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Extraction and Characterization Study on *Allium cepa L. var. aggregatum* Residual- A Sustainable Fibre for Textile Application

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Abstract

Objective: Currently, most research in the textile field focuses primarily on developing new eco-friendly fibres extracted from plants and their residues, which are incorporated into various textile products. This study investigates the extraction, characterization, and analysis of fibres derived from the roots of *Allium cepa L. var. aggregatum.* This vegetation was selected due to its widespread cultivation as food ingredient; however, its roots are non-consumable and are typically discarded as landfill waste. Therefore, we selected this and examine the potential of these fibres for textile applications. **Methods:** Residues were collected from fields, and fibres were extracted using the water retting method. Subsequently, fibre characterization was conducted to analyse their properties. **Findings:** Fibre characterization include fibre identification, structural analysis, and elemental composition, examined using FESEM and EDX analysis. The presence of chemical groups and the percentage of chemical groups and the percentage of chemical compounds in the fibres were determined using FTIR and chemical composition test. The crystallographic structure was analysed using XRD, a peak point found at 22.09Θ position, revealing a crystallinity index of 78.82%. Thermal stability and melting point under heat exposure were assessed using TGA, which showed a residual mass of 26.97% and DTA which determined a melting point of 436.0^{0}C. Additionally, fibre toxicity was evaluated through an in vitro cytotoxicity test, which indicated 94% cell viability in PBS extract and 89% cell viability in cottonseed oil extract. **Novelty:** This study details the extraction of fibres from the agro-residue of *Allium cepa.L.var.aggregatum,* a material that has not been previously utilized for textile applications. It evaluates the structural thermal, mechanical and biocompatibility properties to assess its suitability for textile applications.

Keywords: Agro- residual; Fibre extraction; Fibre characterization study; Fibre chemical analysis; Thermal stability; Sustainable textile fibre

1 Introduction

Globally, the extensive cultivation of food crops and the operations of food processing industries generate substantial volumes of agro-residuals and processed waste, respectively. While a minor fraction is repurposed, the majority is discarded through environmentally detrimental methods like burning, landfill disposal and water body dumping, contributing significantly to waste management challenges with adverse ecological and potentially lethal consequences. However, these underutilized resources present opportunities for sustainable valorisation into raw materials and by-products, aligning with growing global consumer interest in recycled products and offering a pathway to mitigate environmental pollution.[1] Following food, clothing necessitates substantial resources for its production, with the textile industry requiring approximately 113.8 million metric tons of fibres annually at considerable cost. Utilizing agro-industrial residuals as textile raw materials offers dual benefit: reducing the disposal burden of these wastes and potentially lowering fibre production costs within the textile sector by providing an alternative fibre source.[2] Integrating these concepts, this research explores the valorisation of collected agro-industrial residue as raw material for textile production, a strategy with the potential for significant and sustainable practical impact. The emergence of sustainable fibres derived from these residuals is increasingly evident in the textile industry, supported by numerous studies investigating diverse agro-industrial sources for various textile applications.[3]

Recent research has focused on various plant sources for fibre extraction, though these processes often require substantial resources. In contrast, utilizing agro-industrial residues is more resource-efficient, as fibre extraction from residues does not necessitate specific cultivation methods. Simple mechanical, manual or water retting processes suffice. Accordingly, the roots of *Allium cepa L. var. aggregatum* were selected for this study. To extract fibres from *Allium cepa L. var. aggregatum* roots, they were collected from agricultural lands, sorted based on strength and structure and subjected to the water retting process. Their characteristics were then analysed for potential textile applications. The following factors justify conducting this research study on the extraction and characterization of fibres from *Allium cepa L. var. aggregatum* residual. It is one among the key food ingredient, so its availability is abundant, particularly in the southern regions. Its roots are non-consumables, discarded in landfill as residue that pollutes the environment and creates health hazards. The roots are shorter in length and slightly brittle texture, by utilizing them as textile raw-material are more suitable for composite preparation. The extraction method is relatively simple, requiring only few days. Mainly it is an eco-friendly alternative for traditional textile fibres because it doesn't go through any chemical process for extraction and environmentally safe. To the best of our knowledge, studies on the extraction and characterization of *Allium cepa L. var. aggregatum* root are not yet available.

