Construction and Building Materials 306 (2021) 124906

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Influence of short-term degradation on coir in natural fibre-cement composites

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ARTICLE INFO

Keywords:
Natural fibre
Bio-based material
Cement-fibre composites
Cement-fibre compatibility
Alkaline degradation
Short-term degradation
Alkaline hydrolysis
Fibre mineralisation

ABSTRACT

Natural fibres are regarded as a potential bio-based alternative to traditional fibre material used in cement-based composites. A well-known complication of natural fibres is their instability the alkaline cement environment, which causes degradation. This work presents a study of mechanical performance, microstructure, and composition of coir fibres exposed to alkalinity, focusing on short-term degradation mechanisms of degradation. Fibre performance was found to be unrelated to the alkalinity of the medium and only hemicellulose was found to readily degrade in a highly alkaline environment. It was therefore concluded that alkaline hydrolysis is not the main factor involved in natural fibre degradation.

1. Introduction

Ever since the latter half of the twentieth century, natural fibres (NF) have been receiving increased attention in academic literature as a building material. The main precursors to this interest reflect various technological as well as socio-economic developments of the late twentieth century. Firstly, as we became more informed about the carcinogenic effects of asbestos, the search for a suitable fibrous replacement commenced [1]. Alternatives were found in steel and carbon fibres, although these are relatively expensive, or synthetic and glass fibres, which have a substantial environmental footprint due to their production methods [2,3]. Natural fibres are, regarding these aspects, a more sensible alternative. Environmental awareness has simultaneously been a trend that characterised the last few decades of development in the field of building materials, with an increasing demand for sustainable building materials [4–6]. NF are inherently sustainable due to benefits such as renewability, biodegradability, low dependence on non-renewable energy sources, and low emission of pollutants [3,4,7–10]. Lastly, there have been socio-economic developments in the arid or tropical areas of Latin America and South Asia, such as migration to the urban environment [7], that sparked research interest into NF. As a highly available material which incurs very few production costs and allows use of local labour, NF are a suitable building material for low-cost housing in the impoverished rural areas [1,7,11,12].

Coir, or coconut fibre, is one NF type which may benefit tremendously from valorisation as a building material since its production rate is very high and it currently accrues mostly as a waste product [8,13]. Fibrous material such as coir may be used in building products such as thin sheets and boards for non-structural applications. Combination of NF with cement forms a NF-cement composite [14,15], which can be used to make a variety of building products such as wood-wool cement board. Composites with other binders, such as polymers, are also available for constructive or automobile applications [16]. These products are greatly enhanced by the inclusion of fibres, which help to increase flexural strength, toughness, and impact resistance [7,14]. Additionally, the resulting product provides some thermal and acoustic insulation [15]. Currently, research is still continuing towards the best practice of application of NF-cement composites and is directed mostly towards the interactions between NF and cement, several niche applications, or environmental benefits of using NF [17,18].

As a novel building material, natural fibres have brought a set of limitations and difficulties of their own, which are often the topic of scientific research. Many report the highly heterogeneous nature of the material [4] and water absorption capability, which causes swelling and shrinking and ultimately debonding of the fibres and the binder [10]. An additional disadvantage is the release of saccharides from fibres into the pore solution of a NF-cement composite during curing. These inhibit cement hydration by chelating calcium ions and through surface adsorption to cement grains, therefore preventing the formation of C-S-

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https://doi.org/10.1016/j.conbuildmat.2021.124906
Received 5 July 2021; Received in revised form 7 September 2021; Accepted 10 September 2021

H gel [19–21]. The most challenging element relates to the instability of NF in the alkaline environment of the cement matrix [1,22,23]. This is defined as an interaction of alkali attack on fibres and fibre mineralisation; the latter being greatly enhanced by the high water absorption capacity of fibres. Alkali attack may affect natural fibres on several structural levels. Whereas degradation often starts as decomposition of lignin and hemicellulose components, which are associated with the fibre surface [22], advanced degradation will affect also the fibre's structural cellulose polymer chains [1,24]. Wei and Meyer [23] provided a four-step sequential model explaining the progression of alkali attack:

1. Dissolution of lignin and, partly, hemicellulose causes exposure of holocellulose, a term for the combined occurrence of hemi (cellulose);
2. Further dissolution of hemicellulose decreases integrity and stability of the cell walls;
3. After degradation of lignin, hemicellulose, and intramolecular hydrogen bonds, the cellulose microfibrils disperse in the pore solution since no further binding remains;
4. Failure of cellulose micro-fibrils due to alkaline hydrolysis in amorphous regions.

Exposure of NF to alkalinity may also lead to positive effects, such as the removal of unnecessary surface material, which creates a rougher fibre that can provide better adhesion in a NF-based composite [2,4]. Alkaline pre-treatment (or mercerisation) is therefore a viable fibre modification method, although treatment with chemicals such as NaOH or KOH may incur high costs while these also have low recovery rates due to the formation of various by-products [25].

Bentur and Akers [24] found that there was little correlation between the degree of polymerisation of cellulose chains and the degradation of NF in composites, however, which led to the belief that another mechanism was involved. It was later found that this mechanism was a combination of the hydrophilic character of NF and the presence of soluble and alkaline calcium hydroxide (CH) in the cement matrix [1,9,26,27]. These two factors cause fibre–matrix debonding and CH crystallisation according to the following model by Claramunt et al. [9]:

1. Transversal section of fibres reduces during the first dry period due to loss of water;
2. Water dissolves CH during the next wet period which the fibres readily absorb;
3. Water is lost during the second dry period while CH precipitates.

This mechanism is named fibre mineralisation and causes embrittlement and loss of flexibility and strength in NF [22]. It is typically an effect related to long-term durability of NF-cement composites. There is some reciprocity in the two mechanisms, since transport of alkaline CH into the fibres will increase alkalinity, thus speeding up alkaline hydrolysis. Altogether, alkali attack on natural fibres is a well-studied topic and most past research focused on NF-cement composites in the later stages of curing, preferably after a number of wetting and drying cycles [1,28,29]. The logic is that durability defects, especially mineralisation, manifest at a later age; it has been found for pulp fibres, for instance, that the first damage damage of wetting and drying cycles is observable after 5 wetting and drying cycles, and fibre mineralisation fully establishes itself after 10 cycles [27]. Drawing on a conclusion from Melo Filho et al. [22], who stated that the impact of accelerated aging with wetting and drying cycles on natural fibres is more severe during the first few wetting and drying cycles, more thorough investigation may be warranted into the immediate and short-term effects of alkali attack on NF. A study into short-term degradation of NF could aid in understanding the degradation process better in its entirety, by finding out exactly how and where it begins. Moreover, since the focus on early degradation is not common in academic literature, a short-term study could cover this literature gap.

The aim of this work is therefore to characterise short-term degradation of NF in the alkaline environment of NF-cement composites, with the logic that knowing how and where degradation starts, seeking solutions to prevent it may become more effective. Understanding short-term degradation can, in that sense, help to increase the use of NF as a sustainable fibrous material in the building industry. Coconut fibre, or coir, was found to be vulnerable to the degradation mechanisms of alkaline hydrolysis and fibre mineralisation [8], while being less problematic in terms of leaching of saccharides in a cement-based composite [20]. It is therefore a suitable material to evaluate short-term degradation of NF. Coir is also an abundant material with a large application potential. For these reasons, coir will be used in this research study. Focus will lie on a real cement environment as well as a modelled alkaline environment for the first 28 days of curing and the first three wetting and drying cycles after that, in order to study the mechanisms of alkali attack and fibre mineralisation individually.

