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Aging behaviour assessment of cellulosic fibres in alkaline media: A green technology approach in construction materials

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ABSTRACT

With the increasing demand for buildings, the utilisation of green materials represents a novel approach toward achieving sustainable development goals. Cellulose-based materials, such as jute, exemplify eco-friendly substances for civil engineering applications. The present study aims at studying the aging behavior of jute fibers in an alkaline environment. For this purpose, three alkali environments (NaOH, KOH, $Ca(OH)_2$) with three different concentrations were selected. Samples were treated at three different periods (7 d, 14 d, and 28 d) to assess the impact of treatment on the fiber properties. This study introduces a pioneering concept of the alkali treatment of jute-based fibres to evaluate tensile strength and weight loss, marking the first of its kind. This approach incorporates various characterisation techniques, including Differential Scanning Calorimetry (DSC), Thermo-Gravimetric Analysis (TGA), and Scanning Electron Microscopy (SEM) to assess the treatment process. In our experimental procedures, we evaluated key parameters such as the type of alkali, alkali concentration, and curing time for their influence on tensile strength. Subsequently, we applied Response Surface Methodology (RSM) in conjunction with bagging techniques to develop mathematical models based on the acquired data. Results demonstrated varied tensile strengths based on alkali type, concentration, and treatment duration. Fibres treated with 15 g/L concentration of NaOH exhibited an increase in tensile strength from 7 days (79.66 MPa) to 28 days (225.05 MPa). The highest tensile strength of jute fibres treated with KOH was observed with 15 g/L concentration at 14 days (237.58 MPa). For $Ca(OH)_2$ treatments, the highest tensile strength was observed with 30 g/L concentration at 14 days (111.28 MPa). It was also observed that prolonged exposure to very high alkali concentrations could adversely affect the tensile strength, as observed in the case of 30 g/L concentration of NaOH at 28 days (84.73 MPa). Among the various alkali materials tested, NaOH and KOH demonstrated greater effectiveness compared to $Ca(OH)_2$. According to RSM analysis, the time of sample curing was found to be the most significant factor in tensile strength, with a P-value of

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0.009. On average, the tensile strength changes by 54 %, 52 %, and 35 % for NaOH, KOH, and $Ca(OH)_2$, respectively, when the curing time shifts from 7 days to 28 days.

1. Introduction

The construction sector has been undergoing rapid transformation with the incorporation of creative and eco-friendly materials, with the objective of decreasing harmful ecological effects, whilst concurrently increasing the effectiveness of cementitious materials. In this perspective, fibres are gaining much more attraction by researchers as they offer enhanced strength, durability, and resilience to construction materials [1]. Natural fibres, which have long been recognised for their significant role in the textile industry, have recently found a novel application in the field of construction, specifically in cementitious composites [2]. In the past, steel was predominantly used to reinforce concrete, but with the introduction of fibre technology, a wider range of fibres, such as polypropylene, glass, carbon, basalt, jute and others, are being employed in construction industry [3]. When these fibres are incorporated into building materials such as concrete or asphalt, they are able to bridge cracks in their initial stages, prevent the propagation of cracks and increase the tensile strength and fatigue resistance of the material [4]. Furthermore, fibres also enhance toughness, reduce shrinkage, and improve fire resistance [5]. In addition to enhancing structural performance, they also contribute to the sustainability of the construction sector. For instance, natural and recycled fibres are eco-friendly alternatives that can reduce the carbon footprint [6]. As the industry continues to evolve, the utilisation of fibres promises to revolutionise the way structures are built, making them more resilient, sustainable, and efficient.

During the process of cement hydration in cementitious composites, the presence of specific alkalis plays an essential role. As cement hydrates, it releases calcium ions and hydroxide ions, leading to the formation of $Ca(OH)_2$, also known as Portlandite. This compound contributes to the high alkalinity of the cement paste, which is crucial for the stability and formation of the calcium-silicate-hydrate (C–S–H) gel [7]. The alkaline components in cement can also generate sodium and potassium ions that can dissolve to form NaOH and KOH [8,9]. These alkalis can accelerate some hydration reactions, thereby boosting early-age strength. However, excessive amounts of NaOH and KOH can lead to complications, such as Alkali-Silica Reaction (ASR), that occur when these chemicals react with certain reactive silica in some aggregates. The resultant gel from ASR, when exposed to moisture, expands, exerting pressure internally, and causing cracks, which ultimately undermines the concrete's integrity [10].

Jute being an abundant, biodegradable, and natural fiber, offers an eco-friendly substitute for synthetic fibres and provides distinctive benefits when integrated into cement-based materials. The incorporation of jute within cementitious composites underlines the present-day transition of the construction industry towards sustainable methodologies, employing renewable resources in fabricating construction components that exhibit both durability and eco-friendliness. Moreover, it possesses an extensive range of applications [11,12]. Jute fibres are mainly composed of cellulose and non-cellulose parts. The non-cellulose part is comprised of lignin, pectin and hemi-cellulose [13]. Due to this, jute fiber represents a complex nature and needs to be addressed carefully, especially its behavior in an alkaline environment, which is frequently encountered in cement-based materials [14]. In such circumstances, jute fibres generally experience degradation due to the hydrolysis of hemicellulose and lignin present within the fiber [15]. This degradation can lead to a reduction in the mechanical properties of the technical jute fiber, resulting in the production of dust (elementary fibres) and loss of both strength and elasticity over time. Furthermore, the alkaline medium can lead to the swelling and ultimate disintegration of the technical jute fiber [16], thereby presenting significant challenges when technical jute fibres are utilised as reinforcement in cementitious composites without adequate pre-treatment [17].

To tackle this issue, researchers have examined and executed various treatments, such as the application of alkali-resistant coatings or modifications to the fiber structure, to boost the durability and stability of technical jute fibres in alkaline environments, thus ensuring their longevity and efficacy in composite applications. On the other hand, while dealing with alkalis such as NaOH, KOH and $Ca(OH)_2$, their concentrations and reactions must be carefully handled to ensure that the cementitious composites exhibit the desired performance and durability. Natural fibers have gained more interest because of their mechanical properties and low cost as well as the growing demand for environmental concerns [18]. Cellulosic fibers used in the fabrication of fiber reinforced cementitious composites require little energy to process [19]. One of the uses of cellulosic fibres in the construction and building industry is aimed at reinforcing cementitious composites while replacing traditional reinforcing materials more sustainably thus reducing the dependence on traditional materials to reduce carbon emissions, decrease waste generation, and improving the sustainability of construction materials. These fibres provide a promising approach to meeting environmental challenges and contributing to the environmentally friendly practices in the construction materials. A study conducted by Shireesha revealed that about 26 % of plant fibres were used in the construction sector, the second highest after the textile industry [20]. Cementitious composites having short and long cellulosic fibres affect positively the flexural strength of the matrix [18]. In a study conducted by Onuaguluchi et al., the optimal bending strength of cellulosic fibers in a cementitious composite was found to be 8–10 % [21]. Ramakrishna and Sundararajan revealed that the toughness of cement mortar reinforced with cellulosic fibers increased 3–18 times that of the samples without reinforcement [22]. In a recent study, jute fiber was pretreated by combining hot alkali solution soaking and chloroprene latex impregnation and was employed in the cementitious composite as a reinforcement. The results revealed that the mechanical properties including flexural strength, and splitting tensile strength increased considerably whereas the compressive strength remained unsatisfactory [23].

Cellulosic fibers, like jute, show improved durability in alkaline environments when modified with alkali and polymer, forming a protective coating. This reduces fiber mineralization, preserving tensile strength. Therefore, applying the treated fibers in alkaline environments can be useful for many structural and non-structural applications in some industries such as buildings [17].

Cellulose activation typically involves alkali treatment, commonly with sodium hydroxide due to its availability and cost-effectiveness. Mercerization using NaOH causes cellulose fibers to swell, morphologically change, and dissolve residual hemi-celluloses, albeit with risks of oxidative degradation. Control over activation hinges on alkali concentration and temperature, with diluted solutions widening micropores and higher concentrations splitting fibrillar aggregates. Concentrated solutions lead to increased swelling and partial cellulose transformation [24]. The alkaline treatment process is demonstrated in Fig. 1. Alkaline mercerization causes morphological alterations in cellulosic fibers. Fig. 1 shows how the morphological changes of cellulose fibers (such as their initial bean-shaped shape) are first caused by swelling and then by longitudinal fibers Shrinkage produces circular structures after elliptic structures first [24]. According to the scheme (Fig. 1), it can be understood that the morphological structure of the fibers is changing to a circular one, and it can be useful for increasing the mechanical specifications of some types of concrete [25].

In evaluating the aging process of fibers, various factors are considered, often dependent on the materials used and the intended application of the synthesised fibers. For instance, Scheffler, et al. [26] examined the aging of alkali-resistant glass and basalt fibers in alkaline solutions, focusing on parameters such as time and temperature of exposure, the inhibiting effect of calcium ions, and corrosion mechanisms. Similarly, Bentur et al. [27] investigated the aging of Glass-Fiber-Reinforced Cements in alkali environments, analysing microstructure changes, the influence of different environmental conditions on flexural behavior, and the growth of hydration products between glass filaments. Additionally, Akonda et al. [28] conducted a study on the aging behavior of glass/silicate composites, evaluating physical, thermal, and tensile properties.

Fig. 2 highlights the share of cellulose-based fibres consumption in recent years. It can be observed from the scheme that the use of cellulosic fibres has been increasing but still represents a small amount of the total fiber consumption. This indicates an excellent opportunity to use them as an alternative to traditional materials such as steel reinforcement in the construction industry to meet the growing demands of green building practices [80].

Some of the latest research works done with the technical jute fiber in cementitious materials are illustrated in Table 1. The literature review assessment is done based on different studies' aims and key findings. The main goal of this section is related to the findings of the latest research done with technical jute fibres in this research area.