In this study, residuals were initially collected from agricultural fields, cleaned and placed in a water bath for up to 10-14 days. Afterward, the fibres were shade-dried and prepared for further characterization analysis. Fibre morphology studies such as Field Emission Scanning Electron Microscopy (FESEM), were conducted to analyse the physical structure of the fibres. Energy Dispersive X-ray Spectroscopy (EDX) was used to determine the types of chemical compounds present. Fourier Transform Infrared Spectroscopy (FTIR) was performed to analyse the chemical bonds within the fibre particles. X-ray Diffraction (XRD) analysis was conducted to examine the crystallographic structure of the fibres. Thermogravimetric Analysis (TGA) was used to assess the thermal stability of the fibres, while Differential Thermal Analysis (DTA) was performed to determine their melting point. Additionally, a cytotoxicity test was conducted to evaluate the toxicity range of the fibres and assess their environmental safety. The results of these analysis are discussed in this study and compared with some other plant fibres such as *Cymbopogon flexuous* root, *M. Autopurpurea* fibre, *Cortaderia selloana* grass stem fibre, *Momordica charantia* fibre, *Cordia dichotoma* fibre, *Borassus flabellifer L.* Fibres, *Juncus effusus L.,* and *Cissus quadrangularis* root fibre that have been selected, extracted, and analyzed by various researchers. Significance of this study is to utilize the disposal agro-waste to introduce and incorporate as eco-friendly textile fibre.[3]

About *Allium cepa L. var. aggregatum*

Allium cepa.L.var.aggregatum, commonly known as shallots or small bulb onions, belongs to the *Amaryllidaceae* family- a herbaceous biennial plant.[4] It is a well-known crop, cultivated at an estimated 6.77 million metric ton annually in worldwide.[5] It is typically cultivated in the tropical and sub-tropical regions.[6] In terms of its plantation characteristics, the roots are soft and fibrous in nature, while the leaves are linear and semi-cylindrical, growing up to 40cm in length and 1 cm in diameter. The shallots typically reach a height of 30-60cm, with an outer portion that is thin and dry while inner portion is fleshy.[7] India is one of the largest producers of *Allium cepa L. var. aggregatum,* with a high production rate across the country.[8] In South India, the use of shallots in cooking is indispensable; it is a key ingredient, appetizer in nearly all varieties of spicy foods.[9] Beyond its culinary applications, it is also valued as a medicinal product in traditional medicine and home remedies.[6] It is commonly used to treat hair-related problems, reduce fever and heal wounds.[7] This plant possess significant antioxidant properties and helps regulate cholesterol level due to the presence of flavonoids and quercetin. It is also used in treatment of cardiovascular levels as well as neurodegenerative disorders such as Alzheimer disease. Additionally, it exhibits antibacterial, antifungal, antitumor, anti-larval, anti-allergic, and anti-inflammatory properties.[10]

2 Methodology

2.1 Extraction of *Allium cepa L. var. aggregatum* fibre

In this study, agro-residual of *Allium cepa L. var. aggregatum* (roots) were collected from cultivated fields in Vellode-Erode, where shallots are grown on a large scale. The discarded roots were thoroughly washed to remove dirt, soil, ashes, and other foreign bodies and then sorted based on strength and length. Roots with comparatively higher strength and length were selected for the extraction process. The collection and separation of roots from shallots are shown in Figure 1.[11]

Fig 1. ACF roots are collected from the agro field after harvesting

For this study, watter retting method was chosen for the extraction of *Allium cepa L. var. aggregatum* fibres that yields high-quality fibres. Mechaical extraction does not preserve fibre strength, while chemical retting compromises medicinal properties and it is not eco-friendly. For the water retting process, collected roots of *Allium cepa L. var. aggregatum* were thoroughly cleaned and immersed in water bath for retting, which lasted for 10-14 days. This process was conducted in an open chamber to facilitate microbial growth and anaerobic reactions that reduces the lignin content of the fibre and loosens the fibre layer. Prolonged retting loosens the fibre structure and reducing its strength, whereas shorter duration results in insufficient reaction, producing weak, uneven fibres. For this process, 1 Litre of water at room temperature was placed in a vessel and 50gm of roots were immersed in an open water bath under room temperature conditions to allow microbial reaction.[12] After retting period, the roots were removed and thoroughly washed with running water.[13] During this stage, the swallen outer layer of the shallot roots was removed, exposing the thin, fibreous inner portion. The extracted fibres were shade-dried at room temperature to eliminate excess moisture, yielding thin, lightweight, beige-brown colour fibre. These fibres were then prepared for identification and characterization studies to assess their suitability for textile applications.[11] alternative extraction methods, including mechanical, chemical and water-boiling techniques did not produce fibres of satisfactory quality or quantity. Figure 2 illustrate the process stage: the first image shows the root samples of *Allium cepa L. var. aggregatum* fibres (ACF), the second depicts the retting stage, and the third represents the extracted fibres.

