Mechanical and Physical Properties of Cement Mortar Incorporating Pine Needle Fibers, Silica Fume, Metakaolin

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This study investigated the physical and mechanical properties of cement mortar incorporating pine needle fibers, silica fume (SF) and metakaolin (MK). The study had two phases. In Phase I, we compared six combinations of silica fume (SF) and metakaolin (MK), including an unmodified mortar control (S0-M0) and five mixtures with 20% total cement replacement. The mix with 10% SF and 10% MK achieved a 28-day compressive strength of 75.3 MPa, which is a 37.7% increase compared to the plain mortar. In Phase II, pine needle fibers were added at 0% to 10% by mass of total binder, and the water-to-binder ratio varied from 0.35 to 0.60. A total of 270 specimens were tested at 7, 14, and 28 days. Among the Phase II mixtures, the 4% fiber mix showed the highest average compressive strengths of 60.3, 71.0, and 76.6 MPa at 7, 14, and 28 days, and the highest 28-day modulus of rupture (MOR) of 14.7 MPa. Compared with the Phase II fiber-free reference mix (S10-M10-F0), the 4% fiber mix showed increases in average compressive strength of 5.4%, 7.9%, and 4.5%, and increases in MOR of 15.7%, 11.7%, and 14.0% after 7, 14, and 28 days, respectively. The same mixture also had 6.9%, 3.1%, and 1.2% lower oven-dry density and 115.4%, 57.5%, and 77.1% higher water absorption at 7, 14, and 28 days, respectively. These comparisons are based on reported mean values and were not tested for statistical significance. Because fiber content and water-to-binder ratio varied together, their individual effects cannot be separated. The results indicate that the 10% SF–10% MK binder combination and the 4% fiber formulation are the best-performing mixtures among those tested, although the individual effects of fiber content and water-to-binder ratio cannot be separated.

Keywords: Pine Needle Fibers; Silica Fume; Metakaolin; Compressive Strength; Water Absorption

Introduction:

Cement is the principal binder in mortar; sand, water, and admixtures are selected according to the intended application. Cement mortar composites are renowned for their versatility in construction and are widely used in activities such as plastering, masonry, concrete structure repairs, patching, floor leveling, rendering, and crafting precast structures and products. The need for high-performance cement composites is crucial, especially in applications such as prompt concrete repair, roof treatments, high-compressive-strength precast structures, rigid pavements, flooring, and rehabilitation projects. To enhance the mechanical and physical properties of cement composites, both artificial and natural fibers are utilized as reinforcements within their matrices. These fibers play a crucial role in improving toughness, ductility, bending strength, resistance to fatigue and the ability to withstand high impact loads. These fibers can also help reduce shrinkage and bleeding while the mortar is still wet. Once the mortar hardens, how much the fibers affect permeability depends on the fiber type, the amount used, and how well they are mixed. Moreover, natural fibers possess several advantageous qualities, including renewability, cost-effectiveness, non-abrasiveness, widespread availability, and reduced health and safety concerns throughout their processing and handling [1]. Pine needle fibers have been investigated as reinforcement in cementitious composites due to their availability as an agricultural byproduct. Some natural-fiber composites have been reported to exhibit reduced thermal conductivity, depending on the fiber type and composite composition [2][3][4]. The incorporation of natural fibers in traditional cement composites was a common practice. Adding materials to binding agents can be traced back to the ancient Greek era, when wood fibers and straw were used to improve volume stability and durability [5]. Similarly, jute straws in the time of Indo-Muslim architecture were used for strengthening of bonding and decreasing of cracks in members [6]. Natural fibers can improve toughness and tensile strength and may help control plastic-shrinkage cracks. How they affect durability depends on the fiber type and the mix [7][8]. The functions and performance of natural fibers in the composite mix depend primarily on geometric properties such as fiber length, diameter, shape, and mixing proportion [9]. Additionally, the performance of natural fibers in the composite mix also depends on the orientation and dispersion of the natural fibers in the composite mix as well as the type of binding material used [9][10]. To improve the mechanical performance, including compressive and bending strength, pine needle fibers can be used as natural reinforcement in cement composites at an appropriate dosage [11] [12]. Over time, synthetic fibers such as glass and polypropylene, as well as steel fibers, have also been used as reinforcement in cement composites. The American Concrete Institute Committee (ACI 544) groups fiber reinforced concrete by the type of fiber used. The four main groups are steel fiber reinforced concrete, glass fiber reinforced concrete, synthetic fiber reinforced concrete, and natural fiber reinforced concrete [13]. Numerous researchers have worked to understand the role of fibers in cement composites [14]. Kim and Khaloo tested 84 specimens in which the steel fibers were randomly distributed for tensile, compressive and flexural strength. The fibers in the concrete matrix were varied from 0 to 1.5 % by its volume. The results showed that compressive strength improved considerably with steel fiber contents up to 1%, while tensile strength did not depend on the compressive strength of the concrete matrix [15]. [16] examined the mechanical properties of high-strength steel fiber concrete by varying the steel fiber volume fraction from 0.5% to 2%. The results indicated that the compressive strength has been improved by 15.3 % with 1.5 % volume fraction of steel fibers. Moreover, Alsadey investigated the effects of polypropylene fibers on the mechanical properties of cement mortar at volume fractions ranging from 0.5% to 1.5%. The results indicated that compressive strength after 28 days was numerically higher in some fiber-containing mixtures [17]. [18] cement composites with carbon nanofibers. The results showed that compressive strength improved, probably because

the matrix porosity was reduced, but the flexural strength did not change. [19] looked at basalt fibers in concrete and compared them to glass and carbon fibers. The results showed that basalt fibers had lower strength, but they were more stable at temperatures above 600°C.

A study [20] investigated the use of synthetic fibers in lime-based mortars at various dosages from 0.5% to 2% by volume. The results of their study showed that increasing synthetic fiber content also enhanced the toughness of the composites. The results also indicate that 0.5% fiber content gave the best dispersion and highest toughness compared to other mixtures.

Due to environmental concerns and the vulnerability of artificial fibers, the demand for sustainable alternatives has increased, directing attention back to natural fibers. Natural fibers are economical, abundantly available, lightweight, and possess excellent energy absorption capacity. Common types of natural fibers include pine needle, palm, coconut coir, jute, hemp, rice straw, sisal, bamboo, and flax. Over the past decade, pine needle fibers have attracted significant interest as reinforcement in low-cost structures due to their toughness and abundance as forest waste. Pine needles are widely available in parts of Pakistan and can be collected from forest waste to use as natural reinforcement. While fallen pine needles and other forest waste might also be useful as fuel, we did not test their fuel potential in this study. Despite these benefits, several studies indicate that incorporating pine needle fibers in cement composites typically reduces compressive strength due to increased porosity. To compensate for these losses, a common approach is to incorporate dense materials that enhance the densification of the cement composite. Research has demonstrated the effectiveness of metakaolin in improving both the physical and mechanical properties of cement paste and concrete in the short and long-term. Previous research found that replacing 10% to 15% of cement with metakaolin gave the best results [21]. Metakaolin is a supplementary cementitious material produced by calcining kaolin-rich clay at elevated temperatures. The pottery industry refers to kaolinite clay as china clay. Metakaolin is made by heating kaolin clay. This process changes the structure of the original clay and creates reactive compounds that help strengthen the mortar. Metakaolin reacts with calcium hydroxide to form additional cementitious compounds, including calcium silicate hydrates and aluminates. Metakaolin can be a very useful material in construction that can partly replace regular cement.