According to Tables 1 and it can be found that over recent years, advancements in construction materials have intensely focused on harnessing the benefits of natural fibres, especially jute, in cement composites. Research by Gwon et al., Mawlood, and Khan et al. spotlighted the transformative impact of these fibres on the rheology, mechanical properties, and eco-friendliness of such composites. Studies by Alomayri et al. and Kurda have further underscored the synergy between jute fibres and additives such as silica fume, revealing substantial enhancements in strength, durability, and environmental sustainability. At the same time, efforts to use waste materials and compare different types of fibers, as shown in studies by Malagar and Singh, Choi, and Kurpińska et al. have expanded the possibilities for eco-friendly and creative building solutions [29–37].

Fig. 3 is obtained based on VOSviewer analysis of Scopus databank according to keyword occurrences. The scheme proves that the application of jute as fiber in alkali environments is not considered by previous investigations strongly which will be evaluated in this study. More specifically, this study aims to analyse the tensile tolerance of each sample as a cost function of optimisation process in which some preparation factors including the type of alkali, their concentration, and curing time are assessed. Therefore, different alkali effects on cement paste preparation with green fiber additives are considered as a research gap that will be filled in this practice. Besides, Fig. 3 demonstrated that research on jute fibres was developed after 2015, intensively.

Fig. 3 illustrates the distribution of published documents based on the frequency of specific keywords repetitions across different years around 2015. It is worth noting that the accumulated published research items were around 2015 in this field of research. This visualization represents a clustering process applied to keywords within various research items. By examining this scheme, we can discern the interconnection among different features and their prevalence over time. The data mining process employed in this method

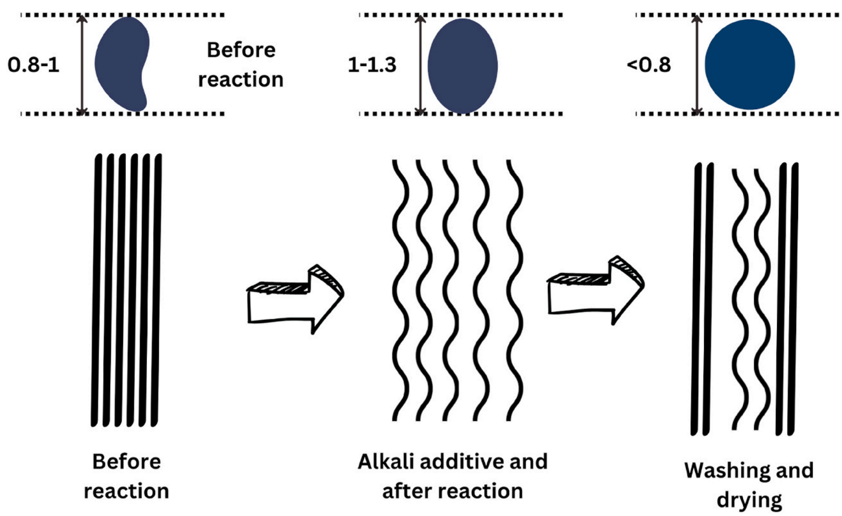

Fig. 1. The schematic plan of morphological changes of cellulose fibers in alkali environments (adapted from [24].

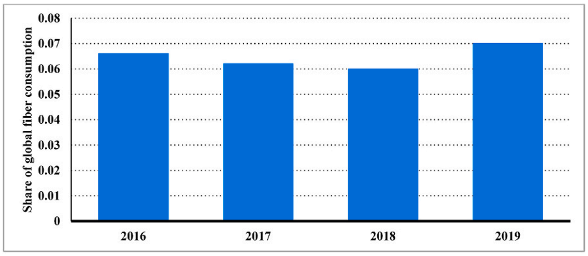

Fig. 2. Cellulosic fiber consumption around the world in recent years [80].

Table 1
Summary of the latest research works with jute fiber in cementitious materials.

Aims	Achievements/key findings	Researchers
Exploration of kenaf and jute fibres effects on the rheology and mechanical properties of cement composites, considering varied fiber lengths and volumes.	Jute's unique effects on composite properties identified optimal fiber configurations, and established correlations between fiber content and material properties, with kenaf fibres ensuring the best dispersion.	[29]
Enhancement of the flexural capacity and ductility of cementitious ferrocement composites using jute and polypropylene fabric reinforcements.	The fiber type and layer count significantly affected composite strength and ductility. Notably, jute-reinforced plates outperformed those with polypropylene, suggesting potential construction industry applications for these eco-friendly composites.	[30]
Exploring the potential of jute fiber (JF) in enhancing the mechanical strength of concrete and in reducing its carbon emissions, addressing a gap in current literature.	The optimal addition of 0.10 % JF improved various concrete strengths, offering both mechanical and environmental benefits. Using the RSM model, equations were derived, revealing the promising role of JF in future concrete advancements.	[31]
Effects of jute fiber volumes on high-performance concrete properties and the impact of silica fume as a secondary binder in JF-reinforced HPC.	Incorporating 0.3 % vol. Jute Fiber (JF) with Silica Fume (SF) enhanced High-Performance Concrete (HPC) compressive strength by 30 %. SF also improved JF's influence on HPC's durability and reduced chloride-ion permeability.	[32]
Understanding the effects of jute fibre (JF) and silica fume (SF) on enhancing plain concrete (PC) properties and environmental impacts.	JF and SF notably improved concrete's mechanical performance, with a 28 % increase in compressive strength. SF also addressed JF's water absorption and chloride penetration challenges.	[33]
The integration of waste materials (Bagasse ash, Waste Foundry Sand, Jute fiber) into high-strength concrete.	Bagasse ash decreased workability and strength beyond certain levels, Waste foundry sand maintained consistent performance, and Jute fiber, beneficial up to 2 %, reduced shrinkage. This incorporation promotes eco-friendly construction by reducing waste.	[34]
Combined effects of ground granulated blast furnace slag (GGBS) and jute fiber (JF) on creating eco-friendly, ductile concrete.	Adding JF with GGBS improved tensile and flexural strengths, especially when plasticisers were used, while GGBS countered JF's adverse effects on water absorption and chloride ion penetration.	[12]
Predicting the 28-day compressive and splitting tensile strength of concrete integrated with varying percentages of jute and coconut fibres and quarry dust, using both the response surface methodology and artificial neural networks.	Identifying optimal strengths using specific mixtures of fibres and dust. Both ANN and RSM effectively predicted strength values, with high correlations to experimental data, though RSM demonstrated superior accuracy.	[35]
The impact of natural fibres (abaca, hemp, jute) on cement hydration reactions and their potential as internal curing materials.	Natural fibres were found to delay cement hydration, affecting setting time, while their moisture absorption influenced the compressive strength and long-term strength improvement rate of the cement composites.	[36]
Evaluating the impact of both natural (e.g., cotton, sisal, jute) and synthetic (polymer, polypropylene) short fibres on the properties of cement composites.	Natural fibres improved cement composite compressive strength by 27 %, whereas synthetic fibres reduced it by 4 %, with specific fibres influencing consistency, strength variations, shrinkage, and water absorption.	[37]

relies on natural language processing algorithms to extract meaningful insights from the textual content of these documents (www.vosviewer.com). The analysis demonstrated that in the beginning, just fundamental ideas of jute as a fiber were studied by different research items. In the next cluster, the main idea of jute fiber applications in cement is presented. Finally, the main trend was related to polymer modification and jute application in different subjects such as concrete, cement paste, and 3D printing.

Kumar and Singh focued on assessing the degradation of composites comprising ethylene–propylene copolymer and jute fiber, examining their performance under accelerated aging and biotic environments [77]. Likewise[38] delve into the influence of accelerated aging on the interface of jute textile reinforced concrete. Also, Sanvezzo and Branciforti [39] investigate the recycling of industrial waste through the utilisation of jute fiber-polypropylene, aiming to produce sustainable fiber-reinforced polymer composites. The study further characterizes these composites both before and after accelerated aging to evaluate their durability. Finally, Wang, et al. [40] researched enhancing water aging resistance in jute fiber composites by adding alumina ceramic particles. They analyzed the effects of temperature, vibration frequency, and loading time on viscoelastic properties pre and post-water aging, noting reversible and irreversible performance degradation. All the research studies evaluated the aging of jute fibers in a specific way. However, for the first time the present study studies the aging of cellulosic fibres in alkaline media. Plus, for the first time, the interactions of effective

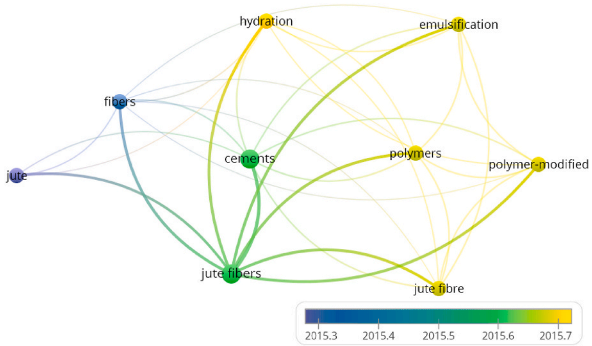

Fig. 3. The output of bibliometric analysis of jute fiber application in cement paste structure as per Scopus databank.

features including alkali type, alkali concentration, and time are assessed based on tensile strength. The interaction of the features with the strength is assessed with the application of the RSM method.

Therefore, the objectives of this study include (1) lab-scaled sample preparation of different jute-based fibres in alkali environments subject to weight loss and tensile strength tolerance assessment by the One-Factor-at-A-Time (OFAT) method; for this purpose, three different alkalis (NaOH, KOH, and $Ca(OH)_2$) with three different concentrations (5 g/L, 15 g/L and 30 g/L) were chosen. The samples were treated for three different periods (7 d, 14 d, and 28 d) (2) characterisation of prepared samples by the application of Differential Scanning Calorimetry (DSC), Thermo-Gravimetric Analysis (TGA), and Scanning Electron Microscope (SEM) instruments to evaluate the morphological and structural alterations and to investigate the thermal properties and degradation behavior of alkali treated jute fibers; (3) optimisation, sensitive analysis, and mathematical modelling of effective factors influence tensile function with Response Surface Methodology (RSM) technique.

