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The Effect of Fiber Surface Modification on the Microstructure and Strength of Sisal-Reinforced Concrete

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Abstract

This study examined how modifying sisal fibre surfaces affects both the workability and durability of sisal fibre-reinforced concrete (SFRC). Three concrete mixes were compared: untreated sisal fibres (USFRC), alkali-treated fibres (ATSFRC), and paraffin- alkali-treated fibres (PATSFRC). Workability and durability improved as fibre treatment increased. Based on sorptivity (water absorption behaviour) at 28 and 56 days, USFRC had the highest sorptivity, while PATSFRC had the lowest. PATSFRC achieved 0.08 mm/min^{1/2} at 28 days and 0.05 mm/min^{1/2} at 56 days—reductions of 55% and 64%, respectively, compared with USFRC. For chloride conductivity, USFRC performed worst, with the highest values of 920 C (28 days) and 600 C (56 days). PATSFRC performed best, showing the lowest values (reported down to 0.05 mm/min^{1/2} at 56 days for sorptivity, consistent with the best overall durability trend. Overall, paraffin treatment provided the greatest improvements, making PATSFRC a more effective option against moisture and chloride ingress. Across curing ages (3, 7, 14, 28, and 56 days), adding sisal fibres increased compressive, tensile, and flexural strengths, with ATSFRC showing the best strength results after 56 days (37 MPa compressive, 3.4 MPa tensile, 5.3 MPa flexural). Finally, microstructural analysis using SEM, FTIR, and SEM-EDX supported these performance results.

Keywords: *chloride permeability, concrete, durability, mortar, sorptivity, sisal fibre.*

1. INTRODUCTION

The construction industry is increasingly driven by sustainability principles, with a focus on renewable, low-energy, and environmentally friendly materials [1, 2, 3, 4]. Over the years, conventional steel or synthetic reinforcements, while effective, have involved high embodied energy and carbon emissions. Therefore, there is a need for more sustainable and cost-effective reinforcement materials in the construction sector. In this context, natural vegetable fibres have

emerged as a promising alternative for reinforcing cementitious composites due to their low density, renewability, biodegradability, and cost-effectiveness, while also providing crack-bridging and post-crack load-carrying capacity in cement matrices [5, 6, 7, 8, 9, 10, 11]. However, the inherent hydrophilic nature of their cellulosic components and microstructural heterogeneity make these fibres prone to moisture swelling, dimensional instability, and alkaline degradation in cementitious environments [11, 12]. These limitations compromise interfacial bonding and reduce the long-term durability of the composite, necessitating appropriate fibre modifications prior to application [13,14].

Among natural fibres, sisal is notable for its advantageous tensile properties, abundance, and renewability. Its distinctive fibrillary structure, combined with surface pectin and lignin layers and varying crystallinity, significantly influences its bonding behaviour within the cementitious interfacial transition zone (ITZ). Multi-scale studies, from single-fibre pull-out tests to composite-level flexural evaluations, demonstrate that fibre strength variability, embedded length, and ITZ characteristics impact post-crack performance. Consequently, many studies have focused on modifying sisal fibre surfaces to reduce moisture absorption, improve chemical compatibility and surface roughness, and decrease mineralization within the ITZ.[6, 12, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25,26, 27].

A wide range of treatment techniques has been investigated to enhance fibre-matrix compatibility, reduce water uptake, and delay alkaline deterioration [21, 28, 29, 30, 31, 32, 33, 34]. Common approaches include alkali (NaOH) treatment, silane coupling, esterification/acetylation, paraffinic coatings, hydrophobic paraffin or oil immersion, and pozzolanic or matrix chemical modification, among others [35,42,43,44,45,46,47]. These treatments generally follow two strategies: surface modification to decrease hydrophilicity and improve interfacial bonding, and matrix modification using supplementary cementitious materials (SCMs) or alkaline waste solutions to lower matrix alkalinity and slow fibre mineralization [13,14,41]. Each treatment yields distinct microstructural signatures, including alterations in surface roughness, crystallinity, ITZ morphology, and hydration product distribution, that directly influence the tensile, flexural, and compressive responses of the composites [12, 21, 42, 43, 44].

Alkali treatment with NaOH remains one of the most common and effective methods of modification [45, 46, 47, 48, 49, 50, 51, 52, 53]. The treatment removes amorphous components such as pectin and hemicellulose, revealing crystalline cellulose microfibrils and increasing surface roughness [54]. Optimal NaOH concentrations (typically 1–10 wt.%) and exposure durations (1–48 hr) have been reported to enhance fibre-matrix adhesion and flexural behaviour [55,11,36]. However, excessively strong or prolonged alkali exposure can lead to embrittlement and loss of mechanical performance [56, 57, 58, 59, 60, 61, 62]. Microstructural investigations confirm that NaOH treatment increases fibre crystallinity, modifies the morphology of calcium deposits in the ITZ, and alters hydration behaviour. Nevertheless, it also raises environmental and waste disposal concerns, highlighting the need for optimized, controlled processes [63, 64, 65].

In contrast, paraffin or hydrophobic oil treatments work by forming a physical barrier which limits moisture ingress and suppresses alkaline mineralization of fibre walls [65, 66, 67, 68]. Scanning Electron Microscopy (SEM) and X-ray Diffractogram (XRD) analyses show thinner calcium layers and less aggressive mineral deposition around treated fibres [71,72]. Mechanically, paraffin-treated sisal fibres demonstrate an improved moisture resistance and thermal insulation while maintaining satisfactory elastic modulus values [11]. Despite these advantages, long-term adhesion and durability under cyclic mechanical loading remain insufficiently explored, underscoring the need for systematic investigation [69,72].

Existing studies show notable improvements in compressive, tensile, and flexural strength when sisal fibres are properly treated [7, 22, 60, 61, 62, 63, 64, 65, 66, 73, 74, 75, 76]. For example, treated sisal fibre composites have achieved a 46% increase in compressive strength over 7 days and a 27% increase in split tensile strength at 28 days [51, 54, 77]. Using pozzolanic additives such as metakaolin and fly ash in the matrix has also been effective in reducing calcium hydroxide content by up to 64%, resulting in denser ITZs and significant gains in bond strength and pull-out energy [75] [38][39][40]. However, variability across studies due to differences in treatment methods, fibre dosages, and testing scales limits the comparability and reproducibility of the results [60, 63,76, 77, 78, 79].

A research gap exists due to limited mortar-level comparative data that include both NaOH and paraffin treatments under standardized testing conditions. Existing studies lack comprehensive

datasets linking treatment parameters with microstructural features (SEM, XRD, porosity analysis) and mechanical properties (compressive, flexural) over both short- and long-term curing periods (≥ 56 days). Therefore, this research addresses this gap by systematically examining the mechanical and microstructural performance of cement mortar reinforced with sisal fibres treated with various methods, focusing specifically on NaOH- alkali-treated sisal fibre reinforced mortar (ATSFRM) and paraffin-modified alkali-treated sisal fibre reinforced concrete (PATSFRC). The study aims to establish correlations between fibre treatment, interfacial mechanics, and composite behaviour to identify optimal, sustainable, and durable configurations for sisal-reinforced mortar systems.

2. Materials and Methodology

2.1. Materials

The materials used in this study were selected to conform to relevant South African standards, ensuring consistency and local applicability. They include:

2.1.1. Cement: 42.5 Ordinary Portland cement (OPC) was used as the primary concrete binder and was supplied by Afrisam Roodepoort concrete product supplier. This cement conforms to the requirements of **SANS 50197-1** Cement - Part 1 Composition, specifications and conformity criteria for common cements, which is harmonized with the European **EN 197-1** standard.

2.1.2. Quartz Sand: Supplied by Express building material was used as the fine aggregate. The sand was separated into two distinct particle-size fractions (0.11–0.21 mm and 0.21–0.38 mm) to allow precise grading control in the HPC mix, in accordance with the **SANS 1083** standard on Aggregates from natural sources: Aggregates for concrete.

2.1.3. Sisal Fibre: Characterized by a unique set of physical and mechanical properties, including high tensile strength (400-700 MPa) and a moderate modulus of elasticity (9-20 GPa), making it a suitable material for reinforcing brittle cement matrices. However, its performance is mainly influenced by its chemical composition, predominantly cellulose, hemicellulose, and lignin, as summarized in **Table 3**. This composition offers various advantages, while cellulose provides strength, the amorphous hemicellulose and lignin are highly susceptible to alkaline degradation in cement paste, and the fibre's hydrophilic nature leads to poor matrix adhesion. Therefore, a

comprehensive understanding of these properties is essential for developing effective chemical treatments to mitigate these drawbacks and to utilize sisal fibre's full potential in producing durable concrete composites.

2.1.4. Sodium Hydroxide: The chemical agent specified for the alkali treatment process is Sodium Hydroxide, commonly known in industrial and laboratory contexts as Caustic Soda. Its definitive chemical formula is NaOH, and it is uniquely identified by the Chemical Abstracts Service (CAS) **Registry Number 1310-73-2**. This high-purity material, supplied as white, deliquescent pellets or flakes, was verified through visual inspection and used as a primary reactant for the surface modification of sisal fibres. The description is presented in **Table 1**. It has a minimum purity (assay) of $\geq 97.0\%$, tested in accordance with **SANS 50896**, with carbonate impurities (as Na_2CO_3) limited to $\leq 2.0\%$, as specified by **SANS 50897**.

Table 1: Specification for sodium hydroxide

Property	Specification	Test Standard
Form	White, deliquescent pellets or flakes	Visual inspection
Assay (Purity)	$\geq 97.0\%$	SANS 50896
Carbonate (as Na_2CO_3)	$\leq 2.0\%$	SANS 50897
Chloride (as Cl)	$\leq 0.1\%$	Method referenced in SANS 50896
Heavy Metals (as Pb)	≤ 10 ppm	Standard heavy metals test

Chloride content (as Cl) does not exceed 0.1%, as determined by the method referenced in **SANS 50896**. Heavy metals (as Pb) are limited to a maximum of ≤ 10 ppm, measured using the standard heavy metals test. Additionally, the properties of the Paraffin used in this study are listed in **Table**

2. It is a white or off-white, odourless solid, assessed through visual and olfactory inspection, with a melting point ranging from 58°C to 62°C, as determined in accordance with **SANS 342**.

Table 2: Specification for Paraffin

Property	Specification	Test Standard
Form	White or off-white, odourless solid	Visual/olfactory inspection
Melting Point	58°C - 62°C	SANS 342
Oil Content	≤ 0.5% (by mass)	SANS 342
Penetration at 25°C	≤ 20 (0.1 mm)	SANS 342
Flash Point	≥ 200°C	SANS 342

The material contains an oil content of $\leq 0.5\%$ by mass, and its penetration at 25°C is ≤ 20 (0.1 mm), both measured in accordance with **SANS 342**. Additionally, it has a flash point of $\geq 200^\circ\text{C}$, in compliance with **SANS 342** requirements. The specification of sisal fibre, shown in **Table 3**, describes a lightweight natural material with a density of 1.30–1.45 g/cm³ and a diameter of 100–300 μm . It exhibits strong mechanical performance, with a tensile strength ranging from 400–700 MPa and a modulus of elasticity of 9–20 GPa.

Table 3: Physical and mechanical properties of sisal fibre

Property	Typical Value Range
Tensile Strength	400 - 700 MPa
Modulus of Elasticity	9 - 20 GPa
Elongation at Break	2.0 - 4.0 %
Density	1.30 - 1.45 g/cm ³
Diameter	100 - 300 μm

The fibre shows 2–4% elongation at break, indicating moderate flexibility and reliable load-carrying capability, making it suitable for composite reinforcement applications. Sisal fibre is primarily composed of cellulose, ranging from 67% to 78%, which contributes to its strength and structural stability. It also contains 10% to 14% hemicellulose, influencing its moisture absorption and flexibility, and 8% to 11% lignin, which provides rigidity and resistance to microbial degradation [3], [69], [70]. The chemical composition of sisal fibre is shown in **Table 4**. The remaining approximately 4% consists of minor components such as pectin, ash, and waxes, which affect surface characteristics and bonding behaviour in composite applications.

Table 4: Chemical composition of sisal fibre

Component	Composition (% by mass)
Cellulose	67% - 78%
Hemicellulose	10% - 14%
Lignin	8% - 11%
Other (Pectin, Ash, Waxes)	~ 4%

2.2. Sample Preparation

The sisal fibres utilized in this study were sourced from mature leaves of *Agave sisalana* plants, collected from a plantation in Florida, Johannesburg, South Africa, as presented in **Fig. 1**. The raw fibres were then carefully washed with clean water to remove residual plant pulp, chlorophyll, and soluble impurities. Afterwards, the fibres were evenly spread and air-dried in a shaded, well-ventilated area for 48 hours to prevent photodegradation. As it is shown in **Figure 4** The final preparation step involved combing the dried fibres to separate them into individual filaments and remove any short or weak fibres, after which they were cut to a standardized length of 30 mm using a precision cutter. This specific length was chosen because it is optimal for bridging microcracks in concrete without inducing significant balling during mixing [71, 72].



Fig. 1: Sisal fibre collection process from Florida, Johannesburg, South Africa

To enhance the fibre-matrix interface, two distinct chemical treatment methods were employed. as it is shown in **Figure 2** the first involved an alkali treatment with Sodium Hydroxide (NaOH), a process designed to remove natural waxes, oils, and amorphous components such as hemicellulose and lignin. This treatment aims to create a rougher surface topography to improve mechanical interlocking, expose more cellulose hydroxyl groups, and enhance interfacial bonding. Solutions were prepared at 1% based on the findings of previous studies [73, 57, 59]. By dissolving

laboratory-grade NaOH pellets in distilled water. A critical safety procedure was followed during pellet preparation. Pellets were always added slowly to water with constant stirring, and the solution was allowed to cool to room temperature ($27 \pm 2^\circ\text{C}$) before use due to the highly exothermic nature of dissolution. The treatment process involved fully immersing a predetermined weight of dried sisal fibres in each NaOH solution at room temperature for 2 hours. Subsequently, the fibres were removed, rinsed under running tap water, neutralized with a mild acetic acid solution (1% v/v), and given a final rinse with distilled water until the runoff achieved a neutral pH of ~ 7 . The treated fibres were then air-dried for 24 hours, followed by oven-drying at 60°C for 6 hours to achieve a consistent moisture content.



Fig. 2: Sample Preparation – Soaking of Sisal fibre in distilled water and sodium hydroxide

The second method involved coating the fibres with paraffin wax. This process aims to create a physical hydrophobic barrier on the fibre surface, thereby significantly reducing water absorption and minimizing the damaging cycles of swelling and shrinkage within the concrete matrix [74, 75]. This results in a more stable and durable fibre-matrix interface. The procedure started by pre-drying the sisal fibres in an oven at 60°C for 2 hours to remove moisture and optimize wax adhesion. Commercial-grade paraffin wax was melted in a double boiler to $70\text{--}80^\circ\text{C}$. The pre-dried fibres were then fully immersed in molten wax for 5-10 minutes, with gentle agitation, to ensure a uniform coating.

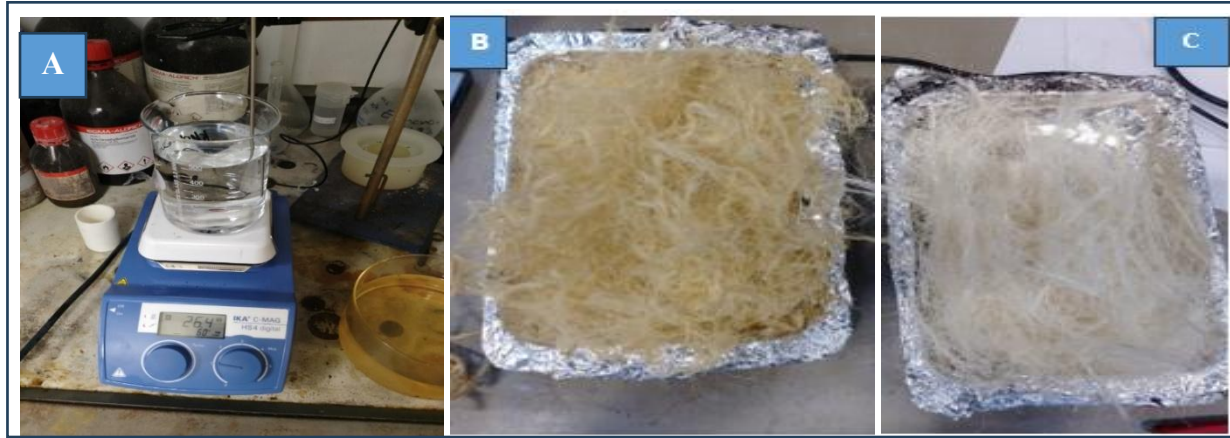


Fig. 3: (A) Distilled water, (B) Treated sisal fibre with sodium hydroxide and paraffin, (C) Untreated sisal fibre

After immersion, the fibres were carefully removed, allowed to drain, and spread on a wire mesh to cool and solidify at room temperature. as it is shown in **Figure 3** the coated fibres were left to cure for 24 hours before use. Unlike the chemical modification achieved with NaOH, alkali and paraffin treatment is a physical process that does not enhance chemical bonding but is highly effective at mitigating moisture-induced degradation, a primary failure mechanism for natural Fibres in cementitious composites. The total cycle of treatment and production of mortar is illustrated in **Figure 5**.



