

Article

Investigation of Physical and Mechanical Properties of Cement Mortar Incorporating Waste Cotton Fibres

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Abstract: There is a lack of effective disposal methods for the increasing amount of textile waste that is being generated worldwide. This is creating environmental concerns and burdening waste management facilities. In this study, we propose that cotton fibres that have been recycled from textile waste could be used as fibre reinforcement in cement mortar. Seven mix designs were prepared, which were based on the quantity (0.4%, 0.8%, 1.6% and 2.0% by the weight of the cement) and length (20 mm, 30 mm and 40 mm) of the cotton fibres. The physical properties, including workability, compressive strength, flexural strength, density and water absorption, were investigated. The workability of the cement mortar was reduced with the addition of the cotton fibres. The flexural strength of the cement mortar with the added cotton fibres was improved by up to 9%, compared to the flexural strength of the control samples. The compressive strengths of the samples generally decreased with the increase in the fibre content and length. However, the C0.8 mix showed a comparable compressive strength to the control mix at all curing ages, with a slight decrease of 2.5% on day 56 of curing. The results were further clarified using SEM images. The improvement in the flexural properties showed that the cotton fibres could be implemented as fibre reinforcement in cementitious composites.

Keywords: cotton fibre; cement mortar; waste management; natural fibre; cement composites



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1. Introduction

Textile waste is an increasingly common issue for waste management facilities. There have been extensive investigations, globally, to find the most effective way to reduce, reuse and recycle textile waste materials; however, there is still an overwhelming amount of excess textile waste ending up in landfill. In Australia alone, on average, 27 kg of new clothing is acquired per person annually, of which 23 kg ends up in landfill [1,2]. In total, more than 800,000 tonnes of leather, rubber and textiles were discarded in 2018–2019, with a recycling rate of just 7% [3].

To reduce the environmental impact and relieve the pressure on waste management facilities [4], the cotton fibres extracted from textile waste could be recycled for use in construction applications. The current methods used for the fibre reinforcement of cementitious materials typically use synthetic fibres to improve their mechanical properties. However, the cost and environmental impacts associated with the manufacturing of synthetic fibres are unsustainable and comparatively worse than processing natural fibres such as cotton [5]. The use of waste/recycled material will lead to sustainable development in the construction industry [6,7]. Saleh et al. [8] used waste fibres to improve the properties of mortar and recommended the use of waste fibres for various construction applications.

Cement-based composites are widely utilised materials in civil engineering practices. Though, due to their brittle nature and poor resistance to crack formation, there have been significant studies exploring the potential use of natural fibres in cement composites to prevent these mechanical defects [9–12]. In addition, the fibre reinforcement could enhance the durability of cementitious composites. Jiang et al. [13] concluded that the inclusion of fibres improved the energy dissipation characteristics of cementitious composites. Due to

the relatively high tensile strength and complex orientation of natural fibres, they have the ability to reinforce brittle cementitious composites [14]. Some recent studies have indicated that the implementation of natural fibre reinforcement in cement composites can significantly improve their physical, mechanical and microstructural properties [15]. Furthermore, Eskander and Saleh [16] used natural fibres to reinforce cement mortar and concluded that the addition of fibres showed resistance against radioactive waste and freeze–thaw cycles. However, there has been inconclusive research regarding the influence of fibre quantity and fibre length on specific mechanical properties.

The mechanical properties of individual cotton fibres depend on a fibre's dimensions, density and orientation. In general, increasing a fibre's length tends to decrease its tensile strength. Studies have shown that increasing the lengths of fibres can lead to a higher probability of kink banding and fibre entanglement, compared to shorter fibre lengths. This leads to a reduced mechanical response from individual fibres [10]. Kink banding is another phenomenon that can occur. It involves the dislocation of fibres on the surface when under axial loads [17]. The fibre entanglement can cause multiple fibres to interlock, forming complex fibrous structures that can alter the bonding between the cement matrix and the fibres [18].

Previous studies have compared the use of synthetic fibres with natural fibre reinforcement and have found comparable improvements in the flexural properties of natural-fibre-reinforced cement composites [12,19]. Pichardo et al. [12] reported a 40% and 7% improvement in the compressive and flexural strengths, respectively, of concrete by incorporating cotton fibres. Some studies have demonstrated the feasibility of using recycled cotton fibres as a renewable alternative to inorganic fibre reinforcement. Alomayri et al. [20] reported improvements of up to 50% in the flexural strength of composites with the addition of 2.1% of cotton fibres. Several studies [21–24] have been conducted on the use of various types of natural fibres that can be used; however, very few studies have focused on the cotton fibres extracted from textile waste. Therefore, further investigations are required to determine the effect of different percentages of cotton fibres to obtain an optimum quantity for improving the properties of mortar. In addition, there is a limited amount of literature available on the effect of the length of cotton fibres on the properties of mortar. The use of cotton fibres, unlike synthetic fibres, will reduce the carbon footprint of the construction industry.

In this research, to enhance textile recycling and to promote sustainability in the construction industry, cotton-derived fibres were incorporated in cement-based mortar. This was carried out by shredding recycled cotton sheets down to a fibrous state for use in the reinforcement of cement composites, and to assess the fresh, hardened and microstructural properties. Primarily, four mixes were prepared based on the addition of cotton fibres by the weight of the cement, in quantities ranging from 0.4%, 0.8%, 1.6% and 2.0%. The influence of the fibre content on the mechanical properties were then investigated. Moreover, to assess the influence of fibre length on the properties of mortar, cotton fibres with lengths of 20 mm, 30 mm and 40 mm were studied.

To determine the effects of fibre length and fibre quantity on the cement composites in this study, the physical and mechanical properties were assessed through workability, density, absorption, compressive strength and flexural strength tests. The microstructural properties were analysed through scanning electron microscopy (SEM) imaging, to assess the morphology of each mixture and to identify the interfacial bonding between the cotton fibres and the cement matrix.

2. Materials and Methods

2.1. Materials

The materials used to prepare mortar samples included cement, fine aggregates, cotton fibres and superplasticizer. In this research, Boral general-purpose cement, complying with Australian Standard AS 3972-2010 [25], was used. The fine aggregates, with particle sizes ranging from 0.075 mm to 4.75 mm and a bulk density of 2800 kg/m³, were used.

The bed sheet and extracted cotton fibres are shown in Figure 1. To maintain a consistent water-to-cement ratio, MasterGlenium SKY 8100 [26] superplasticiser was added to all mixtures.



Figure 1. Raw materials: (a) bed sheet and (b) extracted cotton fibres.

The cotton fibres used in this research were extracted from recycled, white bed sheets, obtained from Lifeline Newcastle (Newcastle, Australia)—a textile recycling facility. The white sheet was utilized as it did not contain any dye material. The sheets were cleaned and then cut into strips. Each strip was then cut to the desired width and then further cut into squares, according to the required length. The individual fibres were collected from these square sheets to obtain the required amount of cotton fibres. The process for obtaining the required fibres is summarised in Figure 2.

