Mauritius Research Council

INNOVATION FOR TECHNOLOGY

CHARACTERIZATION OF THE MECHANICAL AND CHEMICAL PROPERTIES OF LOCAL INDIGENOUS FIBRES WITH A VIEW TO YIELDING THE STRONGEST POLYMER COMPOSITE

Final Report

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Mauritius Research Council

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Characterization of the mechanical and chemical properties of local indigenous fibres with a view to yielding the strongest polymer composite.

Mauritius Research Council Funded Project URIGS (PMC 42.K.23)

by

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MAURITIUS RESEARCH COUNCIL FINAL REPORT

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Mauritius Research Council

DECLARATION FORM

I certify to the best of my knowledge (1) the statement herein (excluding scientific hypotheses and scientific opinion) are true and complete, and (2) the text and graphics in this report as well as any accompanying publications or other documents, unless otherwise indicated, are the original work of the signatories or of individuals working under their supervision. I understand that willfully making a false statement or concealing a material fact in this report or any other communication submitted to MRC is a criminal offense.

Project Title: Characterization of the mechanical and chemical properties of local indigenous fibres with a view to yielding the strongest polymer composite.

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Abstract

The main objective of the research work was to characterise the mechanical and chemical properties of fibres from a number of endemic and exotic plant species available in Mauritius in order to identify potential fibres that can be used as reinforcing materials in the bio-composites.

To that effect, a set of twenty-five plants were investigated, and useful fibres could be extracted and chemically treated from a final set of fifteen plants. The tensile strength of the fibres (both untreated and treated) was determined as well as the characterization of the chemical properties by FTIR, DSC and TGA tests. The Helium pycnometer density test was performed on two fibres, namely, Dracaena concinna and Ravenala, which are among those with the highest tensile strength.

The results have revealed good tensile strength for the following fibres: Ravenala Madagascariensis, Dracaena floribunda, Latania loddigesii, Dracaena concinna, and Thysanolaena latifolia. The FTIR results have shown that in most cases, the 5 % NaOH treatment at room temperature for 24 hours significantly removed hemicellulose from the fibres. The DSC and TGA results have confirmed the improved thermal stability of the chemically treated fibres. However, there is a need to determine the optimum chemical treatment which would yield the maximum tensile strength for each of the above-mentioned five fibres.

The results of the tensile strength of the bio-composite manufactured with Ravenala Madagascariensis, Dracaena floribunda, Dracaena concinna, and Thysanolaena latifolia, have shown a significantly lower strength as compared to the strength of the resin alone. The SEM image analysis has revealed a poor wettability of the fibres to the resin. This shows that there is a need for further research in order to improve the wettability of the fibres as well as the fibre characteristics in terms of length, density and orientation which can lead to reinforcement in the bio-composites.

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Chapter 1 Introduction

1.1 Brief on the Opportunity of using Natural Fibres

Glass fibre composites are widely used in many countries as well as in Mauritius for various applications ranging from structural applications such as boat hull, swimming pools and domestic water tanks to semi-structural applications such as interior of automobile, letter box, roof tiles, vehicle canopy, domestic furniture, kiosk shelter, etc.

The use of natural fibres in composite applications, when compared to that of glass fibres, has numerous advantages when it comes to environmental and sustainability issues. However, the tensile strength, fibre morphology, and wettability properties remain the main challenges to their wide spread utilisation in different applications in the industrial sectors. It is therefore important to investigate the mechanical and chemical properties of a natural fibre in order to determine the potential of using it for any engineering application. Moreover, the effect of any mechanical and/or treatment on the fibres should also be studied with a view to yielding a fibre with the highest performance in any engineering application while, at the same time, ensuring the least impact on the environment.

Mauritius is blessed with several fibrous indigenous plants, which offer a huge potential for their uses as an alternative to glass fibres in several semi-structural applications as defined above. Also, the investigation of the chemical and mechanical properties of the extracted fibres from the local plants may open avenues for uses of their uses in paper-making, technical textiles and further research in generating nano-materials from them.

In the medium term, the development of a new natural fibre-polymer group of composites can potentially lead to the creation of new job opportunities in terms of new and greener production facilities, growing, harvesting and processing of the leaves. The reduced health hazards as compared to the use of fibre glass is also an important aspect.

1.2 Problem Statement

Composite materials, particularly those manufactured with carbon fibres, fibre glass, aramid fibres, and hybrid (carbon and aramid) fibres have been around for several decades. The main objective of the composite material is to reinforce a low strength substrate such as an epoxy or unsaturated polyester material with thin fibres in order to improve considerably the tensile strength of the composite material.

Currently, suppliers provide different forms of glass fibres, namely, chopped strand mat (csm) of different specific weight (300/450/600 g/m^2), woven roving, tissue surfacing type; which can be used as reinforce materials. The csm normally consists of about 35-50 mm chopped glass fibres which have been randomly oriented and embedded into a thin mat. The mat is

supplied as a roll, and the composite manufacturer can just cut the required shape of the mat and place same in the mould together with the application of a gel coat, and the epoxy or polyester polymer by using the hand lay- up technique.

Glass fibre is commonly used as a reinforcing material in many polymers for a variety of products ranging from structural parts such as speedboat frame and water tank, to non-structural components such as letterbox, interior panel of cars, etc. The significant energy consumption for producing hand-made fibres such as glass fibres, the associated threat to the landfill due to their disposal as well as the global concerns about environmental issues are all favouring the use of naturally available and recyclable materials. The use of the latter in the manufacture of composite materials has found increasing applications in various industrial sectors. For example, abaca-fibres-reinforced composites are being used in under-floor protection of passenger cars by Daimler Chrysler [1]. Studies during recent years on plant based cellulosic fibres [2] like hemp, flax, kenaf, jute, bamboo, pineapple or sisal showed that natural fibre reinforced composite enjoy significant advantages in terms of lower densities, lower abrasiveness, recyclability, high bending resistance, biodegradability, income generation for rural areas and job creation [3]. According to Sena Neto *et al.* [3] in 2010 the world annual production of natural fibres was 28.4 million tons, out of which 7.5 million tons were produced in India. Moreover, Sena Neto *et al*. [3] reported that in 2007, 12 % of the research groups in the area of Metallurgical and Materials Engineering were involved in research related to composites with lignocellulosic fibres. Bledzki *et al*. [4] reported that the consumption of natural fibres in the automotive industry in Europe from 1996 to 2010 has increased significantly from about 4,300 tons in 1996 to more than 100,000 tons in 2010. These authors also reported that the natural fibres are also being used in applications such as packaging in Europe, such as egg cartons in Germany [4]. So it can be seen that there is a worldwide trend to develop natural substitutes to glass fibres for different engineering composite applications.

However, the relatively low-strength of fibre composites have limited their applications to non-structural parts for the automotive industry, namely, covers, car doors panels and car roofs [5]. The low-strength has been attributed to a poor wetting property of the fibre to the polymer matrix which in turn depends on the surface roughness and surface energy of the fibre.

In Mauritius, there are a few companies which are involved in the use of fibre composites on an industrial scale, namely, Resiglas Ltd, Fibre Marine Ltd, Nabridas Ltd and Legend Marine

Ltd. Legend Marine Ltd (G. R. N. W Boat Yard Ltd) is involved principally in the manufacture of pleasure boat, Nabridas in the manufacture of outdoor swimming pools, and Fibre Marine in the manufacture of boat and swimming pools. Resiglas Ltd is also involved in the manufacture of pleasure boat, large water tanks (30 m^3), swimming pools but also smaller items such as meter boxes, letter boxes, electric protection enclosure, roof tile, etc. The larger products such as large water tanks and swimming pools are produced by the spray up technique using unsaturated polyester and fibre glass as reinforcing material. Boat hulls and kayak are normally produced by the hand lay-up technique while the smaller items such as meter boxes are produced from Dough Moulding Compound (DMC) using compression moulding technique.

In the above-mentioned study, Sena Neto *et al*. [3] investigated the mechanical and other properties of six varieties of pineapple leaf fibre to be used in composite. These authors obtained different results in terms of cellulose crystallinity (48.7 – 64.4 %) and tensile strength (212 – 691 MPa) for the different pineapple varieties. The statistical analysis revealed significant differences among the six varieties of leaf, except for the stress-strain. The authors further concluded that the mechanical properties have a direct relationship with the crystallinity. Thus it can be noted that different species of a plant can have different mechanical and chemical properties.

According to Sena Neto *et al*. [3], multiple types of lignocellulosic fibres are available according to their species, places of origin, seasonality and mode of obtainment. They further reported that the generally, higher cellulose percentages, lower microfibrillar angles, higher cellulose degrees of crystallinity and aspect ratios lead to better mechanical properties and lower extensibility.

From the Calvin cycle, it is shown how CO_2 is taken up from the atmosphere and the carbon is ultimately incorporated into organic compounds along with the energy obtained in the light reactions. It is mentioned that in C3 plants (plants with the most common type of photosynthetic pathway) large amounts of the enzymes that catalyze the reactions of the Calvin cycle are dissolved in the chloroplast stroma. The fixation of carbon is thus powered by the light reactions, and the light energy captured by photosynthesis is ultimately stored in the chemical bonds of carbohydrates and other organic molecules. From a diagram of the Calvin cycle it can be noted that the CO_2 (atmosphere) and energy from the light reaction, will influence the cellulose of the leaf [6].

Several methods have been studied to enhance the mechanical, surface properties as well as the adhesion mechanism of natural fibres, namely, physical methods (stretching, calandering and thermotreatment), and chemical methods such as alkali treatment, acetylation, peroxide treatment, and graft copolymerization [7]. Among the numerous chemical treatments, alkali treatment has been acknowledged as the mostly used, least expensive, effective and environment friendly method to improve mechanical and interfacial properties of the fibres [8]. Alkali (sodium hydroxide - NaOH) treatment causes the fibres to fibrillate [9]; a situation whereby the surface area and surface roughness of the fibres increase. The former effect encourages greater absorbance of resin to the fibre while the increased surface roughness will engage with the resin better, known as mechanical interlocking. These mixes of effects create a higher fibre aspect ratio and leads to a better fibre/matrix adhesion.

Plant based fibres consist of three main components; cellulose, hemicellulose, lignin and other surface impurities [2, 8, 9, 10]. Dittenber *et al*. [9] stressed that lignin and other impurities such as wax must be removed if fibres were to be effective as composite reinforcement. Since strength and stiffness of plant based fibres is mainly dependant on their cellulose content, increasing cellulose content is therefore a key factor.

The effects of alkali treatment on tensile and compressive strength of agave fibres were explored by Mylasamy et al [11]. It was found that the alkali-treated reinforced composites offered superior mechanical properties compared to untreated one. This is because alkali treatment has enhanced the interfacial adhesion, surface energy and wetting of fibres. The study also showed that NaOH treatment increases the cellulose percentage through the removal of hemicellulose and lignin [11, 12]. Towo et al [13] evaluated the fatigue properties of sisal fibre composites with epoxy and polyester matrix. They found that composite containing mercerized fibres proved to be better than those with untreated fibres as the fatigue life has increased. Epoxy matrix had longer fatigue life than polyester matrix composites. Constant life diagrams of epoxy matrix composite showed the superiority of alkali treated fibre composite especially for low cycle fatigue [13].

There are several native plants in different countries which have been in use for decades in the textile and composite industries [14]. In Mauritius, there are many local plants where fibres

may be extracted, namely, filao (casuarinas), sugar-cane, pineapple, banana, screwpine and coconut. Given that in Mauritius, different plants grow in different regions, with different edaphic (soil) and climatic conditions and that their ecology can vary (for example some species grow in the vegetation canopy while others are understorey species), it is to be expected that different species would possess variable morphological and anatomical traits that would suit them to their particular environmental conditions. Characteristics of their fibres would be one of those traits.

A recent study by Deesoruth [15] has been undertaken at the Faculty of Engineering, University of Mauritius in order to evaluate the effect of alkali treatment with NaOH on the wetting property of screwpine fibres to an epoxy polymer by investigating the compressive and fatigue strength. The results have shown that at 10 % fibre fraction, the compressive strength of glass fibre composite is marginally higher by 3.7 % as compared to alkali treated fibres. The fatigue strength at a low loading condition, namely, 32.4 MPa, is more than 3 times higher for the treated fibre as compared to the untreated fibres.

Another study has been completed by the authors to further investigate the use of the *Pandanus utilis* fibres as reinforcement in resin. The authors have compared the mechanical and boiling methods to extract the fibres as well as performing tensile tests and chemical tests in order to better evaluate the properties of the Pandanus *utilis* fibres as a reinforcing material in composite. Table 1 shows a list of selected local plants (either non-endemic native or endemic to Mauritius) which could potentially be used as reinforce fibres in composite materials.

Glass fibres are used extensively in Mauritius in the construction of water tanks, boats, swimming pools and furniture [16]. The replacement of glass-fibres, even partially, by any locally-available natural fibre would yield obvious benefits to the Mauritian economy in terms of employment and currency savings.

Table 1: Short list of some local fibrous plants

Endemic/Exotic plant
Acanthophoenix rubra
Afromomum angustifolium
Crinum mauritianum
Cyperus latifolius
Dianella ensifolia
Dictyosperma album
Dracaena concinna
Dracaena floribunda
Flagellaria indica
Hyophorbe lagenicaulis
Hypolytrum mauritianum

Endemic/Exotic plant
Latania loddigesii
Machaerina anceps
Machaerina iridifolia
Phragmitis mauritianus
Scleria sieberi
Typha dominguensis
Ravenala Madagascariensis
Rhapis excelsa
Ficus benghalensis
Ananas Bractaetus
Sporobolus africanus
Thysanolaena latifolia
Panicum maximum

Aim of the Project

Given the above, the proposed study thus aims at identifying local fibres with suitable properties for use as reinforcing material in natural fibre composites.

The main objectives are to evaluate the mechanical and chemical properties of the fibres, and to assess the mechanical performance of the composites made from the selected best fibres.

It is believed that on the long term, the conservation of indigenous plants could also help in creating job opportunities in term of growing, harvesting and processing of the leaves for engineering applications. This would thus lead to the emergence of a different economic sector of activities for Mauritius.

Plan of Work (Methodology)

The following is a list of main research activities to be carried out:

1. Collection of leaves from a range of identified plants. The investigators are proposing to collect green leaves of a proper maturity level in order to obtain the longest fibres from each type of selected plant.

2. The leaves will be washed, cleaned before being passed through a mechanical fibre extractor in order to separate the fibres from their epidermal layers.

3. The thermal analysis of the extracted fibres will be carried. These methods would help in identifying the presence of cellulose, hemi-cellulose and lignin in the fibres prior to any chemical treatment.

4. Scanning Electron Microscopy (SEM) analysis will be done in order to characterise the morphology of the different fibres as well as the cross section view of the fibres.

5. Tensile testing of the fibres will be done on a Testometric Universal Testing Machine (Machine No: 0500-10112) located in the Metrology & Tribology laboratory.

6. The fibres will be subjected to an appropriate chemical treatment solution in order to evaluate the removal of surface impurities.

7. The density of the different fibres will also be evaluated by using the gas pycnometry method.

8. Following the chemical processing of the fibres, chemical analysis will be done in order to investigate the removal of non-cellulosic constitutents.

9. Furthermore, tensile testing of the chemically treated fibres will be carried out to evaluate the Young's Modulus and tensile strength.

10. SEM analysis will again be conducted on the treated fibres to investigate the change in the morphology.

11. The fibres with the highest mechanical strength will be selected for use in the manufacture of natural fibre-polymer composite. An appropriate technique will be used to manufacture composites.

12. A final set of experiments will then be carried out in order to test the tensile strength of the composites.

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CHAPTER 2: LIST OF NATURAL PLANT FIBRES

2.1 Exploitation of natural fibres in Mauritius

In many parts of the world, besides the agricultural purposes, different parts of plants and fruits of many crops have been found to be viable sources of raw material for industrial purpose. Mauritius, being a tropical island, is endowed with vast resources of local indigenous fibres. Only a limited number of plant fibres have been investigated in this island for their suitability in the manufacture of composites structures. It is thought that there is a huge potential in this area to tap into; thus the challenge is to identify the fibrous plants, find the most economical way of extracting and treating the fibres as well as ensuring little environmental impact of the processes. In order to evaluate the sustainability of plant fibres for composite applications, it is preferred to utilise non-food crops. In the Mauritian context, this research focuses on panoply of non-edible natural plant species as tabulated in Table 2.1

Table2.1: List of plants under study , Source: ("Flora of China @ efloras.org," n.d., "Global Plants on JSTOR," n.d., "Useful Tropical Plants," n.d., "Wikipedia," n.d.)

Family/Genus/species/common name	Plant Description
Arecaceae *Acanthophoenix rubra/* **Palmiste rouge** 	**General info:** Acanthophoenix rubra is a single-stemmed, evergreen palm tree growing up to 12 m tall. The unbranched stem, which can be up to 18cm in diameter, is topped by a crown of up to ten red leaves that can each be up to 3 m long. Mature plants have around 10 leaves in total, which radiate from the crown in a spiral arrangement. These comprise a multitude of small, creamy-white flowers suspended from a central stem. The fruits are roundish drupes, up to 1 cm long, which contain a single seed (Royal Botanic garden, 2008). The fibres are aligned parallel to each other, the midrib of the leaves being thicker than those closest to the margin. The plant is classified as 'Critically Endangered' in the IUCN Red List of Threatened Species **Habitat:** Mixed, moist forests at elevations from 250 - 650 m **Uses:** The plant is harvested from the wild for its edible young leaves and medicinal uses. It is cultivated for its edible leaf bud within its native range, and is also widely grown as an ornamental, especially in Mauritius.

Zingiberaceae *Afromomum augustifolium/* **Zedoaire du pays** 	**General info:** It is a herbaceous perennial plant producing dense strands of leafy stems to 1.90 m high, from a tuberous rootstock and also having flowers narrow in short inflorescences, pink or carmine. **Habitat:** Mostly situated in moist shady places in hilly regions. **Uses:** The fruit-pulp is used for Food, while the General root for Medicines and the Stem is served for painkillers. The stems are also beaten for use as a rope in Nigeria to tie up guine. The seed extract has also been shown to have anti-aging properties. Other applications include: farming, forestry, hunting and fishing apparatus.
Amaryllidaceae *Crinum Mauritianum* 	**General info:** *Crinum mauritianum* is an herbaceous plant, being endemic to Mauritius. Plants reaches upto 3-4 ft overall height. The 1-1.3 m long leaves are not distichous. The flowers are pure white with faint purple tinge along margins of perianth parts. It was believed to be extinct, but was rediscovered in 1973, near Midlands Dam (Barrage de Midlands) in Mauritius. Due to its white flowers, it has become an ornamental in Mauritius, frequently used in landscaping. **Habitat:** Crinum. M is usually Grown in muddy soil on the shores of shallow river side pools. It usually forms large colonies locally on moist soil banks and in shallow river pools, at and above water level in full sun.

Cyperaceae *Cyperus latifolius* 	**General info:** *Cyperus latifolius* belongs to the sedge species. It is a tall plant, growing up to 1 m in height, with creeping rhizomes and erect, triangular stems, each terminating in an inflorescence. The species is native to Mauritius. **Habitat:** It grows in shallow water or on damp ground, such as at pond edges.
Xanthorrhoeaceae *Daniella ensifolia/* **Reine des bois** 	**General info:** *Dianella ensifolia* is an evergreen, perennial herb with grass-like leaves growing from a branched, gradually spreading rhizome. It can grow up to 1.5 m tall, spreading at the roots to form quite large clumps. **Habitat:** In its native range, this species occurs in a broad ecological range, from seashores, grasslands, and low altitude evergreen rainforest, often beside rivers, in deep shade of forest floor, usually forming dense stands. Forests and grassy slopes from near sea level to elevations of 1,700 m **Uses:** The plant is sometimes gathered from the wild for use as a medicine, insect, pesticide and dye. It is grown as an ornamental, especially in rockeries

Arecaceae *Dictyosperma album*/ **Hurricane palm** 	**General info:** Hurricane Palm is a single-stemmed, slender, evergreen feather palm that can grow up to 20 m tall. The unbranched stem can be up to 16cm in diameter with a swollen base; it is topped by a crown of 10 - 20 leaves that can each be up to 3 m long. The leaves are dark green in color, and emerge from the rachis in a single plane. It is commonly called princess palm or hurricane palm, the latter owing to its ability to withstand strong winds by easily shedding leaves. The ovoid fruit ripen to purple or black in color, containing one brown, ellipsoidal seed. (Riffle *et al*., 2003) **Habitat:** Grows at low elevations generally up to 600 m **Uses:** A very attractive palm, it is widely grown as an ornamental in the tropics
Asparagaceae *Dracaena concinna*/ **Bois de Chandelle** 	**General info:** This plant is endemic to Mauritius. It has large and straight leaves arranged in whorls (as in a bouquet). The branches are straight up, with only very individuals having many branches and big orange fruits. It is an ornamental plant, the endemic green gecko reside within its leaves. (World environment day, 2014). Note: The fibres in the leaves are concentrated in the middle.
Asparagaceae *Dracaena floribunda* 	**General info:** Thin straight leaves arranged in whorls on the tip of the branches, flowers are like white brushes that attract birds and insects. The fruits are little white coloured, appreciated by birds. **Uses:** It is an effective air cleaner and is among the best plants for removing formaldehydes (toxic organic compounds found into tobacco, vehicle exhaust, and nail polish). (World environment day, 2014) Note: The fibres in the leaves are concentrated in the middle.