Local clays in Pakistan are often used by the pottery industry. These natural materials might be a good local source for making metakaolin. However, we still need to test if they are suitable and practical for construction use.

[22] did a similar study. They used silica fume, metakaolin, and coconut fibers in cement mortar. Their best mix was 10% SF + 10% MK with 6% coconut fibers. It gave a compressive strength of 78.6 MPa. But pine needle fibers were not tested in this SF–MK system. This is the gap. The performance of pine needle fibers in the specific silica fume–metakaolin mortar system investigated here remains insufficiently established. In this work, we replaced coconut fibers with pine needle fibers while keeping the same SF-MK binder system and experimental framework. The novel contribution of this study is the investigation of pine needle fibers as reinforcement in the SF-MK mortar system, which has not been previously reported. In this study, Phase I binder optimization was repeated using the experimental framework established in our previous study [22]. While the framework was similar, the specimens for Phase I were newly cast and tested for this investigation, which led to slight variations in the results. The primary novelty of this work is the investigation of pine needle fibers in the Phase II program. All experimental results reported in this manuscript were generated from specimens newly cast and tested for the present investigation. This allows the results of the present study to be discussed in the context of the related coconut fiber study, although a controlled direct comparison between fiber types was not performed.

Silica fume is finer than metakaolin. Previous studies show that adding it can improve

the strength of cement mortar. For example, replacing 5% to 15% of cement with silica fume increased compressive strength by 20% to 30% [23]. Metakaolin and silica fume are often added separately to fiber-reinforced mortar. But they can also be combined. Metakaolin improves workability of mortar, while silica fume reduces it. However, if both are utilized together, the negative effect of silica fume on workability is reduced. Both materials can improve strength. When used together, they can produce synergistic improvements in strength [24].

Using pine needles as mortar reinforcement could provide a way to utilize this underused natural resource, which is locally available in many parts of Pakistan. However, the performance of plant-based fibers depends on moisture interaction, their geometry, dispersion, dosage, and compatibility with the cement matrix. Silica fume and metakaolin both are supplementary cementitious materials that can partially replace Portland cement and influence the hydration and packing of the binder. Combining these materials with pine needle fibers gives us a way to study both the physical properties and mechanical performance in a cement mortar system. We did not measure the exact environmental benefits (like carbon emissions) in this study, but we consider sustainability here in terms of replacing some cement and using a plant-based resource.

Recent Literature and Research Gap:

Recent studies have looked at how fiber type and fiber amount affect the properties of cement materials. [25] studied natural fibers and noted that fiber dosage, workability, and durability are important factors. [26] compared natural fibers and synthetic fibers and showed that checking of both mechanical strength and physical properties like density and water absorption is necessary. [27] also reviewed these materials and pointed out ongoing challenges like moisture absorption, fiber dispersion, and durability.

Some studies have looked at pine needles specifically. [28] investigated pine needles in concrete. However, these studies used different mixtures of concrete, fiber amounts, and testing methods compared to our mortar study. Therefore, their numerical results cannot be directly compared to our results.

[22] studied a mortar with silica fume, metakaolin, and coconut fibers. In our study, we used a similar binder system but replaced coconut fibers with pine needle fibers. This work extends the research to a new natural fiber.

The research gap is that there is little experimental information on using pine needle fibers with a binder of 10% SF and 10% MK in mortar. We tested fiber contents from 0% to 10% at 7, 14, and 28 days. Since the water-to-binder ratio also changed with fiber content, our results show the combined effects of these mixture conditions, not just the effect of the fibers. More controlled experiments are needed to separate these effects.

Novelty of the Study:

The main contribution of this research is the investigation of pine needle fibers in a mortar mix containing 10% SF and 10% MK by mass of total binder. Study [22] investigated a similar binder system using coconut fibers. Our study uses pine needle fibers and tests moisture content, compressive strength, water absorption, MOR, and oven-dry density at 7, 14, and 28 days. This extends the research on this SF-MK binder system to a new natural fiber. It also shows how the mechanical and physical properties change with different amounts of pine needle fibers. The fiber mix of 4% performed best in Phase II for average compressive strength and MOR. However, this must be viewed within the study's limits: the water-to-binder ratio changed with fiber content, we didn't have individual specimen data for statistical analysis, and we didn't run microstructural tests. So, the novelty is the new material combination and the experimental comparison, rather than a claim that 4% is a universally perfect fiber amount or that we isolated the fiber's effect.

Research Objectives:

The objectives of this study are:

To compare the compressive strength of six mortar mixes with different silica fume and metakaolin amounts, and to identify the best-performing mix among those tested.

To evaluate modulus of rupture (MOR) and compressive strength of mortar with 0%, 2%, 4%, 6%, 8%, and 10% pine needle fibers at 7, 14, and 28 days.

To measure moisture content, water absorption, and oven-dry density for all tested fiber mixes at different curing ages.

To compare the mechanical and physical results of these mixes and discuss possible reasons based on previous research.

To identify the limitations of this experimental design, especially the changing water-to-binder ratio, and to suggest future controlled experiments.

Experimental Program:

Constituents:

The materials used in this study included Portland cement, sand, metakaolin, silica fume, pine needle fibers, and a water-reducing admixture. Details of each material are provided below.

Cement:

The mortar was made up of locally available ordinary Portland cement (OPC). Table 1 lists its setting times and compressive strengths, and Table 2 shows its chemical composition. The values in Tables 1 and 2 are as reported by the cement manufacturer.

Table 1. Cement setting times and reported compressive strengths

Property	Test age / parameter	Value	Unit
Compressive strength	3 days	27.4	MPa
Compressive strength	7 days	33.50	MPa
Compressive strength	28 days	48.7	MPa
Initial setting time	—	180	min
Final setting time	—	217	min

Table 2. Cement chemical composition

Chemicals	Value (%)
CaO	62.91
SiO2	21.45
Al2O3	4.97
Fe2O3	4.16
SO3	2.50
MgO	2.44
Loss on ignition (LOI)	1.57

Sand:

Locally sourced river sand was utilized in this study. Tables 3 and 4 show the properties of the fine aggregate.