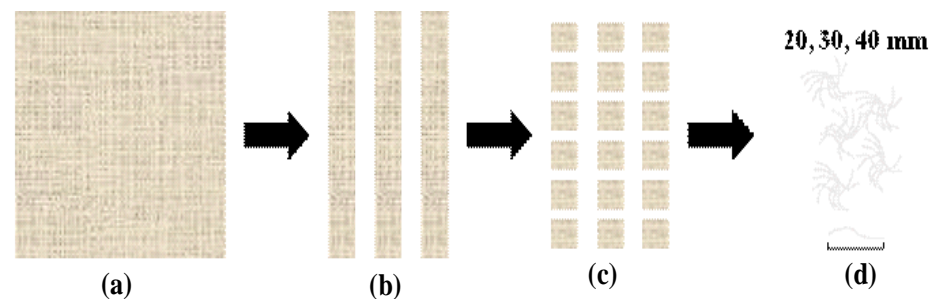


Figure 2. Recycled cotton fibre extraction process: (a) recycled bed sheet, (b) cut into strips, (c) cut into square of required lengths and (d) cotton fibres.

The SEM image of cotton fibres is shown in Figure 3, below. The diameter of cotton fibres ranged from 5 μm to 20 μm .

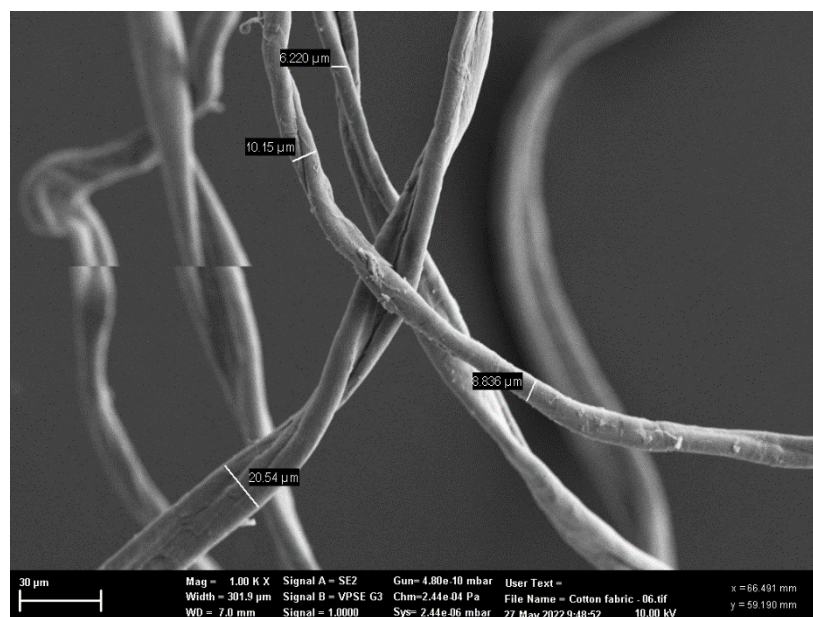


Figure 3. SEM image of cotton fibres.

2.2. Mix Design

The mix design for the preparation of mortar samples is presented in Table 1. The mix design was based on the trial mixes used to obtain high-strength mortar samples. The amounts of cement and fine aggregates were kept constant in all mixes. The quantity of water used in each mix was maintained to account for the absorption of water by cotton fibres and fine aggregates. Seven mixes were prepared, and the mixes were based on the quantity and length of the cotton fibres. The four mortar mixes were prepared with the addition of 0.4%, 0.8%, 1.6% and 2.0% of cotton fibres, by weight of cement in each mix. Cotton fibres of 30 mm in length were used for these four mixes. Two mixes were prepared with cotton fibres of 20 mm and 40 mm in length and the percentage of cotton fibres added to these two mixes were kept 0.8% of the weight of the cement. One control mix was prepared without the addition of cotton fibres to compare the effect of different amounts and lengths of cotton fibres on the properties of mortar. The percentage of cotton fibres added was based on previous studies [14,27].

Table 1. Designed mix proportions.

Mix Label	Fine Aggregates (kg)	Cement (kg)	Water (kg)	Cotton Fibre (wt%)	Cotton Fibre (kg)	Superplasticiser (L)
C0.0	1750	525	233	0.0	0.0	3.75
C0.4	1750	525	233	0.4	2.1	3.75
C0.8	1750	525	233	0.8	4.2	6.25
C1.6	1750	525	233	1.6	8.4	11.25
C2.0	1750	525	233	2.0	10.5	15.00
C0.8-S	1750	525	233	0.8	4.2	6.25
C0.8-L	1750	525	233	0.8	4.2	6.25

To identify each mix, a symbolic naming convention was adopted. For example, the letter 'C' at the start of each mixture indicates the presence of cotton fibres in each mix, and the amount of cotton fibres in that mix is then indicated by the decimal number following the letter 'C'. Where symbols 'S' and 'L' are present, this indicates that the smaller (20 mm) and larger (40 mm) cotton fibres were used. For example, C0.8-S indicates the mix contains

0.8% cotton fibre, relative to the cement weight, and that the smaller (20 mm) fibres were used. A water-to-cement ratio of 0.5 was maintained for all mixes, and superplasticiser was added to maintain comparable workability.

2.3. Preparation of Samples

All mixes were prepared in the engineering department's laboratory at the University of Newcastle and followed AS 2350.12-2006 [28] for the preparation of standard mortar and the moulding of samples for testing.

After all ingredients had been weighed and prepared, half of the fine aggregate was poured into the mortar mixer, followed by the entire cement bucket. The remaining fine aggregate was then added and the machine was turned on and mixed for 2 min. After 2 min, the dry materials were inspected to ensure adequate mixing. After inspection, the mortar mixer was then turned back on, and cotton fibres were added incrementally to reduce clumping phenomena between fibres. Fibres were mixed for 2 min, then the mixing machine was stopped for an additional inspection to observe the dispersion of cotton fibres, as shown in Figure 4.



Figure 4. Dry mixture with cotton fibres.

If fibres were well-dispersed in dry materials, the mortar mixer was then turned on and the water (with superplasticiser) was added slowly. The wet mixture was blended for an additional 2 min before stopping to check the homogeneity of the mix and dispersion of cotton fibres. The workability of the mix was determined using a flow table test, in accordance with ASTM C1437 [21]. After mixing, the fresh mortar was placed in different moulds for mechanical tests. The samples were removed from the moulds after 24 h and cured in the climate-controlled fog room.

2.4. Physical and Mechanical Testing

2.4.1. Water Absorption of Cotton Fibres

To estimate the 24 h water absorption of the cotton fibres, a test was conducted before the mortar mixing. To do this, 1 gram of cotton fibres was weighed in a precision, vacuum-chambered scale. The fibres were then submerged in water for 24 h. After 24 h, the fibres were removed from the water and placed on a drying rack to be air-dried for 1 h, ensuring the fibres were well dispersed.

