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Effect of alkali-silica treatments of miscanthus fibres on chemical and micro-morphological modifications

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ABSTRACT

Chemical treatments can remove waxes and sugar components from the surface of bio-fibres, increasing their compatibility with mineral binders. This paper investigates the effects of alkali solutions treatments on miscanthus shives. For 8, 24, and 48 h, fibres were immersed in NaOH solutions at 1.5, 2.5, and 5.0% concentrations, with and without a 2.5% sodium silicate solution, having a silica modulus (SiO_2/Na_2O) of 2.0. SEM and Fourier Transform Infrared Spectroscopy (ATR-FTIR) were used to investigate the effect of different treatments on the microstructure and surface chemistry of miscanthus. The SEM results show that the morphologies of miscanthus fibres were significantly altered in the case of 5.0% NaOH treatment, with a weakening of the inner cell structures in some locations. Furthermore, the ATR-FTIR patterns of raw and treated shives were analysed, suggesting that treating miscanthus with 2.5% NaOH and 2.5% sodium silicate results in the required chemical modifications while retaining the cellular structure of miscanthus fibres. For all treatments, the absorbance was reduced by 31 to 77% at 450 cm^{-1} and 48–80% at 1035 cm^{-1}.

1. Introduction

Miscanthus is a locally available resource with proven environmental benefits in terms of carbon absorption, capture and storage in Southwest England (Ntimugura et al., 2021). The chemical compatibility of vegetal fibres with mineral binders, as observed for wood-cement composites (Fan et al., 2012), and hemp concretes (Wang et al., 2021), is one of the barriers to their use. Mineral binders' setting and hardening hindrance are triggered by water soluble carbohydrates from lignocellulosic fibres. Chemicals such as alkali-hydrolysed polysaccharides produce organic acids. Compounds, such as hemicelluloses and pectins, have the ability to form stable chelates with calcium ions in mineral binders, interfering with setting and hardening of binders (Jorge et al., 2004). Several physico-chemical treatments, including hornification, hydrophobic coating of fibres, and surface treatment with silanes, were investigated to improve the compatibility of lignocellulosic fibres with mineral binders (Magniont et al., 2017). Alkali treatments have been extensively investigated on several materials, including miscanthus (Haque et al., 2013), due to their effectiveness at low temperatures and pressures. Alongside this, it is also a relatively inexpensive method to use of an industrial scale, as noted for hemp shives (Sedan et al., 2008).

The influence of alkali-treatment on physical and chemical properties of miscanthus particles (Boix et al., 2016), and its effects on bio-based concrete strength development were investigated (Boix et al., 2020). It was reported that high NaOH concentration (5.0 wt% NaOH for 30 min) enables the removal of most of carboxylic group components (hemicellulose and lignin) (Boix et al., 2016). It has been shown that this treatment reduces water absorption, increases strength of bio-composites, and improves fibre–matrix adhesion in bio-cementitious materials. However, these results are extremely dependent on the alkali type, concentration, temperature, and duration of exposure.

The alkali treatment of biomass materials aims at modifying the chemistry of the cell membrane to improve the compatibility with mineral binders. The alkaline pre-treatment of biomass materials can dissolve lignin and a portion of hemicellulose by breaking polymer bonds (Badiei et al., 2014). NaOH, KOH, NH_4OH, NH_3, and $Ca(OH)_2$ have all been investigated for the pre-treatment of lignocellulosic materials, and NaOH has proven to be highly efficient (Kim et al., 2016). The addition of silicates in the treatment of biomass fibres for their use in building materials has the potential to trigger the nucleation of calcium silicate hydrates in the binding gel (Vo and Navard, 2016), and the

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Table 1

Description of different treatments of miscanthus particles. SS(l): sodium silicate (liquid solution), SH(l): sodium hydroxide, SM: Silica Modulus. tH: time of treatment in hours t = 8, 24, 48 h.