2. Methodology

2.1. Materials

Dried brown coir used in this research was supplied by Wageningen Food & Biobased Research, the Netherlands. These fibres were analysed in a previous study [20], and their chemical composition is given in Table 1. Comparing the composition to a review study on coconut fibre properties [16], shows that the coir used in this research is enriched in hemicellulose, with 37% of hemicellulose compared to 0.15–15% which is more common. Lignin content is slightly lower at 22% than commonly found (30–46%).

Coir can be seen as a collection of fibre cells covered and protected by a surface layer, forming a circular fibre, see Fig. 1a. The surface layer serves as a protective barrier to the structural fibre cells by covering them with a layer of lignin, oils, and wax, see Fig. 1b. The surface is covered in silica bodies, which are spherical and spiked protrusions of silica [30] also known as phytoliths. Before use, the coir was brushed to homogenise orientation, washed in lukewarm water, and dried in an oven at 60 °C.

In terms of physical properties, a review study found that coir fibres may have a density varying from 1.1 to 1.5 g/cm^3 and show water absorption of 10 to 180% of their original weight [16]. Due to the scope of this study, physical properties of the coir fibres used in this research were not investigated. Mechanical properties of raw fibres were studied, however, and these will be discussed in later sections.

Cement used in this research is CEM I 42.5 N Portland cement originating from ENCI in the Netherlands. Bulk chemical composition was analysed with X-ray fluorescence (XRF) spectrometry, to study the elemental composition making up the clinker phases. The bulk chemical composition is presented in Table 2.

Alkaline solutions were used to model the effect of alkalinity alone on coconut fibres, i.e. in absence of fibre mineralisation. In the literature, NaOH or $Ca(OH)_2$ are often used to modify natural fibres, with a slight preference to use NaOH for alkaline pre-treatment [8,31,32] and $Ca(OH)_2$ to model the effect of the cement matrix [1,7]. In this research, six solutions of NaOH of increasing pH are used, to exclude the possibility of lime crystallisation, and one solution of $Ca(OH)_2$ for comparison. The pH range investigated was based on the typical pH value of fresh cement mortar for the high end [33,34], and lower pH values to

Table 1
Chemical composition of coir measured by HPAEC after H_2SO_4 hydrolysis, from [20].

	Concentration [%]				
	Cellulose	Hemicellulose	Lignin	Ash	Extractive
Coir	36.6%	37.0%	22.2%	1.9%	4.2%

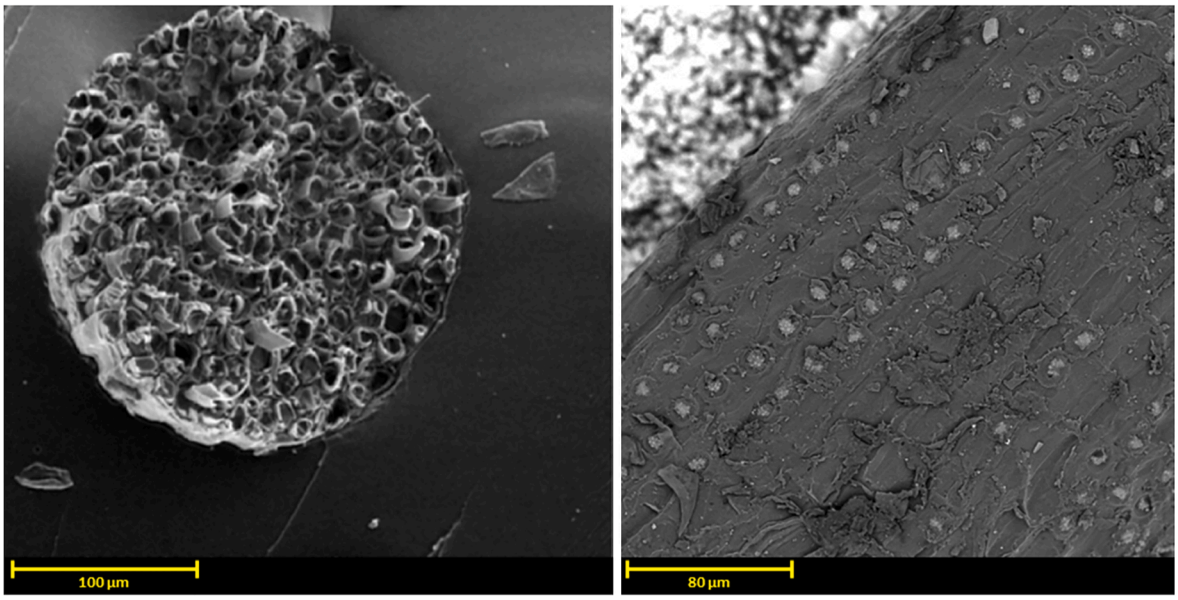

Fig. 1. a) SEM micrograph of coir fibre cross-section [16]; b) SEM micrograph of coir fibre surface (own work).

Table 2
Chemical composition of ordinary Portland cement CEM I 42.5 N used in this study.

	Oxide composition [wt.%]									
	CaO	SiO_2	Fe_2O_3	SO_3	Al_2O_3	K_2O	MgO	P_2O_5	TiO_2	Others
Portland cement	76.3%	9.8%	4.8%	3.6%	2.0%	0.9%	0.8%	0.5%	0.4%	0.7%

account for the possibility to lower the matrix pH by blending pozzolanic materials into the cement [26]. Table 3 shows the label name, base, and pH of each solution.

2.2. Sample preparation

To evaluate the influence of alkalinity on fibres, coir samples were fully immersed in the premade alkaline solutions and stored in air-tight containers. A small batch was retrieved once a week for five weeks, washed with water, and dried in a ventilated oven at 60 °C. These samples were labelled with S and a number from 1 to 7 to specify the solution and using a second number to specify the weeks of soaking. For example: S1.1 are fibres from solution 1 soaked for 1 week.

To evaluate the influence of the cement environment (which exposes the fibres to alkalinity and mineralisation), a simple cement paste was mixed to make a model NF-cement composite. Cement and tap water were mixed at a water-to-binder ratio of 0.4 in a bench-mounted mixer, first at low speed for 1 min, and then at high speed for 2 min. Finally, cement paste was cast in polystyrene moulds (40 × 40 × 160 mm). Fibres were immersed into the matrix in a different way for each analysis method, as will be clarified in the following section.

Table 3
Set of used alkaline solutions and their details.

	Characteristics	
Sample	Base	pH
S1	NaOH	9.0
S2	NaOH	10.0
S3	NaOH	11.0
S4	NaOH	12.0
S5	NaOH	12.5
S6	NaOH	13.0
S7	$Ca(OH)_2$	12.0

Moulds were wrapped in PVC foil, demoulded after 24 h, and then left to cure in a controlled humid environment (RH > 95%, T = 21 °C) for 28 days. After curing, some specimens were also exposed to wetting and drying cycles to accelerate aging, a method which has been repeatedly used in academic literature [1,6,19]. A wetting and drying cycle was chosen that would assure full wetting and drying after each step:

- Wetting: 168 h (7 days) immersed in tap water at laboratory room temperature (21 °C);
- Drying: 72 h (4 days) in a ventilated oven at elevated temperature (60 °C);
- Cooling: 3 h of cooling at laboratory room temperature (21 °C) to prevent heat shock.

