain size and CEM I Portland cement conforming to EN 197-1 (from CEMEX –UK) was used in this study. Pulverised fly ash (FA) was obtained from HCCP Hargreaves Coal Combustion Products Limited (UK), which is compliant with BS 3892-1:1982 for use as type II additive in the production of mortar. Ground granulated blast furnace slag (GGBS) was obtained from Hanson Heidelberg Cement Group (UK) which complies with EN 15167-1 (see Fig. 1 for microstructures). The natural fibre selected as reinforcement in this study is flax/wool twine (see Fig. 2), which was cut to the length of approximately 25–30 mm. The diameter of the twine was 5 mm, which results in the aspect ratio of 5–6. The density of the flax/wool twine was 1.5 g/cm³. Flax, Linum usitatissimum, belongs to the bast fibres where it is most often used in the higher value-added textile markets. Wool, on the other hand, is a protein fibre containing disulphide groups in molecular architecture which gives this fibre great elasticity.

![Fig. 1. Microstructure of (a) ordinary Portland cement (b) pulverised fly ash and (c) ground granulated blast furnace slag.](image1)

![Fig. 2. (a) Flax/wool twine and (b) sample size.](image2)
The twine was “wrap-spun” and the wool was used for the wrapping fibre. Axson FastCast PU resin F190 and West System epoxy resin were obtained from East Coast Fibreglass supplies for coating the twines prior to mixing in cementitious matrices.

2.2. Mix design and formulations

The NFR samples had a 1% by volume ratio of flax/wool twine, chosen based on previous works done by our group [11] on jute fibre reinforced cementitious composites. The mix proportion ratio used for NFR mortar was 1:1.5 for cement to sand ratio with water to cement ratio of 0.53, while for the UNR samples, the water to cement ratio was reduced to 0.38 based on several optimisation cycles of mix workability. The matrices used for making mortars consisted of OPC, FA and GGBS which were mixed at 70%:30% and 50%:50% based by weight. Moreover 100% samples of OPC cementitious matrix was produced for comparison and benchmarking (Fig. 3).

2.3. Sample preparation and testing

The initial experimentation of uncoated flax/wool twine samples was subdivided into natural fibre reinforced (NFR) and unreinforced (UNR) groups (Fig. 3). Three samples were made for each of the tests and coefficient of variance was calculated to ensure the reliability of data. Flexural strength testing at 7 and 28 days (BS EN 196-1:2005) was conducted, followed by compressive strength and water absorption testing (BS EN 772-21:2011). The specimens were weighed at the interval of 5 min, 10 min, 15 min, 30 min, 1 h, 4 h, 6 h, 24 h and 48 h. The flow table test (BS EN 1015-3:1999) was used to differentiate the consistency/workability of different mix designs and investigate the influence of flax/wool twine on the rheology (see Fig. 4). The tensile strength of flax/wool twine used in this study was also investigated to determine one of the most influential mechanical properties of reinforcement fibres in mortars. Twenty tensile tests were carried out on flax/wool twine specimens using a displacement control Instron testing machine. From the previous literature it was gathered that the tensile strength can reach more than 1000 MPa in case of flax fibre and vary from about 100 MPa to 300 MPa in case of wool fibre [19,20]. In this study, the flax/wool twine’s tensile strength for 20 samples was 580 MPa with a coefficient of variance of 9%.

Once the uncoated flax/wool twine reinforced samples had been tested, two cementitious matrices were selected for resin coated addition of flax/wool twine: (1) 50%OPC–50%FA (OF) and (2) 50%OPC–50%GGBS (OG). The resin coated flax/wool twines were allowed 24 h to cure prior to mixing with the aforementioned cementitious matrices (see Fig. 5).

Samples of the failure region for uncoated and resin coated NFR specimens were taken for microstructure analysis. Field emission gun-scanning electron microscopy, Zeiss Supra 35 VP FEG-SEM was used to examine the mortar samples. It is important to emphasise that for the interfacial bonding properties for every mortar, 10 different samples were selected and tested, therefore considerable amount of SEM images were analysed. This analytical technique is subjective and is important to have extensive analysis prior to making conclusive statements.

3. Results and discussion

3.1. Flexural strength

The comparison for flexural strength was done on two fronts: (1) the influence of different cementitious matrices and (2) the effect of flax/wool twine as resin coated and uncoated reinforcing element of cementitious composite.