After an hour, the fibres were transferred to a clean aluminium dish and placed in an oven at 50 °C, for 24 h. After 24 h, the fibres were re-weighed and the change in water content was measured using Equations (1)–(3), as follows:

$$24 \text{ h Absorption \%} = \frac{M_{\text{Dry final}} - M_{\text{Dry Initial}}}{M_{\text{Dry Initial}}} \times 100 \quad (1)$$

$$M_{\text{Dry Final}} = \text{Mass of Cotton Fibres after 24 h in Oven} \quad (2)$$

$$M_{\text{Dry Initial}} = \text{Mass of Cotton Fibres before being submerged in water} \quad (3)$$

2.4.2. Workability and Density

The workability of the fresh mortar was assessed using the flow table test, in accordance with ASTM C1437 [29]. Two readings were taken and the average was reported. The densities of hardened mortar samples at 7, 28 and 56 days were determined in accordance with AS 1012.12.1-1998 (R2014) [30]. Three cube samples of each mix were used and the average was reported.

2.4.3. Mechanical Tests

For this research study, mechanical tests involved compressive and flexural strength. These tests were performed to observe the effect of cotton fibres on the mechanical properties of mortar. The compressive strength test was carried out following the method outlined in AS 1012.9:2014 [31]. The tests were performed on cube samples with dimensions of 70 mm, using a universal testing machine and keeping a loading rate of 0.5 N/mm²/s. They were tested on days 7, 28 and 56 of curing.

The flexural strength tests of the cement mortar prisms were conducted using a three-point bending test in accordance with ASTM C348 [32]. The flexural tests were performed on the prisms of 40 × 40 × 160 mm, using a Geo-con 10 kN loading frame, at a displacement rate of 0.05 mm/min. Tests were carried out on day 28 of curing.

2.4.4. Water Absorption

The water absorption test involved the preparation of 3 samples on day 56 of curing. The samples followed AS1012.21-1999 (R2014) [33] for the water absorption of hardened concrete. The mass of each sample was recorded before being submerged in room-temperature water for 48 h. After 48 h, the samples were removed and re-weighed at surface-dry conditions (M1). The samples were then moved to a 100 °C oven for a further 24 h. After 24 h, the samples were removed and re-weighed (M2). The total absorption for each sample was calculated using Equation (4), as follows:

$$\text{absorption (\%)} = \frac{M1 - M2}{M2} \times 100 \quad (4)$$

2.4.5. Scanning Electron Microscopy (SEM) Analysis

To observe the microstructure of each mix, a trimming of a sample from each mix was prepared for scanning electron microscopy (SEM) analysis. The SEM used to complete this analysis was a Zeiss Sigma VP, with low-vacuum conditions for very porous materials. To obtain clear images of the microstructure, backscattered detection was used at 15 kV acceleration voltage. To confirm the chemical composition and location of cotton fibres throughout each mix, energy dispersion X-ray spectroscopy (EDS) was also used.

3. Results and Discussion

3.1. Water Absorption of Cotton Fibres

A water absorption test on the cotton fibres indicated that their 24 h absorption was approximately 2 grams per 1 gram of cotton fibre. Therefore, the additional water added

to each mix was two times the quantity of the cotton fibres. While there is insufficient literature to support the exact absorption rate of cotton fibres, this 24 h approximation should be sufficient for the low quantity of fibres added in this research study.

3.2. Workability

As the cotton fibre content increased, a steady decrease in the workability of the mixture was observed, and the added superplasticiser began to have a minimal effect on the flow diameter. The results are shown in Table 2. There were minimal improvements to the workability of the mix as the amounts of superplasticiser were increased. This was likely to be because the cotton fibres absorbed some of the superplasticiser while in the wet mixture; therefore, it was not well dispersed considering the cement particles [11]. Similar results were reported in [34]: the addition of fibres absorbed the superplasticizer and nullified its effect in cementitious composites. It is believed that the absorbing phenomenon is generalized for every type of superplasticizer and must be considered while working on the mix design of cement composites with cotton fibres. The dosage of superplasticizer used was similar to the dosage used for the optimum mix, C0.8, and the flow diameter of mixes with 20 mm and 40 mm fibres was 170 mm. It seems that the fibre length showed no significant effect on the workability of the mortar, compared to the flow diameter of mix C0.8. Similar results were reported by Banfill et al. [35]: increasing the fibre length and fibre content increased the workability of the cement mortar.

Table 2. Flow table workability measurements.

Mix	Average Flow Diameter (mm)	Superplasticiser Used (mL)
C0.0	170	15
C0.4	170	15
C0.8	160	25
C1.6	140	45
C2.0	130	60
C0.8-S	170	25
C0.8-L	170	25

3.3. Density

The density of the mortar samples in their hardened states on days 7, 28 and 56 of curing are demonstrated in Figure 5. The reported densities were the average of the three samples for each mix at the specific curing intervals. The density of the control mix increased on days 28 and 56, compared to the density on day 7. The densities of the mixes with cotton fibres showed irregular patterns; however, there was not much difference among the densities of the mixes with cotton fibres on days 28 and 56. The C0.8 sample had the highest density—2401 kg/m³—on day 7 of curing, but this reduced by 8.45% and 10.58% on day 28 and 56, respectively. The slight change in the densities may have been because of the amount of superplasticizer used in each mix. The presence of superplasticizer affected the workability and compaction of the samples and had a relative effect on the densities of the mixes with cotton fibres. The presence of superplasticizer induced air entrainment in the mixes and caused slight variations in the densities of the mortar samples [36].

When we analysed the influence of the length of the cotton fibres on the density of the mortar mixes, as shown in Figure 5, we observed a slight difference among the densities of the mortar samples. The samples with 30 mm fibre lengths showed the highest densities on days 7, 28, and 56 of curing. The densities of all the mortar samples, with varying fibre lengths, showed a similar trend, with decreases in their densities on days 28 and 56 of curing. The density of the samples with 20 mm cotton fibres on day 28 of curing were exceptions to this trend; a slight increase in density was observed on these 28-day mortar

samples, with 20 mm cotton fibres. Primarily, this was because of the lower rate of water loss compared to the other samples. These density results indicate that the presence of cotton fibres produces additional pores in the microstructure, causing a reduction in density. This aligns with the results in a previous study [37].