In the following the methodology section is conveyed in Section 2 with a concentration on both experimental and numerical techniques. Then, the results and outputs of this research item focusing on their impacts are presented in Section 3. Finally, the main achievements of this effort plus some suggestions for future studies are illustrated in Section 4.

2. Materials and methods

The research roadmap of this study is depicted in Fig. 4. According to the research scheme, this study is divided into three phases including sample preparation and characterisations, optimisation by HDA-RSM method, and prediction system execution. The experimental and numerical assessments are conducted sequentially as per data preparation, categorisation, and modelling.

Preparation involves meticulous selection of chemicals and substrate, followed by precise preparation of alkali solutions. Samples undergo controlled treatment for 7, 14, and 28 days, with vigilant monitoring. A rigorous sampling protocol ensures accurate analysis, promising insightful outcomes.

Analysis includes weight loss assessment for material degradation, tensile strength testing for mechanical integrity, and DSC and TGA for thermal properties. These methods collectively reveal treatment impact on sample characteristics, enabling comprehensive evaluation.

RSM is employed to systematically explore the parameter space for optimising treatment conditions. Sensitivity analysis identifies critical factors influencing outcomes, guiding focus. Statistical assessment ensures the robustness and reliability of results. Through RSM, optimal factors are pinpointed, maximizing desired responses efficiently.

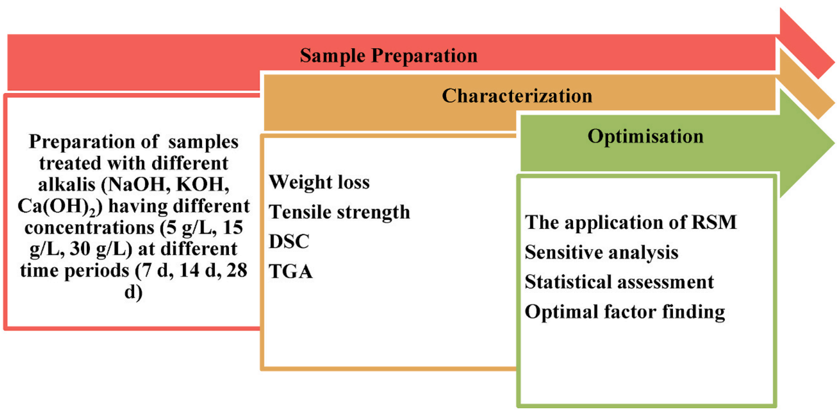

Fig. 4. The research roadmap of the present study including sample preparation, characterisation, and optimisation process.

2.1. Materials and applied instruments

In this study, the list of applied materials and compounds and also, the used instruments are summarised in Table 2 and Table 3, respectively. The utilised materials are provided from different sources and the instruments were available at the Technical University of Liberec. For sample preparation, jute fibers were prepared by taking in each sample twenty threads with each thread fifty centimetrs long. For alkali solution preparation, 5 g, 15 g, and 30 g of each alkali were mixed in 1 L of distilled water. The solution was poured into a 200 ml container and jute fibers were immersed in it. The samples were kept for 7 days, 14 days, and 28 days at room temperature. After the treatment period, the samples were taken out, rinsed with distilled water carefully, and kept for drying at room temperature (see Table 4).

2.2. Measurement protocols and techniques

The stages of experimental sample preparation and data gathering in this study are shown in Fig. 5. In the experimental stages (Fig. 5), jute fibres along with solutions having different concentrations (5 g/L, 15 g/L and 30 g/L) of NaOH, KOH and $Ca(OH)_2$ were prepared as described earlier. Therefore, it is important to emphasise that there is room for variance in the tests depending on three main factors: the kind of alkali, the alkali concentration, and the length of exposure. It is also important to remember that, to maintain uniformity across experimental conditions, all tests were carried out at room temperature. Furthermore, because of the significance of these factors, careful control and modification were used to clarify their individual and combined effects on the results of the experiment.

Fig. 6 presents that after sample preparation, three characterisations including TGA, SEM, and DSC are first carried out on the samples to observe the behavior of jute technical fibres under varying thermal conditions followed by conducting the tensile strength test for each sample by the LAP TEST 2.010 instrument. The collected data are then evaluated to find the impact of the process performance on the increasing of quality of samples. All experimental practices of this study are done based on ČSN EN ISO 2062 [87], ČSN EN ISO 5079 [86], ČSN EN ISO 11358–1 [84], ČSN EN ISO 11357–1 [83], and ISO/TS 21383:2021 [82].

2.3. RSM modelling

This study adopts RSM (Response Surface method) modelling as statistical data analysis approach that involves creating a mathematical model to predict how the response changes based on variations in input variables. The methodology also facilitated the identification of the most influential factors and their interactions, which would be pivotal in steering the tensile strength to desired levels. The general second-order polynomial equation applied in RSM for this three-factor system is expressed as [41]:

$$y=\beta_0+\beta_1x_1+\beta_2x_2+\beta_3x_3+\beta_{12}x_1x_2+\beta_{13}x_1x_3+\beta_{23}x_2x_3+\beta_{11}x_1^2+\beta_{22}x_2^2+\beta_{33}x_3^2 \quad \text{Eq. (1)}$$

x_1: Time
x_2: Alkali type (coded numerically for different types)
x_3: Alkali concentration
y: represents the predicted response (tensile strength).
β_0 is the constant term.
β_1, β_2, and β_3 are the linear coefficients.
β_{12}, β_{13}, and β_{23} are the interaction coefficients.
β_{11}, β_{22}, and β_{33} are the quadratic coefficients.
In this research, Design Expert 7.0.0 is applied to implement the RSM analysis.

3. Results and discussion

The results of weight loss of jute fibres after treatment with various concentrations of NaOH, KOH and $Ca(OH)_2$ at different time durations are represented in Fig. 7. It can be seen from Fig. 7a that the weight loss tends to increase with both concentration and duration. A clear trend is observed where the highest weight loss occurs at 30 g/L concentration over 28 days, reaching up to 19.17 %. Even at the shortest duration of 7 days, NaOH causes significant weight loss, with 13.13 % being observed at 30 g/L concentration. The pattern of weight loss for KOH is slightly different than NaOH. Although weight loss increases with time, there is no consistent trend as it increases with concentration; however, the weight loss percentages are relatively closer across the different concentrations, especially for longer durations, as shown in Fig. 7b. $Ca(OH)_2$ on the other hand appears with the lowest weight loss percentages across all conditions when compared to NaOH and KOH as shown in Fig. 7c. Jute fibre is mainly composed of cellulose (61–71.5 %), hemicellulose (12–20.4 %), and lignin (11.8–13 %) [42]. During the alkali treatment, leaching of lignin or hemicellulose from the structure of the jute fibre occurs and this can lead to weight loss of the fibre [43]. This was further supported by Kataoka and Luz [81].

Table 2
The utilised materials in the present research.

Material	Company	Application goal
NaOH	Lach-Ner, Czech Republic	Preparation of Alkaline environment
KOH	Lach-Ner, Czech Republic	Preparation of Alkaline environment
$Ca(OH)_2$	Lach-Ner, Czech Republic	Preparation of Alkaline environment
Jute Fiber	Saifan, S.R.O, Czech Republic	Determination of tensile strength

Table 3
The applied instruments during this study.

Instrument	Company	Model
Tensile Testing Machine	Labor Tech	LAP TEST 2.010
Thermogravimetric Analyser	Mettler Toledo	TGA/SDTA851e
Differential Scanning Calorimeter	Mettler Toledo	DSC 3^+ Star Systém
Scanning Electron Microscope	TESCAN	TESCAN VEGA3

Table 4
The data used in the RSM modelling.

Alkali type	Alkali concentration (g/L)	Time (day)	Tensile Strength (MPa)
NaOH	5	7	41.62
NaOH	15	7	79.66
NaOH	30	7	60.77
KOH	5	7	69.38
KOH	15	7	49.05
KOH	30	7	82.67
$Ca(OH)_2$	5	7	51.48
$Ca(OH)_2$	15	7	67.4
$Ca(OH)_2$	30	7	41.41
NaOH	5	14	84.2
NaOH	15	14	58.68
NaOH	30	14	181.27
KOH	5	14	194.81
KOH	15	14	237.58
KOH	30	14	103.7
$Ca(OH)_2$	5	14	74.38
$Ca(OH)_2$	15	14	62.73
$Ca(OH)_2$	30	14	111.28
NaOH	5	28	140.34
NaOH	15	28	225.05
NaOH	30	28	84.73
KOH	5	28	130.7
KOH	15	28	131.5
KOH	30	28	163.06
$Ca(OH)_2$	5	28	67.75
$Ca(OH)_2$	15	28	95.92
$Ca(OH)_2$	30	28	86.72

Dissolution of the fibre bundle into smaller fibres, is caused by the alkali treatment, which also breaks the hydrogen bonds that bind the hydroxyl groups (-OH) of cellulose, hemicellulose, and lignin together [44,45]. Lignin and hemicellulose are known to impede the matrix-fiber bonding in composite materials; hence, their removal can enhance the mechanical properties of the fiber [46]. During the alkali treatment of the fibres, hydrogen bonding in the network structure is disrupted leading to increased surface roughness and removing lignin, hemicellulose, and other impurities [47]. Moreover, eliminating these components exposes the cellulose content of the fiber, which is primarily responsible for its tensile strength. In natural fibers, cellulose content and the orientation of microfibrils in the cell wall are mainly responsible for the strength and stiffness [48]. Alkaline treatment of natural fibers increases surface roughness for enhanced mechanical interlocking as well as the amount of cellulose exposed on the surface of the fibre, therefore, enhancing the possible reaction following a durable effect on the mechanical properties of the fibre [49,50]. Thus, alkali treatment is not only responsible for some weight loss of the fibres but also has an indirect indication of the potential enhancement in the fibre's performance when incorporated into composite materials. For instance, improvements in tensile and fatigue properties were noted for 1 % and 5 % alkali treatment whereas, for a higher concentration of 10 %, no improvements in the mechanical properties of the fibre were observed [51].