Table 3. Physical properties of fine aggregates

Specific gravity	Fineness modulus	Bulk density loose	Water absorption
2.71	2.66	1.53 kg/L	1.23%

Table 4. Fine aggregate sieve analysis results

Sieve size	Mass retained (g)	Percentage retained (%)	Cumulative percentage retained (%)	Percentage Passing (%)
4.75mm	15.5	1.55	1.55	98.45
2.36mm	101.5	10.15	11.70	88.30
1.18mm	249.5	24.95	36.65	63.35
600 µm	139	13.90	50.55	49.45
300 µm	221	22.10	72.65	27.35
150 µm	202	20.20	92.85	7.15
Pan	71.5	7.15	100.00	0.00
Total	1000	100		

Fine aggregate fineness modulus = 266 /100 = 2.66
Total weight taken = 1000 g

Metakaolin:

Metakaolin is a pozzolanic material and can be made by heating kaolinite clay. Its particle size is intermediate between those of cement and silica fume. We used Sikacrete M-100 (Sika), which meets ASTM C618 Class N requirements. Its properties, as reported in the manufacturer's technical data sheet for Sikacrete M-100, are presented in Tables 5 and 6. The powder form of the metakaolin used in this study is shown in Figure 1.

Table 5. Metakaolin chemical composition

Composition	Value (%)
SiO_2	55.50
Al_2O_3	36.50
Fe_2O_3	2.00
TiO_2	1.00
K_2O	1.70
Na_2O	0.10
LoI (Loss on Ignition)	3.20

Table 6. Metakaolin physical characteristics

Property	Observed Characteristic
Appearance	Off-White
Average grain diameter	1.78 micrometers
Relative Density	2.60
Shape of particle	Rounded
Specific surface area	17,000 m^2/kg



Figure 1. Powder form of metakaolin

Silica Fume:

Silica fume, which is also called microsilica, is a byproduct of silicon and ferrosilicon

production. It contains fine amorphous silica particles (ACI 548.6R-96). Sika Fume from Sika brand has been used in this research. Its properties, as reported in the manufacturer's technical data sheet for Sika Fume, are presented in Tables 7 and 8. The silica fume powder used in this study is shown in Figure 2.

Table 7. Silica fume physical properties

Parameter	Value
Particle average size	0.15 micrometers
Relative Density	2.20
Geometry of particle	circular
Specific surface area	23000 m^2/kg

Table 8. Silica fume chemical composition

Composition	**Value (%)**
SiO_2	92.3
Fe_2O_3	1.0
Al_2O_3	1.3
CaO	1.6
MgO	0.9
SO_3	0.11
Na_2O	0.25
K_2O	0.79
Loss on Ignition (LOI)	1.53

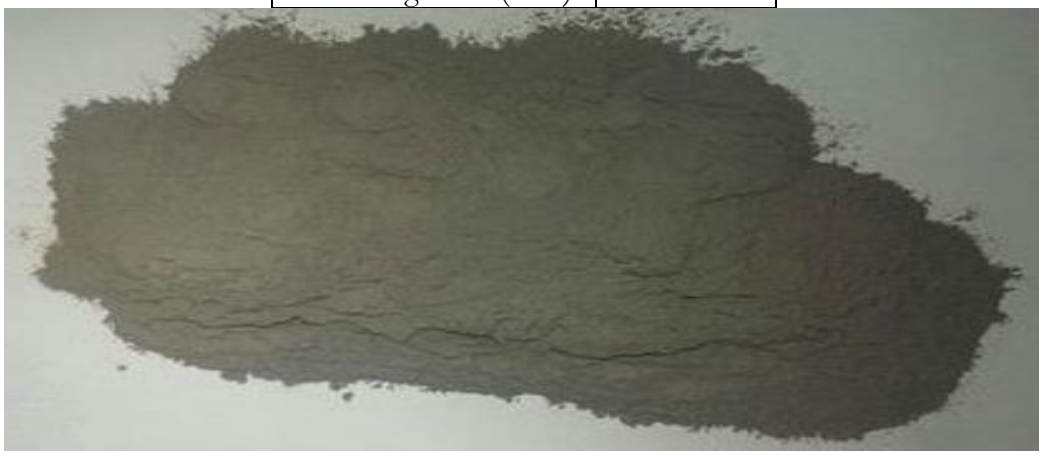

Figure 2. Powder of silica fume

Pine Needle Fibers:

Pine needles were collected from locally available pine trees in the Swat region of Khyber Pakhtunkhwa, Pakistan. After collection, they were washed and sun-dried. Before mixing, the needles were cut into shorter segments to ensure that they could fit inside the 40 mm molds and be distributed evenly throughout the mortar. The fiber percentage was calculated based on the total mass of cement, silica fume, and metakaolin. The collected and prepared pine needle segments are shown in Figure 3.

Regarding fiber characterization: Because the focus of this study was on the macro mechanical behavior of the mortar, the exact botanical species of the pine tree and the precise average diameter of the needles were not measured in the original laboratory records. The fibers were visually verified as local pine needles and cut to fit the specimen molds. This lack of detailed botanical and dimensional data is acknowledged as a limitation for the exact reproduction of the fiber preparation procedure, although the manual layered mixing was used to ensure uniform distribution.



Figure 3. Pine needle fibers

Admixture:

A superplasticizer (Ultra Fluid-235) was added at a dosage of 0.7% by mass of total binder. The admixture was used in accordance with the manufacturer's recommendations, while ASTM C494/C494M (2019) was used as a general reference for chemical admixtures. According to the manufacturer's technical data sheet, this admixture is reported to improve workability, reduce water demand, and enhance finishability. The experimental workflow is shown in Figure 4.

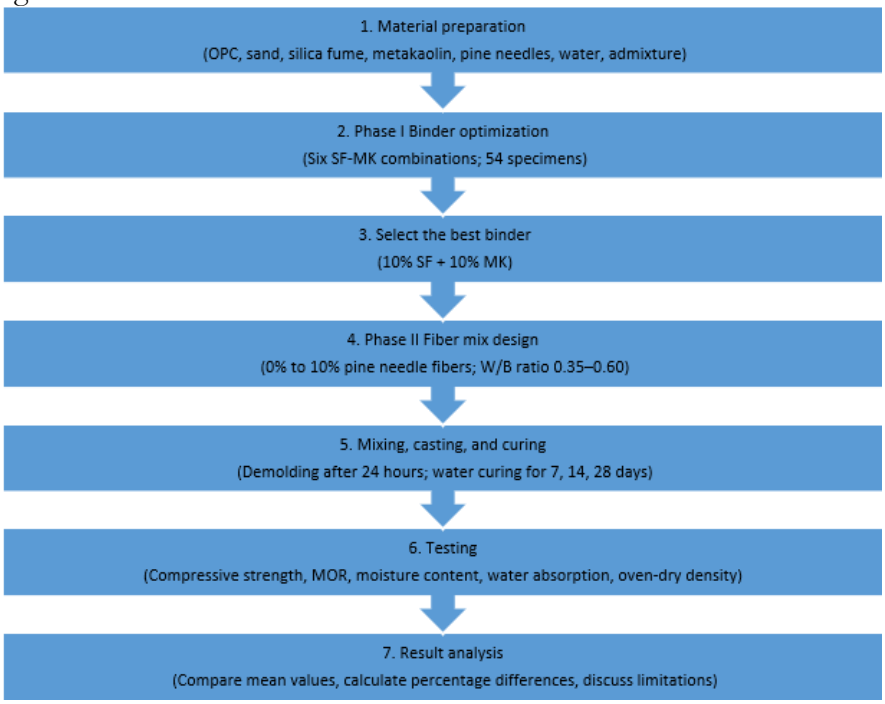

Figure 4. Flowchart of the experimental methodology

Cement Mortar Samples:



Figure 5. Casting process for plain cement mortar

A total of 270 specimens were cast in two different phases. In Phase I, 54 plain mortar specimens (without fibers) were prepared using six SF and MK combinations, namely 0/0, 20/0, 15/5, 10/10, 5/15, and 0/20%, with nine specimens per mix. The specimens (160 × 40 × 40 mm) were tested for compressive strength at 7, 14, and 28 days. The casting process for the plain mortar specimens is shown in Figure 5.