Fig. 4: Samples of treated and untreated sisal fibers

2.3. Mix design

This mix design is developed for a C30/37 strength class concrete (characteristic cube strength of 30 MPa) in accordance with the **SANS 10400** standards.

Table 5: Mix design for sisal fiber-reinforced fibrous concrete

Material	Proportion	Quantity	Unit	Remarks
Cement	1	450	kg	Ordinary Portland Cement
Fine Sand	3	1350	kg	Clean, dry, well-graded
Water	w/c = 0.50	225	kg (L)	Adjust for workability
Sisal Fibre	1% of cement	4.5	kg	Cut length 20–30 mm

2.4. Test Methods

2.4.1. Fibre Characterization

2.4.1.1. Scanning Electron Microscopy (SEM) analysis of sisal fibre and mortar

The fibre-matrix interface and microstructural features were examined using scanning electron microscopy of fractured surfaces from flexural test specimens. This analysis provided visual evidence of interfacial bonding quality, fibre pull-out mechanisms, and any signs of fibre degradation or matrix micro-cracking.

2.4.1.2. X-Ray Diffraction (XRD) analysis of sisal fibre and mortar

Crystalline phase composition of the mortar matrix was characterized using X-ray diffraction. This technique identified and semi-quantified hydration products, particularly Portlandite content, to understand the chemical environment surrounding the fibres and potential reaction products at the interface.

2.4.1.3. Fourier-Transform Infrared (FTIR) spectroscopy of sisal fibre

Chemical modifications caused by fibre treatments were confirmed using FTIR spectroscopy. This analysis identified key functional groups and chemical bonds, verifying the success of the surface treatments by detecting the removal or addition of chemical moieties.

2.4.2. Tests for mortar properties

2.4.2.1. Flow table test

The workability of the fresh mortar mixtures was evaluated using the flow table test in accordance with SANS 5865-1. This test measured the spread diameter of the mortar after a specified number of jolts, providing quantitative data on how incorporating treated and untreated sisal fibres affected the mixture's consistency and flow properties.

2.4.2.2. Initial and final setting time of mortar

The initial setting time is the period from when water is added until the mortar begins to lose its plasticity. The final setting time marks when the mortar has completely lost its plasticity and becomes sufficiently rigid. These parameters are crucial for managing handling time, finishing tasks, and removing formwork. The presence of sisal fibres may affect the hydration process by absorbing some of the mixing water and influencing the cement-water interaction, potentially altering both the initial and final setting times.

2.4.2.3. Consistency of mortar

The consistency of mortar refers to its flowability or ease of deformation under applied force. It shows how much water is needed to make a workable mix. Proper consistency ensures uniform mixing, sufficient compaction, and better bonding between cement paste and aggregates. The addition of sisal fibres, which have high water absorption capacity and a rough surface texture, can change the rheological behaviour of mortar. Fibres tend to decrease flowability due to increased internal friction and water demand, making consistency testing vital for optimizing mix proportions.

2.4.3. Mechanical Properties Tests

The process flow for material preparation, mortar casting, and testing of mechanical properties is illustrated in Fig. 5.

2.4.3.1. Compressive strength test

The compressive strength of the hardened mortar was measured after curing for 7, 28, and 56 days using 40×40×40 mm cube specimens, as specified in **SANS 5865-3**. This test provided essential data on the composite's capacity to resist axial loads, with any significant reduction in strength indicating potential issues with fibre dispersion or interfacial bonding.

2.4.3.2. Flexural strength test

The flexural performance was evaluated using three-point bending tests on 40×40×160 mm prism specimens in accordance with **SANS 5865-4**. Conducted under displacement control, this test captured the complete load-deflection response, enabling calculation of both the modulus of rupture and the toughness index, a critical measure of the composite's energy absorption capacity and post-cracking ductility.

2.4.3.3. Tensile strength test

Tensile strength, defined as the maximum stress a material can withstand when stretched or pulled apart, is one of the most critical yet often lacking properties in conventional concrete. It is tested in accordance with **SANS 10100**. While concrete can endure substantial compressive forces, its capacity to resist tensile stress is notably low, typically ranging from only 8% to 15% of its compressive strength. This inherent weakness results from its heterogeneous composition, which includes a brittle cement matrix, aggregates of varying strengths, and a porous interfacial transition zone that acts as a primary site for crack initiation.

2.5. Durability Performance Assessment

2.5.1. Accelerated ageing via wet-dry cycling of mortar

Long-term durability was evaluated using an accelerated ageing process involving repeated wet-dry cycles. Specimens underwent alternating immersion and drying phases to simulate harsh environmental conditions, and retained mechanical properties were assessed to determine degradation resistance.

2.5.2. Sorptivity resistance for mortar

Sorptivity resistance measures how quickly water is absorbed into mortar through capillary action and is a key indicator of its durability. In sisal fibre-reinforced mortar, adding natural fibres can affect the pore structure, water absorption, and microcrack development due to their hydrophilic properties and fibre-matrix interaction. Assessing sorptivity helps determine the mortar's resistance to moisture ingress and related deterioration, ensuring that the inclusion of sisal fibres does not harm long-term durability [74, 75].

2.5.3. Chloride permeability test

The chloride permeability test assesses the ability of mortars to resist chloride ion penetration, which greatly contributes to reinforcement corrosion and structural deterioration. In sisal fibre-reinforced mortar, the addition of fibres may alter the internal pore structure and crack propagation behaviour, thereby affecting ionic transport properties. This test is crucial for determining whether the inclusion of sisal fibres improves or compromises the mortar's resistance to chloride ingress and overall durability [76, 77, 78].



Fig. 5: (A)Processed sisal fiber, (B) Casting of mortar, (C) Curing of mortar, (D) Mortar samples in oven (E) Samples left out to cool and dry (F) Result of mortar testing at seven

3. RESULTS AND DISCUSSION

3.1. Initial and final setting time

The penetration time curves for the three concrete mixtures exhibit distinct behaviours by sisal fibre treatment type, revealing clear differences in the initial and final setting times. In the untreated sisal fibre concrete, the penetration depth remains nearly constant at around 38–40 mm for an extended period, indicating that the mix remains in its plastic state for several hours. The initial setting occurs at around 5.5–6 hours, when penetration begins to exceed the 35 mm limit. The curve then gradually increases, with noticeable fluctuations, demonstrating unstable stiffening due to the fibres' natural water absorption and swelling. Final setting is reached around 7.5–8 hours, marking the point where the penetration stabilizes near the 5–6 mm range. The setting times of untreated, alkali-treated, and alkali-plus-paraffin-treated sisal fibres are presented in **Fig. 6, 7, and 8**, respectively.

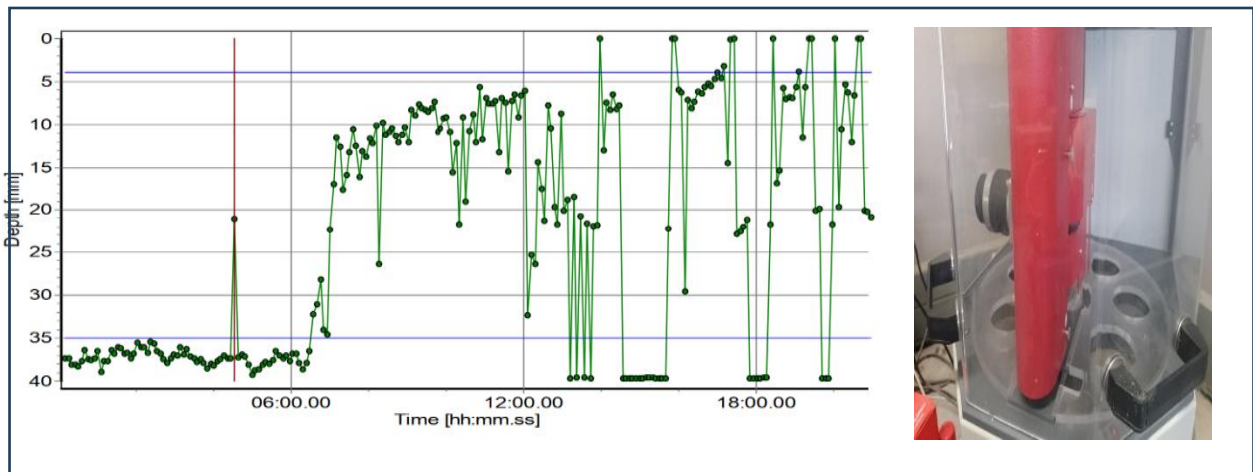


Fig. 6: Final setting time for untreated sisal fibre reinforced concrete

The delayed and irregular setting pattern reflects the hydrophilic nature of untreated sisal fibres, which draw water from the mix, slow early hydration, and cause fibre swelling that disrupts uniform stiffening. In the NaOH-treated sisal fibre concrete, the behaviour differs significantly. Here, the paste remains plastic until approximately 6 hours, similar to the untreated mix, but once hydration accelerates, the penetration depth increases sharply.

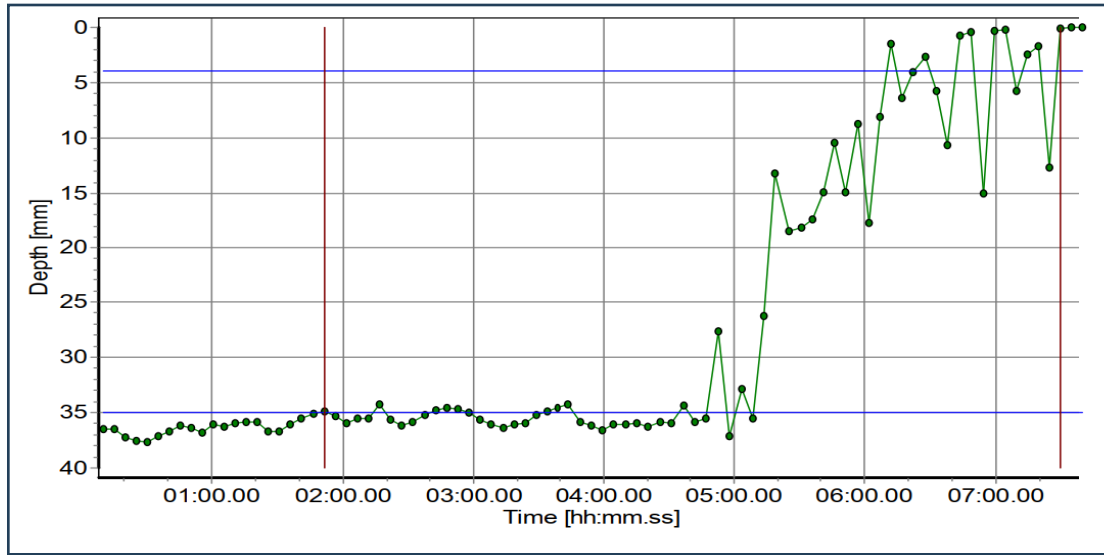


Fig. 7: Setting time of alkali-treated sisal fibre concrete

This rapid transition is characteristic of alkaline-treated fibres, which develop a rougher surface and stronger fibre–cement adhesion. However, the overall setting process takes longer to stabilize, with final setting occurring only around 10–11 hours. The extended final setting time is because of alkaline treatment, which removes surface impurities and increases fibre hydrophilicity. Although this improves mechanical interaction with the cement matrix, it also raises water absorption by the fibres and slows hydration kinetics. Consequently, the NaOH-treated mix shows the longest setting time among all treatments, despite offering the greatest potential to enhance tensile behaviour and crack-bridging capacity.

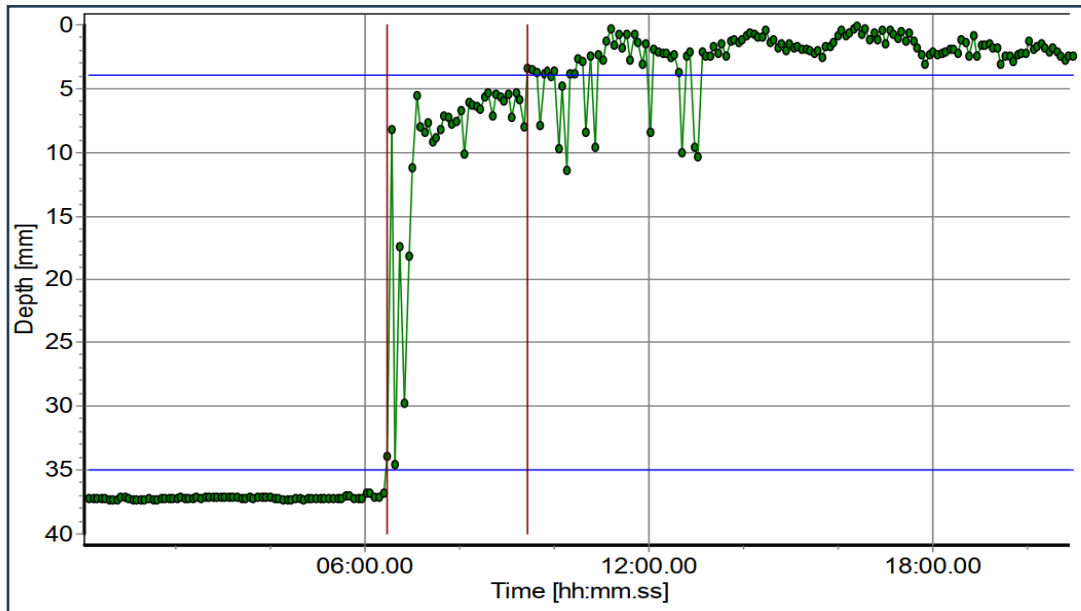


Fig. 8: Paraffin and alkali-treated sisal fibre reinforced concrete

The paraffin-treated sisal fibre concrete shows the quickest and most stable setting profile because paraffin forms a hydrophobic coating around the fibres, preventing water absorption during mixing [79, 80, 81]. This makes freer water available for cement hydration, helping the mixture to stiffen faster [80, 81]. Initial setting occurs earlier, around 5–5.5 hours, slightly sooner than the non-treated and NaOH-treated mixes. The final setting is also achieved more quickly, at around 7–7.2 hours, and the penetration curve shows fewer fluctuations, indicating a stable, continuous hydration process. Unlike untreated fibres, paraffin-coated fibres do not swell, preventing micro-instabilities within the matrix during setting [80]. Although this treatment reduces fibre matrix bonding strength compared to NaOH treatment, it greatly improves workability and early hydration and reduces the risk of early-age cracking.

Comparing the two treatments, alkali- and paraffin-treated fibres provide the quickest and most uniform setting, making them ideal for applications requiring rapid strength development or early finishing operations. Untreated fibres cause a moderate delay in setting due to their natural hydrophilicity, while NaOH-treated fibres cause the greatest delay, despite offering superior mechanical bonding. From a performance perspective, NaOH treatment is expected to produce the strongest fibre- concrete interface and the greatest mechanical improvements. Meanwhile, alkali

and paraffin treatments enhance workability, stability, and early-age hydration. Untreated fibres fall between these two extremes, offering moderate performance while suffering from water absorption and swelling.

