FACULTY OF TEXTILE ENGINEERING TUL

Master Thesis

Characterization and Modification of Coir Fibers

Study programme:	N0723A270002 Textile Engineering
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Thesis Supervisors:	Mohanapriya Venkataraman, M.Tech., M.F.Tech., Ph.D. Department of material engineering

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Master Thesis Assignment Form

Characterization and Modification of Coir Fibers

Name and surname:	**Mahalakshmi Subramanian**
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Rules for Elaboration:

1. Literature review of coir fibers used in various products like composites, blends for construction, and textile materials.
2. Analysis of the physical and chemical properties of two types of coir fibers.
3. Modification of fiber properties using suitable chemical treatments and re-analysis of properties.
4. Comparison of fiber properties before and after modification and data analysis.
5. Adhesion and wettability test between geopolymer and coir fibers for future work.

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- Ichim, M., Stelea, L., Filip, I., Lisa, G., Muresan, E.I. Thermal and Mechanical Characterization of Coir Fibre–Reinforced Polypropylene Biocomposites. *Crystals* **2022**, *12*, 1249. DOI: 10.3390/cryst12091249.
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- Aravind, D., Senthilkumar, K., Diwahar, P., Chandrasekar, M., Senthil Muthu Kumar, T., Parameswaranpillai, J., Rajini, N., Siengchin, S. Chapter 4 – Review on coir fiber surface modification by various techniques, Editor(s): Jawaid, M. *In Woodhead Publishing Series in Composites Science and Engineering*, Coir Fiber and its Composites, Woodhead Publishing **2022**, 55-7. DOI: 10.1016/B978-0-443-15186-6.00044-8.
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Anotace

Cílem této diplomové práce s názvem "Charakterizace a modifikace kokosových vláken" je analyzovat a porovnat fyzikální vlastnosti hnědých kokosových vláken ze dvou různých odrůd kokosovníku před a po chemické modifikaci. Pro ověření kompatibility kokosových vláken před a po modifikaci jako plniva zahrnuje tato diplomová práce přípravu geopolymerních kompozitů s různými délkami kokosových vláken. V neposlední řadě si tato práce klade za cíl přispět k hlubšímu pochopení vlastností hnědých kokosových vláken dvou vybraných odrůd a jejich kompatibility s geopolymerními kompozity.

Klíčová slova

Kokosový strom, kokosová vlákna, fyzikální vlastnosti, chemická modifikace, termická analýza, geopolymerní kompozity, ohybové vlastnosti.

Annotation

The objective of this diploma thesis titled "Characterization and Modification of Coir Fibers" is to analyze and compare the physical properties of brown coir fibers from two different varieties of coconut trees before and after chemical modification. To test the compatibility of the coir fibers before and after modification as fillers, this thesis includes the preparation of geopolymer composites with different lengths of coir fibers. Lastly, this thesis aims to contribute to a deeper understanding of the characteristics of brown coir fibers of the two selected varieties and their compatibility with geopolymer composites.

Keywords

Coconut tree, Coir fibers, Physical properties, Chemical modification, Thermal analysis, Geopolymer composites, Flexural properties.

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I would like to express my deepest gratitude to Dr. Mohanapriya Venkataraman, M.Tech., M.F.Tech., Ph.D., for her invaluable guidance, unwavering support, and expert mentorship throughout my diploma thesis. Her profound knowledge, insightful feedback, and encouragement have played a pivotal role in shaping this research endeavor and have enriched my academic journey. I would also like to thank Prof. Ing. Jakub Wiener, Ph.D., for his invaluable contributions. His expertise, constructive criticism, and scholarly insights have significantly enhanced the quality of this thesis and broadened my perspective on the subject matter. My sincere gratitude to Ing. Miroslava Pechociaková, Ph.D. , Ms. Jana Stránská and Ing. Jaroslava Frajová for helping me with the experiments and assisting me in the laboratories at various stages of this research project. I would also like to extend my gratitude to the faculty members of the Department of Clothing Technology and Materials who have provided assistance and support. Lastly, I want to thank my family and my friends for always being there for me and supporting me through thick and thin.

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List Of Abbreviations

AM	After Modification
BM	Before Modification
F_{end}	Force at the end of the test
F_{max}	Maximum Force
KP	Kavilipathiram (Chowghat Orange Dwarf)
MK	Metakaolin
NaOH	Sodium hydroxide
NT	Naatu Thengai (Indian Country Coconut)
RWD	Root Wilt Disease
S_{end}	Track at the end of the test
S_{max}	Track at maximum power3
σfB	Bending Stress at the moment of fracture
σfM	Bending Strength

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Abstract

Two classes of coconut tree fibers, Naatu thengai (Indian Country Coconut) and Kavilipathiram (Chowghat Orange), were tested for their physical properties in a chosen stage of maturity (dried coconut - brown fiber). Chemical modifications were carried out to retest their physical properties and the results were compared. Geopolymer composites were prepared with and without different sizes of the two types of coir fibers to analyze their compatibility. The findings showed significant differences in the physical properties between the modified and unmodified fibers and between the geopolymer composites with and without fibers.

1. Introduction

Natural fibers have long been an integral part of human civilization, finding applications in a myriad of industries ranging from textiles to construction. They are used as cheap construction materials and as an alternative of synthetic fibers in composites (Ali et al., 2012). Vegetable fiber is a type of natural fiber extracted from various parts of plants such as stems, leaves, roots, fruits, and seeds (Rao and Rao, 2007). Almost all plant fibers are used as sound absorbers (Kassim et al., 2023).

Coir, a lignocellulosic fiber with high lignin content, derived from the fibrous mesocarp of coconuts, sourced from coconut trees (*Cocos nucifera*), have gained prominence due to their abundant availability, biodegradability, and impressive mechanical properties. It serves as a raw material for various traditional items such as furnishing materials, ropes, bags, mats, brushes, hats and mattresses (Geethamma et al., 2005; Arsyad et al., 2015; Rao and Rao, 2007). This research aims to delve into the intricate world of coir fibers, aiming to comprehensively characterize their physical, chemical, and mechanical properties. Furthermore, this study will explore various methods of modification, seeking to enhance their performance characteristics and expand their potential applications.

1.1 Coconut Tree

Coconut, the fruit of *Cocos nucifera* belongs to the Arecaceae (Palm family), a large plant variety that has an average height of about 80 - 100 ft (Main et al., 2014; Perera et al., 2003; Chan and Elevitch, 2006). With over 80% of coconut production concentrated in the Asia-Pacific region, coconuts emerge as a pivotal economic contributor for many countries in this area (Louis et al., 2010). Their significance extends beyond national borders, playing a crucial role in both global exports and local economies, while also bearing cultural importance (Beveridge et al., 2022). The outer morphology of the coconut fruit exhibits diversity in terms of shape, color, and size. Shapes range from elliptic and oblong to round, while colors include yellow, green, orange, brown, or red.

Their varieties include tall, dwarf semi-tall and semi-dwarf. Tall varieties' immature fruits often display varying shades of green and bronze, occasionally with yellow hues. Conversely, self-pollinating dwarf varieties can present orange, yellow, green, and light brown colors (Beveridge et al., 2022; Jayasekara and Amarasinghe, 2010). The genetic diversity within the species reflects in varying biomass proportions of husk, shell, and kernel. Wild coconut populations are characterized by an increased proportion of husk, contributing to a long and angular fruit shape, advantageous for water dispersal. Wild coconuts typically exhibit slow germination, with approximately 60% of their biomass in the husk, 15% in the shell, and 25% in the endosperm (Beveridge et al., 2022). Figure 1 shows the coconut tree parts and their applications.

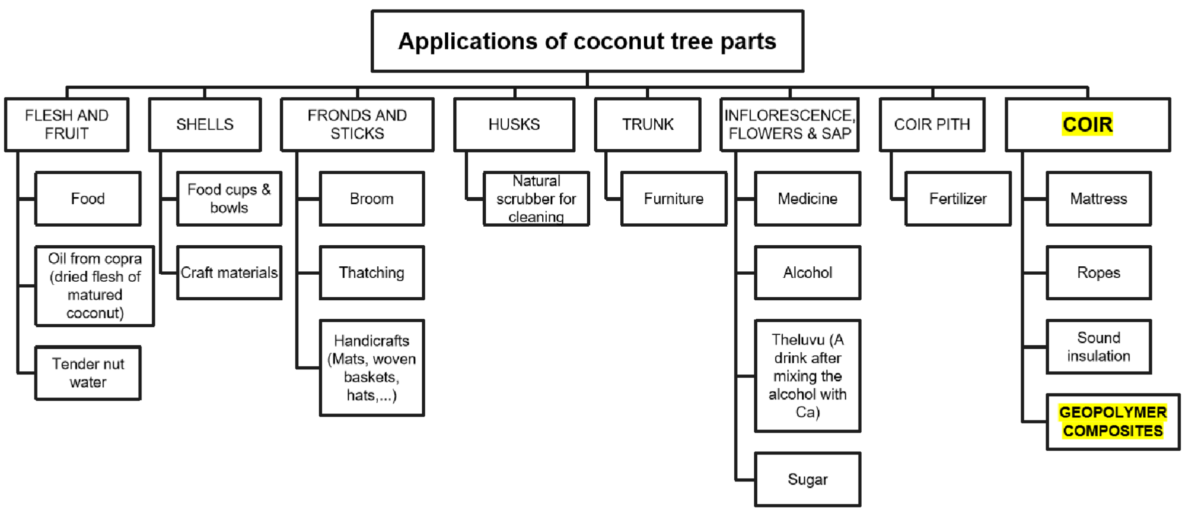


Fig 1. Application of coconut tree parts

1.2 Types of Coconut Tree

Coconut trees are divided into 2 types based on the type of growth - Dwarf and Tall (Freitas Neto et al., 2016). Dwarf trees are self pollinated and that's the reason they can't be found much in nature (Lantican et al., 2019). Tall trees are cross pollinated, resulting in significant variability which is evident in the traits of size and color of the fruits and surface morphology (Chan and Elevitch, 2006; Perera et al., 2003; Swaminathan and Nambiar, 1961). Conversely, there's a third type called 'hybrids' that are the result of cross-breeding between different varieties of tall and dwarf coconut trees, combining the characteristics of both parent varieties. Dwarf and tall coconut trees are shown in Fig 2 (a) and (b).

(a) (b)

Fig 2. (a) Dwarf coconut tree **(b)** Tall coconut tree

1.2.1 Dwarf

This variety produces fruits at an early age and are around 1-2 m tall while some can even grow up to 7 m. Dwarf coconut trees are shorter compared to other types and their fruits are easily reachable. They are mostly used for tender nut purposes. The properties of a few different types of dwarf variety are given in Table 1. (The places mentioned in Tables 1,2 and 3 are states of India).

Table 1. Dwarf coconut tree types (Kasaragod, 2023)

NAME OF THE COCONUT TREE	PROPERTIES
Chowghat Orange Dwarf	• Starts flowering after the 3rd year of cultivation. • Gives fruits from the 9th year. • Average annual yield is around 63 nuts/palm. • They are best for tender nut water. • This cultivar was released in 1991 by ICAR-CPCRI for cultivation as a tender nut variety. • The fruits are in round or oval shape. • They can be affected by water scarcity.
Kalpasree	• Earliest flowering cultivar where it starts flowering in 2.5-3 years. • Mean annual yield is around 90 nuts/palm and .0963 kg copra/nut.

