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Investigating the Effects of Alkaline Aging on Jute Fibers and Their Reinforcement Capabilities in Modified Cement Composites

Author

Aamir Mahmood, M.Sc.

SUMMARY OF THE THESIS

Title of the thesis:	**Investigating the Effects of Alkaline Aging on Jute Fibers and Their Reinforcement Capabilities in Modified Cement Composites**
Author:	**Aamir Mahmood, M.Sc.**
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Department:	**Department of Material Engineering**
Supervisor:	**Ing. Miroslava Pechočiaková, Ph.D.**

Committee for defense of the dissertation

Chairman: prof. Ing. Michal Vik, Ph.D.	FT TUL, Department of Material Engineering
Vice-chairman: doc. Ing. Lukáš Čapek, Ph.D.	FT TUL, Department of Technology and Structures
prof. Ing. Luboš Hes, DrSc., Dr.h.c.	FT TUL, Department of Textile Evaluation
prof. Ing. Michal Šejnoha, Ph.D., DSc.	CTU in Prague, Faculty of Civil Engineering, Department of Mechanics
doc. Ing. Antonín Potěšil, CSc.	LENAM, Ltd.
doc. Ing. Veronika Tunáková, Ph.D.	FT TUL, Department of Material Engineering
Ing. Miloš Jerman, Ph.D. (oponent)	CTU in Prague, Faculty of Civil Engineering, Department of Materials Engineering and Chemistry,

opponent who is not a member of the committee

Prof. Dr. Yasir Nawab	Department of Textile Engineering, National Textile University Faisabalad

The dissertation is available at the Dean's Office FT TUL.

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Annotation

This thesis is divided into two main parts. The first part investigates the degradation (refining) of jute technical fibers under alkaline conditions. Various types of alkali concentrations, and treatment times were systematically reviewed to assess their effects on fiber strength. Results indicate that fibers treated with NaOH significantly reduced the tensile strength over time. Sodium hydroxide was found to be the most aggressive alkali according to Response Surface Methodology (RSM) analysis.

The second part of the study evaluates the effects of fly ash (FA), Laponite (LAP), and Bentonite (BENT) on the mechanical properties of cement paste. Fly ash improved 3-point bending stress but reduced compressive strength and toughness. Conversely, laponite negatively affected all properties, but 1% of Laponite showed a maximum value for all the functions. while Bentonite enhanced both 3-point bending stress and compressive strength. Statistical regression and Ordered Weighted Averaging (OWA) models indicated that a mixture of 5% fly ash and 1% Laponite was optimal for construction purposes, balancing safety and performance. The best performance in terms of 3-point bending, compressive strength, and toughness was observed in cement mixtures containing 5% fly ash and 1% Laponite, reinforced with different amounts of jute fibers.

Keywords: Aging behavior, Jute fiber, Alkali treatment, Mechanical properties, Response Surface Methodology, Cement mixture, Fly ash, Laponite, Bentonite, Ordered Weighted Averaging, Environmental Impact Assessment.

Anotace

Tato diplomová práce je rozdělena na dvě hlavní části. První část zkoumá degradaci (zjemňování) technických jutových vláken v alkalických podmínkách. Různé koncentrace alkálií a doby působení byly systematicky přezkoumány za účelem posouzení jejich vlivu na pevnost vláken. Výsledky ukazují, že vlákna ošetřená NaOH výrazně snížila pevnost v tahu v průběhu času. Hydroxid sodný byl podle analýzy metodou “Response Surface Methodology” (RSM) nejagresivnějším alkáliem.

Druhá část studie hodnotí účinky popílku (FA), Laponitu (LAP) a Bentonitu (BENT) na mechanické vlastnosti cementové pasty. Popílek zlepšil pevnost při tříbodovém ohybu, ale snížil pevnost v tlaku a houževnatost. Naproti tomu Laponit měl negativní vliv na všechny vlastnosti, ale 1 % Laponitu vykázalo maximální hodnotu u všech funkcí. Bentonit zlepšil jak pevnost při tříbodovém ohybu, tak pevnost v tlaku. Statistické regresní modely a modely „Ordered Weighted Averaging “(OWA) naznačily, že směs s 5 % popílku a 1 % Laponitu je optimální pro stavební účely, vyvažující bezpečnost a výkon. Nejlepší výsledky z hlediska tříbodového ohybu, pevnosti v tlaku a houževnatosti byly pozorovány u cementových směsí obsahujících 5 % popílku a 1 % Laponitu, zesílených různými množstvími jutových vláken.

Klíčová slova: Stárnutí materiálu, Jutové vlákno, Alkalické ošetření, Mechanické vlastnosti, Metoda Response Surface, Cementová směs, Popílek, Laponit, Bentonit, Ordered Weighted Averaging, Hodnocení dopadu na životní prostředí.

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1 Introduction

Industrialization and growth are another important factors that have raised many concerns over the increasing degradation of the environment over the years across the globe. As the world population is increasing and with the coming up of industries, there is a need to expand construction facilities. In the same way, through emerging construction activities, more pressure is being placed on natural resources; the construction industry is among the major negative contributors to the environment through utilizing a large percentage of raw natural resources and releasing a high amount of greenhouse gases [1]. Cement concrete being the widely used construction material is not without a few drawbacks at least within the context of this research; it is a brittle material that cracks easily, possesses low tensile strength, and endures early failure due to freeze-thaw cycles and chemical attack. According to estimations, concrete production itself contributes to the contribution of global CO_2 emissions that range from 5% to 8%. Out of these, 95% of carbon dioxide emissions are associated with the production of cement which is the key material used for concrete production [2]. It has been established that the process of manufacturing Portland cement leads to emissions almost similar to carbon dioxide for every individual cement produced. Furthermore, apart from being a major source of CO_2 release in concrete production, the process also consumes a lot of raw materials, depleting natural resources and polluting our environment. For instance, in making Portland cement, it is often observed that the raw material consumption is approximately double the amount of cement produced [3]. Hence, pursuing the efficient use of other materials that at least partially or totally act as cement is extremely significant to minimize the influence of cementitious materials. Under this condition, there are a range of materials such as Waste and Clays replacing cement which should be further explored [4]. By using these waste materials in cement mixtures one gets to reduce the use of cement required for the construction as well as offer a sustainable, functional way to dispose the waste products and yet going a long way in preserving natural resources. The other possibility for the use of clays in concrete is that they can act as cement replacement material. Calcined clay or metakaolin has additional properties, namely the pozzolanic properties, which enable the clay to react with calcium hydroxide thus producing more cementitious compounds [5]. As mentioned above concrete has relatively low tensile strength, Earlier used materials for the reinforcement of concrete include steel; however, due to the advancement of fibre technology the modern construction world has more fibres including polypropylene, glass, carbon, basalt, jute and many more [6]. These fibres improve strength, durability, and toughness of constructions materials [7]. Natural fiber, which has always been appreciated in the textile industry, is now widely incorporated in construction, particularly cementitious composites [8]. Apart from enhancing the efficiency of structures, they help enhance the sustainability of the building industry. Developing natural and

fibres or using natural fibre blends can reduce the carbon footprint [9]. With advancing technology fibers' importance is expected to revolutionize construction and make structures to be stronger, more environmentally friendly, and efficient.

Cementitious composites are highly alkaline [10] which can cause different levels of degradation in fibres with varying polymeric compositions. Therefore, it is essential to investigate the performance of various fibres when added to concrete. Some fibres are chemically inert, making them more stable compared to others [11]. Thus, understanding the durability and life-cycle performance of fibres in cementitious composites remains a significant technical challenge [12][13].

1.1 Purpose and Aim of the Thesis

The current research is intended to analyze the effect of aging with alkaline solutions, including different concentration levels and exposure periods of NaOH, KOH, and $Ca(OH)_2$ solutions on fibre jute properties and mechanical features. It also explores the influence of Supplementary Cementitious Materials (SCMs) on the mechanical characteristics and durability of jute fibre reinforced cement paste keeping into consideration the characteristic such as compressive strengths, flexural strength, and toughness. Furthermore, in order to study the fundamental information of the morphological and composition changes occurred within the cementitious matrix this study will employ analysis using scanning electron microscopy (SEM) and particle size distribution studies.

2 Overview of the Current State of the Problem

The construction Industry is one of the largest industries all-over the world and going on with the increase in human population and Structural development. It is worth to emphasize that this industry is heavily rely on natural resources with the predicted virgin aggregates production within the next 13 years estimated at around 60 billion tons worldwide [14]. According to the sources, the construction industry and building materials around the world have been projected to grow at the rate of 4.2 % from 2018 to 2023, attaining an estimated value of $ 10.5 trillion By 2023 [15]. As construction demands are increasing with time, its impact on the environment is alarming; it is therefore very relevant to look for materials that help reduce impacts on the environment. As it stands today, most constructions are done using conventional materials that are not environmentally friendly to cater to the growing needs of the developing society [16].

It is worth noting that construction activities in the world produce around 1 billion tonnes of masonry and concrete waste [17]. The major energy consumption is in the process of production of cement clinker, which ranges between 20 - 40% percent of the total energy in the cement industry [18]. Cement production is one particular industrial process through which a huge amount of CO_2 is emitted and accounts for about 5% – 7% of global CO_2 emissions while CO_2 estimated contributes 65% of global greenhouse gases [19]. Interestingly, to create one tonne of cement, 0.8 tonnes of CO_2 is released to the atmosphere [20]. Concrete is a material that does not possess the elasticity to support tensile forces, which is why it requires reinforcement. In this regard, conventional deformed steel bars are the ones in use. Yet of high importance is the fact that steel production is an energy demanding process that contributes to enormous carbon emissions. In 2016 there was production of 1,202 million tonnes of steel. According to the steel production statistics, around 2.3 tonnes of CO_2 emission is released for every tonne of steel produced [21]. This situation shows how important it is to create and use materials that are satisfactorily both in terms of sustainability and expenditure. Among the solutions, one is the use of waste and other materials in the creation of concrete. Disposal of industrial by-products is becoming voluminous, expensive, and complex due to the high costs of treatment involved, landfill operating costs, and scarcity of available disposal sites. Therefore, they encourage the use of industrial by-products in construction as it serves as a good option [22]. The outcome of the properties of concrete is the work of waste materials from industries, including fly ash, which has been investigated by previous researchers extensively [23][24]. Using these materials as a replacement or partial replacement for cement in cementitious materials helps solve the waste disposal problem and reduces the energy demand needed for cement production, thereby decreasing carbon emissions. Investing in such materials is crucial for addressing both environmental sustainability and the mechanical properties required for construction. The following sections will discuss the overall properties of fly ash, laponite, and bentonite.

Concrete, primarily composed of cement, is the most widely used material in construction activities. It is estimated that concrete is used at a rate of one ton per person on Earth [25]. However, concrete is known to be a brittle material with low tensile strain and strength capacities [26]. Its low toughness and susceptibility to cracking limit its applications. The propagation of cracks further deteriorates its mechanical properties, compromising the safety of structures [27][28]. Consequently, concrete can only be used in non-critical sections with small gravity loads [29]. For critical infrastructure development, concrete requires reinforcement to be effective. Typically, this is achieved by embedding deformed steel bars or welded wire fabric into freshly cast concrete [30]. The following are the issues related to this type of reinforcement: their costs are relatively high as compared to other rebars, they are prone to corrosion, and are quite dense. Also, steel production has been widely known to call for large quantities of energy and therefore lowers the well-being of our environment. Industrialized production of iron and steel is one of the most energy and carbon intensive ones across the globe and the manufacturing processes are still coal-dependent and contribute immensely to the emission of CO_2 [31]. The manufacturing industry across the globe is currently emitting up to 40 percent of the overall global emissions as estimated by the IEA, with the iron and steel sector manufacturing industry representing the highest emissions of over 27 percent of carbon emissions from manufacturing industries globally [32][33].

Keeping in view the problems mentioned above, there is a need to apply the concept of reducing, reusing, and recycling in the construction industry and material fabrication [34] to cater to the growing environmental challenges in a more sustainable way [35]. Therefore, the construction industry's interest in innovative sustainable solutions from recycling and reusing processes with minimum energy intake and reduced carbon emissions has been increasing day by day [36]. In this sense, fibers are getting their way as a promising alternative to steel reinforcement [37]. Cementitious materials incorporated with certain types of fibers have shown an improvement in many properties of the material, including toughness, energy absorption capacity, post-cracking residual strength, decreased shrinkage potential, and enhanced durability [38]. Previously, many types of short fibers, like asbestos, steel, glass, and polymeric, have been employed as reinforcing elements in cement-based composites. These fibers, besides their advantages, have shown some disadvantages, such as detrimental health effects associated with asbestos, high costs associated with steel and polymeric fibers, as well as a notable environmental footprint [39][40].