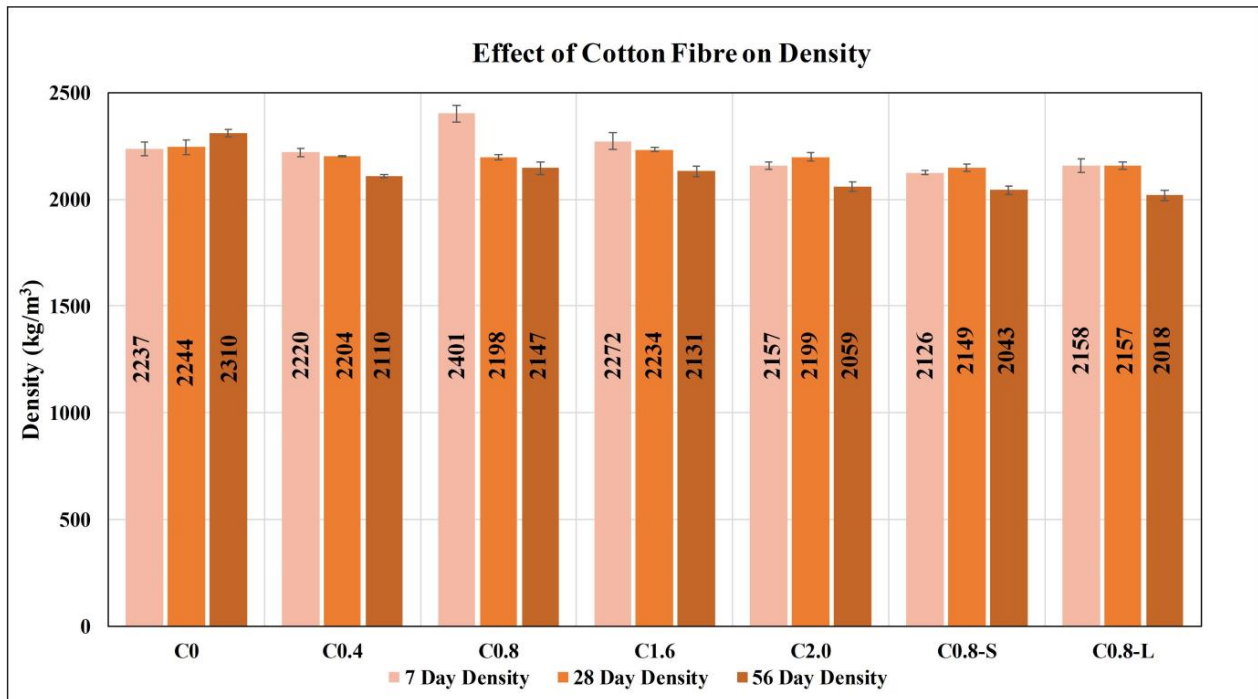


Figure 5. Density comparison at all curing ages for varying fibre contents.

3.4. Compressive Strength

The compressive strength tests were performed for all of the mixes on days 7, 28 and 56 of curing. Three samples per curing age were tested to determine the average compressive strengths. The failure pattern in the samples with and without cotton fibres are shown in Figure 6. The control samples without cotton fibres typically failed in a brittle and explosive manner, while the samples with cotton fibres showed ductile failure. The ductile failure pattern indicated the bridging effect provided by the cotton fibres.

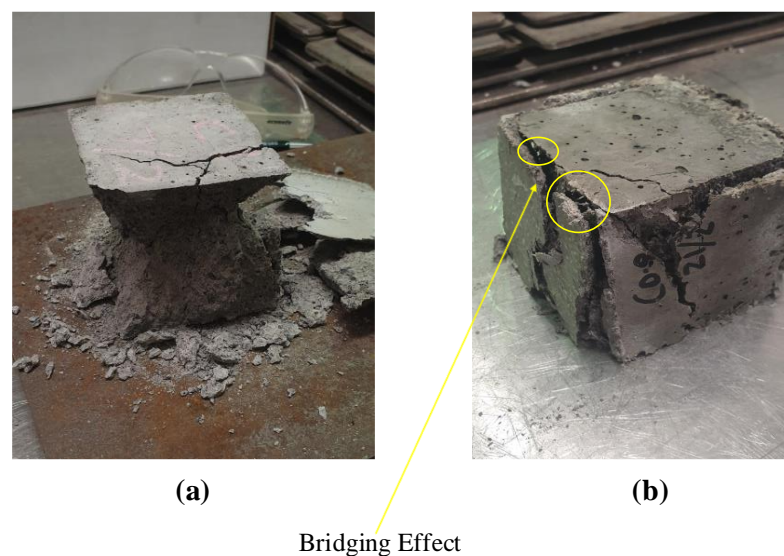


Figure 6. Failure mechanism: (a) without cotton fibres and (b) with cotton fibres.

When comparing the compressive strengths at all of the curing ages, the incorporation of cotton fibres showed an overall reduction in compressive strength, as shown in Figure 7. The mixes containing lower cotton fibre quantities showed better compressive strength than the mixes with a higher cotton fibre content at all curing ages.

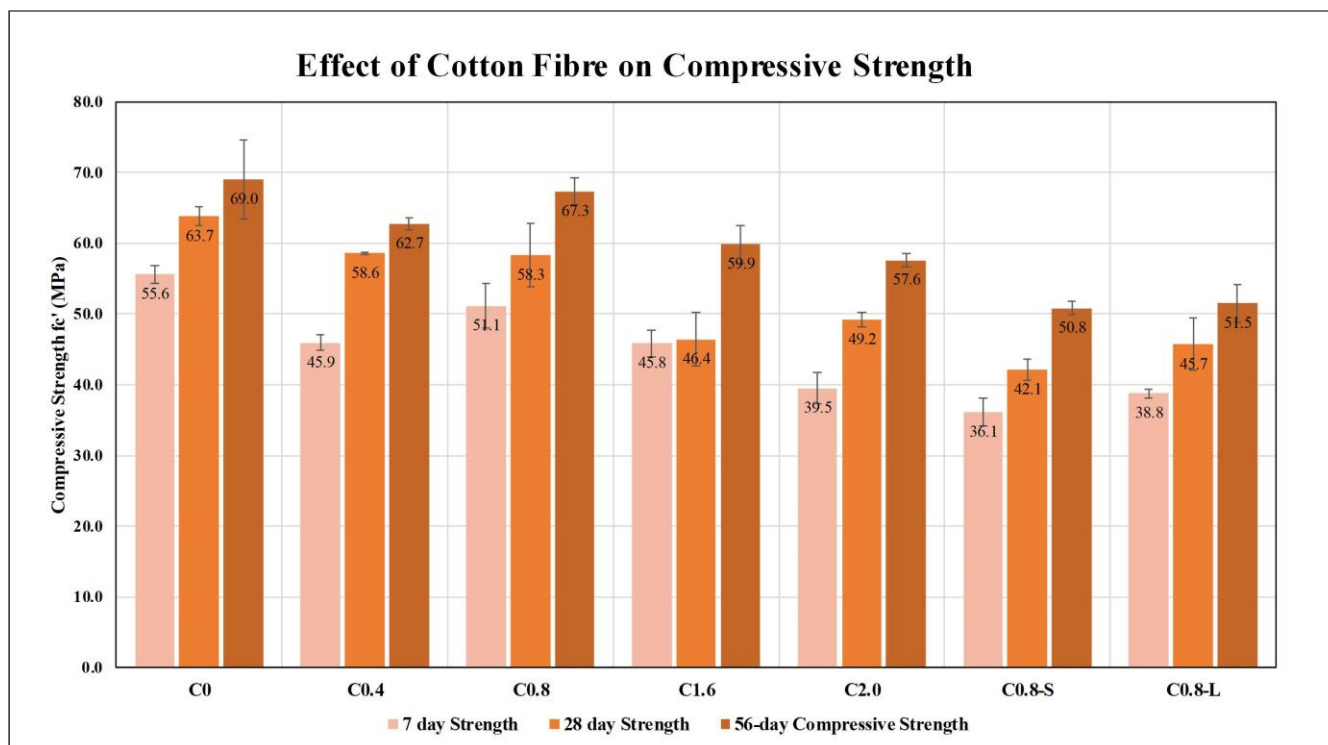


Figure 7. Effect of cotton fibre content on compressive strength.