	• Have superior quality coconut oil and sweet tender nut water and meat. • They bear green fruits. • High tolerance towards Root Wilt Disease (RWD) which is one of the most devastating diseases of coconut palms. Phytoplasma is responsible for the occurrence of root (wilt) disease in India (Krishnakumar et al., 2015). • Major symptoms of this disease are necrosis of leaflets, wilting, drooping, flaccidity, paling/yellowing and ribbing of leaves. • It is cultivated in RWD prevalent areas of Kerala, a state in South India.
Kalpa Jyoti	• They bear yellow fruits. • Average yield is 114 nuts/palm and copra is 16 kg/palm. • Recommended for cultivation in Kerala and Karnataka for tender nut purposes.
Kalpa Suriya	• They bear orange fruits. • Annual yield is 123 nuts/palm and copra is 23 kg/palm. • Recommended for cultivation in Kerala, Karnataka and Tamil Nadu.

Kalparaksha	• They are a semi-tall variety that gives green fruits with sweet tender nut water. • They have a high resistance to RWD. • Starts flowering after 54 months from planting. • Annual yield is around 87 nuts/palm, 16.38 kg copra/palm and 10.65 kg oil/palm. • And if it is affected by RWD, it produces around 65 nuts/palm. • The quantity of tender nut water is about 250ml/nut.

1.2.2 Tall

Tall coconut trees are the most commonly found trees in nature as they cross-pollinate. They take longer to bear fruits compared to the dwarf variety. They have good copra content and the fruits come in different shades of green and orange depending on the type. The properties of a few different types of tall variety are given in Table 2.

Table 2. Tall coconut tree types (Kasaragod, 2023)

NAME OF THE COCONUT TREE	PROPERTIES
Chandrakalpa / Lakshadweep Ordinary (LCT)	• This variety grows in all types of soil and can withstand moisture stress. • The average annual yield is 100 nuts/palm. • They bear green fruits. • They are recommended for cultivation in Kerala, Karnataka, Andhra Pradesh and Maharashtra.
Kalpatharu	• This variety is recommended for ball copra production. (Ball Copra – Mature coconuts are dried for a minimum of 11 months where the water in the nuts is dried and they attain a sweet taste. It is then de-husked and de-shelled to get copra which looks like a ball) • Annual yield is 116 nuts/palm and copra content is 176 g/nut under rainfed conditions.

Kera Keralam	• Fruits of this variety weigh about 800-900 g and are suitable for ball copra production. • Annual yield is around 109 nuts/palm under rainfed conditions, copra is 176 g/nut and copra oil content is 68% • It is recommended for large-scale commercial cultivation in Tamil Nadu, Kerala and West Bengal.
Kalpa Haritha	• These are the superior high-yielding tall selection. • They have a lower incidence of Eriophyid Mite Infection (Eriophyid mites penetrate plant cells and suck up the cellular contents causing visible deformities or abnormalities) (Anon., 2023b).
Kalpa Ratna	• This variety is high-yielding and is suitable for inflorescence sap production. (It is an exudate obtained from the inflorescence. Fermented and non-fermented beverages are produced from the exudate and sugar and jaggery are produced from the sap) (Samsudeen et al., 2013). • Annual yield is around 133 nuts/palm and copra outturn is 24.47 kg/palm. • Suitable for cultivation in Kerala and Tamil Nadu.

1.2.3 Hybrids (Tall X Dwarf/Dwarf X Tall)

A hybrid variety comprises trees that are intentionally bred by combining the genetic traits of tall and dwarf coconut trees. This breeding process aims to achieve a desired resultant type that inherits and showcases the desirable qualities of both the tall and dwarf parent trees. The properties of a few different types of hybrid variety are given in Table 3.

Table 3. Hybrid coconut tree types (Kasaragod, 2023)

NAME OF THE COCONUT TREE	PROPERTIES
Chandra Sankara	• This variety is a hybrid between West Coast Tall and Chowghat Orange Dwarf. (WCT is indigenous to India and is an ordinary tall variety cultivated since ancient times) (Nair and Nair, 2021). • Annual production range is around 100-150 nuts/palm and copra is around 160-230 g/nut. • This variety is susceptible to drought. • It was first cultivated in 1985 in the regions of Kerala and Karnataka.
Chandra Laksha	• This variety is a hybrid between Lakshadweep Ordinary and Chowghat Orange Dwarf (Nair and Nair, 2021). • It bears fruits in 4-5 years of cultivation. • Annual yield is 109 nuts/palm and copra is around 150-210 g/nut.

Kera Samrudhi	• Kera Samrudhi is a hybrid between Malayan Yellow Dwarf and West Coast Tall. • Annual yield is 117 nuts/palm, copra is around 4.38 t/ha and oil is 3.04 t/ha. • This variety is cultivated particularly for tender nut purposes in the areas of Kerala and Assam.
Kalpa Sankara	• This is a hybrid between RWD free Chowghat Green Dwarf and RWD free West Coast Tall (Nair and Nair, 2021). • Annual production is 85 nuts/palm, copra is 2.5 t/ha and oil is 1.69 t/ha. • This variety is cultivated in RWD prevalent tracts of Kerala.
Kalpa Sreshta	• This variety is a hybrid between Malayan Yellow Dwarf and Tiptur Tall. • Annual yield is 167 nuts/palm. • It has a high copra outturn of around 35.9 kg/palm/year (or) 6.28 t/ha. • This variety is used for tender nut purposes and is cultivated in Kerala and Karnataka (Kasaragod, 2023).

1.3 Soil and Environmental Conditions

Coconut palms will not survive in prolonged freezing temperatures. So, in case of some rare or occasional freezing temperatures during chilly weather, the soil around the tree must be thoroughly watered the day before as it helps in insulating the roots and acts as an added layer of protection. Nitrogen and water stress causes delay in flowering compared to the trees grown in favourable conditions (Kumar et al., 2008).

Best soils for coconut palms

- Red sandy loam, Laterite, Alluvial soil, Coastal and reclaimed soils
- pH ranging from 5.2 - 8.0
- Minimum depth for the soil must be 1.2 m
- Deep well drained soil
- Soil with good water holding capacity
- Proper supply of moisture either through well distributed rainfall or irrigation
- Need sufficient drainage
- The best is soil with alternate layers of sand and clay.

Unsuitable soil conditions

- Heavy and imperfectly drained soil
- Shallow soils with underlying hard rock, low-lying areas subject to water stagnation and heavy clayey soil (Anon., 2018; Ranasinghe and Thimothias, 2012).
- Root wilt disease occurs in various soil types, with a faster spread observed in sandy, sandy loam, alluvial, and heavy textured soils compared to laterite. Areas characterized by waterlogging, particularly those situated near rivers and canals, tend to exhibit a relatively higher incidence of this disease (Solomon and Geetha, 2004).

1.4 Field Layout

Field layout plays a key role in productivity and is followed to avoid common field problems like overlapping of plant parts and uneven distribution of water, nutrients and light. Some of the most commonly followed systems of layout include Square, Rectangular, Triangular, Hedge and Contour systems. Selecting the most appropriate layout depends on the variety of the plant, climatic conditions and soil type (Anon., 2023a). An example of field layout for coconut trees is shown in Fig 3.

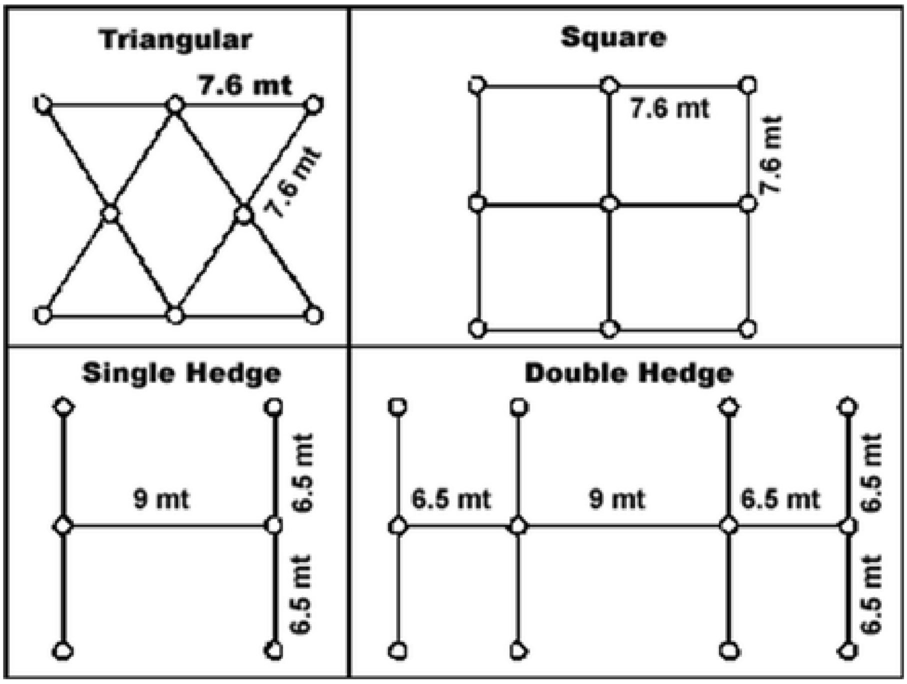


Fig 3. Example of field layout (Thomas et al., 2018)

1.5 Coir Fibers

Coir fibers, extracted from coconut husks, are categorized based on when they are removed from the husks. Brown coir fibers are obtained from fully ripe coconuts and are exceptionally durable against abrasion (Arrohman et al., 2020; Mathura et al., 2014) They find primary applications in products like brushes, floor mats, and upholstery padding due to their strength. Conversely, white coir fibers are derived from husks of coconuts that are harvested just before ripening. This variant is softer and less robust compared to brown fibers (Ali et al., 2012; Rajan and Abraham, 2007). Typically, white coir fibers are woven into mats or twisted to form twine or rope. The distinction between brown and white coir fibers allows for versatile usage in various industries, from durable applications in upholstery to more delicate applications like mat weaving and ropemaking (Jayasekara and Amarasinghe, 2010). The thickness of a coconut fruit shell ranges from 1cm to 5cm, varying with different varieties. This shell comprises three distinct layers: the outer exocarp, the porous and fibrous mesocarp in the middle, and the inner hard endocarp. This natural sandwich structure provides the coconut shell with unique properties and characteristics (Wang and Huang, 2009). Fig 4 shows the illustration of the cross-section of coconut. SEM cross-section of a single coir fiber is shown in Fig 5.