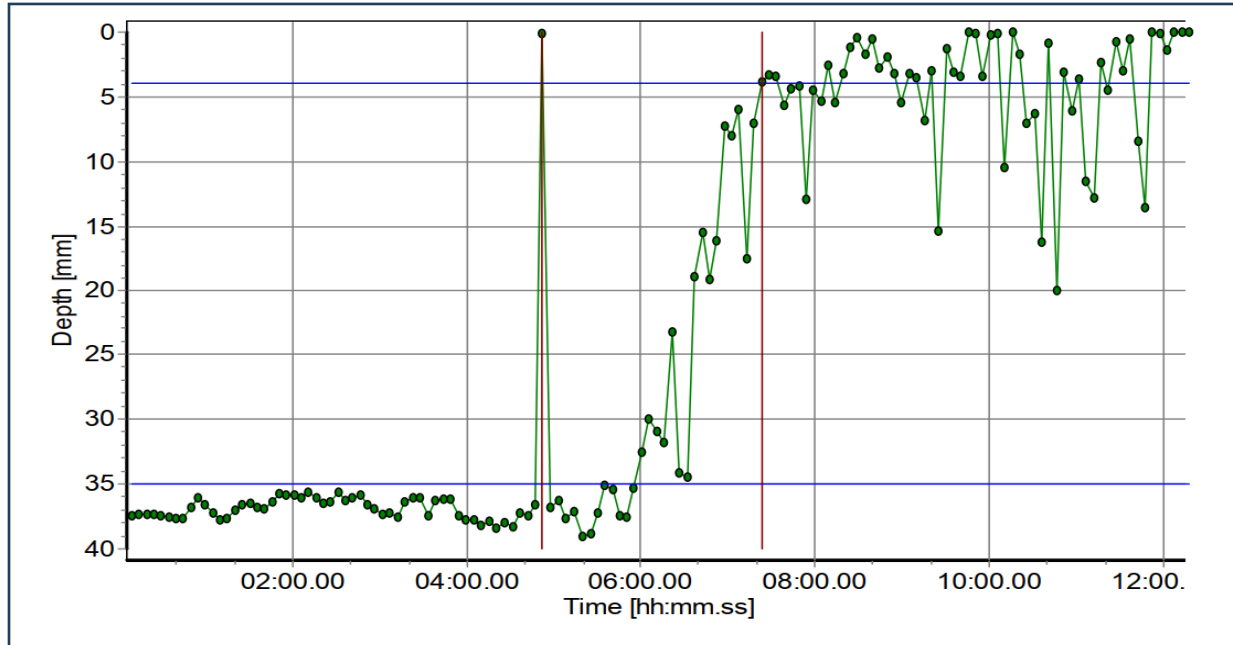


Fig. 9: Setting time of optimum treated sisal fibre

Fig. 9 shows the optimum setting time for concrete with treated sisal fibre, and overall, the results demonstrate that fibre treatment has a direct and significant influence on hydration behaviours and setting characteristics. NaOH treatment enhances mechanical properties but extends the setting time. Alkali and Alkali with paraffin treatments improve early-age behaviours and rheology, although they slightly compromise fibre cement bonding. Untreated fibres exhibit intermediate performance, with inherent limitations stemming from their natural hydrophilicity. These findings underline the importance of fibre modification in designing sisal fibre reinforced concrete with

tailored setting behaviours and structural performance. The above results are consistent with the findings of [24, 79, 80, 81].

A summary of the initial and final setting times for USFRC, ATSFRC, and PATSFRC is shown in **Table 6**, where a notable increase in both initial and final setting times is observed from USFRC to ATSFRC and further to PATSFRC.

Table 6: Summary of initial and final setting time

Parameter	PATSFRC	ATSFRC	USFRC
Initial Setting Time	165 min	120 min	75 min
Final Setting Time	255 min	190 min	135 min

The results show that hydration accelerates as the mix transitions from PATSFRC to ATSFRC to USFRC: the initial setting time decreases from 165 minutes (PATSFRC) to 120 minutes (ATSFRC) and then to 75 minutes (USFRC). This indicates that untreated sisal fibre concrete stiffens fastest because water can interact with cement more quickly, while the paraffin coating in PATSFRC delays hydration by acting as a barrier to water absorption. Similarly, the final setting time follows the same pattern: it reduces from 255 minutes (PATSFRC) to 190 minutes (ATSFRC) and finally to 135 minutes (USFRC). This suggests that the paraffin coating slows moisture exchange and delays full stiffening, thereby extending the workable period. ATSFRC performs in between, implying alkali treatment improves fibre–matrix interaction but does not greatly hinder hydration. Overall, surface treatment alters hydration kinetics—paraffin enhances workability and prolongs setting, while untreated fibres accelerate both initial and final setting.

3.2. Consistency Test

The consistency of mortar is a critical property that determines its workability, ease of placement, and overall performance in construction [79, 80]. Mortar, which is a mixture of cement, lime, sand, and water, serves as a bonding material for masonry units such as bricks and blocks. The term “consistency” refers to the relative fluidity or stiffness of the mortar, which is influenced primarily

by the water-cement ratio, the type and grading of sand, and the presence of admixtures.



Fig. 10: Consistency of optimum treated sisal fibre

Achieving the correct consistency is crucial because it directly impacts the mortar's ability to fill joints, adhere to surfaces, and maintain structural integrity after setting. As it is shown in **Figure 10** mortar that is too stiff may not spread easily, leading to poor adhesion and voids, whereas overly fluid mortar can cause sagging, excessive shrinkage, or segregation of the mix components. Consequently, measuring and controlling mortar consistency is a vital step in quality assurance during construction.

Table 7: Visual workability and fibre dispersion notes

Sample Description	Initial Spread Diameter (mm)	Final Spread Diameter After 15 Drops (mm)	Flow Increase (%)	Visual Workability & Fibre Dispersion Notes
Control (Plain M25 Mortar)	140 ± 2	165 ± 3	17.9%	Excellent flow, smooth surface, homogeneous.
Mortar + Untreated fibre	110 ± 5	125 ± 6	13.6%	Poor flow, harsh mix, visible fibre clumps/balling, rough surface texture.

Mortar + NaOH-Treated fibre	135 ± 3	155 ± 4	14.8%	Good flow, uniform mix, fibres well-dispersed, cohesive with slight fibre visibility.
Mortar + Paraffin- Treated fibre	128 ± 4	145 ± 5	13.3%	Fair flow, slightly stiff, minor clustering of fibres, glossy surface from wax.

As shown in **Table 7**, fibre treatment significantly affects the workability and dispersion properties of sisal-fibre-reinforced mortar. The control mix (plain M25 mortar) is expected to perform the best, with an initial spread of 140 mm and a final spread of 165 mm, indicating a 17.9% increase in flow. Its excellent flow, smooth finish, and homogeneity set the standard for workability. Since no fibres are present, there is minimal internal friction, allowing the fresh mortar to move freely. Conversely, adding untreated sisal fibres leads to a noticeable decrease in workability, with lower initial (110 mm) and final (125 mm) spread diameters and a smaller flow increase of 13.6%. The rough surface texture and visible fibre clumping suggest poor compatibility between untreated fibres and the cementitious matrix. The natural surface impurities, waxes, and hemicellulose likely increase water absorption by the fibres, reducing the amount of free water and raising internal friction. This results in dispersion problems and fibre balling, which impair the overall uniformity of the fresh mortar [33, 81, 82].

The NaOH-treated sisal fibre mix displays improved behaviour compared to untreated fibres, with an initial spread of 135 mm and a final spread of 155 mm, indicating a 14.8% increase in flow. Alkaline treatment removes surface impurities, waxy layers, and weak amorphous constituents, exposing more cellulose and increasing surface roughness in a beneficial manner. This enhances compatibility between the fibres and the cement paste, leading to better dispersion and less clumping. As a result, the mortar shows good flowability, a more cohesive texture, and a generally uniform appearance with only slight fibre visibility [22, 33, 83, 84, 85, 86].

3.2.1. Slump test

Workability of each mix design was assessed in accordance with **SANS 5862-1:2006**. The inclusion of untreated sisal fibres resulted in a slump value of 65 mm, reflecting reduced

workability due to the fibres' hydrophilic nature, which absorbs mixing water and increases internal friction. Alkali-treated sisal fibres improved workability to 78 mm by removing non-cellulosic impurities, resulting in stiffer, less absorbent fibres. Lastly, paraffin-treated sisal fibres achieved the highest workability, with a slump of 85 mm, attributed to the paraffin coating, which seals the fibre surface, reduces water absorption, and improves fibre dispersion within the matrix.

3.3. X-ray Diffraction Analysis

X-ray Diffraction (XRD) is a powerful, non-destructive analytical technique used mainly to identify the crystalline phases in a material. In mortar studies, XRD is essential for determining the mineralogical composition of both raw binders (such as cement or lime) and hydrated reaction products. This analysis allows for the identification of key compounds like Portlandite (Ca(OH)_2), Calcite (CaCO_3), various silicate hydrates (C-S-H), and ettringite. By providing a "fingerprint" of the crystalline components, XRD aids in understanding the degree of hydration, potential carbonation, and the presence of unreacted precursors, all of which are crucial for assessing the quality and long-term performance of mortar. This study examines the X-ray diffraction (XRD) patterns of untreated, sodium hydroxide (NaOH)-treated, and paraffin-coated sisal fibres to elucidate the structural effects of each modification process. The control sample displays the characteristic XRD profile of native cellulose I, with prominent diffraction peaks at $2\theta \approx 15.5^\circ$, 22.5° , and 34.5° , corresponding to the (110), (200), and (004) crystallographic planes of cellulose I, respectively. A significant amorphous background, with an intensity of approximately 30,000 counts at $2\theta \approx 18^\circ$, is attributed to non-crystalline components such as lignin, hemicellulose, and pectin [54, 87, 88].

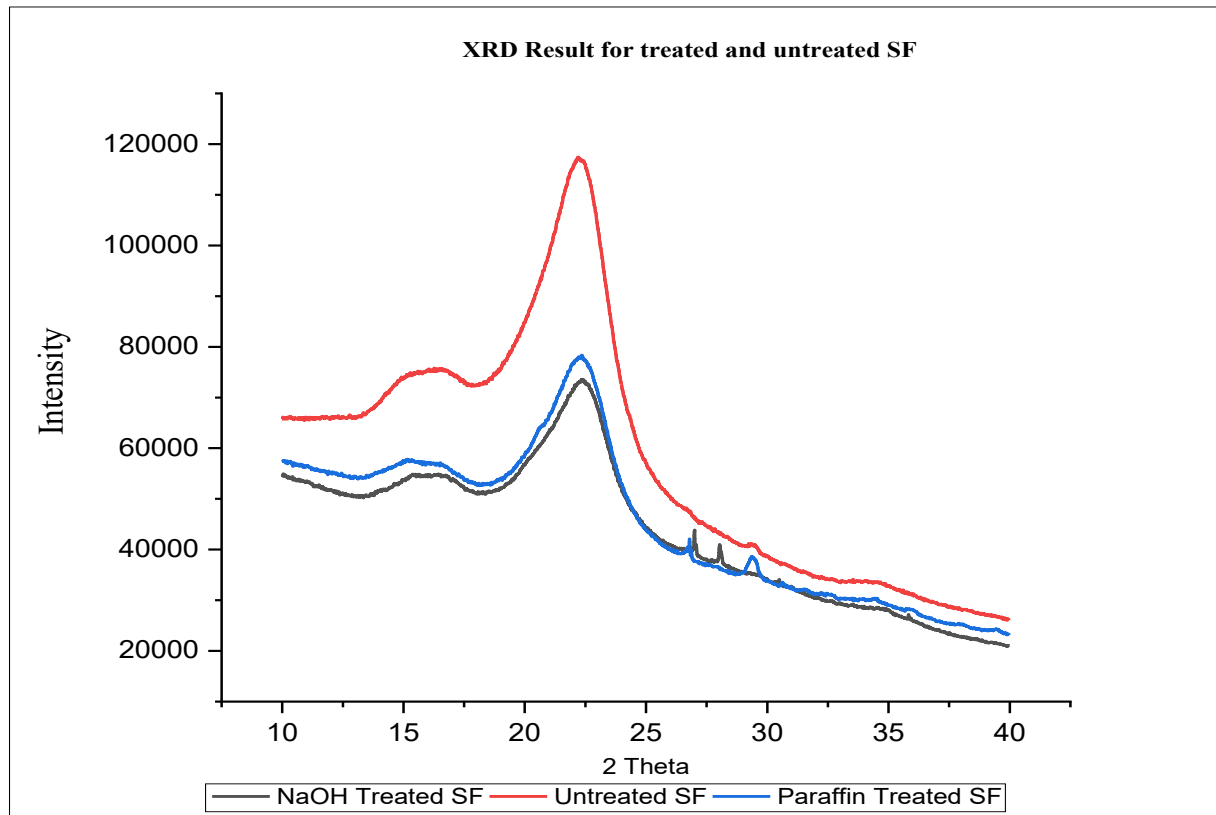


Fig. 11: XRD result for treated and untreated sisal fibre

As it is illustrated in **Figure 11** the NaOH-treated sample shows a significant change in its crystalline profile. While the peak positions remain consistent with cellulose, I indicating no polymorphic transition to cellulose II the relative intensities change markedly. The intensity of the (200) peak at approximately 22.5° becomes more prominent, and the amorphous background is considerably reduced. This results directly from alkali treatment, which effectively dissolves and removes the amorphous matrix materials, thus purifying the cellulose microfibrils and increasing the relative proportion of crystalline domains [54, 89]. Using the empirical Segal method, the crystallinity index (CI) increases from approximately 62.5% in the control to about 75% post-treatment. This enhancement in CI correlates with the well-documented effect of mild alkali treatment in removing non-cellulosic components, leading to improved structural order without inducing a phase change [90].

In contrast, the XRD pattern of paraffin-coated sisal (Sisal 2) shows a distinctly different modification mechanism. The main cellulose I peaks remain visible, confirming that paraffin

deposition is a surface-level physical process that does not change the core crystal structure of the cellulose. However, the diffraction profile displays noticeable changes: a decrease in peak sharpness and the appearance of a broad scattering feature across the 15–25° 2 θ range. This broad feature is typical of semi-crystalline paraffin wax, which exhibits weak diffraction peaks typically around 21° and 24°, corresponding to the orthorhombic phase of hydrocarbon waxes [71, 72, 91]. The paraffin coating acts as a physical barrier, forming a hydrophobic layer on the fibre surface and within superficial pores. In XRD analysis, this adds a non-cellulosic scattering signal to the cellulose pattern, effectively raising the measured amorphous intensity at ~18° 2 θ . Therefore, applying the Segal method to this composite pattern without deconvolution would yield an apparently lower CI, misleadingly indicating reduced crystallinity. This highlights that Alkali and paraffin treatments alter surface properties and moisture resistance without increasing—and in fact, slightly obscuring—the underlying cellulose crystallinity [92].

The comparative XRD analysis clearly distinguishes two orthogonal modification strategies. NaOH treatment is a chemical purification process that selectively etches amorphous content, resulting in sharper cellulose peaks, a cleaner baseline, and a higher crystallinity index. Alkali and paraffin treatment is a physical coating process that adds a secondary material phase, producing a composite diffraction pattern where the cellulose signature is maintained but overlaid by the broad scattering of the wax. These structural differences directly impact composite applications. NaOH treatment enhances intrinsic fibre strength and stiffness by increasing crystallinity, whereas paraffin coating improves interfacial adhesion and moisture barrier properties through surface modification. XRD is a powerful tool to differentiate between chemical and physical fibre treatments. For sisal fibres, NaOH treatment improves cellulose crystallinity by removing non-crystalline components, while paraffin coating offers surface hydrophobicity without altering the crystalline core. The choice of treatment should therefore align with the desired final properties of the fibre for specific engineering applications, particularly in natural fibre-reinforced composites.

3.4. Scanning Electron Microscopy (SEM)

The Scanning Electron Microscopy (SEM) images shown in **Fig. 12** depict the surface morphology of sisal fibres under three different conditions: untreated fibres (A and B), alkali-treated fibres (C and D), and alkali-treated fibres coated with paraffin (E and F). These micrographs provide

important insights into how chemical treatment and surface coating modify the fibre structure, which directly affects fibre–matrix interaction in concrete composites.

SEM images A and B show the surface features of untreated sisal fibres. The fibres appear fairly smooth and dense, with continuous longitudinal grooves, which are typical features of natural plant fibres. These grooves result from the alignment of cellulose microfibrils along the fibre axis. However, the surface is coated with impurities such as lignin, hemicellulose, waxes, and other natural extractives that remain attached to the fibre during natural growth.

In image A, the fibre bundles are clearly visible, with fibrils aligned in parallel, indicating the natural hierarchical structure of sisal fibres. The surface appears dense with limited surface roughness. Such smoothness can reduce mechanical interlocking between the fibre and the cement matrix, potentially leading to weaker interfacial bonding when the fibre is incorporated into concrete.

Image B further highlights the presence of surface layers and deposits, likely comprised of waxy substances and organic materials. These layers serve as a barrier between the fibre surface and cement hydration products, lowering adhesion and bond strength. Additionally, untreated fibres display micro-cracks and weak zones along their surfaces, which may accelerate premature fibre degradation when exposed to the highly alkaline environment of cement paste. As a result, untreated sisal fibres often exhibit poor interfacial transition zone (ITZ) bonding, higher water absorption, and reduced durability when used directly in cement-based composites.

Images C and D show the surface morphology of alkali-treated sisal fibres, typically treated with sodium hydroxide (NaOH). Alkali treatment removes non-cellulosic components such as hemicellulose, lignin, pectin, oils, and surface waxes from the fibre surface. This process exposes the cellulose microfibrils and significantly alters fibre morphology. These findings are similar to the findings of [91, 92, 93, 94] .