In Phase II, the mix with the highest observed compressive strength (10% SF and 10% MK) was used with 0–10% pine needle fibers. 216 specimens were cast: 54 specimens for compression (160 × 40 × 40 mm), 54 for MOR (400 × 100 × 16 mm), and 108 for moisture and water absorption (100 × 40 × 40 mm). The same specimens were used for both moisture content testing and density measurements. Therefore, the 108 physical property specimens comprised 54 for moisture content and density measurements and 54 for water absorption. The total number of specimens in Phase II is 216 (54 compression + 54 MOR + 54 moisture/density + 54 water absorption). The fiber-free specimens in Phase I and Phase II were cast in separate batches. The fiber reinforced specimens prepared for compressive strength testing are shown in Figure 6. The specimens prepared for modulus of rupture testing are shown in Figure 7, while the specimens used for physical property tests are shown in Figure 8.



Figure 6. Pine fiber-reinforced mortar specimens for compressive strength testing



Figure 7. MOR test samples reinforced with pine needle fibers



Figure 8. Specimens for physical property tests (density, moisture, and water absorption)

The experimental materials used in this study included locally available ordinary Portland cement (Bestway brand), locally sourced river sand as fine aggregate, natural pine needles, silica fume, metakaolin, and a water-reducing admixture. The binder consisted of 80% ordinary Portland cement, 10% silica fume, and 10% metakaolin by mass of total binder. The total binder-to-sand mass ratio was 1:1, while the water-to-binder ratio varied between 0.35 and 0.60 based on natural fiber content. Water was added in stages during mixing to accommodate the required consistency; however, quantitative workability measurements are not reported. A superplasticizer (Ultra Fluid-235) was added at a dosage of 0.7% by mass of total binder. The admixture was used in accordance with the manufacturer's recommendations, while ASTM C494/C494M (2019) was used as a general reference for chemical admixtures.

The experimental program consisted of two phases. Phase I aimed to find the best SF/MK combination for compressive strength. The mix with the highest observed compressive strength (10/10% SF/MK) was then carried forward to Phase II with fiber reinforcement.

In Phase II, mortar samples were reinforced with pine needle fibers using the silica fume and metakaolin percentages determined in Phase I. Various fiber contents ranging from 0% to 10% were tested to evaluate mechanical and physical properties. The mix proportions are detailed in Table 9.

Table 9. Mix design of mortar samples

Sample	Cement%	SF%	MK%	Sand-to-Binder Mass Ratio	W/B ratio	Fiber content (%)
1	80	10	10	1	0.35	0
2	80	10	10	1	0.40	2
3	80	10	10	1	0.45	4
4	80	10	10	1	0.50	6
5	80	10	10	1	0.55	8
6	80	10	10	1	0.60	10

The mixture proportions are reported as percentages of total binder and mass ratios. The original records that contain absolute batch masses were unavailable; therefore, the table reports the documented mixture proportions rather than verified original batch quantities. For clarity, an illustrative normalized mixture can be calculated on a basis of 1,000 g of total binder. On this basis, the binder comprises 800 g of ordinary Portland cement, 100 g of silica fume, and 100 g of metakaolin. The sand mass is 1,000 g for a binder to sand mass ratio of 1:1. Fiber masses corresponding to 0%, 2%, 4%, 6%, 8%, and 10% of total binder are 0, 20, 40, 60, 80, and 100 g, respectively. The corresponding water masses for water-to-binder ratios of 0.35, 0.40, 0.45, 0.50, 0.55, and 0.60 are 350, 400, 450, 500, 550, and 600 g, respectively. These are calculated normalized quantities, not recovered original batch masses. The superplasticizer dosage was reported as 0.7% of total binder.

The water-to-binder ratio increased from 0.35 to 0.60 as the pine needle fiber content increased from 0% to 10%. According to the recorded procedure, water was added in stages in order to accommodate the required consistency during mixing. However, quantitative workability measurements were not recorded, and the original data do not establish a measured relationship between fiber dosage and water demand. The increasing water-to-binder ratio should therefore be regarded as a feature of the tested mixture design rather than as a separately validated effect of fiber content. The illustrative normalized batch quantities calculated from the documented mixture proportions are presented in Table 10.

Table 10. Normalized mix quantities (based on 1,000 g total binder)

Mixture	Cement (g)	SF (g)	MK (g)	Sand (g)	Fiber (g)	Water (g)
F0	800	100	100	1,000	0	350
F2	800	100	100	1,000	20	400
F4	800	100	100	1,000	40	450
F6	800	100	100	1,000	60	500
F8	800	100	100	1,000	80	550
F10	800	100	100	1,000	100	600

Note: A superplasticizer was added at 0.7% of the total binder weight. On this 1,000 g basis, this equals 7 g of admixture. It is not listed in the table above.

The mix proportions are reported as mass ratios and percentages of the total binder because the original records containing absolute batch quantities were unavailable. Although the nominal mixture proportions and water-to-binder ratios are provided, the absence of verified batch masses limits exact reproduction of the original mixtures. The binder consisted of 80% ordinary Portland cement, 10% silica fume, and 10% metakaolin by mass of total binder. The total binder-to-sand mass ratio was 1:1. Pine needle fiber content was calculated as a percentage of total binder mass (cement + SF + MK). All components were measured by mass. The water-to-binder ratio increased across the fiber-reinforced mixes. However, because we did not record quantitative workability measurements, the available data do not establish that this increase was directly caused by the presence of fibers.

For the preparation of the pine needle fiber-reinforced mix, a systematic layering approach was adopted. Fine aggregate was placed at the bottom of the mixing pan, followed by layers of pine needles, cement, and pozzolans. This layering sequence was repeated for the second and third layers. The mechanical mixer ran for 3 minutes and 30 seconds. Water was then added in stages, and mixing continued for another 3.5 minutes. Care was taken to avoid fiber clumps and ingredient segregation. Molds were filled with the mixture in three separate layers, and each layer was compacted to remove entrapped air. After 24 hours, the specimens were demolded and cured in clean water for the specified curing ages of 7, 14, or 28 days.

Testing Procedures:

The cast specimens were handled carefully to avoid damage or contamination. The study was conducted at the Building Material and Construction Laboratory, Department of Civil Engineering, University of Engineering and Technology, Peshawar, Pakistan. The laboratory location is shown in Figure 9.