Fig. 8 presents the results of SEM characterisations of jute fibres based on different alkali solutions with different concentrations. According to Fig. 8, all SEM images are reported in 250× magnifications.

The outcomes of OFAT experiments are presented in Fig. 9. Based on Fig. 9a, at a shorter duration of 7 days, the tensile strength appears to peak at 15 g/L NaOH concentration (79.66 MPa) and then decrease slightly at 30 g/L (60.77 MPa). This suggests that there might be an optimal alkali concentration for enhancing the tensile strength within a short treatment period. However, for a duration of 14 days, the tensile strength is highest at 30 g/L (181.27 MPa) compared to both 5 g/L (84.2 MPa) and 15 g/L (58.68 MPa). At 28 days, the 15 g/L concentration again exhibits the highest tensile strength (225.05 MPa), with a drop at 30 g/L (84.73 MPa) and a lesser value at 5 g/L (140.34 MPa) as shown in Fig. 8a and b. This might suggest that prolonged exposure to very high alkali concentrations could adversely affect the tensile strength, as previously mentioned by Ref. [52], while moderate concentrations (15 g/L) seem optimal. For the 5 g/L NaOH concentration, a noticeable increase in tensile strength with time can be observed, moving from 41.62 MPa (7 days) to 84.2 MPa (14 days) and then to 140.34 MPa (28 days). This suggests that longer exposure to a mild alkali solution significantly benefits the tensile properties of the jute fibres. According to a study conducted by Hoyos et al., fique fibres treated with 5w/v% NaOH solution

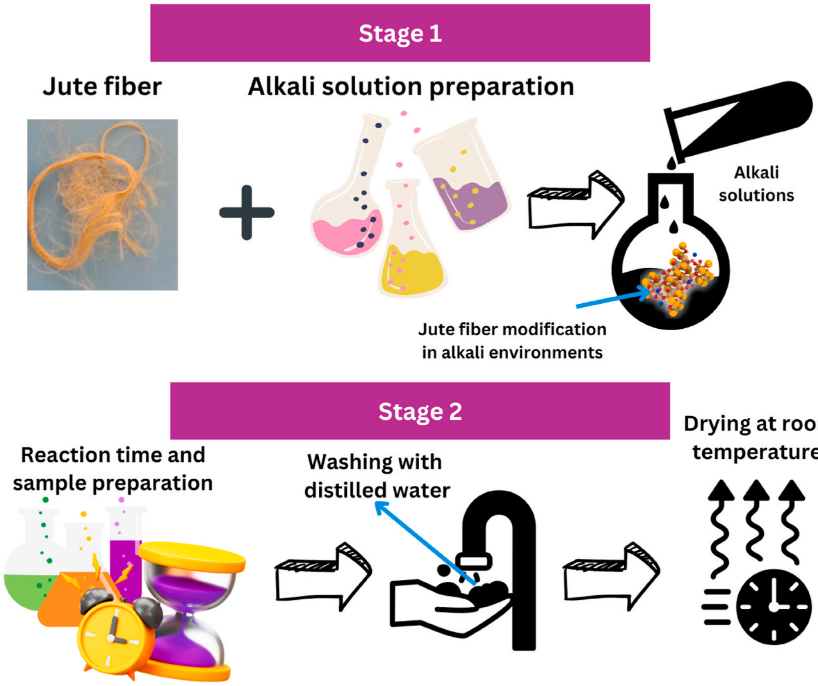

Fig. 5. The schematic plan of sample preparations in this study.

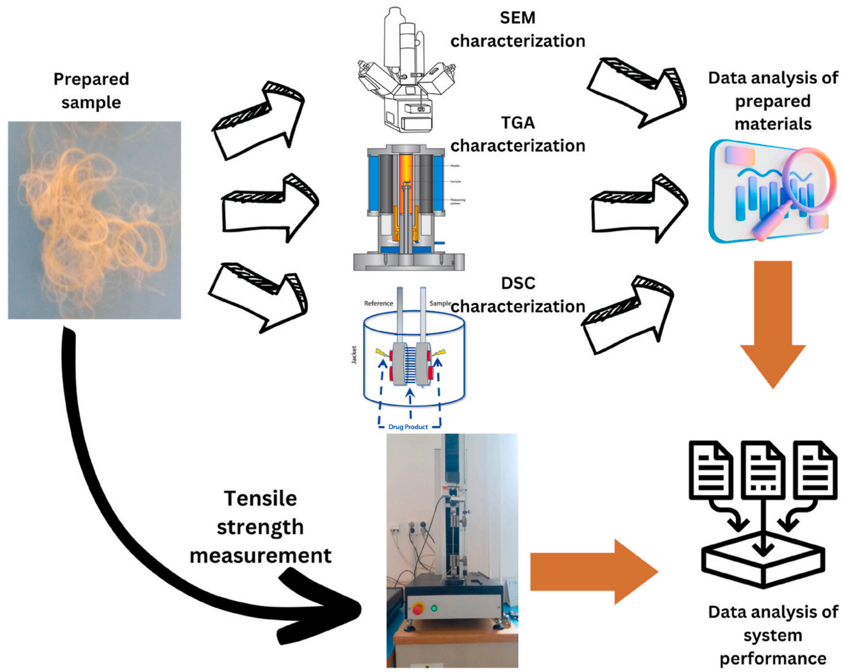

Fig. 6. The steps of various tests and analyses on the sample in this study.

for 24 h showed an increase in tensile strength of the fibres [53]. Conversely, for the 30 g/L concentration, there is a fluctuation in strength. The tensile strength first increases from 60.77 MPa (7 days) to 181.27 MPa (14 days) but drops to 84.73 MPa (28 days), reinforcing the idea of possible damage with prolonged high-concentration treatments. The 15 g/L treatment displays a steady increase with time: 79.66 MPa (7 days) to 58.68 MPa (14 days) and then a significant jump to 225.05 MPa (28 days). The decrease from 7 to 14 days might require further investigation.

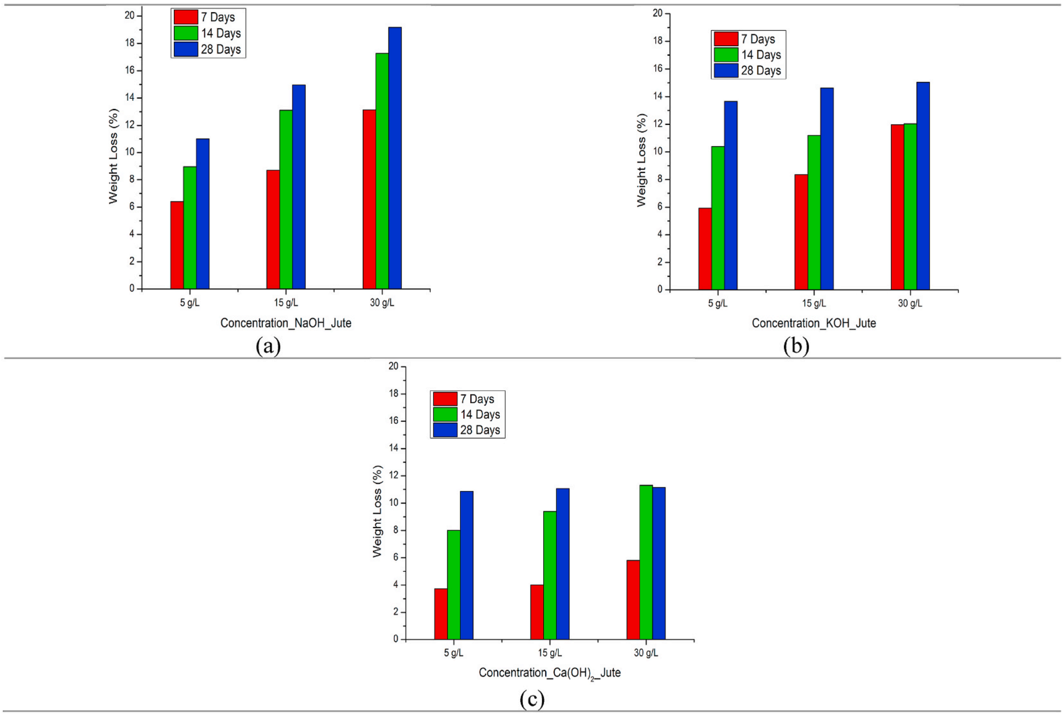

Fig. 7. The Weight loss (%) of different alkali treated jute fibres by (a) NaOH, (b) KOH and (c) $Ca(OH)_2$.

Based on Fig. 9b, For the 7-day duration, the tensile strength peaks at the 30 g/L KOH concentration (82.67 MPa). The 15 g/L concentration (49.05 MPa) shows a decrease compared to the 5 g/L concentration (69.38 MPa) as shown in Fig. 8e. This suggests that, in a short treatment time, the jute fibres might respond better to higher concentrations of KOH. At the 14-day mark, the tensile strength is highest for the 15 g/L concentration (237.58 MPa), followed closely by the 5 g/L (194.81 MPa) and then a significant decrease at 30 g/L (103.7 MPa). This indicates an optimal response at a moderate alkali concentration for this treatment duration. By 28 days, the tensile strengths are fairly close across the three concentrations: 130.7 MPa (5 g/L), 131.5 MPa (15 g/L), and 163.06 MPa (30 g/L). This indicates a more uniform response to KOH treatment over an extended period, with a slight advantage for the highest concentration. For the 5 g/L concentration, the tensile strength steadily increases from 69.38 MPa (7 days) to 194.81 MPa (14 days), then slightly decreases to 130.7 MPa (28 days). This could suggest a plateau or limit to the fiber enhancement after a certain treatment time. The 15 g/L concentration starts with 49.05 MPa (7 days), surges to 237.58 MPa (14 days), and then slightly decreases to 131.5 MPa (28 days). This again suggests a potential plateau effect after two weeks. The 30 g/L concentration shows a variable trend: 82.67 MPa (7 days), 103.7 MPa (14 days), and 163.06 MPa (28 days). The increase from 14 to 28 days might suggest that longer treatments are beneficial at high concentrations, but not as effectively as at the 5 g/L or 15 g/L levels.