The control mix demonstrated the highest compressive strengths: 55.6 MPa, 63.7 MPa and 69 MPa on days 7, 28 and 56, respectively. When comparing the 7-day compressive strength of the control mix with the compressive strength of the mixes containing cotton fibres, there was a noticeable decline, with mixes C0.4, C0.8, C1.6 and C2.0 reducing by 17.4%, 8.1%, 17.6% and 29%, respectively. A similar trend was observed on both days 28 and 56 of curing.

The C0.8 mix had the highest compressive strength of all mixes containing cotton fibres and was the most comparable to the control mix at all curing ages. The decrease in the compressive strength of mix C0.8 compared to the control mix were only 8.1%, 8.5% and 2.5% on days 7, 28 and 56 of curing, respectively.

When comparing the influence of the fibre content on the compressive strength of cement mortar, an optimal quantity (C0.8) was found. At this quantity, we observed improvements in compressive strength, and a decrease in the samples' compressive strengths were observed as the quantity of cotton fibres were increased [12,38]. The decrease in the compressive strength of the mixes containing cotton fibres was due to the complex structure of the cotton fibres, which occupied additional space in the matrix in a highly variable orientation. The orientational variability that was interfering with the interfacial bonding between the cement and aggregate led to a reduction in the compressive strength of the mortar mixes containing cotton fibres. Vailati et al. [38] reported that the orientation of fibres played an important role in the strength mechanism of mortar. In addition, the evaporation of the initially absorbed water by the cotton fibres led to a decrease in the compressive strength of the mixes containing cotton fibres. Kubica and Galman [39] reported that the retention of the initially absorbed water has a significant influence on the strength of the mortar. When observing the effects of the fibre length on the compressive strengths

of the mixes, the 30 mm fibre length had the highest compressive strengths at all curing ages, as shown in Figure 7.

There was a significant decrease in the compressive strength of the mixes containing 20 mm and 40 mm fibres—29.3% and 24.2%, respectively—compared to the 7-day compressive strength of the mix containing 30 mm fibres. Similarly, the 28-day compressive strengths of the mixes with 20 mm and 40 mm fibre lengths were 27.7% and 21.6% lower, respectively, than the 28-day compressive strength of the mixes with 30 mm fibre lengths. Furthermore, the 56-day compressive strengths for the 20 mm and 40 mm fibre lengths were 24.5% and 23.5% lower, respectively, than the 56-day compressive strength of the mixes with 30 mm fibre lengths. As all the mixes were based on the C0.8 mix design, it can be concluded that fibre length does affect the compressive strength of mortar. The lower compressive strength for the samples containing 20 mm and 40 mm fibres could be attributed to the quantity and dimensions of the fibres in the matrix. As found in the literature, the dimensions of the fibre correlate to a higher probability of defects such as kink banding and fibre entanglement [10]. While fibre entanglement would be the reason for the reduced compressive strength in the samples containing 20 mm fibres, kink banding could be the reason in samples containing 40 mm cotton fibres. This is because an increased fibre length is more likely to develop kink banding in individual fibres, thus reducing the compressive strength of the mortar.

3.5. Flexural Strength

The flexural strength of each mix was determined using the three-point bending test, after 28 days of curing. All the mixes containing cotton fibres displayed a brittle failure mechanism, with some fibres visibly bridging between the failure surface. When comparing the cotton fibre content of all of the mixes to the control mix, there was a general decrease in flexural strength with the addition of cotton fibres, as shown in Figure 8. Mixes C0.4, C1.6 and C2.0 had an average flexural strength of 8.94 MPa, 9.1 Mpa and 8.95 Mpa, respectively, which were approximately 13.5% lower than the control mix. However, the C0.8 mix displayed an improved flexural strength of 11.35 MPa, which was a 9% improvement on the control mix.

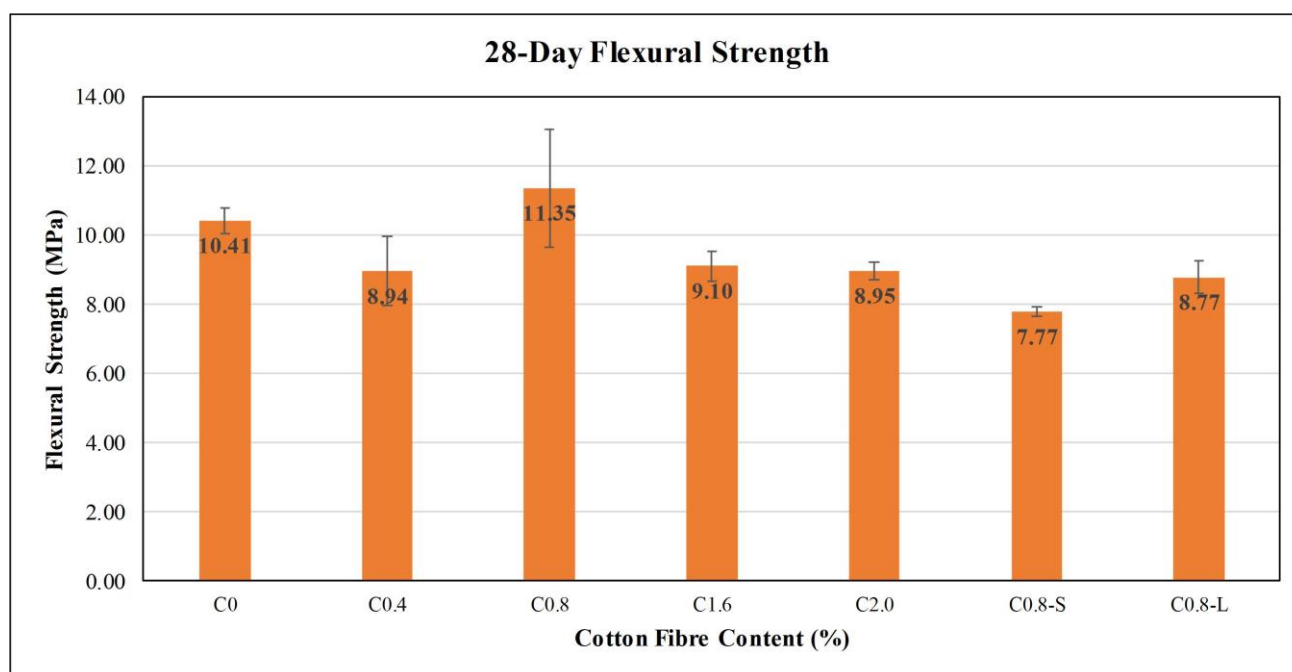


Figure 8. Effect of cotton fibre content on flexural strength.