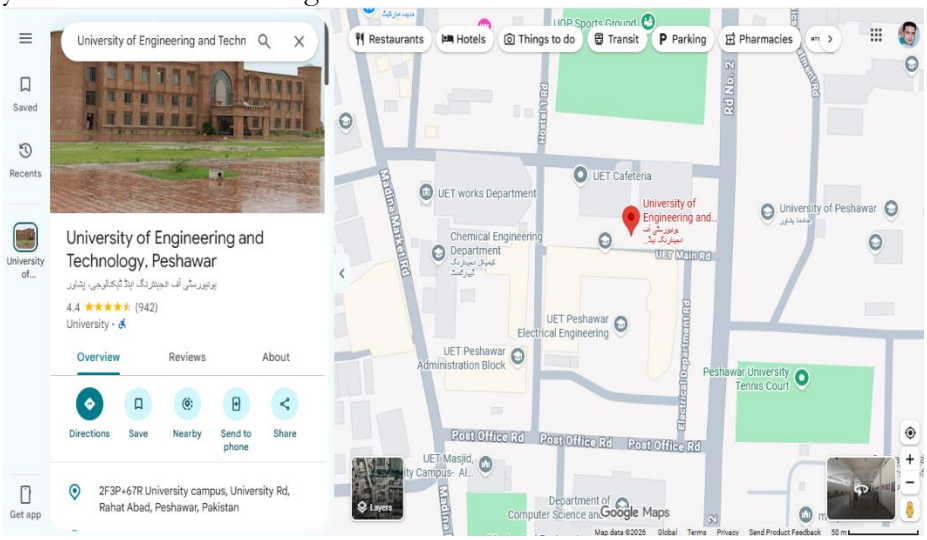

Figure 9. Site map showing the location of the Building Material and Construction Laboratory, UET Peshawar.

The testing procedures described below were carried out using the laboratory-specific methods available at this site. For the compressive strength test, we used a universal testing machine (UTM) with a loading rate of 1.8 kN/s. The dimensions of the samples for this test were 160 mm × 40 mm × 40 mm. Although ASTM C109/C109M (2020) is a recognized standard for hydraulic cement mortar, our specimen dimensions differ from the standard 50 mm cubes. Therefore, the results are reported based on our laboratory method without claiming strict standard compliance. The original records specifying the complete compressive test setup, including the loading orientation and the standard applied, were unavailable for independent verification. The reported compressive strength values are therefore presented as recorded results, with this limitation acknowledged in relation to independent reproducibility.

For the modulus of rupture test, we used specimens measuring 400 mm × 100 mm × 16 mm. The beam was supported at both ends and loaded at the two third points of the support span until failure. MOR was calculated using the standard beam theory equation. The specimen dimensions and loading setup were specific to our laboratory and are not claimed to strictly comply with ASTM C348 (2021). Because the original records did not include the exact support span or fracture location, we cannot independently verify these results for every specimen.

$$MOR = (P \times L) / (b \times d^2)$$

This equation is used because the beam is loaded at the third points, and the maximum bending moment occurs within the middle third of the span. In this equation, MOR is the modulus of rupture (MPa), P is the maximum total applied load at failure (N), L is the distance between the supports (mm), b is the specimen width (mm), and d is the specimen depth (mm). The original records state that third point loading was used, but the exact support span and fracture location were not recorded. Therefore, while the equation matches the intended loading method, strict compliance for every specimen cannot be independently verified.

For the moisture content, water absorption, and density tests, the specimens with dimensions 100 mm × 40 mm × 40 mm were used. For determination of moisture content, the specimens were oven dried at 100°C for 24 hours and weight changes were noted. For the water absorption test, the specimens were oven dried, weighed and immersed in water for 24 hours, and then weighed again. The drying temperature and duration used before immersion were not documented in the original laboratory records. The procedures for removing surface water before weighing specimens for the moisture content and water absorption tests were not documented in the original laboratory records. While ASTM C642 (2021) provides a recognized method for density, absorption, and voids in hardened concrete, the procedures used here are reported transparently as laboratory specific methods rather than fully compliant standard tests.

The moisture content, water absorption, and density were calculated using the following equations:

$$\text{Moisture content (dry basis, \%)} = [(\text{mass before oven drying} - \text{oven-dry mass}) / \text{oven-dry mass}] \times 100$$

$$\text{Water absorption (\%)} = [(\text{mass after immersion} - \text{mass after drying}) / \text{mass after drying}] \times 100$$

$$\text{Density} = \text{mass after drying} / \text{volume}$$

where mass before drying is the mass of the specimen before oven drying, mass after drying is the mass after oven drying, and mass after immersion is the mass after water immersion. Density was calculated using the oven-dry mass. The volume of each specimen was determined by measuring its dimensions (length × width × depth). Two separate specimen groups were used for the physical property tests. One group was used to determine moisture content and oven-dry density, while a second group was used to determine water

absorption. For the moisture content and density measurements, the specimens were weighed before and after oven drying. For water absorption, the specimens were oven dried, weighed, immersed in water for 24 hours, and weighed again. The post immersion surface water handling procedure was not documented.

For each test condition, three replicate specimens were cast from a single batch. These specimens represent within batch reproducibility and not batch to batch variability. Each reported value represents the mean of three specimens tested under identical conditions. Individual measurements were not available for standard deviation analysis; therefore, error bars are not included in the figures. Because individual specimen measurements were unavailable, the reported means could not be independently recalculated and statistical variability could not be quantified. The comparisons are therefore descriptive, and no claims of statistical significance are made.

Results:

Compressive Strength of Phase I Mortar Mixes:

The compressive behavior of the mortar composites was evaluated using compression tests. The compressive strength results at 7, 14, and 28 days are shown in Figure 10 and summarized in Table 11. Each value represents the average of three specimens prepared from the same mix and tested under identical conditions. Figure 10 shows the compressive strength results of all the composite mixtures.

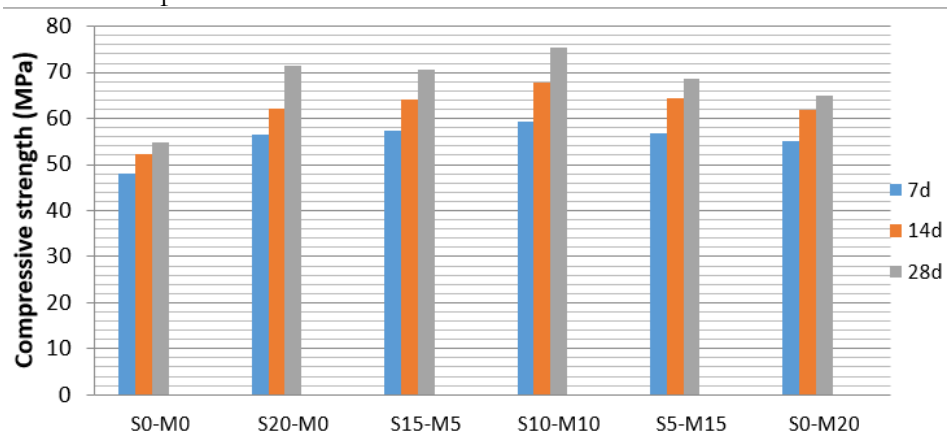

Figure 10. Compressive strength results in MPa for different SF and MK contents

Table 11. Phase I compressive strength results (mean of 3 specimens)