In image C, the surface becomes significantly rougher and more fibrillated compared to the untreated fibres. The removal of amorphous materials causes the fibre bundles to partially separate, creating micro-scale grooves, pits, and irregularities. This increased roughness enhances the mechanical interlocking between the fibre and cement matrix, thereby strengthening the interfacial

bond. The exposed microfibrils also increase the available surface area for interaction with hydration products such as calcium silicate hydrate (C–S–H).

Image D depicts further defibrillation and separation of fibre bundles, demonstrating that alkali treatment has successfully disrupted the outer layers of the fibre structure. The fibres seem more porous and textured, enabling better penetration of cement hydration products into the fibre surface. This morphological change enhances stress transfer between the fibre and cement matrix, thus improving mechanical properties such as tensile and flexural strength. However, the increased porosity and exposed cellulose may also lead to higher water absorption, which could affect long-term durability if not properly controlled.

The SEM images E and F depict the morphology of alkali-treated sisal fibres that were subsequently coated with paraffin. This treatment combines the benefits of alkali modification with a protective hydrophobic layer. In image E, the fibre surface still retains the roughened texture resulting from alkali treatment, although parts of the surface appear to be covered by a thin layer of paraffin. The coating partially fills the micro-pores and irregularities formed during alkali treatment. This leads to a more uniform and protected surface, which helps minimize the direct exposure of cellulose fibres to the alkaline cement environment.

Image F shows localized paraffin deposits and smoother coated areas, indicating that the paraffin layer acts as a protective barrier around the fibres. This coating significantly reduces water absorption and prevents excessive swelling of the fibres when embedded in cementitious materials. Additionally, the hydrophobic nature of paraffin enhances the dimensional stability and durability of the fibres, lowering the risk of fibre degradation and mineralization during long-term exposure to cement paste. Despite the coating, the underlying alkali-induced roughness remains partly visible, meaning that mechanical interlocking with the cement matrix is still maintained. This balance between surface roughness and hydrophobic protection helps optimize fibre performance by improving bonding while increasing durability.

The SEM images clearly show that surface modification significantly alters the morphology of sisal fibres. Untreated fibres have smooth surfaces with natural impurities that reduce bonding with cement matrices. Alkali treatment effectively removes these impurities, resulting in a rough and

fibrillated surface that improves mechanical interlocking and fibre–matrix adhesion. However, the increased porosity might lead to higher water absorption.

The additional paraffin coating applied to alkali-treated fibres creates a hydrophobic protective layer that reduces moisture absorption and shields the fibres from alkaline degradation while preserving sufficient surface roughness for bonding. As a result, alkali-treated and paraffin-coated sisal fibres display a more balanced microstructure, combining better interfacial bonding with increased durability in cement-based composites. These microstructural changes, as observed by SEM, provide strong evidence that chemical treatment and surface coating are effective strategies for improving the compatibility and long-term performance of natural fibres in concrete applications.

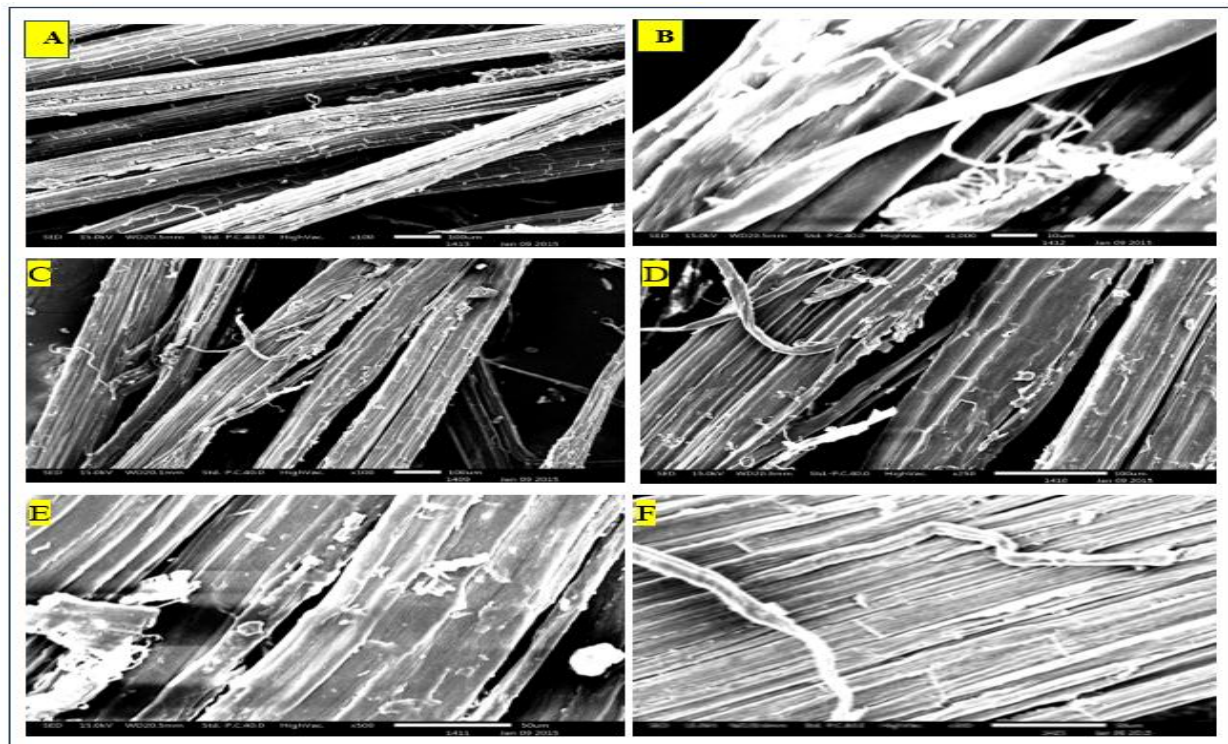


Fig. 12: SEM result for treated and untreated sisal fibre

This involves examining the formation and structure of hydration products like C-S-H gel, the occurrence of microcracks, the bond between the aggregate and the cement paste, and the overall porosity. SEM, along with Energy-Dispersive X-ray Spectroscopy (SEM-EDX), can also offer

semi-quantitative elemental analysis, providing a detailed view of the mortar's internal structure and its connection to macroscopic properties.

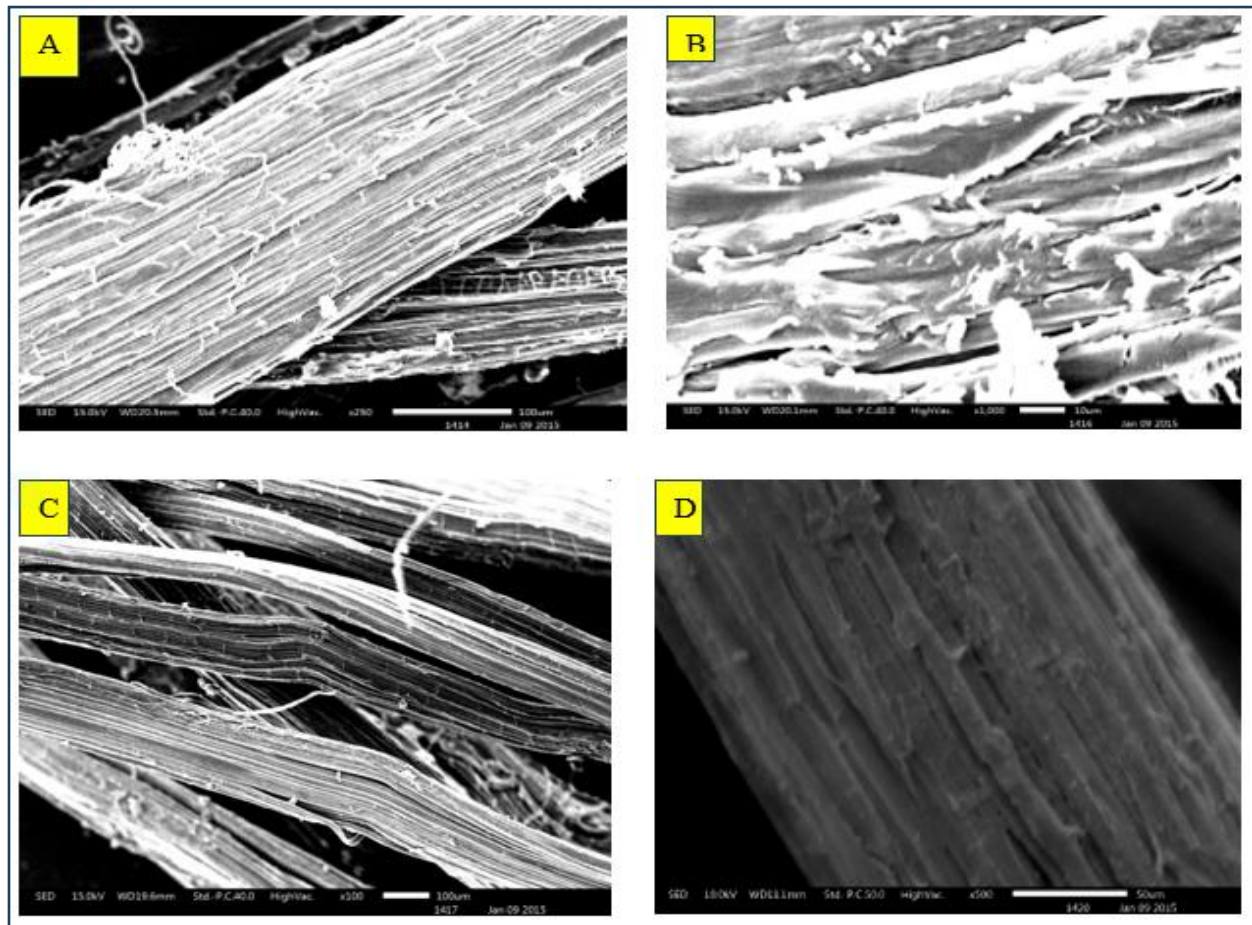


Fig. 13: SEM result for paraffin and alkali - treated sisal fibre

In contrast, the sodium hydroxide (NaOH) treated fibre, examined at a higher magnification of 250x, displays a notably altered and activated surface morphology. The alkali treatment effectively dissolves and removes the amorphous hemicellulose, lignin, pectin, and surface impurities that coated the untreated fibre. This cleansing process results in significant defibrillation, in which the main fibre bundle separates into individual cellulose microfibrils. The surface becomes rougher and more textured, showing exposed fibrillar structures with increased surface area. This improved topography is highly advantageous for composite applications, as it provides more sites for mechanical interlocking with cement hydration products. The cleaner, more reactive cellulose surface promotes denser, closer contact with the matrix, encouraging stronger physicochemical

bonds. Consequently, NaOH treatment mainly functions as a bonding optimization process, aiming to address the weak interface issue and likely enhance the composite's flexural strength, fracture toughness, and resistance to crack propagation, although the fibre's susceptibility to moisture may still be a concern without additional modification.

Conversely, as seen in **Fig. 13 (A) and (B)**, alkali-treated and paraffin-coated sisal fibre, examined at an even higher magnification of 500x, exhibits a fundamentally different surface engineering approach centred on passivation rather than activation. The treatment involves applying a thin, continuous film of paraffin wax to the fibre. The resulting morphology displays a smooth, uniform, and occluded surface, in which the natural grooves, pores, and fibrillar features of the fibre are visibly filled or bridged by the deposited layer. This wax coating acts as an effective physical barrier, sealing the micro-pores and capillaries that give the fibre its hydrophilic nature. By forming this hydrophobic barrier, alkali and paraffin treatment directly addresses the primary durability concern for sisal fibre in concrete—its high-water absorption, which leads to vulnerability to alkaline hydrolysis and swelling. Although this treatment should markedly decrease the composite's water uptake and enhance its resistance to moisture-related degradation, the smooth, non-porous coating may also diminish the potential for mechanical anchorage with the cement matrix. The findings align with previous research of [95, 96, 97], which suggests this may lead to a weaker physical interlock compared to the roughened NaOH-treated fibres, potentially offsetting some mechanical benefits.

In summary, the SEM analysis highlights a clear trade-off influenced by the surface treatment applied to sisal fibre. The untreated fibre has inherent flaws such as a weak surface layer and high porosity, which limit its effectiveness. NaOH treatment enhances the fibre's mechanical performance by improving the fibre-matrix bond through surface roughening and purification, making it suitable for applications where increased toughness and crack control are important. Alkali and paraffin treatments improve the fibre's durability by imparting hydrophobicity and sealing its surface, making it more appropriate for environments where moisture resistance is essential. For advanced composite applications that require a balance of strong bonding and long-term durability, a sequential treatment—such as an initial NaOH cleaning followed by a paraffin or silane coating—is often recommended in contemporary research. This dual approach utilizes the mechanical interlocking capacity of the roughened surface while protecting it with a durable

hydrophobic layer, thereby mitigating the inherent limitations of sisal fibre in cementitious composites.

3.5. Energy Dispersive X-ray (EDX)

Energy Dispersive X-ray (EDX) analysis is a sophisticated analytical method used to determine the elemental composition of materials, including concrete and cement-based composites. Since concrete is a heterogeneous material made up of cement, aggregates, water, and various admixtures, it requires accurate characterization to understand its chemical and microstructural features. EDX, often combined with Scanning Electron Microscopy (SEM), provides both qualitative and quantitative data on the elements in a sample, enabling researchers and engineers to examine chemical interactions, hydration products, and potential impurities within the concrete matrix.

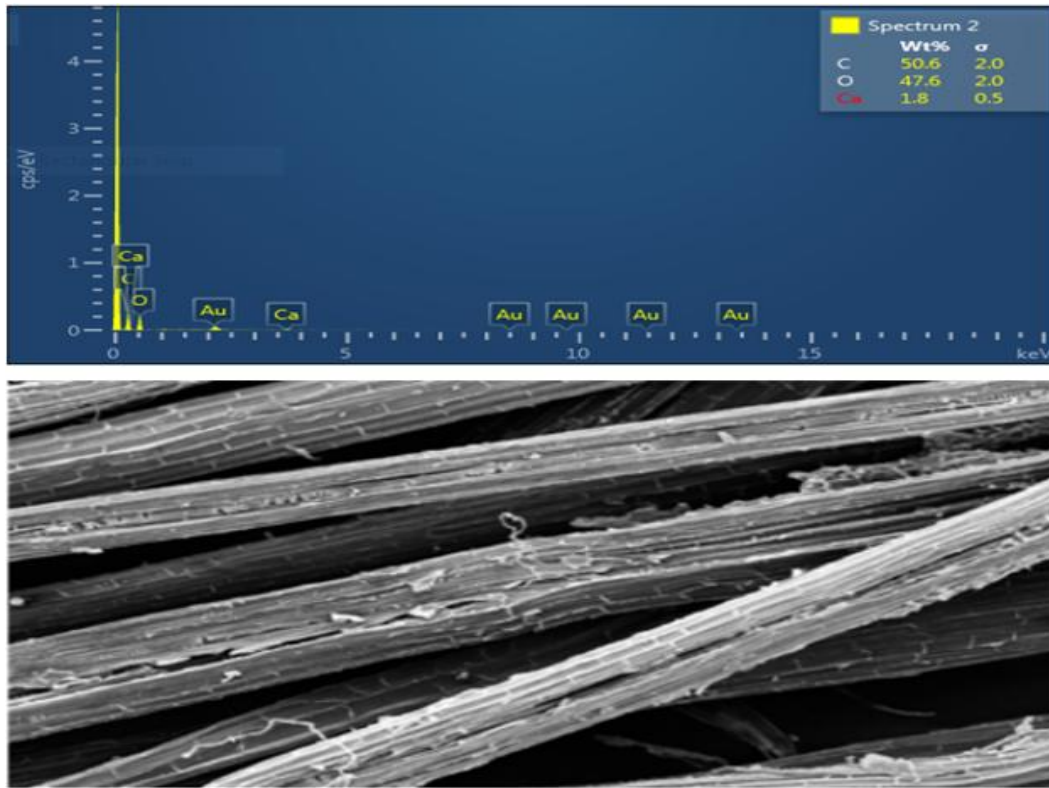


Fig. 14: EDX for untreated sisal fibre

EDX analysis plays a crucial role in exploring concrete durability, performance, and how additives or fibres influence the material. By detecting elements like calcium, silicon, aluminium, and iron, as it is shown in **Figure 14** EDX assists in evaluating cement hydration quality, the dispersion of supplementary materials, and the presence of harmful compounds that could impact strength or lifespan. This method is commonly employed in both research and quality assurance to refine concrete mixes and to better understand the relationships among composition, microstructure, and mechanical properties. The SEM (Scanning Electron Microscopy) and EDX (Energy Dispersive X-ray Spectroscopy) data provide essential insights into the characteristics of raw, untreated sisal fibre. This analysis is vital for understanding the fibre's inherent qualities, which directly affect its performance when integrated into a concrete matrix. The SEM micrographs display the fibre's surface morphology, typically showing a rough, fibrillated texture with longitudinal grooves and visible pores. This roughness can create sites for mechanical interlocking with cement hydrates, which may partly explain the observed improvements in post-cracking toughness and flexural strength in composite tests. However, the SEM also probably reveals uneven coatings and impurities, such as residual lignin, pectin, and waxy substances, on the fibre surface. These impurities form a weak boundary layer that hampers the formation of a strong chemical bond with the cement paste, resulting in a weak interfacial transition zone (ITZ). This weak ITZ is a primary cause of the common reduction in compressive strength when untreated fibres are incorporated into concrete.