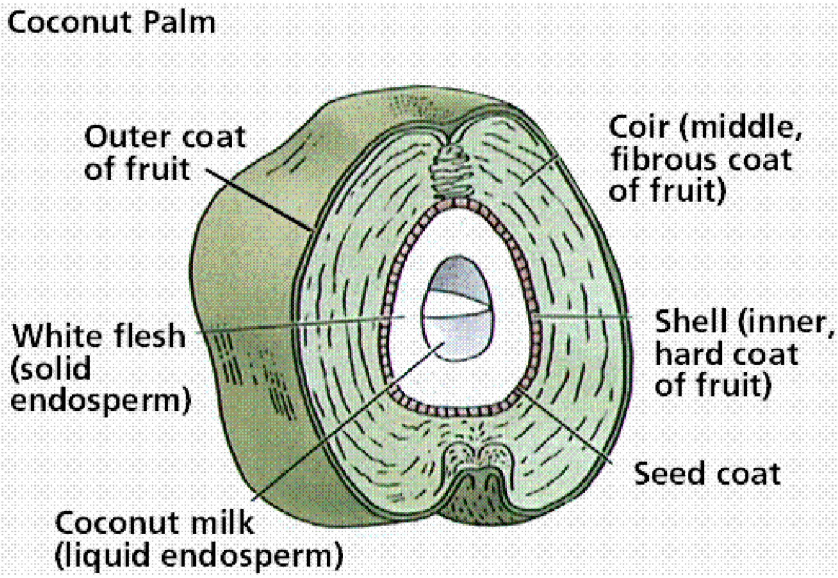


Fig 4. Schematic cross-section of coconut fruit (Nwankwojike et al., 2012)

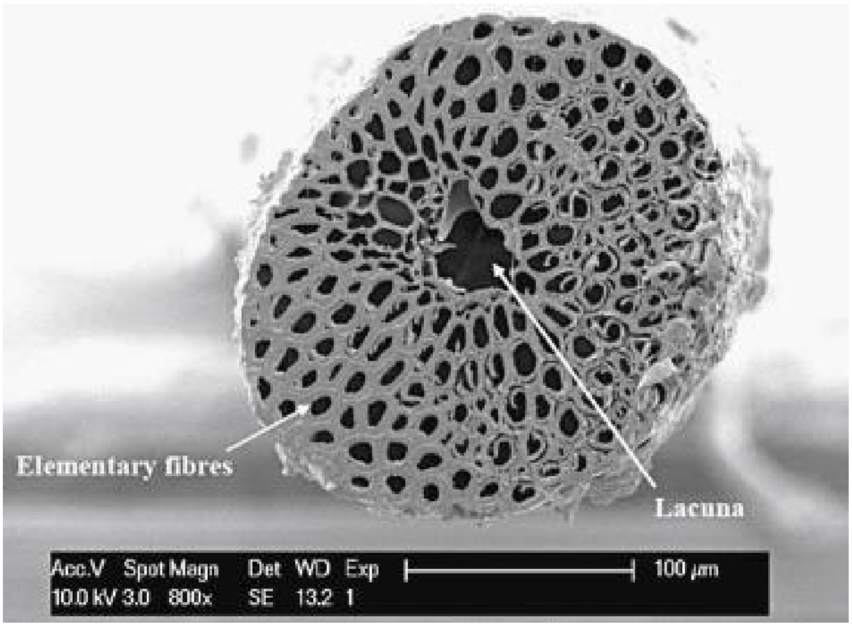


Fig 5. Cross section of a single coir fiber (Tran et al., 2015)

1.6 Geopolymer Composites

Geopolymers, also known as alkali-activated binders, are a highly effective and sustainable alternative to traditional binders. Aluminosilicate sources like fly ash, volcanic ashes, and metakaolin, which are either natural or waste, can be combined with alkali activators such as sodium hydroxide and sodium silicate to create a material that is durable. The resulting hardened gel products have desirable mechanical properties and other benefits such as speedy curing, exceptional fire resistance, high resistance to acid, strong adherence to aggregates, the ability to immobilize hazardous and toxic materials, and substantially lower energy usage and greenhouse gas emissions. that make them suitable for various applications (Chen et al., 2014). These materials are also referred to as 'inorganic polymers' or 'mineral polymers' in academic literature. The alkali activators work to dissolve the aluminosilicate precursors, freeing aluminate and silicate monomers that undergo a polycondensation reaction at a later stage. This reaction results in a superior product that is resistant to chemicals, fire, and extreme weather conditions (Ahmari et al., 2012; Bernal et al., 2012; Duxson et al., 2007; Provis and Van Deventer, 2009; Ranjbar and Zhang, 2020).

Geopolymer technology was introduced by the scientific community in the 1970s, and it was claimed that materials produced from thermally treated natural clays like metakaolin present structural stability at high temperatures due to their ceramic-like structure and high melting points. However, studies have shown that MK-based geopolymer binders often experience a significant loss of mechanical performance at temperatures above 200 °C due to dehydration and dehydroxylation of the reaction products, which causes severe shrinkage. Structural rearrangement, softening, and viscous sintering occur at temperatures between 800 °C and 1000 °C. These inorganic substances can be cured and solidified at temperatures close to ambient to create materials similar low-temperature ceramics, with typical heat resistance and strength. Although they possess a range of desirable qualities, such as relatively high strength, elastic modulus, and low shrinkage, geopolymers tend to exhibit brittle failure, similar to most ceramics (Alomayri et al., 2014). As a result, the use of these is limited in applications that require high volumetric stability. However, introducing appropriate fillers into brittle matrices has demonstrated the ability to alleviate shrinkage stresses and elevate the fracture toughness of resulting composite materials. Notably, the inclusion of fibers has emerged as a particularly effective strategy for augmenting mechanical properties, including fracture toughness (Bernal et al., 2012; Korniejenko et al., 2016).

Fibers act as a barrier against cracking, reduce the width of cracks, prevent brittle behavior, and improve ductility. This has the added benefit of mitigating damage caused by cracking. Another advantage of using fiber reinforcement is its ability to increase the flexural strength of composites. In addition, fibers can enhance the energy absorption and deformation resistance properties of geopolymer composites. The introduction of short fibers can especially contribute to improving the physical and mechanical properties of the specific geopolymer. Reinforcement with fibers is one of the most effective methods of strengthening and toughening geopolymer materials, particularly due to the ease of fiber dispersion and aspect ratio (Korniejenko et al., 2016).

Geopolymer composites can display a diverse range of properties and characteristics that depend on the selection of raw materials and processing conditions. These can include high compressive strength, low shrinkage, fast or slow setting, acid resistance, fire resistance, and low thermal conductivity. However, these characteristics are not universal to all geopolymeric formulations, as they depend on the selection of raw materials and processing conditions (Duxson et al., 2007).

Metakaolin is a form of kaolin clay that has been dehydroxylated. Kaolin clays are one of the most commonly mined minerals worldwide and are typically used in the production of ceramics such as porcelain. These clays are generally amorphous aluminosilicate hydrates with limited long-range order due to their layered structure. Heating kaolin clay to temperatures between 100 and 200 °C releases absorbed water that is not chemically bound to the aluminosilicate structure. At higher temperatures between 500 and 800 °C, the water that is chemically bound to the aluminosilicate structure is also removed. The exact temperatures at which kaolin is burnt to create metakaolin, as well as other variables in the process, are unique to each producer and are considered proprietary. Burning for too long or at too high a temperature can reduce the reactivity of the final product (Sakulich, 2011; Shekarchi et al., 2010).

1.7 Export Data of Coir Products

The detailed information regarding the exportation of coir products from India, specifically encompassing the months of April for the years 2021 and 2022, has been systematically compiled by accessing the comprehensive export data. This valuable dataset was meticulously sourced from Tamil Nadu Agricultural Department, a reputable authority in agricultural statistics and information. The specifics and nuances of India's coconut product exports, particularly focusing on coir fiber products, are vital components in understanding the broader landscape of the country's agricultural and economic contributions on the global stage. This data is given in Table 4.

Q - Quantity in Million Tonnes

V - Value in Rs. Lakhs

Table 4. Export data

	APRIL 2022		APRIL 2021		% GROWTH	
ITEM	**Q**	**V**	**Q**	**V**	**Q**	**V**
Coir Fibre	23490	3303.96	37823	6683.60	-37.9	-50.6
Coir Yarn	460	317.77	504	359.20	-8.,7	-11.5
Handloom Mat	1347	1631.02	1632	1974.00	-17.5	-17.4
Powerloom Mat	25	46.87	33	55.72	-26.1	-15.9
Tufted Mat	6496	7087.75	5607	5636.34	15.9	25.8
Handloom Mattings	72	127.01	135	161.90	-47.1	-21.6
Powerloom Matting	0	0.00	0	0.00	-	-
Geo textiles	974	908.99	708	571.15	37.6	59.2

Coir Rugs & Carpet	7	13.15	42	50.43	-84.2	-73.9
Curled Coir	367	80.89	881	240.00	-58.4	-66.3
Rubberized Coir	13	29.95	84	128.32	-84.4	-76.7
Coir Pith	69474	21372.84	95234	29898.37	-27.0	28.5
Coir Other Sorts	109	189.02	55	98.29	96.7	92.3
Total	**102859**	**35131.11**	**142738**	**45859.51**	**-27.9**	**-23.4**

Country-wise export to some European countries of a few coir products as of April 2022, also acquired from the Tamil Nadu Agricultural Department shows the quantity and values of coir products exported to each country. Table 5 shows the export data of coir fiber products to four different countries.

Q - Quantity in Tonnes

V - Value in Rs. Million

Table 5. Export data to 4 countries

ITEM	BELGIUM		GERMANY		NETHERLANDS		ITALY	
	Q	V	Q	V	Q	V	Q	V
Coir Fibre	17.50	0.504	40.42	1.085	114.00	3.757		
Coir Yarn	30.68	4.240	14.43	1.431	181.53	14.547	174.36	7.275
Handloom Mat	30.15	5.669	37.99	6.398	30.56	4.289	26.88	2.718
Tufted Mat	83.00	8.398	183.85	18.227	122.96	12.766	282.76	29.411
Handloom Mattings	1.05	0.123	10.55	2.683				
Geo textiles			92.42	10.156	19.46	2.109	52.66	4.237
Coir Pith	1334.8	38.095	1084.8	32.929	10380	293.79	2436.8	72.932

2. Motivation

Being one of the global leaders in coconut production, Asia, possesses an unparalleled surplus of coir fibers, a byproduct of this thriving industry. This abundance presents a unique opportunity to not only tap into a readily available, natural resource but also to invigorate the economy through value-added processes. While previous literature has extensively explored coir fibers across various maturity stages, there seems to be a notable gap regarding the characterization of coir fibers from different varieties of coconut trees. Hence, this study undertakes a novel exploration to bridge this gap and is one of the main reasons, this research is being started from scratch. This would open paths to learning about the differences in fiber strength, thermal properties, and chemical properties between the varieties. In this way, each application can use the best-suited coir fiber variety.

3. Objectives

1) Literature review of coir fibers used in various products like composites, blends for construction, and textile materials.
2) Analysis of the morphological, physical and chemical properties of the selected two types of coir fibers.
3) Modification of fiber properties using suitable chemical treatments and re-analysis of properties.
4) Comparison of fiber properties before and after modification and data analysis.
5) Preparation of coir fiber based geopolymer composites.
6) Adhesion and wettability test between geopolymer and coir fibers.

4. Materials

In this endeavor, two distinct varieties of coconut trees have been chosen for their unique properties. The first selected variety is country coconut and the second is Chowghat Orange. The first, known as the country coconut, is renowned for its versatility and adaptability. It is a tall variety. The second variety, a dwarf known as Chowghat Orange, stands out as it is specially cultivated for its tender nut water, making it an ideal choice for this process. These materials were imported from India. The details of the chosen materials are given in Table 6. Fig 6 (a) shows the stage of the selected coconuts. Fig 6 (b) and (c) represent Chowghat Orange and Country Coconut.