Mix ID	7 days (MPa)	14 days (MPa)	28 days (MPa)
S0-M0 (control)	48.1	52.3	54.7
S20-M0	56.5	62.2	71.4
S15-M5	57.4	64.1	70.6
S10-M10	59.2	67.8	75.3
S5-M15	56.9	64.5	68.7
S0-M20	55.2	61.8	64.9

Among all the tested composites, the mixture containing 10% silica fume and 10% metakaolin exhibited the highest observed compressive strength at all curing ages. At 7 days, the strength was 59.2 MPa. It increased to 67.8 MPa at 14 days and reached 75.3 MPa at 28 days. This mixture exhibited higher compressive strength than the mixture containing 20% silica fume. Compared with the control mixture, the compressive strength increased by 23.1%, 29.6%, and 37.7% at 7, 14, and 28 days, respectively.

Results of Phase II Experiments

The complete numerical results for all Phase II tests are summarized in Table 12.

Table 12. Phase II results – mean values for all tests (7 / 14 / 28 days)

Mix ID	Compressive strength (MPa)	MOR (MPa)	Oven-dry Density (kg/m³)	Moisture content, dry basis (%)	Water absorption (%)
S10-M10-F0	57.2 / 65.8 / 73.3	10.2 / 11.1 / 12.9	2258 / 2144 / 2092	3.5 / 4.1 / 4.4	1.3 / 1.27 / 0.96
S10-M10-F2	58.1 / 67.9 / 75.0	11.0 / 11.9 / 13.4	2138 / 2098 / 2072	3.9 / 4.2 / 4.7	2.4 / 1.6 / 1.4
S10-M10-F4	60.3 / 71.0 / 76.6	11.8 / 12.4 / 14.7	2103 / 2078 / 2067	4.1 / 4.6 / 5.0	2.8 / 2.0 / 1.7
S10-M10-F6	55.0 / 63.1 / 68.4	11.5 / 12.0 / 14.1	2071 / 2038 / 2017	5.1 / 5.6 / 6.0	3.1 / 2.2 / 2.0
S10-M10-F8	37.7 / 41.3 / 43.8	10.2 / 11.1 / 12.0	1988 / 1960 / 1938	6.2 / 6.4 / 6.6	4.0 / 3.1 / 2.7
S10-M10-F10	23.1 / 25.7 / 26.4	8.5 / 9.2 / 9.9	1856 / 1838 / 1820	6.8 / 7.2 / 7.5	4.7 / 3.6 / 3.4

Values are presented as 7 days / 14 days / 28 days; n = 3 for each condition.

The fiber-free mix in Phase II (S10-M10-F0) showed slightly lower compressive strengths at all curing ages (57.2, 65.8, and 73.3 MPa at 7, 14, and 28 days) compared to the Phase I S10-M10 mix (59.2, 67.8, and 75.3 MPa). This difference may reflect batch-specific or other experimental variability; however, its cause could not be established because independent batches were not tested.

Compressive Strength of Natural-Fiber-Reinforced Mortar:

The mixture exhibiting the highest observed compressive strength in Phase I was selected, and different amounts of pine needle fibers were subsequently added. Then these new composites were tested for compressive strength at 7, 14, and 28 days. Figure 11 shows the results for all these different mixes. The results presented here reflect the combined effects of fiber content and water-to-binder ratio, as both variables changed together in the tested formulations.

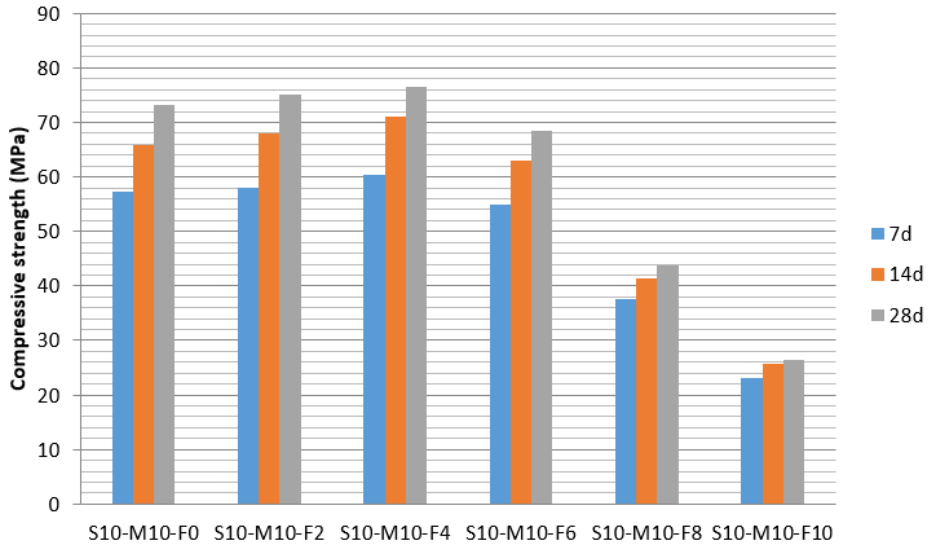

Figure 11. Compressive strength results in MPa for different pine needle fiber contents (wt%)

Among all the pine needle fiber-reinforced cement composites, the highest compressive strength was observed at 4% fiber content for all curing ages. At 7 days, it was 60.3 MPa. At 14 days, it was 71.0 MPa. At 28 days, the strength reached 76.6 MPa. The second-

highest compressive strength was observed in composites with 2% fiber content, with values of 58.1, 67.9, and 75.0 MPa at 7, 14, and 28 days, respectively. However, when the pine needle fiber content exceeded 4%, a notable decline in compressive strength was observed. The lowest compressive strength values were recorded for composites with 10% fiber content, registering 23.1 MPa, 25.7 MPa, and 26.4 MPa at 7, 14, and 28 days, respectively.

Modulus of Rupture (MOR):

The flexural strength of the composites was determined at 7, 14, and 28 days. Each reported value is the mean of three specimens prepared from the same batch and tested under the same conditions. The flexural strength results for all composite mixtures are presented in Figure 12.