Flagellariaceae *Flagellaria indica*/ **Rotin du pays** 	**General info:** Also known as False rattan, Flagellaria indica is a semi-woody climbing plant with stems up to 15- 20 m long. The leaves are unusual in as much as they terminate in a coiling tendril that helps the plant to climb. **Habitat:** It is grown in Moist littoral forests, mangrove swamps, freshwater swamps; from near sea level to elevations of 1,500 m. **Uses:** The plant is often gathered from the wild for local use, mainly as a source of material for food and medicine. The bark and tough stems are used for making mats, baskets, fishtraps, and for constructing huts and roofs
Arecaceae *Hyophorbe lagenicaulis/* **Bottle palm** 	**General info:** The bottle palm is naturally endemic in Mauritius. Bottle Palm has a large swollen trunk. It is a myth that the trunk is a means by which the palm stores water. Bottle Palm has only 4-6 leaves open at any time. The flowers of the palm arise from under the crownshaft (Page,1998). The fibres are similarly arranged in the leaf structure. While habitat destruction may destroy the last remaining palms in the wild, the survival of the species is assured due to its ubiquitous planting throughout the tropics and subtropics as a specimen plant. **Habitat:** Dry coastal forest

Cyperaceae *Hypolytrum mauritianum* 	**General info:** *Hypolytrum mauritianum* is assessed as Vulnerable. This species is known to occur in less than 10 locations and there is a continuing decline in the extent and quality of the habitat. **Habitat:** The species is distributed only in low altitude (0-800 m) evergreen humid forests which are extremely threatened by habitat destruction as a result of slash and burn cultivation. In Mauritius only an estimated 5% of natural vegetation survives (Schipper 2001). Additional problems, include introduced animals (grazers and herbivores) and invasive alien plant species.
Arecaceae *Latania loddigessi/* **Latanier bleu** 	**General info:** *Latania loddigesii* is a species of palm tree. It is endemic to Mauritius, where the only remaining populations are located on the offshore islands. Its common names include *Latanier de l'Ile Ronde* and *Latanier de Maurice* The leaf color is a pale waxy blue to blue-green. The leaf sheath splits to form a V-shape below the petiole. The petiole is smooth and sharp-edged or armed with minute teeth along the margin near its base, and the hastula is flat and somewhat arrow-shaped. The fibres are concentrated in the middle part of the leaves **Habitat:** Coastal, dry forest **Uses:** It is grown as an ornamental plant

Cyperaceae *Machaerina anceps* 	**General info:** The name Machaerina comes from the Greek word machaira meaning big knife, referring to the shape of the leaves. There are two species; Machaerina anceps and Machaerina iridifolia. The species in the genus are rhizomatous perennials. The leaves are mainly basal, with a few cauline, laterally compressed, distichous and equitant at base. The culms are tufted and pithy. The inflorescence consists of several partial panicles. The flowers may be bisexual or male. The two species are very close. Machaerina anceps is differentiated by little inflorescences (small panicle, few glomeruli) and a seed with a thick beak and the latter is endemic in Mauritius and seems to be a species ostensibly extinct in Réunion. **Habitat:** Wetlands, understory, open areas
Cyperaceae *Machaerina iridifolia/* **Canne marronne** 	**General info:** Machaerina iridifolia belongs to the Cyperaceae family Machaerina iridifolia, whicch is a perennial herb that develops a scaly rhizome. The firm, smooth, dark green leaves are persistent, and grow fan-shaped. They are ensiform (in the form of a sword). Inflorescence paniculated at the top of the stem. The branches are erect, almost smooth with ultimate branches more or less arched, solitary or geminated glomeruli very dense, oval spikelets, subcoriaceous glumes, brownish with sharp keel. The fruit is a round triangular achene. **Habitat:** This fairly common species occurs in the wetlands, riverside and understory of the island

Poaceae *Phragmitis mauritianus*/ **Roseau du pays** 	**General info:** *Phragmites mauritianus* is vigorous and fast growing robust perennial grass with very long creeping rhizomes and stolons. The stem reaches about 4-8 m high whilst the leaves are arranged alternately. The growth rate after cutting is about 30 t/ha of dry matter in a period of 4 months. **Habitat:** *Phragmites mauritianus* is common along rivers, lakes and dams and in swamps, floodplains and vleis, often growing partially submerged. It occurs at altitudes of 580–1500 m. **Uses:** *Phragmites mauritianus* can be used for soil stabilization, especially along shores of lakes and streams. It is planted in basins and constructed wetlands to treat mainly domestic waste water, but also waste water from industrial sources. Dry stems are viable fuel sources. The stems are used for thatch and to make walls and partitions in houses, fences, and as insulation material. They are also split and used for plaiting and wickerwork
Cyperaceae *Scleria sieberi* 	**General info:** *Scleria* are known commonly as nutrushes. They are distributed throughout the tropical world, and some have ranges extending into temperate areas. There are about 200 species. In Mauritius, there are 2 species of scleria genus, whereby one of them is extinct and the other species left is the *scleria sieberi* solely. Plants of this genus reaches upto 1 m long. These are mostly perennial, but sometimes annual. Some have rhizomes. They produce solitary stems or clumps. They have few leaves. The inflorescence is variable, bearing a single spikelet to over 100. **Habitat:** Understory and open areas

Thyphaceae Typha dominguensis/ **Voune** 	**General info:** Typha domingensis is a very vigorous, herbaceous perennial plant. It is a perennial growing 1- 3 m long with spongy strap-like leaves. Typha domingensis is available abundantly in nature and is renewable. Growing from a fast-spreading rhizomatous rootstock, it forms a colony of unbranched leafy stems 150 - 400cm tall. **Habitat:** In frost-free climates that plant grows all year round. It is mostly found in regions where there is a stock of water, sewage water in open places, and also in waste lands. **Uses:** These plants are helpful in some cases, such as chemical wastewater from industry where they absorb harmful water. The plant is also planted to provide wildlife habitats and for wetland restoration. It is grown as an ornamental, though it needs to be restrained at the roots in all but the largest areas. **Problem:** The plant can become a serious weeds in managed aquatic systems worldwide, where it can invade canals, ditches, reservoirs, cultivated fields, and farm ponds. It can be a nuisance in recreational lakes; and can reduce biodiversity and displace species more desirable for certain kinds of wildlife.
OTHERS	

Poaceae *Chrysopogon zizanoides*/ **Vetivert** 	**General info:** *Chrysopogon zizanioides* is a coarse, evergreen, perennial grass forming large, dense clumps 1 – 3 m long. The plant has a stout, compact, aromatic, branched, spongy rhizome and fibrous root system growing to a depth of 4 m **Habitat:** It is a plant of the tropics, where it is found at elevations up to 2,500 m. It grows best in areas such as floodplains and the banks of streams and rivers where annual daytime temperatures are within the range 22 - 35°c. **Uses:** Vetiveria grass is the source of a valuable essential oil, for which purpose it is often grown commercially. Amongst its many other uses, it provides material for thatch, has many medicinal applications and is cultivated to protect the soil from erosion.
Cyperaceae *Cyperus distans*/ **slender cyperus** 	**General info:** Slender cyperus is a grass-like perennial plant producing solitary or small clumps of culms 35 - 110cm tall from a short, corm-like rhizome. **Habitat:** It is located in Marshes at sea level, a pantropical sedge occurring in wet grasslands and along waysides Forests, swamps, grasslands, slightly dry or wet places on slopes, river margins, along trails; near sea level to 1,800 m. **Uses:** The plant is sometimes gathered from the wild for local use as a food, medicine and material for weaving.

Moraceae *Ficus benghalensis*/ **Banyan** 	**General info:** The banyan tree is an evergreen tree with a wide, spreading crown; it can grow 20 - 30 m or more tall. The plant usually begins life as an epiphyte, growing in the branch of another tree; as it grows older it sends down aerial roots which, when they reach the ground quickly form roots and become much thicker and more vigorous. They supply nutrients to the fig, allowing it to grow faster than the host tree. The aerial roots gradually encircle the host tree, preventing its main trunk from expanding, whilst at the same time the foliage smothers the foliage of the host. Eventually the host dies, leaving the fig to carry on growing without competition. It can become a very large, spreading tree in time, with some specimens several 100 m across and producing aerial roots from the spreading branches that eventually become new trunks and allow the crown to spread even further **Habitat:** Monsoon and rain forests, Evergreen to deciduous lowland forest **Uses:** The tree is harvested from the wild for its edible fruit and medicinal uses. It also supplies a timber and a material for tying. The tree is considered sacred by Hindus and is commonly planted for religious purposes, it is also grown as an ornamental and to provide shade along roads as well as in parks and large gardens

Poaceae *Panicum maximum*/ **Fatak** 	**General info:** *Panicum maximum* is a perennial, tufted grass with a short, creeping rhizome. The stems of this robust grass can reach a height of up to 2 m. As the stems bend and nodes touch the ground, roots and new plants are formed. The leaf sheaths are found at the bases of the stems and are covered with fine hairs. **Habitat:** It is situated in Grasslands, open woodlands and shady places. **Uses:** This species is one of the most important cultivated range and fodder grasses of lowland and can also be grown to produce biomass for making alcohol. It is widely cultivated as pasture and is especially used to make good quality hay. It is also used locally as a source of materials and for a few minor medicinal uses.
Thysanolaeneae *Thysanolaena latifolia*/ **Fatak Broom** 	**General info:** Asian broom grass is an evergreen, clump-forming grass with bamboo-like culms 2 - 4 m tall. **Habitat:** It is found mostly in valleys and lightly shaded slopes; ravines; river banks; forest margins; open grasslands. Usually in association with trees (such as bamboo), solitarily or in small groups, not in full sunlight; at elevations from 150 - 2,000 m **Uses:** Due its durability, the plant is commonly used to make a light dust brush and brooms which is extensively sold in local markets. It is also cultivated as a hedge or screen, and is also grown as an ornamental.

Poaceae *Sporobolus africanus*/ **Patte poule** 	**General info:** *S. africanus* is an erect, tough, caespitose, perennial tussock grass usually growing to 60 cm- 1 m in height. According to Parsons and Cuthbertson (2001), stems are dark green, upright and slender. Leaves are dark green, glabrous, mostly occurring around the base, and are slender and stiff, to 18 cm long, acuminate and with in-rolled margins. The roots are fibrous. **Problems**: It is an invasive tussock grass of low palatability and regarded in Australia as a very serious and declared weed. It is dispersed very easily by several mechanisms and once established it can quickly dominate existing pastures, causing loss of productivity and reduced land values. Its presence may also be an indicator of reduced soil fertility and pasture mismanagement. Control on extensively grazed properties is problematic and every effort must be made to prevent its introduction to clean properties.
Strelitziaceae *Ravenala Madagascariensis* 	**General info:** Also known as Traveller's tree, Ravenala is an evergreen tree with an open, fan-like crown and it usually grows up to 20- 30 m tall. The cylindrical stem can be solitary, or branched at the base. The plant produces large, banana-like leaves that can each be 2.5 - 4 m long and 80 - 150cm wide on top of a stout petiole that is 3 - 6 m long. These are held in a fan-shaped formation of 20 or more leaves. The plant produces an edible seed that is sometimes gathered from the wild for local use. It gains its common name from the fact that copious amounts of rainwater and liquid plant secretions can collect in the leaf bases and leaf bracts of the plant. And, these have been used as refreshment by thirsty travelers. **Uses:** The tree is widely cultivated as an ornamental in the tropics. The leaves are used for roofing and as a packing material. The leaf petioles and midribs are used for making walls. The bark is used for making floors in houses. The stem is used in house construction.

Bromeliaceae *Ananas bracteatus*/ **Ananas marron** 	**General info:** *Ananas bracteatus* (common name, red pineapple) is a species of the pineapple. *Ananas bracteatus* is a large terrestrial species of bromeliad that grows 100 cm dark green leaves that fade red to pink when exposed to sunlight. The long spiny leaves are characterized by broad, cream and green, longitudinal stripes that are suffused with pink when grown in good light. **Uses:** *Ananas bracteatus* is grown as an ornamental plants for its decorative red fruit. The leaves are long with sharp spines, so it can be used as a protective hedge for home security. In colder places they can be grown indoors as a houseplant.
Arecaceae *Rhapis excelsa* 	**General info:** A slow-growing, miniature fan palm with shiny, dark green leaves. The unbranched, rigid, bamboo-like stems are covered with a coarse, brown fibre. Rhapis grows wild in Mauritius and is named after the Greek word for needle, referring to its thin leaves and fine, elegant form. The latter also gave rise to its common name “Lady palm”, where it has been in cultivation as a pot plant for hundreds of years. **Uses:** The Rhapis excelsa gained popularity throughout the world during the 1800's when it became a highly prized and admired feature in the homes and gardens of the Aristocracy. It is regarded as a classic interior and a fine exterior landscape palm.

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3.0 NATURAL FIBRES

3.1 Resurgence of reinforcing materials

For over 3000 years ago, lignocellulosic fibres have been wielded as reinforcing agents, in combination with polymeric matrices. Their use dates to antiquity, such as the Great Wall of China whose construction started initially in 121 B.C. as earth works were strengthened by clay bricks made of local materials initially using red willow reeds, stone, sand, twigs and gravel during the Han dynasty (209 B.C.) and the Qin dynasty (221–206 B.C.). Likewise, the Egyptians have also been acquainted to use grass and straw as reinforcing fibres in mud and clay bricks for the building of walls since time immemorial (Bledzki *et al.*, 2002) Besides, bows adhesively made with wood or silk were used by the Mongolians in 1200 A.D. Cotton–polymer composites are cited to be the first fibre reinforced plastics used by the military for radar aircraft (Lubin, 2013; Piggott, 2002). Another example, include the East German Trabant car in 1950 whose frame was constructed from polyester reinforced with cotton fibres. References have been made to the use of linen and hemp textiles as reinforcements of ceramics as early as 6500 BC (Pickering, 2008) However, since the 1960s, the use of synthetic fibres has increased dramatically, causing the natural fibre industry to lose from its market share. While synthetic fibres, specifically glass, conquer today's market, consciousness of the scarcity of non-renewable resources and a demand for environmental sustainability have led to a resurgent interest in bio composites (Jawaid and Abdul Khalil, 2011)

3.2 Classification of natural and synthetic fibres

Fibres are a class of hair-like materials that are continuous filaments. They can be swivelled into strand, thread and rope. Natural fibres can be grouped into different types depending upon their origin such as plant, animal, mineral, organic and inorganic fibres (Bogoeva-Gaceva et al., 2007; Eichhorn et al., 2010; George et al., 2001). Figure 3.1 below shows the different classification of fibres.

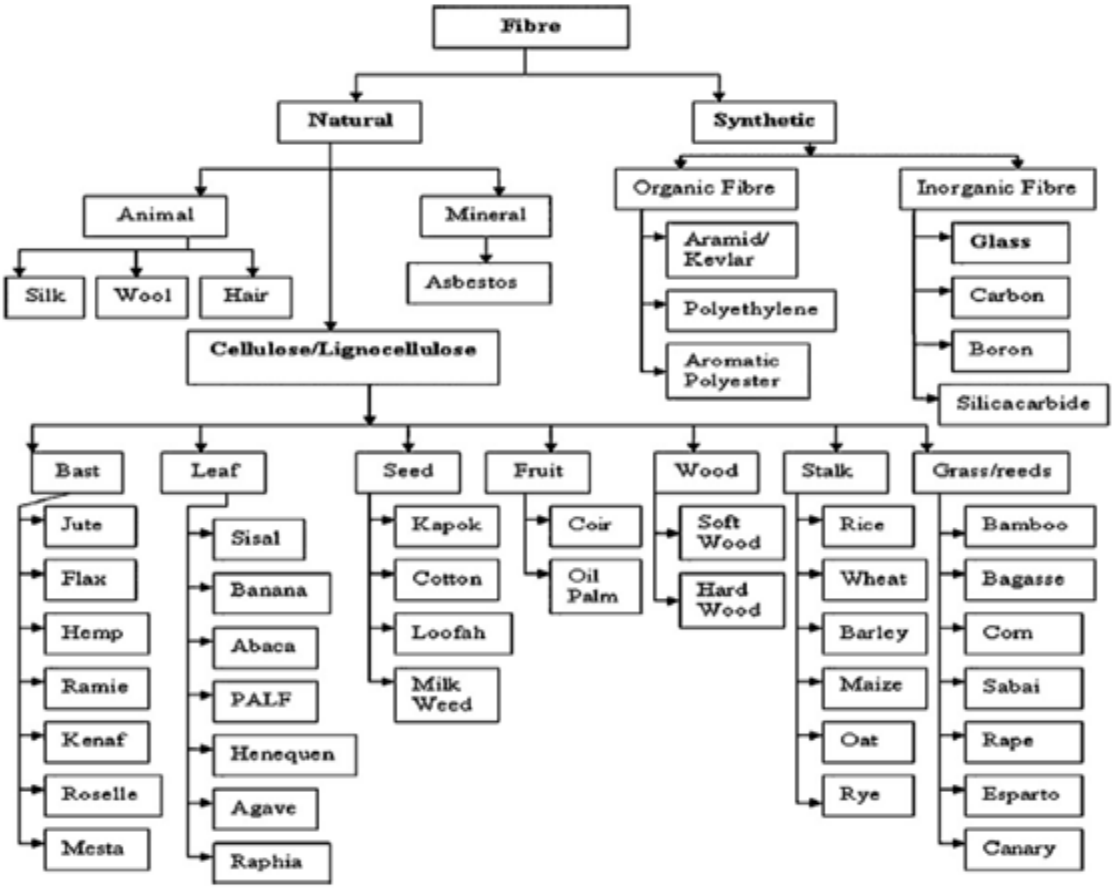

Figure 3.1: Classification of natural and synthetic fibres

Sources: (Jawaid and Abdul Khalil, 2011)

3.3 Plant fibre structure

A quantitative approach involves a conception of the influence of the chemical and physical structure of plant fibres on their mechanical properties. Each elementary plant fibre is a single cell with an elongated thick cell wall surrounding a central luminal cavity, portrayed in Figure 3.2. The cell wall is responsible for the structural integrity of the living plant whilst the luminal cavity facilitates the transportation of nutrients. Although having a high aspect ratio (length/diameter), the cross-sectional shape and dimensions of the cells are highly variable (Lewin and Pearce, 1998). Typically, elementary plant fibres are found in bundles, where the middle lamella (a pectin layer) cements the cell walls of two adjoining cells together. As depicted in Figure 3.2, the cell wall has a hierarchical structure, including a thin primary (P) cell wall, and a thick secondary (S) cell wall which exists in three sub layers (S1, S2, S3). The fibre structure develops in the primary cell wall and is deposited during its growth. Each layer in the secondary wall has a long chain of

helical cellulose microfibrils (Faruk *et al.*, 2012). In accordance with a specific type of fibre, cellulose microfibrils have their own cell geometry which is a factor responsible for the properties of the fibre (Reux, 2012). Typically, the primary cell wall and secondary cell wall accounts for less than 2% and 90% of the total cell wall thickness respectively (Placet et al., 2012). Notably, the S2 cell wall is the main sub-layer, accounting for more than 80% of the total cell wall thickness (Placet et al., 2012).

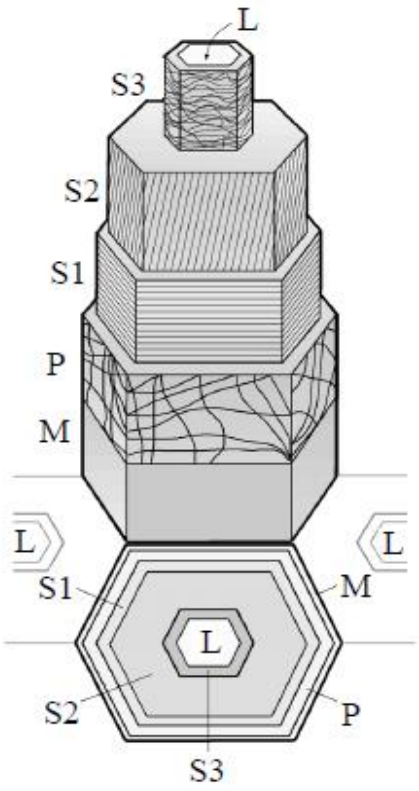

Figure 3.2 : The structure of an elementary fibre (i.e. a unit cell) in a technical fibre bundle, where the middle lamella (M) glues adjacent cells together, and each unit cell composes of primary (P) and secondary (S) cell walls and a central Lumen (L) Sources: (Placet et al., 2012).