Table 6. Details of the chosen materials

S.No	Type of tree	Type of fiber	Type of soil
1	Chowghat Orange Dwarf	Brown (matured)	Alluvial / Laterite
2	Country Coconut (Tall)	Brown (matured)	Alluvial / Laterite

(a) (b) (c)

Fig 6. (a) Matured coconuts **(b)** Chowghat Orange Dwarf **(c)** Country Coconut

4.1 Coir Fiber Extraction

The extraction process of coir fibers involves a meticulous series of steps, carefully designed to obtain high-quality fibers. It was done in two different methods for comparison of experiment results.

Processing method 1

The initial phase of this extraction process entails the removal of the husks from the coconuts of both selected varieties. This step is crucial, as it lays the foundation for obtaining the fibers and the quality of it varies depending on the method with which the fibers were extracted (Jayasekara and Amarasinghe, 2010). Once the husks are separated, they undergo a thorough soaking in water, a crucial step that lasts for an approximate duration of 15 days (Wang and Huang, 2009). This soaking process is pivotal, as it facilitates the softening of the husks, preparing them for the subsequent stages (Prasad et al., 1983; Rajan and Abraham, 2007). Following the soaking phase, the husks are subjected to mechanical methods, which aid in the gradual separation of the fibers from the husk material (Mathura et al., 2014). This step is repeated 2 to 3 times, ensuring that all the fibers are effectively loosened and liberated from the husk. Once the fibers have been successfully extracted, they are transferred to a drying stage, a critical component of the process. The extracted fibers are laid out in the open, allowing them to bask in the natural sunlight for an extensive period of approximately 10 days (Jayasekara and Amarasinghe, 2010). This meticulous drying process serves to eliminate any residual moisture, ensuring that the fibers are thoroughly desiccated and ready for further processing (Nguyen et al., 2016). Upon completion of the drying process, the fibers emerge transformed, and their moisture content is significantly reduced. This renders them more resilient and suitable for a diverse range of testing. To keep them away from regaining moisture, the fibers and the husks were put in zip-lock bags and were sealed shut (Nguyen et al., 2016). The soaked husks look like in Fig 7 (a). Fig 7 (b) & (c) show the coir fibers after removal and separation.

(a) (b) (c)

Fig 7. (a) Husks after being soaked **(b)** Coir fibers after removal **(c)** Removed coir fibers being separated

Processing method 2

Following the husk removal from the coconut, a visual representation in Fig 8(a) illustrates the subsequent appearance. The husks are then subjected to mechanical treatment, involving the use of a hammer to beat and loosen the fibers, allowing for their extraction. Fig 8(b) visually captures this mechanical process. The fibers obtained through this mechanical method tend to be shorter and contain more impurities in contrast to fibers extracted using the first method.

Fig 8. (a) Coconut after the removal of husk **(b)** Removal of fibers from husks

6. Methodology

6.1 Chemical Modification of Coir Fibers

Four chemical modification methods were selected from literature to choose the best pre-treatment for this research.

Method 1

Coir fibers were soaked in 5% NaOH for 48 hours followed by washing and air drying (Geethamma et al., 2005).

Method 2

Coir fibers were soaked in 2% detergent solution for 1 hour at 70℃. It is then washed with running tap water at first, followed by a thorough washing with distilled water. The washed fibers are dried at 70℃ in a vacuum oven. These fibers were then dewaxed in a mixture of 1:2 ethanol and benzene at 50℃ for 72 hours and then washed with distilled water. These dewaxed fibers were treated in 5% NaOH solution at 30℃ for 30 mins, followed by a thorough washing with distilled water (Bismarck et al., 2001).

Method 3

Coir fibers were soaked in 5%,10%,15% and 20% conc., of NaOH solution for 3 hours followed by drying in the oven at 90℃ for 5 hours and cooled to room temperature (Arsyad et al., 2015).

Method 4

Coir fibers were soaked in 3% sodium sulfite (Na_2SO_3) solution and subjected to sonication for 2 hours on ultrasound apparatus. This step is to ensure the penetration of reactants into the fibers (Calado et al., 2000). It is then washed and dried at room temperature for 1 day, followed by drying in the oven at 80℃ for 10 minutes.

6.1.1 Sonication of Coir Fibers

Method 4 was chosen based on visual analysis by Scanning Electron Microscopy. To modify the fibers using this method, 40 grams of AM-KP and AM-NT fibers were immersed in a 3% solution of sodium sulfite (Na_2SO_3) in 2500 ml of distilled water. The mixture underwent sonication for 2 hours using the K-2LE ultrasound apparatus at 30℃ and the highest intensity level, 9. Subsequently, the fibers were thoroughly rinsed and air-dried at room temperature for 24 hours, followed by further drying in an oven at 80℃ for 10 minutes. Fig 9(a) shows the sonication of fibers and (b) shows the remaining solution with impurities after sonication.

Fig 9. (a) Sonication of fibers **(b)** Remaining solution after sonication

6.2 Fabrication of Geopolymer Composites

The geopolymer selected for the preparation of the composites is Baucis LBNa. This aluminosilicate binder has 2 parts - metakaolin (part A), activated by liquid alkaline (part B). The reinforced fibers are classified into 3 sizes. Table 7 shows the proportion of the fibers and the geopolymers.

Table 7. Geopolymer composite mixture proportion

SAMPLE NAME	PART A	WEIGHT	PART B	WEIGHT	MIXTURE WEIGHT	FIBER LENGTH (cm)	FIBER WEIGHT	FIBER %
0	5 parts	10 g	4 parts	8 g	18 g	-	-	-
1 (AM-KP)						Full length	0.45g	3%
I (AM-NT)						Full length		
2 (AM-KP)						0.5 - 1.2		
II (AM-NT)						0.5 - 1.2		
3 (AM-KP)						0.2 - 0.5		
III (AM-NT)						0.2 - 0.5		

The prepared geopolymer mixture was mixed with the fibers in a silicone mold and dried in the oven at 70℃ for 1 hour. The silicon mold is covered completely to prevent evaporation. The geopolymer composites were then wrapped separately and kept untouched for a day. This step facilitates the identification of any additional reactions occurring. The composites are subsequently allowed to air dry at room temperature for one day, ensuring complete evaporation of residual water. This process significantly enhances the strength and stability of the composites. The preparation steps are shown in Fig 10.

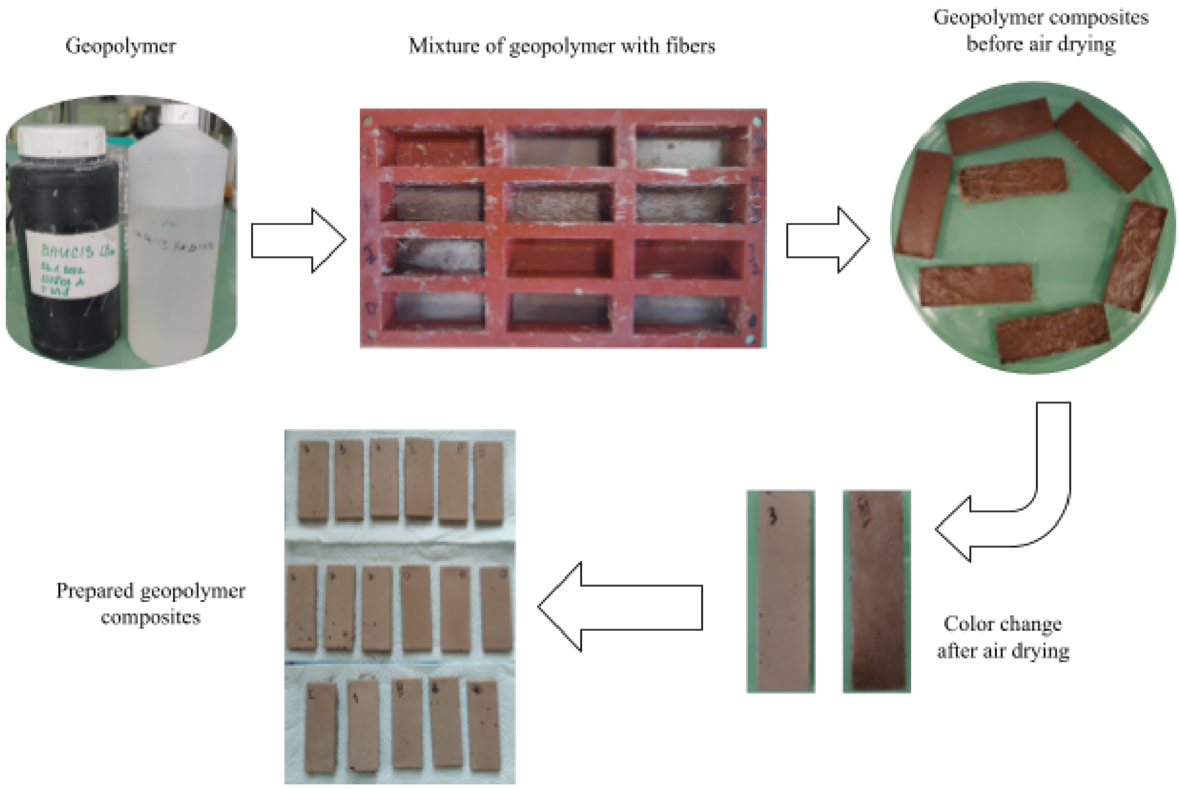


Fig 10. Preparation of geopolymer composites with and without fibers

6.3 Experimental Techniques

Scanning Electron Microscope (SEM)

The morphologies of the treated and untreated coir fibers were observed by Scanning Electron Microscope (SEM), Nova nanoSEM 230 (FEI Europe B.V., Eindhoven, The Netherlands). The surface and cross-section of the fibers were observed at a voltage of 10 kV.

Diameter

100 fibers of each type were randomly selected and were captured under a digital microscope, BRESSER DST 1028. The diameter was calculated using image analysis from Bresser software for each fiber and the average was taken. Fig 11 shows the digital microscope.

Fig 11. Digital microscope

Aspect Ratio

The aspect ratio of the two different types of fibers were calculated using the formula AR = L/D, where L is the average length of the coir fibers and D is the mean diameter.

Fourier-Transform Infrared Spectroscopy (FT-IR)

The untreated and treated, two types of coir fibers were studied and analyzed with the Fourier-Transform Infrared Spectrometer (FT-IR), Nicolet iS50 FT-IR. The data were recorded with the wavenumber range from 400 to 4000 cm^{-1}.

Differential Scanning Calorimetry (DSC)

The changes in the heat flow of the samples were analyzed using Differential Scanning Calorimetry DSC3+, (Mettler Toledo), as it is subjected to controlled temperature changes, with the maximum temperature as 40℃ at a heat rate of 10℃. Fig 12 shows the Differential Scanning Calorimetry DSC3+.

Fig 12. Differential Scanning Calorimetry DSC3+

Thermogravimetric Analysis (TGA)

Thermal stability and weight changes of treated and untreated, two types of coir fibers were tested using a thermogravimetric analyzer TGA/SDTA851 (Mettler Toledo). The fibers were cut into approximately 1 mm specimens and were put in the crucibles. The maximum testing temperature was set to 600℃ with the heating rate at 20℃ and nitrogen. Fig 13 shows the thermogravimetric analyzer.