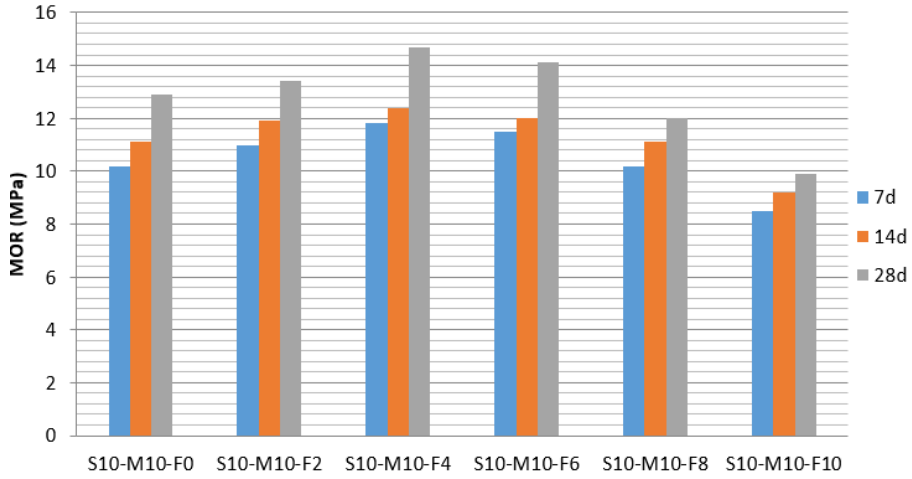

Figure 12. Modulus of rupture (MOR) of mortar mixes with different pine needle fiber contents

The modulus of rupture (MOR) values of pine needle fiber-reinforced cement composites ranged from 8.5 to 11.8 MPa at 7 days, 9.2 to 12.4 MPa at 14 days, and 9.9 to 14.7 MPa at 28 days of curing. The composite containing 4 wt.% pine needle fibers exhibited the highest MOR of 14.7 MPa after 28 days. All composites achieved their peak MOR values at 28 days. The MOR values increased with fiber content up to 4 wt.%, beyond which a substantial decline was observed.

Moisture Content:

The moisture content (dry basis) of all composites was measured after 7, 14, and 28 days of curing. Figure 13 shows the moisture content at each curing age.

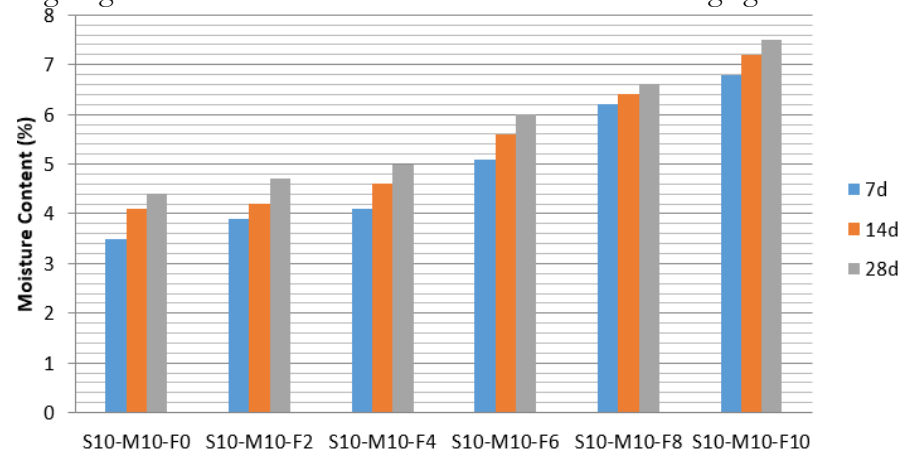

Figure 13. Moisture content of mortar mixes with different pine needle fiber contents

The highest moisture content (7.5%) was recorded for the mixture containing 10% pine needle fibers after 28 days of curing. The Phase II fiber-free reference mixture (S10-M10-

F0) exhibited the lowest moisture content (3.5%) after 7 days of curing. Moisture content increased slowly from 0% to 4% fibers, but then it jumped sharply above 4%.

Water Absorption:

The water absorption of the composites was measured at 7, 14, and 28 days. Figure 14 shows the water absorption results for all composite mixes. Water absorption rose as the natural fiber content increased. Additionally, the water absorption capacity decreased with curing age. The Phase II fiber-free reference mix (S10-M10-F0) exhibited the lowest water absorption compared to all fiber-reinforced composites.

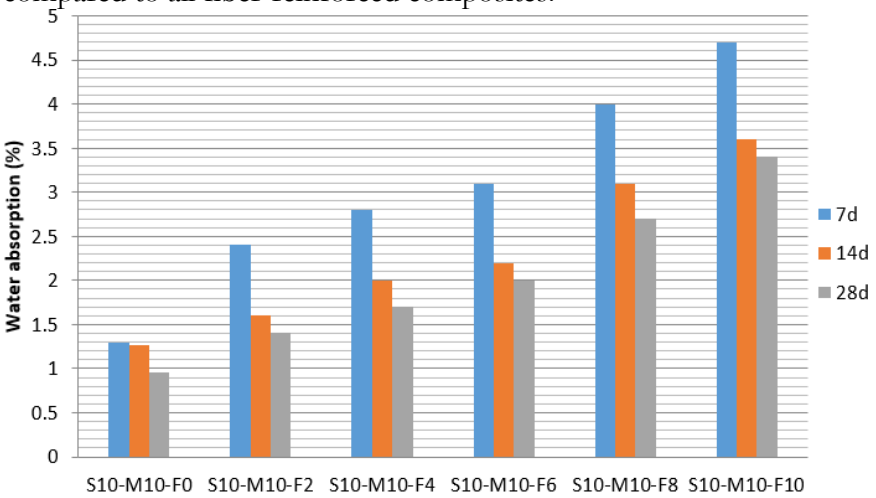

Figure 14. Water absorption of mortar mixes with different pine needle fiber contents

The mixture containing 10% pine needle fibers exhibited the highest water absorption (4.7%) after 7 days, whereas the fiber-free mixture exhibited the lowest value (0.96%) after 28 days. At each curing age, water absorption was higher for the reinforced mixtures than for the fiber-free mixture.

Density:

The density of all composites was determined at curing ages of 7, 14, and 28 days, and the results are summarized in Figure 15. A decrease in density was noted with increasing pine needle fiber content. Additionally, the density declined with increasing curing age. The Phase II fiber-free reference mix (S10-M10-F0) had the highest reported density, 2,258 kg/m³, after 7 days, while the minimum density of 1820 kg/m³ was recorded for the specimen having 10% fiber content at 28 days.

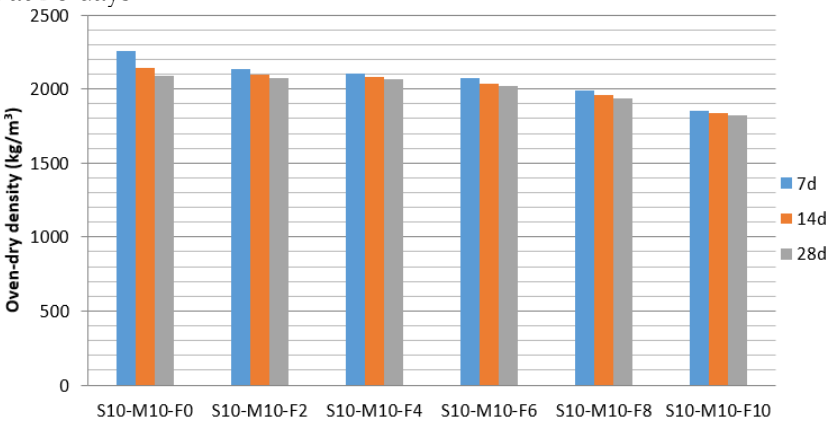

Figure 15. Density results in kg/m³ for different pine needle fiber contents

Discussion:

Effect of Silica Fume and Metakaolin on Compressive Strength:

The Phase I results showed that the mixture containing 10% silica fume and 10% metakaolin achieved the highest compressive strength among the six binder combinations that

were tested. The compressive strength reached 75.3 MPa after 28 days, compared to 54.7 MPa for the plain cement mortar. This shows an increase of approximately 37.7% relative to the control mixture. The mix with 10% silica fume and 10% metakaolin performed better than the mixes with 20% silica fume or 20% metakaolin alone at all curing ages. This matches the findings of previous research [22][24]. However, since only six mixtures were tested, the 10% SF–10% MK combination represents the best-performing mixture among those investigated and cannot be considered universally optimal.