As highlighted in Table 1, the three strongest cementitious matrices in terms of flexural strength achieved at both 7 and 28 days are OG (50%OPC–50%GGBS), O1 (100% OPC) and G2 (70% GGBS–30%OPC), whilst the two weakest cementitious matrices are F2 (70%FA–30%OPC) and OF (50%OPC–50%FA). This is in line with previous observation where the inclusion of FA has shown weak mechanical properties in cementitious composites [11]. Although, some work for production of structural concrete incorporating large quantities of fly ash has been done [21–23].

It is worth pointing out the reduction in flexural strength was observed in all the samples upon the inclusion of flax/wool twines. The highest reduction percentage in flexural strength was observed for G2 (70% GGBS–30%OPC) samples for both testing ages of 7 and 28 days with 44% and 52% reduction respectively (see Table 1). On the other hand, the smallest reduction between the flexural strength of UNR and NFR was observed in O2 (70% OPC–30%FA) samples at both 7 and 28 days with 10% and 22%, respectively.

![Fig. 3. Testing programme chart.](image-url)

![Fig. 4. Flow table test results for reinforced and unreinforced fresh mortar samples.](image-url)
The NFR samples did not shatter in pieces after failure as opposed to the change in flexural extension behaviour cannot be ignored. The flexural strength of cementitious mortar composites when compared to unreinforced (UNR) samples at 7 and 28 days. The highest compressive strengths were observed for OG (50% OPC–50%GGBS) and O1 (100%OPC) cementitious matrices at 58 MPa and 55 MPa, respectively at 28 days for unpaired samples. The flax/wool twines have negative influence on compressive strength of cementitious composites where the reduction in strength is as high as 71% (a drop from 58 to 34 MPa) for the case of OG cementitious matrix at 28 days. This is due to voids developed because of inhomogeneous matrix and fibre interaction.

An interesting observation in Fig. 8 is the higher compressive strength of OG cementitious matrix at 28 days for both UNR and uncoated NFR samples compared to O1 cementitious matrix. This is not the same for 7 days compressive strength, where O1 samples are stronger than OG. This can be related to the delay in hydration process as a result of addition of GGBS. The effect of flax/wool twine as resin coated and uncoated reinforcing elements of cementitious composite were also studied in the compressive strength property of mortar composites. The result in Fig. 9, illustrate that both of the resins used for coating, i.e. NFR (PU) and NFR (EP), have increased the compressive strength when compared to uncoated NFR samples. This trend is observed for both of the cementitious matrices, i.e. OF and OG at 7 and 28 days. Compared to uncoated NFR samples, the epoxy coating of flax/wool twine, NFR (EP), has resulted in 80% and 107% improvement at 7 and 28 days, respectively, for the OF cementitious matrix. The same improvement for the OG cementitious matrix is 93% and 47% for 7 and 28 days respectively. On the other hand, the PU resin coated NFR samples, showed a similar improvement. In the case of 7 days in the OF cementitious matrix the EP and PU resin resulted in the same compressive strength, although, the biggest difference was observed for the same matrix but at 28 days, where NFR (PU) had a compressive strength of 30 MPa compared to 35 MPa for NFR (EP). It can therefore be concluded, that the addition of epoxy coated flax/wool twine can improve the compressive strength of mortar composites.

Interestingly, as observed in Fig. 9, both of the resin coated NFRs showed compressive strength improvements compared to their UNR counterparts, except for OG cementitious matrix at 28 days, which had a compressive strength of 58 MPa compared to 47 MPa and 50 MPa for NFR (PU) and NFR (EP) samples, respectively. The largest improvement of compressive strength between the coated NFR and UNR samples was observed at 7 days in the OF matrix with 27%, whilst at 28 days, 18% improvement was observed. The small compressive strength difference between NFR (EP) and NFR (PU) might be due to stronger interfacial bond between the EP resin and flax/wool twine and therefore, stronger reinforcing effect in the cementitious composite.

Table 1
Flexural strength of unreinforced (UNR) and uncoated reinforced (NFR) samples with different cementitious matrices.

| Sample ID | Flexural strength (MPa) | 7 Days | 28 Days |
|-----------|-------------------------|--------|---------|
|           | UNR NFR Reduction %     | UNR NFR Reduction % |
| O1        | 6.5 (5%) 5.4 (9%) 20%   | 7.8 (8%) 6.1 (4%) 29% |
| O2        | 5.7 (8%) 5.2 (6%) 10%   | 7.0 (7%) 5.8 (9%) 22% |
| F2        | 1.4 (9%) 1.2 (7%) 15%   | 3.4 (5%) 2.6 (7%) 31% |
| G2        | 6.6 (5%) 4.7 (6%) 44%   | 9.1 (3%) 6.0 (8%) 52% |
| OF        | 3.4 (9%) 3.2 (7%) 4%    | 6.0 (5%) 5.1 (4%) 18% |
| OG        | 7.1 (4%) 5.9 (8%) 20%   | 9.4 (9%) 6.4 (8%) 47% |