After preparation, fibres were released from the cement matrix in three steps. First, the matrix was gently crushed to loosen the individual fibres from the composite block; next the fibres were pulling out from the remaining parts of cement; lastly, to remove as much excess cement as possible, fibres were washed using tap water, although some cement particles inevitable remained. Fibres were then dried at 60 °C for about 6 h and labelled C to indicate exposure to cement and with a number to indicate the amount of wetting and drying cycles. For example: C1 are cement composite fibres exposed to 1 cycle. Additionally, unexposed reference fibres were prepared and labelled R.

2.3. Sample analysis

2.3.1. Mechanical performance analysis by tensile strength

Tensile strength of single-strand fibres was tested to evaluate degradation of fibres with specific attention to performance. To this end, fibres were cast into cement by placing two extra perforated walls in the mould, see Fig. 2a, through which fibres of 150 mm length were pulled.

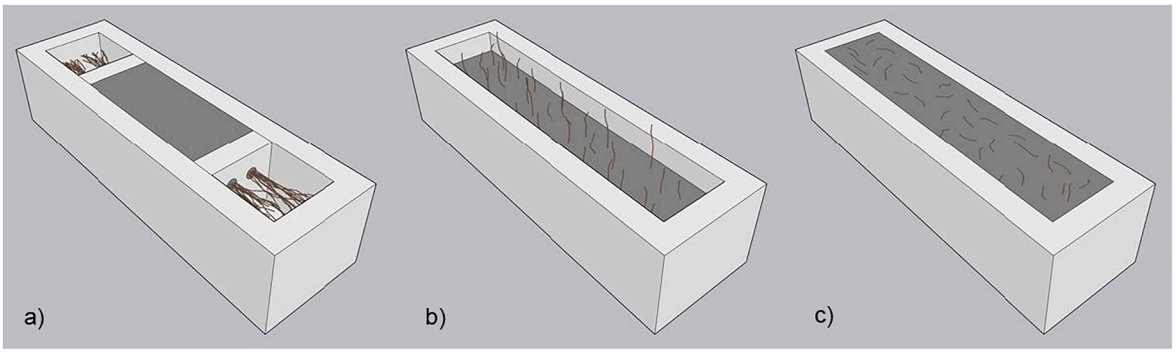

Fig. 2. Visualisation of embedding methods of fibres into cement, a) fibres prepared for tensile strength testing, b) fibres prepared for microstructural analysis, and c) fibres prepared for compositional analyses.

Cement paste was then poured into the mould section in between the extra walls, to result in fibres which were easier to remove from the matrix and were only affected by cement in the middle of their length.

Strength tests were performed on coir kept in either alkaline solutions or cement on an Instron 2967 testing bench, following ASTM D 3822 guidelines [35]. First, average fibre diameter was determined using a calliper at various points along the fibre. Next, fibres were tested with a fibre gauge length (space between apparatus clamps) manually set to 50 mm and a fixed loading rate of 10 mm/min. To account for fibre heterogeneity, twelve fibres per specimen type were tested. After recording elongation and ultimate load data, results were combined with the average fibre diameter to compute the ultimate tensile strength and Young's modulus.

2.3.2. Microstructural analysis by SEM

Microstructural analysis was performed by *scanning electron microscopy (SEM)* to characterise the extent of surface degradation. To this end, fibres cut to 60 mm were placed upright into moulds filled halfway with cement paste, so to have a surface affected by cement and a surface unaffected, see Fig. 2b.

Samples of coir from alkaline solutions and cement were prepared by cutting a short piece from two fibres per specimen type, which were attached to an aluminium stub using carbon tape, and then coated with a thin layer of gold for conductivity. A Phenom Pro X electron microscope was used under an accelerating voltage of 10 kV and all micrographs were taken with a backscattered electrons detector. Indicators of degradation were derived from literature and these include removal of fats, waxes, and silica bodies, removal of external lignin, and damage of fibre cells [6,8,29,30].

2.3.3. Compositional analysis by TGA and FTIR

A compositional analysis was performed to identify degradation of the fibres' main structural components: lignin, hemicellulose, and cellulose. To prepare them for the compositional analysis, fibres cut to 20 mm were immersed into the cement matrix by adding them to the cement paste in the mixer, mixing for 1 additional minute at high speed to homogenise, and finally casting the paste into the moulds, see Fig. 2c.

Firstly, *thermogravimetric analysis (TGA)* was conducted on coir from alkaline solutions and cement: 10 to 30 mg of fine fibre grains (cut to 1 mm) were transferred into alumina crucibles and heated in a Netzsch STA449 F1 Jupiter apparatus under a nitrogen atmosphere with a flow rate of 20 mL/min from room temperature to 800 °C at a constant heating rate of 10 °C/min. Analysis of mass loss data was performed using Netzsch Proteus Thermal Analysis software.

Mass loss analysis was based on the work of Martin et al. [5], who split the mass loss curve into three sections: initial loss, active pyrolysis, and char region. The first region is mostly associated with loss of water, whereas in active pyrolysis most of the (hemi)cellulosic material is burned. The mass loss rate curve shows two separate maxima in the active pyrolysis region, the first of which is related to hemicellulose and the second to cellulose [6], which allows splitting the mass loss even further into a semi-quantification of hemicellulose and cellulose. This method yields semi-quantitative results at best but may give an indication of the relative content of both components. The char region finally is associated with residual matter and lignin, although lignin loses mass steadily across all regions [5], which makes quantification difficult. For this reason, lignin was not considered in TGA.

Secondly, *Fourier Transform Infrared (FTIR) spectroscopy* was performed on fibre grains to supplement the compositional analysis. Fibres cut to approximately 5 mm were placed on the FTIR lens of a PerkinElmer FTIR spectrometer Frontier operated at ATR mode over a wavenumber range of 4000 to 500 cm^{-1} with a resolution of 4 cm^{-1} and results were averaged over 15 scans.

FTIR was performed in reflectance mode using integral fibres instead of powdered fibres, and therefore qualifies only as a surface analysis with semi-quantitative results. Results were analysed based on the work of several authors and several absorption peaks were chosen for the analysis which were unambiguously related to one of the components of lignocellulosic fibres. Of the broad wavenumber spectrum evaluated, the three peaks listed in Table 4 (all in the 1800 to 1500 cm^{-1} segment) were analysed. In the qualitative analysis presence or absence of IR absorption in these regions indicated the presence or absence of the related compound.

3. Results

3.1. Single-fibre tensile strength tests

Tensile strength results of fibres from the alkaline solutions soaked for either 1 or 5 weeks, including the range of the untreated reference fibres, are presented in Fig. 3. Ultimate tensile strength of untreated reference fibres is lower than the average of coconut fibres according to a review study by Adeniyi et al. [16], who found that this property ranges from 105 to 593 MPa. Reference results from the current study are in the range of 86–175 MPa, indicating that these fibres are likely of a generally weaker type of coir.