Effect of Pine Needle Fiber Content on Mechanical Properties:

In Phase II, the mix with 4% fibers gave the highest average compressive strength and MOR among all the tested mixes. A moderate fiber content may help restrict crack propagation, whereas excessive fiber content may lead to fiber clumping and increased void formation in the mortar. Recent studies on natural fiber composites [25][26][27] have also mentioned these same issues with fiber dispersion and bonding. However, microstructural tests such as SEM, XRD, and porosity measurements were not performed, so we cannot prove these mechanisms. Also, since the water-to-binder ratio went up along with the fibers, the 4% mix should just be seen as the best-performing mix we tested, not a universally perfect fiber amount. The compressive strength trend differs from that reported by [29] for coconut fiber reinforced concrete, in which the increasing fiber content reduced compressive strength. However, the studies used different fiber types, matrix compositions, and specimen geometries. Therefore, the findings cannot be compared as if the experimental conditions were identical. The present results instead demonstrate that the mechanical response of the tested pine needle fiber mortar varied with the combined mixture conditions.

Moisture Content and Water Absorption:

The moisture content increased with fiber content in the tested formulations, reaching 7.5% at 28 days for the mixture containing 10% pine needle fibers. The Phase II fiber-free reference mix (S10-M10-F0) had the lowest reported moisture content, at 3.5% after 7 days. The water absorption followed a similar overall pattern: the 10% fiber mixture reached 4.7% at 7 days, whereas the fiber-free mixture had the lowest reported value of 0.96% at 28 days. Water absorption generally decreased with curing age in the tested mixtures. The higher moisture content observed in mixtures with greater fiber content coincided with lower compressive strength. However, because fiber content and water-to-binder ratio varied together, the independent contribution of moisture-related effects to the strength trend cannot be established from the present experimental design.

The higher moisture content and water absorption observed at greater fiber contents may be associated with changes in pore structure, water retention, or the fiber–cementitious matrix interface. The reduction in density may also accompany changes in the internal structure of the composite. Previous research on natural fiber reinforced cement composites has discussed the relationships between density and water absorption [3][30]. Nevertheless, the porosity and fiber matrix interfacial characteristics were not directly measured in the present study. These explanations should therefore be treated as possible mechanisms rather than confirmed causes.

Changes in Oven-Dry Density:

Oven-dry density decreased as the pine needle fiber content increased. The highest reported density was 2,258 kg/m³ for the fiber-free mixture at 7 days, whereas the lowest was 1,820 kg/m³ for the mixture containing 10% fibers at 28 days. The decrease in density with increasing fiber content may be related to the lower density of the incorporated natural fibers and possible differences in the air voids or packing within the mortar. Similar density trends have been reported in previous studies of natural fiber reinforced cementitious composites [31][32]. Density also decreased with the curing age in the reported results. However, the original individual specimen mass and dimension records were unavailable for independent

verification. The cause of this age-related trend therefore cannot be established from the available data and should not be attributed to a specific physical mechanism without further evidence.

Interpretation and Limitations:

Because the fiber content and water-to-binder ratio increased together, we cannot separate their individual effects on compressive strength, MOR, moisture content, water absorption, or density. Also, each reported value is the mean of three specimens from a single batch. We did not have the individual measurements, so we could not calculate standard deviations, add error bars, or run statistical significance tests. For future work, researchers should vary the fiber content while keeping the water-to-binder ratio fixed, test independent batches, keep individual specimen data, measure workability, and check long-term durability and microstructure.

Conclusion:

This study examined the physical and mechanical properties of cement mortar. In Phase I, we tested six binder formulations, including a plain control. In Phase II, we tested six pine needle fiber contents using the selected 10% SF and 10% MK binder. The study evaluated different binder combinations and pine needle fiber contents to address its research objectives. In the first phase, 54 specimens were cast and tested to identify the best-performing silica fume and metakaolin mix for enhancing the compressive strength of the cement matrix. In the second phase, a comparative analysis was carried out considering multiple parameters, including density, compressive strength, moisture content, water absorption, and MOR. Based on the test results, the following conclusions were drawn:

The mix with the highest observed compressive strength was 10% SF and 10% MK. This mix gave the highest compressive strength of 75.3 MPa. This mixture performed better than the other five mixtures tested. The fiber-free mix in Phase II showed a slightly lower 28-day strength of 73.3 MPa compared to the Phase I mix. This difference may reflect batch-specific or other experimental variability; however, its cause could not be established because independent batches were not tested.

Among all the Phase II mixes, the 4% fiber mix gave the highest average compressive strength and MOR, reaching 76.6 MPa and 14.7 MPa after 28 days. However, because fiber content and water-to-binder ratio varied together, this mixture represents the best-performing formulation among those tested rather than a universally optimal formulation.

The highest density was 2,258 kg/m³ for the Phase II fiber-free reference mix (S10-M10-F0) at 7 days, while the lowest was 1,820 kg/m³ for the 10% fiber mix (S10-M10-F10) at 28 days.

The Phase II fiber-free reference mix (S10-M10-F0) exhibited the lowest moisture content (dry basis) at 3.5% after 7 days. Moisture content increased gradually for fiber contents up to 4%, beyond which a sharp rise was observed, reaching the maximum value at fiber content 10%.

The Phase II fiber-free reference mix (S10-M10-F0) exhibited the lowest water absorption at 0.96% after 28 days. Water absorption increased with fiber content but generally decreased with curing age.

Practical Implications: The results show that the 4% pine needle fiber mix has good potential. However, it cannot be recommended for actual construction yet. More testing is needed on workability, dimensional stability, long-term durability, and performance under real service conditions.

Limitations:

The fiber content and water-to-binder ratio changed together, so we cannot separate their individual effects. The exact pine needle species and dimensions were not recorded. We also did not have raw specimen data for statistical analysis, and we did not run SEM, XRD, or

porosity tests. Some original testing details were also missing.

Future Research:

Future work should test different fiber contents at fixed water-to-binder ratios, use independent batches, and keep individual specimen data for statistics. Researchers should also study long-term durability (like water transport and shrinkage) and use microstructural tests to better understand the fiber matrix interface.

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