In cementitious composites, some alkalis that are contained in the materials take on a major role during the process of cement hydration. During cement hydration, it releases calcium ions and hydroxide ions to form a paste with the name of calcium hydroxide, commonly referred to as Portlandite. This is due to the high of alkalinity of the cement paste that is an essential factor for density of the Calcium Silicates Hydrates (CSH) gel [41]. The elements such as sodium and potassium, can dissolve in cementitious mixtures to form NaOH and KOH [42], [43]. Some of these alkalis can prompt to hydrate at a faster pace, hence improving early-age strength. Nevertheless, the use of high concentrations of NaOH and KOH may cause some negative effects, like the Alkali-Silica Reaction (ASR), which is the reaction of some reactive silica in the aggregates with these chemical reagents. The gel formed due to ASR, when exposed to moisture swells forcing more pressure internally, leading to cracks and overall weakening of the concrete [44]. Therefore, it is important to study the effects of alkaline environment on the properties of the fibres. The present thesis focuses on two important issues described earlier namely replacing cement with waste and clay materials and studying the behaviour of natural fibres in alkaline environments from the perspective of their use in cementitious materials.

Jute is an abundant, biodegradable, and natural fiber and because of these attributes, it can be a worthy green option for synthetic fiber reinforcement and also offers unique advantages when incorporated into cementitious composites. The use of jute in conjunction with cementitious composites is certainly the focus of the current modernization of the construction industry in terms of utilizing a sustainable approach to using renewable materials in the creation of construction elements that possess high durability and sustainability. Furthermore, it has several important applications [45], [46]. Jute is an important bast fiber which is largely formed of cellulose and other non-cellulose fragments. The non-cellulose parts are lignin, pectin, and hemicellulose [47]. Because of this, jute fiber has several characteristics that make its use in concrete complex and require careful consideration, especially in the way the fiber reacts in an alkaline environment and the fact that concrete is an alkaline material [48]. Under such conditions, it is a common phenomenon that jute fibers tend to undergo degradation because of hydrolytic action on hemicellulose and lignin present in the fiber [49].

Cellulosic fibers used in the fabrication of fiber-reinforced cementitious composites require little energy to process [50]. One of the uses of cellulosic fibres in the construction and building industry is aimed at reinforcing cementitious composites while replacing traditional reinforcing materials thus reducing the dependence on traditional materials to reduce carbon emissions, decrease waste generation, and improve 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 [51]. Cementitious composites having short and long cellulosic fibres affect positively the flexural strength of the matrix [52]. 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% [53]. Ramakrishna and Sundararajan revealed that the toughness of cement mortar reinforced with cellulosic fibers increased 3-18 times that of the samples without reinforcement [54]. 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 [55].

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 [56]. 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 hemicelluloses, 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 [57]. Alkaline mercerization causes morphological alterations in cellulosic fibers. Morphological changes in cellulose fibers are first caused by swelling and then by longitudinal fibers. Shrinkage produces circular structures after elliptic structures first [57]. Changing of morphological structure into a circular one can be useful for increasing the mechanical properties of some types of concrete [58].

3 Materials and Methods

Tables 3.1 and 3.2 describe the materials and instruments used in the study of aging behavior of technical jute fibers in an alkaline environment. this study and the instruments for measuring certain properties.

Table 3. 1 Materials used for aging of jute fibers.

Material	Company
NaOH	Lach-Ner, Czech Republic
KOH	Lach-Ner, Czech Republic
$Ca(OH)_2$	Lach-Ner, Czech Republic
Jute Fiber	Saifan, S.R.O, Czech Republic

Table 3. 2 The applied instruments during this study.

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

The steps involved in this study are illustrated in Fig. 3.1, showing the aging process of jute fibers in an alkaline medium in three steps: sample preparation and characterization, optimization using HDA-RSM, and prediction system execution.

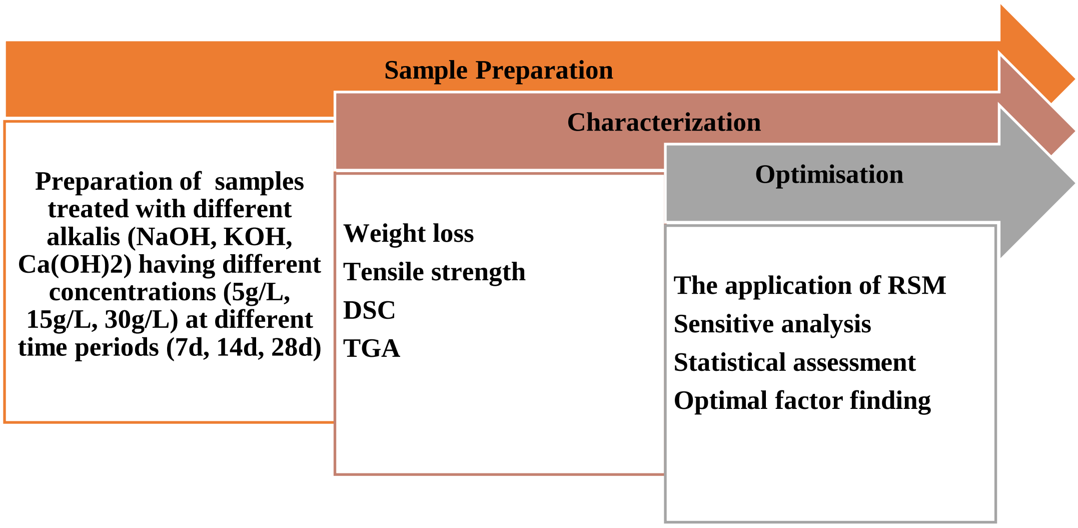


Figure 3. 1 The research roadmap of the present study including sample preparation, characterization, and optimization process.

A preparatory step involved was to obtain twenty threads of jute fiber, each with measurement of fifty centimeters. For preparation of alkali solution, the distilled water was used. Afterward, a 200ml container was used to soak the jute fiber in the prepared solution. The samples were then stored for 7, 14, and 28 days at 23 ^{o}C temperature. The samples were kept in the treatment for some time and after that, the samples were washed thoroughly with distilled water and then air-dried.

The stages of experimental sample preparation and data gathering in this study are shown in Fig. 3.2. In the experimental stages (Fig. 3.2), jute fibres along with solutions having different concentrations (5g/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 emphasize 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. Fig. 3.3 presents that after sample preparation, three characterisations including TGA, SEM, and DSC are first carried out on the samples to observe the behaviour 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. All experimental practices of this study are done based on ČSN EN ISO 2062 [59], ČSN EN ISO 5079 (*Textiles - Fibres - Determination of strength and ductility of individual fibres at break*, 2021), ČSN EN ISO

11358-1 [61], ČSN EN ISO 11357-1 [62], and ISO/TS 21383:2021 [63].

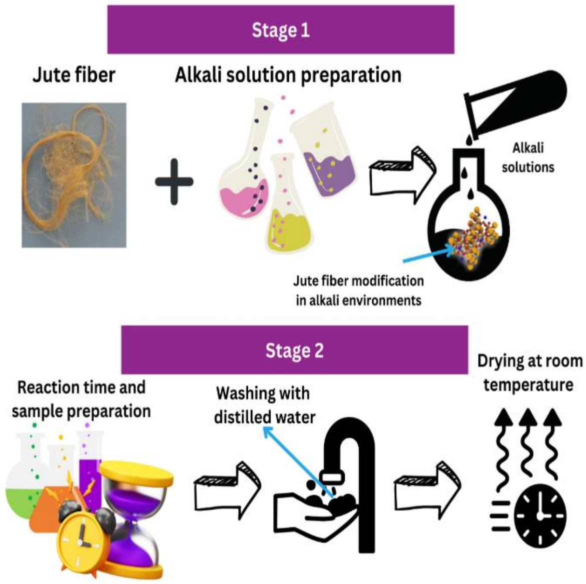


Figure 3. 2 The schematic plan of sample preparations in this study

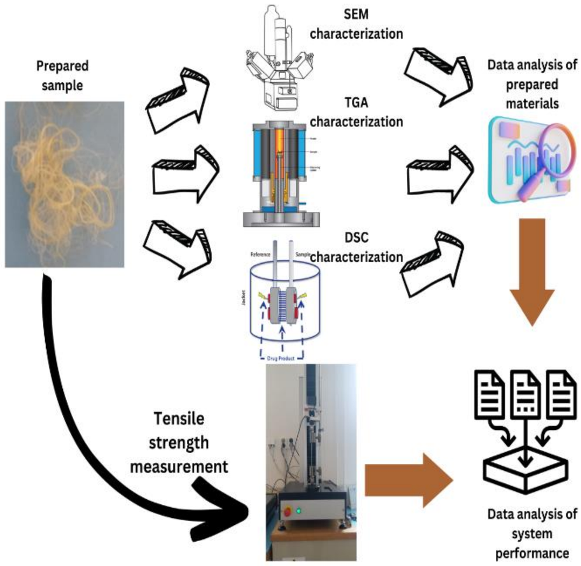


Figure 3. 3 The steps of various tests and analyses on the sample in this study

For the partial replacement of cement with Fly ash and clays and their reinforcement with jute technical fibers, applied materials are illustrated in Table 3.3.

Table 3. 3 The specifications of applied materials in the study.

Material	Specification
Cement	Ordinary Portland Cement ČSN EN 197-1, Denmark
Fly ash	Fly ash for concrete as per DIN EN 450, Betoment OP Germany

Laponite RD	SYnL-1 (Synthetic layered silicate, Hydrous Sodium Lithium Magnesium Silicate), Clay Minerals Society Source Clays Repository P.O.Box 460130, Aurora, Colorado 80046-0130 USA
Bentonite	Bentonite Clay Ekokoza s.r.o, Czech Republic
Jute Technical Fiber	Saifan, S.R.O, Czech Republic

The instruments used in this research are listed in Table 3.4.

Table 3. 4 The applied instruments in the study.

Device	**Specification**
Measuring Scale	Table Digital Accurate**,** Czech Republic
Mixer	KENWOOD XL TITANIUM, Great Britain
Vibrating Table	VSB-40 NS, Brio Harnice s.r.o., Czech Republic
Universal Testing Machine	Tira TEST 2300, Germany
Charpy Hammer LAB TEST	CHK 50J LABOR Tech, Czech Republic
SEM and EDS	VEGA3, TESCAN, Czech Republic
PAMAS SSBS particle counting system	PAMAS SLS-25/25, Germany

The research roadmap of this research is depicted in Fig. 3.4.

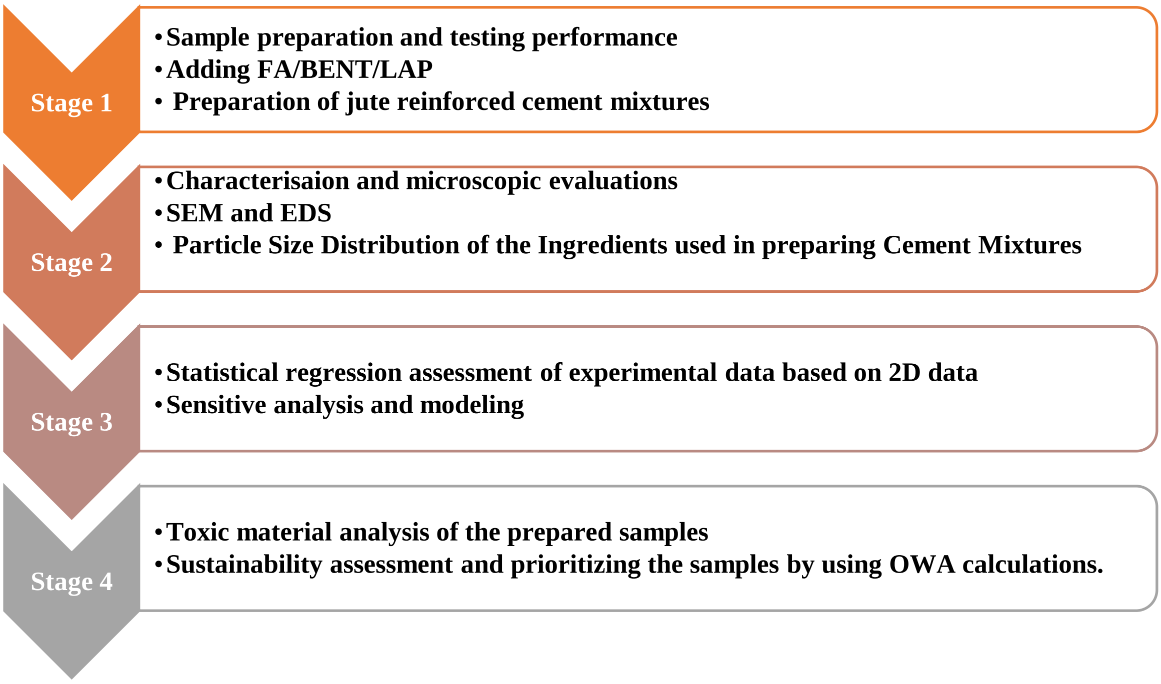


Figure 3. 4 The research roadmap of the investigation

Due to experimental activities in the present investigation, three protocols are applied for sample preparation using mixer (ČSN EN 1008 (732028))[64], determination of flexural and compressive strength of hardened mortars (ČSN EN 1015-11 (722400)) [65], and purpose of impact strength by the Charpy method (ČSN EN ISO 179-2 (640612)) [66]. For particle size distribution calculations, PAMAS SSBS particle counting system followed by ISO 4406 was utilized. The stages of sample preparation and experimental practices are shown in Figs. 3.5 and 3.6, respectively. According to Fig. 3.5, in the first step, different samples were mixed with individual formulation, demonstrated as per Table 3.5.