*The values in () are the coefficient of variance percentages.
3.3. Water absorption properties

In order to evaluate the performance of different cementitious mixtures when exposed to wet conditions, all samples were tested for water absorption at 7 and 28 days for a duration of 48 h. As illustrated in Fig. 10 and following the same trend of mechanical properties results, F2 and OF mixtures absorbed the highest amount of water for both uncoated NFR and UNR samples when tested after 7 days. However, an evident enhancement in the performance of both mixtures was observed at 28 days. The higher water absorption rate at 7 days, when compared with other mixtures, may refer to: (1) the presence of high amount of fly ash in both mixtures (70% and 50% for F2 and OF, respectively) works on delaying the hydration process of mortar at early ages. Additionally, based on the microstructural properties analysis in Fig. 1, the large surface area of fly ash will in general contribute in increasing the rate of water absorption [24,25]. (2) The presence of uncoated flax/wool twine in the NFR mix will work on absorbing

![Fig. 6. Flexural strength behaviour of (a) UNR and (b) NFR samples at 7 days and (c) UNR, (d) NFR at 28 days for six cementitous matrices.](image)

![Fig. 7. Flexural strength comparison of UNR, uncoated and coated NFR specimens with 50%OPC–50%FA (OF) and 50%OPC–50%GGBS (OG) cementitous matrices.](image)
high amounts of water, at early ages (before 7 days), due to their high absorption rate that reaches more than 25% [26]. (3) The higher porosity of both mixtures at 7 days [27,28]. (4) The high workability of the F2 and OF matrices (Fig. 4) that works on increasing air voids and micro-cracks in the internal structure. However, after 28 days the hydration process would reach its higher levels, which contributes to reducing the porosity of the cementitious composite and consequently reduces the intake of water through the pores. On the other hand, lower fly ash content, 30% in the O2 cementitious matrix, was observed to have lower water intake levels.

The presence of GGBS (OG and G2) either in the UNR or NFR cementitious matrices (Fig. 10) has been observed to enhance the performance of mixtures at 7 days (compared to other mixtures). However, water absorption was noticed to increase when testing the mixtures at 28 days. This can be attributed to the influence of the GGBS on the temperature rise of cementitious matrices at early ages. GGBS works on reducing temperature rise at early ages which reduces the development of thermal cracks in the pores and consequently decreases the water intake of cementitious matrices [29]. In contrast, the influence of GGBS at 28 days was noticed to be less effective in reducing water absorption of cementitious matrices, which might refer to their role in increasing the dry shrinkage [30].

More specifically, uncoated flax/wool twine showed a high capacity for absorbing water when compared to control samples. This comes from the porous morphology of the fibres and their distribution in the cementitious composites. The hydrophilic properties of the uncoated flax/wool twine will initiate higher rates of water absorption when integrated in cementitious matrices.

3.4. Interfacial bonding properties

The microstructural investigation of resin coated and uncoated NFR samples were carried out with the rationale to elucidate the micro interaction mechanism between the different cementitious pastes and flax/wool twine and how this could explain the performance of samples. As shown in Table 1, the flexural strength of uncoated NRF samples were reduced compared to UNR counterparts, which could be explained by investigating the microstructure of several, i.e. 10 samples for each mortar, failure zone of uncoated NRF cementitious composites (Fig. 11). The fact that during the mixing of mortar the twines have been untwined and made into separate wool and flax fibres can lead to inhomogeneous mix and development of voids into the cementitious matrix. A smoother surface would lead to less friction between the two bonding surfaces and therefore result in fibres requiring a lower load to be pulled out [31]. It is evident that there is not sufficient bonding between the fibres and cementitious matrix, which resulted in fibre pull out failures, e.g. no residues of cementitious paste on the surface of fibres was observed (see Fig. 11). Moreover, the increased water/cement ratio to make the mix workable could

![Fig. 8. Compressive strength of UNR and uncoated NFR at 7 and 28 days for six cementitious matrices.](image1)

![Fig. 9. Compressive strength comparison of UNR, uncoated and coated NFR specimens with 50%OPC–50%FA (OF) and 50%OPC–50%GGBS (OG) cementitious matrices.](image2)
negatively influence the mechanical strength of samples. All the aforementioned points negatively influence the properties of uncoated NFR samples compared to the UNR counterparts (see Table 1 and Fig. 8 for flexural and compressive properties, respectively). It is worth noting that despite NFR samples displaying a decreased peak flexural strength, they prolonged softening branch in comparison to unreinforced cementitious composites (see Fig. 6).