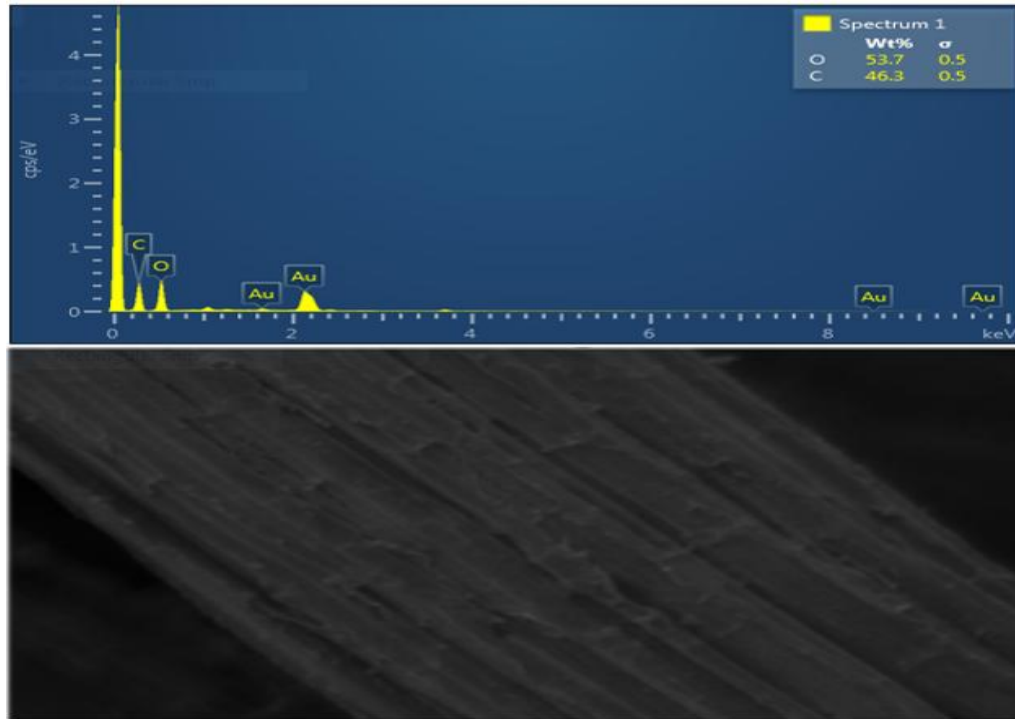


Fig. 15: EDX for alkali-treated sisal fibre

Complementing the morphological data, the EDX spectrum provides an elemental fingerprint of the fibre's surface. As it is illustrated in **Figure 15** The dominant peaks clearly indicate Carbon (C) and Oxygen (O), confirming the organic, lignocellulosic nature of sisal fibre. This composition is key to its limitations in concrete. The high cellulose content makes it susceptible to alkaline hydrolysis in the high-pH environment of the cement pore solution, causing long-term degradation. Minor peaks for elements such as Calcium (Ca) or Potassium (K) are often present due to natural biominerals or soil contamination, but the notable absence of significant cementitious elements (such as silicon or aluminium in bonded forms) confirms that the analysis relates solely to the pristine fibre. The purely organic EDX signature underscores a fundamental chemical incompatibility with the inorganic cement matrix; the fibre lacks reactive sites for strong chemical adhesion and has no inherent resistance to the alkaline environment.

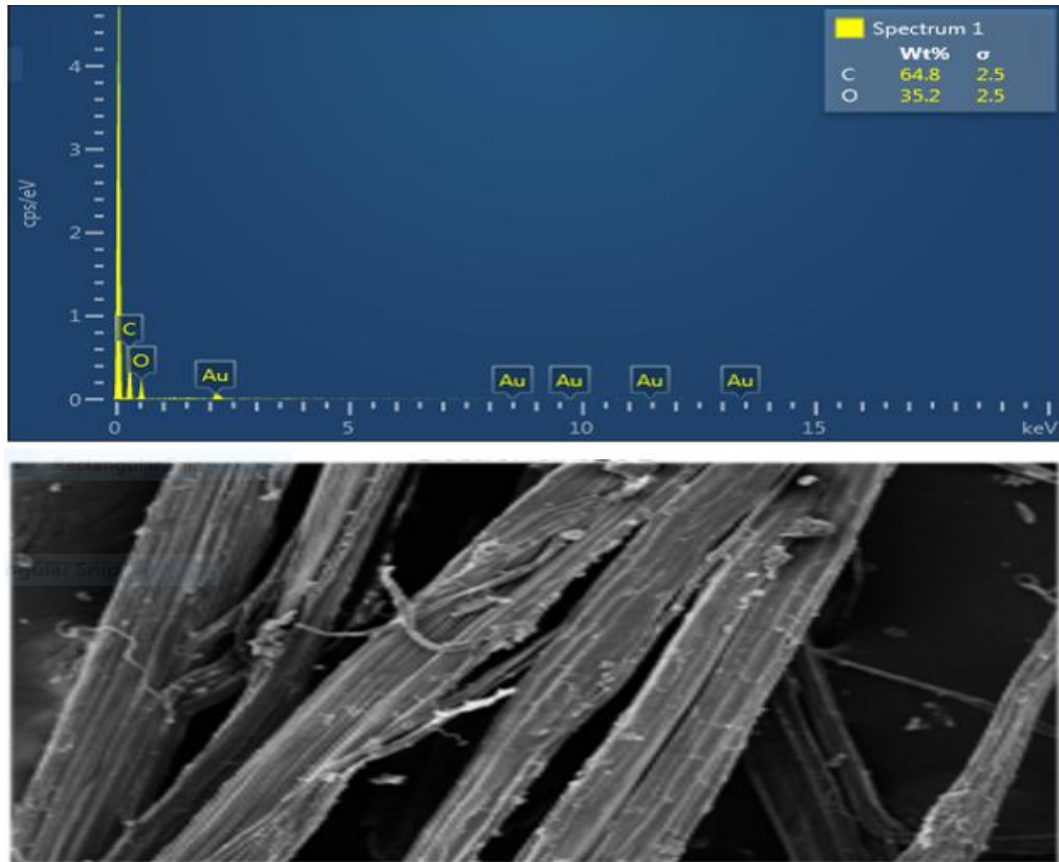


Fig. 16: EDX alkali and paraffin-treated sisal fibre

The characterization of sisal fibre after treatment with sodium hydroxide (NaOH), commonly known as alkali treatment, reveals significant changes in its morphology and surface chemistry compared to the untreated fibre. as it is shown in **Figure 16** these modifications are crucial for enhancing the fibre's performance as a reinforcement in cement composites. The SEM micrographs of the NaOH-treated fibre likely show a markedly different surface topography. The most noticeable change is the removal of the amorphous surface layer, which primarily consists of hemicellulose, lignin, pectin, and waxy substances. This cleaning action exposes the underlying fibrillar structure of the cellulose microfibrils, resulting in a rougher and more textured surface. The fibres may appear more separated and well-defined, with increased surface areas. This improved roughness is highly advantageous for mechanical interlocking with the cement matrix, as it offers more sites for hydration products to anchor. Additionally, the treatment often reduces the diameter of individual fibres by dissolving non-crystalline components, thereby enhancing fibre flexibility and dispersibility within the mix. The visible pores may still be present, but the

overall surface becomes clearer, leading to denser, more intimate contact with the cement paste, thereby boosting interfacial bond strength.

Concurrently, the EDX spectrum of the treated fibre would reflect this chemical cleansing. While the dominant peaks for Carbon (C) and Oxygen (O) remain, confirming the fibre's organic base, their relative ratios may shift. More importantly, the spectrum would show a significant reduction or complete elimination of peaks associated with impurities commonly found in untreated fibre, such as Potassium (K), Magnesium (Mg), or the specific Calcium (Ca) from non-cementitious sources like oxalates. The treatment effectively washes away these soluble extractives and minerals. The surface primarily becomes cellulose, which, while still organic, provides a more uniform and reactive substrate for potential secondary treatments or direct interaction with the cement. The absence of contaminant peaks clearly indicates a purified fibre surface. However, it is crucial to note that alkali treatment alone usually does not introduce new inorganic elements into the fibre surface; its primary role is to promote subtraction and exposure. For a more radical chemical modification, such as creating a hydrophobic barrier, subsequent treatments like salinization are required.

In summary, NaOH treatment significantly enhances the suitability of sisal fibre for composite applications. SEM evidence of a cleaner, rougher surface directly addresses the weak interfacial bond issue by encouraging better mechanical adhesion. The EDX confirmation of a purified cellulose surface explains the improved chemical consistency at the fibre-matrix interface. This dual improvement results in a stronger, more stable Interfacial Transition Zone (ITZ), which should lead to better retention of compressive strength and improved flexural performance in the concrete composite. Additionally, by removing hydrophilic hemicellulose and lignin, the treatment can moderately reduce the fibre's tendency to absorb water, supporting better long-term durability. Therefore, alkali treatment is an essential, foundational processing step that mitigates the inherent drawbacks of the raw fibre, transforming it from a problematic inclusion into a more reliable and effective reinforcing agent in cementitious materials.

The characterization of sisal fibre after treatment with paraffin shows a clear and specific change in its surface properties, primarily aimed at enhancing its compatibility with a cementitious matrix. Unlike chemical treatments that modify the fibre's structure, alkali and paraffin treatments primarily serve as physical coatings, forming a barrier on the fibre surface. This approach directly tackles one of the key weaknesses of natural fibres in concrete: their high hydrophilicity and related durability concerns [95, 96, 97, 98].

The SEM micrograph of the paraffin-treated fibre is expected to show a significant change in surface texture. Instead of the exposed, rough fibrils seen after alkali treatment, the surface likely appears smoother and more uniform, as it is partially or fully covered by a thin film of paraffin wax. The longitudinal grooves and pores characteristic of the raw fibre may be filled or bridged by this coating. While this reduces the microscopic roughness needed for mechanical interlocking, it creates a more consistent, non-porous interface. The main advantage observable in the SEM is the sealing of micropores and capillaries on the fibre surface. This physical barrier is crucial for preventing the fibre from acting as an internal wick, which is the primary cause of high-water absorption in untreated fibre-reinforced composites.

Concurrently, the EDX spectrum of the paraffin-treated fibre would reflect the presence of this superficial coating. The dominant peaks would still be for Carbon (C) and Oxygen (O), as both cellulose and paraffin (a hydrocarbon) are carbon-based. However, a key indicator of the treatment would be a noticeable increase in the relative intensity of the carbon peak compared to the oxygen peak. This shift occurs because paraffin is a long-chain alkane with a higher carbon-to-oxygen ratio than the cellulose and hemicellulose of the native fibre surface. The spectrum would likely show a significant reduction in the signal from other elemental impurities (e.g., K, Mg, Ca), not because they are dissolved, but because they are effectively shielded or covered by the overlying paraffin layer, attenuating their X-ray emission. The EDX thus confirms the successful application of an organic, hydrophobic coating on the fibre surface.

In summary, alkali and paraffin treatments enhance the sisal fibre by directly addressing durability rather than bond strength. The SEM evidence of a sealed surface effectively reduces water ingress and fibre swelling. The EDX confirmation of a hydrocarbon-rich surface layer explains the increased hydrophobicity. This improvement should lead to a significant decrease in the

composite's water absorption capacity and improved resistance to the moist, alkaline environment of concrete, thereby boosting long-term durability. However, this treatment does not enhance and may slightly reduce the mechanical interlocking potential, as the smooth coating can weaken the frictional bond with the cement paste. Therefore, alkali and paraffin treatments are most effective when used on fibres that have already been cleaned (e.g., through a mild alkali treatment) to ensure proper coating adhesion, or in applications where moisture resistance is more important than maximum mechanical performance. It changes the fibre from a hydrophilic vulnerability into a more chemically inert inclusion within the cement matrix.

3.6. Fourier-Transform Infrared (FTIR) Spectroscopy

The chemical structure of a fibre influences not only its mechanical properties but also its interfacial compatibility with the matrix and its resistance to environmental degradation. Fourier-Transform Infrared (FTIR) Spectroscopy is a powerful analytical technique used to identify the molecular bonds and functional groups within a material. By measuring the absorption of infrared light at specific wavelengths, FTIR provides a molecular "fingerprint" that can reveal details about the fibre's chemical composition, detect the presence of surface treatments or sizing agents, and identify chemical changes resulting from processing or exposure to aggressive environments (such as the alkaline resistance test). In this study, as it is shown in **Fig. 17, 18, 19**, FTIR spectroscopy is employed to characterize the virgin fibres and to analyze any chemical modifications that occur after accelerated ageing, thereby linking macroscopic performance changes to alterations at the molecular level [99, 100, 101, 102, 103, 104, 105].

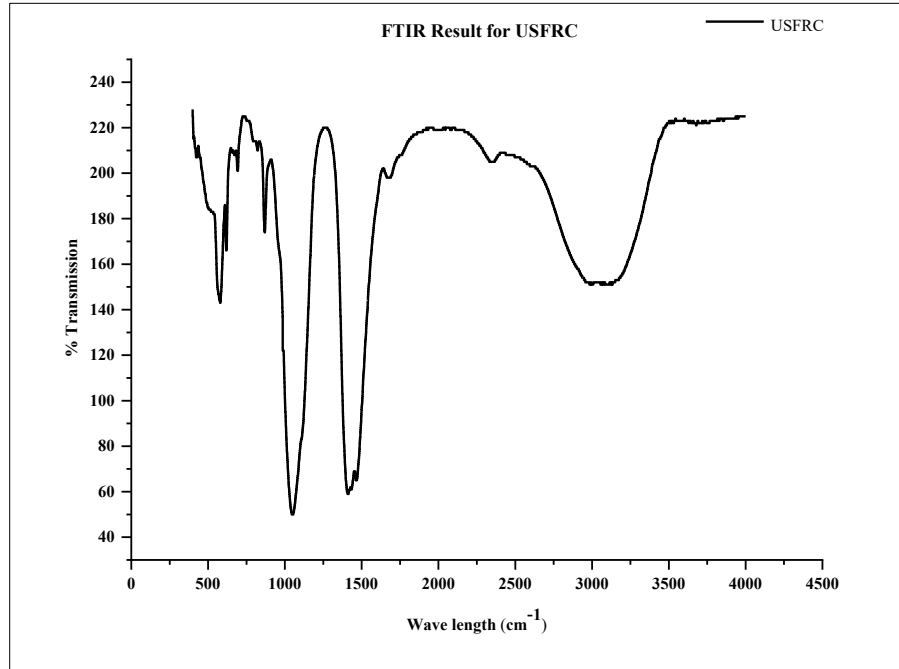


Fig. 17: FTIR results for USFRC

As shown in **Fig. 18 and 19**, the FTIR spectrum of the paraffin-coated and alkali-treated sisal fibre-reinforced material (PATSFRC) clearly indicates significant chemical modifications resulting from the combined treatment process, thereby improving its suitability for concrete applications. The broad band observed in the range of 3200–3500 cm⁻¹ corresponds to the O–H stretching vibrations of hydroxyl groups found in cellulose, hemicellulose, and lignin. The noticeable decrease in the intensity of this peak suggests the effective removal of hemicellulose and partial lignin during alkali treatment, as well as the hydrophobic effect of the paraffin coating. This reduction confirms a decrease in the fibres' hydrophilic nature, which is advantageous for limiting moisture absorption in concrete. The peaks around 2900 cm⁻¹ are attributed to C–H stretching vibrations of chains, and their relative prominence indicates the presence of paraffin hydrocarbons, confirming successful coating and increased hydrophobicity.