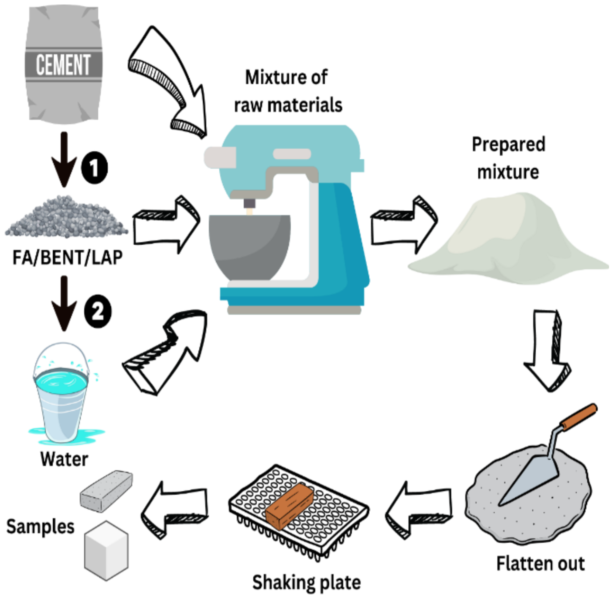


Figure 3. 5 The process of sample preparation in this research.

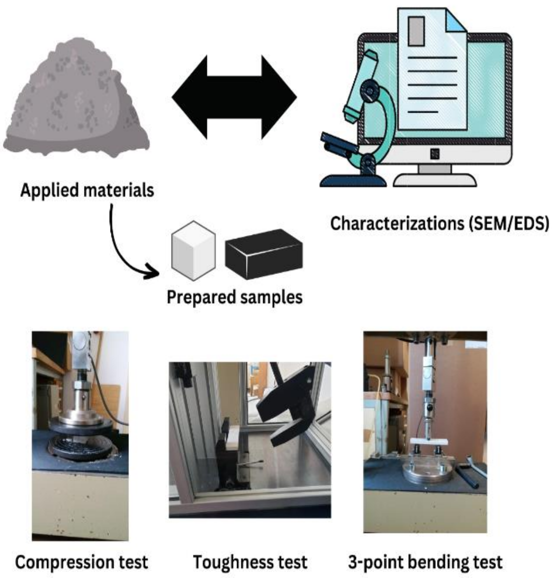


Figure 3. 6 The experimental performance assessment stages of cement paste in the investigation.

It should be noted that in all the samples in Table 3.5, the Water to Binder Ratio (WBR) was equal to 0.4. The prepared samples were cast in simple rectangular and cube shapes

(30mm*30mm for cubes and 140mm*30mm*10mm for rectangular-shaped samples) following the shaking of the samples for uniform distribution and compaction. The samples were then cured for 28 days under standard conditions.

Based on Fig. 3.6, the applied materials were characterized by both SEM and EDS tests. Likewise, the cured samples were utilized for three mechanical tests, including 3-point bending, toughness, and compression tests in the lab.

Table 3. 5 Samples with different fillers in the present study.

Sample name	Fly Ash (FA)	Laponite (LAP)	Bentonite (BENT)
Sample 1 (S1)	5%	0	0
Sample 2 (S2)	10%	0	0
Sample 3 (S3)	20%	0	0
Sample 4 (S4)	0	1%	0
Sample 5 (S5)	0	3%	0
Sample 6 (S6)	0	5%	0
Sample 7 (S7)	0	0	1%
Sample 8 (S8)	0	0	3%
Sample 9 (S9)	0	0	5%

In the following, the obtained results of experimental practices are modelled with linear regression models. Regression analysis was conducted using Excel software. First, the data were categorized, and a curve fitting was done between different percentages of fillers and cement paste functions separately. After quantifying the Environmental Impacts (EIs), the nine prepared samples (Table 3.5) were evaluated concerning sustainability criteria, including economic and EI performance criteria [67].

In the next step, cement mixtures having fly ash and laponite as partial replacements of cement and reinforced with jute fibers (approximately 12 mm in length) were realized. Cement was replaced by 5% fly ash and 1% laponite in each sample. Jute fiber was added to the cement mixture in fractions of 0.2wt.%, 0.5wt.%, 0.7wt.% and 1wt.%. The prepared samples were tested for 3-point bending stress, compressive strength, and toughness properties. Table 3.6 represents the samples with different percentages of the jute fibers. Particle size distribution of cement, fly ash, laponite, and bentonite, a fixed amount of each material (in our case, 0.299 g) was mixed in 100 ml of isopropyl alcohol. The mixture was mixed well and was put in PAMAS particle counting system. Stirring in the system was adjusted accordingly and three readings for each sample were taken.

Table 3. 6 Samples with different percentages of jute fiber in the present study.

Sample name	Fly Ash (FA)	Laponite (LAP)	Jute Fiber
Sample 1 (S1)	5%	1%	0.2%
Sample 2 (S2)	5%	1%	0.5%
Sample 3 (S3)	5%	1%	0.7%
Sample 4 (S4)	5%	1%	1%

4 Results and Discussion

The weight loss of jute fibers treated with various concentrations of NaOH, KOH, and $Ca(OH)_2$ over different time durations is illustrated in Fig. 4.1. It is evident from Fig. 4.1a that weight loss increases with both concentration and duration. The highest weight loss is observed at a 30g/L concentration over 28 days, reaching up to 19.17%. Even after 7 days, NaOH causes significant weight loss, with 13.13% at a 30g/L concentration. The pattern for KOH, as shown in Fig. 4.1b, is slightly different, with no consistent trend, but weight loss percentages are relatively similar across different concentrations, especially for longer durations. In contrast, $Ca(OH)_2$ shows the lowest weight loss percentages across all conditions compared to NaOH and KOH, as indicated in Fig. 4.1c. 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 [68].

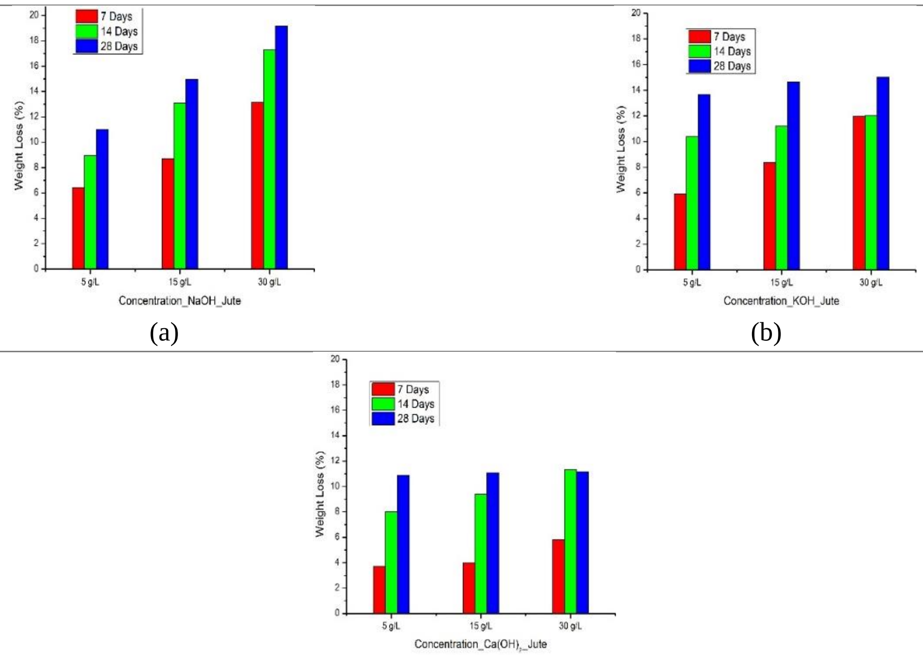
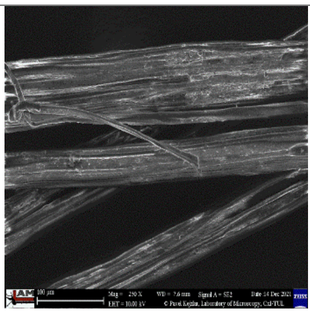
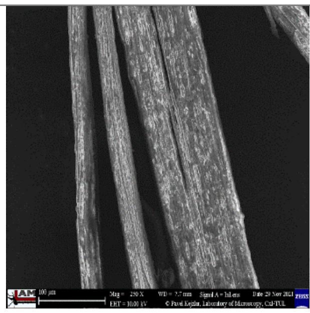


Figure 4. 1 The Weight loss (%) of different alkali treated jute fibres by (a)NaOH, (b) KOH and (c) $Ca(OH)_2$.

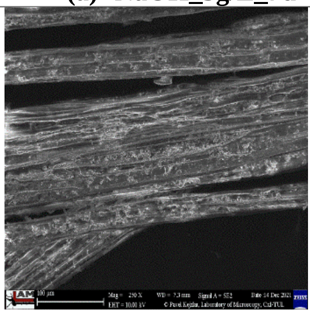
Fig. 4.2 presents the results of SEM characterisations of jute fibres based on different alkali solutions with different concentrations. According to Fig. 4.2, all SEM images are reported in 250 X.



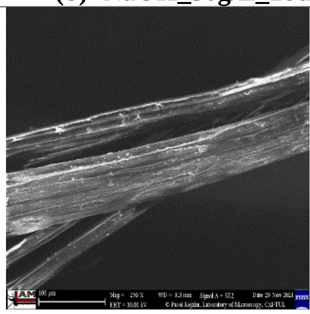
(a) NaOH_5g/L_7d



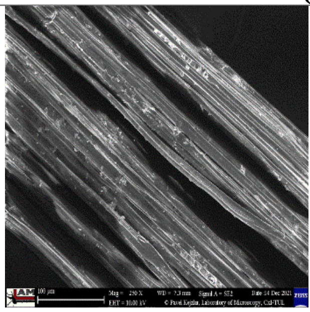
(b) NaOH_30g/L_28d



(c) $Ca(OH)_2$_5g/L_7d



(d) $Ca(OH)_2$_30g/L_7d

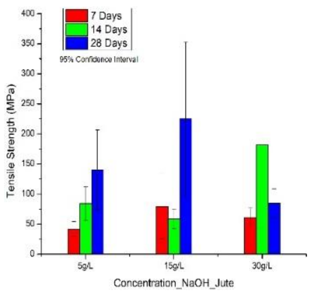


(e) KOH_5g/L_7d

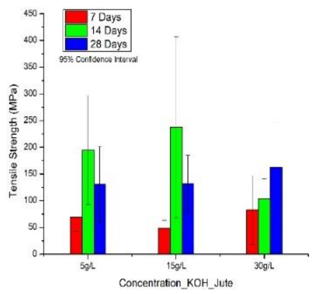
Figure 4. 2 The SEM outputs of different alkali-treated jute fibres (a,b) NaOH (c,d) $Ca(OH)_2$ (e) KOH.

The outcomes of OFAT experiments indicate that the tensile strength of jute fibers varies with NaOH concentration and exposure duration. At 7 days, the tensile strength peaks at 15g/L (79.66 MPa) and decreases at higher concentrations. At 14 days, the highest strength is observed at 30g/L (181.27 MPa), but at 28 days, 15g/L again shows the highest tensile strength (225.05 MPa). Longer exposure to moderate NaOH concentration (15g/L) generally benefits tensile strength, while very high concentrations (30g/L) may cause damage over time. The 5g/L concentration shows a steady increase in tensile strength with prolonged exposure. Further investigation is needed to understand the decrease in strength from 7 to 14 days at 15g/L.