Stress–strain curves of reference fibres showed a knuckle pattern with three phases: a short initial elastic deformation, a prolonged plastic deformation, and finally breakage. This deformation pattern and failure mechanism has previously been found for coir [39]. The plastic phase is

Table 4
FTIR absorption peaks assigned as indicators of constituents of coir, adapted from [4,36–38].

Wavenumber [cm^{-1}]	Functional group	Polymer
1730–1740	Ketone/aldehyde C = O stretching	Mostly hemicellulose
1595	Aromatic ring vibration	Lignin only
1500	Aromatic ring vibration	Lignin only

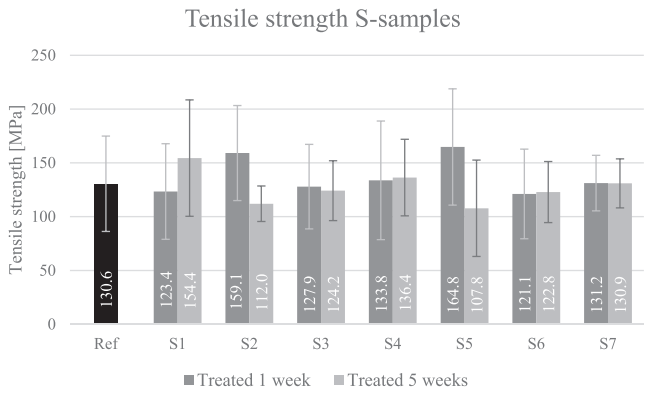

Fig. 3. Results for ultimate tensile strength of coir from alkaline solutions, reference range in green.

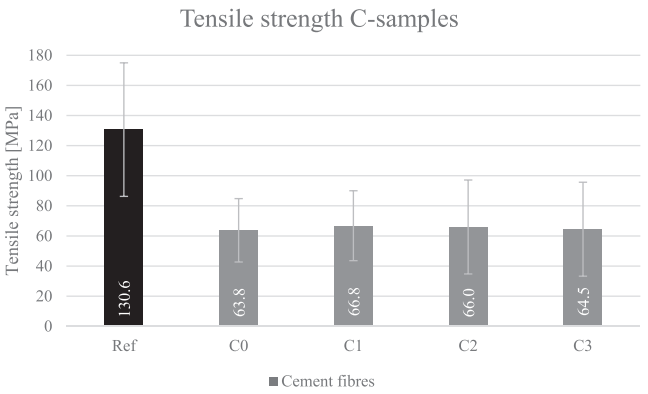

Fig. 5. Results for ultimate tensile strength of coir from cement, range of reference in green.

typical for ductile materials such as natural fibres, and a considerable elongation (around 20%) is achieved before breakage. In all reference fibres, the fracture strength is also the ultimate tensile strength.

Results for fibres from alkaline solutions fluctuate heavily, due to fibre heterogeneity, without a visible trend in the progression of results after increased soaking duration or increased pH. The absence of such a trend indicates that the tensile strength stays stable and does not deteriorate due to the alkalinity of the solutions. Fibres soaked in the most alkaline solution (S6 with pH = 13) perform nearly as well as the reference fibres, suggesting that alkaline hydrolysis is not occurring within the fibres. Also, the choice of the base does not influence the results, since S4 with NaOH and S7 with $Ca(OH)_2$ yield very similar test results, both at a pH of 12.

Young's modulus (E) data for fibres from alkaline solutions are presented in Fig. 4. For this property the reference results agree well with average values found in the review paper of Adeniyi et al. [16], who state E is in the range of 2–8 GPa. The results for this property are similar to those for ultimate tensile strength, in the sense that E does not increase or decrease upon exposure to alkalinity. Longer exposure does generally lead to lower values, however, which is apparent when comparing week 1 to week 5 data. Loss of elasticity may be ascribed to dissolution of any of the three main components of natural fibre, cellulose, hemicellulose, or lignin, and also to a decrease of density in general [40–42]. Since the effect is seen in treated fibres regardless of the solution they were in, results indicate that long soaking time and contact with water may be of larger influence than the alkalinity of the solution, and may cause, for instance, a decrease of density.

Ultimate load data for fibres retrieved from the cement matrix were analysed similarly to those from the alkaline solutions and results are presented in Fig. 5 for ultimate tensile strength. Contrary to the fibres from the solutions, those retrieved from cement show a very pronounced effect on ultimate tensile strength: a reduction of 50% is the norm, regardless of the amount of wetting and drying cycles to which the samples have been exposed. This result compares well to Juárez et al. [7], who stated that fibres exposed to the high humidity of a cement matrix may lose up to 50% of their strength.

Results for Young's modulus are presented in Fig. 6. The Young's modulus, contrary to the tensile strength, did not decrease due to contact with the cement matrix and remains within the reference range even after several wetting and drying cycles. Since the Young's modulus may be considered an intrinsic material property [43], the results suggest that cement does not cause intrinsic changes in the fibres, and degradation must therefore be of extrinsic nature, governed by surface flaws. Since degradation is indeed experienced as loss of tensile strength, this means that surface flaws are causing degradation, or in other words, cement is causing degradation at the surface level.

3.2. Microstructural analysis

With the overall mechanical performance of the fibres characterised, the microstructural surface analysis may provide some visual information relating to chosen degradation indicators, which may be correlated to persistence or deterioration of tensile properties. Three main degradation indicators were chosen, which are removal surface material (impurities such as fats, oils, and waxes, but also lignin and hemicellulose), removal of silica bodies, and damage of fibre cells, because these three were easiest to establish visually. An example of removal of surface impurities and silica bodies would be Fig. 7c, whereas damage of fibre cells is shown in Fig. 7b.

Untreated reference fibres (see Fig. 7a) showed no sign of surface layer removal or fibre cell damage, although minor removal of silica

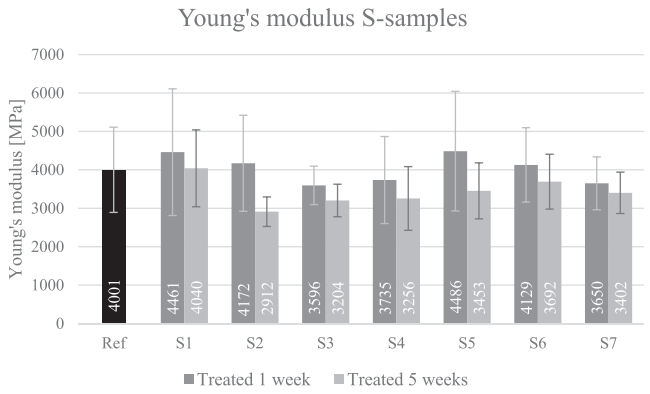

Fig. 4. Results for the Young's modulus of coir from alkaline solutions, range of reference in green.