3.4 Constituents of plant fibres

All natural lignocellulosic fibres primarily contain cellulose, hemicellulose and lignin as their indispensable constituents (Bogoeva-Gaceva et al., 2007; Eichhorn et al., 2010; George et al., 2001). The cellulose content increases steadily from primary to secondary layers, the hemicelluloses amount are similar in each layer whilst the lignin content decreases in this sequence. Hemicellulose molecules are hydrogen bonded with cellulose fibrils and they form cementing materials for the fibre structure. Lignin and pectin are coupled with the cellulose hemicellulose network and provides an adherent quality to hold the molecules together. This adherent quality is the reason for the strength and stiffness properties of the fibre. The figure 3.3 illustrates the position of the major fibre constituents in the fibre cell wall.

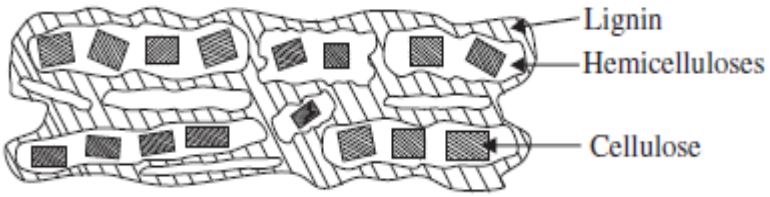

Figure 3.3 : Structural organisation of the three major constituents in the fibre cell wall

Source: (Kabir et al., 2012)

3.4.1 Cellulose

Cellulose (40–90%) is the major framework component of the fibre structure. It provides strength, stiffness and structural stability of the fibre. Owing to its high tensile strength, cellulose is the main structural component, which is responsible for the resistance of plants to mechanical stress. The chemical structure of cellulose as in figure 3.4 consists of three hydroxyl groups (OH). The hydroxylic groups (OH−) are responsible for the hydrophilic character in natural fibres. Two of them form hydrogen bonds within the cellulose macromolecules (intramolecular) whilst the rest of the group forms hydrogen bond with other cellulose molecules (intermolecular) (Sabo et al., 2013). The repeating unit is cellobiose, composed by two molecules of glucose. Each repeating glucose unit contains the three hydroxyl groups, which enables cellulose to form strong hydrogen bonds with its own chains to form fibrils, and with neighbouring chains to form microfibrils. The mechanical properties of cellulose are affected by its degree of polymerisation, which varies. Cellulose has a good resistance in hydrolysis, even if all chemical processes degrade it ito some extend (Akil et al., 2011).

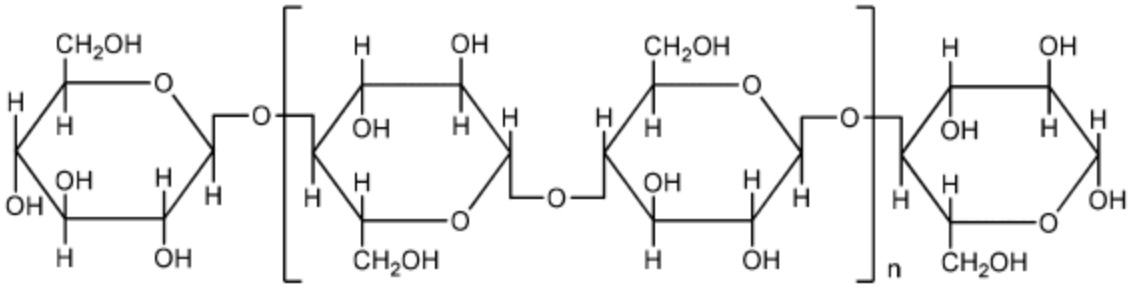

Figure 3.4 : Molecular Structure of cellulose (Akil et al., 2011)

3.4.2 Hemicellulose

Along with cellulose, one of the essential element of natural cellulosic fibres is hemicellulose (2–20%),which is the subsequent abundant family of naturally occurring polymers (Thakur et al., 2013a, 2013b). Hemicelluloses, having a branched structure, reside in plant cell walls and are build up of an association of 5- and 6-ring carbon ring sugars (Figure 3.5). Hemicelluloses consists a group of polysaccharides (excluding pectin), of much lower molecular weight than cellulose (Thakur et al., 2013a, 2013b). Hemicellulose can be degraded by both chemical and enzymatic hydrolysis. In contrast with cellulose, the degree of polymerization of hemicelluloses is 10–100 times lower. Hemicellulose has useful applications such as gelling agents, tablet binders viscosity modifiers (Akil et al., 2011; Bogoeva-Gaceva et al., 2007; Eichhorn et al., 2010; George et al., 2001).

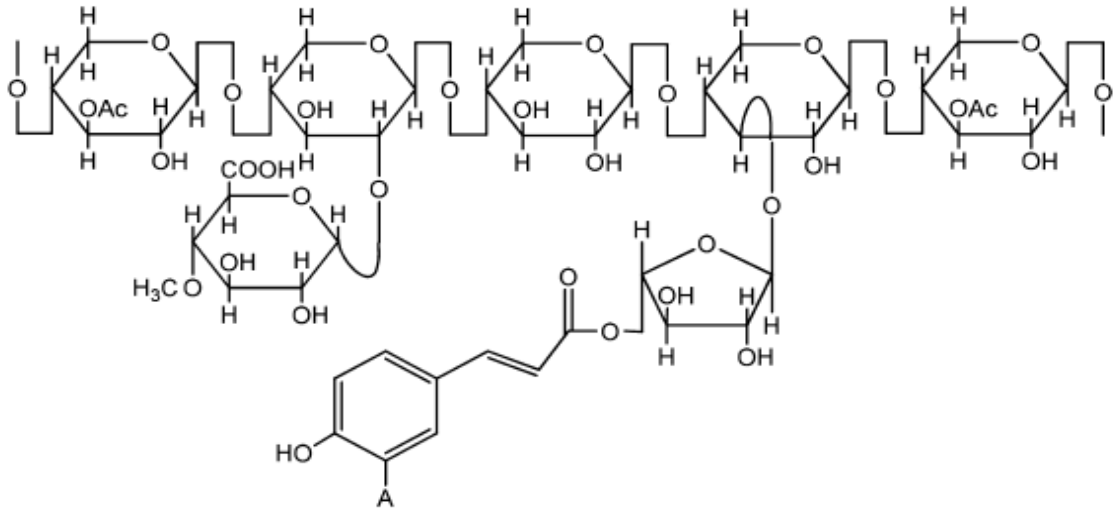

Figure 3.5 : Molecular structure of hemicellulose (Thakur et al., 2013a, 2013b)

3.4.3 Lignin

Among all the constituent of cell wall, lignin is the highly branched polymers (Thakur et al., 2013a, 2013b). The lignin content of plant fibres influences its structure, properties and morphology. Lignin acts as the cementing agent between cellulose and hemicellulose. Lignin structure is very complex consisting of phenyl propane units arranged in a three-dimensional structure (Akil et al., 2011; Bogoeva-Gaceva et al., 2007; Eichhorn et al., 2010; George et al., 2001). Different units in lignin are affiliated together through various types of carbon–carbon and ether bonds. As the plant grows, more lignin is produced, providing mechanical stability (Sperry, 2003). It is resilient to most microorganisms, since its aromatic groups are resilient to anaerobic procedures, while its aerobic degradation is slow. The figure 3.6 illustrates the structure of lignin.

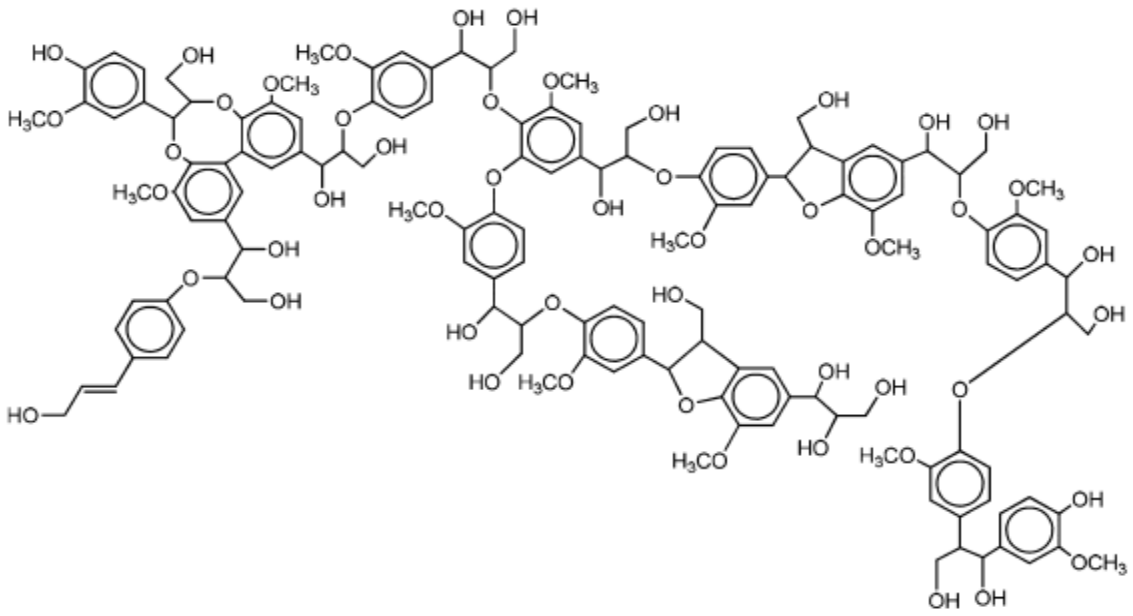

Figure 3.6 : Molecular structure of lignin (Thakur et al., 2013a, 2013b)

3.4.4 Miscellaneous constituents of plant fibre

Other components present in the fibre are: pectin, organic and inorganic. Pectin comprises of complex polysaccharides. Additionally, small amounts of organic (extractives) and inorganic (ash) components are present in the fibre structure. Organic extractives are responsible for colour, odour and decay resistance whilst inorganic constituents enhance the abrasive nature of the fibre.

3.5 Properties of plant fibre

The Secondary thick layer (S2) determines the mechanical properties of the fibre, is therefore of particular interest. Generally, fibres of the S2 layer with a higher cellulose content and a lower microfibrillar angle (the angle between the fibre axis and cellulose microfibrils) have better strength properties. Mechanical properties of natural fibre over synthetic fibre are presented in table 3.1. Although natural fibres have comparatively lower strength properties compared to the synthetic fibres, the specific modulus and elongation at break designates the possibility of these fibres to conquer synthetic fibres in engineering polymer composit

Table 3.1 : Comparative properties of natural fibres with conventional synthetic fibres (Indran and Raj, 2015)

Fibre	Chemical properties					Physical properties			
	Cellulose (%)	**Hemicellulose (%)**	**Lignin (%)**	**Wax (%)**	**Moisture (%)**	**Density (g/m3)**	**Elongation (%)**	**Tensile strength (MPa)**	**Young'smodulus (GPa)**
Cotton	82.7	5.7			1.0	1.6	7-8	400	5-12
Coir	32-43	0.15-0.25	40-45			1.2	30	593	4-6
Jute	64.4	12	11.8	0.7	1.1	1.3	1.5-1.8	393-773	
Flax	64.1	16.7	2	3.3	3.9	1.5	2.7-3.2	500-1500	
Hemp	68	15	10	0.8	6.2	1.47	2-4	690	70
Kenaf	45-57	8-13	21.5	0.8	6-12	1.31	1.6	427-519	23.1-27.1
Ramie	68.6-85	13-16.7	0.5-0.7	0.3	7.5-17	1.5	2-3	220-938	44-128
Sisal	60-78	10-14.2	14	2	10-22	1.5	2-25	511-635	9-22
Pineapple leaf	70-83		5-12.7		11.8	1.4	0.8-1	413-1627	34-82
Banana	56-63	20-25	7-9	3		1.35	50-250	529-759	17.85
Palmyrah	40-52	42-43				1.09	7-15	180-215	7-60
Bamboo	73.83	12.49	10.15	3.16		0.91	1.4	503	35.91
Curaua	70.7	9.9	7.5-11			1.4	3.7-4.3	500-1150	11
Bagasse	55.2	16.8	25.3			1.2			
Alfa	45.4	38.5	14.9	2		0.89	5.8	350	22
E-glass						2.5	0.5	2000-3500	70

Aramid						1.4	3.3-3.7	3000-3150	63
Carbon						1.4	1.4-1.8	4000	230-240

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CHAPTER 4: LITERATURE REVIEW

4.1 Fibre extraction

Parallel to the advantages natural fibres in composites reinforcements, there are also shortcomings with respect to their performance, their behaviour in polymeric matrix systems and their processing. Plant fibres, even of the same species, do not have a constant pattern of physical properties throughout a given year (Baley *et al.*, 2006). The variability in properties, which dictate the fibre quality, are influenced by plant growth conditions and fibre extraction and preparation process. The end properties of natural fibres depends on many variables such the same cultivation period of the crops, fibre location in plant, age/maturity of plant, geographic location, soil characteristics, climate, maturity, harvesting period, decortications, retting degree, disintegration, type of extraction method (steam explosion treatment, mechanical, manual). (Bledzki and Gassan, 1999; Le Duigou et al., 2015; Morvan et al., 2003). To ensure that the quality of their products is consistent (the variability in properties is within acceptable limits) and independent of plant growth conditions, it is recommended to blend several batches that are themselves of different crops/harvests/years.

Regarding optimising fibre extraction and processing, scientific studies emphasises that an increase in number of mechanical processing steps heighten the defect count: in the form of kink bands, reduction in extent of polymerisation of the cellulose chains and all that results in a consequent reduction in fibre mechanical properties (Hänninen *et al.*, 2012). Notably, the rise in twist level imparted to the fibre at each stage have various detrimental effects on composite properties, including obstructed resin impregnation, dwindled wettability, augment in void formation and a significant quantifiable drop in tensile properties (Chen *et al*., 2011; Goutianos *et al.*, 2003). It is therefore important, to minimise the numbers of steps associated with fibre extraction.

Table 4.1 summarises the different types of fibre-extraction techniques used in the different research works undertaken.

Table 4.1 Different modes of fibre extraction techniques.

Source	Extraction Technique
El-Abbassi,F.E., *et al.,* 2015. Effect of alkali treatment on Alfa fibre as reinforcement for polypropylene based eco-composites: Mechanical behaviour and water ageing, *Composite Structures*,133,451-457	Dried stems were manually cut to bundles of 4–6 cm length. A special mill was used to mechanically crush these bundles because the Alfa stems are very hard to break. The fibres are sieved in order to keep only those with a length of 8 ± 2 mm and a diameter of 400 ± 100 microns.
Wang,H *et al.,* 2015. Properties of *Xanthoceras sorbifolia* Husk Fibers With Chemical Treatment for Applications in Polymer Composites. *Journal of Materials science and technology*, 31(2), 164-170	The fibers were obtained by smashing the X. *sorbifolia* husks. The smashed fibers were sieved to the size of 16-32 meshes (about 0.6-1.18 mm), then the raw fibers were obtained.
Arrakhiz, F.Z et al., 2013.Mechanical and thermal properties of natural fibers reinforced polymer composites: Doum/low density polyethylene, *Materials and Design*, 43, 200-205	Raw and treated fibers were grinded in a precision grinder equipped with a 1 mm sieve size the fibers were then sieved in a 250 um sieve size to remove the presence of any waxes.
Haameem *et al.,* 2016. Mechanical properties of Napier grass fibre/polyester composites, *Composite Structures*, 136,1-10	The Napier grass fibres were extracted by using a conventional water retting process to allow the bast fibres to detach from the woody centre. The stems were crushed with a mallet and immersed in a water tank filled with tap water for approximately 1 month.
Ramanaiah, K., Ratna Prasad, A.V., Chandra Reddy, K.H., 2011. Mechanical properties and thermal conductivity of Typha angustifolia natural fiber–reinforced polyester composites. International Journal of Polymer Analysis and Characterization 16, 496–503.	The extraction of fibers involves the retting process followed by decortication. The stems of Typha angustifolia were cut at their base and immersed in a water tank for three weeks. Then they were removed; the fibers were stripped from their stalks by hand, washed, and dried in the sun. After drying, any unwanted matter that might still be adhering to them was removed by washing it with NaOH. The extracted fibers were used for composite manufacturing.

Milan, S., Christopher, T., Jappes, J.W., Siva, I., others, 2015. Investigation on Mechanical Properties and Chemical Treatment of Sea Grass Fiber Reinforced Polymer Composites. Journal of Chemical and Pharmaceutical Sciences www. jchps. com ISSN 974, 2115.	The sea grass were extracted using microbial degradation technique. After removing the dirt and other foreign materials, the roots were immersed in water for two weeks to allow microbial degradation. They were then taken out and thoroughly washed with fresh water and dried in the sun light for a week to remove the moisture content. The soil particles and outer layer of the root were then removed by traditional combing process with metal teeth brush.
Alsaeed, T., Yousif, B.F., Ku, H., 2013. The potential of using date palm fibres as reinforcement for polymeric composites. Materials & Design 43, 177–184. doi:10.1016/j.matdes.2012.06.061	All fibres from the date palm were obtained from the outer layer of the tree stem. Only fibres from the same bunch were used to ensure the consistency. The fibres were carefully extracted from the stem manually.
Zakikhani, P., Zahari, R., Sultan, M.T.H., Majid, D.L., 2014. Extraction and preparation of bamboo fibre-reinforced composites. Materials & Design 63, 820–828. doi:10.1016/j.matdes.2014.06.058	The bamboo bark was removed and the cylindrical part of the culm was peeled to obtain strips. The strips of bundles were kept for three days in water. Then, to separate the fibres, the wetted strips were beaten, scraped with a sharp edged knife and combed. In this method, the process of scraping the fibre surface had a strong effect on the quality of the fibres, and the fibres broke less along the length of fibres.
Zakikhani, P., Zahari, R., Sultan, M.T.H., Majid, D.L., 2014. Extraction and preparation of bamboo fibre-reinforced composites. Materials & Design 63, 820–828. doi:10.1016/j.matdes.2014.06.058	Bamboo fibres were extracted by first cutting the raw bamboo into small pieces by a roller crusher. Then, the small pieces were extracted into coarse fibre by a pin-roller. Before the coarse fibres were put in a dehydrator, they were boiled at 90 ^{0}C for 10 h to remove their fat and later dried in the rotary dryer. The problem with this process is that it yields short fibres, which become powdered after mechanical over-processing

Zakikhani, P., Zahari, R., Sultan, M.T.H., Majid, D.L., 2014. Extraction and preparation of bamboo fibre-reinforced composites. Materials & Design 63, 820–828. doi:10.1016/j.matdes.2014.06.058	The compression moulding technique (CMT) and roller mill technique (RMT) are usually used after alkali and chemical treatment to extract fibres. In one research study, the CMT technique was used to pressurise a bed of alkaline treated bamboo strips between two flat platens under a load of 10 tons. In this method, the starting bed thickness and compression time are important factors to separate high quality fibres. In the RMT technique, the treated bamboo strips were forced between two rollers, with one fixed and the other rotated. In both methods the bamboo strips were flattened, and the combined alkaline and mechanical process enabled the easy separation of strips into individual fibres. In addition, the size of the compression mould and the diameter of the rollers are two factors that limit the ability to extract fibres with smaller strip sizes in both techniques.
De Rosa, I.M., Kenny, J.M., Puglia, D., Santulli, C., Sarasini, F., 2010. Morphological, thermal and mechanical characterization of okra (Abelmoschus esculentus) fibres as potential reinforcement in polymer composites. Composites Science and Technology 70, 116–122. doi:10.1016/j.compscitech.2009.09.013	After collecting the fresh plant, about two months old and around 2 m high, the central part of the stems was removed and kept under water to allow microbial degradation. Within 15–20 days the stems degraded appreciably to allow fibre extraction. The fibres were isolated from the degraded stems by being washed three times, using deionised water, then tied with ropes, dried in open air and kept in moisture-proof container.
Ridzuan, M.J.M., Abdul Majid, M.S., Afendi, M., Aqmariah Kanafiah, S.N., Zahri, J.M., Gibson, A.G., 2016. Characterisation of natural cellulosic fibre from Pennisetum purpureum stem as potential reinforcement of polymer composites. Materials & Design 89, 839–847. doi:10.1016/j.matdes.2015.10.052	Purpureum plants were harvested and following the water retting process, the fibres were manually extracted from the stem internodes. To separate the fibre strands, the stems were initially cleaned and subsequently crushed into small parts with a mallet. Subsequently, the short plant stems were immersed under running tap water for a few weeks to facilitate the separation process. Finally, the fibres were cleaned with distilled water and subsequently dried under the sun to remove the moisture content.