Fig. 10. Water absorption of (a) UNR and (b) NFR samples at 7 days and (c) UNR, (d) NFR at 28 days for six cementitious matrices.

Fig. 11. Interfacial bonding morphology between the uncoated flax/wool twine and 50%OPC–50%GGBS (OG) matrix.
When cementitious composites, with the pH of wet cement being around 13.1–13.5, are combined with natural fibre reinforcements, there is bound to be mass loss from the fibres caused by alkaline hydrolysis. There would be a possibility of shrinkage of the reinforcement away from the margins of the encapsulating cement paste. However, this phenomenon was not observed in the case for resin coated flax/wool twine (see Figs. 12 and 13).

The resin coating, i.e. EP and PU, of flax/wool twines and their influence on the microstructural properties of two cementitious matrices of OG (50%OPC–50%GGBS) and OF (50%OPC–50%FA) were also investigated. With reference to Figs. 12 and 13, it is immediately apparent that the microstructure of cementitious composites are more compact without any obvious voids and/or delamination at interfaces. The interfacial bonding is likely to get stronger for resin coated flax/wool twine, and consequently, improve the load transfer process at the interface. This was proven by the stronger mechanical performance, e.g. flexural and compressive strength in comparison to UNR and uncoated NFR samples (see Figs. 7 and 9). In the case of OG as cementitious matrix, the flexural strength of EP coated samples at 28 days increased by 127% compared to uncoated counterpart (see Figs. 11 and 12b). The same phenomenon was observed for PU coated samples, where an increase of 84% was observed in flexural strength at 28 days. The superior mechanical performance of coated flax/wool twine mortar samples mainly depends on the positive synergetic crack arresting and bridging effect delaying the occurrence of micro cracks and preventing it from developing into macro cracks. Crack deflection mechanism happens as a result of interface delamination and changes in the crack path. Such crack arresting and bridging mechanisms leads to a ductile cementitious composite as shown by the flexural responses (see Fig. 6).

By comparing the performance of the two resins, i.e. EP and PU, as the coating agent of flax/wool twine, it is not immediately evident which one has better interfacial bonding properties by study-

Fig. 12. Interfacial bonding morphology between the EP coated flax/wool twine and (a) 50%OPC–50%FA (OF); (b) 50%OPC–50%GGBS (OG) cementitious matrix.
ing the microstructures in Figs. 12 and 13. Although, from Figs. 7 and 9, it can be seen that EP has performed slightly better in terms of flexural and compressive strength at both testing ages and cementitious matrices, i.e. OG and OF.

4. Conclusions

In this paper, the mechanical, physical and interfacial bonding properties of various cementitious matrices (OPC, FA and GGBS) reinforced with uncoated and resin coated flax/wool twine were investigated. The influence of different parameters and the synergetic effect of resin coated reinforcement on mechanical properties of cementitious composites were discussed. The following conclusions can be drawn from the results presented in this study:

1. Uncoated flax/wool twine may act as flaws in the mortar and lead to the decreased compressive and flexural strength compared to unreinforced samples. This is probably owing to the poor dispersion of flax/wool twine which were untwined into individual fibres during mixing. The observed phenomenon cannot achieve the goal of reinforcement effect.
2. The resin coatings of flax/wool improved the mechanical performance of mortars samples compared to unreinforced and uncoated samples. The epoxy resin coating led to a better performance in terms of strength improvements compared to polyurethane resin.

3. Combination of OPC and GGBS by the weight ratio of 50:50 outperformed the OPC and FA cementitious matrix with the same proportions. Both of these matrices can be suitable for natural fibre reinforced cementitious composites.

4. The incorporation of GGBS within NFR cementitious composites enhanced their impermeability after 7 days of curing. Whereas, incorporating the mixtures with fly ash enhanced impermeability at 28 days.

5. The microstructural investigation was able to elucidate the reasons for mechanical improvements in the coated flax/wool twine mortar samples in comparison to the uncoated counterparts.

For future prospective of natural fibres in cementitious composites, detailed research studies on the effect of natural fibres on flexural performance cementitious composites via the internal curing technology should also be explored.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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