Treatment ID	Surface chemical treatment description	SM
1.5SH-0SS-tH*	Low concentration of NaOH: 1.5%	–
1.5SH-2.5SS-tH	Low concentration of NaOH (1.5%) + 2.5% sodium silicate	0.43
2.5SH-0SS-tH	Medium concentration of NaOH: 2.5%	–
2.5SH-2.5SS-tH	Medium concentration of NaOH: 2.5% + 2.5% sodium silicate	0.28
5.0SH-0SS-tH	High concentration of NaOH: 5%	–
5.0SH-2.5SS-0.5-tH	High concentration of NaOH: 5% + 2.5% sodium silicate	0.15

effects of silane treatment has been investigated in the literature (Bilba and Arsene, 2008). Sodium silicate can contribute to increase the strength of mineral binders (lime-cement) due to the reaction with residual calcium hydroxide and consequent production of further binding C-S-Hs. However, some sources of silica, including siloxanes, and silanes, part of the organosilicon family, exhibit hydrophobic properties that can interfere with the reaction chemistry of mineral binders (Grabowska and Koniorczyk, 2022).

The conventional alkali-treatment methods simply dissolve lignin and hemicellulose to reduce the chemical interaction of these chemical species with the setting mechanisms of Ca-based mineral binders. The approach followed in this research is a combination of alkali and silica treatments that aimed on one hand at removing lignin and hemicellulose (due to the presence of NaOH), and on the other hand at fostering the stickingof Si-O- radicals on the surface of the treated surfaces of the fibres (due to reactive silica). The Si-O- radicals could then trigger the C-S-H nucleation and hence lead to a stronger binder-fibre interfacial zone. In a similar approach, silane chemicals of the form Y-Si-(O-R) have been successfully used for the treatment of hemp fibres, creating Si-O-Si bonds with the fibres (Benitha Sandrine et al., 2015).

Si-O radicals are expected to foster CSH nucleation on the fibre surface, and thus to increase the strength of the mineral matrix-binder interface. The presence of Si-O-Si bonds is typically detected by FTIR analysis in the wavenumber range 1200–950 cm^{-1} (Ellerbrock et al., 2022). However, cellulose, hemicellulose and lignin have strong absorbances in the same range, thus the reduction of their peak intensities might hide the appearance of -Si- related bonds. Spectra deconvolution techniques could be adopted for further investigating this effect. However, the aim of the research was to check the efficiency of NaOH in dissolving lignin and hemicellulose even in the presence of sodium silicate, which typically reduces the pH of the solution. The simultaneous effect of the two chemicals is of interest when reactive silicates are purposely added in the mix for enhancing the chemical reactions in the binding matrix. The substantial equivalence of FTIR spectra with and without silicate is positive evidence of the suitability of the combined solution in the delignification of miscanthus biomass.

The purpose of this paper was to investigate the effect of NaOH concentration (1.5–5.0 wt%) and sodium silicate solution treatments on the microstructure and chemical features of miscanthus fibres at time intervals up to 48 h using scanning electron microscopy (SEM) and Fourier Transform Infrared Spectroscopy (ATR-FTIR). The addition of sodium silicate provides a potential to further enhance the interface with alkali-activated binders (Ntimugura et al., 2022). As main activators in alkali-activated binders, sodium hydroxide and silicate solutions were selected for use as in-process treatments. The overall objective of this paper was to provide a foundation for the applicability of the alkali-silica treatment to miscanthus fibres, from chemical modifications and microstructural perspectives.