4.1.1 Conclusions (Fibre Extraction)

It can be seen that some plant that have been minimally processed fibres followed by retting splitting and hammering produced high quality fibres that would in turn yield composites of higher strength. Water retting is recognised to be least energy intensive, followed by dew retting, bio-retting, manual scraping and mechanically extracted fibres. In terms of environmental impact in the manufacture of fibre-reinforcements composites, minimum energy requirement is suitable. It is expected that mechanical extraction process would generate significantly higher fibre-yields as the degree of decortication increases as the material passes along the mechanical extractor. The above studies show that mechanical extraction is better for rigid bast (*Ravenala Madagascariensis* Leaf sheath) and thick leaves (*Draceana concinna*) whilst manual scraping is better for extracting fibres from thin leaves (such as *Acanthophoenix rubra*). Retting process followed by hammering could be adopted for bast, sedge and the grass stem (*Raphis excelsa*, *Cyperus latifolius*, *Cyperus distans*, Banyan), i.e. for finer plants.

4.2 Fibre surface modification

Although natural fibres offer numerous benefits over their synthetic counterparts, certain challenges obviously prevail. The major drawback of natural fibre composites originate from the hydrophilic nature of the highly polar plant fibre and hydrophobic nature of the non-polar polymer matrices (Abdelmouleh *et al*., 2007). The inherent incompatibility between these two phases results weakening bonding at the interface. Additionally, pectin and waxy substances cover the reactive functional groups of the fibre and act as a barrier to interlock with the matrix. Other weaknesses associated with these fibres include being sensitive to water/moisture absorption, poor wettability, inadequate adhesion, non-uniform dispersion of fibres within the matrix, and their poor dimensional and thermal stability, leading to comparatively low dispersion of force and ultimate severe decline of overall mechanical properties (Akil et al., 2011; Bogoeva-Gaceva et al., 2007; Eichhorn et al., 2010; George et al., 2001)

Research activists have explored various avenues to enhance the effectiveness of fibre/matrix interfacial properties (Faruk et al., 2012). The two fundamental routes are fibre surface physical/chemical modification. The purpose of physical modification techniques, such as (plasma, steam explosion, clandering, UV gamma) is to roughen the fibre surface topography and to eradicate surface impurities (such as oils, waxes, pectin), enabling improved mechanical adhesion between the fibre and the matrix. In chemical modification techniques, a third material is introduced, as a compatibiliser or reinforcing agent, between the fibre and the matrix. (John and Anandjiwala, 2008; Kabir et al., 2012; Kalia et al., 2009)

As compared to physical treatment, chemical modification techniques have been postulated to boost the performance of biocomposite materials by reducing water sorption, increase resistance against fungal decay and by exposing more reactive groups on the fibre surface thus facilitates efficient coupling with the matrix. Different chemical surface modification techniques have been used to modify the surface of natural cellulosic fibres, including alkali, silane, acetylation, benzoylation, acrylation, maleated coupling agents, permanganate, peroxide, isocyanate, stearic acid, sodium chloride, triazine, fatty acid derivative, and fungal. (Kabir et al., 2012)

4.2.1 Alkali treatment

Among all the chemical modification treatment aforementioned, alkali treatment is acknowledged as being the most popular, commercially efficient, cost effective, and eco-friendly method for improving interfacial properties. The aim of the alkali treatment is to mitigate the hemicelluloses, split the fibres in the fibrils, and produce a closely packed cellulose chain owing to the release of the internal strain, which consequently improves the mechanical properties of the fibre (Kabir et al., 2012; Vallo et al., 2016). The alkali treatment also breaks the hydrogen bonds and increases the number of free hydroxyl groups of the fibre, thus increasing the fibre reactivity. Following the alkali treatment, the fibrillation of the fibres also increases the effective surface area available for wetting by the resin, and enhances the bonding between the fibre-matrix interfaces within the polymer composites (Kabir et al., 2012; Vallo et al., 2016). The chemical reaction between fibre cell and NaOH is shown in Figure 4.1. Figure 4.2 illustrates the schematic view of the fibre before and after the alkali treatment. Table 4.2 elucidates some of the previous research studies on alkali-fibre treatment.

$$\text{Fibre-cell-OH} + \text{NaOH} \longrightarrow \text{Fibre cell-O}^{-}\text{Na}^{+} + \text{H}_2\text{O} + \text{impurities}$$

Figure 4.1 (Source :*Kabir et al., 2012)*

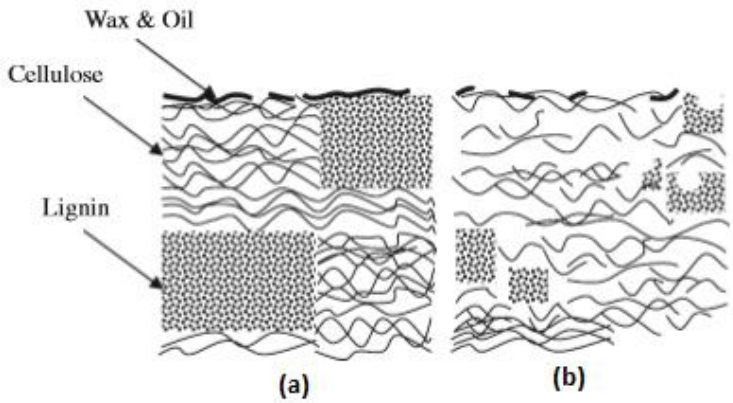


Figure 4.2 (a) Structure of untreated fibre (b) treated fibre Source: (Mwaikambo and Ansell, 2002)

Table 4.2 Alkali treated of natural fibre for the manufacture of reinforced polymer composites.

Article references	Applied treatment methods	Mechanical properties of treated fibres/composite
El-Abbassi,F.E., *et al.,* 2015. Effect of alkali treatment on Alfa fibre as reinforcement for polypropylene based eco-composites: Mechanical behaviour and water ageing, *Composite Structures*, 133,451-457	10% NaOH in room temperature for 24 hr	The alkali treatment enhances the mechanical properties of the fibres
Le, T.M and Pickering, K.L., 2015.The potential of harakeke fibre as reinforcement in polymer matrix composites including modelling of long harakeke fibre composite strength, *Composites:Part A,76*, 44-53	5% of NaOH at 170 °C for 120 min	For the 5 % alkali conc,The tensile strength of harakeke fibre composites (223 ± 14 MPa) is greater than that of sisal fibre composites (211 ± 12 MPa) at the same conc.

Herrera-Franco, P.G and Valadez-Gonzalez, A., 2005. A study of the mechanical properties of short natural-fibre reinforced composites, *Composites: Part B*, 36, 597-608	Six different fibre surface treatments: 1. Without treatment 2. fibre treated with NaOH 3. fibre pre-impregnated with dissolved HDPE 4. fibre treated with NaOH aqueous solution and then pre-impregnated with dissolved HDPE 5. fibre treated with a silane coupling agent 6. fibre treated with NaOH aqueous solution and then with silane	The tensile modulus of HDPE/henequen-fibre (80:20 v/v) composite, is higher for the fibre surface treatment with NaOH only (910 MPa).
Ibrahim, M.M *et al*., 2010. Banana fibres and microfibrils as lignocellulosic reinforcements in polymer composites, *Carbohydrate Polymers*,81, 811-819	10 % NaOH and liquor to fibre ratio was 10:1 at 170 ^{0}C and for 2 hr	Steam explosion (Physical treatment) was compared with alkaline treatment (chemical treatment), which demonstrates better mechanical properties.

Wang,H *et al.,* 2015. Properties of *Xanthoceras sorbifolia* Husk fibres With Chemical Treatment for Applications in Polymer Composites. *Journal of Materials science and technology*, 31(2), 164-170	5 % NaOH solution/fibre ratio of 20:1, followed by KH550, KH570 and JN-9A titanate were chosen as the coupling agent. Ambient temperature, 1 hour.	Tensile strength of the alkalized, KH550 treated, and KH570 treated fibres is increased by 24%, 17.2%, and 6.4% respectively However, the tensile strength of the JN-9A treated fibre is reduced by 25.6%.
Rwawiire *et al.,* 2015. Development of a biocomposite based on green epoxy polymer and natural cellulose fabric (bark cloth) for automotive instrument panel applications. *Composites: Part B,* 81, 149-157	Bark cloth fabrics were treated in 5% alkali solution at room temperature for one hour.	Tensile properties of Bark cloth biocomposites is 33 MPa, higher compared to the synthetic counterparts (30 MPa)
Alawar *et al.,* 2009. Characterization of treated date palm tree fibre as composite reinforcement, *Composites: Part B,* 40, 601-606	fibres were soaked in 5%, 2.5%, 1.5%, 1%, and 0.5% NaOH at 100^0C for 1 h.	1 % NaOH attain the highest tensile strength of fibres followed by 0.5 %, 2.5%, 5% and untreated.
Alsaeed, 2013. The potential of using date palm fibres as reinforcement for polymeric composites, *Materials and Design*,177-184	Three different NaOH concentrations were prepared as 3, 6, and 9 wt. %. The selected fibres were immersed in NaOH aqueous solution for 24 h at room temperature.	The highest tensile strength (400 MPa) is exhibited when the fibre was treated with low NaOH concentration (3%),

Saba *et al.*, 2015. Mechanical properties of kenaf fibre reinforced polymer composite: A review, *Construction and Building Materials* 76, 87–96	The kenaf with 15/15 v/v kenaf–glass fibres on treatment with 6% sodium hydroxide using mercerization method for 3 h Temp not mentioned	This treatment yields better mechanical strength to the composite.
Elkhaoulani *et al.*, 2013.Mechanical and thermal properties of polymer composite based on natural fibres: Moroccan hemp fibres/polypropylene, *Materials and Design*, 49, 203-208	6.5% NaOH in room temperature for 48 hr followed by coupling Temp not mentioned	
Haameem *et al.*, 2016. Mechanical properties of Napier grass fibre/polyester composites, *Composite Structures*, 136,1-10	The treatments were conducted using NaOH concentrations of 5, 10, 15, and 20%. The ratio of Napier grass fibre to the soaking solution was maintained at 1:10.	The 10% alkaline-treated fibres yielded the highest tensile strength
Kommula *et al.*, 2013Study on Impact strength of Untreated and Alkali treated Napier grass fibre strands reinforced Epoxy composites, International Journal of Research in Engineering and Technology, 2(3),101-106	The processed Napier grass fibre strands were treated with 5, 10, and 15% (w/v) sodium hydroxide solution for 2 h at room temperature, maintaining a liquor ratio of 20:1 to remove the surface impurities from the fibre strands.	The 10% alkaline-treated fibres yielded the highest tensile strength

Ridzuan *et al.*, 2016. Characterisation of natural cellulosic fibre from Pennisetum purpureum stem as potential reinforcement of polymer composites, *Materials and Design*, 89, 839-847	Alkali solutions of 5, 7, 10, 12, and 15% were applied for 24 h at room temperature. A liquor ratio of 40:1 was used to remove the hemicelluloses and surface impurities of the fibre.	The 5% alkali-treated fibre achieved the maximum ultimate tensile stress. The 10, 12, and 15% alkali treatments reduced the ultimate tensile stress of the fibres; this was confirmed by SEM observations, which demonstrated the damaged textures and the twisted fibres.
Ishak *et al.*, 2013 Sugar palm (Arenga pinnata): Its fibres, polymers and composites, *Carbohydrates polymers*,91, 699– 710	1 % and 2 % NaOH at 8 hours. Temp not mentioned.	Increased in tensile strength was noted at 2 % alkali treatment.
Essabir *et al.*, 2016. Structural, mechanical and thermal properties of bio-based hybrid composites from waste coir residues: fibres and shell particles, *Mechanics of Materials*, 93,134-144	6.5 % NaOH in room temperature for 48 hours followed by coupling.	The filler/polymer compatibility was improved.
Li X, Tabil LG, Panigrahi S. Chemical treatment of natural fibre for use in natural fibre-reinforced composites: a review. Polym Environ 2007;15(1):25–33.	Alkaline treatment.	30 % increase in tensile properties.

Li X, Tabil LG, Panigrahi S. Chemical treatment of natural fibre for use in natural fibre-reinforced composites: a review. Polym Environ 2007;15(1):25–33.	0.5 %, 1 %, 2 %, 4%, 10 % NaOH at room temperature Duration not mentioned.	4 % alkali treatment resulted greatest tensile strength
Mwaikambo LY, Tucker N, Clark AJ. Mechanical properties of hemp fibre reinforced euphorbia composites. Macromol Mater Eng 2007;292(9): 993–1000.	0.16 % NaOH for 48 hr Temp not mention	30 % increased in tensile strength
Ray D, Sarkar BK, Rana AK, Bose NR. Effect of alkali treated jute fibres on composite properties. Bull Mater Sci 2001;24(2):129–35.	5 % NaOH for 4,6, 8 hr Temp not mention	4 hr alkali treated demonstrates better mechanical properties
Dhanalakshmi *et al.*, 2015. Effect of Chemical Treatments on Tensile Strength of Areca fibre Reinforced Natural Rubber Composites, *Journal of Applied Chemistry*, 8(5),43-53.	Areca fibres were soaked in a stainless steel vessel containing 6% NaOH at room temperature for 1h. The alkali treated fibres were immersed in distilled water for 24 h to remove the residual NaOH.	The tensile strength values of alkali treated areca fibre reinforced natural rubber composites with 40%, 50%, 60% and 70% fibre loadings are 92.26 MPa, 116.82 MPa, 130.82 MPa and 109.26 MPa respectively.
Cyras VP, Vallo C, Kenny JM, Vazquez A. Effect of chemical treatment on the mechanical properties of starch-based blends reinforced with sisal fibre. J Compos Mater 2004;38(16):1387–99.	10 % NaOH for 1,3,24,48 hours. Temp not mention	Increased in elastic modulus with prolong reaction time

Ouajai S, Shanks RA. Composition, structure and thermal degradation of hemp cellulose after chemical treatment. Polym Degrad Stab 2005;89(2):327–35.	8 % NaOH Duration, temp not mentioned	Thermal stability raised by 4 %.
Prasad SV, Pavithran C, Rohatgi PK. Alkali treatment of coir fibres for coir–polyester composites. J Mater Sci 1983;18(5):1443–54.	5 % NaOH for 72 hours, 28℃	Mechanical properties increased by 40 % compared to untreated composites.
de Oliveira P., Marques M., 2015. Chemical treatment of Natural malva fibres and preparation of green composites with poly(3-hydroxybutyrate), *Chemistry and Chemical technology*, 9(2),211-222	5 %, 7 %, 10 %, 15%, 20 % NaOH at room temperature for 48 hours.	The results showed that the crystallinity index and thermal resistance significantly increased when the alkaline treatment was conducted with 7 % NaOH solution. Higher concentrations of NaOH, such as 10, 15 and 20 %, did not considerably increase the fibre crystallinity index.
Pandurangadu, V and Palanikuamr.K., 2011. Mechanical properties of green coconut fibre reinforced HDPE polymer composite, *International Journal of Engineering Science and Technology*, 11(3), 7942-7952	The green coconut fibres(100g) were pre-treated with 1L alkaline solution which is prepared in different concentrations as 2, 3 and 4% of NaOH for an hour under constant stirring and for 24 hours at room temperature.	Green coconut fibre treated with NaoH 2% concentration has the highest tensile strength.

4.2.2 Conclusions (Alkali treatments)

In light from table research works on alkali treatment of natural fibres, it could be deduced that a significant low concentration of alkali (1- 5%) can alleviate the waxy layers and improve the chemical and mechanical properties of the fibres whilst on the other hand an increase in alkali concentration (>10 %) cause weakening of the resulting fibre properties.

It is observed that the the range of NaOH used is 1-10 % depending on the fiber species. Therefore, for the current study, an average of 5% NaOH can be regarded as the recommended acceptable concentration for improving fibre strength and surface characteristics, for yielding sufficient enhancement to the interfacial adhesion of the fibre /matrix. This alkali treatment can be optimised at room temperature and reaction time of 24 hr, thereby reducing any additional heating effect and heating costs, and also for providing adequate time for fibres to immerse completely in the liquor solution.

4.3 The polymer matrix material

The polymer matrix is a binder material that is used to hold fibres in position and transfer external loads to internal reinforcements. Generally, polymer can be classified into two classes, thermoplastics and thermosettings (Malkapuram et al., 2008). In natural fibre reinforced polymer composites the widely used matrix for composites applications are depicted in Figure 4.3. These matrices have distinct chemical structures and undergo different reactivities with the surface molecules of fibres in composites. Table 4.3 represents the comparison of thermoset and thermoplastic matrices, along with reported studies on these two types of polymers in Tables 4.4 and 4.5.

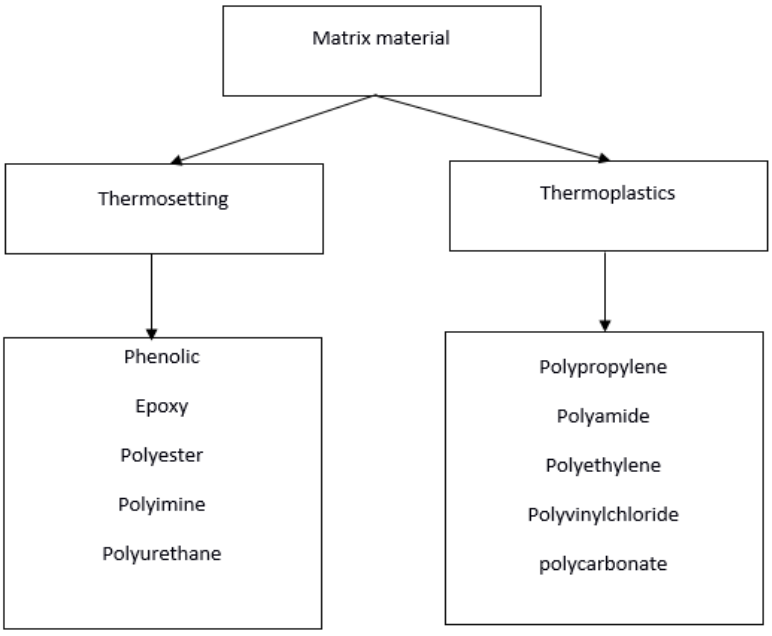


Figure 4.3: Different polymer matrix material

Table 4.3: Comparison of thermoset and thermoplastic matrix

Advantages	Disadvantages
Thermoset	
Low resin viscosity	Brittle
Good fibre wetting	Non-recyclable via standard techniques
Excellent thermal stability once polymerised	
Chemically resistant	
Thermoplastic	
Recyclable	Poor melt flow
Easy to repair by welding and solvent bonding	Need to be heated above the melting point for processing purposes
Post formable	
Tough	

Table 4.4: Reported studies on natural fibre hybrid thermoplastic polymer composites (Jawaid and Abdul Khalil, 2011)

Hybrid fibre	Matrix polymer
Oil palm EFB/ glass	Polypropylene (PP)
Jute/glass	Polypropylene (PP)
Sisal/glass	Polypropylene (PP)
Banana/glass	Polyethylene (PE)
Bamboo/glass	Polystyrene Polypropylene (PP)
Hemp/glass	Polypropylene (PP)
Flax/glass	Polyethylene (PE)
Cotton/flax	Polyethylene (PE)
Wood floor/glass	Poly vinyl chloride (PVC)
Cotton/waste silk	Polycarbonate (PC)
Coir/sisal	Natural rubber
Oil palm/ EFB glass	Natural rubber
Kenaf/glass	Natural rubber
Sisal/oil palm	Natural rubber

Table 4.5: Reported studies on natural fibre hybrid thermoset polymer composites Source : (Jawaid and Abdul Khalil, 2011)

Hybrid fibre	Matrix polymer
Oil palm EFB/ jute	Epoxy resin
Oil palm EFB/ glass	Epoxy resin Polyester Phenol formaldehyde Vinyl ester
Jute/glass	Polyester (isothalic) Unsaturated polyester Polyester Epoxy phenolic resin Epoxy resin
Jute/cotton	Novolac phenolic Polyester
Jute/bagasse	Epoxy
Cotton/ramie	Polyester
Kapok/glass	Unsaturated polyester
Sisal/kapok	Unsaturated polyester
Sisal/glass	Unsaturated polyester Polyester Epoxy resin Phenolic
Sisal/cotton	Polyester
Sisal/silk	Unsaturated polyester
Sisal/roselle	Unsaturated polyester Polyester

Table 4.5: Reported studies on natural fibre hybrid thermoset polymer composites (Jawaid and Abdul Khalil, 2011) *Continued*

Banana/kenaf	Unsaturated polyester
Kenaf/glass	Epoxy resin
Sisal/banana	Polyester
Banana/glass	Phenol formaldehyde Unsaturated polyester
Palmyra/glass	Rooflite resin
Bamboo/glass	Unsaturated polyester Polyester Epoxy resin Vinyl ester
Biofibre/glass	Polyester
Natural fibre/glass	Epoxy vinyl ester
Coir/glass	Polyester Phenolic resin
Coir/silk	Unsaturated polyester
Cellulose/glass	Expoy resin
Ridge gourd/glass	Phenolic resin
Jute/biomass	Bisphenol-C-formaldehyde

4.3.1 Selection of polymer matrix material

Several matrix materials originating from renewable resources are suitable components for application in a green composite either being biodegradable or non-biodegradable. In the automotive industry, there is a general tendency of the increased use of thermoplastic matrices. This is primarily because the latter are faster to process, are less expensive (for high volume production), are fabricated by a cleaner process (dry systems with no toxic by-products), and are easier to recycle. On the other hand, traditional thermoplastics have processing limitations as high processing temperatures (up to 200 °C) and high melt viscosity(100-10000 Pas) (Summerscales et al., 2010) Notably, thermoplastic-based matrices that are recycled by remolding into new parts exhibit severely deteriorated mechanical properties due to repeated thermal exposure as in the case of injection molding processing (T et al., 1999).