Based on Fig. 9c, at 7 days, the tensile strength peaks at the 15 g/L concentration of $Ca(OH)_2$ (67.4 MPa) as shown in Fig. 8c and d. The strength is lower at 5 g/L (51.48 MPa) and a considerable decrease in tensile strength can be observed at 30 g/L (41.41 MPa). This might suggest that, for short durations, a moderate concentration is optimal. At 14 days, the 30 g/L concentration gives the highest tensile strength (111.28 MPa), more than the 5 g/L (74.38 MPa) and 15 g/L concentrations (62.73 MPa). This indicates that the fibres benefit from a higher alkaline concentration with slightly longer treatment [54]. By 28 days, the tensile strength at 15 g/L (95.92 MPa) is higher than both the 5 g/L (67.75 MPa) and 30 g/L concentrations (86.72 MPa), suggesting that prolonged exposure to an extremely high concentration might not be as effective as a moderate one. At the 5 g/L concentration, there's a progressive increase in tensile strength from 51.48 MPa (7 days) to 74.38 MPa (14 days), and a minor decrease to 67.75 MPa (28 days). This indicates that the effect of a mild alkali solution may plateau after two weeks. The 15 g/L concentration sees an initial increase from 67.4 MPa (7 days) to 62.73 MPa (14 days), and then another increase to 95.92 MPa (28 days). This nonlinear response suggests that the fibres may undergo different mechanisms of interaction at this concentration over time. For the 30 g/L concentration, the tensile strength first decreases from 41.41 MPa (7 days) to 111.28 MPa (14 days) and then drops slightly to 86.72 MPa (28 days). This fluctuation indicates that longer exposure times at high concentrations can lead to variable outcomes, with potential damage or saturation effects [55].

Jute fibres demonstrate varied tensile strengths based on alkali type, concentration, and treatment duration. While NaOH-treated fibres emphasise the necessity of balancing concentration and time to maximise tensile strength, the KOH series showcases distinct behavioural patterns. The $Ca(OH)_2$ treatments further underscore these differences, with the peak tensile strength achieved at a 30 g/L concentration over 14 days. These variances across alkali types underline the pivotal role of alkali selection in treatment outcomes. Some recent studies indicating different treatments of various cellulosic fibres with alkalis include [56–65].

Fig. 10 presents the DSC curves of all the samples. Before 100 ^oC, there is one single peak of all the samples. By combining with the

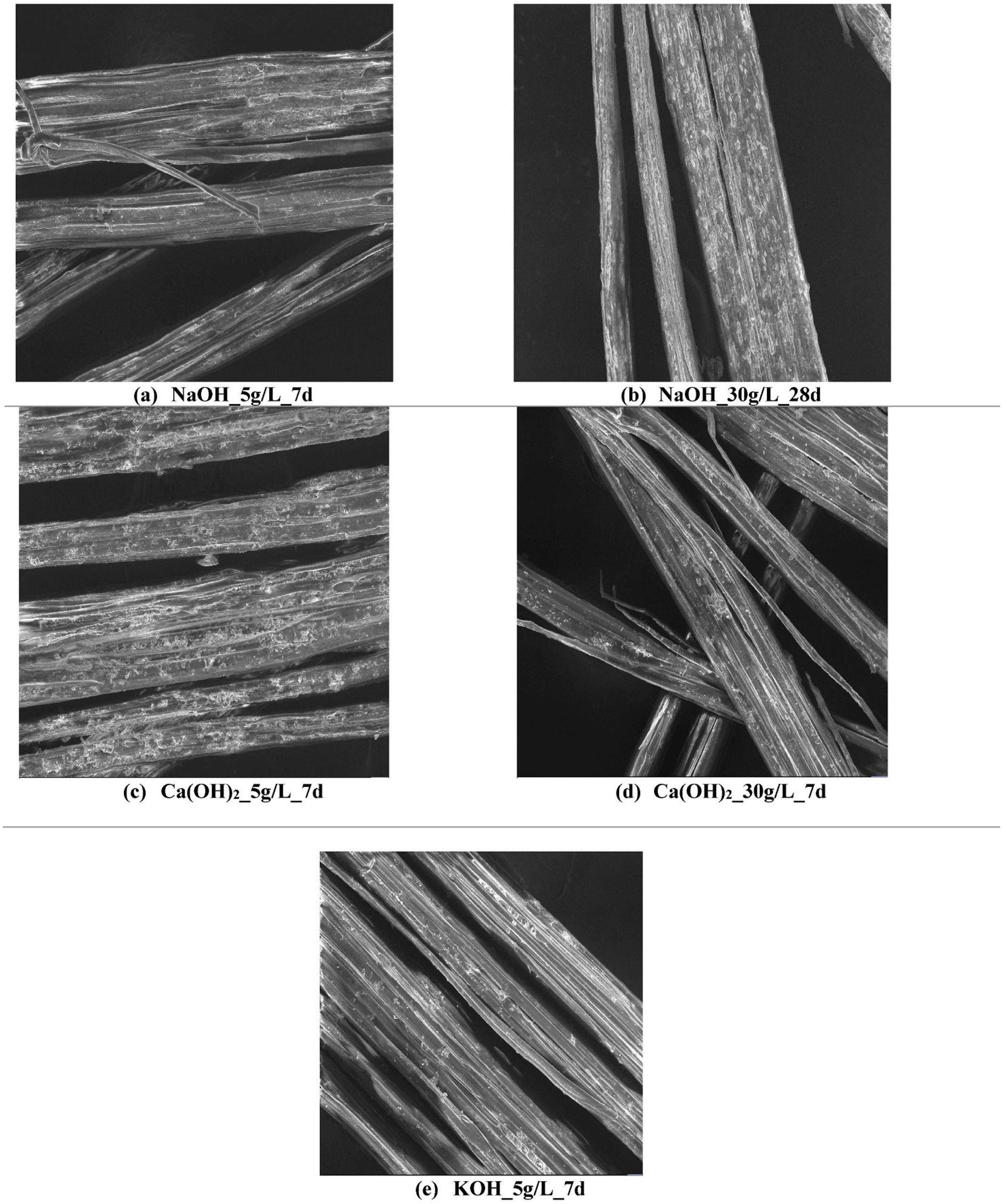

Fig. 8. The SEM outputs of different alkali-treated jute fibres (a,b) NaOH (c,d) $Ca(OH)_2$ (e) KOH; magnification: 250 X.

following analysis of TGA, it is caused by moisture evaporation [88]. In the temperature range from 300 ^{o}C to 400 ^{o}C, DSC curves of all the samples experience a fluctuation. By combining with the following analysis of TGA, the fluctuation is caused by thermal decomposition.

The endothermic peaks, associated with energy-absorbing processes such as melting or evaporation [66], appear to have somewhat similar behavior for the NaOH-treated jute fiber and the KOH-treated sample. The exothermic peaks that is usually related to energy-releasing processes such as decomposition or oxidation [67] have also a somewhat similar trend for NaOH-treated and KOH-treated samples with varying specific temperatures and intensities. In contrast, $Ca(OH)_2$-treated fibres seem to undergo different thermal events, as indicated by exclusively exothermic reactions. The peaks above 300 °C in all samples likely relate to cellulose decomposition [68,69]. The DSC data suggests that the alkali treatment indeed affects the thermal behavior of jute fibres [85].

Thermogravimetric Analysis (TGA) provides information on the thermal stability of materials by recording weight loss as the sample is heated [70]. Fig. 11 has three curves representing jute treated with NaOH, KOH, and $Ca(OH)_2$. All three curves exhibit a similar trend, showing a gradual weight loss up to around 300°C followed by a steep decline. This indicates that the jute fiber

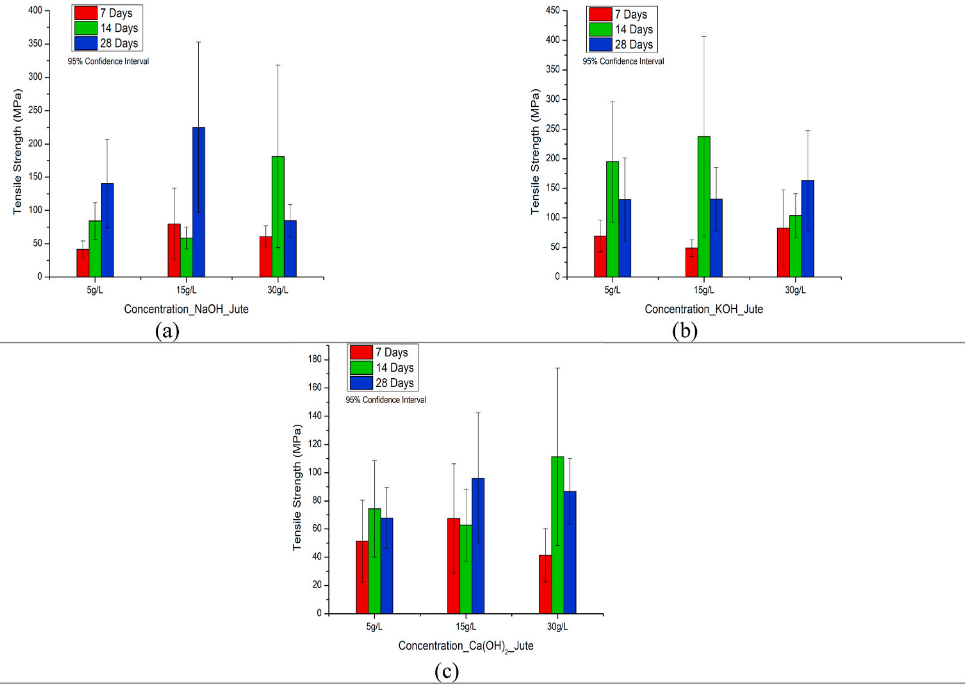

Fig. 9. The tensile strength of jute fibres treated with different alkalis, concentrations, and time periods (a) NaOH (b) KOH (c) $Ca(OH)_2$.