2. Experimental methods

Miscanthus shives were procured from Lower Marsh Farm (Taunton), Somerset, UK. The average fibre length was 2–10 mm, while the width was 3.7–4.0 mm, and the moisture content was 9.8%. Miscanthus fibres were chemically treated by soaking 10 g of fibres in 1.5, 2.5, and 5.0 wt% NaOH and solutions, with and without 2.5% sodium silicate solution inclusion (Table 1). Sodium hydroxide solutions were obtained by dissolving NaOH pellets (99% purity) in tap water. Sodium silicate solution with Na_2O 12.8%, SiO_2 25.5% was supplied by Fisher scientific, with an average silica modulus (SM) of 2.0. All treatments were applied by soaking fibres in solutions for 8, 24, and 48 h in a laboratory environment $\sim 22\ ^\circ C \pm 0.5\ ^\circ C$. Miscanthus fibres were air-dried under the same room conditions for 72 h after treatment before SEM and FTIR analysis. Alkali reactions have an impact on vegetal fibres because they can disrupt/remove hydrogen bonds and ionise hydroxyl groups, increasing surface roughness and changing the chemical composition of fibres, according to equation (1) (John and Anandjiwala, 2008). Silicate treatment on other side, attaches silica on the surface of fibres (equation

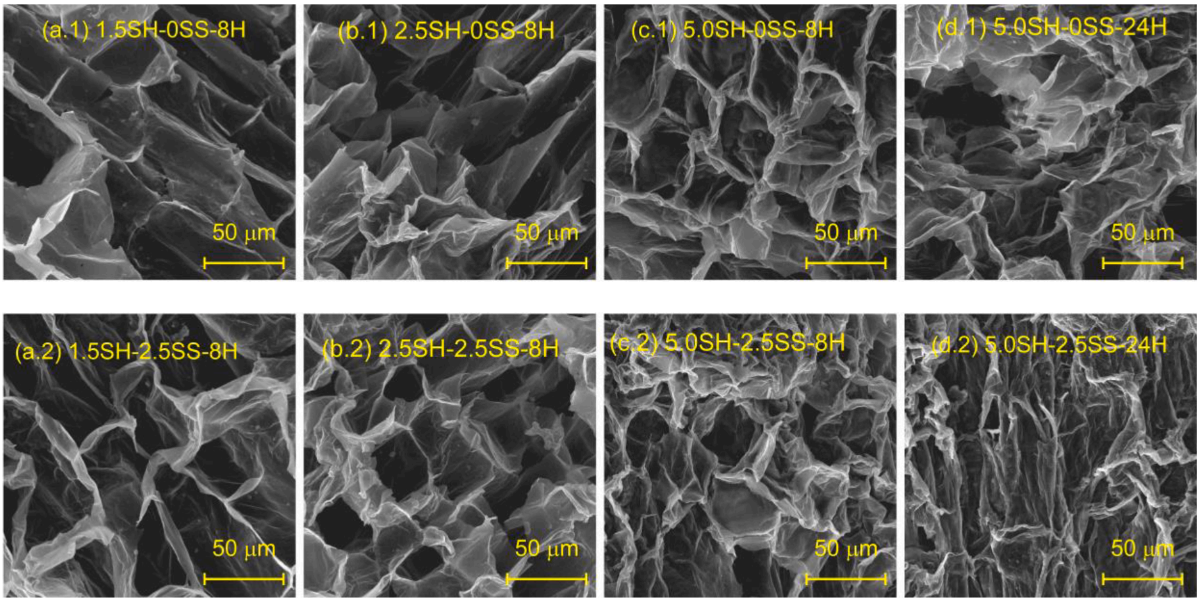

Fig. 1. SEM micrographs showing the inner surfaces of miscanthus following treatments for 8 h and 24 h. The scale bar applies to all images in the row.