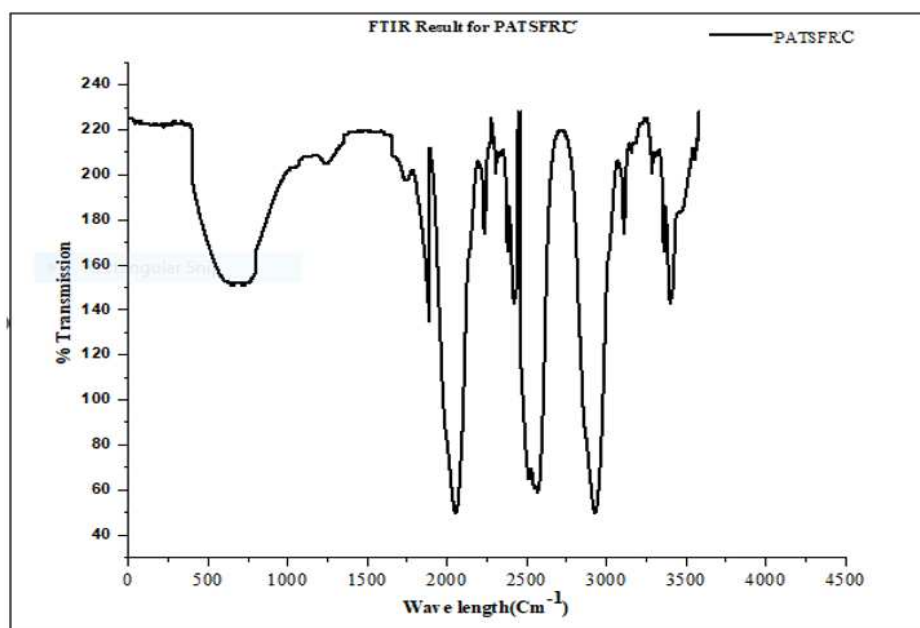


Fig. 18: FTIR results for PATSFRC

Furthermore, as shown in **Fig. 17**, the reduction or disappearance of the peak near 1730–1750 cm^{-1} , typically associated with C=O stretching of hemicellulose and ester groups, indicates the effective removal of amorphous components during alkali treatment, thereby increasing cellulose content. The region between 1500 and 1600 cm^{-1} , which relates to aromatic skeletal vibrations of lignin, shows decreased intensity, further confirming partial delignification and improved fibre surface condition. Strong peaks in the range of 1000–1200 cm^{-1} correspond to C–O and C–O–C stretching vibrations of the cellulose backbone, indicating that the cellulose structure remains intact while non-cellulosic components are reduced. Minor peaks at lower wavenumbers reflect structural rearrangements and interactions caused by chemical treatment and coating.

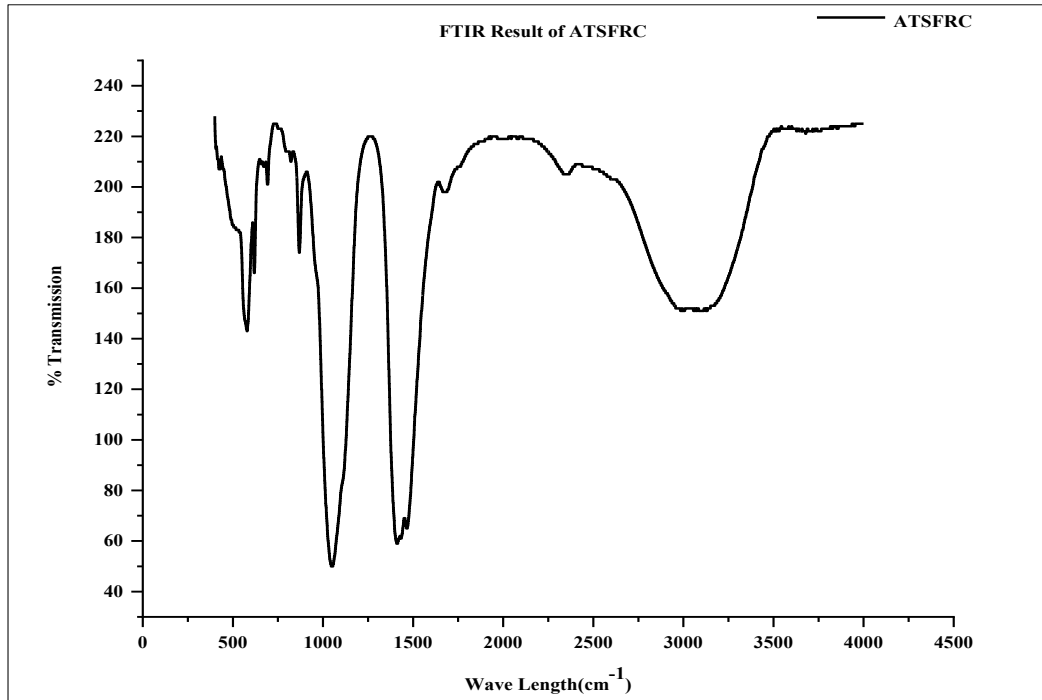


Fig. 19: FTIR results for ATSFRC

Overall, these spectral changes confirm that alkali treatment combined with paraffin coating significantly enhances the physicochemical properties of sisal fibres. The treatment reduces water absorption by lowering hydroxyl content and creating a hydrophobic barrier, improves fibre–matrix bonding through increased surface roughness, and boosts durability by shielding fibres from alkaline degradation within the cement matrix. Additionally, removing unstable components such as hemicellulose and lignin increases chemical stability, while better dispersion and interfacial bonding improve mechanical properties, including tensile and flexural strength. As a result, PATSFRC demonstrates superior performance, making it a promising material for durable, high-performance fibre-reinforced concrete applications.

3.7. Physical Properties of Concrete

The physical properties of mortar are key indicators of its quality and suitability for construction use. These properties, which result directly from its mix design and microstructural development, influence the material's capacity to resist applied loads and environmental stresses. Assessing these

characteristics is vital for meeting standards and for forecasting the long-term structural performance and service life of masonry and plasterwork.

3.7.1. Compressive Strength

The compressive strength results of the concrete mixtures at curing ages of 3, 7, 14, 28, and 56 days are shown in **Fig. 20**. Compressive strength is one of the most important mechanical properties of mortar because it indicates the material's ability to resist axial loads without failure. The results clearly demonstrate that compressive strength increases with curing age across all mixes, primarily due to ongoing cement hydration and the gradual development of a denser microstructure. For the control mix, compressive strength increased steadily with curing time. It was approximately 15 MPa in 3 days, rising to about 21 MPa at 7 days, 24 MPa at 14 days, 28 MPa at 28 days, and around 31 MPa at 56 days. This gradual rise is typical of conventional mortar and is primarily due to cement hydration, which produces calcium silicate hydrate (C–S–H) gel and calcium hydroxide. The formation of C–S–H gel enhances the bond between the cement paste and fine aggregates, thereby increasing the load-bearing capacity of the mortar over time.

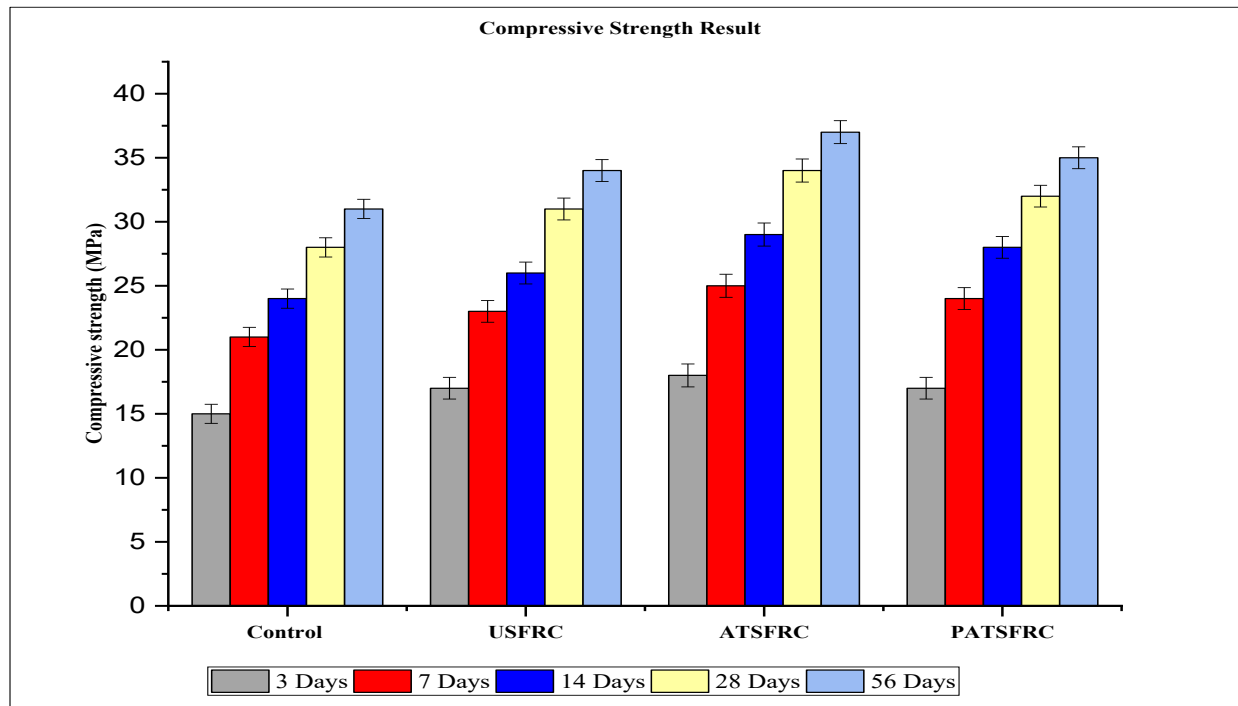


Fig. 20: Compressive strength of sisal fibre reinforced concrete

The USFC mix exhibited higher compressive strength than the control mix at all curing ages. The compressive strength increased from approximately 1.7 MPa at 3 days to 23 MPa at 7 days, 26 MPa at 14 days, 31 MPa at 28 days, and about 34 MPa at 56 days. The improvement in strength can be attributed to the addition of fibres and supplementary materials, which enhance the concrete's internal structure. Fibres help control microcrack development, while supplementary materials act as micro-fillers that reduce voids within the matrix. In addition, the pozzolanic reaction between the supplementary material and calcium hydroxide forms additional C–S–H gel, which further enhances compressive strength.

Among all the mixes, the ATSFRC mixture showed the highest compressive strength values at all curing ages. The compressive strength rose from approximately 1.8 MPa at 3 days to 25 MPa at 7 days, 29 MPa at 14 days, 34 MPa at 28 days, and reached around 37 MPa at 56 days. The superior performance of this mix indicates that the combination of materials used in ATSFRC provides a strong synergistic effect. The presence of fibres enhances crack-bridging capacity and delays crack propagation, while supplementary cementitious materials increase the density of the cement matrix through pozzolanic reactions and filler effects. This results in a stronger, more compact microstructure, which improves the mortar's load-bearing capacity.

The PATSFRC mix also showed significant improvement in compressive strength compared with the control mixture. The compressive strength increased from approximately 17 MPa at 3 days to 24 MPa at 7 days, 28 MPa at 14 days, 32 MPa at 28 days, and reached about 35 MPa at 56 days. Although the strength is slightly lower than that of the ATSFRC mixture at later ages, it still demonstrates a considerable enhancement over the control mix. This improvement is likely due to the combined effect of fibre reinforcement and improved bonding within the cementitious matrix, which contributes to better stress distribution and crack resistance.

Overall, the results show that all mortar mixes exhibit a consistent increase in compressive strength with curing age, attributable to continued hydration. The incorporation of fibres and supplementary cementitious materials significantly improves the compressive strength compared with the control mortar. These materials contribute to a denser microstructure, reduced porosity, and improved bonding within the matrix. In general, the ranking of compressive strength performance at later curing ages can be summarized as $\text{ATSFRC} > \text{PATSFRC} > \text{USFRC} > \text{Control}$. The ATSFRC mix

demonstrated the best overall performance, indicating that the combination of fibres and supplementary materials provides the most effective improvement in mortar's mechanical properties. These findings suggest that such modified mortar mixtures have strong potential for use in construction applications requiring enhanced strength and durability. The above result is supported by the findings of similar studies [1, 2, 61, 106, 107, 108].

3.7.2. Flexural Strength

The flexural strength results of the mortar mixtures at different curing ages (3, 7, 14, 28, and 56 days) are presented in **Fig. 21**. Flexural strength is an important parameter that reflects the ability of concrete to resist bending stress and cracking, which is particularly significant for materials reinforced with fibres and supplementary cementitious materials. The control mortar exhibited the lowest flexural strength at all curing ages. The strength increased gradually from approximately 2.2 MPa at 3 days to about 4.4 MPa at 56 days, indicating normal strength development due to continued cement hydration. As hydration progresses, more calcium silicate hydrate (C–S–H) gel forms, enhancing the bond between the cement matrix and aggregates and improving flexural resistance over time.

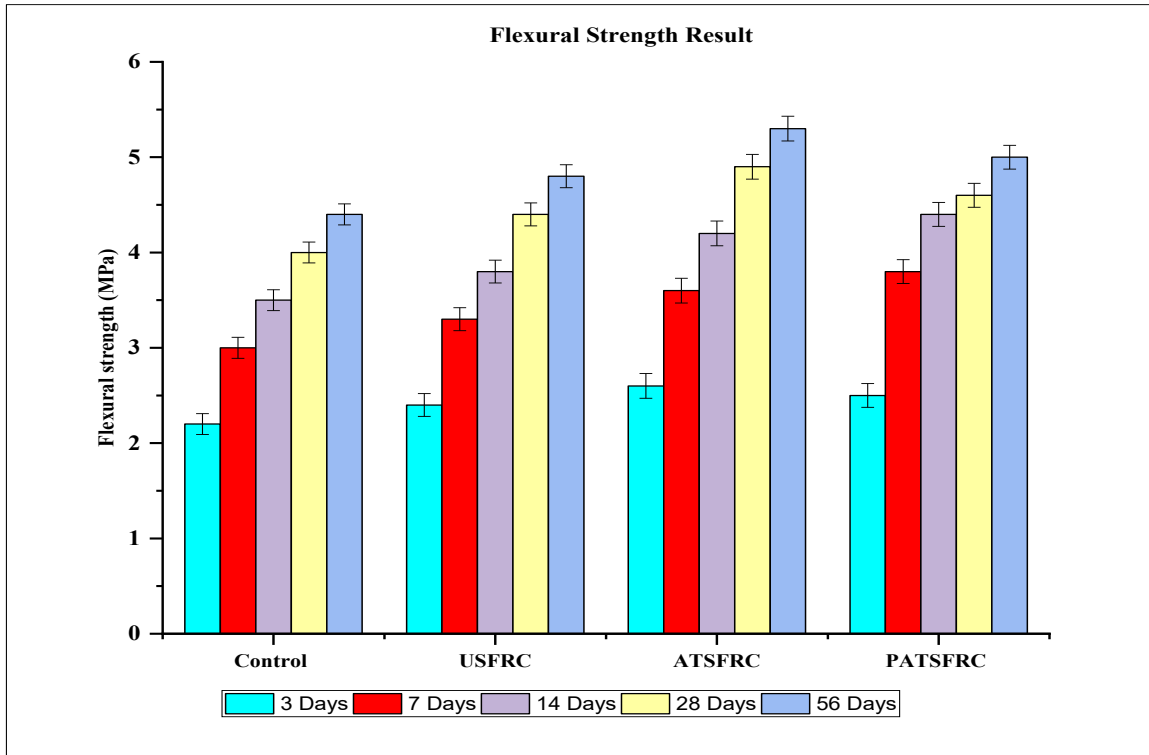


Fig. 21: Flexural strength of sisal fibre reinforced concrete

The USFRC mix showed higher flexural strength than the control specimen at all curing ages. The strength increased from about 2.4 MPa in 3 days to nearly 4.8 MPa at 56 days. This improvement can be attributed to the presence of fibres and the modification of the matrix structure. Fibres act as crack-bridging elements, which delay crack propagation and improve the post-cracking behaviour of mortar. In addition, supplementary materials contribute to a denser microstructure through filler effects and secondary pozzolanic reactions, thereby enhancing bonding within the matrix.

The ATSFRC mixture exhibited the highest flexural strength among all the mixes throughout the curing period. The value increased from approximately 2.6 MPa in 3 days to about 5.3 MPa at 56 days. The superior performance of this mixture can be attributed to the synergistic effect of fibre reinforcement and to improved matrix densification resulting from the incorporation of supplementary materials. The fibres help distribute tensile stresses more evenly across the mortar matrix, while the pozzolanic reaction produces additional C–S–H gel, reducing porosity and

strengthening the interfacial transition zone. This combination significantly enhances the mortar's resistance to bending stresses.

Similarly, the PATSFRC mixture also demonstrated improved flexural strength compared with the control mortar, increasing from around 2.5 MPa at 3 days to approximately 5.0 MPa at 56 days. Although its strength is slightly lower than that of ATSFRC at later curing ages, it still shows a considerable improvement compared to the control mixture. The enhanced performance may be due to better fibre dispersion and improved matrix bonding, which help resist crack initiation and propagation.

In general, all mortar mixtures exhibited a consistent increase in flexural strength with curing time. This trend is primarily associated with ongoing hydration and the progressive development of the internal microstructure. The incorporation of fibres and supplementary materials significantly enhanced flexural performance compared with the control mortar. These materials improve crack resistance, reduce brittleness, and increase the energy absorption capacity of the mortar matrix.