Fig 13. Thermogravimetric analyzer

Tensile Testing

The change in mechanical properties of the untreated and treated fibers were tested using the tensile tester LabControl, LabTest 2.010 with 50 N, 4 cm gauge length and a speed of 50 mm/min. 10 samples were measured for each type of treated and untreated fibers. Fig 14 shows the tensile tester.

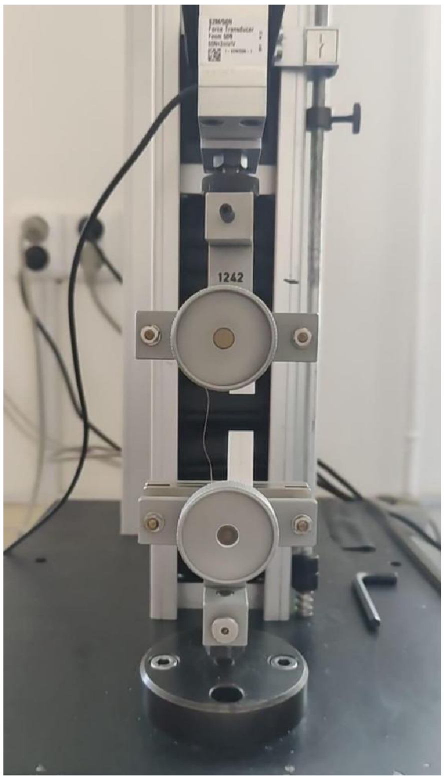


Fig 14. Tensile tester

Flexural Strength Testing

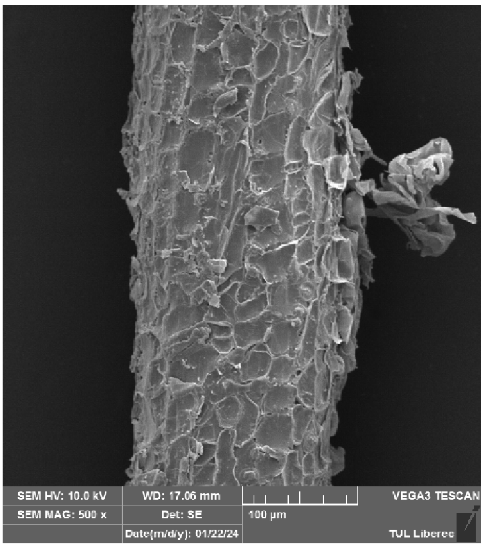
Flexural strength of the geopolymer composites were tested using a 3 point bending machine, TIRA test 2300. 3 samples were tested for different lengths of each fiber sample. Fig 15 shows the testing of flexural strength.

Fig 15. Flexural strength testing

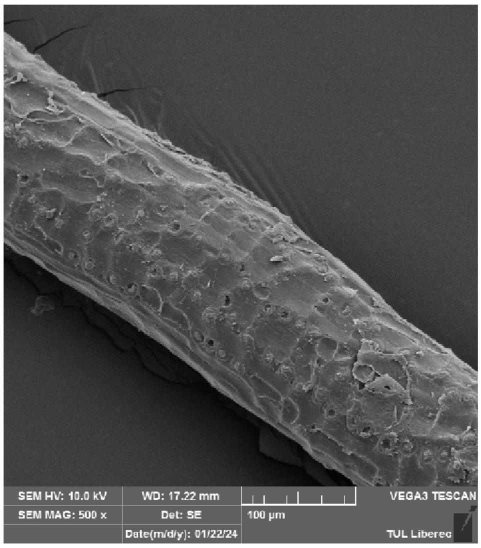
7. Results And Discussion

Morphological Analysis

SEM micrographs of different chemical modification methods are shown in Fig 16. KP and NT fibers before modification are tightly wrapped by impurities (Fig 16 (a), (b)). Due to this, uneven surface structures can be observed. SEM images of fibers modified by methods 1 & 2 (Fig 16 (c), (d), (e), (f)) still show impurities on the surface, while fibers from method 3 are damaged due to the lingering NaOH on the surface and the damages are higher for the fibers treated with a higher concentration of NaOH (Fig 16 (g), (h), (i), (j)). Method 4 has the best results on both the fiber types, with a smoother surface indicating the removal of impurities and the least damage caused in the process as shown in Fig 16 (k) & (l).



(a)



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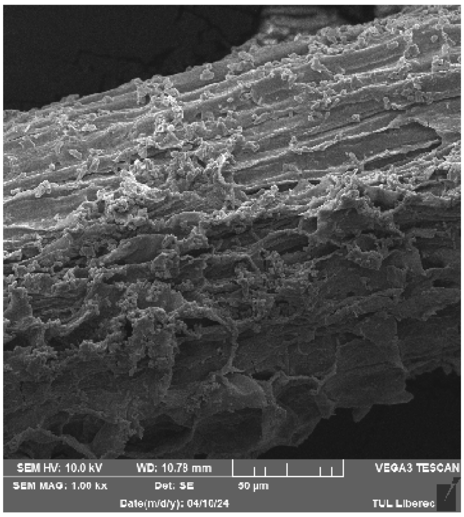
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VEGA3 TESCAN
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Det: SE
50 µm
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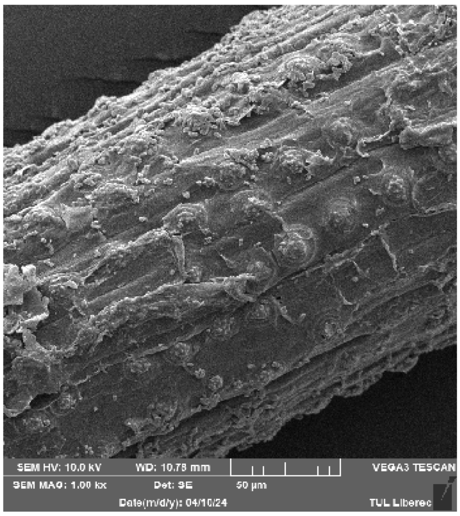
TUL Liberec

(c)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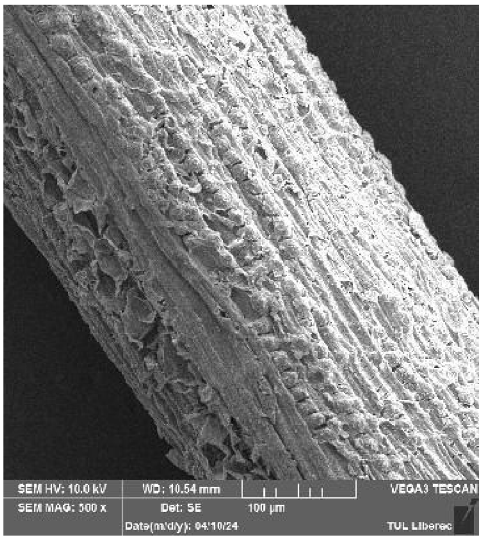
(d)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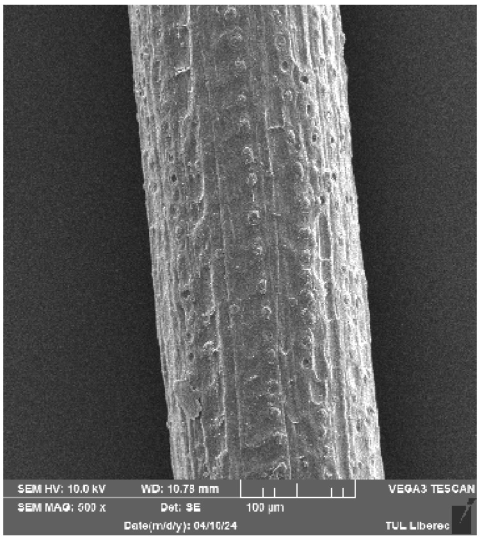
(e)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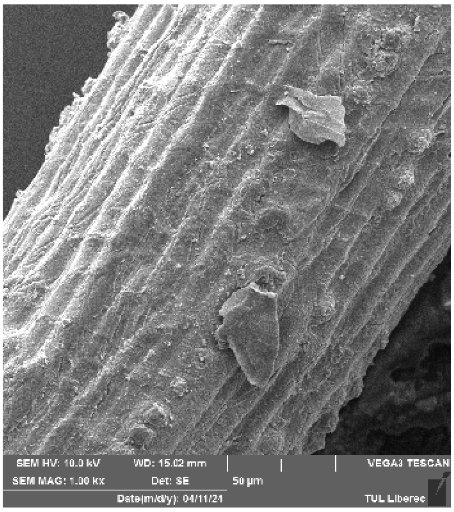
(f)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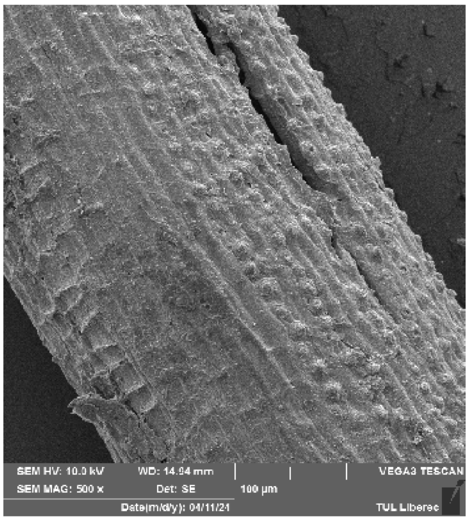
TUL Liberec

(g)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Det: SE
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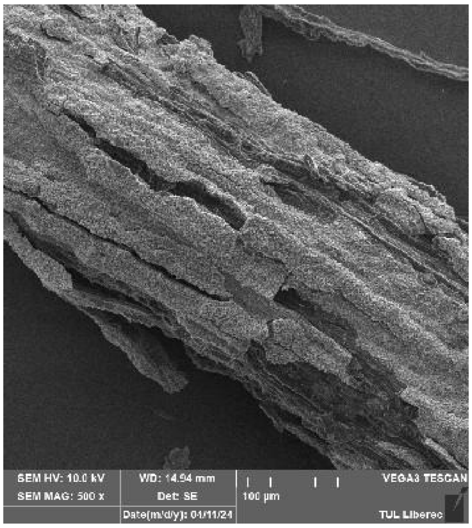
TUL Liberec

(h)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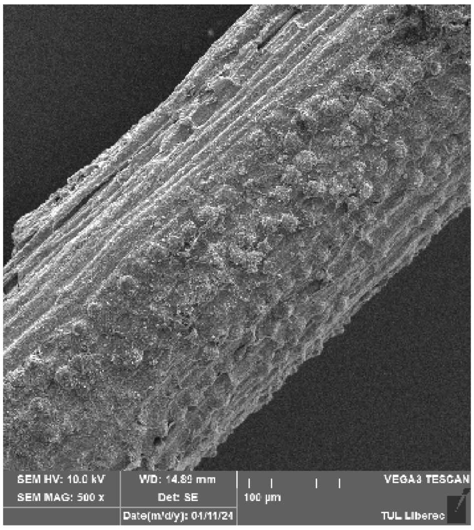
TUL Liberec