Fig 2. Fibres are extracted from ACF roots by the method of water retting process

2.2 Characterization studies of *Allium cepa L. var. aggregatum* (ACF)

Various analytical techniques, including FESEM, FTIR, XRD, TGA, DTA, chemical composition test and cytotoxicity test were conducted to analyse the characteristics of the fibre and assess their suitability for textile applications. FESEM (Field Emission Scanning Electron Microscopy) was performed to examine the fibre structure and elemental composition.[14] FTIR (Fourier

Transform Infrared Spectroscopy) was used to identify the functional groups in different components based on peak values in infrared spectrum.[15] XRD (X-ray Diffraction Analysis) was employed to determine the crystallographic structure, physical properties, and chemical composition of the fibre sample.[16] TGA (Thermogravimetric Analysis) was conducted to assess the thermal stability of the material and measure the proportion of volatile components by analysing weight changes as the sample was subjected to heat at constant rate.[17] DTA (Differential Thermal Analysis) measured temperature variations and heat flow in the material during thermal transitions.[18] chemical composition test was conducted to assess the presence of cellulose, hemicellulose, lignin, ash, wax, moisture percentage and its density range. Cytotoxicity testing was performed to evaluate the potential harmful effects of the fibres on the living cells, ensuring their safety, and assessing their impact on biological tissues.[19]

3 Results & Discussion

3.1 Characterization Studies

Field Emission Scanning Electron Microscopic Analysis of ACF:

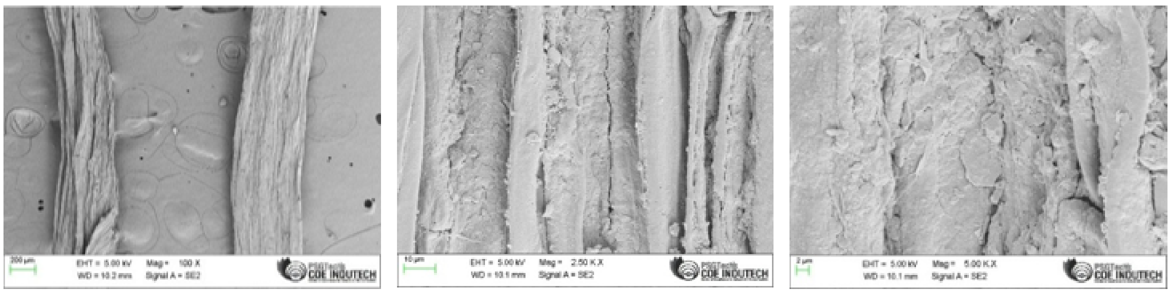

Fig 3. FESEM analysis of ACF

A morphological study of *Allium cepa L. var. aggregatum* agro-residue fibre was conducted using Field Emission Scanning Electron Microscopy (FESEM). The analysis was performed using a BRUKER machine. Non-coated fibre samples were examined at magnifications of 100x, 250Kx, 5Kx, with an accelerating voltage of 5.00kV applied to assess the fibre's morphology and elemental composition. The FESEM images revealed a wax-coated, rigid structure with vertically aligned hollow particles and the presence of dust and foreign bodies. The fibre exhibits a rough, irregular surface with visible longitudinal striations, which may be attributed to the presence of cellulose microfibrils and lignin. And in fibre, minute pores like structure, wax flakes, and nodules distributed randomly across the surface. This composition contributes to the fibre's compact structure and low internal transmission due to the presence of wax flakes; it is suitable to use for thermal and sound insulation applications and by enhancing the fibre structure can be helpful to implement the fibre for eco-friendly textiles, filtration, and disposable applications.