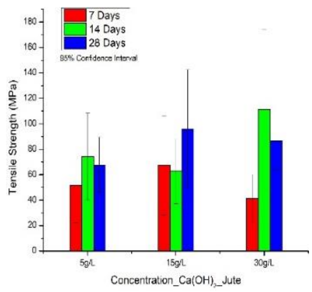
The OFAT experiments for KOH-treated jute fibers reveal varying tensile strength trends based on concentration and duration. At 7 days, the highest tensile strength is observed at 30g/L (82.67 MPa). At 14 days, the optimal tensile strength is found at 15g/L (237.58 MPa), followed by 5g/L (194.81 MPa), and a significant decrease at 30g/L (103.7 MPa). By 28 days, tensile strengths across all concentrations are closer, suggesting a more uniform response over time. The 5g/L concentration shows an increase in tensile strength from 69.38 MPa (7 days) to 194.81 MPa (14 days), then a slight decrease to 130.7 MPa (28 days), indicating a possible enhancement plateau. The 15g/L concentration follows a similar trend, peaking at 237.58 MPa (14 days) and then decreasing to 131.5 MPa (28 days). The 30g/L concentration shows variability, with tensile strength increasing from 82.67 MPa (7 days) to 163.06 MPa (28 days). The OFAT experiments for $Ca(OH)_2$-treated jute fibers show varying tensile strengths based on concentration and treatment duration. At 7 days, the highest tensile strength is at 15g/L (67.4 MPa). At 14 days, the tensile strength peaks at 30g/L (111.28 MPa), higher than at 5g/L (74.38 MPa) and 15g/L (62.73 MPa). By 28 days, the 15g/L concentration shows the highest tensile strength (95.92 MPa). For 5g/L, there is a steady increase from 51.48 MPa (7 days) to 74.38 MPa (14 days), then a slight decrease to 67.75 MPa (28 days), indicating a plateau effect. The 15g/L concentration shows a nonlinear response: an initial increase to 62.73 MPa (14 days) followed by a rise to 95.92 MPa (28 days). The 30g/L concentration fluctuates: decreasing from 41.41 MPa (7 days) to 111.28 MPa (14 days), then dropping to 86.72 MPa (28 days), suggesting variable outcomes with prolonged high-concentration exposure.



(a)



(b)



(c)

Figure 4. 3 The tensile strength of jute fibres treated with different alkalis, concentrations, and time periods (a) NaOH (b) KOH (c) $Ca(OH)_2$.

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 30g/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 [69]–[78].

Fig. 4.4 presents the DSC curves of all the samples.

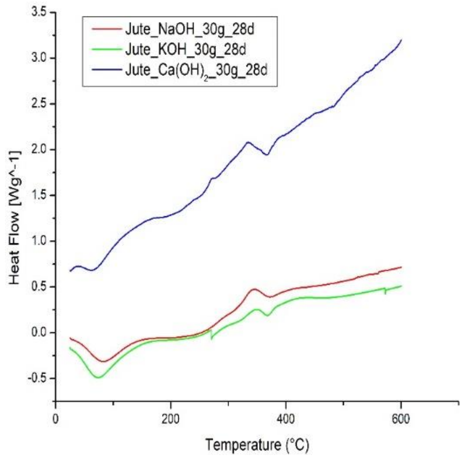


Figure 4. 4 The DSC analysis outputs of different jute samples.

Before 100 °C, there is one single peak of all the samples. By combining with the following analysis of TGA, it is caused by moisture evaporation [79]. In the temperature range from 300 °C to 400 °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 DSC data suggests that the alkali treatment indeed affects the thermal behavior of jute fibres [80], with $Ca(OH)_2$ treatment showing the most distinct differences. It has almost little or no effect on the jute fiber.

The Thermogravimetric Analysis (TGA) diagram shows the weight loss of jute fibers treated in different alkaline environments as a function of temperature. Fig. 4.5 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 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. The major weight loss between 300°C and 400°C represents the decomposition of cellulose, the main component of jute fiber. The final weight loss after 400°C corresponds to the breakdown of lignin and hemicellulose, which are the other major components of the fiber [79] as lignin decomposes at larger temperatures [81]. The range for T_g is from 220 °C till 350 °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.

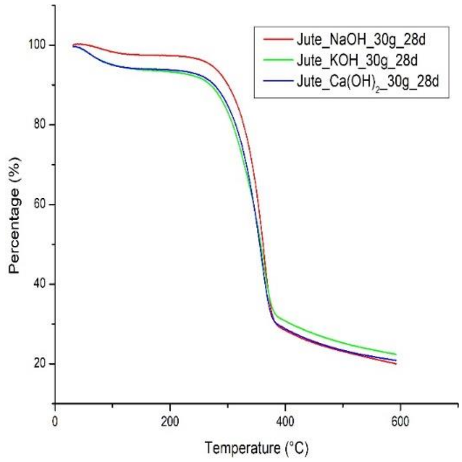


Figure 4. 5 The TGA results of different jute samples.

The RSM analysis data are presented in Table 4.2. 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.

Table 4. 1 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

Alkali type	Alkali concentration (g/L)	Time (day)	Tensile Strength (MPa)
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

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 will investigate the connections between the alkali type, concentration, time, and the observed responses using RSM. The mathematical expression of the model is demonstrated in Equation 2.

$$Yi = f\,(Ai, Ci, ti) \qquad \text{Eq. (4.1)}$$

Yi representing the outcome of the ith response. This function f will be a mathematical expression that captures the behaviour 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 4.2. As indicated in Table 5, it becomes evident that the quadratic model outperforms the others due to its minimal standard deviation and maximal R-squared value.

$$Tensile\ Strength = 221.65 * AlkaliType + 4.18 * AlkaliConcentration + 21.14 * Time - 0.047 * AlkaliType * AlkaliConcentration - 1.42 * AlkaliType * Time - 0.029685 * AlkaliConcentration * Time - 39.41 * AlkaliType^2 - 0.09 * AlkaliConcentration^2 - 0.42 * Time^2 \qquad \text{Eq. (4.2)}$$

Table 4. 2 The results of statistical indicators in different regression models.

Source	Std. Dev.	R^2	Adjusted R^2
Linear	50.88	0.24	0.1412
2FI	53.19	0.28	0.1864
Quadratic	46.15	0.54	0.4802
Cubic	55.78	0.6	0.548

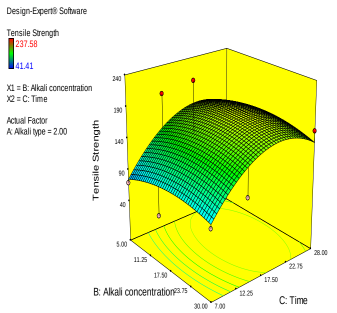
Based on the insights gleaned from Table 4.3, it becomes apparent that the Time of sample curing, with the highest F-value of 8.65, emerges as the most pivotal feature in our analysis. As we delve further into our investigations, we find that Alkali type assumes the mantle of being the most influential factor in our experimental endeavours. This hierarchical order of significance among the variables underscores the importance of these factors in shaping our outcomes.

Table 4. 3 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			

Analysing Fig. 4.6a, 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 Figs. 4.6b-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. 4.6 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. 12-c, the residual values derived from the variance between actual and predicted 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.

(a)	(b)



(c)

Figure 4. 6 The sensitive analysis of the effective features as per tensile strength (a-c).

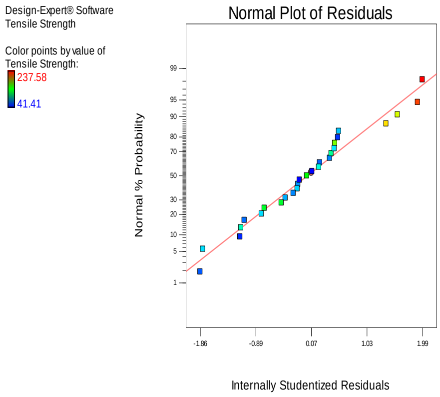


Figure 4. 7 The data distribution of experimental practice in the present research.

Based on the observations from Fig. 4.7, 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.

As illustrated in Table 4.4, 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 visualisation in Fig. 4.6.

Table 4. 4 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

Analysing the data presented in Fig. 4.8, 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. According to Fig. 4.8, the use of NaOH results in the highest tensile strength among the three alkali types considered. Moving on to alkali Type 2, potassium hydroxide (KOH), we observe a similar trend in Fig. 4.8. The tensile strength of jute-based fibres treated with KOH is also notably higher than that of Type 3 ($Ca(OH)_2$) treatment. In contrast, the data for alkali Type 3, calcium hydroxide ($Ca(OH)_2$), show the lowest tensile strength improvement among the three alkali types. These findings from Fig. 4.8 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.

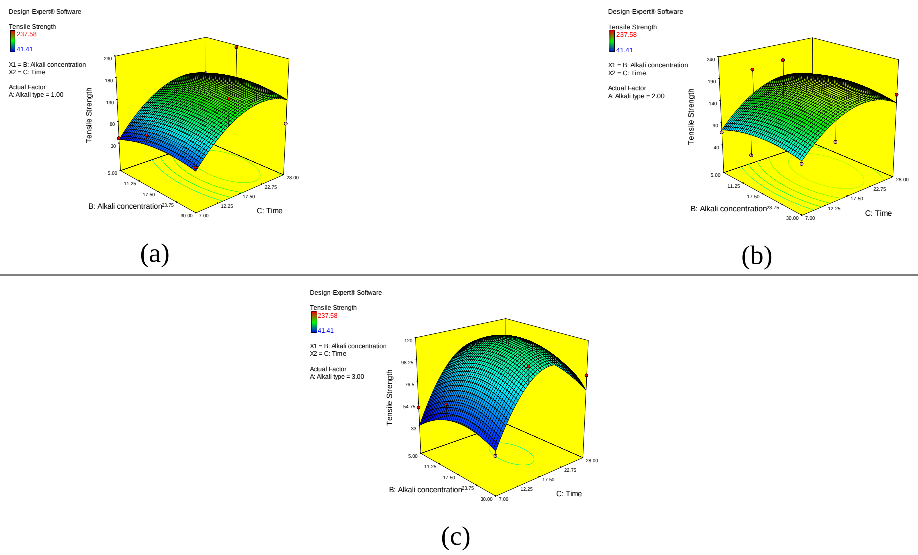
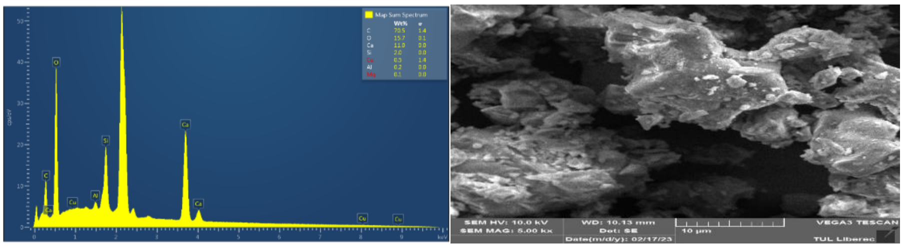


Figure 4. 8 The graphical result of the optimum condition in (a) alkali type: NaOH, (b) alkali type: KOH, and (c) alkali type: $Ca(OH)_2$.

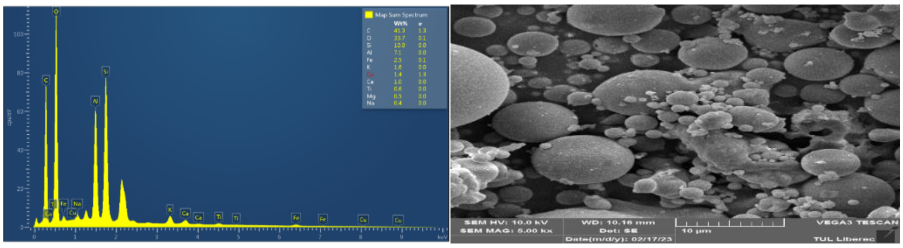
For achieving the task of partial replacement of cement by inorganic additives, different percentages of FA (5%, 10%, 20%), LAP (1%, 3%, 5%) and BENT (1%, 3%, 5%) were used to prepare the cement based mixtures. Later, new cement mixtures with partial replacement of cement by fly ash (5%) and Laponite (1%) reinforced with jute fibers (0.2%, 0.5%, 0.7%, 1%) were realized. Fig. 4.9 shows the results of EDS and SEM characterisations based on different raw materials and prepared samples. As can be seen, all SEM images are reported in 5kx.