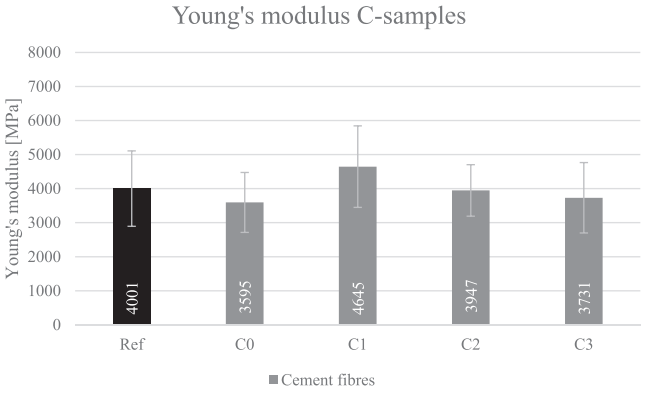

Fig. 6. Results for the Young's modulus of coir from cement, range of reference in green.

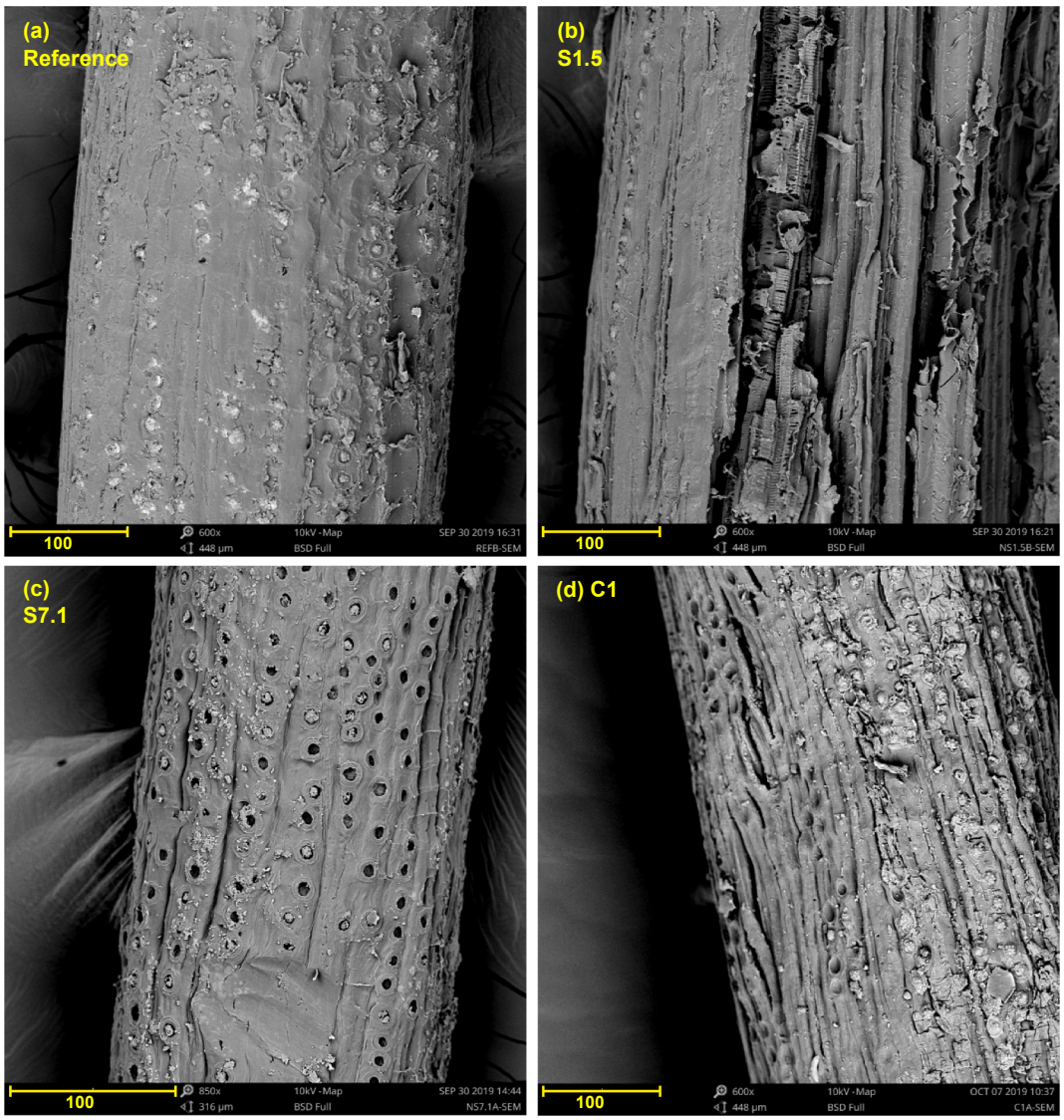

Fig. 7. a) SEM micrograph of an untreated reference fibre; b) SEM micrograph of a fibre treated for 1 week for a NaOH solution of pH 9; c) SEM micrograph of a fibre treated for 5 weeks with a $Ca(OH)_2$ solution of pH 12; d) SEM micrograph of a fibre from a cement matrix exposed to 1 wetting and drying cycle.

bodies was visible, which possibly occurs naturally or may be caused by the pre-treatment step of washing the fibres with lukewarm water.

Fibres treated with increasingly alkaline solutions showed that increasingly more silica bodies were removed and that the surface layer became thinner, evidenced by the grooved surface which is revealed once the underlining fibre cells become visible. It is interesting to note that the solution with $Ca(OH)_2$ at pH 12, Fig. 7c, showed very pronounced effects, even more than the NaOH solutions of pH 12.5 and 13, showing that there are some differences in how $Ca(OH)_2$ and NaOH affect the coconut fibres.

Mechanical testing was performed on fibres soaked for either 1 or 5 weeks, SEM analysis, on the other hand, was performed at a weekly interval from 1 to 5 weeks. Consecutive results after 1 week of soaking showed little progression of the degradation effects which established in fibres during the first week. These results suggest that alkaline degradation or modification is a process which establishes itself on the short term.

To illustrate this further, Ng et al. [32] found that alkaline solutions were capable of dissolving 36–44% of total hemicellulose from coir within 6 h, depending on the basicity of the solution. In another SEM study on alkaline treatment of coir, Prasad et al. [44] found that within 72 h, the cross-section of coir had visibly changed to a more compacted fibre with thicker cell walls and a thinner lacuna, while the fibre's surface layer had become thinner, due to removal of surface impurities, and silica bodies were removed.

In this study, removal of surface impurities and silica bodies highly correlated with increasing pH, but there was no correlation whatsoever between damage of fibre cells (Fig. 7b) and increasing pH. This defect occurred randomly across fibres of all solutions and ages. Therefore, it appears that fibre cell damage is related to mechanical damage that was inflicted prior to alkaline treatment. Mechanical damage may explain why the tensile strength of fibres in this study is slightly lower than commonly experienced with coir.

Fibres pulled from the cement matrix showed high surface layer removal; the specimen in Fig. 7d is slightly covered with hydration products but shows a thin surface layer, evident from the highly jagged surface. These fibres generally showed less removal of silica bodies than was the case for fibres from alkaline solutions; this may be explained by the absence of excessive water to solubilise the silica bodies which was present in the solutions. Samples from series C2 and C3 showed increased removal of silica bodies, which indicate that longer contact with water is indeed a prerequisite from silica body removal.

3.3. Compositional analysis

The surface layer, which has been characterised in the microstructural study, consists next to oils, fats, and waxes also of lignin and hemicellulose, so removing this layer results in removal of these components. This can be studied in a compositional analysis which may corroborate results so far, in addition to revealing how the cement environment affects the fibres *below* the surface. Changes in chemical composition of the fibres may also help to explain the persistence or loss of mechanical performance in fibres treated with alkaline solutions or cement, respectively.