When observing the influence of cotton fibre lengths on the flexural strength of the samples, the mix containing 30 mm fibres had the highest flexural strength, at 11.35 MPa, as shown in Figure 8. This improvement might be due to a combination of fibre-bridging in the microstructure and of a reduction in the crack propagation, as discussed above (see Figure 6). Furthermore, sufficient fibre bonding might occur in the interfacial transition zone (ITZ). A further discussion on microstructure can be found in Section 3.7, where the microstructure is discussed in detail, alongside microscopy images. Ozerkan et al. [40] also reported similar findings: the addition of fibres improved the flexural performance of the mortars but decreased their compressive strength. This could be attributed to the orientation of the cotton fibres in the matrix [41]. The orientation of the fibres in the cement composites played a vital role. The fibres that were oriented perpendicular to the crack may have helped in delaying or reducing the crack opening through a bridging effect [42].

The mix containing 20 mm and 40 mm fibres showed a reduction in flexural strength. The flexural strength of the mixes containing 20 mm and 40 mm fibres were 31.5% and 22.7% lower, respectively, than in the mix containing 30 mm fibres. This suggested that the geometry of the 20 mm fibres could be too small to cause the flexural improvements needed for the cotton fibres to bond effectively with the cement matrix. However, the 30 mm fibres were of sufficient dimensions to influence the microstructure and to develop a fibre-bridging effect. The reduced flexural strength of the mix containing 40 mm fibres could be a result of fibre entanglement throughout the sample, which could have led to a weaker bonding with the matrix.

3.6. Water Absorption

The water absorption test was performed for all mixes on day 56 of curing. Given the hydrophilic properties of cotton fibres, it was expected that the mixes with an increased fibre content would retain additional water. When comparing all of the mixes to the control mix, a steady increase in absorption could be observed, as shown in Figure 9. Mixes C0.4, C0.8, C1.6, C2.0, C0.8-S and C0.8-L showed an increase of 16.5%, 9.0%, 49.5%, 55.1%, 58.6% and 64.0%, respectively. The mix containing 40 mm fibres had the greatest increase in water absorption. By comparing the average 56-day densities to the 56-day water absorption of all the mixes, a strong correlation was observed between the absorption and density of all in them.

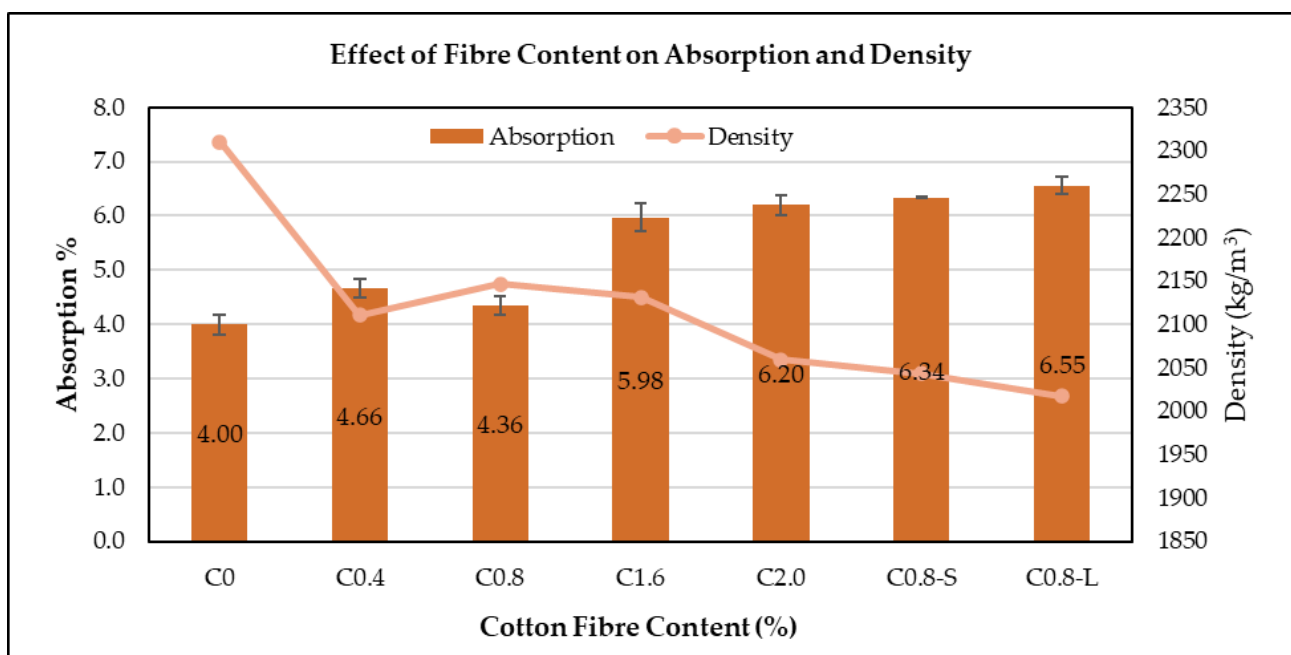


Figure 9. Effect of fibre content on 56-day water absorption and density.

As shown in Figure 9, the mixes with higher densities had a lower absorption rate. This suggests that mix C0.0 and mix C0.8 contained less voids than C0.4, C1.6, C2.0, C0.8-S or C0.8-L. When comparing the effects of fibre length, the mix containing 30 mm fibres had the highest density and lowest absorption, whereas the 40 mm mix had the highest absorption and lowest density. This further confirmed that the increased presence of fibres generated more pores in the microstructure. Similar results were reported by Ziada et al. [43]: the addition of fibres increased the water absorption of cementitious composites. In other studies, natural fibres have been pre-treated using various techniques to reduce the water absorption [44]. In this study, the cotton fibres were pre-soaked and dried for this reason. Although the synthetic fibres might have lower water absorption, the use of natural fibres enhances the sustainability of the building materials [45].

3.7. Scanning Electron Microscopy (SEM) Results

The microstructural formation of the mortar mixes with cotton fibres were observed through scanning electron microscopy (SEM). The SEM analysis was conducted on each of the mixes containing 30 mm cotton fibres. To validate the SEM findings, an additional energy dispersion X-ray spectroscopy (EDS) was used to determine the chemical composition corresponding to each SEM image. The morphologies of all of the samples are shown in Figure 10. It was confirmed that the microstructure was uniform in the cement regions of all of the mixes and that, overall, it was very dense, with some porous regions present.