Energy-Dispersive X-ray Spectroscopic (EDX) Analysis of ACF:

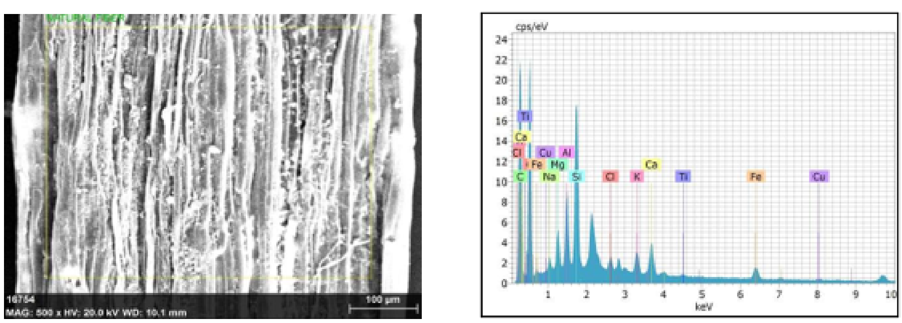

Fig 4. EDX spectrum shows structure of ACF and the elements present in it

In this EDX spectrum analysis, the atomic and weight percentages of elements in *Allium cepa L. var. aggregatum* fibres were calculated. The fibre primarily contains carbon and oxygen with weight percentages of 43.01% and 42.87% atomic percentages of

53.41% and 39.97% respectively. Additionally, the following elements were detected: silicon (Si): 4.67wt%, 2.48 at%, Aluminium (Al):2.37 wt.%, 1.3 at.%, Iron (Fe): 2.05 wt.%, 0.55 at.%, Magnesium (Mg): 1.31 wt.%, 0.80 at.%, Calcium (Ca): 1.82 wt.%, 0.68 at.%, Titanium (Ti): 0.18 wt.%, 0.06 at.%, Copper (Cu): 0.26 wt.%, 0.06 at.% The high concentration of additional elements indicates a significant level of contamination on the fibre surface.[2] Based on the presence of Si Al, and Ti, it indicates the fibre has hydrophobicity, thermal resistance and UV-blocking properties, presence of Cl and Cu indicates the anti-microbial property.

Quantification of chemical composition of ACF:

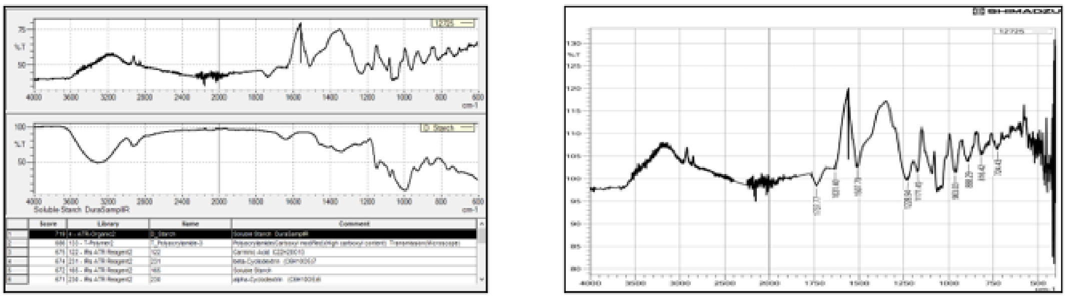

Fig 5. FTIR analysis shows the peak points of chemical compounds in ACF

The Figure 5 presents the Fourier Transform Infrared Spectroscopy (FTIR) analysis of *Allium cepa.L.var.aggregatum* fibres, conducted using a SHIMADZU spectrometer. Infrared light was transmitted through the disc containing the ACF sample, with the wavelength range of 4000 to 500 cm^{-1}. The scanning rate was maintained at 2mm/s. The FTIR spectrum revealed nine distinct peaks, indicating the presence of various chemical compounds. The identified functional groups include- C=O (Carbonyl group): Present at 1737.77 cm^{-1}, indicating the presence of ketones and aldehydes, commonly found in aromatic compounds contributing to smell and taste. C=C, C=N, NH Bonds: Stretching observed at 1631.40cm^{-1}, representing alkenes and nitrogen-containing compounds. Ethylene, an alkene is known for its sweet and musty odour. C-O-C (Ester group): Presence confirmed at 1228.94$cm^{-1}$and 1171.45cm^{-1}. Esters are associated with fats, waxes, and vitamin-C. Ethers, which are structurally similar are widely used as solvents, perfumes, anaesthetics, refrigerants, and disinfectants. Si-O (Siloxane bond) and P-O Phosphorus-oxygen bond): identified at 963.03 cm^{-1}, 888.26cm^{-1}, 816.42 $cm^{-1.}$ C-Halogen (Aromatic rings): Stretching observed at 724.43 cm^{-1}. Soluble starch (D-starch/Dcera): Identified in the fibre and represented in the FTIR graph. These results confirm the presence of multiple functional groups, indicating that ACF fibres contain significant chemical compounds suitable for various applications heat resistance, UV protection, microbial protection, Filtration textiles etc.