3.3.1. Thermogravimetric analysis

The method of Martin et al. [5] has resulted in the division of the TGA temperature range visible in Fig. 8, where the initial loss region ends at the mass loss rate maximum between 150 and 200 °C and the active pyrolysis region ends at 400 °C where mass loss rate stabilises again. Active pyrolysis is split into hemicellulose and cellulose mass loss by the mass loss maximum ("shoulder peak") around 300 °C. Mass loss results divided into the four sections according to this method are listed in Table 5 for the fibres from alkaline solutions.

The fibres soaked in alkaline solutions show much less mass loss than the untreated reference, typically about 75% for solutions with low basicity and 70% for solutions with high basicity, compared to 85% for the reference. This suggests that material has already been removed from the treated fibres, leaving less material for combustion than originally present. The high char loss shows that the reference fibres require more time to burn, which may be explained by the presence of an intact surface layer in the reference fibres, which is removed after soaking and exposure to (even moderate) alkalinity.

With increasing alkalinity, however, another effect establishes, which is the increased absence of hemicellulose evidenced by the disappearing mass loss rate maximum pertaining to this component. This shows that the first and second steps of Wei and Meyer's alkaline hydrolysis model [23] indeed occur in these fibres.

Mass loss results for the fibres from cement are listed in Table 6. Hemicellulose is absent in all fibres which have come in contact with cement, suggesting again that the initial steps of alkaline hydrolysis have occurred. These fibres show slightly higher initial and char mass losses, which may be explained by the presence of cement hydration products of which C-S-H gel and ettringite will dehydrate in the initial region and calcium carbonate may decompose in the char region [45].

The effect of additional wetting and drying cycles is minimal, except for a slightly decreasing trend in initial mass loss and increasing trend in cellulose region loss. This may suggest that due to wetting and drying, cellulose becomes slightly more vulnerable to decomposition.

With results from both sample types reviewed, it appears that hemicellulose readily degrades in an alkaline environment, starting at a

Table 5
Mass loss results for the samples from alkaline solutions, categorised per region. Hemicellulose is abbreviated as Hemicell.

Samples	Initial loss	Active pyrolysis			Char region	
	Mass loss	Mass loss	*Hemicell.*	*Cellulose*	Mass loss	Total mass loss
S1.1	2%	59%	*20%*	*40%*	11%	72%
S1.5	6%	58%	*23%*	*35%*	11%	75%
S2.1	4%	59%	*21%*	*38%*	11%	74%
S2.5	6%	57%	*25%*	*32%*	18%	81%
S3.1	3%	57%	*21%*	*36%*	11%	72%
S3.5	6%	56%	*23%*	*33%*	12%	75%
S4.1	3%	56%	*19%*	*37%*	10%	69%
S4.5	6%	54%	*28%*	*26%*	12%	72%
S5.1	2%	52%	*0%*	*52%*	10%	64%
S5.5	3%	52%	*0%*	*52%*	10%	66%
S6.1	6%	51%	*0%*	*51%*	11%	68%
S6.5	8%	51%	*0%*	*51%*	14%	73%
S7.1	3%	60%	*21%*	*39%*	11%	75%
S7.5	6%	60%	*21%*	*40%*	12%	78%
Reference	7%	55%	*30%*	*25%*	23%	85%

Table 6
Mass loss results for the samples from cement, categorised per region. Hemicellulose is abbreviated as Hemicell.

Samples	Initial loss	Active pyrolysis			Char region	
	Mass loss	Mass loss	*Hemicell.*	*Cellulose*	Mass loss	Total mass loss
C0	12%	42%	*0%*	*42%*	14%	69%
C1	10%	45%	*0%*	*45%*	17%	72%
C2	8%	46%	*0%*	*46%*	14%	69%
C3	8%	47%	*0%*	*47%*	14%	69%
Reference	7%	55%	*30%*	*25%*	23%	85%

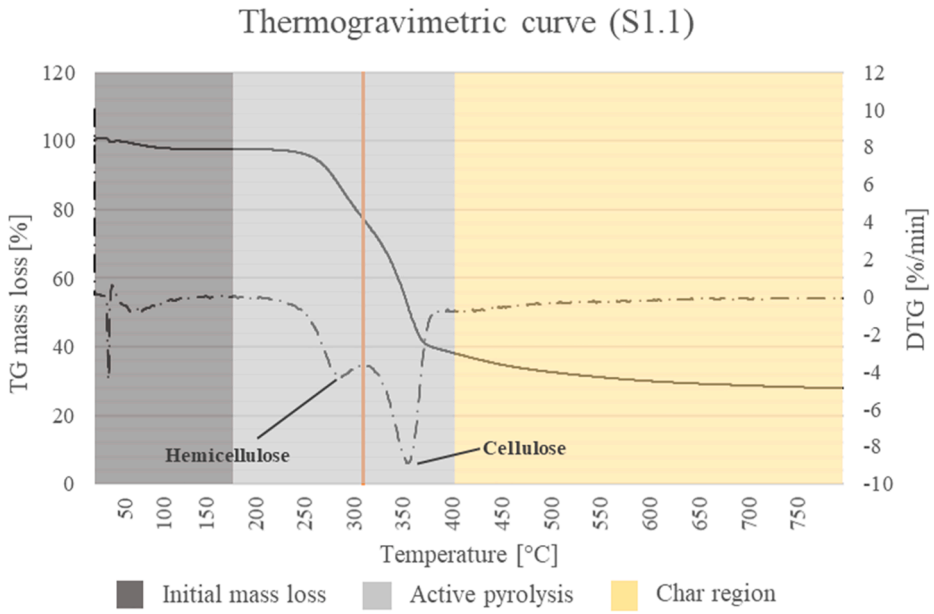

Fig. 8. Example thermogravimetric curve visualising the division of the curve into regions, sample: S1 week 1.

pH of 12.5 (as in S5). This criterion is certainly met by fresh cement paste, which may have a pH of about 13 [33]. Therefore, all samples exposed to cement show absence of hemicellulose. This finding shows that the solutions S5 and S6 are able to model the chemical changes of coir in cement, although the solutions are unable to fully reflect the changes in tensile properties, as found before.

3.3.2. *Fourier Transform Infrared spectroscopy*

Since characterisation of lignin is difficult with thermal analysis, FTIR surface analysis was additionally performed to analyse whether lignin is removed from degrading fibres. FTIR results for the fibres from alkaline solutions are presented in Fig. 9c and d. These may be compared to the untreated reference in Fig. 9a, which is based on four separate measurements (R1–R4). Results for the S1 and S6 fibres are shown because these represent lowest and highest pH. Since the method is used qualitatively, it is only possible to judge removal of components by the presence or absence of their characteristic absorption peaks.

The reference and S1 samples show clear transmittance minima for all of the three investigated absorption peaks, signifying the presence of both lignin and hemicellulose. The S6 samples show, on the other hand, no transmittance dip for the hemicellulose indicator, suggesting that hemicellulose has been removed. Absence of hemicellulose, in fact, occurs first for the solutions S4 and S7 (both of pH 12), but only slightly, and increases going on to solutions S5 and S6. In accordance with TGA results, the degradation of hemicellulose occurs already in the first week and remains stable thereafter. This also agrees with previous research which found that hemicellulose largely dissolves over the course of several hours upon contact with alkaline solutions [32].