Overall, the results indicate that the combined use of fibre reinforcement and supplementary cementitious materials can effectively improve the flexural behaviour of mortars. The ATSFRC mixture demonstrated the best performance, suggesting that this combination provides an optimal balance between matrix densification and crack-bridging mechanisms, thereby enhancing the mortar's structural performance. These findings are supported by the findings of similar studies [4, 11, 55, 69, 109].

3.7.3. Tensile Strength

The diagram illustrates the development of split tensile strength for different mortar mixes (Control, USFRC, ATSFRC, and PATSFRC) at curing ages of 3, 7, 14, 28, and 56 days. The results show a consistent increase in tensile strength with curing time across all mixes, indicating continuous cement hydration and improved microstructure.

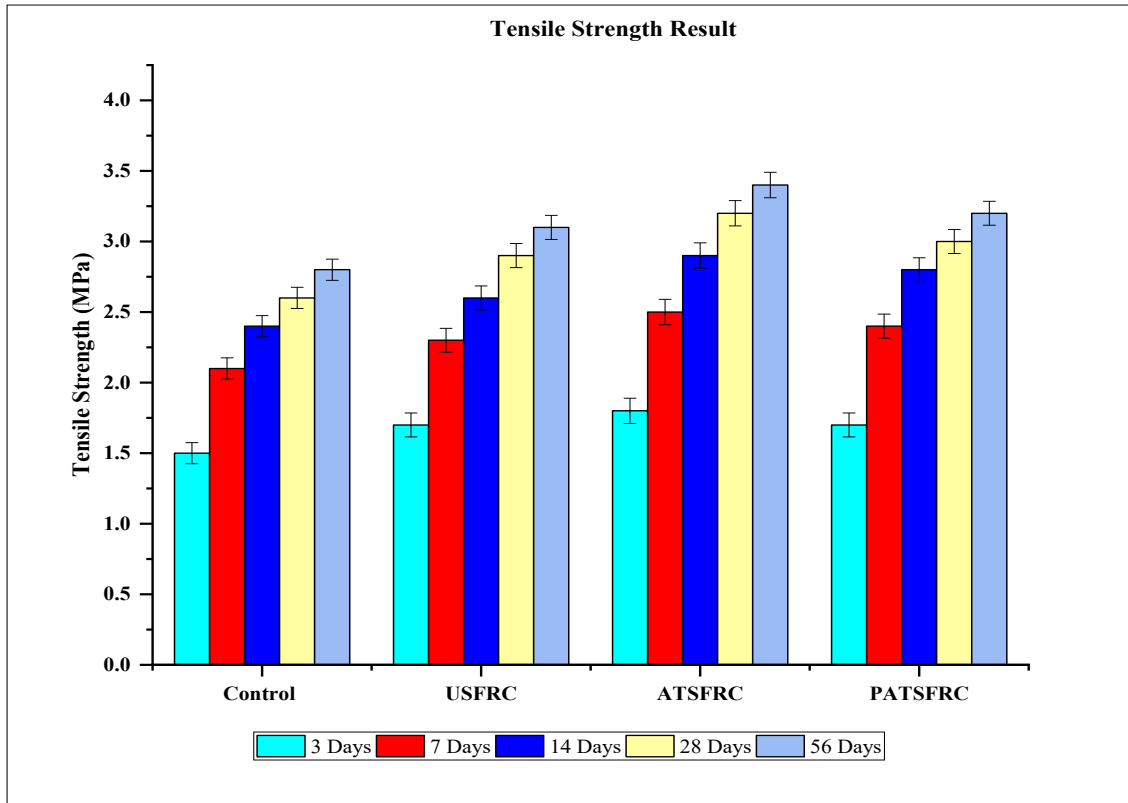


Fig. 22: Tensile strength of sisal fibre reinforced concrete

Fig. 22 presents the split tensile strength results for different concrete mixes (Control, USFRC, ATSFRC, and PATSFRC) at curing ages of 3, 7, 14, 28, and 56 days. The results show a consistent increase in tensile strength with curing time across all mixes, indicating continuous cement hydration and an improved microstructure. The control mix (plain M25 mortar) exhibited the lowest tensile strength at all curing ages. Strength increased gradually from about 1.5 MPa at 3 days to approximately 2.7 MPa at 56 days. This increase is mainly attributed to the normal hydration process of cement, which forms calcium silicate hydrate (C-S-H) gel that enhances bonding within the mortar matrix.

The USFRC mix exhibited higher tensile strength than the control mix across all curing periods. Strength rose from about 1.7 MPa at 3 days to approximately 3.3 MPa at 56 days. This enhancement arises from the micro-filler effect and pozzolanic activity of the supplementary material, which refines the pore structure and strengthens the interfacial transition zone between cement paste and aggregates. Consequently, crack propagation is reduced, and tensile resistance

is improved. The ATSFRC mix showed an additional gain in tensile strength compared with both the control and USFRC mixes. Its tensile strength increased from around 1.8 MPa at 3 days to about 3.4 MPa at 56 days, the highest among all mixtures. This improvement mainly stems from the combined effect of pozzolanic materials and fibre reinforcement, which enhances crack-bridging ability and delay crack propagation under tensile loading.

Similarly, the PATSFRC mix showed significantly higher tensile strength than the control mix throughout the curing period. The strength rose from about 1.7 MPa at 3 days to around 3.1 MPa at 56 days. The fibres in this mixture enhanced tensile performance by bridging microcracks and distributing stress more evenly within the mortar matrix.

Overall, the results clearly demonstrate that fibre reinforcement and supplementary cementitious materials significantly enhance the split tensile strength of mortar compared to the conventional control mix. The increase in strength with curing time indicates improved hydration and microstructural densification. Among the tested mixtures, ATSFRC performed best, suggesting that combining pozzolanic materials with fibres provides an effective strategy for enhancing the tensile behaviour and crack resistance of concrete. These findings are supported by similar studies [18, 42, 70, 110, 111].

3.8. Durability Performance Assessment

Durability refers to the ability of mortar to withstand deterioration from environmental and service conditions over its intended lifespan. Unlike strength, which is a short-term characteristic, durability is a measure of long-term performance. Evaluating durability involves subjecting the mortar to tests that mimic harsh environments, such as water ingress, freeze-thaw cycles, or chemical attack. A thorough durability assessment is essential for predicting service life, reducing maintenance costs, and ensuring the long-term sustainability of construction.

3.8.1. Alkaline Resistance Evaluation

The long-term durability of fibre-reinforced cementitious composites depends largely on fibre stability within the high-pH environment of the cement matrix. Portland cement pore solutions are highly alkaline, typically with a pH exceeding 13.5, which can cause chemical degradation of

many organic and some synthetic fibres through hydrolysis and dissolution. This degradation reduces tensile strength and embrittles the fibres, ultimately diminishing the composite's post-cracking ductility and toughness. Therefore, assessing a fibre's resistance to alkaline attack is vital for predicting its service life and performance. This section outlines the methodology for assessing fibre alkaline resistance, in which specimens are exposed to a simulated cement pore solution under controlled conditions. Subsequent analysis of changes in mechanical properties or chemical structure provides essential data for selecting suitable fibres for durable cement-based applications.

Sample Description	Initial 28-Day Compressive Strength (MPa)	Residual Compressive Strength After 30 Cycles (MPa)	Strength Retention (%)	Mass Change (%)	Visual & Physical Degradation Observations
Control (Plain M25 Mortar)	26.4	25.8	97.7%	+0.3%	Minor surface scaling, no cracks.
Mortar + Untreated Fibre	20.1	14.1	70.1%	+2.1% / -1.8%*	Severe surface erosion, extensive microcracking, fibres swollen/decomposed, spalling at fibre locations.
Mortar + NaOH-Treated Fibre	25.3	23.2	91.7%	+0.8%	Good integrity. Fine surface crazing, fibres intact, but some minor debonding visible.
Mortar + Paraffin-Treated Fibre	23.0	22.0	95.7%	+0.5%	Excellent surface integrity. Minimal cracking; fibres well-protected; best overall preservation.

The alkali resistance results show significant differences in the long-term stability of mortar exposed to alkaline environments when using untreated, NaOH-treated, and paraffin-treated sisal fibres. The control mix remained stable, retaining 97.7% of its initial strength with only minor surface changes, indicating that plain mortar is largely unaffected by alkali exposure. Mortar with untreated fibres performed worst, with strength retention falling to 70.1% and visible signs of degradation, including fibre swelling, decomposition, extensive microcracking, and localized spalling. This is because untreated sisal fibres contain hemicellulose and lignin, which are highly

susceptible to alkaline attack. These components dissolve or weaken in high-pH environments, leading to loss of fibre integrity, reduced adhesion, and ongoing internal damage.

In contrast, NaOH-treated fibres showed significantly improved alkali resistance, retaining 91.7% of their strength. Alkaline treatment removes unstable components such as waxes, hemicellulose, and partial lignin, leaving a more resistant, cellulose-rich structure with enhanced surface roughness. As a result, the fibres maintain better stability, exhibit only minor debonding, and preserve the mortar's internal cohesiveness under alkaline exposure. Paraffin-treated fibres showed the best resistance, with 95.7% strength retention and minimal visible degradation. The hydrophobic coating protects the fibres from direct chemical attack by limiting moisture and alkali ingress, helping them retain structural integrity and preserving the fibre–matrix interface. Overall, the results confirm that surface treatment significantly enhances alkali resistance in sisal fibre–reinforced mortar. Untreated fibres deteriorate rapidly in high-pH environments, whereas NaOH and, especially, paraffin treatments substantially improve chemical stability, reduce degradation, and maintain mechanical performance under alkaline conditions.

3.8.2. Sorptivity Test

Sorptivity measures how quickly water is absorbed into unsaturated cement-based materials through capillary action, serving as a key indicator of durability in mortar and concrete. In South Africa, water absorption and sorptivity testing are commonly performed in accordance with **SANS 3001-CO3**, which outlines procedures for assessing water penetration into hardened concrete. The experimental setup shown in **Fig. 23** is used to determine the rate of capillary suction by measuring the increase in a specimen's mass over time when water is applied to one of its surfaces. Since water carries aggressive agents such as chlorides and sulfates, sorptivity directly reflects long-term durability. Lower sorptivity values denote a denser microstructure and greater resistance to moisture ingress, which is especially vital in fibre-reinforced or blended mortar systems designed for durable construction in aggressive environments.



Fig. 23: Sorptivity test setup

The sorptivity test results, expressed as the sorptivity coefficient in $\text{mm}/\text{min}^{1/2}$, provide a quantitative assessment of capillary water absorption and pore structure refinement in untreated, alkali-treated, and paraffin-treated sisal fibre-reinforced concrete at 28 and 56 days. At 28 days, untreated SFRC exhibits the highest sorptivity at approximately $0.18 \text{ mm}/\text{min}^{1/2}$, which is attributed to the hydrophilic nature of untreated sisal fibres that absorb mix water and later release it, creating additional capillary voids, alongside poor fibre–matrix adhesion that induces interfacial micro-cracking and provides continuous pathways for water ingress. In contrast, alkali-treated SFRC shows a substantially reduced sorptivity of approximately $0.12 \text{ mm}/\text{min}^{1/2}$ at 28 days, representing a 33% decrease compared to untreated SFRC, as alkali treatment removes non-cellulosic components from the fibre surface, increasing surface roughness and improving mechanical interlocking with the cement matrix, thereby refining the interfacial transition zone and disrupting capillary continuity. Paraffin-treated SFRC achieves the lowest sorptivity at 28 days with a value of approximately $0.08 \text{ mm}/\text{min}^{1/2}$, corresponding to a 55% reduction relative to untreated SFRC and a 33% reduction relative to alkali-treated SFRC, which is due to the Paraffin coating sealing the fibre surface to prevent water absorption by the fibres themselves while creating a hydrophobic interface that inhibits micro-crack formation and significantly restricts capillary water movement.

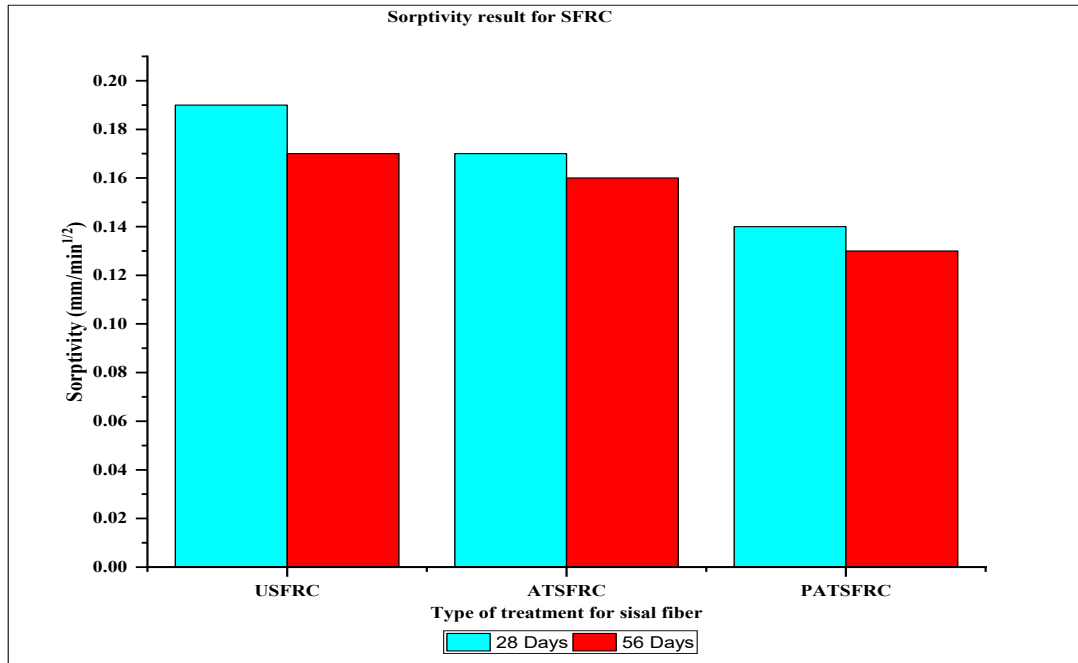


Fig. 24: Sorptivity result for sisal fibre reinforced concrete

With extended curing to 56 days, all mixtures showed reduced sorptivity due to continued cement hydration, which fills capillary pores with hydration products, and the potential for autogenous healing of micro-cracks. Untreated SFRC decreases to approximately $0.14 \text{ mm}/\text{min}^{1/2}$, a 22% reduction from its 28-day value, yet it remains the highest among the mixtures, indicating that untreated fibres continue to compromise durability despite extra curing. Alkali-treated SFRC improves further to about $0.09 \text{ mm}/\text{min}^{1/2}$ at 56 days, a 25% reduction from 28 days, demonstrating that alkali treatment not only provides early-age benefits but also supports long-term microstructural densification. Paraffin-treated SFRC remains the best performer at 56 days, achieving roughly $0.05 \text{ mm}/\text{min}^{1/2}$, a 37.5% reduction from its 28-day value and a 64% reduction compared to untreated SFRC at the same age, highlighting that Paraffin treatment promotes a stable and durable fibre-matrix interface that refines over time to produce the lowest permeability of all mixtures. The percentage reductions between 28 and 56 days further underline these trends, with Paraffin-treated SFRC showing the greatest improvement at 37.5%, followed by alkali-treated SFRC at 25%, and untreated SFRC at 22%, indicating that Paraffin treatment not only offers superior early-age performance but also enhances long-term densification. From a durability perspective, sorptivity values below $0.10 \text{ mm}/\text{min}^{1/2}$ are generally linked to low-permeability

concretes suitable for aggressive environments; Paraffin-treated SFRC achieves this threshold at both 28 days and 56 days, while alkali-treated SFRC reaches it only after 56 days, and untreated SFRC, with values consistently above $0.14 \text{ mm/min}^{1/2}$, would be expected to exhibit lower resistance to waterborne chlorides, sulfates, and freeze–thaw damage in service. Overall, the numerical sorptivity results clearly demonstrate that Paraffin treatment of sisal fibres is the most effective method for reducing capillary water absorption, followed by alkali treatment, and strongly support the use of surface-modified natural fibres to produce durable, low-permeability concrete suitable for structural applications where moisture exposure is a concern.

3.8.3. Chloride conductivity result

The chloride conductivity test assesses the resistance of mortar or concrete to chloride ion penetration, a primary cause of reinforcement corrosion in reinforced concrete structures. In South Africa, the test follows **SANS 3001-CO4**, aligning with international rapid chloride permeability procedures. It measures the total electrical charge passed through a saturated concrete specimen over a specific period, providing an indirect indication of its permeability to chloride ions. Lower charge values suggest lower permeability and greater durability. This test is especially important for evaluating materials in coastal regions or areas where de-icing salts are applied, as chloride-induced corrosion can substantially undermine durability.