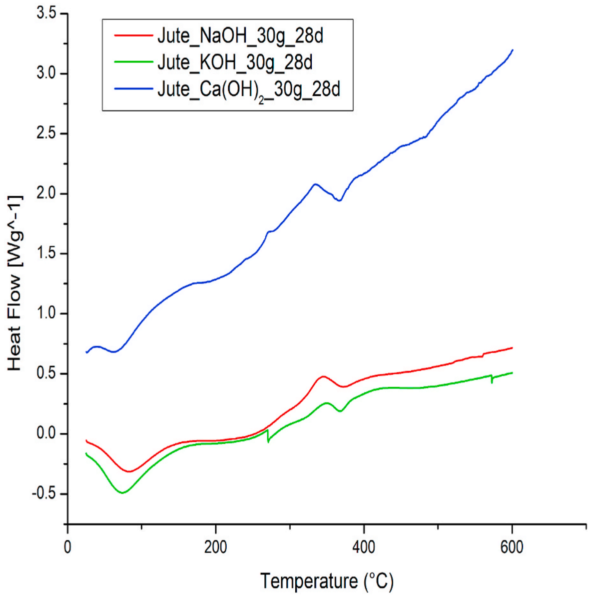

Fig. 10. The DSC analysis outputs of different jute samples.

undergoes thermal degradation in multiple stages. The initial weight loss is likely due to the evaporation of moisture and other volatile compounds from the beginning [71]. The major weight loss between 300°C and 400°C represents the decomposition of cellulose, the main component of jute fiber [72]. The final weight loss after 400°C corresponds to the breakdown of lignin and hemicellulose, which are the other major components of the fiber [88] as lignin decomposes at larger temperatures [89]. The range for T_g is from 220 ^{o}C till 350 ^{o}C. The TGA data suggests that the different alkaline treatments significantly impact the thermal stability of jute fibers. For instance, $Ca(OH)_2$ treatment imparts the highest thermal stability, followed by KOH and NaOH. This could be due to the different chemical interactions between the alkaline agents and the jute fiber components. The treatment with NaOH weakens the fiber structure more than the other two, resulting in a lower thermal degradation temperature and faster decomposition.

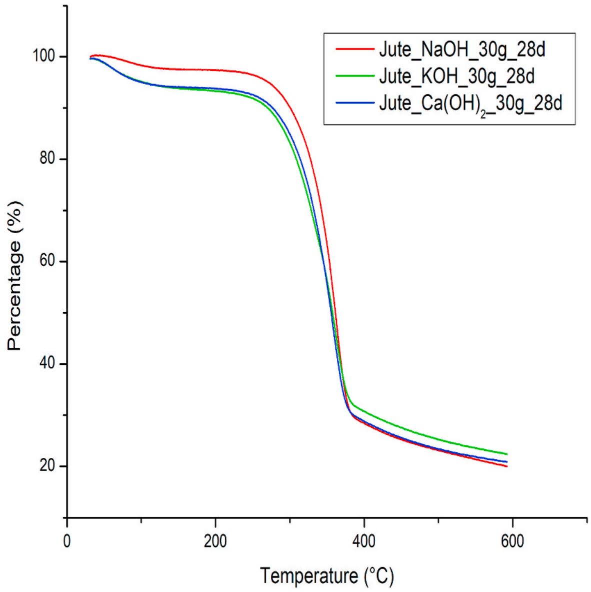

Fig. 11. The TGA results of different jute samples.

The RSM analysis data are presented in Table 5. As indicated in the table, we evaluated the impact of three parameters: alkali type, alkali concentration, and time, on the tensile strength as the response variable in our model.

Different combinations of alkali type (Ai), alkali concentration (Ci), and time (ti) are used in each of these tests. Different test results are a result of these changing parameters. We investigated the connections between the alkali type, concentration, time, and the observed responses using historical data and RSM. The mathematical expression of the model is demonstrated in Equation (2).

$$Yi = f\ (Ai,\ Ci,\ ti) \quad \text{Eq. (2)}$$

Yi representing the outcome of the ith test's response. This function f will be a mathematical expression that captures the behavior of the system under study. In the course of conducting regression analysis on the experimental data, we derived a mathematical model as depicted in Equation (3). As indicated in Tables 5 and it becomes evident that the quadratic model outperforms the others due to its minimal standard deviation and maximal R-squared value.

$$\begin{aligned} \mathit{Tensile\ Strength} = {} & 221.65x\mathit{AlkaliType} + 4.18x\mathit{AlkaliConcentration} + 21.14x\mathit{Time} - 0.047x\mathit{AlkaliTypexAlkaliConcentration} \\ & - 1.42x\mathit{AlkaliTypexTime} - 0.029685x\mathit{AlkaliConcentrationxTime} - 39.41x\mathit{AlkaliType}^2 - 0.09x\mathit{AlkaliConcentration}^2 \\ & - 0.42\mathit{Time}^2 \end{aligned} \quad \text{Eq. (3)}$$

Based on the insights gleaned from Table 6, it becomes apparent that the Time of sample curing, with the highest F-value of 8.65, emerges as the most pivotal feature in our analysis. As we delve further into our investigations, we find that Alkali type assumes the mantle of being the most influential factor in our experimental endeavours. This hierarchical order of significance among the variables underscores the importance of these factors in shaping our outcomes.

Analysing Fig. 12a, it becomes evident that the variations in slope for alkali type are more pronounced when compared to those of alkali concentration. This observation highlights the greater significance of alkali type in contrast to alkali concentration in our study. Moreover, examining Fig. 12b and c, we discern that the time of sample curing exhibits more pronounced slope changes than both alkali type and concentration. This finding underscores the heightened importance of time of sample curing, as reflected in the magnitude of its slope variations, in shaping our research outcomes. Taking Fig. 12 into account, it becomes evident that the extremum points, indicative of absolute maximums, can be qualitatively identified. For precise values, however, the equation presented must be solved using classical methods. Similarly, in Fig. 11-c, the residual values derived from the variance between actual and predicted

Table 5
The results of statistical indicators in different regression models.

Source	Std. Dev.	R-Squared
Linear	50.88	0.24
2FI	53.19	0.28
Quadratic	46.15	0.54
Cubic	55.78	0.6

Table 6
The outputs of ANOVA assessment in this study.

Source	Sum of Squares	Mean Square	F-Value	p-value
Model	42917.42	4768.60	2.24	0.0729
A-Alk. type	5827.50	5827.50	2.74	0.1165
B-Alk Conc.	154.27	154.27	0.072	0.7911
C-Time	18421.18	18421.18	8.65	0.0091
AB	4.21	4.21	1.979E-003	0.9650
AC	2772.40	2772.40	1.30	0.2697
BC	191.42	191.42	0.090	0.7680
A^2	9322.30	9322.30	4.38	0.0518
B^2	1207.60	1207.60	0.57	0.4618
C^2	9835.74	9835.74	4.62	0.0463
Residual	36210.37	2130.02		
Cor Total	79127.79			

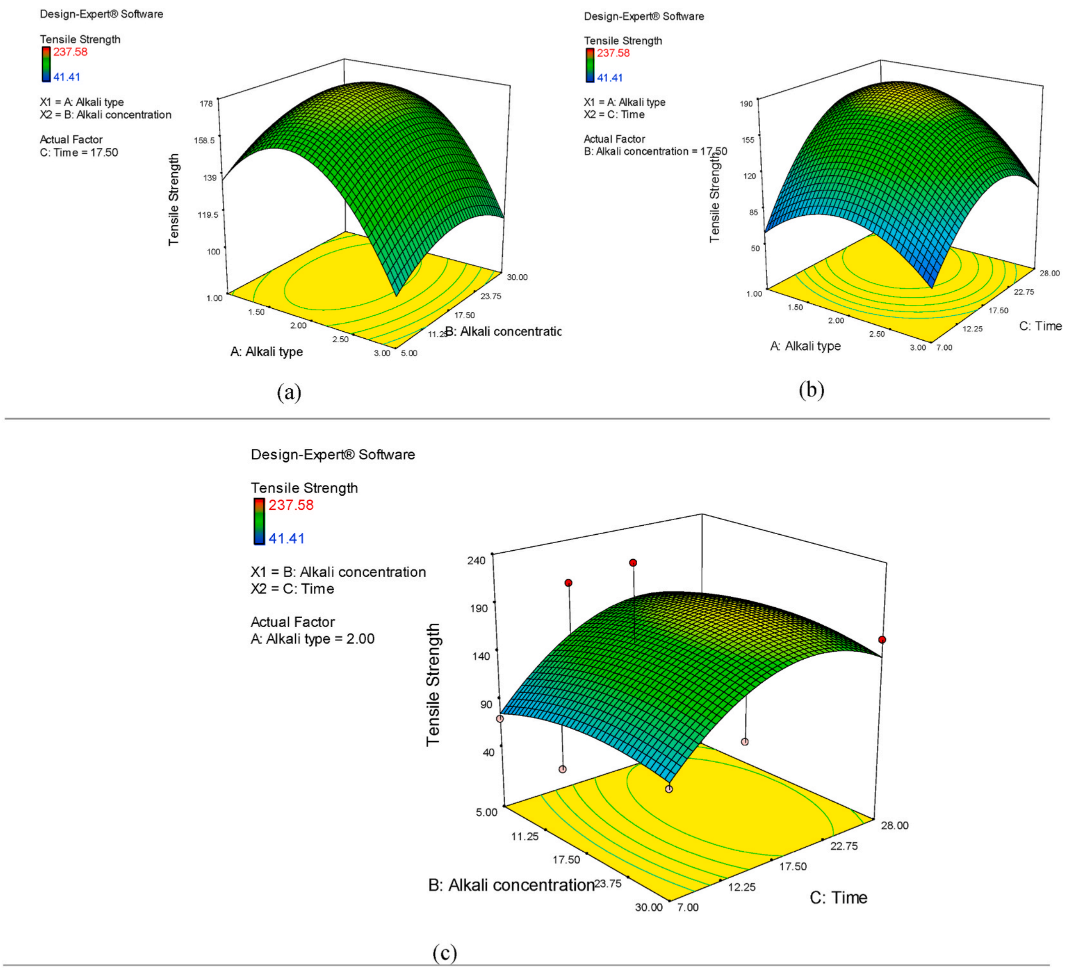

Fig. 12. The sensitive analysis of the effective features as per tensile strength (a–c).

values are observable. This suggests that the model's accuracy regarding alkali type versus time and alkali type versus its concentration is notably higher due to the insignificant residual values.