Results for the fibres from cement are presented in Fig. 9b. The similarity to the fibres exposed to the highly alkaline solution (Fig. 9d) is large: hemicellulose appears absent in all fibres which were treated with cement, while the lignin content is barely affected. The results remain similar after each consecutive wetting and drying cycle, which shows that hemicellulose degradation is not related to the fibre mineralisation process but only to alkaline hydrolysis, while also showing that wetting and drying does not affect lignin.

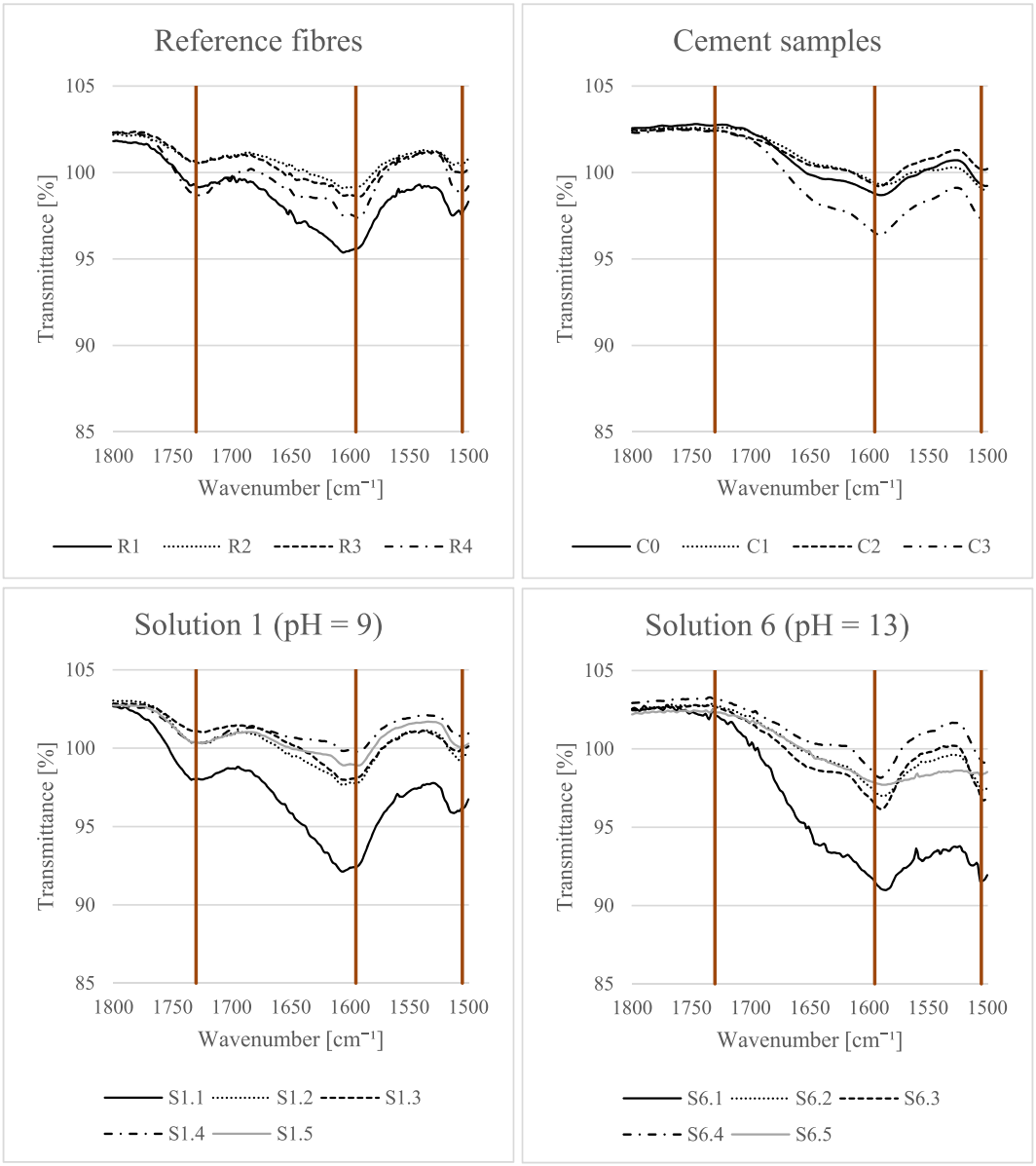

Fig. 9. a) FTIR spectra for four untreated reference fibres; b) FTIR spectra for the fibres from the cement matrix after varying number of wetting and drying cycles; c) FTIR spectra for the fibres treated with a NaOH solution of pH 9; d) FTIR spectra for the fibres treated with a NaOH solution of pH 13.

4. Discussion

Results for fibres soaked in an alkaline solution suggest that the degree of alkalinity does not influence tensile properties. This is contrary to results from some previous studies on alkaline pre-treatment of NF, which is typically done to enhance mechanical properties [8,32,44,46]. A critical comparison to other studies shows, however, that the pH level and the soaking time do influence the outcome of the treatment.

Santos et al. [36] applied a $Ca(OH)_2$ solution with a pH of approximately 13.3 and found that properties were slightly improved. Gu [8], on the other hand, used a set of NaOH solutions with a pH range of 13.7 to 14.4 to find that the two least alkaline solutions only deteriorated properties by 5% compared to the reference, whereas the highly alkaline solutions affected fibres strongly. Results from the current study fit into this pattern, and it may be deduced that NF are relatively stable up to a pH of 14, after which alkaline hydrolysis will play an increasingly larger role in deterioration.

This does not explain, however, why certain studies find that alkaline pre-treatment increases tensile properties, which was not observed in the current study. It was previously found that soaking up to 3 or 4 days may increase tensile properties considerably, due to physical alteration of the fibre cells, e.g. closure of lacunae and thickening of cell walls, whereas prolonged soaking (8 days) again decreases tensile properties due to dissolution of hemicellulose and lignin [44], which are the first steps of alkaline hydrolysis [23]. From these results combined it seems that both physical and chemical alterations lead to smaller fibre diameter, because lacunae close and surface lignin and hemicellulose are removed, while the structural backbone (cellulose) remains intact. All this allows the fibre to withstand higher stress, because it can endure the same load at a smaller diameter, leading to higher tensile strength after some contact with an alkaline solution.

Prolonged soaking could lead to lignin and hemicellulose removal to such an extent, that the fibre is affected structurally, thus offsetting any positive effects of initial alkaline treatment. Results after 7 days of soaking are almost identical to the untreated reference, which corresponds well with these findings of Prasad et al. [44] after 8 days. This could indicate that prolonged soaking (7–8 days) indeed offsets initial enhancement of properties (within 3–4 days). Alternatively, absence of property deterioration may also indicate that the alkalinity of the solution is not high enough to inflict any damage at all.

Compared to the fibres from alkaline solutions, those removed from the cement matrix showed a much larger decrease in tensile strength. Although the main effects deteriorating the fibre *in* the matrix are alkaline hydrolysis and fibre mineralisation, there is another factor influencing the results, which is the chance that upon removal from cement, fibres surface may have become damaged. Such surface flaws could easily reduce tensile strength through the introduction of local weaknesses. This concern was also expressed in the literature by Wei and Meyer [6], who proposed an alternative method for embedding NF into a cement matrix to avoid mechanically damaging them when pulled out.