(i)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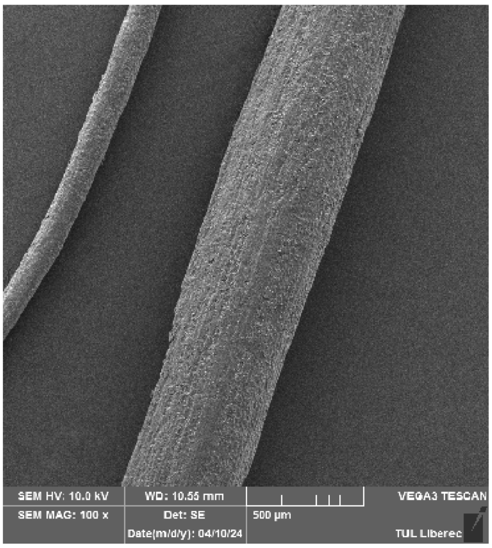
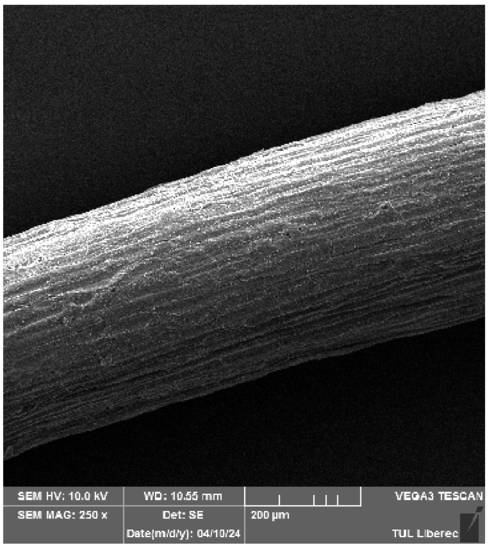
100 µm
Date(m/d/y): 04/11/24
TUL Liberec

(j)



(k)



(l)

Fig 16. SEM images of **(a)** BM-KP **(b)** BM-NT **(c) & (d)** Method 1 - KP & NT **(e) & (f)** Method 2 - KP & NT **(g), (h), (i), (j)** Method 5 - 15% KP, 15% NT, 20% KP, 20% NT **(k), (l)** Method 4 - KP & NT

Diameter and Aspect Ratio

Table 8 shows the mean diameter, length, thickness and aspect ratio of the two types of coir fibers.

Table 8. Physical properties of coir fibers

SAMPLE NAME	MEAN DIAMETER (mm)	LENGTH RANGE (mm)	AVERAGE LENGTH (mm)	AVERAGE THICKNESS (mm)	ASPECT RATIO (L/D)
KP	0.25	65-245	150	0.31	581.62
NT	0.23	55-255	150	0.26	628.14

Aspect ratio is an important parameter in many applications such as composites and textiles. Fibers with higher aspect ratio tend to have a larger surface area per unit volume, which can enhance the bond between the fiber and the matrix in a composite material, leading to improved mechanical properties. And from Table 8, it is evident that there are no significant differences in the physical properties between KP and NT fibers.

Fourier-Transform Infrared Spectroscopy (FT-IR)

The use of FT-IR helps identify the molecules and functional groups that are present in natural fibers. By analyzing the FTIR spectra within the range of 400 to 4000 cm^{-1}, we can get a better understanding of the composition of these fibers. Cellulose, for instance, has a strong crystalline structure that makes it resistant to hydrolysis. However, it is susceptible to acid hydrolysis, which breaks it down rapidly. Hemicellulose, on the other hand, is characterized by a random, amorphous, and hydrophilic structure that functions as a support matrix for cellulose. It has a low specific gravity, can be equally dissolved in alkali and hydrolyzed in acid. Lignin, an amorphous hydrophobic complex polymer, provides plants with stiffness and is not prone to hydrolysis in acid. However, it can be dissolved in hot alkali. Pectin, which is a structured heteropolysaccharide, also has unique characteristics. Fig 17 and Table 9 show the stretching of functional groups at different wavelengths.

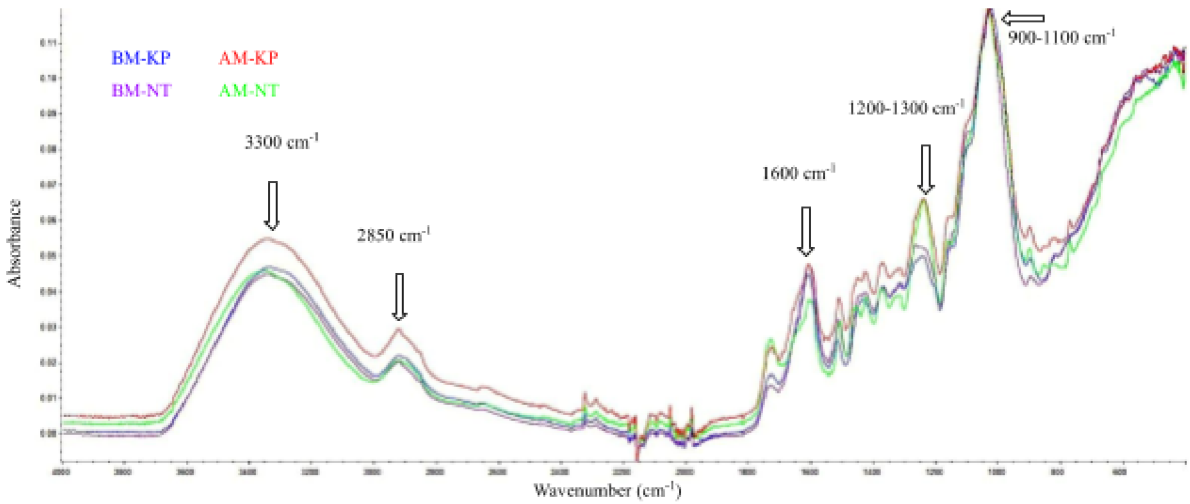


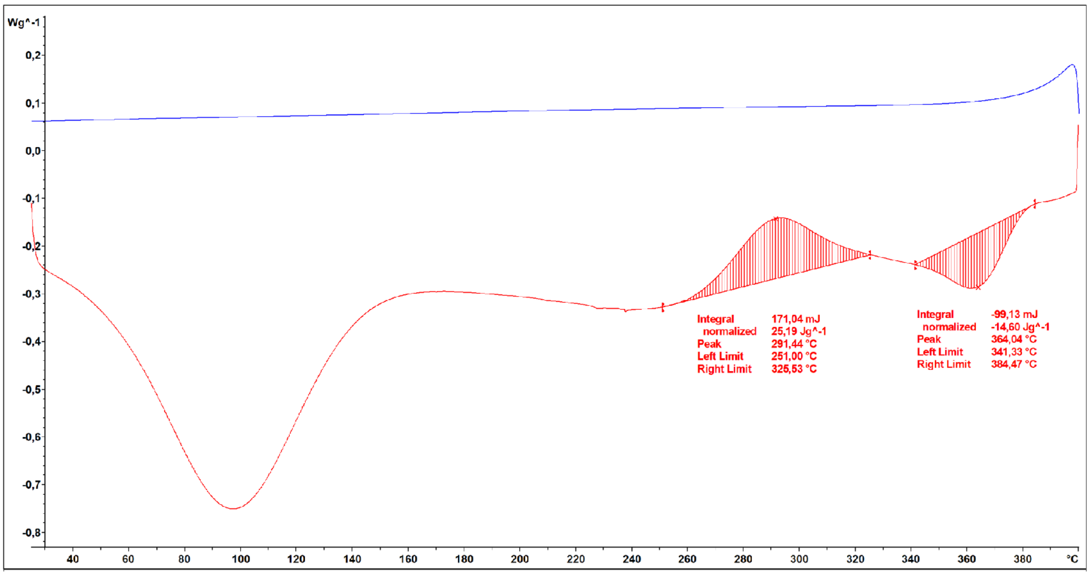
Fig 17. FT-IR spectrum of coir fibers

Table 9. Functional groups at different wavelengths

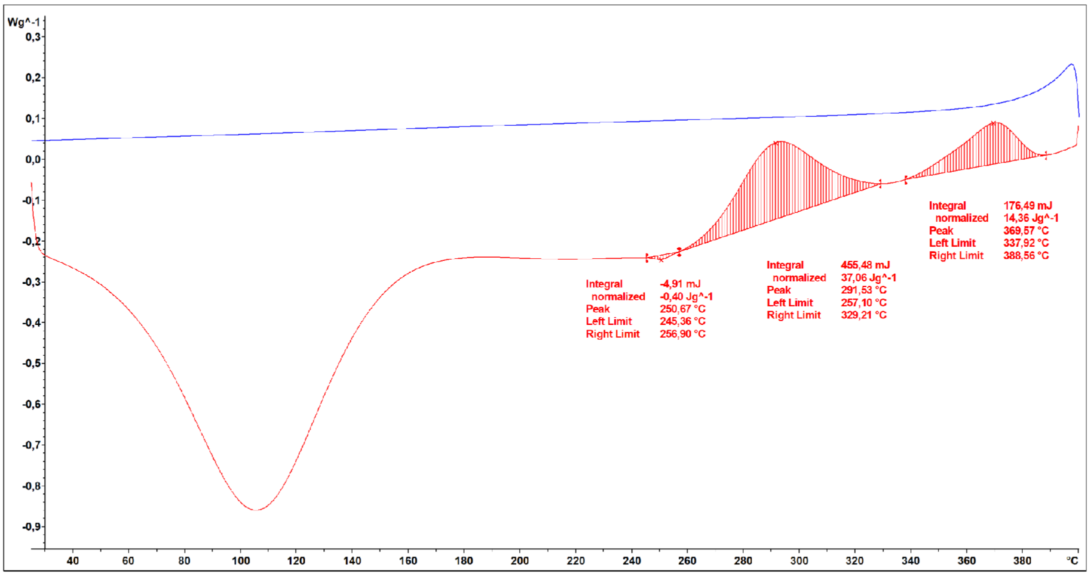
Wavenumber (cm^{-1})	Functional groups
900-1100	C-O stretching of hemicelluloses, lignin C-H out of plane deformation of lignin
1200-1300	O-H in plane deformation of cellulose C-O stretching of hemicelluloses, lignin
1600	C-O Stretching, indicating the presence of hemicellulose.
2850	C≡C stretching. Presence of dispersed wax materials in coir fibers.
3300	Availability of O-H (Hydroxyl group).