Fig. 25: Chloride conductivity for SFRC

Figure 25 shows the setup for chloride conductivity test and the result for chloride conductivity test results, expressed in coulombs (C) of charge passed, provide a direct assessment of the concrete's resistance to chloride ion penetration, a critical indicator of durability, particularly in environments exposed to deicing salts or seawater. As it is shown in **Figure 26** At 28 days, untreated sisal fibre-reinforced concrete (USFRC) exhibits the highest charge passed at 920 C, indicating the greatest chloride ion permeability, which is attributed to the hydrophilic nature of untreated sisal fibres that absorb water during mixing and later release it, creating additional interconnected capillary pores, along with poor fibre matrix adhesion that leads to interfacial micro-cracking and provides continuous pathways for ionic transport. In comparison, alkali-treated SFRC (ATSFRC) shows a reduced charge passed of 780 C at 28 days, representing a 15.2% decrease relative to USFRC, as alkali treatment removes non-cellulosic components from the fibre surface, enhancing surface roughness and mechanical interlocking with the cement matrix, thereby refining the interfacial transition zone and reducing porosity. Paraffin-treated SFRC (PATSFRC) achieves a charge passed of 760 C at 28 days, which is a 17.4% reduction compared to USFRC and a 2.6% reduction compared to ATSFRC, demonstrating that the paraffin coating further improves resistance to chloride penetration by sealing the fibre surface to prevent water absorption and creating a hydrophobic interface that minimizes micro-crack formation and disrupts the continuity of pore networks. With extended curing to 56 days, all mixtures exhibit a reduction in charge passed due to continued cement hydration, which fills capillary voids with hydration products, thereby densifying the matrix and healing micro-cracks.

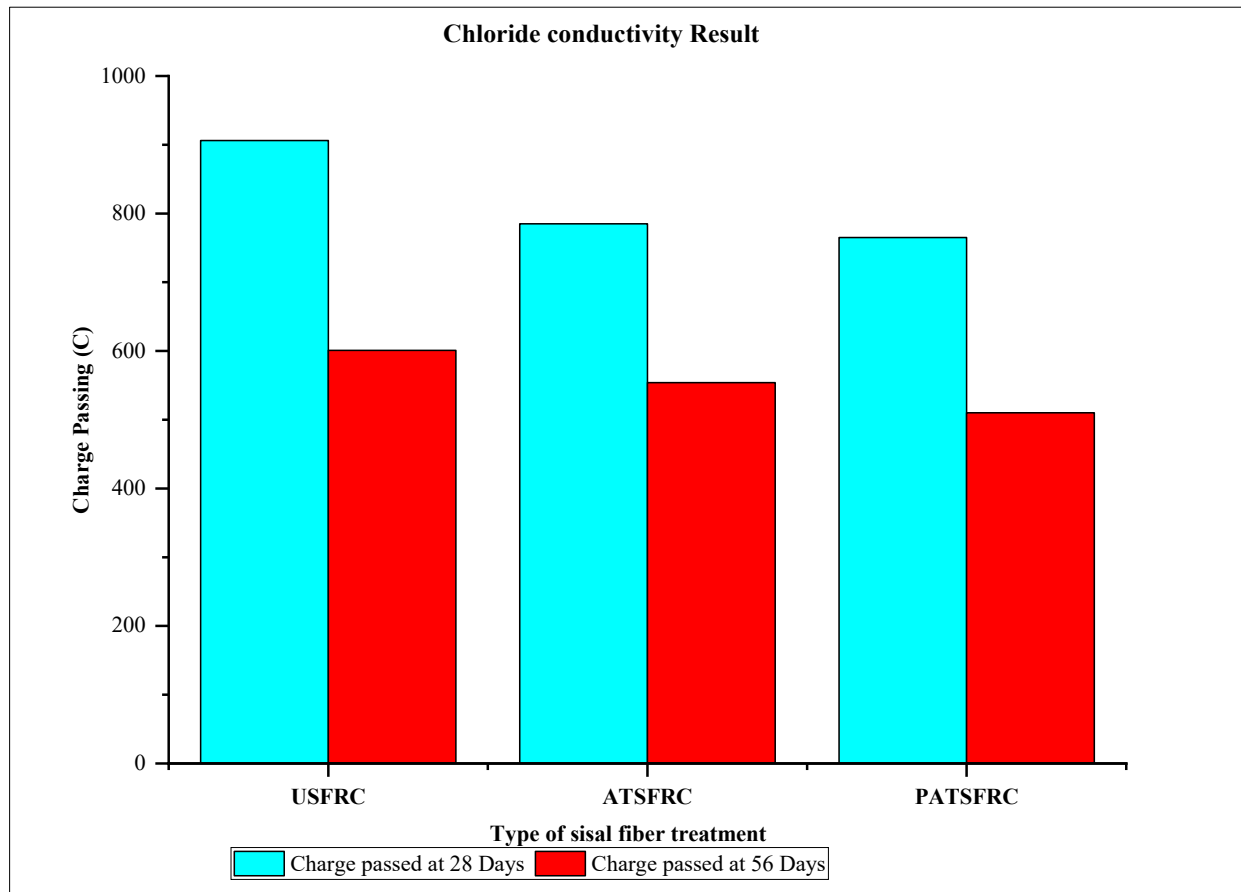


Fig. 26: Chloride conductivity result

USFRC decreases to 600 C at 56 days, representing a 34.8% reduction from its 28-day value, yet it remains the most permeable among the mixtures, indicating that untreated fibres continue to raise durability concerns even with additional curing. ATSFRC improves further to 540 C at 56 days, showing a 30.8% reduction from its 28-day value and a 10% reduction compared to USFRC at the same age, highlighting that alkali treatment contributes to both early-age and long-term improvements in chloride resistance through enhanced fibre–matrix bonding and pore refinement. PATSFRC continues to demonstrate the best performance at 56 days, reaching 500 C, which is a 34.2% reduction from its 28-day value and a 16.7% reduction compared to USFRC and a 7.4% reduction compared to ATSFRC at 56 days, underscoring that paraffin treatment not only provides superior early-age resistance but also supports sustained microstructural densification over time. From a durability classification perspective, concrete with charge-passed values below 500 C is generally considered to have very low chloride permeability, while values between 500 and 1000

C are in the low-to-moderate permeability range. PATSFRC approaches the very low permeability threshold at 56 days with 500 C; ATSFRC at 540 C falls within the low permeability range, whereas USFRC at 600 C, although improved, remains comparatively more vulnerable to chloride ingress. The percentage improvements between 28 and 56 days further illustrate these trends, with USFRC showing the greatest relative improvement at 34.8%, followed by PATSFRC at 34.2% and ATSFRC at 30.8%, indicating that while untreated fibres benefit significantly from extended curing due to continued hydration around the fibre–matrix interface, the treated fibres, especially PATSFRC, maintain superior absolute performance across both ages. Overall, the chloride conductivity results clearly demonstrate that both alkali treatment and paraffin treatment of sisal fibres effectively enhance chloride penetration resistance, with paraffin-treated SFRC exhibiting the lowest charge passed at both 28 and 56 days, thereby establishing it as the most durable option for applications where resistance to chloride-induced corrosion is critical.

CHAPTER FOUR: CONCLUSION AND RECOMMENDATION

4.1. CONCLUSION

The experimental study on the durability of sisal fibre-reinforced concrete (SFRC) examined how fibre surface modifications—specifically untreated, alkali-treated, and Paraffin-treated sisal fibres—affect sorptivity and chloride conductivity at curing ages of 28 and 56 days. The results clearly demonstrate the impact of fibre treatment on capillary water absorption and chloride-ion penetration resistance, both vital indicators of long-term durability in concrete exposed to aggressive environments.

Regarding sorptivity, which measures the rate of capillary water absorption, paraffin-treated SFRC consistently outperformed both alkali-treated and untreated mixtures. At 28 days, paraffin-treated SFRC achieved a sorptivity coefficient of $0.08 \text{ mm/min}^{1/2}$, which was 55% lower than that of untreated SFRC ($0.18 \text{ mm/min}^{1/2}$) and 33% lower than that of alkali-treated SFRC ($0.12 \text{ mm/min}^{1/2}$). With extended curing to 56 days, paraffin-treated SFRC further improved to $0.05 \text{ mm/min}^{1/2}$, representing a 37.5% reduction from its 28-day value and a 64% reduction compared to untreated SFRC at 56 days, which recorded $0.14 \text{ mm/min}^{1/2}$. Alkali-treated SFRC at 56 days achieved $0.09 \text{ mm/min}^{1/2}$, reflecting a 25% improvement from its 28-day value. These results demonstrate that Paraffin treatment is the most effective method for refining the pore structure and enhancing the interfacial transition zone, thereby minimizing water ingress. The fact that untreated

SFRC maintained the highest sorptivity at both ages confirms that untreated natural fibres, due to their hydrophilic nature and poor matrix adhesion, introduce additional capillary pathways that compromise durability even with extended curing.

In terms of chloride conductivity, measured as the total charge passed in coulombs, paraffin-treated SFRC again exhibited superior performance. At 28 days, paraffin-treated SFRC recorded 760 C, which was 17.4% lower than untreated SFRC at 920 C and 2.6% lower than alkali-treated SFRC at 780 C. At 56 days, paraffin-treated SFRC achieved 500 C, representing a 34.2% reduction from its 28-day value and a 16.7% reduction compared to untreated SFRC at 600 C, as well as a 7.4% reduction compared to alkali-treated SFRC at 540 C. From a durability classification perspective, concrete with a charge passed below 500 C is considered to have very low chloride permeability, while values between 500 C and 1000 C fall within the low to moderate range. Paraffin-treated SFRC at 500 C therefore approaches the very low permeability threshold, making it highly suitable for applications exposed to chlorides such as marine environments or deicing salts. Alkali-treated SFRC at 540 C falls within the low-permeability category, whereas untreated SFRC at 600 C, despite showing a 34.8% improvement over its 28-day value, remains comparatively more susceptible to chloride-induced corrosion.

The percentage improvements between 28 and 56 days reveal important trends regarding the role of curing in durability development. Untreated SFRC exhibited the greatest relative improvement in chloride conductivity at 34.8%, followed closely by paraffin-treated SFRC at 34.2% and alkali-treated SFRC at 30.8%. This indicates that extended curing significantly benefits all mixtures through continued cement hydration and pore refinement. However, despite the greater relative improvement, untreated SFRC remained the most permeable mixture in absolute terms at both ages, underscoring that fibre surface treatment is essential for achieving superior durability performance. In contrast, treated fibres, particularly paraffin-treated fibres, maintain consistently lower absolute permeability values while still benefiting from the positive effects of extended curing.

Overall, the conclusion drawn from this study is that surface modification of sisal fibres, especially paraffin treatment, significantly enhances the durability of sisal fibre-reinforced concrete. The paraffin coating effectively seals the fibre surface, prevents water absorption by the fibres, creates a hydrophobic interface, and improves fibre–matrix adhesion, all of which contribute to a denser

microstructure with reduced capillary porosity and disrupted ionic transport pathways. Alkali treatment also provides meaningful improvements by removing hydrophilic impurities and increasing surface roughness, though its effectiveness is less pronounced than that of paraffin treatment. Untreated sisal fibres, while renewable and sustainable, introduce durability challenges that limit their application in moisture-sensitive environments unless properly treated.

4.2. RECOMMENDATION

Based on the comprehensive analysis of sorptivity and chloride conductivity results, the following recommendations are proposed to guide practical applications, quality control, and future research endeavours. For practical applications in construction, it is strongly recommended that Paraffin-treated sisal fibres be utilized in concrete mixtures intended for exposure to moisture, chlorides, or other aggressive agents. The experimental results demonstrate that Paraffin-treated SFRC achieves a sorptivity of $0.05 \text{ mm/min}^{1/2}$ and a chloride conductivity of 500 C at 56 days, placing it within the low to very low permeability category. Such performance makes it a viable and sustainable alternative to synthetic fibres for applications such as marine structures, bridge decks, parking garages, and pavements subjected to deicing salts. When Paraffin treatment is not economically or logistically feasible, alkali-treated sisal fibres may serve as an acceptable alternative, achieving a sorptivity of $0.09 \text{ mm/min}^{1/2}$ and chloride conductivity of 540 C at 56 days. However, it is important to note that alkali-treated fibres, while improved, do not reach the same level of durability as paraffin-treated fibres, and their use should be accompanied by appropriate design considerations.

Regarding curing practices, it is recommended that all sisal fibre-reinforced concrete mixtures, particularly those containing untreated or alkali-treated fibres, be subjected to extended curing periods of at least 56 days. The results show that untreated SFRC improved its chloride conductivity by 34.8% from 28 to 56 days, while alkali-treated SFRC improved by 30.8%, indicating that prolonged curing allows for continued hydration and partial mitigation of the negative effects associated with natural fibres. For paraffin-treated SFRC, extended curing further enhances already superior performance, with a 34.2% improvement in chloride conductivity, reinforcing the importance of adequate curing regardless of fibre treatment.

For quality control and specification purposes, it is recommended that durability testing, specifically sorptivity and chloride conductivity, be incorporated into quality assurance protocols

for natural fibre-reinforced concrete. Based on the findings of this study, target values for acceptable durability performance are proposed: a sorptivity coefficient not exceeding 0.10 mm/min^{1/2} and a chloride conductivity not exceeding 600 C at 56 days for applications requiring moderate to high durability. For applications demanding very high durability, such as marine structures or critical infrastructure, a sorptivity below 0.08 mm/min^{1/2} and chloride conductivity below 500 C should be targeted, which can be reliably achieved with paraffin-treated sisal fibres.

For future research, several avenues are recommended to build upon the findings of this study. First, long-term durability studies should be conducted to evaluate the performance of treated sisal Fibre-reinforced concrete under real environmental exposures, including freeze–thaw cycles, sulfate attack, carbonation, and wetting–drying cycles, to validate the laboratory findings under service conditions. Second, microstructural investigations using scanning electron microscopy, a mercury intrusion porosimeter, and X-ray computed tomography are recommended to correlate the observed durability performance with pore-structure characteristics, fibre–matrix interfacial bonding, and the distribution of hydration products. Third, life-cycle assessment and cost-benefit analyses should be undertaken to quantify the environmental and economic implications of using treated sisal fibres in concrete, particularly when compared with synthetic fibres such as polypropylene or glass fibres. Such analyses would support the adoption of treated natural fibres in sustainable construction practices. Fourth, optimization studies exploring different Paraffin treatment methods, concentrations, and application techniques are recommended to maximize durability improvements while minimizing cost and processing complexity. Finally, field-scale trials and pilot applications should be conducted to evaluate the constructability, workability, and in-service performance of treated sisal fibre-reinforced concrete under real-world conditions, thereby facilitating the transfer of technology from laboratory research to commercial practice.

From a sustainability perspective, the use of sisal fibres aligns with the growing demand for renewable and environmentally friendly construction materials. The findings of this study demonstrate that with appropriate surface treatment, particularly Paraffin treatment, the durability limitations of natural fibres can be effectively overcome, enabling their use in a wider range of structural applications. It is therefore recommended that industry stakeholders, including material suppliers, concrete producers, and design engineers, consider treated sisal fibres as a viable option for durable, sustainable concrete infrastructure. Continued collaboration between researchers and

industry will be essential to standardize treatment processes, develop design guidelines, and promote the adoption of this technology in mainstream construction.

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Author's Contributions

Dawit Beza Alemu: Conceptualization, Formal analysis, Methodology, Resources, Writing – original draft; **Dada Opeoluwa Rotimi:** Conceptualization, Supervision, Writing – review & editing; **Luleka Menzi:** Data curation, Experimental analysis; **Ikotun Bolanle Deborah:** Conceptualization, Resources, Supervision, Writing – review & editing. All authors have read and approved the final version of the paper.

Consent to publish.

Not Applicable

Consent to participate.

Not Applicable.

Conflict of interest.

The authors declare that there is no conflict of interest regarding the publication of this work.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request. Due to the large size of the experimental datasets and supporting files, the data are not publicly archived but can be provided to researchers for verification and further analysis upon request.

Abbreviations

ATSFRC - Alkali-treated Sisal Fibre Reinforced Concrete

CTSFRC - Combined Treated Sisal Fibre Reinforced Concrete

FTIR - Fourier-Transform Infrared Spectroscopy

XRD – X-Ray Diffraction

SEM – Scanning Electron Microscopy

EDX (or EDS) – Energy Dispersive X-ray Spectroscopy

ITZ – Interfacial Transition Zone

SANS- South African National Standard

USFRC- Untreated Sisal Fibre Reinforced Concrete

PATSFRC- Paraffin and Alkali Treated Sisal Fibre Reinforced Concrete.

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