Based on the observations from Fig. 13, it is evident that the distribution of the results data in our experimental practices conforms to a normal distribution and closely adheres to the Gaussian model. This adherence to a Gaussian distribution is an important characteristic, signifying the robustness and reliability of our experimental data, which can facilitate more robust statistical analyses and inferences. The normal plot of residuals reveals a linear pattern, indicative of a well-fitted model. Moreover, the distribution of data points tends to cluster predominantly around the center and lower sections, suggesting that the majority of observations fall within these regions. This concentration is further reflected in statistical descriptors such as the mean and median, which likely exhibit values

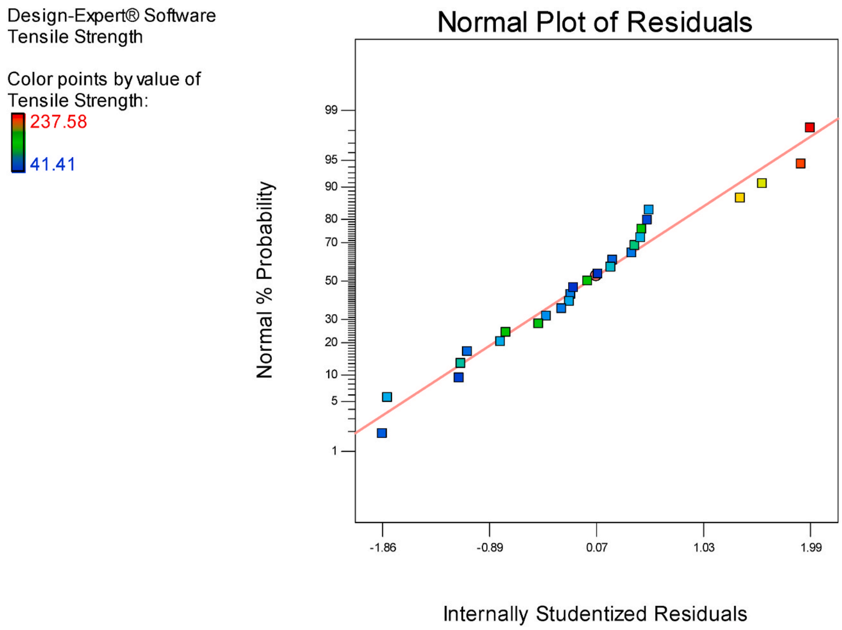

Fig. 13. The data distribution of experimental practice in the present research.

close to each other due to the symmetrical distribution of the data. The absence of significant outliers in the upper tail of the distribution reinforces the adequacy of the model without the need for transformation functions. Thus, based on these statistical insights, the data and its associated model exhibit robustness and reliability for further analysis or predictive purposes.

As illustrated in Table 7, our model has identified optimal conditions for the fiber curing process. This analysis reveals distinct characteristics for three different types of alkalis, namely NaOH (type 1), $Ca(OH)_2$ (type 3), and KOH (type 2). These findings provide critical information for selecting the most suitable alkali and its associated concentration, thereby influencing the curing time of the fibres. When using NaOH (type 1) as the preferred alkali, it is recommended to employ a concentration of approximately 23 g/L. Under these conditions, the fibres reach their desired state within a remarkably short curing time of just 7 days. This suggests that NaOH (type 1) is an ideal choice for those seeking a rapid preparation technique. Conversely, for KOH (type 2), the optimal concentration is lower, around 8 g/L, but it results in a longer curing time of 28 days. This implies that KOH (type 2) might be preferred in situations where an extended curing process is acceptable or beneficial. For those interested in using $Ca(OH)_2$ (type 3), our model suggests applying an alkali concentration of approximately 21 g/L, which is lower than that of NaOH. This offers the advantage of lower alkali concentration while still achieving effective curing. Furthermore, to provide a visual representation of the first optimal condition (type: NaOH, Concentration: 8 g/L, and Time: 7 days), we have included a surface visualization in Fig. 12.

Analysing the data presented in Fig. 14, it becomes evident that when it comes to enhancing the tensile strength of jute-based fibres, the choice of alkali treatment plays a crucial role. In this discussion, we will delve into the findings from the figure and explore the implications of these results in the context of jute fiber reinforcement. Fig. 14 clearly illustrates that there are three different alkali types under consideration: Type 1 (NaOH), Type 2 (KOH), and Type 3 ($Ca(OH)_2$). Each of these alkalis is used for fiber treatment, to evaluate the tensile strength of jute-based fibres. The tensile strength is a critical parameter in determining the durability and applicability of these fibres in various industries, including textiles, construction, and automotive.

It first need focusing on the data regarding alkali Type 1, which is sodium hydroxide (NaOH). According to Fig. 14, the use of NaOH results in the highest tensile strength among the three alkali types considered. This finding suggests that NaOH treatment effectively modifies the jute fibres, enhancing their ability to withstand tensile forces. The mechanism behind this improvement may involve the removal of impurities, lignin, and hemicellulose from the jute fibres, which leads to better alignment of cellulose chains and increased intermolecular forces, ultimately boosting tensile strength.

Moving on to alkali Type 2, potassium hydroxide (KOH), we observe a similar trend in Fig. 14. The tensile strength of jute-based fibres treated with KOH is also notably higher than that of Type 3 ($Ca(OH)_2$) treatment. This suggests that KOH treatment is an effective method for strengthening jute fibres. The chemical properties of KOH likely contribute to breaking down non-cellulosic components, thus enhancing the fibre's structural integrity and tensile strength.

In contrast, the data for alkali Type 3, calcium hydroxide ($Ca(OH)_2$), show the lowest tensile strength improvement among the three alkali types. This result implies that $Ca(OH)_2$ treatment may not be as effective as NaOH or KOH in enhancing the tensile properties of jute-based fibres. The reasons for this could be related to differences in the chemical reactions between $Ca(OH)_2$ and jute fibres, or it may indicate that $Ca(OH)_2$ treatment is less efficient in removing impurities and altering the fibre's structure compared to NaOH and KOH. These findings from Fig. 14 have significant implications for industries that rely on jute-based materials. Manufacturers and researchers can use this information to select the most appropriate alkali treatment method for their specific applications. For

Table 7
The results of optimisation in this research.

Alk. type	Alk Conc. (g/L)	Time (d)
NaOH	22.7	7.1
KOH	8.3	27.3
$Ca(OH)_2$	21.3	9.4

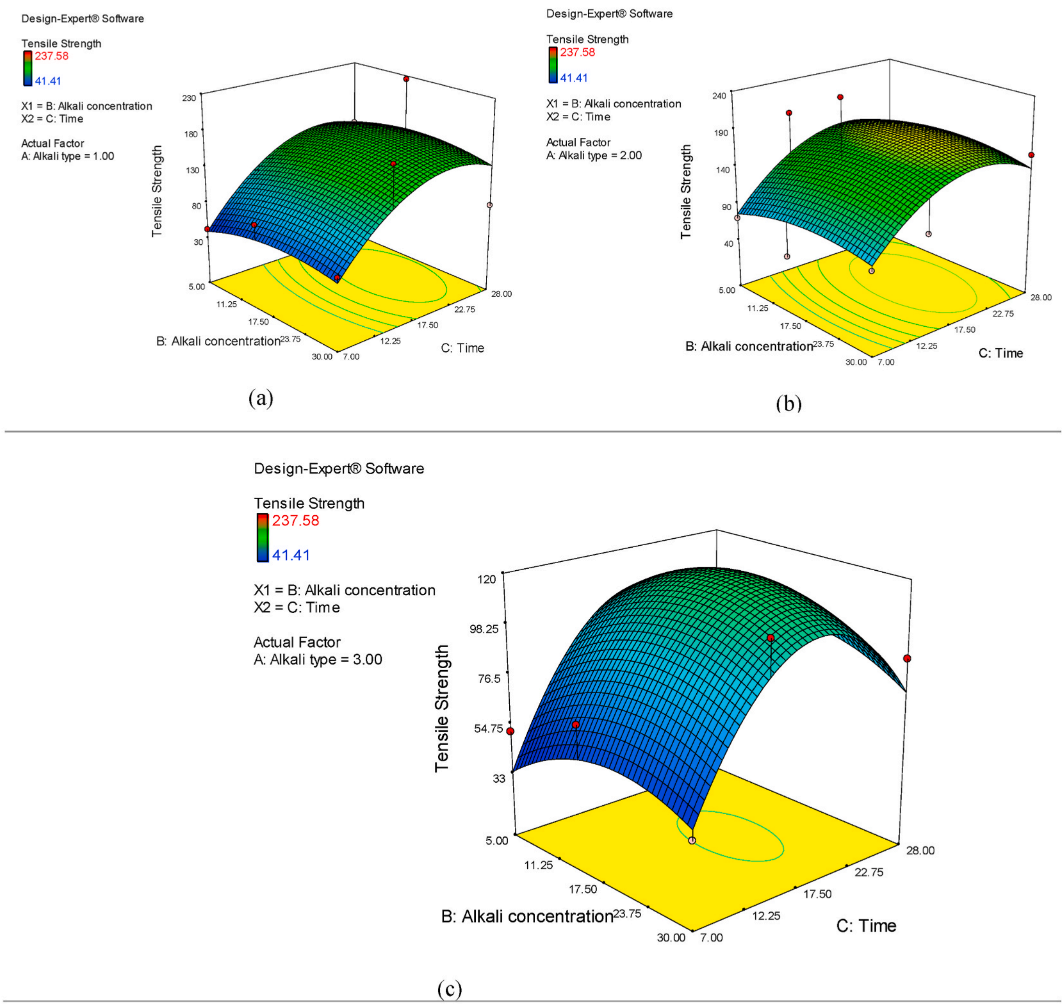

Fig. 14. The graphical result of the optimum condition of tensile strength in (a) alkali type: NaOH, (b) alkali type: KOH, and (c) alkali type: $Ca(OH)_2$.

applications where maximum tensile strength is a priority, such as in the production of high-strength textiles or composite materials, NaOH or KOH treatment should be considered as the preferred options. Conversely, $Ca(OH)_2$ treatment may find more relevance in applications where tensile strength is less critical, but other properties like flame resistance or biodegradability are more important.