Analysis of Thermal Stability of Coir Fibers

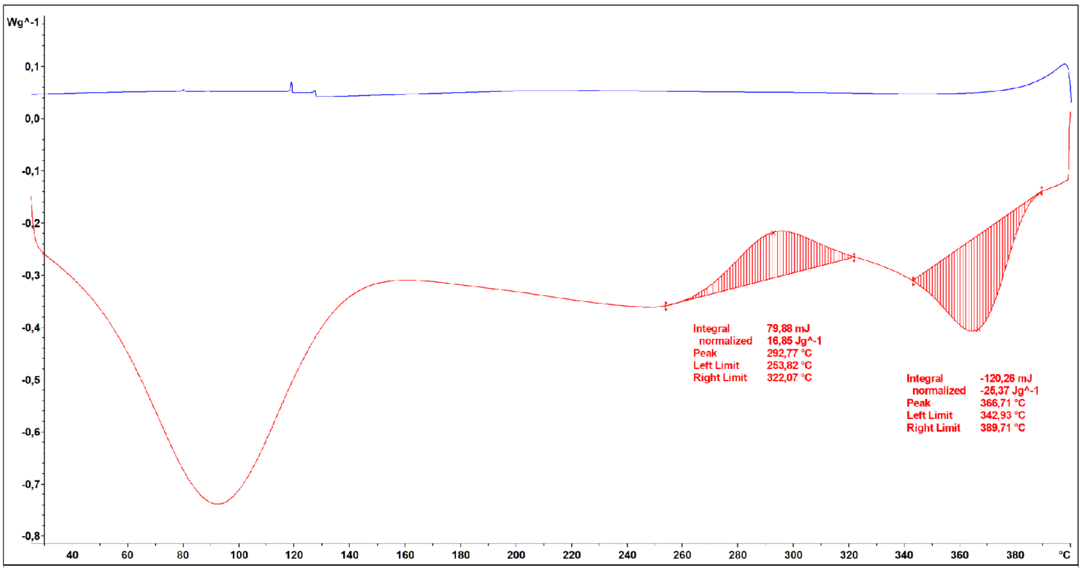
The behavior of the samples due to change in temperature is given in Fig 18. The first curve in the graphs shows the evaporation of water from the sample. In BM-KP & BM-NT (Fig 18 (a) & (c), some exotherm changes are observed between 251℃ & 325.53℃ and 253℃ & 322.07℃ with the peak at 291.44℃ and 292.77℃, which shows the crystallization and indicates the changes in hemicellulose and cellulose in the fibers. An endothermic peak is observed at 364.04℃ and 366.71℃ with limits from 341.33℃ to 384.47℃ and 342.93℃ to 389.71℃ indicating the degradation process of the fibers and changes in lignin. Whereas in AM-KP (Fig 18 (a)), three curves are observed. The first one is an endothermic curve with a peak at 250.67℃, indicating the changes in hemicellulose and the second is an exothermic curve with a peak at 291.53℃, indicating the changes in cellulose. AM-NT (Fig 18 (d)) also has an endothermic curve as the first with the peak at 292.83℃. But, in contrast with fibers before modification, the final curves are observed to be an exothermic change and not much energy was needed to supply to the system with the peak at 369.57℃ and 353.89℃. These images show a significant difference in the second half of the heating process among the fibers before and after modification and the fibers after modification show better results.


Wg^-1
0,2
0,1
0,0
-0,1
-0,2
-0,3
-0,4
-0,5
-0,6
-0,7
-0,8
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
°C
Integral 171,04 mJ
normalized 25,19 Jg^-1
Peak 291,44 °C
Left Limit 251,00 °C
Right Limit 325,53 °C
Integral -99,13 mJ
normalized -14,60 Jg^-1
Peak 364,04 °C
Left Limit 341,33 °C
Right Limit 384,47 °C

(a)


Wg^-1
0,3
0,2
0,1
0,0
-0,1
-0,2
-0,3
-0,4
-0,5
-0,6
-0,7
-0,8
-0,9
40
60
80
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
°C
Integral -4,91 mJ
normalized -0,40 Jg^-1
Peak 250,67 °C
Left Limit 245,36 °C
Right Limit 256,90 °C
Integral 455,48 mJ
normalized 37,06 Jg^-1
Peak 291,53 °C
Left Limit 257,10 °C
Right Limit 329,21 °C
Integral 176,49 mJ
normalized 14,36 Jg^-1
Peak 369,57 °C
Left Limit 337,92 °C
Right Limit 388,56 °C

(b)



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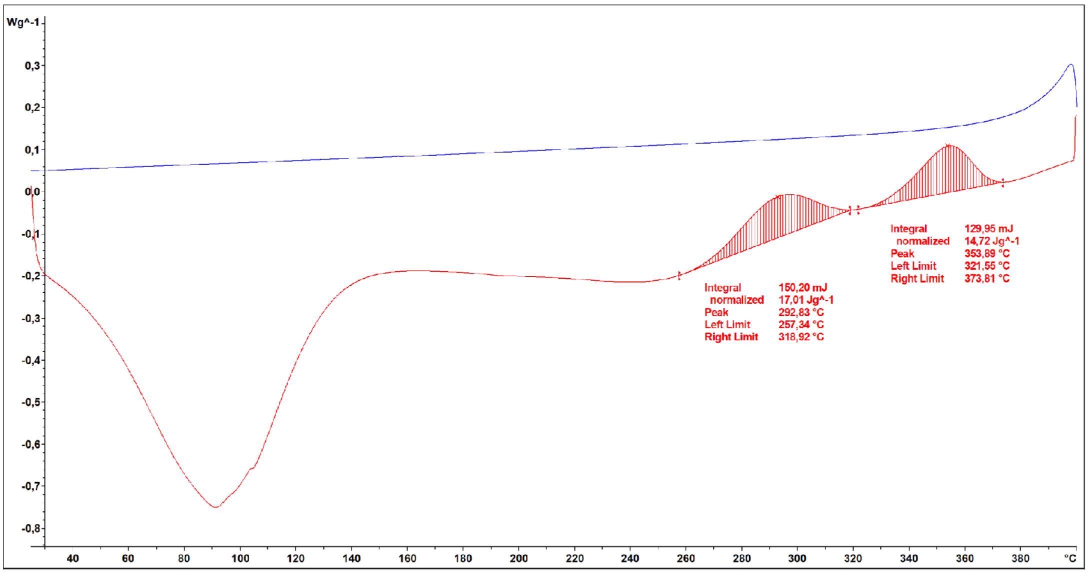


(d)

Fig 18. DSC analysis of **(a)** BM-KP **(b)** AM-KP **(c)** BM-NT **(d)** AM-NT

Thermogravimetric Analysis (TGA)

The thermogravimetric analysis of the two types of coir fibers before and after modification are shown in Fig 19 and Fig 20 shows the weight loss percentage. In the first step, there is a loss in mass due to the removal of water from the samples. The heavy change in the BM-KP's weight due to decomposition happens between the onset temperatures of 277.83℃ & 388.39℃ and the approximate weight loss is observed as 55.86%. This shows that BM-KP fibers are thermally stable until 277℃ and they can't be exposed to a thermal environment higher than this. The onset temperatures AM-KP are observed to be 281.83℃ & 387.91℃ and the weight loss is observed around 55.64%. BM-NT fibers have an onset temperature between 282.13℃ and 391.77℃ with a loss of around 59.64%. The onset temperatures of AM-NT are observed to be 284.20℃ & 375.34℃ and the weight loss is observed as 56.13% which is similar to the KP fibers. This analysis shows that the fibers before and after modification don't have a significant difference between the starting temperatures but the AM fibers have slightly lower ending onset temperatures than the BM fibers.

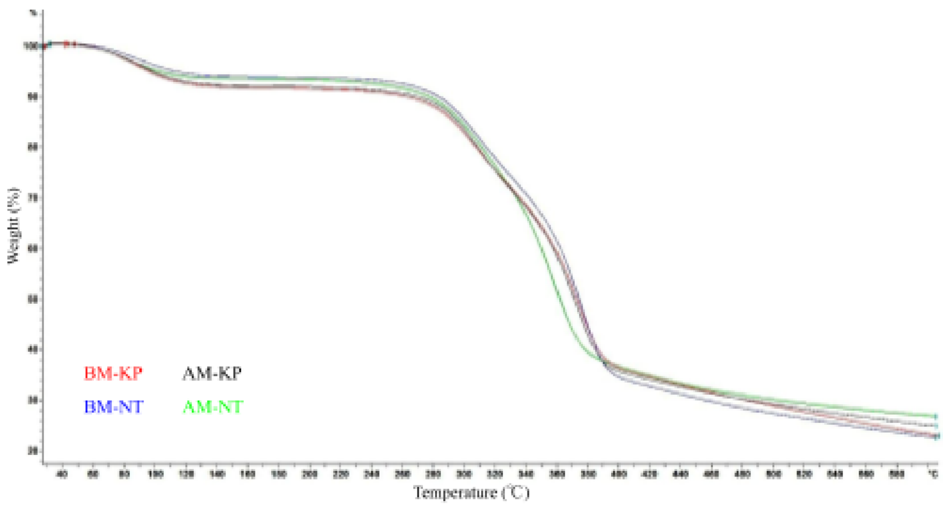


Fig 19. Thermogravimetric analysis of coir fibers

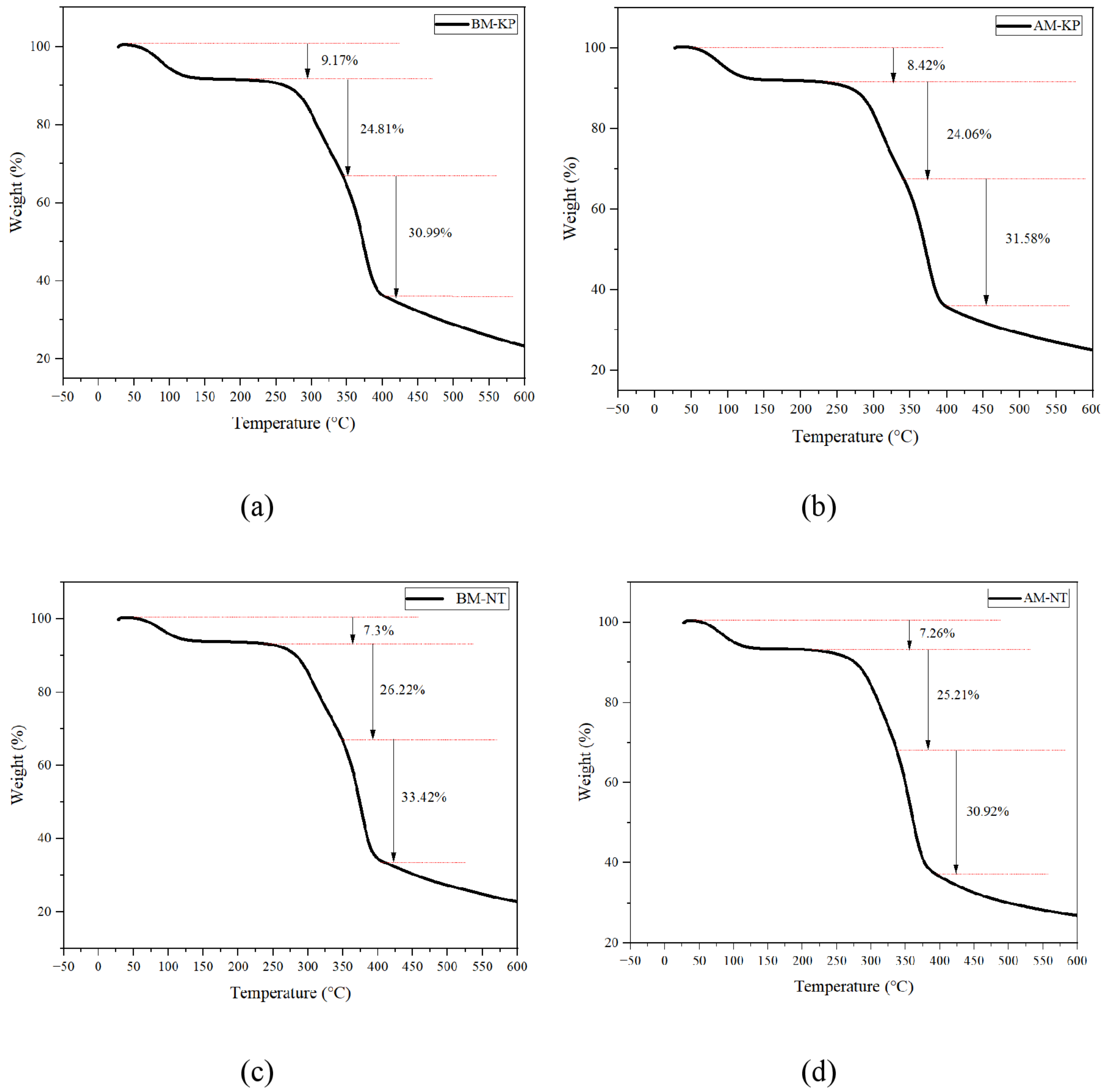


Fig 20. Weight loss graph of **(a)** BM-KP **(b)** AM-KP **(c)** BM-NT **(d)** AM-NT

Analysis of Tensile Properties

The elongation at break, tensile modulus and elongation are shown in the table 10. Fig 21 shows the 3D plot of variations in the reading of all the 10 samples of each fiber type before and after modification. Analyzing Fig 21 and table 10, it is evident that AM-NT shows the highest tensile modulus E, AM-KP has the highest elongation at break compared to the other samples while BM-NT shows the lowest. AM-NT is shown to have the most flexibility and the resistance to deformation than the other samples. Overall, the fibers after modification show a greater increase in the elongation at break showcasing an increased ability to withstand a greater strain without fracturing. So these fibers are suitable for composite materials used in flexible structures.