Cement powder



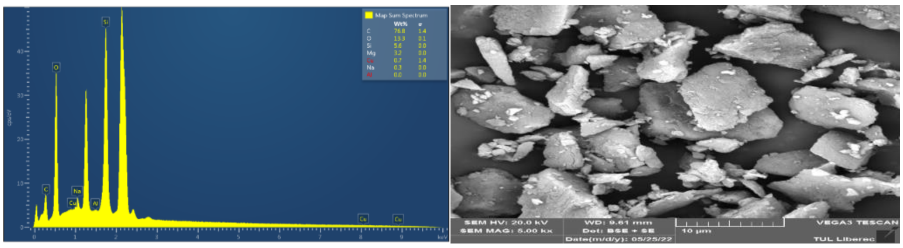
(a)

Fly ash



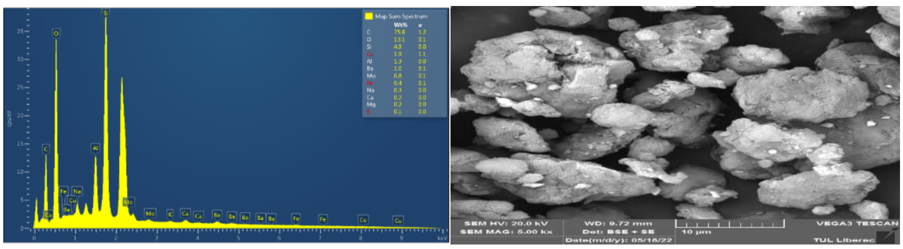
(b)

Laponite



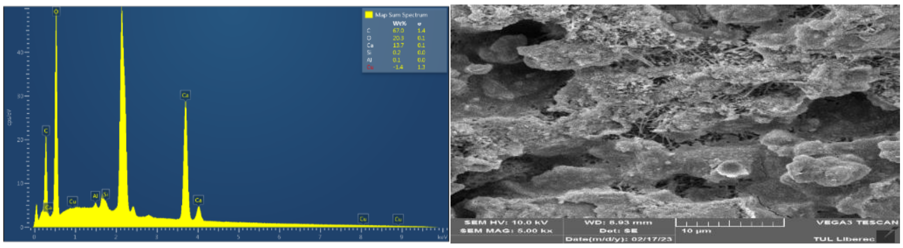
(c)

Bentonite



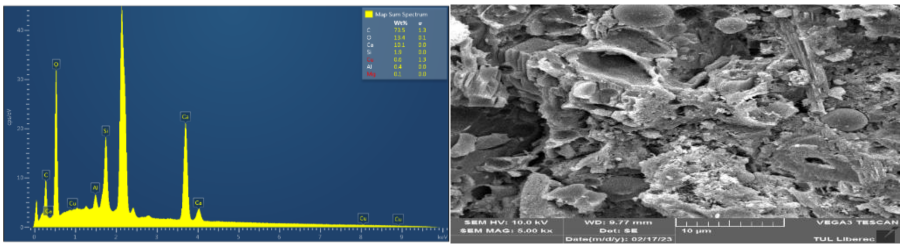
(d)

FA 5%



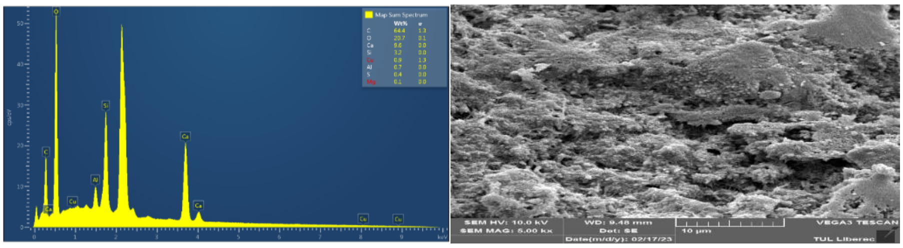
(e)

FA 10%



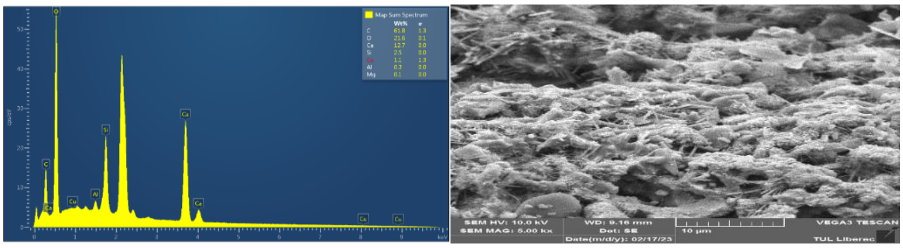
(f)

FA 20%



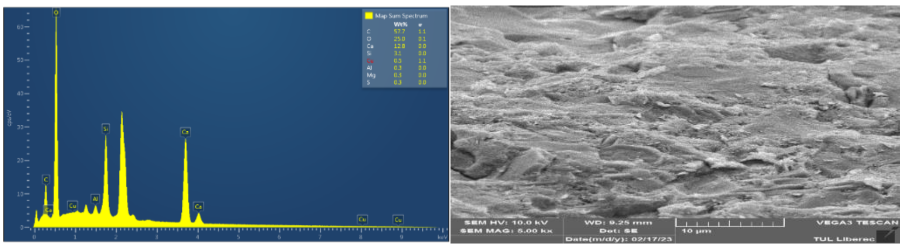
(g)

LAP 1%



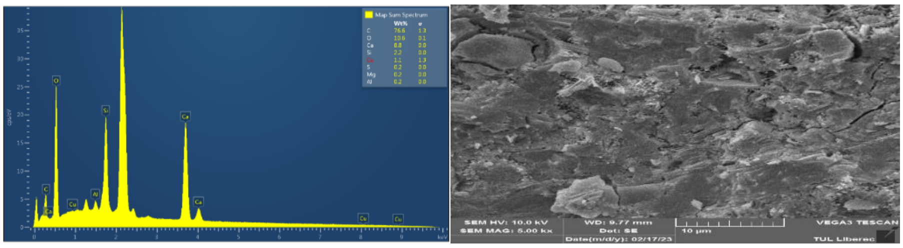
(h)

LAP 3%



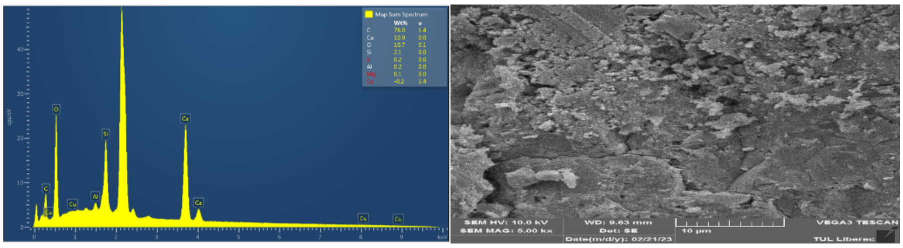
(i)

LAP 5%



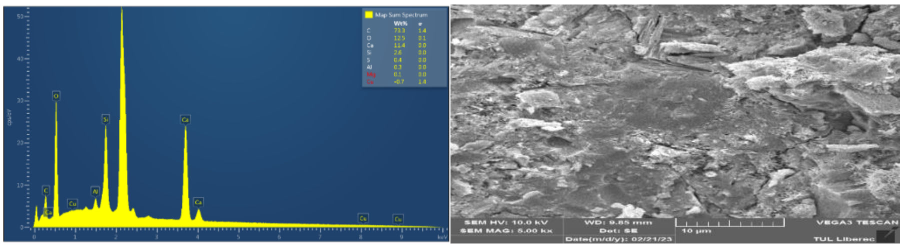
(j)

BENT 1%



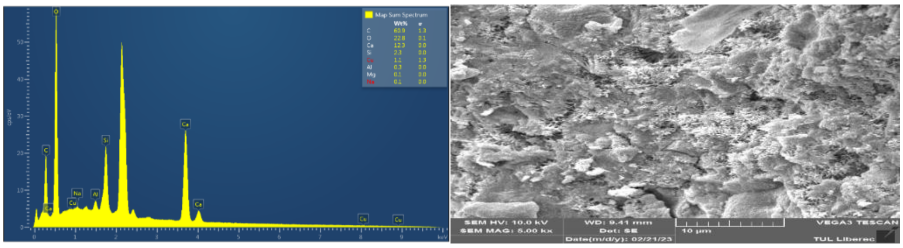
(k)

BENT 3%



(l)

BENT 5%



(m)

Figure 4. 9 The results of characterisation study for the three cement paste additives with different rates (a-m).

Fig. 4.10 summarises the results of the OFAT experiments. By comparing Figs. 4.9 and 4.10, it can be seen that adding higher percentage of fly ash in the mixture of cement paste would increase the percentage of C, O, Al, and Si elements and therefore increase, the 3-point bending value. However, increasing the mentioned elements would considerably reduce, the amounts of toughness and compressive strengths. Comparing Figs. 4.9 and 4.10 can justify the reason for increasing in the percentages of C, O, Al, and Si elements when adding more fly ash in the mixture of cement paste. These reasons can be attributed to several factors. Firstly, the presence of fly ash in cement paste introduces additional reactive components into the mixture. Fly ash is a byproduct of coal combustion and contains a significant amount of silica (SiO_2) and alumina (Al_2O_3) [82]. These components react with the alkaline compounds in cement, such as calcium hydroxide, during the hydration process, forming additional calcium silicate hydrate (C-S-H) gel. The formation of C-S-H gel contributes to the overall strength and durability of the cementitious material [83]. Moreover, the increase in these elements can be linked to the pozzolanic reaction, which is a key mechanism associated with the incorporation of fly ash in cement paste. This reaction produces additional hydration products, including calcium silicate hydrate and calcium aluminate hydrate (C-A-H) gels. These gels fill in the pore spaces within the cement paste, resulting in a denser microstructure and improved mechanical properties [84]. Additionally, the increase in the carbon and oxygen percentages can be attributed to the carbon content present in fly ash. The presence of carbon can influence the microstructure of the cement paste, affecting its mechanical properties [85]. However, while the addition of fly ash and the subsequent increase in the mentioned elements can enhance certain properties, it is worth noting that there are trade-offs in terms of toughness and compressive strengths. The incorporation of fly ash generally results in a decrease in toughness and compressive strengths. This can be attributed to the dilution effect caused by the addition of fly ash, which leads to a reduction in the overall cement content and a decrease in the inter-particle bonding within the cementitious matrix. Consequently, the material becomes more brittle and less resistant to applied forces. By increasing LAP in the structure of cement paste mixture, all functions (3-point bending, compressive, and toughness strengths) would reduce. However, with evaluation of BENT (main elements: C, O, Al, Si, Cu) the 3-point bending and compressive strengths are increased and reversely, the capacity of toughness is decreased. Thus, the Cu and Al have positive influences on 3-point bending. While just Cu has direct relationship on compressive strength. The presence of LAP can impact the hydration process of cement, which is essential for the development of strength in the paste [86]. The water associated with LAP, along with the water required for cement hydration, influences the availability of water molecules for the chemical reactions. Excess water from LAP can dilute the cementitious system, affecting the formation of stable calcium silicate hydrate (C-S-H) gel, which is responsible for the strength and durability of the cement paste. This dilution effect can lead to reduced 3-point bending strength, compressive strength, and toughness. In the case of BENT, its chemical composition primarily comprises elements such as Si, Al, O, C, and Cu. These elements can interact with the cementitious system and influence its properties differently. The presence of Cu in BENT can have a positive effect on the mechanical properties of the cement paste. Copper ions can act as a catalyst in the hydration reactions, promoting the formation of a denser and stronger cementitious structure [87]. This can lead to an increase in 3-point bending strength and compressive strength. On the other hand, Al in BENT can also influence the cement paste, particularly the 3-point bending strength. Aluminum ions can react with the silicate compounds in the cement, leading to the formation of additional hydration products. These products can contribute to improved bonding and enhance the material's resistance to bending stresses. Also, by comparing the SEM images, it can be concluded that with increasing FA, LAP, and BENT, the porosity of cement paste is reduced. While there are different behavior in all functions. Therefore, it can be concluded that the strength outputs are independent in comparison of sample porosities.

According to Fig. 4.9, fly ash is a fine powder that is primarily composed of spherical particles. The chemical composition of fly ash can vary depending on the composition of the coal burned, but it generally consists of silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), iron oxide (Fe_2O_3), calcium oxide (CaO), and small amounts of other elements. The morphology of fly ash particles is typically irregular, with varying sizes ranging from a few micrometres to several tens of micrometres. The particles are often hollow and porous, giving them a light and powdery texture. The surface of fly ash particles can be rough and angular. Laponite is a synthetic clay-like material that belongs to the class of layered silicates. Its structure consists of a two-dimensional sheet of silica and magnesium ions, with water molecules located between the sheets. Laponite particles are disc-shaped. These particles can stack together to form aggregates or gel-like structures in water or other polar solvents. Bentonite is a type of clay formed from the weathering of volcanic ash. The structure of bentonite consists of individual clay platelets stacked on top of each other. These platelets have a layered structure, with each layer being composed of two silica tetrahedral sheets sandwiching an alumina octahedral sheet. The layers are held together by weak van der Waals forces, allowing them to slide and swell in the presence of water. They have a flake-like or needle-like morphology, with a high surface area due to the presence of numerous platelets. The interlayer spaces between the clay platelets can accommodate water molecules and other ions, giving bentonite its unique swelling and adsorption properties.

The results of this study (Fig. 4.10) show that with increasing fly ash content, the compressive strength of the cement paste decreases. It is well known that fly ash is a pozzolanic material that reacts with calcium hydroxide during hydration of cement to form additional cementitious compounds [88]. However, excessive amount of fly ash in the cementitious composites cannot fully participate in the pozzolanic reaction thus resulting in a reduction of hydration products in cement-based composites. This can result in the reduction of the compressive strength [89]. It is important to note that higher compressive strengths are possible with fly ash content in cementitious composites when fly ash will be mechanically activated, the outcomes of different studies such as [90]–[92]. Also, the declared research items conform to the present investigation outputs and explanation.