Crystallographic Information of ACF:

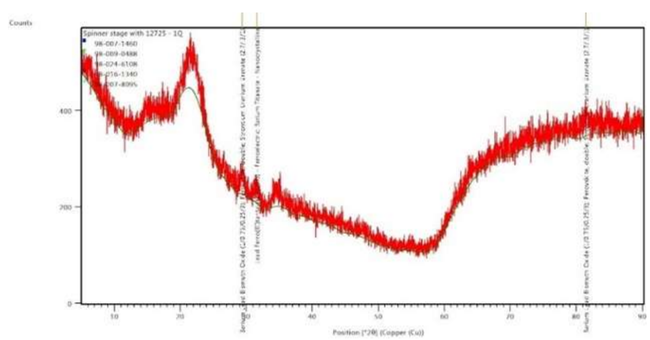

Fig 6. XRD analysis Structure and chemical compounds of ACF

X-ray diffraction (XRD) analysis was performed to determine the crystallographic structure of ACF using a SHIMADZU analyser. In this analysis, fibre samples were placed in a sample holder, and X-rays were passed through the sample at a wavelength of 1.54060Å at a temperature of 25°C. the diffracted X-rays were detected in the range of $2\theta = 5^{\circ} - 20^{\circ}$. The XRD analysis revealed the presence of the following chemical compounds in ACF: Barium lead bismuth chloride

(25%) – Monoclinic crystalline structure, Ferro tantalate (14%)- Tetragonal structure, Perovskite double compound (24%)-Monoclinic crystalline structure, Barium titanate-nanocrystalline (20%)- Tetragonal structure, Strontium uranium uranate (18%)-Monoclinic crystalline structure. The crystallographic investigation of ACF demonstrated a significant decomposition rate with crystalline index of 78.82% calculated with the formula $\mathrm{CI} = \mathrm{Ic} - \mathrm{Ia}/\mathrm{I_c} \times 100 =$ Intensity of the crystalline peak where, Ia = Intensity of the amorphous region. Ic = Intensity of the crystalline peak, and these results indicate that ACF primarily exhibits a high crystalline structure which possess high mechanical strength, low water absorption and better thermal stability. When compared to other cellulosic fibres, Ach has a crystalline index percentage similar to *Cymbopogon flexuosus* root fibre (36.8%) and a higher crystallinity index than *M. autopurpurea* fibre (24.01%), *Cortaderia selloana* grass stem fibre (22%), *Momordica charantia* fibre (21.42%), *Cordia dichotoma* fibre (12.14%), and *Borassus flabellifer* L. (17.13%). Additionally, its relative intensity percentage is comparable to that of *Borassus flabellifer* L. (24.17%). In contrast, common textile fibres such as Banana and Jute exhibit significantly higher crystallinity indices, with Banana fibre at 50.8% and Jute fibre at 63.09%.[20]

Assessment of thermal degradation nature of ACF:

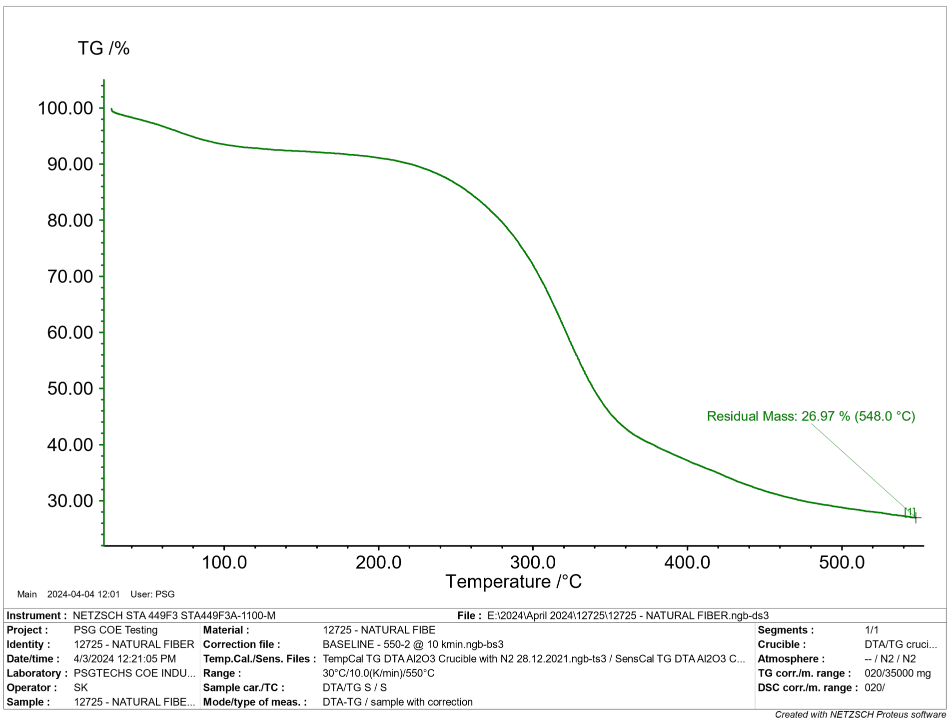

Fig 7. TGA analysis shows residual mass of ACF subjected to thermal contact

Thermogravimetric analysis (TGA) was conducted to determine the optimal processing temperature for plant-based fibres. For ACF sample, TGA was performed using a NETZSCH STA 449 F3 instrument. This analysis supports the findings of the chemical structures of hemicellulose, cellulose, and lignin. Initially, an ACF sample was placed on an alumina pan, and the temperature was set to 30°C. the temperature was then gradually increased to 600°C at a heating rate of 10°C/min. At each interval, the mass of the sample was recorded. The TGA analysis revealed that fibre decomposition occurs in four distinct stages: Stage 1 (30-100°C): weight loss due to the release of absorbed moisture and water content. Stage 2 (100–250°C): weight loss due to the decomposition of cellulose. Stage 3 (250–350°C): Decomposition of lignin content. Stage 4 (350–600°C): Increase in ash content. In this thermogravimetric analysis, the residual mass of ACF was recorded 26.97% at 548.00°C which shows a maximum safe processing temperature and good range of thermal stability of fibre; and the percentage of ash residual shows a moderate range of flame resistance and bio-degradable property of fibre; and low moisture loss results the fibres' hydrophobic structure, this analysis suggests, ACF fibre is more suitable for heat resistance and moisture resistance applications. This TGA range of ACF is better than *Borassus flabellifer* L. (498.2°C), *Mucuna atropurpurea* (298.0°C), *Juncus effusus* L. (300.0°C), *Cissus quadrangularis* root (328.9°C), and Banana fibre (348.6°C). However, it is lower than Jute fibre, which has a residual mass recorded at 687°C under thermal contact conditions.[21]

Assessment of the Chemical Composition of ACF:

The chemical composition analysis of *Allium cepa L. var. aggregatum* fibre revealed that it contains 78.09% cellulose, 29.71% lignin, 8.67% hemicellulose, 16.84% ash, 8.23% moisture, and 1.57% wax which results the better mechanical strength, crystallinity, bio-degradability of the fibre. These values are comparable to other natural fibres, such as: Banana fibre: Cellulose (60–70%), Hemicellulose (10–30%), Lignin (5–12%) Jute fibre: Cellulose (50–70%), Hemicellulose (10–20%), Lignin (7–21%) [22], *Mucuna atropurpurea*: Cellulose (58.74%), Hemicellulose (16.31%), Lignin (14.22%), *Cissus quadrangularis* root: Cellulose (77.17%), Hemicellulose (11.02%), Lignin (10.45%), *Cortaderia selloana* grass stem fibre: Cellulose (53.7%), Hemicellulose (14.43%), Lignin (10.32%), *Momordica charantia* fibre: Cellulose (61.2%), Hemicellulose (17.3%), Lignin (4.8%), *Cymbopogon flexuosus*: Cellulose (74.33%), Hemicellulose (9.76%), Lignin (27.50%), *Screw pine prop* root: Cellulose (73.10%), Hemicellulose (12.58%), Lignin (7.11%) [3]

Assessment of the Melting Point of ACF Subjected to Thermal Contact:

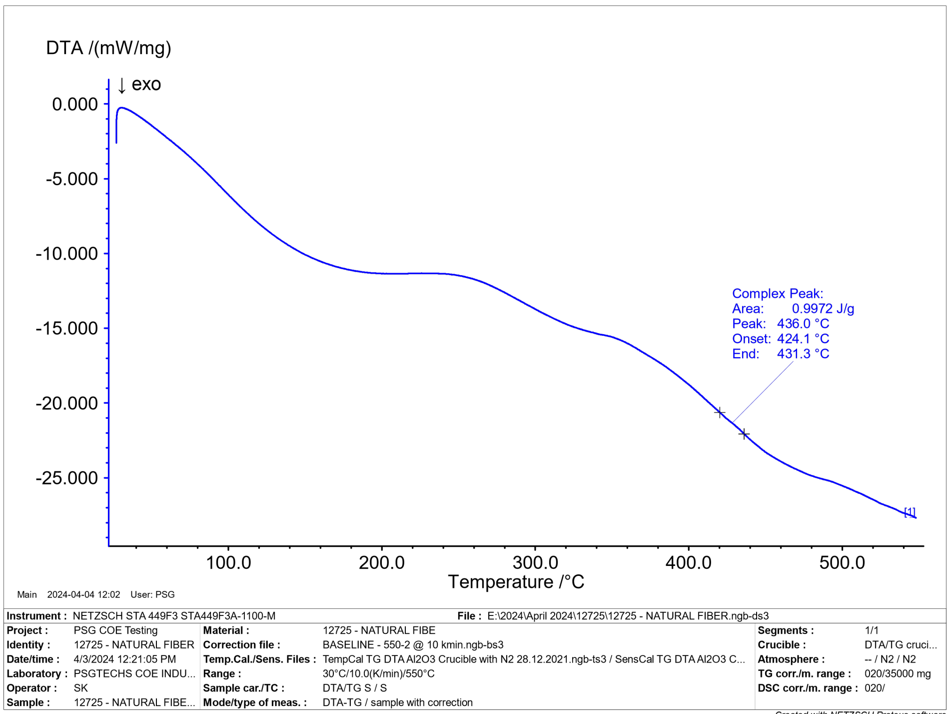

Fig 8. DTA analysis shows melting point of ACF subjected to thermal contact

Differential Thermal Analysis (DTA) is a technique used to measure temperature changes and heat flow in a fibre material during thermal transitions. The DTA analysis of ACF was conducted using the NETZSCH STA 449 F3 analyser to measure its exothermic heat range. The temperature range started at 30°C and increased to 550°C at a heating rate of 10/K min. During DTA analysis, heat transmission was gradually applied to the ACF sample, leading to structural changes in the fibre. The graphical representation of the analysis displays thermal variations in the form of curves. In this analysis, an endothermic peak at 147.8°C was recorded, indicating moisture vaporization from the fibre surface. A glass transition was observed in the range 250-300°C range, as the heat transition increased. The exothermic peak at 436.0°C was attributed to cellulose degradation in the ACF sample.[23] This DTA graph shows exothermic decomposition, and fibre can withstand moderate to high temperature before degradation. It is suitable for aerospace, thermal resistance, and bio-degradable composite applications. When compared to other fibres, the exothermic degradation peak of ACF (436.0°C) was higher than *Borassus flabellifer* L. (332.9°C), *Agave sisalana* (350°C), and Jute fibre (368.8°C), but lower than Banana fibre (481.2°C).[1]

Assessment of the Toxicity Range of ACF (In Vitro Extraction Method):

Cytotoxicity assays are commonly conducted to evaluate the toxicity of fibre samples and assess their potential effects on consumers and the environment. This is achieved by performing contact tests on live cell tissues.[18] In this study, the MTT assay was used to assess the cytotoxicity of ACF fibres on L929 cells. The fibre samples were incubated at 37°C with CO_2 and tested with extracts of Phosphate buffered saline (PBS) and cottonseed oil to determine their reactivity and toxic range. The results indicate that, with PBS extract: The fibre samples exhibit 94% cell viability, releasing 6% cytotoxicity. With cottonseed