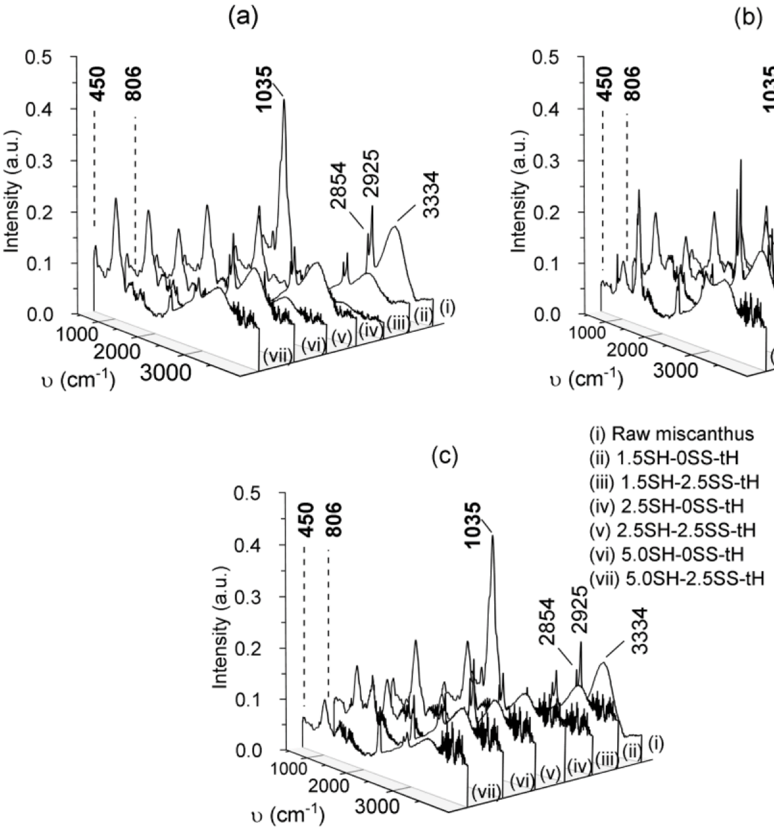

Fig. 2. ATR-FTIR spectra of raw and alkali-treated miscanthus fibres: (a) treatments for 8 h, (b) treatments for 24 h and (c) treatments for 48 h.

(2)) (Jiang et al., 2015), reducing water absorption and stimulating C-S-H formation in cementitious-pozzolanic binder composites.

$$(M) - OH + NaOH\,(l) \rightarrow (M) - O^{-} - Na^{+} + H_2O \tag{1}$$

$$Na_2O\,.\,nSiO_2 + CO_2 + mH_2O \rightarrow\; Na_2CO_3 + nSiO_2\,.\,mH_2O\,(\downarrow) \tag{2}$$

The morphological features of the fibres were observed using a scanning electron microscope (Tescan Vega 3, beam of 20 kV −22A). Samples were mounted on a carbon tape and coated with 15 nm Cr (Quorum Q150T ES Plus coater) prior to SEM observations. The chemical changes of fibres surface were analysed using attenuated total reflectance-Fourier transform infrared (Bruker Alpha II FTIR spectrometer with a Diamond Crystal ATR). The scan rate was fixed at 16 measurements for each record in the range of 400–4000 cm^{-1}.

3. Results and discussion

The morphology of the inner surface exhibits a characteristic honeycomb structure with cavities of dimensions 10-20x50 µm, in a repeating and regular pattern (Ntimugura et al., 2022). The experiment emphasized on the inner surfaces of the fibres because of their higher adherence to mineral binders. Fig. 1.a1-2, b1-2, c1-2 show the SEM of alkali-treated fibres after 8 h of treatment with 1.5% NaOH, 2.5% NaOH, and 5.0% NaOH, respectively, with and without sodium silicate.

The honeycomb structures on the inner surfaces lost their regular shape and exhibit an increasing degree of distortion as the NaOH concentration increases from 1.5 to 5%. At similar NaOH concentrations, the addition of sodium silicate to the treatment solutions increases the alteration of the honeycomb structure of miscanthus particles. As the treatment period increases (Fig. 1.c-1 and d.1), more distortion is observed, even to a higher degree with sodium silicate (Fig. 1c.2-d.2). The alkali-treatment affects the internal microstructure and overall honeycomb structure by dissolving lignin and hemicellulose, producing a change in the shape of the cell membranes. This change is due to the weakening of the primary and secondary cell walls, causing the lumen to collapse. The collapse and change of the honeycomb structure is more pronounced as the concentration and treatment time increase. The collapse of lumen of the lignocellulosic fibres was observed in a similar investigation on abaca fibres treated with 10–15 wt% NaOH (Cai et al., 2015).