Nonetheless, ignorant from the level of recyclability issue, thermoset matrices present enhanced mechanical performance and superior adhesion with reinforcing fibres, and are therefore apt for structural applications in order to satisfy stiffness, resistance, reliability and life-time requirements. Firstly, thermoset matrices have better mechanical properties than thermoplastics, due to the formation of a large cross-linked rigid three dimensional molecular structure upon curing. It is noteworthy that thermoset-based polymers consistently show better tensile properties (absolute and even specific) in contrast to the thermoplastic-based counterparts. Besides, the low processing temperatures (typically below 100 °C) and viscosity (0.1-10 Pas) of thermoset matrices implies that plant fibre mechanical properties are not degraded due to high temperature exposure during composites manufacture, and resin impregnation and preform wettability are easier leading to lower void content and better interfacial properties. The low viscosity of thermoset resins also raises the possibility of using liquid composite moulding techniques, such as vacuum infusion and resin transfer moulding (RTM), which are standard manufacturing procedures in the performance-demanding automotive industries. Finally, thermosets have better shear properties than thermoplastics, and they form a better interface with typically polar plant fibres than thermoplastics (which tend to be non-polar).

It should be noted that in terms of end-of-life disposal, the use of thermosetting matrices, rather than thermoplastic matrices, does not necessarily lower the eco performance of the resulting composite produced. In the case of a hypothetical 100% bio-based composite, even if the material could not be recycled directly there are means to be opted out through incineration for energy recovery. All fibre reinforced composites can be incinerated for energy recovery or re-used as fillers. In the case of incineration, there are no emissions of toxic gases and by decomposition there are no gases at all (Malkapuram et al., 2008). The novel bio-based thermosets (plant oil-based resins) resembling the synthetic thermosets (phenolics, polyesters, epoxies) are indeed difficult to recycle and reuse but can be later decomposed in most cases.

4.3.2 Conclusions (Selection of polymer matrix material)

Based on the above highlighted issues, thermoset matrices are selected as the matrix materials for this study. Eventually, it can be deduced that thermosets are more suitable than thermoplastics, due to their ease of handling, their capacity in high-performance applications such as in the automotive industries, lower viscosity and processing temperatures, chemically resistant, excellent thermal stability and exhibit better compatibility with plant fibres

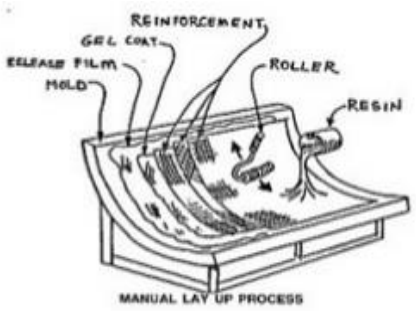
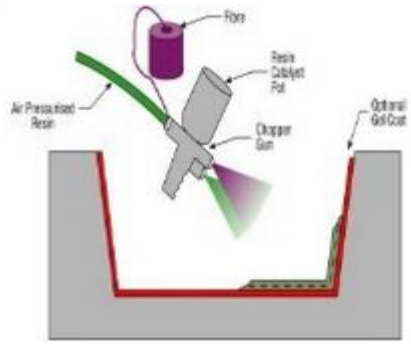
4.4 The composite material

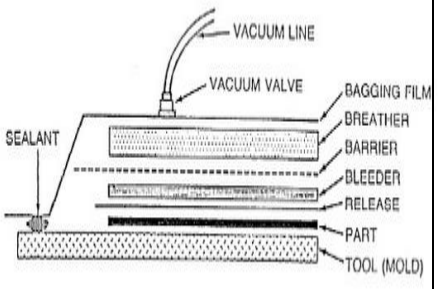
A composite material, like a fibre reinforced polymer (FRP) consists of a polymer matrix imbedded with high strength fibres (Malkapuram et al., 2008). Generally, a composite is a material mixture created by a synthetic/ natural assembly of two or more physically and chemically distinct components, having a discrete interface separating them. The first component is a selected filler or reinforcing agent (discontinuous phase) whilst the other component is a compatible matrix binder (continuous phase). One or more discontinuous phases therefore, are embedded in a continuous phase to form a composite (Bledzki and Gassan, 1999). These two components are combined in order to achieve specific characteristics and properties. For composite reinforcements, different fibre orientation such as long unidirectional, bidirectional, multidirectional, cross ply, weave or discontinuous, such as short-randomly oriented fibre were previously explored (Kabir et al., 2012) Each reinforcement architecture is designed for a particular domain of application. Fibres with a high aspect ratio and randomly distributed in plane can be used to manufacture structural parts with a quasi-isotropic behaviour. This type of reinforcement is useful for panel manufacturing, whereas unidirectional fibres are sought for beams. To ensure full utilisation of fibre properties in a composite, a continuous and aligned reinforcement product is required.

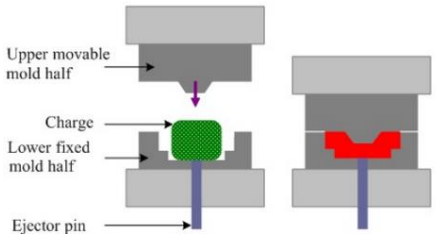
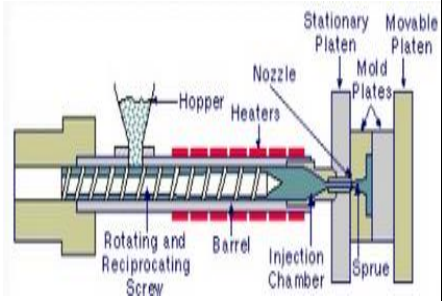
4.5 Composite manufacturing techniques

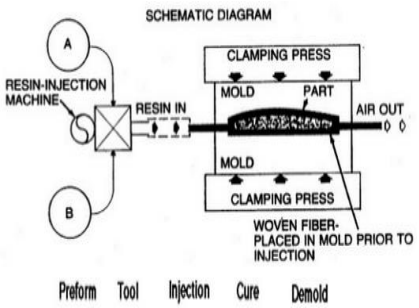
Suitable processing techniques and parameters must be wisely selected in order to yield the optimum composite products. Among the leading methods reporting to achieve high performance of natural fibre composites include hand lay-up, spray lay-up, vacuum bag moulding, compression moulding, injection moulding, resin transfer moulding , pultrusion, filament winding. Table 4.6 summarises the benefits and drawbacks associated with the underlying composite manufacturing techniques.

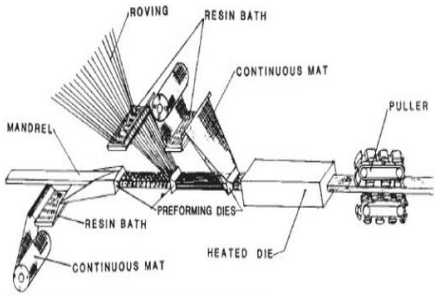
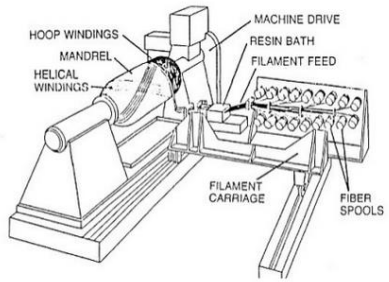
Table 4.6: Different methods of Composite manufacturing techniques Source (Kabir et al., 2012)

Composite fabrication method	Process description	Advantages	Disadvantages
Hand lay-up 	A process wherein the application of resin reinforcement is done manually onto a suitable mould surface. The resulting laminate is allowed to cure in place without further treatment. (used for thermoset matrix)	• Widely used • Low tooling cost • Custom shape • Larger and complex items can be produced	• Labour intensive • Low volume process • Formation of air bubbles when mixing both fibres and resin in atmospheric air • Styrene emission • Quality control is entirely dependent on the skill of laborers
Spray lay-up 	In spray up process liquid resin matrix and chopped reinforcing fibres are sprayed by two separate sprays onto the mould surface. Fibres are sprayed by an air jet simultaneously with a resin spray at a predetermined ratio between the reinforcing and matrix phase.	• Tooling cost is low • Semiskilled workers are easily trained • Design flexibility • Molded- in inserts and structural changes are possible • Minimum equipment investment necessary • The startup lead time and cost are minimal	• Labour intensive • Low volume process • Longer curing times • Production uniformity is difficult • Waste factor is high

Vacuum bag moulding  	Vaccumm bag moulding is a modification of the hand lay-up, in which the lay-up is completed and placed inside a bag made of flexible film and all edges are sealed. The bag is then evacuated, so that the pressure eliminates voids in the laminate, forcing excess air and resin from the mould.	• Higher fibre volume fraction-lower void content • Better fibre wet out due to pressure-resin flow • Heavier fabrics can be wet-out • Volatile organic compounds are largely contained during the curing stage • The additional consolidation pressure can help the reinforcement to conform to tight curvatures • Improved mechanical properties consequent upon the higher fiber volume fraction	• Higher labour skills for the bagging stage • Low production rates due to bagging stage • Joining bagging film for large items • Mould tool must be vacuum tight • The vaccum pump may strip volatiles from resin • Consumable material compatibility with resin

Compression moulding 	For the compression molding reinforcement is mixed with matrix and pressed with a heated plate under pressure (thermoplastic matrix)	• Low cost • Uniform density • Uniform shrinkage due to uniform flow • Improved impact strength • Dimensional accuracy • Internal stress and wrapping are minimized • Short cycle time • High volume production	• Large curing time • Uneven parting lines present • Scrap cannot be reprocesses • High initial capital investment
Injection moulding 	Fibre–resin is added as granulate to the machine and melted into fluid mass, then injected under high pressure into the form, and are used for composite fabrication	• Fast production – Can produce large amount of products/h • Design flexibility- Allows companies to produce complex products in different shapes and designs • High production- creates a high production output while having low labour cost • Little waste- any unused moulding plastic or scrap can be reground and can be recycled for future use	• High cost of machines • Unexpected costs that need to be budgeted for well in advance • Tight schedule- designs must be created before any process begins which could put production behind schedule

Resin transfer moulding  	The principle behind the composite manufacture is the mixing of the reinforcement with the resin and catalyst mixture and the solidifying it using heat and pressure into the net shape desired.	• High fibre volume laminates can be obtained with low void contents • Volatile emissions like styrene are massively reduced • High repeatability can be achieved • The laminate thickness can be closely controlled • Less dependent on the skills of the operator • Automation possible • Can produce a very strong part • Allows one to obtain even very complex neat-shape parts	• Matched tooling is inexpensive and heavy in order to withstand pressures • Impregnated areas can occur resulting in very expensive scrap parts • Higher cycle times • Large sized components needs very heavy and costly moulds • Can produce some thickness variation • Low production • Not possible to determine whether the reinforcement material is properly covered by the resin

Pultrusion  	A process for producing continuous length of shapes with a constant cross section by pulling resin-impregnated fibres through a heated die where curing occurs. (used for both thermosets and thermoplastics)	• Continuous process • Easy to automate, low Labour • High output, very long parts are possible • Uses inexpensive forms of reinforcement • Selective placement of reinforcement relatively easy • Low scrap	• Cross sections must be generally uniform • Difficult to maintain tight tolerances • Quick curing resin systems typically have lower mechanical properties • Complexity of process
Filament winding  	Is a process of winding fibre material and resin around a shape, konwn as a mandre, to create composite product. The process of filament winding is typically used to create circular composite products with a hollow core.	• Fast and economic method • High reproducible nature of process • Continuous fibre over the entire part – high fiber volume is obtainable • Ability to orient fibres in the load direction • Autoclave not necessary • Lack of ductility • Low modulus of elasticity	• Part configuration must facilitate mandrel extraction (no trapped cooling) • Mandrel could be complex and expensive • Inability to wind reverse curvature • Inability to easily change fibre path within one layer • As wound external surface may not be satisfactory for some applications

4.5.1 Conclusions (Composite manufacturing techniques)

In contrast to the various composite fabrication techniques as tabulated in Table 4.6, the hand lay-up technique is regarded as the simplest composites moulding method due to their ease of handling and processing, requiring low skilled workmanship, offering low cost tooling and a wide range of part sizes. Besides, the hand lay-up technique is mostly suitable for handling thermosets polymer matrix such as polyester and epoxy resin. In order to minimise air bubbles entrapped in the polymer resin, a modified hand lay-up technique could also be adopted such as the vaccum bag moulding. In this way, improved mechanical properties of the resulting composites could be achieved, enabling better fibre wet out due to pressure-resin flow and also pressure eliminates void in the composite, forcing excess air from the mould.

4.6 Influential factors of the composite mechanical properties

There are numerous factors that significantly impact the properties and interfacial characteristics of the composites. As highlighted below, technical problems such as the uniformity of fibre distributed within the composites, thermal degradations and weathering effect of fibre and matrix, water absorption of both fibre and matrix, wettability of resin impregnated into spaces between fibrils and breakage of fibres during mechanical stirring/mixing stages during the manufacturing processes limit the use of natural fibres and biodegradable polymers for new composite development.

4.6.1 Fibre anatomy

The critical micro-structural parameters affecting the mechanical characteristics of plant fibres include: the crystal structure, the degree of cellulose crystallinity, the degree of polymerization , microfibril angle, the porosity content, fibre aspect ratio the size of the lumen (center void), and the chemical composition, which are strongly correlated to the tensile properties of plant fibres. For instance, the cellulose structure of the fibres is distinguished through crystalline and amorphous regions. Large numbers of strong intra-molecular hydrogen bonds are formed in the crystallite region. This creates cellulose block and therefore impede chemical penetrations. However, in the amorphous phase, resins are easily absorbed. The hydrophilic hydroxyl groups present in this region are combined with water molecules from the atmosphere. Hemicellulose, lignin, pectin and waxy substances do normally hold these water molecules. This render the fibre hydrophilic and polar in character which lowers the compatibility with the non-polar/hydrophobic matrix (Abdelmouleh et al., 2007; Ansell and Mwaikambo, 1999, 1999). For the expansion of the crystalline region, the dismissal of the hydrophilic

hydroxyl groups and the exclusion of surface impurities (waxy substances), natural fibre necessitates to be chemically modified.

4.6.2 Fibre length, loading and orientation

The mechanical properties of the composites are also depended on fibre length, loading and orientation in the matrix. It is often observed that the increase in fibre length and fibre loading in composites improves its mechanical properties (Kabir et al., 2012). In general, high fibre content is required to achieve high performance of the composites. When a load is applied to the matrix, stress transfer occurs by shear at the interface along the fibre length and ends of the fibre. The extent of load transfer is a function of the critical fibre length (aspect ratio), the direction and orientation of fibre and the compatibility between fibre–matrix interfaces. Depending on the fibre orientation at the matrix, three types of composite are prepared. Firstly, longitudinally aligned fibre composites generally have higher tensile strength but lower compressive strength, due to fibre buckling. Secondly, transversely directed fibres undergo very low tensile strength, which is lower than the matrix strength. Finally, randomly orientated short fibre composites have different mechanical properties. This is due to the complexities of load distribution at different direction along the interfaces, consistent mechanical properties of these composites are recalcitrant. By controlling factors such as the aspect ratio, the dispersion and orientation of fibres, considerable improvements in the properties can be accomplished (Ansell and Mwaikambo, 1999; Joseph et al., 2003).

4.6.3 Moisture absorption of fibres

The lignocellulosic fibres are hydrophilic and absorb moisture. Many hydrogen bonds (hydroxyl groups AOH) are present between the macromolecules in the fibre cell wall. When moisture from the atmosphere comes in contact with the fibre, the hydrogen, bond breaks and hydroxyl groups form new hydrogen bonds with water molecules. The cross section of the fibre becomes the main access of water penetration. The interaction between hydrophilic fibre and hydrophobic matrix causes fibre swelling within in the matrix. This results weakening the bonding strength at the interface, which leads to dimensional instability, matrix cracking and poor mechanical properties of the composites (Zakaria and Poh, 2002). Therefore, the removal of moisture from fibres is an essential step for the preparation of composites. As aforementioned, the moisture absorption of fibres can be reduced by eliminating hydrophilic hydroxyl groups from the fibre structure through different chemical treatments (Wang et al., 2007). Moisture content cause dimensional variation in composites and ultimately affects the mechanical properties of the composites. During manufacturing of thermoset based composites, moisture leads to void contents and affects fibre–matrix bonding that lead to decrease in mechanical properties. (Jawaid and Abdul Khalil, 2011).

4.6.4 Presence of voids

During the insertion of fibre into the matrix, air or other volatile substances may be trapped inside the composites. After the curing process micro-voids are formed along the individual fibre tows and in the matrix rich regions. This causes sudden failure of the composites and exhibit poor mechanical properties. The curing and cooling rate of the composites are also responsible for the void formation (Joseph et al., 2003). High void content (over 20% by volume) is responsible for lower fatigue resistance, greater affinity to water diffusion and increase variation (scatter) in mechanical properties (Bowles and Frimpong, 1992; Vaxman et al., 1989). Composites at higher fibre content initiate more risk for void formation (Vaxman et al., 1989).

4.6.5 Thermal stability of fibres

The degradation of natural fibres is a paramount criteria in the development of composite (Sgriccia et al., 2008; Taj et al., 2007). Natural fibre generally starts degrading at about 240 ^{0}C. Structural constituents of the fibre (cellulose, hemicelluloses, lignin) are sensitive with different range of temperatures. It was reported that, lignin starts degrading at a temperature around 200 ^{0}C while hemicelluloses and cellulosic constituents degrade at higher temperatures(Joseph et al., 2003). Thermal stability of the fibre can be enhanced by removing certain proportion of hemicelluloses and lignin constituents by different chemical treatments. Cellulosic fibres have low thermal stability that results in the exclusion of some manufacturing processes, and also limits the use of the composites to low temperature applications. The low thermal stability increases the possibility of cellulosic degradation and the possibility of emissions of volatile materials that could adversely affect the composite properties. Processing temperatures are thus limited to about 200 ◦C, although it is possible to use higher temperature for short periods of time. (Jawaid and Abdul Khalil, 2011)

4.6.6 Biodegradability

Cellulosic fibres degrade easily when exposed to nature. Some methods for degradation include biological, chemical, mechanical, thermal, photochemical and aqueous. The biodegradability of cellulosic fibre is regarded as a positive attribute, justifying the use of these fibres. Nevertheless, for many outdoor applications it is essential for the composites to be serviceable for several years. In order to surge their service life, it is vital to control this natural degradation. One method of slowing down the natural degradation process is by modifying the cell wall chemistry. Undesirable cellulosic fibre properties such as dimensional instabilities, flammability, biodegradability, and chemical degradation can be eliminated in this way (Rowell and Rowell, 1996). Chemical treatments can reduce the water uptake in the fibres, and can therefore reduce the amount of fibre swelling and biological degradation

by blocking the available –OH group on the fibre surface (Joseph et al., 1999). It is also reported that encasing natural fibres in thermoplastic reduce water uptake.

4.7 Conclusions

The literature review has given an overview of the different existing ways of fibre extraction and its treatment. The fibre is treated so as to render its surface cohesive so that it can 'blend' well with the polymer resin and give the highest possible performance in terms of reinforcement within the matrix and using the different methods of composite manufacture also identified. It has been found that the length of fibres, its moisture content and its orientation in the matrix play a significant role in the resulting strength of the composite. However, during manufacture, air should not be trapped in the matrix since the presence of air bubbles weakens act as weak places in the composite materials.