Degradation of fibres from cement did not increase over the first three wetting and drying cycles, and, moreover, tensile strength was affected more so than the fibres' intrinsic Young's modulus. For these two reasons, it is likely that, in the current study, fibres suffered mechanical damage while being removed from the matrix rather than damage through fibre mineralisation. The absence of fibre mineralisation cannot be fully ruled out, however.

The microstructural study of visual degradation allowed further comparison of fibres exposed to cement or alkaline solutions. To this end, we may compare the SEM micrographs in Fig. 7c and d, which show fibres exposed to a highly alkaline solution and those exposed to cement, respectively. There are signs of surface layer removal in both, although slightly more pronounced in the latter, but otherwise the fibres are rather similar. Any strong precursors to deterioration of tensile strength are absent in fibres from cement. This gives further support to the idea that these fibres were damaged when removed from the matrix, rather than damaged by fibre mineralisation.

Another observation of the microstructural study, although unrelated to the fibres' tensile properties, is that the surface layer can be thoroughly modified by alkaline treatment. The removal of the waxy surface layer exposes the fibre cells underneath, yielding a larger and cleaner surface for fibre–matrix adhesion in NF-based composites [8,32,38,46]. Removal of silica bodies has furthermore been hypothesised to cause better adhesion, for similar reasons [44,47]. Microstructural results therefore show that an alkaline pre-treatment could be helpful in attaining a surface layer that provides a better interfacial transition zone.

The compositional analysis showed that hemicellulose is the fibre component which readily degrades in the alkaline environment of cement, whereas lignin and cellulose are persistent, for pH levels ranging from 12.5 up to 14. This explains why consequences of the alkaline environment on performance are minor: structural cellulose remains unaffected while alkaline hydrolysis is limited to a purifying effect of the surface layer, which is in fact positive rather than detrimental.

Microstructural analysis showed that surface layer refinement occurred in all treated samples, whereas hemicellulose is only absent in fibres treated at high pH. This leads to believe that soaking fibres in solutions of low alkalinity already removes waxes and oils from the surface, whereas as alkalinity increases, hemicellulose will be decomposed all throughout the fibre.

Findings for hemicellulose degradation agree with the hydrolysis model of Wei and Meyer [23], but the findings on lignin do not, nor is the stage of cellulose degradation reached, as is suggested by the unchanged performance in tensile strength tests of the fibres, even when treated at high pH. This suggests that the pH conditions in curing cement are not severe enough to cause cellulose degradation, a positive finding for the use of natural fibres in NF-cement composites.

Results found for lignin in this study do not correspond well with established literature. The hydrolysis model of Wei and Meyer [23] was developed, however, for sisal fibre, and the authors had previously already found that lignin is easily removed from this type of fibre [2], whereas it is more persistent in coir [32]. This can be explained by the low content of lignin in sisal, reported to be 7–8% [48], whereas coir has a far higher lignin content, approximately 22% for fibres used in this research and typically even higher, up to 30–45% [16,28]; for sisal it will therefore be easier to remove all lignin than for coir.

An alternative explanation can be based on a microstructural comparison of sisal and coir, see Fig. 10, which shows that untreated coconut fibres have a much smoother surface layer than sisal fibres, and the latter therefore has a much higher surface area. Since the density and diameter of both fibre types are usually in the same range [49], it may be concluded that lignin degrades faster in sisal than coir, because coir has a more efficient surface layer.

Future work could include an in-depth quantitative analysis to show in more detail whether and to which extent lignin in coir is decomposed in the cement environment. Chemical extraction methods could be suitable for this purpose and have previously been successfully attempted for natural fibre [4,32].

5. Conclusion

This work aimed to obtain a better understanding of short-term degradation of coconut fibre in the alkaline cement environment. The various mechanisms involved in degradation were studied in a real cement environment and a modelled environment using alkaline solutions.

Combination of mechanical performance, microstructural, and compositional analyses showed that performance is not reduced by high alkalinity in the modelled environment. In fact, alkaline treatment could enhance fibre-cement bonding by increasing surface roughness due to the removal of the smooth surface layer of waxes and oils, without

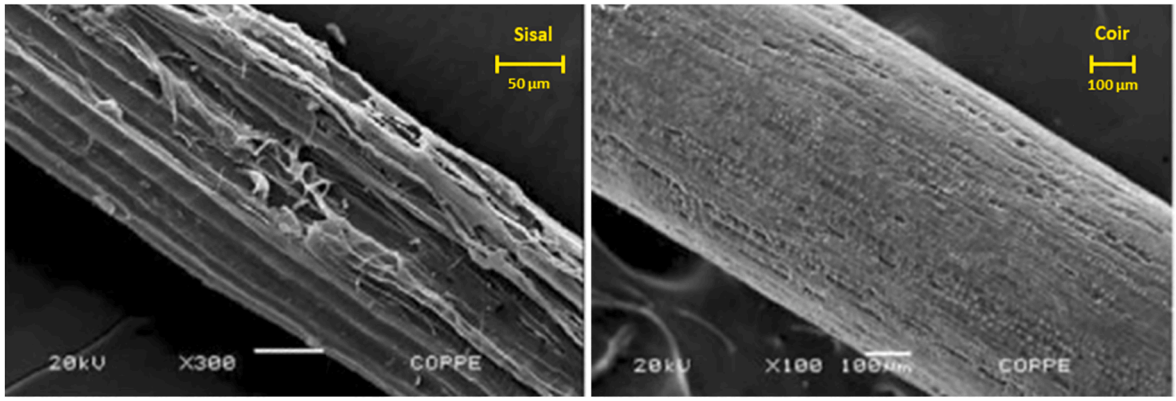

Fig. 10. Comparison of sisal fibre (left) and coconut fibre (right) in terms of microstructural surface, images sourced from [50].

sacrifice of tensile strength.

Furthermore, the alkaline solutions were capable of removing hemicellulose from fibres, while lignin and structural cellulose remained largely intact, which is one of the reasons why performance did not decrease. Recommendations for future studies are to continue research on the compositional changes incurred by alkaline media, for instance by studying the stability of lignin and cellulose or studying the stability of fibres which are enriched in hemicellulose and therefore expected to suffer more from alkaline hydrolysis.

In the real cement environment, tensile performance of fibres did deteriorate, apparently caused by extrinsic surface flaws since the Young's modulus (an intrinsic fibre property) remained constant. These flaws seemed, however, caused by damage inflicted while removing fibres from the cement matrix, so that the extent of fibre mineralisation could not be characterised. Future work could focus on characterisation of fibre mineralisation using test methods such as the strand-in test, which avoid mechanical fibre damage.

Nevertheless, results from this study help understanding the degradation mechanisms of coir (and other NF) in cement, and research may now be directed more efficiently towards understanding and mitigating fibre mineralisation, now that alkalinity has been eliminated as one of the main degradation mechanisms. Also, solving the concern about low stability in alkaline media may bring the use of natural fibres in cement-based composites one step closer to successful application.

Declaration of Competing Interest

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

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