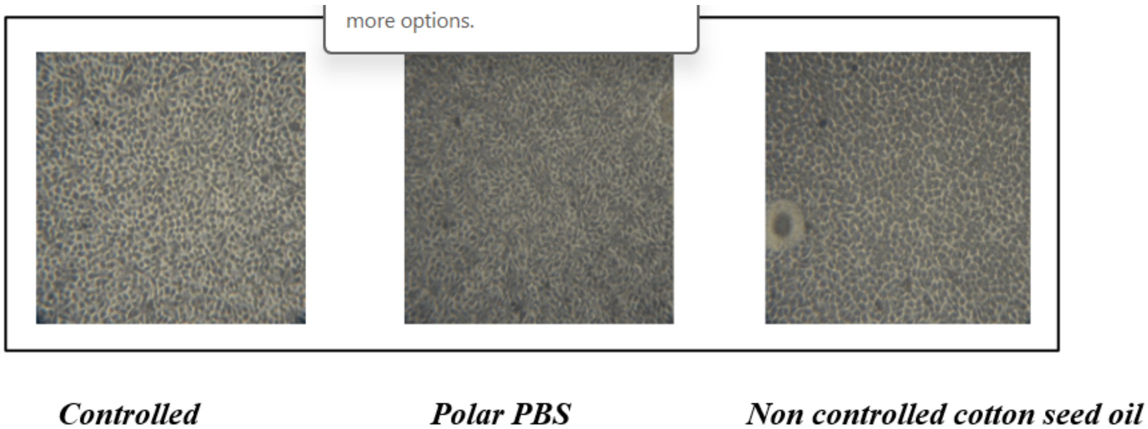

Fig 9. Cytotoxicity assay test shows cytotoxicity percentage of ACF

oil extract: The fibre samples exhibit 89% cell viability, releasing 11% cytotoxicity. The cell viability percentage ranges greater than 80% comes under Grade1, so these results suggest that the sample exhibit no or slight level of toxicity range, which is not harmful for technical textile applications.

4 Conclusion

This study investigated the effects of fibre extraction and analyse the surface structure and chemical properties of *Allium cepaL. var. aggregatum* roots. Classified as agro-residuals, these roots were collected from fields and subjected to water retting for fiber extraction. The extracted fibres were beige to light brown colour, slightly brittle, flexible, and sensitive to extreme pressure.

Surface morphology analysis revealed fibre resulted in minute pores, wax flakes, and nodules distributed randomly across the surface. This composition contributes to the fibre's compact structure and low internal transmission. EDX spectroscopy confirmed the presence of the following elements: carbon (43.01 wt.%, 53.41 at.%), oxygen (42.87 wt.%, 39.97 at.%), silicon (4.67 wt.%, 2.48 at.%), aluminium (2.37 wt.%, 1.3 at.%), iron (2.05 wt.%, 0.55 at.%), magnesium (1.31 wt.%, 0.80 at.%), calcium (1.82 wt.%, 0.68 at.%), titanium (0.18 wt.%, 0.06 at.%), and copper (0.26 wt.%, 0.06 at.%)

TGA analysis showed that significant weight loss occurs in four stages: 30–100°C: Initial weight loss due to the release of absorbed moisture. 100–250°C: Decomposition of cellulose materials. 250–350°C: Decomposition of heavy components such as lignin. 350–600°C: Increase in ash content. At the final stage, ACF retains a residual mass of 26.97% after thermal degradation. DTA analysis revealed an endothermic peak at 147.8°C, indicating moisture vaporization from the fibre surface. A glass transition was observed between 250°C and 300°C, and an exothermic peak at 436.0°C was attributed to cellulose degradation.

X-ray diffraction (XRD) analysis indicated that ACF has a crystalline structure, which contributes to its brittle nature. The material also demonstrated a significant decomposition rate, with a crystalline index of 78.82%. The chemical composition analysis of *Allium cepa.L.var.aggregatum* fibre revealed that it contains 78.09% cellulose, 29.71% lignin, 8.67% hemicellulose, 16.84% ash, 8.23% moisture, and 1.57% wax. Cytotoxicity analysis indicated that ACF samples reacted with PBS extract, maintaining 94% cell viability with a 6% cytotoxicity level, while in cottonseed oil extract, 89% cell viability was recorded with an 11% cytotoxicity level, it comes under grade-1 results no or slight toxicity level that are not harmful to living organisms.

Compared to traditional textile fibres such as cotton, silk, and polyester, *Allium cepa.L.var.aggregatum* fibre has minimal environmental impact. Since it is a plant-based fibre extracted from residual parts, its extraction process does not involve any chemical treatments, unlike traditional fibres, which often require chemical processing. Furthermore, the water used in the retting process can be recycled and reused, making this method sustainable for large-scale production. Based on the characterization, physical, chemical and toxicity analyses, *Allium cepa.L.var.aggregatum* residual fibre demonstrates favourable properties and it is well suited for the production of non-woven sheet composite materials for technical textile applications rather than for woven or knitted fabric production.

5 Competing Interests

The authors declare that they have no competing interests relevant to the content of this article.

6 Acknowledgement

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7 Authors contribution

Conceptualization of study problem, Experimental design, data and report analysis, original article writing: N. Ramya; Research guidance, editing & reviewing: Dr. J. Banu Priya.

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