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CHAPTER 5: METHODOLOGY

The aim of the is work is to assess the potential of using fibres from fibrous plants in Mauritius with a view to using them in composite materials for engineering applications. Hence it is important to identify the plants from diiferent locations, find the best method of fibre extraction from each plant identified and find the optimum fibre treatment method and assess the imact of same in the mechanical and chemical properties of the fibres. Figure 5.1 gives an overview of the methodology used.

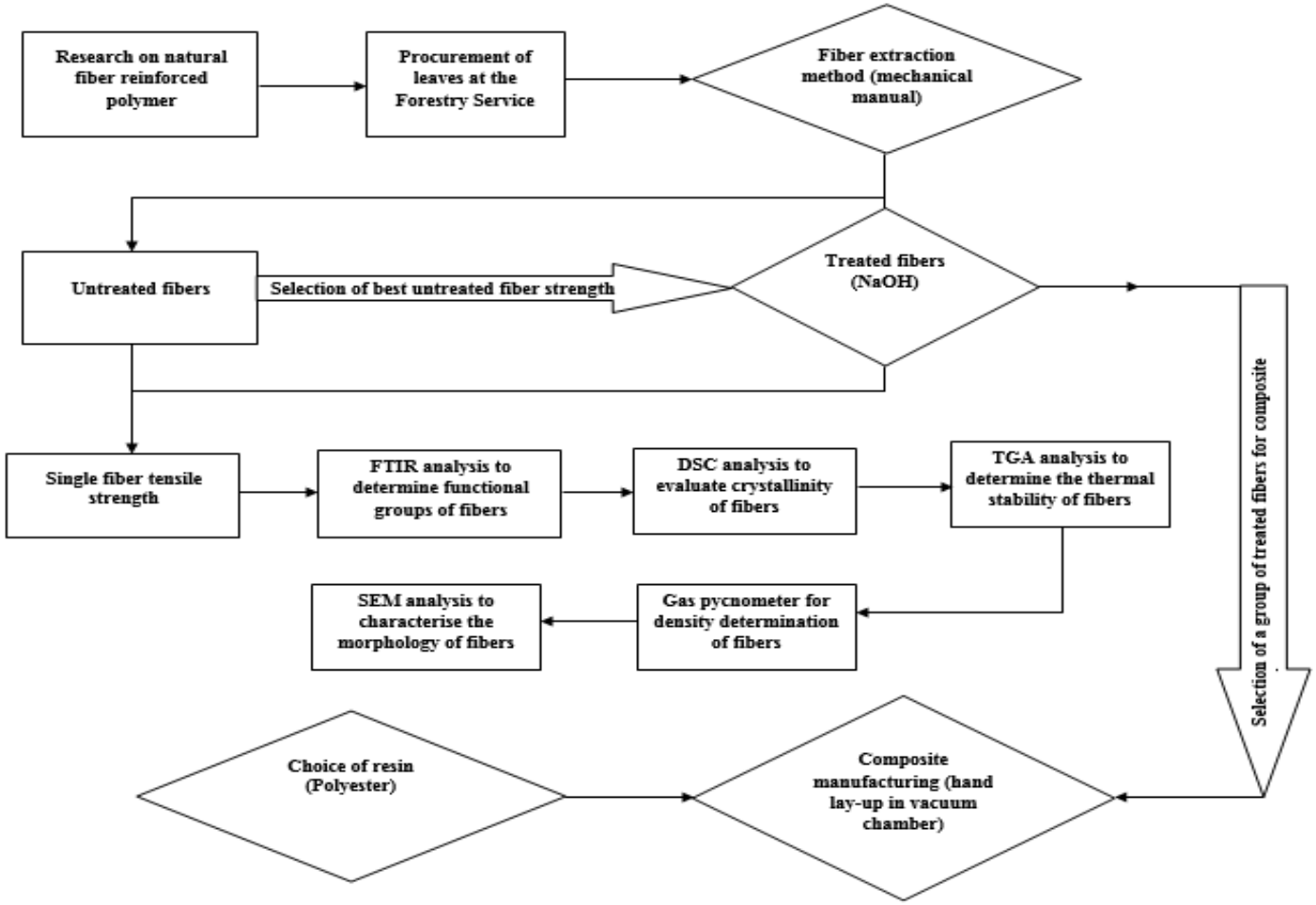


Figure 5.1: The methodological process

5.1 Mode of fibre extraction

The main purpose of fibre extraction achieve the highest fibre yield with minimal damage to the fibres. Thus, the suitable type of extraction for each type of plant should be found.

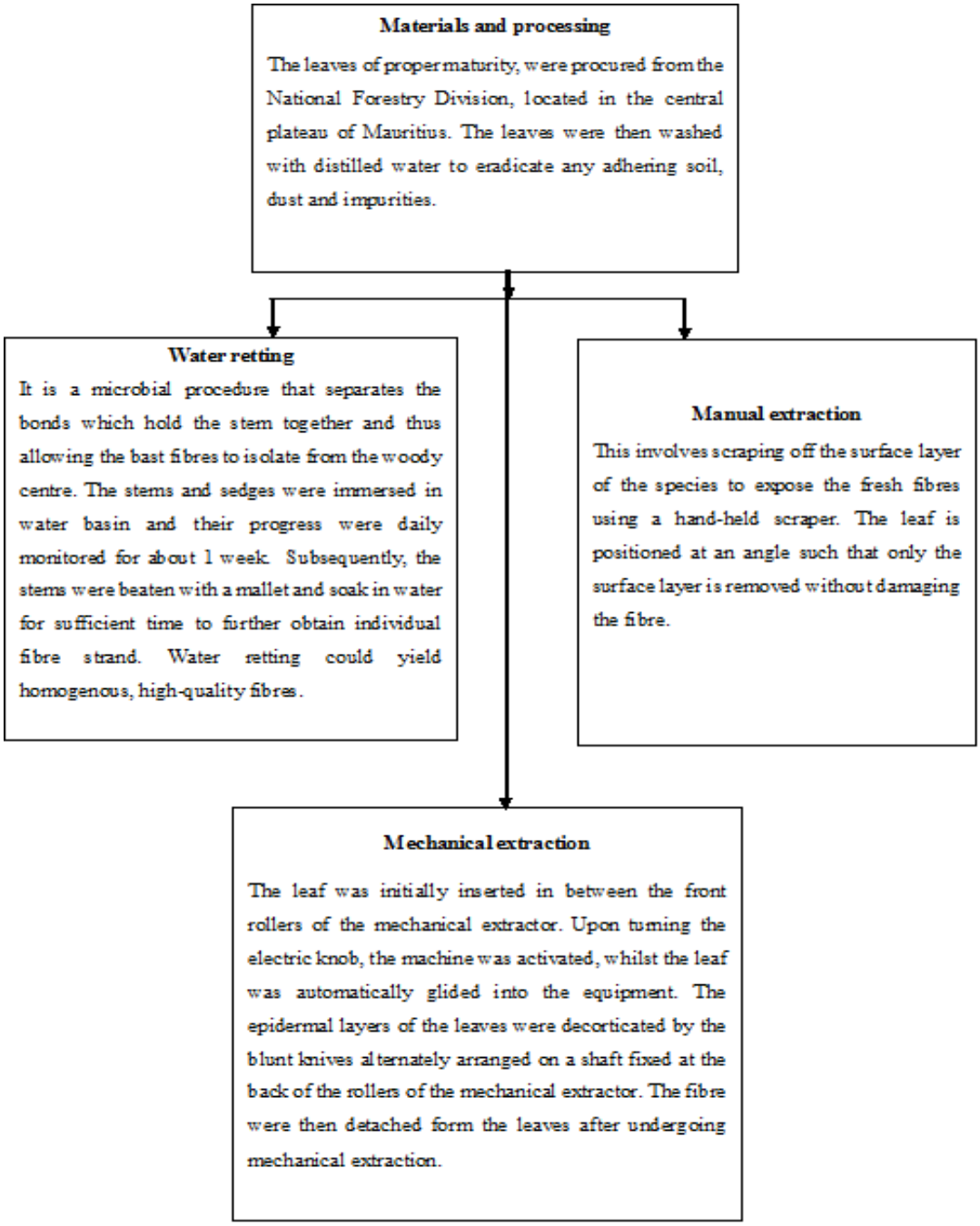


Figure 5.2: Various mode of fibres extraction

5.2 Alkali treatment of fibres

Fibres were chemically treated with 5 % NaOH solution at room temperature and reaction time of 24 hr. The resultant fibres were then removed from the caustic solution and washed several times with

distilled water until the pH of the solution becomes neutral. The materials were then finally dried for 24 hr at 60^0C in oven in order to remove excessive moisture content. The purpose of this treatment was to remove lignins, waxes and oils covering the external surface of the fibre wall and to expose the hydroxyl groups.

5.3 Fibre mat fabrication

After undergoing alkali treatment, mechanically extracted fibres form *Dracaena Concinna, Dracaena floribunda, Ravenala M* and manually extracted fibres from *Thysalaeno Latifolia* were chopped into 50 mm fibre length. A layer of fibre mat were then constructed by using readily available paper glue to stick the fibres together and held them in position in the mould. Experimental parameters were tested by using various weightage of fibre mat layers (3 g and 5 g fibres) to observe the fiber surface adhesion to surrounding matrix.

Figure 5.3: Schematic view of Fibre mat fabrication

5.4 Polymer matrix

C114TS Polyester resin was purchased from AMRO Chemicals Ltd. C114TS is a thixotropic, promoted orthophthalic Unsaturated Polyester Resin, designed for hand lay-up and spray-up of parts where short mould turnover and low peak exothermic are required and is suitable for 3~15 mm application wet-on-wet . Details of the Unsaturated Polyester resin C114TS are illustrated in the Table 5.1.

Table 5.1 : Technical details of C114TS Polyester resin

PERFORMANCE

- Good workability.
- Good wet-out and easy defoaming.
- Has excellent mechanical strength.
- Has high thixotropic efficiency.

SPECIFICATIONS

- Solid Content, % 55 ~ 60
- Acid Value, mgKOH/g 15 ~ 26
- Viscosity, cps, Brookfield, No.3/60rpm at 25℃ 450 ~ 500
- Gel Time, min. at 25℃, 1%MEKPO(50%) 30 ~ 35
- Thixotropic Index 2.7 ~ 3.5

TYPICAL PHYSICAL PROPERTIES OF CURED CASTINGS

- Barcol Hardness 45
- Tensile Strength, kg/mm^2 5.5
- Elongation, % 2.0
- Flexural Strength, kg/mm^2 10
- Flexural Modulus, kg/mm^2 420
- Heat Distortion Temerature, ^oC 65
-

Table 5.1 : Technical details of C114TS Polyester resin (*continued*)

TYPICAL PHYSICAL PROPERTIES OF REINFORCED RESIN,30% CSM FIBRE

- Tensile Strength, kg/mm^2 11
- Flexural Strength, kg/mm^2 22
- Flexural Modulus, kg/mm^2 750

STORAGE STABILITY

The storage life in drums at room temperatures is three months. Storage life decreases with increasing temperature. Avoid exposure to heat sources such as direct sunlight or steam pipes. Keep containers sealed to prevent monomer loss and moisture pick-up.

5.5 Composite Fabrication

The modified Hand-lay-up technique in the vacuum chamber was adopted for composite fabrication. A simple and low -cost vacuum chamber made up of Perspex Acrylic; normally acknowledged as glove box was constructed. An alu-carbon mould (180 ×165 ×7) mm was also designed and shown in Figure 5.4. The mould was first prepared by applying a layer of releasing agent to facilitate the removal of the cured composite specimens from the mould. Previously, prepared fibre mat was then placed in the mould. Weighted amount of C114TS polyester resin were then poured in an aluminium can along with 1 % of catalyst as per the manufacturer's specifications was measured in a syringe . After all the materials (mould, fibre mat, Polyester resin and catalyst) were positioned in the vaccum chamber, the vacuum pressure were then activated to remove air from the chamber. The catalyst was then released in the Aluminium can, containing the C114TS polyester resin with continuous stirring for 3 mins until homogeneity. The resulting mixture was then carefully transferred in the mould for the fibre mat to impregnate into the resin and to increase the fibre wettability. After this process, the vaccum pressure were then turned off. The composite samples were allowed to cure at room temperature and left overnight to cool and solidify in the vacuum chamber. After the composite solidification (Figure 5.5(a)), the latter were removed from the mould and directed on the CNC machine for shaping the test specimen as shown in Figure 5.5(b). The dimensions of the cut samples should conform to the dimensions of the standard as shown in Figure 5.6 and Table 5.2.

Figure 5.4: Schematic view of the Modified hand lay-up technique in a vacuum chamber

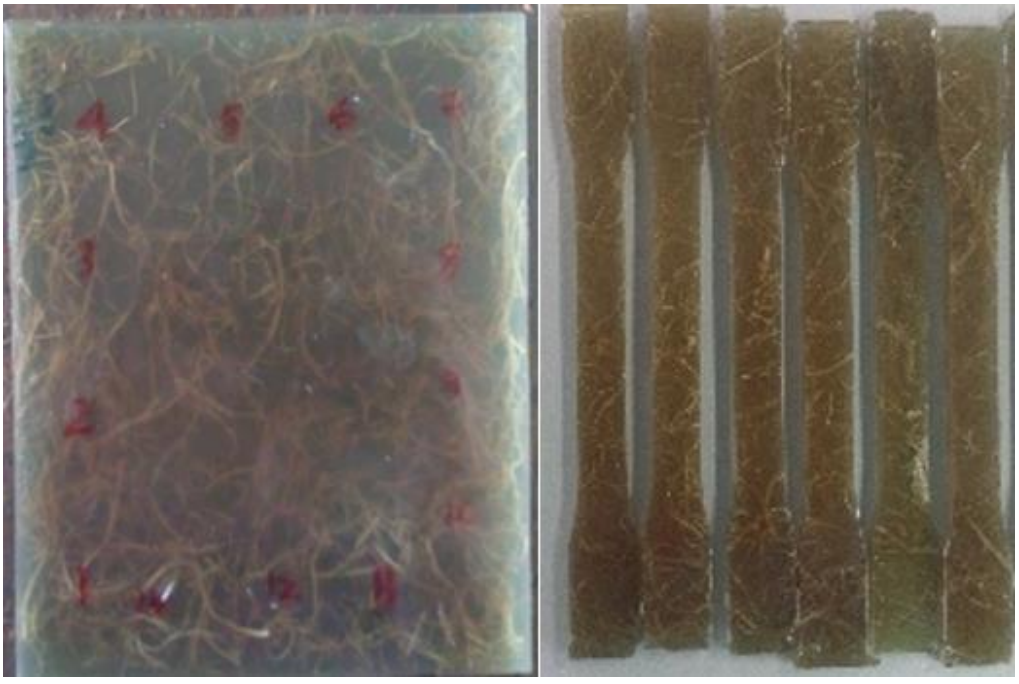


Figure 5.5 (a) Composite Before cutting (b) Composite After cutting

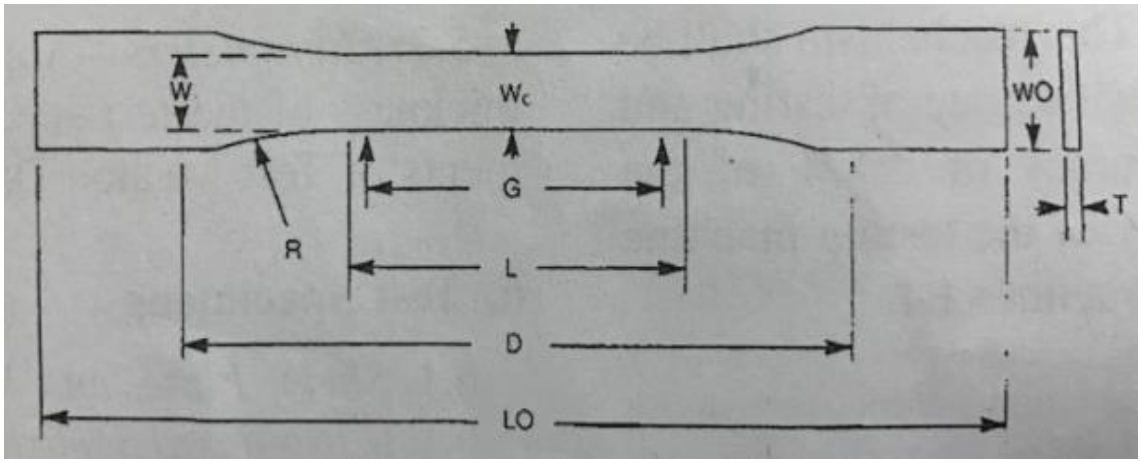


Figure 5.6 Shape and dimensions of test samples (pl refer to Table 5.2 as well)

Table 5.2: Dimensions of the test specimen

Dimensions			Value/mm
W	-	Width of narrow section	13
L	-	Length of narrow section	57
WO	-	Width overall	19
LO	-	Length overall	165
G	-	Gage length	50
D	-	Distance between grips	115
R	-	Radius of fillet	76
T	-	Thickness	7

5.6 Mechanical testing of fibres

Longitudinal fibre tensile tests were conducted according to ASTM C1557 using a Universal Testometric Testing Machine equipped with a 100 N load cell and a cross-head speed of 0.48 mm/min was maintained throughout the testing. The gauge length of each fibre was specified at 25.4 mm, and the fibre were then mounted on the apparatus. To obtain a pragmatic estimate of the fibre mechanical properties, a population of 50 fibre specimen was tested for each batch. The diameter of the broken fibre was then carefully measured using a calibrated optical microscope.

The tensile strength for each fibre were then computed using the equation: $T = \frac{F}{A}$, where

T = Tensile strength, Pa;

F= Force to failure, N;

A= Fibre cross sectional area at fracture plane (normal to fibre axis), m^2.

5.7 Mechanical testing of composites

Tensile strength properties of the reinforced composite samples were characterised in accordance with ASTM D3039 on a Universal Testometric Testing Machine (Figure 5.7) operating at a crosshead speed of 5 mm/min and load cell of 50000 N. The test specimen is held in such a way that the slippage relative to the grips is prevented. 10 specimens were tested to obtain average tensile properties of the composite.

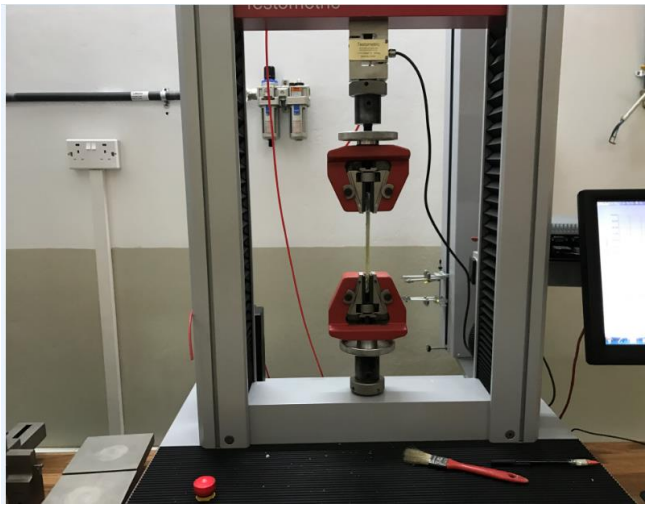


Figure 5.7: Tensile test of the composite specimen in the Universal testing machine

The fractured samples are shown in Figure 5.8.

Figure 5.8: Fracture point of the test specimen

5.8 FTIR analysis

FTIR analysis was performed to characterise the functional groups of the different chemical constituents of each fibre and eventually to identify any alteration in their functional groups after alkaline treatment. The analysis was effected using a Bruker single bounce ATR-FTIR spectrometer (Figure 5.9), equipped with its Opus software. Prior to the analysis of the fibres, a background measurement was evaluated as reference. The fibres to be analysed were then inserted under the probe, clamped firmly and analysed by the spectrometer. Finally, the spectra were subsequently obtained with an accumulation of 25 scans for wave number ranging from 4000 to 400 cm^{-1}. The presence of the three functional groups (cellulose, hemicellulose and lignin) are determined by the FTIR analysis.

Figure 5.9: Schematic view of the Bruker ATR-FTIR spectrometer

5.9 DSC analysis

The Netzsch DSC 200 F3 Maia® thermal analyzer was used. Sample weighing approximately 5mg was placed in an aluminium crucible and sealed using a press. The sample was then heated in nitrogen atmosphere from room temperature to 400°C at a heating rate of 10°C /min. The melting temperature (T_m) was consequently taken as the peak temperature of the melting endotherm.