The research aims outlined in this project are closely aligned with sustainability considerations, particularly in the context of jute-based fibres. The initial aim focuses on the preparation of lab-scaled jute-based fiber samples within alkali environments, with a specific emphasis on assessing tensile strength tolerance using the OFAT method. This pursuit is integral to enhancing the durability and performance of jute-based materials, thus contributing to sustainability by potentially extending their lifespan and reducing the need for frequent replacements [73]. Another crucial aspect of the research involves the characterisation of the prepared samples through advanced techniques such as DSC, TGA, and SEM instruments. This characterisation process offers insights into how jute-based fibres behave under varying conditions. Such knowledge is instrumental in optimising their utilisation across diverse applications, potentially resulting in more sustainable end-products. For instance, understanding thermal properties can facilitate the creation of energy-efficient manufacturing processes [74]. The research project also emphasises the optimisation and mathematical modelling of key factors affecting tensile function using the RSM technique. This endeavour has the potential to yield highly efficient processes for jute-based fiber production and application, leading to reduced resource consumption and waste generation. Analysing historical data provides the opportunity to identify patterns and trends that can inform sustainable practices, further enhancing the project's contribution to sustainability [75,76].

Various studies have explored different aspects of cellulose fibers, as depicted in Fig. 15. Kumar, Singh and Sarwade [77] investigated the evolution of tensile modulus during the aging of composites containing commercial microcrystalline cellulose powder over a span of 300 h. This study shed light on how mechanical properties change over time. Kawashima and Shah [78] examined cellulose fibers' influence on early-age shrinkage behavior, focusing on a timeframe of approximately 10 days. Parameters such as fiber percentage were analyzed for their effect on tensile strength variations during this period. Furthermore, Cai et al. [79] conducted a study on flax fiber reinforced phenolic composites, investigating changes in hydrothermal aging across different temperatures over a duration of 160 weeks. This long-term study provided insights into the durability of such composites under varying environmental conditions.

In another facet of research, the present study focused on the effects of three alkali environments on the treatment of jute fibers. Sensitivity analysis revealed that curing time and alkali type are crucial factors affecting the tensile strength of the samples. Notably, the results indicated that the highest tensile strength was observed in the KOH environment after approximately 14 days, reaching 237.5 MPa. For NaOH treatment, the peak tensile strength occurred on the 28th day, with a value of 225 MPa. Finally, with $Ca(OH)_2$ treatment, the highest tensile strength was observed on the 14th day, albeit with a lower value of 111 MPa. These findings underscore the importance of both the type of alkali treatment and the duration of exposure in enhancing the mechanical properties of jute fibers.

4. Conclusions

The utilisation of green materials represents a pivotal exemplar of sustainability, both in developed and developing nations. Among these, jute stands out as a particularly promising and cost-effective resource for fiber production in various building and construction applications. This study, in particular, explored the optimisation of jute-based materials through diverse alkali treatments, exploring the impact of different alkali types, concentrations, and curing times on tensile strength. Results demonstrated that fibres treated with 15 g/L concentration of NaOH had the highest tensile strength at 28 days (225.05 MPa). The highest tensile strength of jute fibres treated with KOH was observed with 15 g/L concentration at 14 days (237.58 MPa). For $Ca(OH)_2$ treatments, the highest tensile strength was observed with 30 g/L concentration at 14 days (111.28 MPa). It was also observed that prolonged exposure to very high alkali concentrations could adversely affect the tensile strength as observed in case of 30 g/L concentration of NaOH at 28 days (84.73

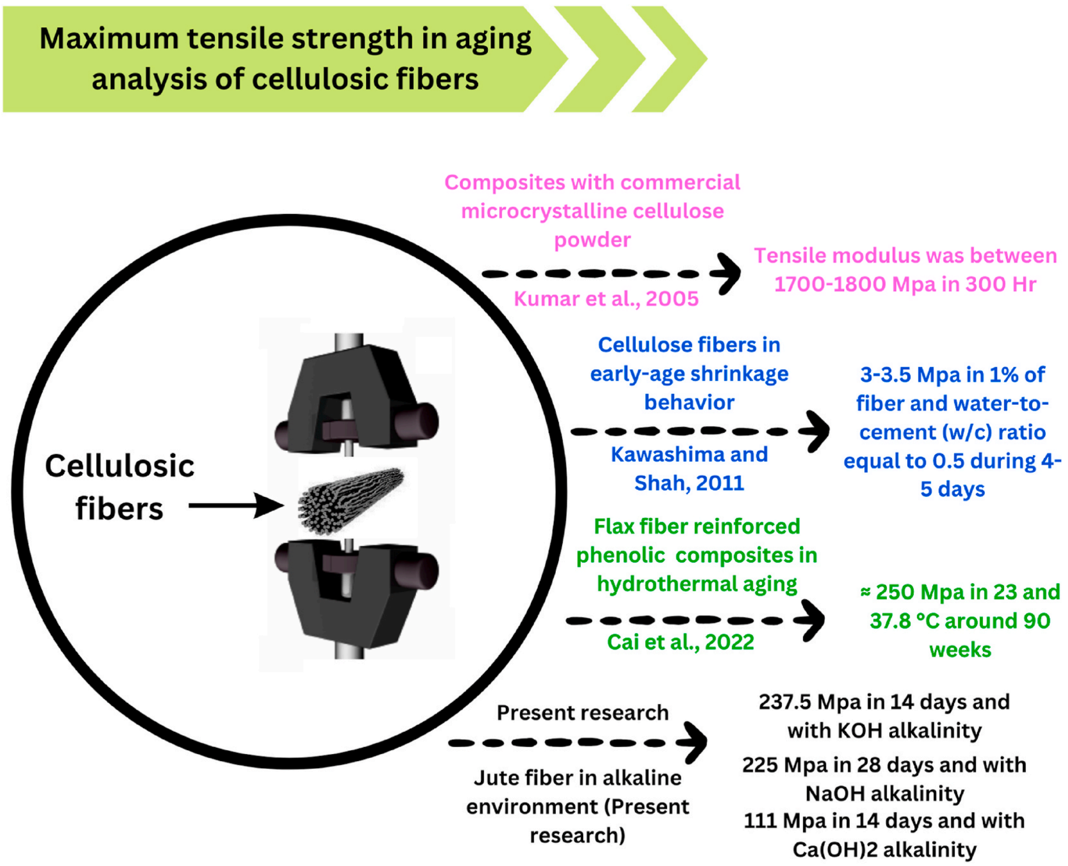

Fig. 15. The comparison of main outputs about cellulose fiber tensile strength evaluations in different studies.

MPa). The RSM analysis showed that the maximum effect on tensile strength is related to time, while the minimum effect is related to alkalinity concentration. For example, with NaOH as the primary suggested alkalinity, the tensile strength changes by around 70 % when transitioning from a 7-day to a 28-day curing time. Similarly, changing the NaOH concentration between 5 g/L and 30 g/L results in a change of around 30 %.

The findings of this research shed light on the crucial role of sodium hydroxide (NaOH) as the most influential alkali compound for enhancing the tensile strength of jute-based samples. Through rigorous statistical analysis, it was determined that curing time emerged as the predominant factor affecting tensile strength performance, as demonstrated by ANOVA assessment.

Looking ahead, future studies could explore the survey of more detailed insight into the jute fibres composition as well as incorporation of alternative cellulose fibres as fillers for composite materials, diversifying the scope of sustainable options. Additionally, the application of electrolysis and heat treatment techniques holds promise in enhancing the properties of jute-based fibres, warranting attention from the scientific community. As a departure from the RSM model, the utilisation of metaheuristic algorithms emerges as an enticing avenue for optimising fiber characteristics, offering new insights and avenues for refining the sustainable potential of jute-based materials. These collective efforts promise to further elevate the role of green materials in advancing sustainability objectives on a global scale.

CRediT authorship contribution statement

Aamir Mahmood: Conceptualization, Data curation, Formal analysis, Visualization, Writing – original draft. **Miroslava Pechočiaková:** Methodology. **Muhammad Tayyab Noman:** Validation, Writing – review & editing. **Stanisław Wacławek:** Validation, Writing – review & editing. **Mohammad Gheibi:** Conceptualization, Formal analysis, Investigation, Methodology, Visualization, Writing – original draft. **Kourosh Behzadian:** Validation, Writing – review & editing. **Jakub Wiener:** Validation, Writing – review & editing. **Jiří Militký:** Validation, Writing – review & editing.

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

Data will be made available on request.

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