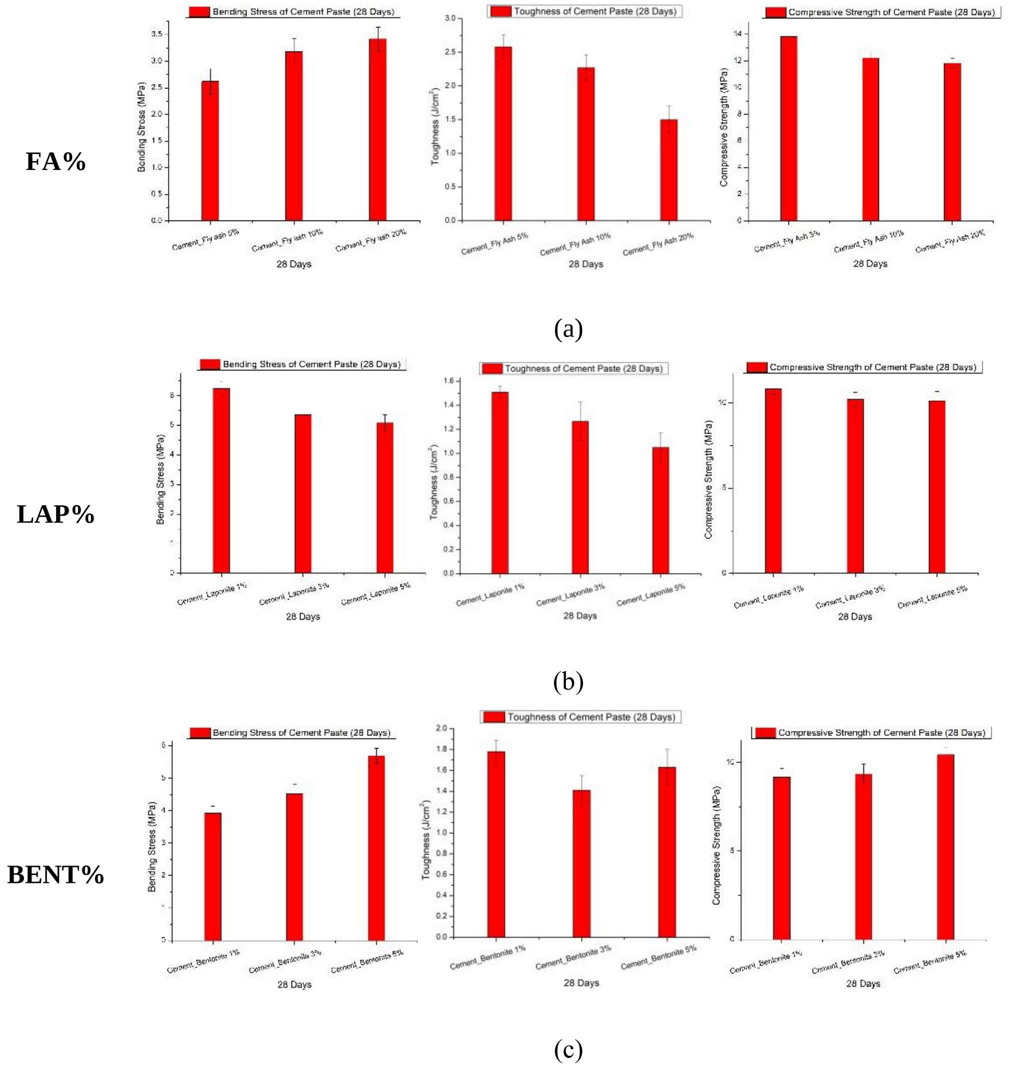


Figure 4. 10 The results of the three evaluation functions (bending stress, toughness, compressive strength) for the three cement paste additives; (a) FA%, (b) LAP%, and (c) BENT% function performances.

Results with LAP as a partial replacement in this study depict that the compressive strength of cement pastes decreased with increasing content of the LAP. The possible reasons for this is that LAP clay being very fine disk like particles belonging to the Smectite group of phyllosilicates [93], [94] possess pozzolanic reactivity and can increase the C-S-H during cement hydration process. Moreover, it can fill the voids thus improving packing of the cementitious system thus increasing the compressive strength of the cement paste [95]. However, with increasing LAP content causes agglomeration and also affects the water demand

and workability of the cement which can negatively affect the hydration procedure. Previous studies [96], [97] reported similar results for compressive strength with different clays. Compressive strength of the cement paste having bentonite as a partial replacement of cement in this research work showed that the higher value for compressive strength was achieved with 5% replacement of cement by BENT. This behavior of BENT in cement pastes may be due to the improved particle packing within the cement paste matrix. Furthermore, it can be due to the better binding and cohesion properties with increased hydration and cementitious products. The outcomes of this study reveal that with increasing FA and BENT content, the 3-point bending property of the cement paste also improved. This can be attributed to the better interfacial bonding between the FA/BENT particles and cement paste thus resulting in improved flexural strength [98]. Whereas in case of LAP, 3-point bending decreased with increasing content. The reduction in strength with increasing content of LAP can be because of dilution effect of the LAP particles which lead to poor participation in interfacial bonding.

Cement paste with FA/LAP/BENT in this study showed a decreasing trend with increasing content of fillers. In case of FA, this behavior may be because of the increasing brittleness and reduced ductility of the cement paste, while the decreasing trend of the cement pastes having LAP may be attributed to the ineffective role of LAP with higher percentages in cement paste leading to poor toughness properties. Cementitious composites with partial replacement of cement by BENT firstly showed a decrease with 3% but at 5% it showed some improvement. Cement paste with 3% showed a reduction in toughness by 20.8% compared to cement paste with 1% BENT, while cement paste with 5% BENT showed an improvement of 13.5% compared to cement paste with 3% BENT. This behavior may be due to the formation of cracks and their propagation due to the internal stresses within the cement pastes.

The results of regression statistical analysis for curve fitting of LAP, FA, and BENT against 3-point bending strength, compressive strength, and toughness are summarised in Fig 4.11. The results show that in adding different percentages of FA in the mixture of cement paste, toughness and 3-point bending strengths have acceptable (R^2> 82%) Coefficient of determination. Likewise, the FA % addition had direct effects onto 3-point bending and had reverse impact on both others. Fig 4.11 depicts that the Coefficient of determination of all functions based on LAP is acceptable (more than 84%). However, the slope of all functions (3-point bending, compressive, and toughness strengths) are minus in the provided equations and it shows reverse relationship between adding LAP in mixture of cement paste. The response of both 3-point bending and compressive strengths for the case of BENT is acceptable (R^2> 84%)

as they had direct relationship with the additive. The reaction of BENT % and cement paste mixture vs toughness was strange, and the linear equation could not describe it.

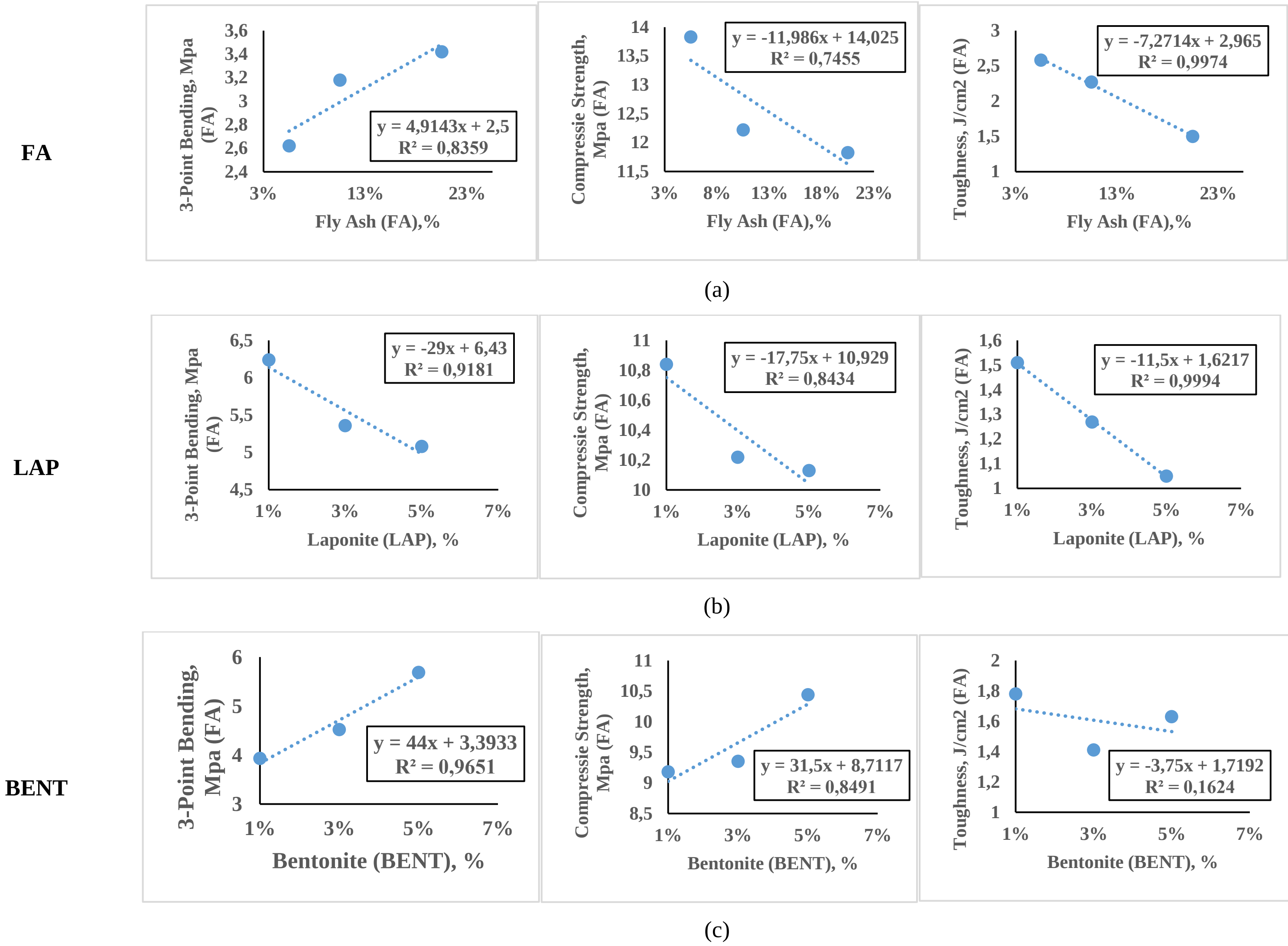


Figure 4. 11 The results of regression statistical analysis for curve fitting of the present research (a-c).

The outcomes of the EIA for nine samples are presented (Fig. 4.12). The diagram illustrates that the presence of Al element in the FA and higher percentages of the additive used in the study result in the highest level of toxicity. Similarly, in the subsequent phase, varying percentages of BENT, due to the presence of Mg and Al elements, exhibit the most toxicity and EIs. Finally, based on its chemical structure, the least hazardous additive is associated with the LAP.

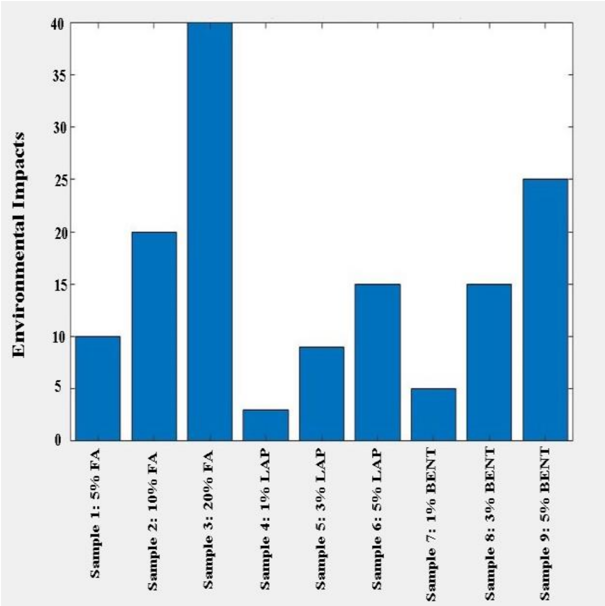
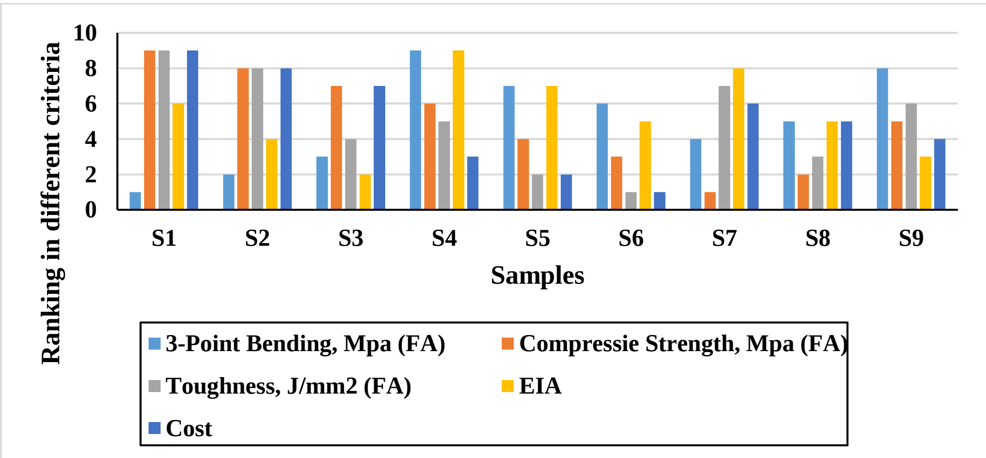
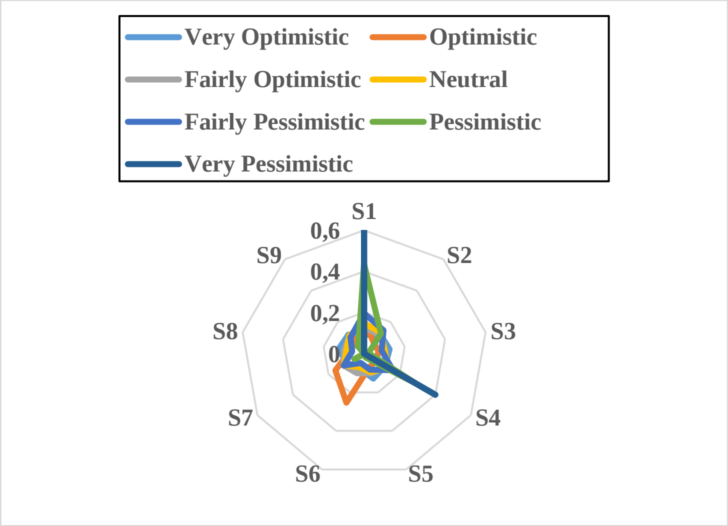


Figure 4. 12 The outputs of EIA assessment of prepared samples in this research.