Figure 5.10: Schematic view of the Netzsch DSC 200 F3 Maia® thermal analyzer

5.10 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis of the samples was conducted on a TG209 F3 Tarsus® Thermo-Microbalance analyzer (NETZCH, India) under nitrogen atmosphere where the fibre samples (6-8 mg) were heated from 25°C to 1000°C using a heating rate of 10°C/ min.

5.11 Scanning Electron Microscopy (SEM)

A Tescan Vega 3 LMU microscope was used to examine the surface and cross-sectional morphology of the fibres with a high voltage of 30.0 kV. The fibre samples were sputter coated with gold/ palladium before subsequent SEM imaging.

CHAPTER 6: RESULTS OF MECHANICAL PROPERTIES

6.1 Results of extraction of natural fibres

Several fibrous plants were investigated during the course of this work. Tables 6.3 and 6.4, at the end of this chapter, show the list of plant species for successful extraction of untreated and treated fibres as well as list of plant species for unsuccessful extraction.

6.2 Results of Tensile strength of natural fibres

Figure 6.1 shows the results of the tensile strength in MPa of the fifteen (15) plant fibres for both untreated and mercerized condition. For most of the fibres, there is an increase in the mean tensile strength which varies between 9.5 % and 92.2 % when treated with 5 wt.% NaOH solution, except for Afromomum, Hurricane palm fibres and Sporobolus africanus (Patte poule). No change in the mean tensile strength of Sporobolus africanus was noted, whereas for Afromomum and Hurricane palm fibres, a decrease of 27.6 % and 32.7 %, respectively in the mean tensile strength was obtained. For untreated fibres, the highest maximum (219 MPa) and mean tensile (186 MPa) were obtained with the Dracaena floribunda fibres, followed by Ravenala (max = 206 MPa, and mean = 166 MPa), and Draceana concinna (max = 184 MPa, and mean = 144 MPa). The same trend was obtained for the alkaline treated fibres, with the highest maximum and mean tensile strength being 287 MPa and 215 MPa respectively for Ravenala.

Haameem *et al.* (2016) have investigated the effect of NaOH concentrations of 5, 10, 15, and 20% on Napier grass fibres. These authors have reported that the maximum tensile strength was obtained with the 10% alkaline-treated fibres.

Hussain *et al.* (2011) investigated the effect of sodium hydroxide (NaOH) solution on green coconut fibres with concentration of 2, 3 and 4 % for 24 hours at room temperature. These authors reported that the maximum tensile strength of 42.09 MPa was obtained when green coconut fibres were treated with 2 % NaOH solution.

Wong *et al.* (2010) have treated bamboo fibres with 1, 3 and 5% of NaOH and, according to them, higher concentration would damage the fibre too much. These authors reported that the untreated fibre was found to have higher yield strength, ultimate tensile strength, and stiffness than treated fibres. The ultimate tensile strength of the untreated fibre was 518 ± 51 MPa

compared to 187 ± 40 MPa at 1 % NaOH, 213±43 MPa at 3 % NaOH and 222 ± 47 MPa at 5 % NaOH. However, the strain at break increased from 10.4 ± 0.2 % from untreated fibre to 31.6 ± 0.5 % for 1 % NaOH, and then decrease to 23.4 ± 0.4 % at 3 % NaOH, and finally 15.2 ± 0.5 % at 5 % NaOH.

Figure 6.2 shows a schematic diagram of a plant fibre. The cell wall of a plant fibre can itself be considered as a composite structure since it consists of several layers, and within each layers there are different constituents. Each fibril has a complex structure consisting of the following: a primary wall which consist of an irregular network of microfibrils, and a secondary wall which consists of three layers with a thick middle layer with orderly arranged cellulose microfibrils.

In the middle layer S_2 of the secondary wall (Figure 6.2) the microfibrils are closely packed and run in a steep helix, and forms a small angle to the fibre longitudinal axis. The angle of inclination that the microfibrils makes with the main fibre axis is called the microfibril angle (MFA), and is a major parameter in influencing the tensile strength of natural fibres. Mwaikambo *et al.* [8] reported the work of other authors where it is mentioned that sisal fibres with a lower microfibril angle (less than 30°) has a higher cellulose content (more than 60 %) as compared to coir fibres which have a lower cellulose content (less than 50 %) with a higher microfibril angle (above 30°). Furthermore the tensile strength of the sisal fibres is higher than that of the coir fibres by about 5 times. This tends to show that the tensile strength is related to the cellulose content as well as the MFA.

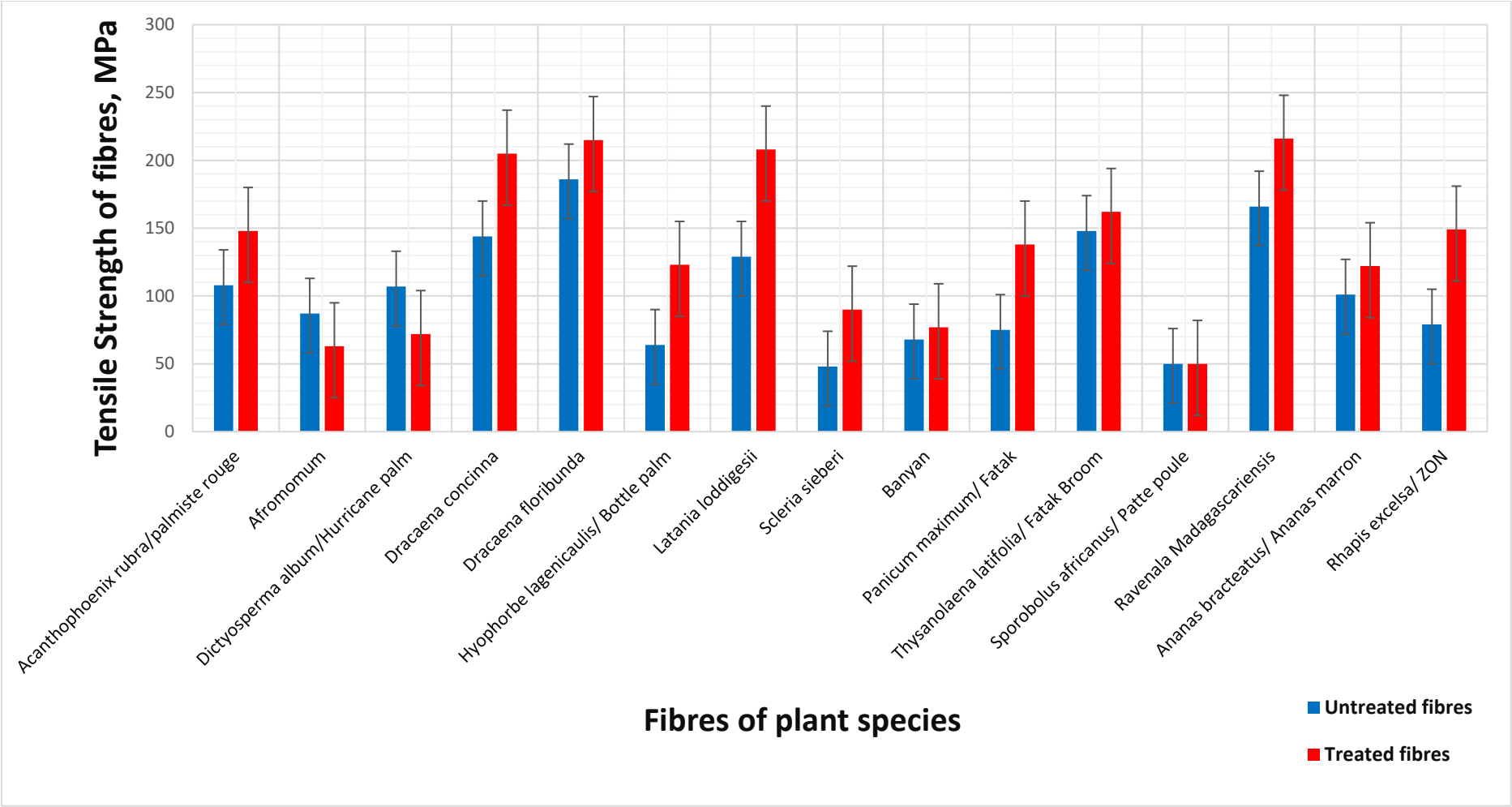


Figure 6.1 Tensile strength of the 15 different fibres

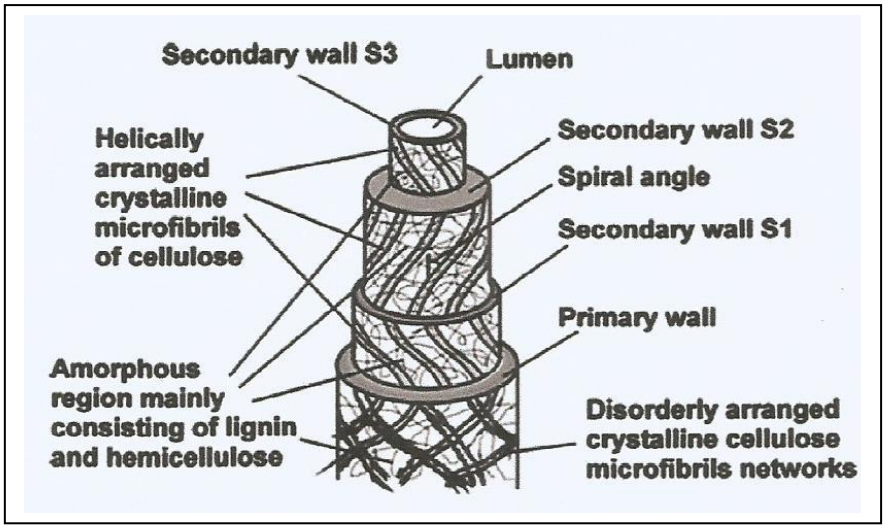


Figure 6.2: Structure of a single fibril cell [M. Ramesh, 2016].

The tensile strength of untreated Dracaena floribunda is more than twice that of the untreated Afromomum, and 73 % higher than that of the Hurricane palm untreated fibres. The tensile strength of the 5 % alkali treated Draceana floribunda fibre is 3 times higher than the treated Hurricane palm, and 3.4 times higher than the treated Afromomum fibre.

The significant decrease in the tensile strength of Afromomum and Hurricane palm fibres can possibly be due to a lower cellulose crystalline content, and a thinner outer (primary) wall layer which contains mainly the hemicellulose and lignin together with disordered crystalline cellulose microfibrils network. Moreover, there is a probability that the microfibril angle of Afromomum and Hurricane palm fibres is higher as compared to that of Dracaena floribunda.

Figure 6.3 shows the results of the tensile strength of fibres from four (4) species. These fibres were generally of low tensile strength, typically less than 100 MPa except for the Cyperus distans/triangular stem grass fibres (106 MPa). But the fibres were also of poor quality, very brittle, non-uniform in dimension, and relative short in length. The extraction process yielded a very low amount of fibres, and this does not offer any useful prospect to further investigate the effect of chemical processing on the fibre properties.

It is seen from Table 6.4 that it was not possible to extract useful fibres from the following five (5) plants: *Phragmitis mauritianus* (stem), *Machaerina iridifolia* (stem), *Machaerina anceps* (stem), *Daniella Ensifolia* (stem), and *Crinum M* (leaf blade).

Asim et al. (2016) obtained 432.01 MPa and 455.74 MPa for Pineapple leaf fibre and kenaf fibre respectively with 6 % NaOH solution. Ridzuan *et al*. (2016) obtained a mean tensile strength of 141 MPa for a 5 % NaOH treatment of Pennisetum *purpureum* (Napier grass) fibres whereas the tensile strength of the untreated fibe was 73 MPa.

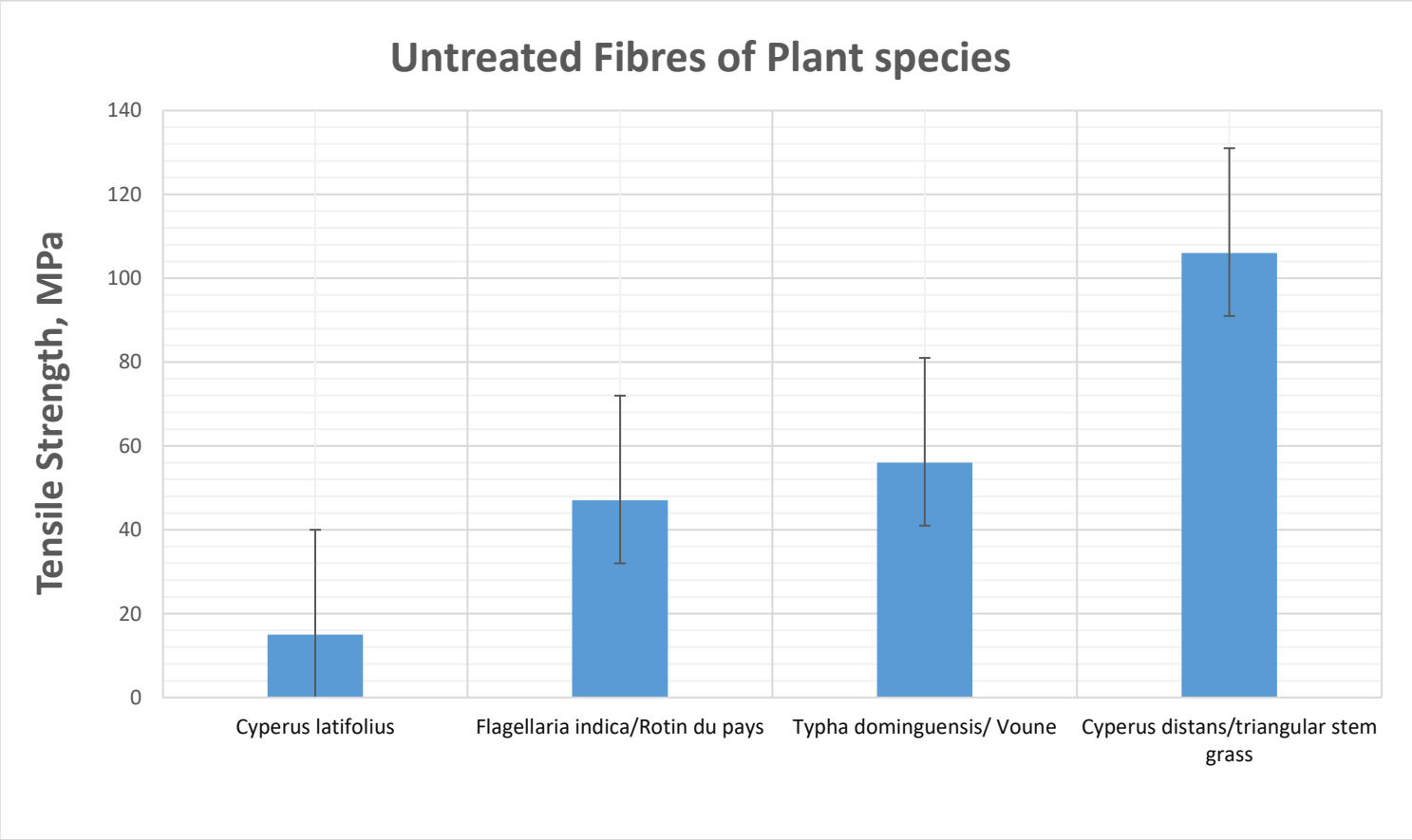


Figure 6.3: Tensile strength of untreated fibre strength

Based on the present experimental work, the mean tensile strength of 5 % alkaline treated Ravenala fibre (216 MPa) as well as that for Dracaena floribunda fibre (215 MPa) are comparable to that obtained for the 3 -5 % alkaline treated Bamboo fibres (213-222 MPa) (Wong *et al*. (2010)), and higher than the tensile strength of alkali treated Napier grass (Ridzuan (2015)). This definitely supports the proposal to use these fibres as reinforcing materials for biocomposites.

6.3 Density of fibres

Table 6.1 shows the results of the He pycnometer density test for the two fibres: Dracaena concinna and Ravenala.

Table 6.1: Density of ten replicates for each of the two fibres

	Density, g/cm^3									
Dracaena concinna	1.6013	1.6074	1.6075	1.6104	1.6117	1.5690	1.5783	1.5832	1.5847	1.5840
Ravenala	1.5848	1.5409	1.5639	1.6004	1.6081	1.5613	1.5583	1.5602	1.5960	1.5732

The density of these two (2) plants are relatively high and comparable to other common fibres such as ramie, cotton and abaca fibres (Table 6.2). This high density implies a low porosity as can be observed in Figure 6.4 where the SEM image of an untreated ravenala fibre revealed a relatively compact internal structure of the fibre.

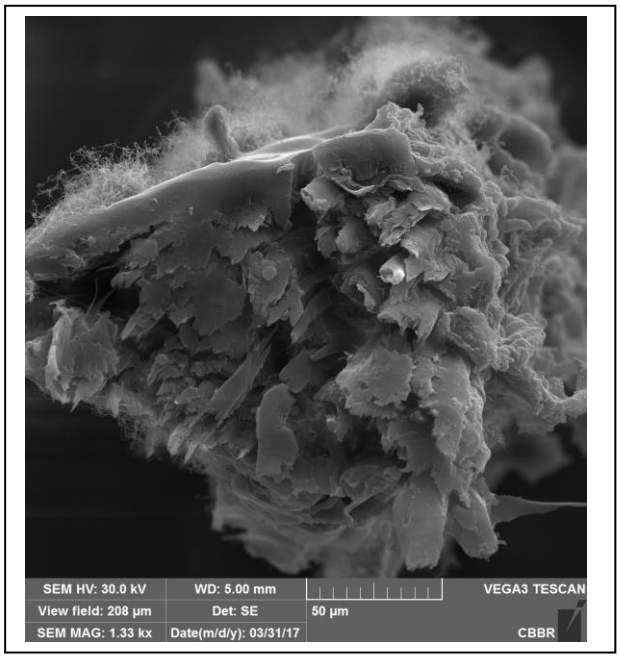


Figure 6.4: SEM image of a Ravenala fibre at 1330 X.

Table 6.2: Density of natural and glass fibres

Density/ g/cm^{-3}	**Fibre**								
	Abaca	Coir	Cotton	Flax	Hemp	Jute	Ramie	Sisal	E-glass
	1.5	1.25	1.51	1.4	1.48	1.46	1.5	1.33	2.55

Source: *Brouwer W. D., FAO*

6.4 Results of Tensile Strength of Composites

Figure 6.5 shows the results of the tensile strength of the polyester resin-fibre composites with the four (4) different fibre species. The mean tensile strength of the polyester resin is 35.24 MPa, and irrespective of the weight of fibre used (3 or 5g) or the fibre used, the tensile strength of the fibre reinforced composites decreased by about 25-35 % except for the reinforcement with the Thysanolaena latifolia (Fatak) fibres where the decrease is more significant (47 %).

The random orientation of the 50 mm chopped fibres do not necessarily improve the tensile strength in the axial direction of the specimen. According to the cited authors, a matrix should include fibres oriented in the axial direction to yield improved strength in that direction. Wong *et al* [49] argue that shorter fibres tend to act as filler within the matrix rather than a reinforcement material. Moreover, a clear gap (Figure 6.6) can be observed between each fibre

and the matrix; showing that the individual fibres have not properly adhered to the matrix, and this can explain the lower tensile strength of the composite. Improvement of fibre adherence to the resin requires further investigation.