The identification of FTIR absorbance bands and their respective chemical bonds assignment was based on literature in (Singh et al., 2020; Xu et al., 2013). The following bands can be identified in the main spectrum of the raw and treated fibres: The band at 3334 cm^{-1} corresponds O-H stretching vibrations of cellulose. The peak near 2925 cm^{-1} can be attributed to the deformation and stretching vibration of the C-H and O-H- groups of glucose units. The absorbance peak near 1734 cm^{-1} can be attributed to the ester linkage between lignin and hemicellulose. The absorbance band near 806 cm^{-1} corresponds to the C-O-C stretching of the cellulose chain. The sharp absorbance peak at 1035 cm^{-1} corresponds to the C-O stretching of hemicellulose secondary alcohols and ether groups, while the band near 450 cm^{-1} can be attributed to the vibration of C-O bonds from acetyl groups in lignin (Chen et al., 2010). The analysis allowed to qualitatively assess the changes in the chemistry of miscanthus fibres and thus to prove the suitability of the proposed treatment. Whilst a quantification of the lignin and hemicellulose contents could in principle be obtained with advanced chemometric analytical techniques, this would have required a significant number of FTIR spectra and the development of calibration models, which were outside the scope of the investigation.

Comparing the FTIR measurements of natural fibres before and after the chemical treatments reveals that the fibre surfaces underwent significant chemical changes (Fig. 2). The FTIR patterns for 1.5SH-2.5SS-8H show the most chemical changes across all spectra bands. The absorbance bands near 450 cm^{-1} and 1035 cm^{-1} respectively, are attributable to lignin, and C–C, C-O stretching or C-OH bending of

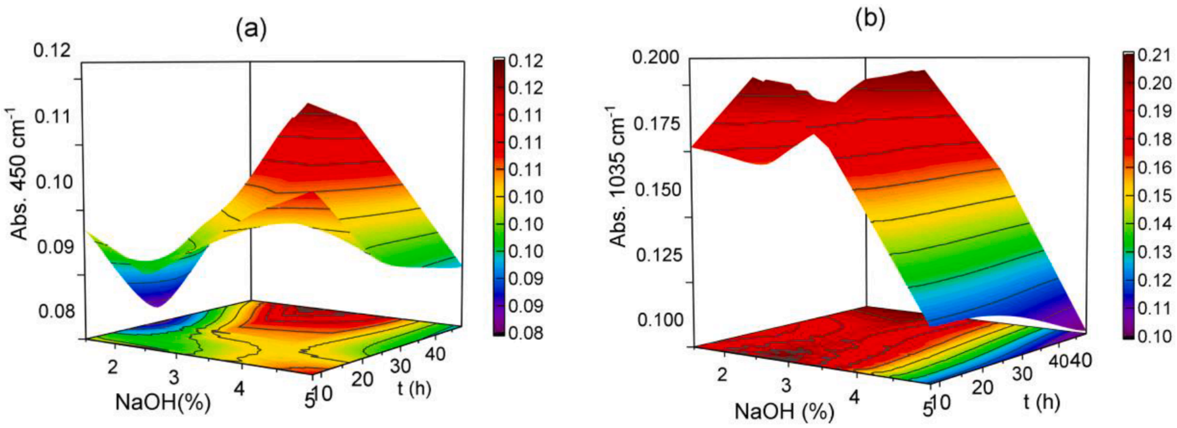

Fig. 3. (a) 3D ATR-FTIR spectrum absorbance levels for bands at 450 cm^{-1} for different NaOH concentrations and treatment time. (b) Idem for 1035 cm^{-1}.