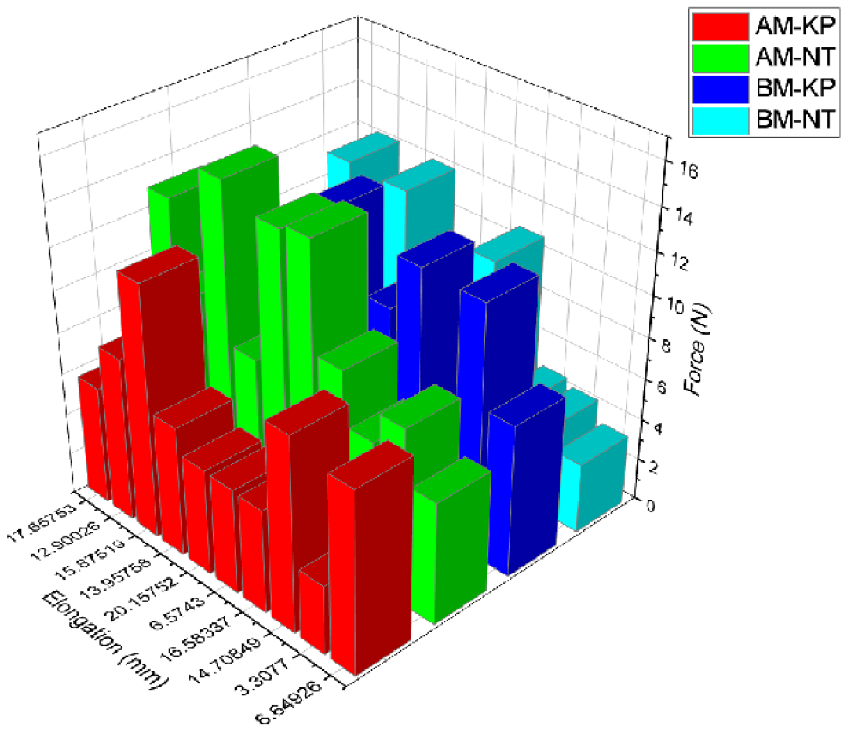


Fig 21. 3D plot of elongation and force of the fibers

Table 10. Tensile properties of coir fibers

SAMPLE	ELONGATION AT BREAK (%)	TENSILE MODULUS E (MPa)	SMAX (ELONGATION)(mm)
BM-KP	22±6.5	236.3±79.5	11.2±3.2
AM-KP	31.9±8.1	183.6±38.6	12.7±3.2
BM-NT	13.3±6.3	205.1±42.8	6.6±3.1
AM-NT	29.8±3.7	274.2±69.6	11.9±1.4

Flexural Strength

Table 11 shows the physical properties of the geopolymer composite samples and Table 12 and Fig 22 shows the flexural testing results of the samples. It can be seen from Fig 22, that the error value for the geopolymer composite with no coir fibers is larger than the composites with the fibers which show a much more reliable value.

Table 11. Physical properties of geopolymer composites

SAMPLE NAME	THICKNESS (mm)	WEIGHT (g)	WIDTH (mm)	LENGTH (mm)
0	4.5	13.6	26.4	75
2 (AM-KP)	4.8	14	26.3	
II (AM-NT)	5	14.1	26.9	
3 (AM-KP)	4.8	14.4	26.6	
III (AM-NT)	5.1	14.5	26.4	

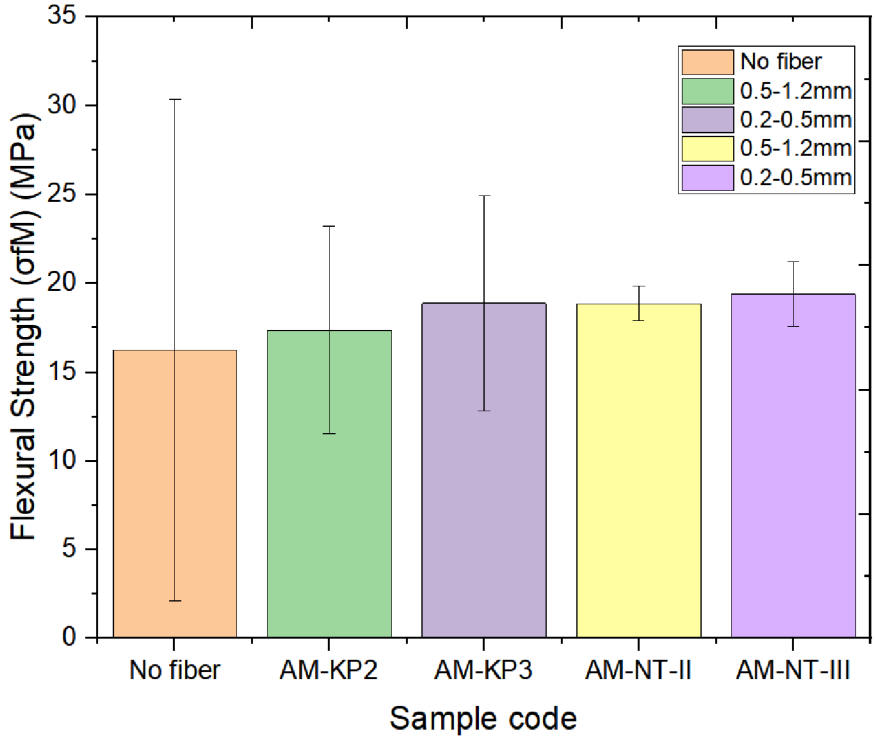
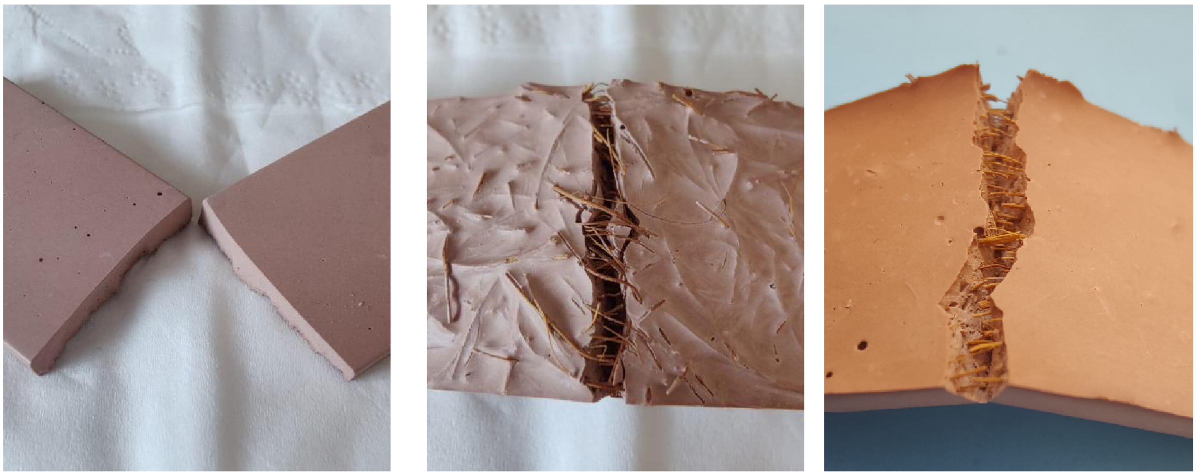


Fig 22. Flexural properties of geopolymer composites

Table 12. Flexural properties of geopolymer composites

SAMPLE	FIBER NAME	FIBER SIZE (cm)	F_{max} (N)	S_{max} (mm)	F_{end} (N)	S_{end} (mm)	σfB (MPa)	σfM (MPa)
0	No fiber	-	29	0.28	10.4	0.7	5.8	16.2
2	AM-KP	0.5 - 1.2	35.2	2.5	15.4	9.3	7.7	17.3
3	AM-KP	0.2 - 0.5	37.6	0.3	7.5	4.3	3.7	18.8
II	AM-NT	0.5 - 1.2	40.4	3.2	18.4	9.9	8.7	18.8
III	AM-NT	0.2 - 0.5	47.2	0.6	12.9	5	5.2	19.3



(a) (b) (c)

Fig 23. Breaking mechanism of geopolymer composites with **(a)** no fibers **(b)** 0.5 - 1.2 cm long coir fibers **(c)** 0.2 - 0.5 cm long coir fibers

Fig 23 shows the breaking mechanism of the geopolymer composites with no and different lengths of coir fibers. On comparing the data in Table 12 and Fig 23, it is evident that samples 2 & II can keep the composites intact for a longer time in comparison to samples as they absorb more energy during the application of force which results in slower deformation and samples 3 & III have a higher flexural strength.

8. Conclusion

This research examines brown coir fibers obtained from two varieties of coconut trees, with the aim of assessing their intrinsic properties. The results demonstrate significant differences between the two types of fibers studied. Notably, the modified fibers exhibit superior surface morphology, with minimal impurities. This improvement makes them highly desirable for subsequent processing stages, presenting a strong foundation for their potential use in various industrial applications.

Furthermore, the study highlights a significant improvement in the thermal response of the modified fibers, positioning them as prime candidates for integration into geopolymer composites. Specifically, the AM-NT fiber variant stands out due to its remarkable flexural strength. Additionally, the research emphasizes the crucial role played by fiber length in enhancing the physical properties of geopolymer composites, highlighting the importance of this parameter in material design and optimization strategies.

In essence, these findings contribute not only to a deeper understanding of characteristics of brown coir fiber of the two selected types but also hold promising implications for advancing sustainable materials with enhanced performance and functionality in various engineering applications. The results of this study provide a valuable foundation for further research and development in this area, with the potential to drive innovation and progress in the field of sustainable materials science.

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