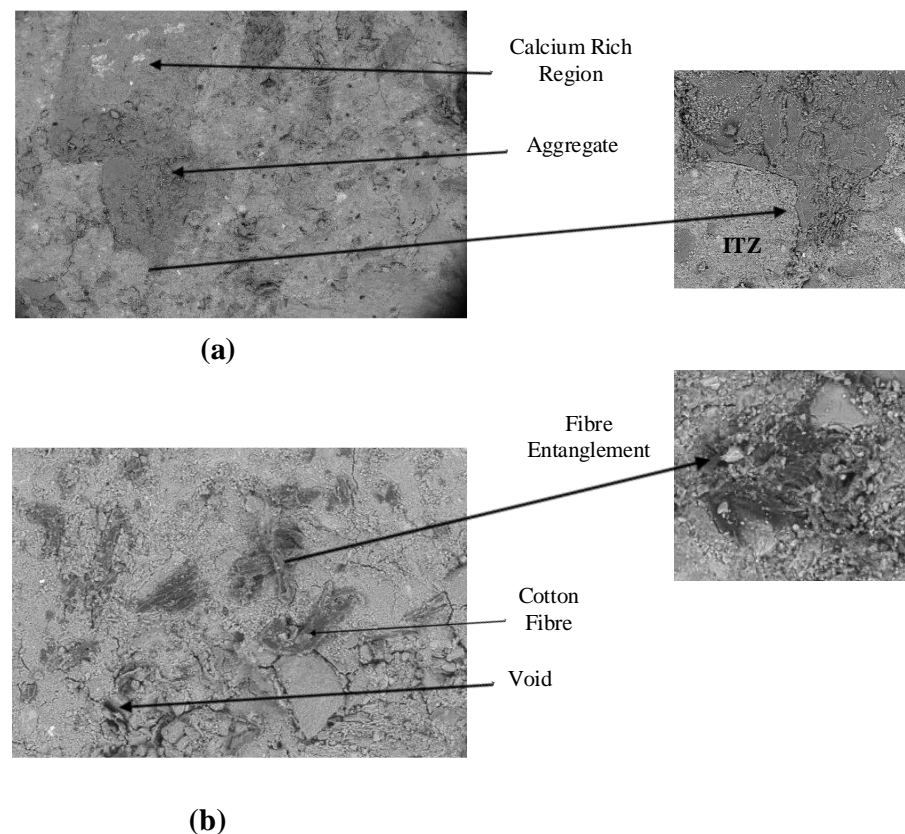


Figure 10. SEM images: (a) without fibres and (b) with cotton fibres (C1.6).

When comparing the SEM images for all the mixes, the C0.0 mix (Figure 10a) displayed very dense cement regions and minor cracking between the ITZ, in a mostly linear orientation. Some dark spots could be observed, but very minimal voids were present. Figure 10b shows a sample with cotton fibres, which showed higher porosity and significant dark spots throughout the matrix. In Figure 10b, two fibres appeared to intersect with one another, causing some fibre entanglement. The close-up image appears to capture an agglomeration

of cotton fibres, causing fibre entanglement. These fibres did not bond effectively with the matrix, and there was significant micro-cracking spalling from the edges of the fibres.

When comparing these SEM images to the mechanical properties of each sample, Figure 10b confirmed that the increased cotton fibre content caused agglomerations of fibres throughout the matrix, increasing the probability of fibre entanglement and kink banding. When cracking propagates at sharp angles it indicates a poor interfacial bonding between the cement and the aggregate, or the presence of aggregates embedded beneath the surface of the matrix. When analysing the microstructural images for the C1.6 mix, we observed improvements in the cotton fibre and cement bonding, as shown in Figure 11.

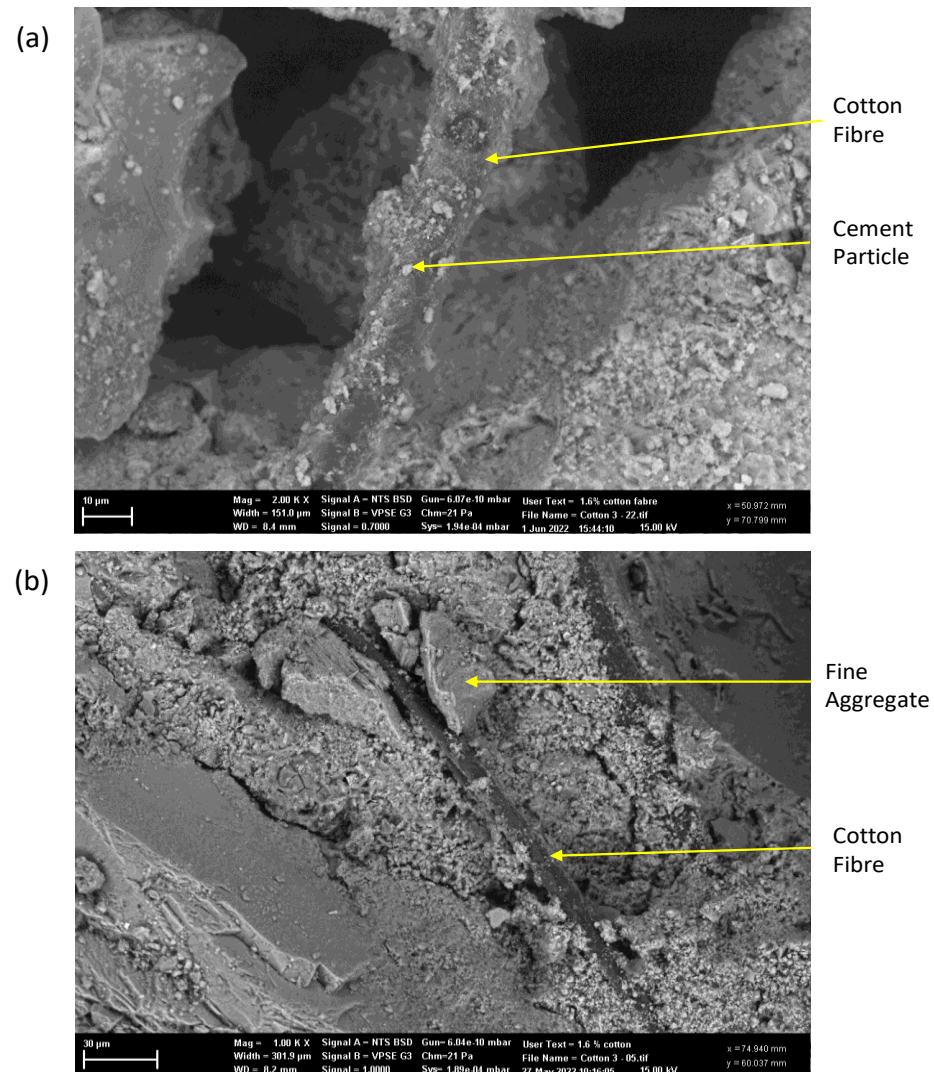


Figure 11. Fibre bridging in sample C1.6: (a) cement-coated fibre and (b) poorly-coated fibre.

Figure 11a shows a single, cement-coated cotton fibre bridging a void, with fine aggregate embedded within the void. The cotton fibre was well coated with cement particles, hence, the strong bonding between the void boundaries. Similarly, Figure 11b shows a cotton fibre bridging between two different cement regions. This fibre was poorly coated with cement particles and was most likely only fixed at the ends of the fibres. The flexural improvements in all the samples could have been because of the fibre bridging effect. However, due to the increased quantity of fibres in the matrix, kink banding and fibre entanglement was most likely the reason for the variability in the flexural strengths [10].

This suggested that the cotton fibre distribution was likely to be the most influential reason for the flexural improvements shown in mix C0.8, rather than the quantity of

the fibres. The use of the proper type and quantity of superplasticizer also helps in the distribution of fibres. In addition, care must be taken while adding the fibres into the matrix to reduce the chances of the agglomeration of fibres in the cement matrix [14].