alcohol and ether groups in cellulose and hemicellulose. The details of the attribution of absorbance bands to chemical bonds and species were reported in (Ntimugura et al., 2022). Fig. 3.a-b show the FTIR absorbance levels at 450 cm^{-1} and 1035 cm^{-1} to highlight the impact of sodium hydroxide and treatment times. The removal of amorphous components comprised of waxes, hemicelluloses, and pectins has a significant impact on the change of the chemical composition of lignocellulosic fibres (Cai et al., 2016). The determination of the crystallinity of cellulose can be achieved using FTIR specific peak ratios (Rafidison et al., (2020) 1922.). However, the latter was not further investigated in this paper. The alkali treatment of miscanthus fibres was effective in dissolving lignin and hemicellulose, resulting in the development of a rougher surface of the fibres, which can improve the bonding with mineral binders. This can enhance the properties of the interfacial transition zone and hence result in composites with higher mechanical strength (Rahimi et al., 2022). Longer treatment times and higher alkali chemical concentrations may result in the attack of cellulose fibre bundles. This is reported to occur as a result of the hydroxyl chemical bonds (–OH) breaking due to treatments using NaOH $>$ 9 wt% for three hours (Shahril et al., 2022). The alkali-silica treatment proposed in this paper targets the non-cellulosic compounds in the biomass, such as lignin, hemicellulose, and eventually pectins. Higher alkali solution concentrations result in more hemicellulose and lignin material breakdown as well as a higher surface ratio for the interfacial contact with mineral binders. Due to the increased surface areas and decreased hydrophilicity of the treated fibres, the treatment may reduce the negative effects of lignin and hemicellulose chemicals on the hydration of cement and lime mineral binders, and at the same time increase the adhesion of the matrix to the surface of the treated fibres (Kabir et al., 2012).

The use of lignocellulosic materials in bio-based construction composites may pose compatibility issues due to the leakage of chemicals from fibres that capture Ca^{2+} and hinder the setting and hardening mechanisms of mineral binders (Magniont et al., 2017). Alkali treatments contribute to the dissolution of these chemicals, thus reducing the risk of chemical interference between the lignocellulosic materials and mineral binders. Furthermore, the residual sodium silicate on the surface of the fibres could help in the activation of portlandite available in lime and cement based mineral binders to produce C-S-H gels (equation (3)) (Santana-Carrillo et al., 2021).

$$Na_2SiO_3 + n\,H_2O + m\,Ca(OH)_2 \rightarrow m\,CaO.SiO_2.n\,H_2O + 2NaOH \tag{3}$$

Silane treatments, such as aminopropyl triethoxy silane, are commonly used to functionalise lignocellulosic fibres in composites (Thakur et al., 2010), with a central -O-Si-O- similar to that found in commonly available sodium silicates. Therefore, the treatment proposed in this research can lead to the development of construction materials with improved strength at the fibres / matrix interface, and at the same time reducing compatibility issues.

4. Conclusion

The analysis of SEM micrographs on miscanthus fibres treated with alkali solutions confirmed that high concentrations at 5% significantly distorts the fibre honeycomb structure. High concentrations of alkaline solution combined with long treatment periods can damage the fibres by destroying the core cellulosic bundles. In this case, higher adhesion of miscanthus particles to mineral binders is expected. The investigated treatments reduced absorbance by 31 to 77% at 450 cm^{-1} and by 48–80% at 1035 cm^{-1}, proving that lignin and hemicellulose were effectively removed from the fibres surface. These findings will provide a simple and effective means for an in-process treatment of fibres applicable in alkali-activated bio-based composites, that has the potential of improving strength and dimensional stability.

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.

Data availability

The research data supporting this publication are available as supplementary information accompanying this publication.

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