The results of the OWA ranking model are demonstrated in Fig. 4.13. According to the scheme (Fig. 4.13 a and b), it can be concluded that based on SA indicators, from very pessimistic and neutral points of view (idea of managers), the samples S1 and S4 should be selected for the construction preparation process. In both the options, the least levels of FA (5%) and LAP (1%) are used as an additive in the structure of cement paste. Besides, in a very optimistic selection perspective, S3 (with 20% FA, weight=0.125), S5 (with 3% LAP, weight=0.129), and S8 (with 3% BENT, weight=0.126) are selected. Therefore, in an optimistic condition, all the materials can be applied based on the viewpoint of managers and decision-makers.



(a)



(b)

Figure 4. 13 The outcomes of OWA computations according to (a) sample rankings as per different performance/ cost/EIA and (b) final OWA weights.

The analysis of cement particle distribution reveals a grain size of 15.18 µm and a mean volume of 38.00 µm. The histogram describing cement particle distribution suggests that a significant portion of particles falls within the 1-10 µm range. The overall shape of the cement particles histogram appears to be right-skewed, with a majority of particles concentrated in the smaller size ranges and a tail extending towards larger sizes. The analysis of fly ash particle distribution reveals a grain size of 22.38 µm and a mean volume of 65.38 µm. The histogram describing fly ash particle distribution submits that a majority of the particles are smaller in size with the number of particles decreasing as the size increases. The analysis of laponite particle distribution reveals a grain size of 25.34 µm and a mean volume of 68.92 µm. The histogram describing laponite particle sizes shows a positively skewed distribution. The analysis of bentonite particle distribution reveals a grain size of 23.25 µm and a mean volume of 49.62 µm. The histogram of the bentonite appears to be positively skewed. The average grain sizes of the materials are given in Table 4.5.

Table 4. 5 Average grain sizes of the powder materials

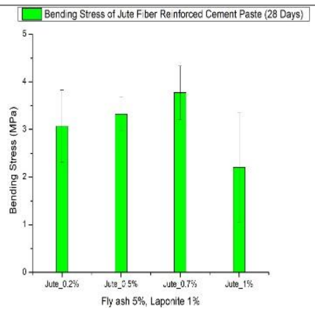
Particle Size Distribution		
#	**Material**	**Average Grain Size, µm**
1	Cement	15.18
2	Fly ash	22.38
3	Laponite	25.34
4	Bentonite	23.25

Particle size distribution data Table 4.5 gives us quantitative data regarding these materials. Particle size distribution data is useful in understanding the materials' behavior in cementitious composites using aggregation tendencies, and overall behavior. Particle size reduction increases surface area, which improves mechanical properties. whereas a decrease in surface area caused by a large particle size could lower the mechanical properties. Therefore, it is recommended to consider the particle sizes of the ingredients of the cementitious composites in future work.

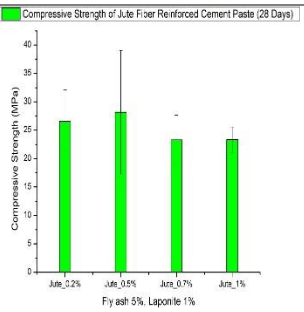
The results of the 3-point bending stress for cement mixtures reinforced with varying weight percentages of jute fiber are shown in Fig. 4.14a. The bending stress increases with higher jute fiber content. At 0.2% jute fiber, the bending stress is 3.07 MPa. With 0.5% jute fiber, the stress increases by 8.2% to 3.32 MPa. At 0.7% jute fiber, the bending stress further increases to 3.77 MPa, representing a 13.6% increase from the 0.5% content and a 22.9% increase from the 0.2% content. The increase in bending stress was previously reported by Shoukry et al. (2013) in a research work where different natural fibers reinforced in cement pastes showed improvement in bending stress up to a 2% fibers content [99]. Similar results were also observed for coated sisal fiber reinforced concrete samples [100]. However, with a further increase in jute fiber content to 1%, we observe a decreasing trend in bending stress with a value of 2.2 MPa showing a 41.6% decrease in bending stress compared to the 0.7% jute fiber content. It can be seen that the optimal value of the jute fiber content against 3-point bending stress was observed at 0.7% fiber content. The decrease in bending stress beyond 0.7% is likely due to several issues such as fiber agglomeration or poor bonding with the cement matrix resulting in a poor composite matrix. Similar explanations have been reported previously such as [101][102]. The compressive strength of cement mixtures reinforced with jute fiber shows an optimal value at 0.5% fiber content, reaching 28.17 MPa. Increasing the fiber content beyond 0.5% results in a decrease in compressive strength: 0.7% fiber content drops it to 23.29 MPa, and 1% fiber content maintains a similar value of 23.31 MPa. Therefore, the highest compressive strength is achieved at 0.5% jute fiber content. Similar trends have been observed in previous studies. Chakraborty et al. (2013) reported an improvement in compressive strength in 1% jute fiber-reinforced cement mortar [103]. In another study, compressive strength of kenaf-reinforced cementitious composites showed improved compressive strength values for 0.5, 1, 1.5, and 2% fiber content and the maximum strength improvement was 96% for 1.5% fiber content [104].

Cement mixtures with 5% fly ash and 1% laponite, reinforced with various percentages of jute fiber, show a clear trend of increasing toughness with higher fiber content. At 0.2% jute fiber, the toughness is 2.3935 J/cm². This increases to 2.8325 J/cm² at 0.5% fiber (18.4% increase), 3.0360 J/cm² at 0.7% fiber (7.2% increase from 0.5%), and 3.5114 J/cm² at 1% fiber (15.7%

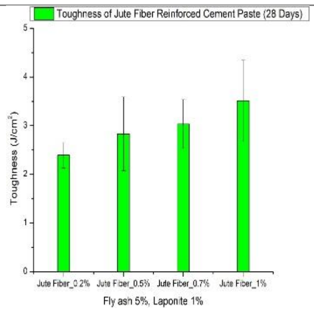
increase from 0.7%). Overall, there is a 46.7% increase in toughness from 0.2% to 1% fiber content. These results indicate that adding more jute fiber enhances the toughness of the cement mixture by improving its energy absorption and post-cracking behavior [101]. The incorporation of short jute fibers into the cement mortar has shown significant improvements in the toughness property of the composites [105]. Overall, the data indicates that jute fiber reinforcement significantly enhances the toughness of the cement mixtures, with the most substantial improvements observed at higher fiber contents.



(a)



(b)



(c)

Figure 4. 14 The results of the jute-reinforced cement mixtures having different fiber contents; (a) 3-point bending stress, (b) Compressive strength, and (c) Toughness.

5. Conclusions

This study optimized jute-based materials through various alkali treatments, examining the effects on tensile strength. The best results were observed with 15 g/L NaOH at 28 days (225.05 MPa), 15 g/L KOH at 14 days (237.58 MPa), and 30 g/L $Ca(OH)_2$ at 14 days (111.28 MPa). Prolonged high alkali exposure reduced tensile strength. NaOH and curing time were most influential on tensile strength. Cement was partially replaced with additives (FA, LAP, BENT) to improve mechanical properties. FA improved 3-point bending stress but reduced compressive strength and toughness. LAP negatively affected all properties, while BENT improved bending stress and compressive strength. Cement mixtures containing 5% FA, 1% LAP, and short jute fibers, with optimal fiber content at 0.7% for 3-point bending, 0.5% for compressive strength, and 1% for toughness.

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Publications of the Author

[1] A. Mahmood et al., “Preparation of Green Sustainable Cement Paste Mixture Based on Inorganic Additives: An Experimental and Modelling Approach,” Buildings, 2024, (Revised Manuscript submitted)

[2] A. Mahmood et al., “Aging behaviour assessment of cellulosic fibres in alkaline media: A green technology approach in construction materials,” J. Build. Eng., vol. 92, p. 109685, 2024, doi: https://doi.org/10.1016/j.jobe.2024.109685.

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Curriculum Vitae

AAMIR MAHMOOD

Address: Ještědská 341/103
46008 Liberec, Liberec VIII- Dolní Hanychov, Czech Republic
Tel: ++923218010483, +420776810051
Email: engr_aamir2002@yahoo.com; aamir.mahmood@tul.cz

OBJECTIVE AND SUMMARY OF QUALIFICATIONS:

Motivational attitude with a goal of conducting result oriented quality research utilizing my extensive twenty years relevant experience in working with construction companies and educational institution, assignments undertaken include site management and staff supervision, estimation and costing, working with subcontractors & material suppliers. Currently teaching Civil Engineering & Environmental Management & Policy courses at university level.

ACADEMIC QUALIFICATIONS:

Vinnitsia State Technical University, Ukraine. Masters of Science in Civil Engineering.	1999
Vinnitsia State Technical University, Ukraine. Bachelor of Science in Civil Engineering.	1997

RELATED PROFESSIONAL EXPERIENCE:

April 2018 to date	PhD Student at Technical University Liberec, Czech Republic
Mar 2005 -April 2018	Balochistan University of Information Technology, Engineering & Management Sciences Quetta, Pakistan. Department of Environmental Management & Policy
July 2004 - Nov 2004 .	Husnain Cotex Limited (H.C.L) Kandahar (Afghanistan). Worked as Civil Engineer on Kandahar – Tirin Kot Road project
Sep 2003 - June 2004.	Participatory Integrated Development Society (PIDS) Worked as Civil Engineer on "European Commission Humanitarian Aid Office (ECHO) Drought Relief Project" for Balochistan.
Oct 2000 - Jan 2003	Saadullah Khan & Brothers (S.K.B) Ashqabad (Turkmenistan). Worked as Civil Engineer on construction projects of Turkmen government.
Apr 1999- Sep 2000	Zareef Khan Kibzai & Brothers Quetta (Pakistan). Site Engineer

EXPERTISE:

- Preparation of project documents, conceptual and detailed estimation of costs.
- Preparation of onsite working drawings for supervisors.
- Well conversant with the processes of project management, disaster management and urban environmental management.
- Well conversant with process monitoring of ongoing projects, onsite support and technical backstopping.
- Development of proposals on construction projects and disaster management.

LANGUAGES

- English, Russian, German (Basic)

Opinion of the supervisor

FAKULTA TEXTILNÍ TUL

SUPERVISOR EVALUATION

TITLE OF WORK: Investigating the Effects of Alkaline Aging on Jute Fibers and Their Reinforcement Capabilities in Modified Cement Composites

AUTHOR: MSc. Aamir Mahmood

The submitted dissertation by Aamir Mahmood focuses on researching the influence of fiber reinforcement on the final properties of concrete composites and the replacement of concrete composite components with more sustainable alternatives. It specifically addresses the following components: fly ash, Laponite, and Bentonite. Additionally, it explores the potential use of cellulose fibers in the preparation of concrete composites and the impact of an alkaline environment on the alteration of their properties.

The dissertation first establishes the objectives of the research. The literature review section focuses on the components of concrete composites and their properties. It describes the components that make up the composite matrix and the jute fibers used in the experiment as composite reinforcement. This section concludes with a chapter dedicated to the theory of testing the properties of concrete composites.

The experimental part of the dissertation is divided into two main sections. In the first section, the doctoral candidate examines the degradation of jute fibers in an alkaline environment and the effect of this environment on the strength of technical jute fibers. In this experiment, three alkalis (NaOH, KOH, and $Ca(OH)_2$) were used to create degradation solutions of specific concentrations to simulate alkaline degradation. The degradation of the fibers is documented with images taken by SEM (Scanning Electron Microscopy).