To confirm the chemical composition of the mixes, an EDS analysis was used to confirm the location of the cotton fibres in the SEM images and to investigate the dark spots throughout the cementitious regions. The EDS analysis was conducted at two locations to compare the atomic differences, and to confirm the darker regions in all of the SEM images where cotton fibres were present.

Provided that the cellulosic chemical composition of cellulose is $C_6H_{10}O_5$ [46], it was expected that a carbon to oxygen ratio of 1.2:1 would be observed in the spectroscopy results. As shown in Table 3, the quantitative analysis of both points showed significant traces of carbon and oxygen, with residual quantities of silicon and calcium.

Table 3. EDS chemical composition of points 1–6.

Point 1		Point 2		Point 3		Point 4		Point 5		Point 6	
Element	Atom (%)	Element	Atom (%)	Element	Atom (%)	Element	Atom (%)	Element	Atom (%)	Element	Atom (%)
O	44.68	C	61.45	C	62.69	C	60.68	O	44.80	O	64.78
C	43.48	O	33.26	O	33.33	O	36.03	C	40.53	C	13.15
Ca	7.19	Ca	2.36	Ca	1.76	Ca	1.75	Ca	8.25	Ca	12.33
Si	2.94	Si	1.88	Si	1.44	Si	1.02	Si	4.02	Si	5.97
Al	0.75	Al	0.48	Al	0.38	Al	0.26	Al	1.01	Al	1.79
Mg	0.30	Na	0.23	Na	0.13	Na	0.11	Na	0.040	Na	0.61
Na	0.22	K	0.14	Fe	0.11	K	0.08	Mg	0.33	Mg	0.44
Fe	0.19	Fe	0.13	K	0.11	Mg	0.06	Fe	0.28	Fe	0.41
K	0.17	Mg	0.07	Mg	0.06			K	0.24	K	0.34
S	0.08							S	0.14	S	0.17

When quantifying the atomic distribution for both point one and two, approximately 66% of the chemical composition was carbon and 30% was oxygen. Calcium and silicon only represented 1% of the chemical composition, and the trace amounts of aluminium, sodium and potassium were 0.45%, 0.24% and 0.31%, respectively. The EDS results at points one and two suggested that the carbon to oxygen content of 2:1 was not expected for a cellulosic fibre. Therefore, additional EDS locations were selected, as shown in Figure 12.

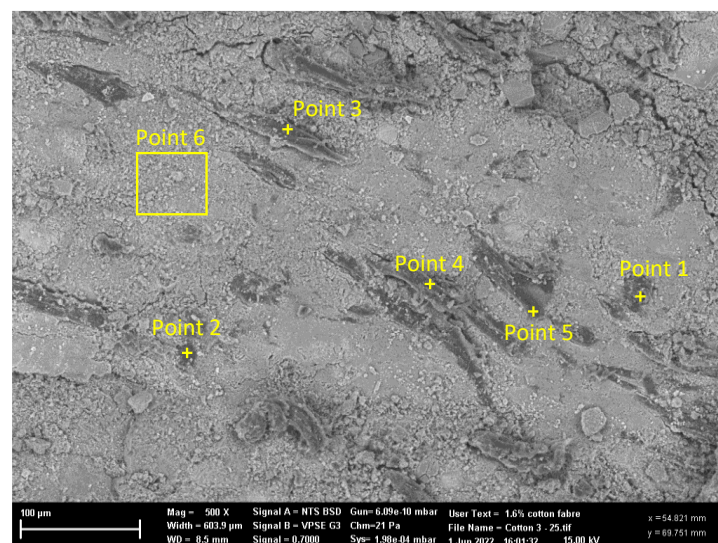


Figure 12. EDS Spectroscopy, points 1–6.

Points one and two were chosen to investigate the dark spots that recurred in all the mixes. Points 3, 4 and 5 were selected as these were expected to display cotton fibres. The area at point six was selected as a control point, to determine the chemical composition of the cementitious region.

As shown in Table 3, the EDS concluded that points one and five contained comparable chemical distributions, with carbon to oxygen ratios of 1.1:1, indicating the expected cotton fibre properties. Points two, three and four showed an increased carbon content, similar to the early cotton fibre findings, where the carbon to oxygen ratio was 1.6:1. The high content of carbon might be due to the presence of superplasticizer, as reported by Ma et al. [47], who suggested that the polycarboxylate-based superplasticizer caused significant concentrations of carbon in cement paste. The cement region at point six contained a typical cement–mortar chemical composition, where higher amounts of oxygen, calcium and silicon were present compared to points one to five [48].

After analysing the spectroscopy for the points shown in Figure 12, we concluded that the superplasticiser was most likely being absorbed by the cotton fibres, which increases the carbon content and leads to a carbon to oxygen ratio of 1.6:1.

When comparing the SEM analysis to the mechanical properties of each sample, we concluded that the inclusion of cotton fibres introduced additional voids in the matrix, therefore, reducing its density. This supports the results obtained through absorption tests, where the samples with higher quantities of fibres increased the voids, and where the water was likely to have been retained. This caused a higher absorption than in the samples with less fibres.

When justifying why the C0.8 mix displayed the best mechanical properties compared to other samples, it was likely that the dispersion of the fibres was more consistent throughout the sample, allowing cement particles to bond more effectively with the fibres. When the fibres were more evenly distributed in the cement matrix, there were less observable void areas and better interfacial bonding between the cement, aggregate and fibres. The microstructural analysis concluded that the dispersion of fibres was more influential on the mechanical properties than the quantity and length of the fibres. In general, the efficiency of the cotton fibres in cementitious composites greatly depends upon the dispersion, orientation, quantity and absorption capacity of the fibres. These factors can be minimized by implementing proper techniques and using a suitable mix design. The impact of the dispersion of cotton fibres on the durability properties of cement mortar could be considered in further investigations.

4. Conclusions

This research investigated the properties of cement mortar that incorporates waste cotton fibres. A total of seven mixes were prepared to determine the effects of the quantity and length of cotton fibres on the physical properties of cement mortar. The percentages of the cotton fibres that were added were 0.4%, 0.8%, 1.6% and 2.0% of the weight of the cement. The lengths of cotton fibres that were used were 20 mm, 30 mm and 40 mm. The addition of the cotton fibres reduced the workability and density of the cement mortar. The flexural strength of the cement mortar containing cotton fibres was enhanced by up to 9%, compared to the flexural strength of the control samples. The compressive strength of the mortar samples generally decreased with the increase in the fibre content and length. However, the C0.8 mix showed comparable compressive strengths to the control mix at all curing ages, with a slight decrease of 2.5% on day 56 of curing. SEM images showed the voids in the mortar samples with high percentages of cotton fibres. Overall, 0.8% by the weight of the cement is the optimum quantity of cotton fibres for improving the flexural performance of cementitious composites. This could enhance the recycling of textile waste and would promote the use of natural fibres rather than synthetic fibres. In future studies, the effect of the addition of cotton fibres on the durability of cementitious composites could be investigated.

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