The second part of the experiment focuses on the effects of fly ash (FA), Laponite (LAP), and Bentonite (BENT) on the mechanical properties of the cement mixture, both without jute fiber reinforcement and with varying concentrations of jute reinforcement. The following properties of the samples were monitored: three-point bending strength, compressive strength, and toughness. The data obtained from the experiment were statistically analysed. Statistical regression models and "Ordered Weighted Averaging" (OWA) models were applied, indicating that the mixture with 5% fly ash and 1% Laponite, reinforced with varying amounts of jute fibers, is optimal for construction purposes.

The research presented in this dissertation involved interdisciplinary connections. The work includes a thorough literature review and utilizes statistical methods for the evaluation of the measured data. The doctoral candidate has widely published partial results in professional journals and continues to do so even after submitting the dissertation. The candidate approached the dissertation work with innovation and also participated in other projects at the Department of Materials Engineering.

A plagiarism check was conducted on June 26, 2024, and no significant similarity with other literature was found. The highest detected similarity rate was 1%. In terms of content, graphical design, and the use of literary references, the work meets the requirements for a dissertation.

I RECOMMEND the submitted dissertation for defence.

Ing. Miroslava Pechočiaková, Ph.D.

In Liberec 18.9.2024

Reviews of the opponents

CZECH TECHNICAL UNIVERSITY IN PRAGUE
Faculty of Civil Engineering
Department of Materials Engineering and Chemistry
Thakurova 7, 166 29 Prague 6
Ing. Miloš Jerman, Ph.D.

In Prague, August 28, 2024

Opponent's review of the Doctoral Thesis

Title of the dissertation: Investigating the Effects of Alkaline Aging on Jute Fibers and Their Reinforcement Capabilities in Modified Cement Composites

Author of the thesis: Aamir Mahmood, M.Sc.
Faculty of Textile Engineering, Technical University of Liberec

Study programme: P3106 Textile Engineering

Study branch: Textile Technics and Materials Engineering

Supervisor: Ing. Miroslava Pechočiaková, Ph.D.

Evaluation of the Dissertation's Significance to the Field

The dissertation focuses on the use of natural fibers and the partial replacement of cement with waste or purely natural materials, which is fully in line with modern principles of sustainability in construction and the reduction of energy consumption in building material production. The results obtained from the investigation of jute fibers represent a significant contribution to the advancement of the scientific field. The second part of the dissertation, dealing with the replacement of cement with fly ash, is, however, assessed somewhat less favorably, as this topic has been extensively studied in the past. For instance, it is well-known that Portland composite cement (CEM II) can contain up to 35% fly ash (EN 197-1).

Comments on the Problem-Solving Approach, Methods Used, and Achievement of the Stated Objectives

The dissertation sets out two main objectives. The first was to explore the potential of using jute fibers as dispersed reinforcement in concrete or cement composites. The second objective was the partial replacement of Portland cement with hydraulically active materials. The introductory chapters of the dissertation describe the current state of knowledge and review the relevant literature. The second chapter focuses on the energy demands of cement and concrete production and considers sustainable alternatives for partial cement replacement with secondary industrial wastes, such as fly ash, Laponite, and Bentonite, while also discussing the possibility of replacing steel reinforcement in concrete with jute fibers, which require alkaline treatment.

The third chapter provides a detailed description of the methods used, including advanced techniques such as SEM analysis, TGA, and DSC measurements, which enable the understanding of key factors leading to the increased strength of jute fibers. Three different concentrations of alkaline solutions were tested, with fibers immersed for varying time intervals, in order to determine the optimal combination for maximizing fiber strength. To achieve the second objective, cement mixtures were prepared, in which the cement was partially replaced with fly ash, Laponite, and Bentonite in different proportions. The samples were tested for three-point bending, compressive strength, and toughness, supplemented by SEM and EDS analyses.

Assessment of the Dissertation's Results and the Original Contribution of the Author

The results are presented in the fourth chapter. For jute fibers, the weight loss due to immersion in alkaline solutions and the tensile strength of the fibers after treatment were examined. These results are clearly summarized in tables and graphs and have also been published by the author in the prestigious journal "Journal of Building Engineering". It would be advisable to consider adding self-citations to the text. This is a highly interesting study that offers potential for further scientific

THÁKUROVA 7
166 29 PRAHA 6
ČESKÁ REPUBLIKA
+420 224 357 128
MILOS.JERMAN@FSV.CVUT.CZ
HTTPS://K123.FSV.CVUT.CZ
IČ 68407700
DIČ CZ68407700

exploration. Regarding the second objective, it was also achieved, but its significance is considered less substantial. The replacement of cement with fly ash at a level of 20% does not introduce a major innovation, particularly in light of the fact that it is already widely known that Portland composite cement can contain up to 35% fly ash. It is unfortunate that reference values for cement paste without additives were not provided, as this would have allowed for a clearer comparison. The tests were conducted after 28 days, which is the standard period; however, from a scientific perspective, it would be interesting to extend these tests to 90 days, during which CSH gels could form, potentially leading to increased strength.

Comments on the systematic, clarity, formal and linguistic level of the dissertation

The dissertation addresses two distinct topics, which somewhat affects its overall coherence. Nevertheless, the work is clearly and comprehensibly structured. The grammatical errors in the Czech abstract can be attributed to the fact that the author is not a native Czech speaker. The main text is written in high-quality academic English. The results are clearly and systematically presented in tables and graphs.

Comments on the publications of the DSP student

The results of the dissertation have been published in foreign publications. Other publications of the PhD student are also extensive and testify to his high professional level.

Final assessment of the doctoral thesis

Despite the above mentioned shortcomings, the dissertation shows that the candidate has the ability to conduct independent research and publish his results in journals included in the Web of Science database. For this reason, I recommend the dissertation to be defended and awarded the degree of PhD.

THÁKUROVA 7
166 29 PRAHA 6
ČESKÁ REPUBLIKA

+420 224 357 128
MILOS.JERMAN@FSV.CVUT.CZ
HTTPS://K123.FSV.CVUT.CZ

IČ 68407700
DIČ CZ68407700

Questions for the defense:

1. The optimal immersion time of 28 days in NaOH solution seems rather long. Do you think it would be possible to shorten this time by increasing the temperature during the immersion process?

2. Some jute fibers treated for 28 days showed a decrease in strength compared to those treated for 14 days. Given that cement composites are known for their high pH, and the fibers will be exposed to high pH levels for many years, do you believe that jute fibers can withstand long-term exposure to high pH?

3. Did you measure the pH of the individual solutions?

Ing. Miloš Jerman, Ph.D.

THÁKUROVA 7
166 29 PRAHA 6
ČESKÁ REPUBLIKA

+420 224 357 128
MILOS.JERMAN@FSV.CVUT.CZ
HTTPS://K123.FSV.CVUT.CZ

IČ 68407700
DIČ CZ68407700

NATIONAL TEXTILE UNIVERSITY

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Thesis Evaluation Report

Faculty/Institute: Faculty of Textile Engineering
Program: PhD
Student's Name: Aamir Mahmood
Thesis Title: Investigating the Effects of Alkaline Aging on Jute Fibers and Their Reinforcement Capabilities in Modified Cement Composites

1 Title

The title of the thesis accurately reflects the content and research focus, providing a clear indication of the study's scope and objectives. For example, it clearly states the dual focus on the effects of alkaline aging on jute fibers and their reinforcement capabilities in cement composites, which is central to the research.

2 Abstract

The abstract offers a concise summary of the research objectives, methods, key findings, and conclusions. However, it includes several abbreviations that are not explained, such as SEM (Scanning Electron Microscope), TGA (Thermogravimetric Analysis), and DSC (Differential Scanning Calorimetry). For instance, when mentioning the use of SEM to analyze microstructural changes in the jute fibers, it would be helpful to spell out the abbreviation for clarity. Additionally, providing more details on the practical implications of the research, such as how the findings can impact sustainable construction practices, would enhance the abstract.

3 Research Problem and Objectives

The background and problem statement are well-defined, addressing a significant gap in the use of natural fibers in construction materials. The objectives of the thesis are clear and relevant to the research problem, and the methodology effectively addresses these objectives.

1. To explore the aging behavior of jute fibers in alkaline environments. For example, understanding how NaOH affects the tensile strength of jute fibers helps identify the most suitable conditions for their use in construction.
2. To perform partial replacement of cement by waste and clay materials in cement paste mixtures. For instance, the study investigates the use of fly ash and bentonite as sustainable alternatives to traditional cement, reducing the carbon footprint.

Sheikhupura Road, Faisalabad-37610, Pakistan. Tel: 041-9230081-82 Fax: 041-9230098

3. To optimize the reinforcement of cement paste mixtures with jute fibers. For example, statistical modeling is used to determine the optimal fiber length and concentration for enhancing tensile strength.
4. To assess the environmental and sustainability impact of the prepared mixtures. For example, the study evaluates the reduction in greenhouse gas emissions achieved by using natural fibers and waste materials in cement composites.

These objectives are aligned with the overall aim of the study, which investigates the deterioration of jute fibers in alkaline solutions and their reinforcement capabilities in modified cement composites.

4 Scope and Relevance

The scope of the study is appropriate for a PhD thesis. The research addresses relevant issues in the field of sustainable construction materials, with a focus on the practical application of jute fibers in cement composites. For instance, the study explores the potential of jute fibers to replace synthetic fibers, contributing to environmental sustainability. The study's outcomes have the potential to make a significant socio-economic impact, particularly in developing regions where natural fibers are abundant and affordable.

5 Literature Review

The literature review is comprehensive, well-organized, and relevant to the research issues. It identifies the knowledge gap effectively and provides a solid foundation for the research. Minor grammatical errors were noted, and the addition of more recent references on similar topics would enhance the review. For example, including studies published in the last five years that focus on the durability of natural fiber composites would strengthen the review. Additionally, insights from the literature to support the sustainability claims made about jute fibers should be included.

6 Research Methodology

The methodology is robust, with a clear explanation of the experimental procedures, including the preparation of cement pastes, the treatment of jute fibers with different alkaline solutions, and the testing methods used. Techniques such as SEM, TGA, and DSC are effectively employed to analyze the changes in fiber structure and thermal properties. For example, SEM images provided in the thesis show the surface degradation of jute fibers after exposure to NaOH, illustrating the impact of alkaline aging. However, the rationale behind the specific selection of alkalis (NaOH, KOH, and Ca(OH)2) could be better explained, particularly in terms of their differing effects on the chemical composition of the fibers.

7 Results and Analysis

The results are presented clearly, with appropriate use of tables, graphs, and microstructural images. The study demonstrates that jute fibers treated with moderate alkali concentrations show improved tensile strength, supporting their potential use in sustainable construction. For instance, the results indicate that jute fibers treated with 5% NaOH exhibited a 15% increase in tensile strength. The analysis is thorough, but the discussion could be enhanced by providing more insights from the literature to support the findings on sustainability. For example, referencing studies that discuss the long-term durability of jute-reinforced composites in construction would add depth to the analysis.

8 Discussion

The discussion effectively interprets the results, linking them back to the research objectives. The candidate explores the implications of using jute fibers as reinforcement in modified cement pastes and addresses the sustainability aspect by highlighting jute as a renewable resource. For example, the discussion could include a comparison between the environmental impact of using jute fibers versus synthetic fibers, supported by recent studies. However, the discussion could be strengthened by integrating additional recent studies to support the findings. For instance, discussing how the alkaline treatment of natural fibers can influence the lifecycle and recyclability of the composites would add value to the discussion.

9 References/Bibliography

The references section is comprehensive and well-structured, covering a wide range of relevant literature. There are 198 references listed, with 92 references from the last five years. For example, recent studies on the application of natural fibers in sustainable construction are well-represented, though more could be included to support the sustainability aspects discussed. The citation style is consistent throughout the thesis. However, there is room for including more recent studies to support the sustainability aspects discussed, particularly in relation to the environmental impact of jute fibers.

10 Overall Structure and Format

The thesis is well-structured, with clear and logical progression from one section to the next. The formatting is consistent, adhering to the guidelines provided by the institution. The writing is generally clear, though the thesis would benefit from minor language improvements, including attention to grammatical mistakes and spelling corrections. For example, correcting minor errors in the literature review and methodology sections would improve the overall readability.

Conclusion

The PhD dissertation titled “Investigating the Effects of Alkaline Aging on Jute Fibers and Their Reinforcement Capabilities in Modified Cement Composites” is well-written and contributes significantly to the field of sustainable construction materials. The research methodology is sound, and the results are thoroughly analyzed. With the minor corrections suggested, the thesis is recommended for the award of the PhD degree.

Prof. Dr. Yasir Nawab
Dean,
School of Engineering and Technology,
National Textile University, Faisalabad, Pakistan
Email: ynawab@ntu.edu.pk
Phone: +92 345 3388914