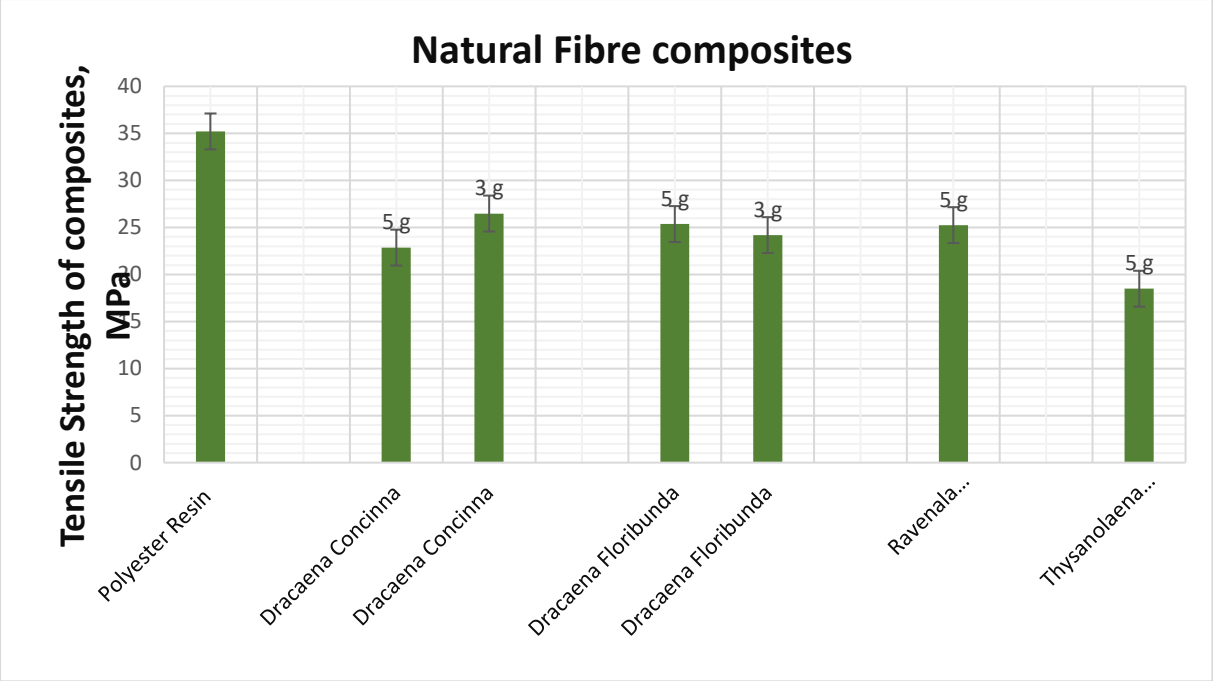


Figure 6.5: Tensile strength of composites

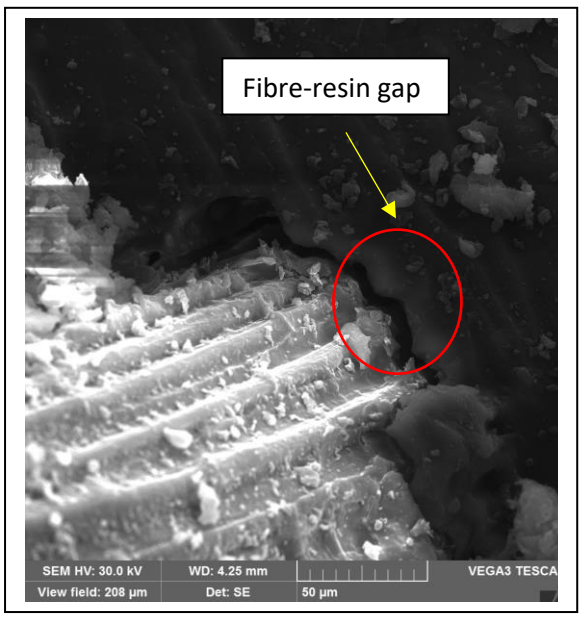
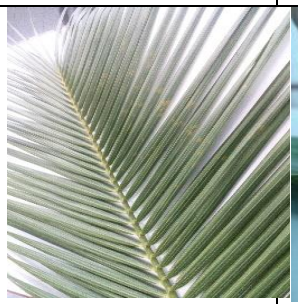
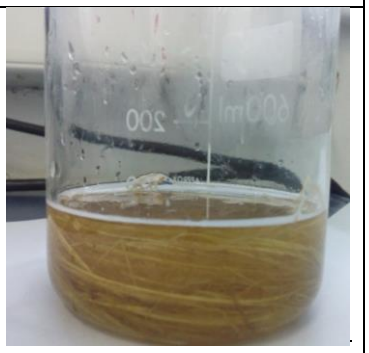
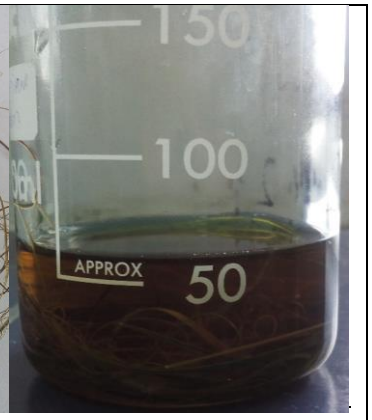
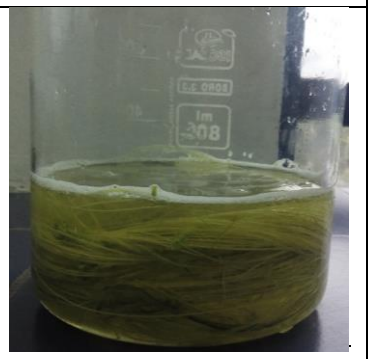
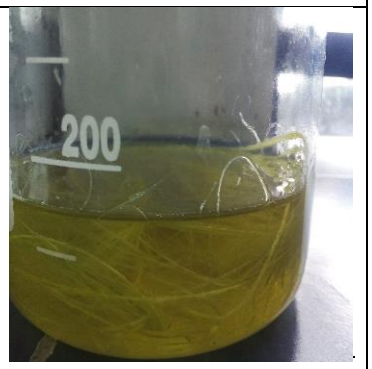
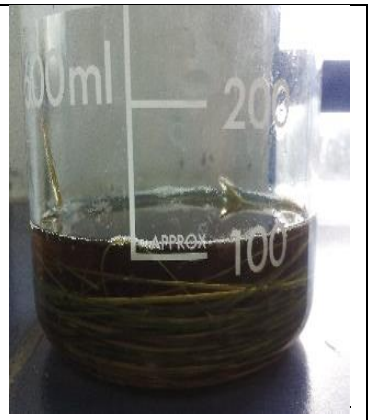
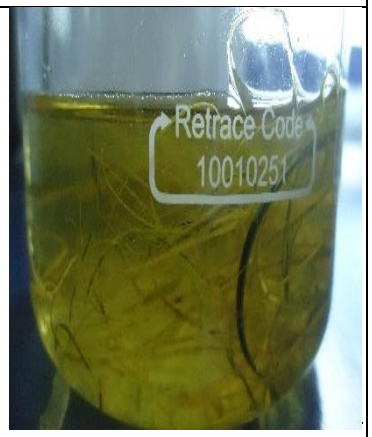


Figure 6.6: SEM image showing gap between the ravenala fibre and the resin at 1330X.

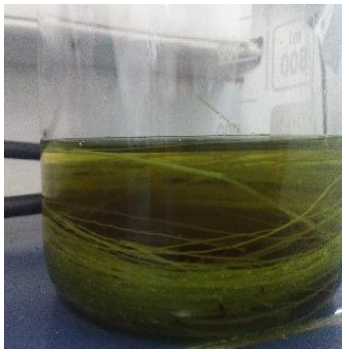
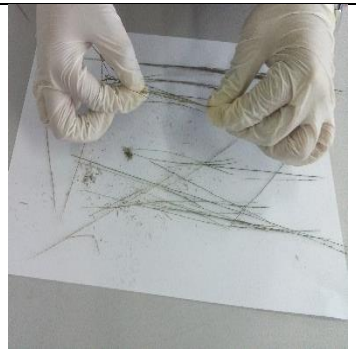
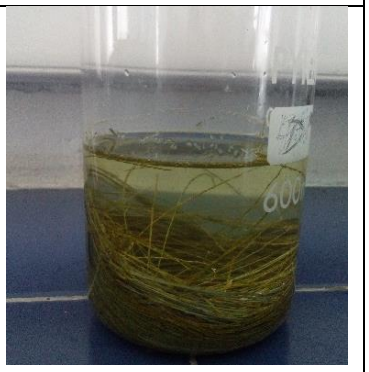
Table 6.3 List of Plant Species for Successful Extraction of Untreated and Treated Fibres

Plant genus/ species (part of plant undergoing extraction)	Photo of plant genus/species	Extraction process/mechanical; manual; hammering and water retting	Untreated fibres	Treated fibres	Alkali treatment
Acantho phoenix rubra/ palmiste rouge (leaf blade)					
Afro momum Augustifoliu m (stem)					

Dictyo sperma album/ Hurricane palm (Leaf blade)					
Dracaena concinna (Leaf blade)					
Dracaena floribunda (Leaf blade)					

Hyophorbe lagenicaulis/ Bottle palm (Leaf blade)						
Latania loddigesii (Leaf blade)						

Scleria sieberi (stem)					
Others					
Banyan (stem)					

Panicum maximum/ Fatak (flower)					
Thysanolaena latifolia Fatak Broom (flower)					
Sporobolus africanus/ Patte poule (stem)					

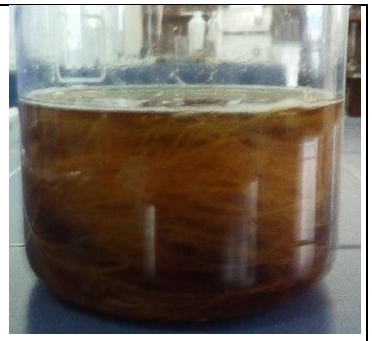
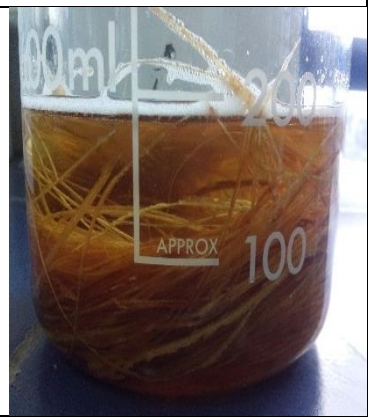
Ravenala. Madagascariensis (leaf sheath)					
Ananas bracteatus/ Ananas marron (leaf blade)					
Rhapis excelsa (stem)					

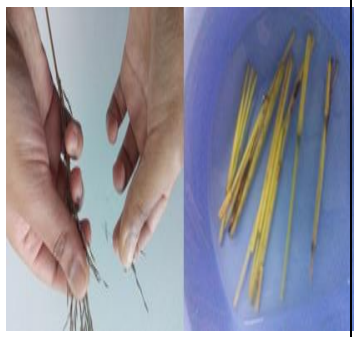
Table 6.4: List of Plant Species for Unsuccessful Extraction of Untreated and Treated Fibres

Plant genus/ species (part of plant undergoing extraction)	**Photo of plant genus/species**	**Extraction process/mechanical; manual; hammering and water retting**	**Untreated fibres**	**Comments**	
Crinum M (leaf blade)					Extraction not possible. The leaves were reduced to pieces after undergoing mechanical extraction
Cyperus latifolius (stem)					No further alkali treatment carried out as confirmed by preliminary tests.

Daniella Ensifolia (stem)					Extraction not possible. No single fibre strand detected in the stem. Presence of very fine fibres in the leaves.
Flagellaria indica/Rotin du pays (stem)					No further alkali treatment carried out as confirmed by preliminary tests.
Hypolytrum mauritianum	Not tested/rare				

Machaerina anceps (stem)					A meagre amount of fibres could only be extracted from this rare species.
Machaerina iridifolia (stem)					Extraction not possible. No single fibre strand detected in the stem. Presence of very fine fibres in the leaves.
Phragmitis mauritianus (stem)					Extraction not possible. No single fibre strand detected in the stem. Presence of very fine fibres in the leaves.

Typha dominguensis/ Voune					No further alkali treatment carried out as confirmed by preliminary tests.
Others					
Cyperus distans/ triangular stem grass (stem)					Fibre deterioration. No further test carried out.

Chrysopogon zizanoides/ Vetivert (Stem and flower)					Extraction not possible neither in stem nor flowers.

6.6 References

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Hussain SA, Pandurangadu V, Palanikuamr K. Mechanical properties of green coconut fiber reinforced HDPE polymer composite. International Journal of Engineering Science and Technology. 2011 Nov;3(11):7942-52.

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Ridzuan, M. J. M., Majid, A., Afendi, M., Aqmariah Kanafiah, S. N., & Nuriman, M. B. M. (2015). Effects of alkaline concentrations on the tensile properties of Napier grass fibre. In *Applied Mechanics and Materials* (Vol. 786, pp. 23-27). Trans Tech Publications.

CHAPTER 7: ANALYSIS OF FTIR RESULTS

7.1 Band Wave numbers used for the analysis of the FTIR

Several authors, namely, El Ghali *et al.* (2012), Cai *et al.* (2015), Krishnaiah *et al.* (2017), Mandal *et al.* (2011) and Sun *et al.* (2004) have investigated the effect of alkali treatment on the removal of hemicellulose and lignin from natural fibres. Table 7.1 shows a summary of the different bond and the associated band wave number.

Table 7.1 IR Absorptions of some Functional Groups

Band Wave number (cm^{-1})	Description	Remarks
1730	C=O from pectin, hemicellulose.	All these 4 bands disappeared completely in the spectra of cellulose fibres obtained after $NaClO_2$ treatment and alkali treatment.
1602	C=C aromatic ring of lignin.	
1505	C=C aromatic ring of lignin.	
1245	C-O of aryl group in lignin.	
3500-3200	Free O-H of OH groups in cellulose molecules.	In all samples (untreated and treated).
2894	C-H	In all samples.
1365	Bending vibration of C-H and C-O bonds in the polysaccharide aromatic rings.	In all samples.
1649-1641	OH bending of the adsorbed water.	In the spectra of cellulose fibres obtained after alkali treatment and the nanocellulose.
1054	C-O-C pyranose ring.	In all samples.
902	Associated with the Beta-glycosidic linkages between glucose units in cellulose which stands for cellulose II, the content of which increases progressively from bagasse to nanocellulose (more present in the alkaline treated fibres as compared to untreated fibres).	
1155	C-C ring polysaccharide component.	Gradually lost in nanocellulose because of hydrolysis and reduction in molecular weight.
1105	C-O-C glycosidic polysaccharide component.	Gradually lost in nanocellulose because of hydrolysis and reduction in molecular weight.

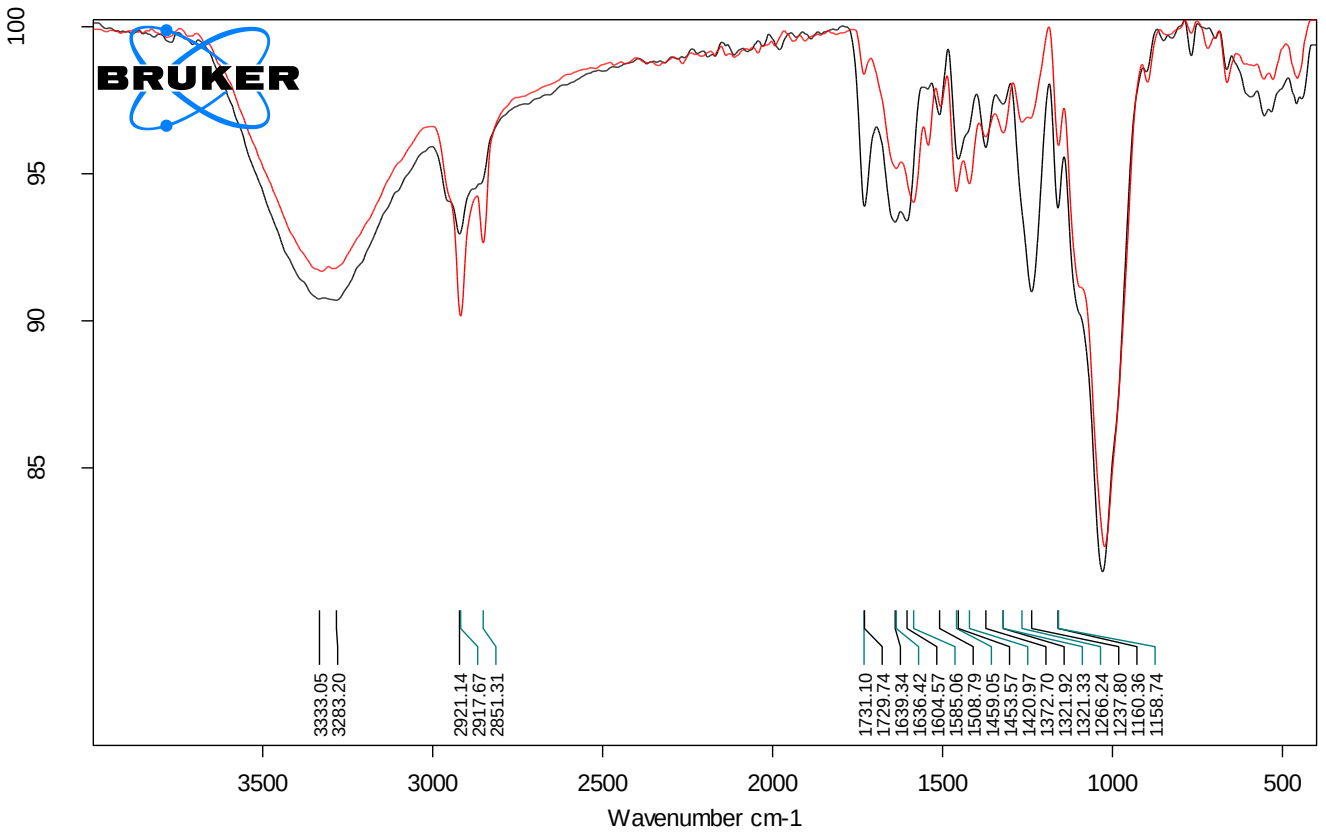

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Figure 7.1a: FTIR for Palmiste Rouge

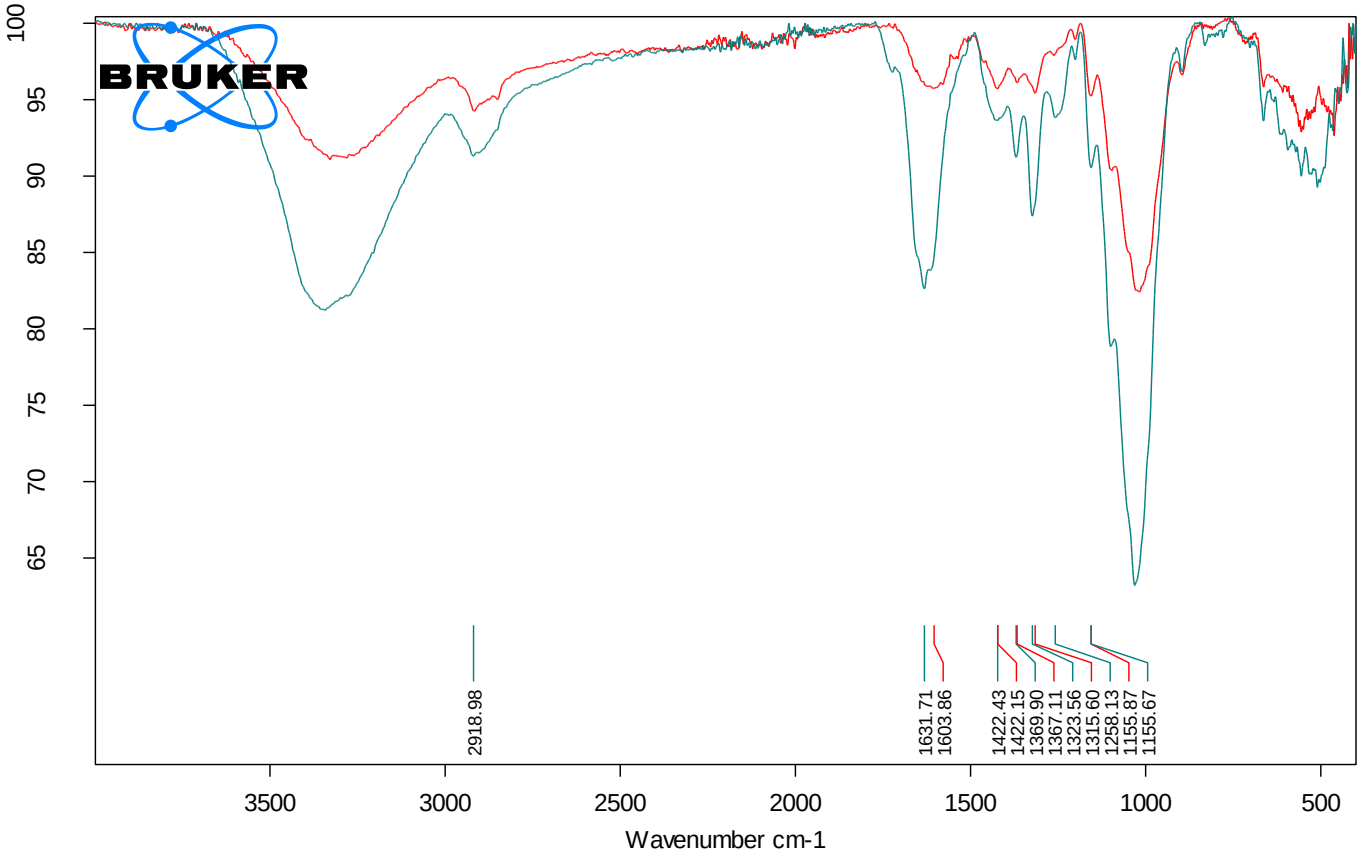

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Figure 7.1b: FTIR for Afromomum

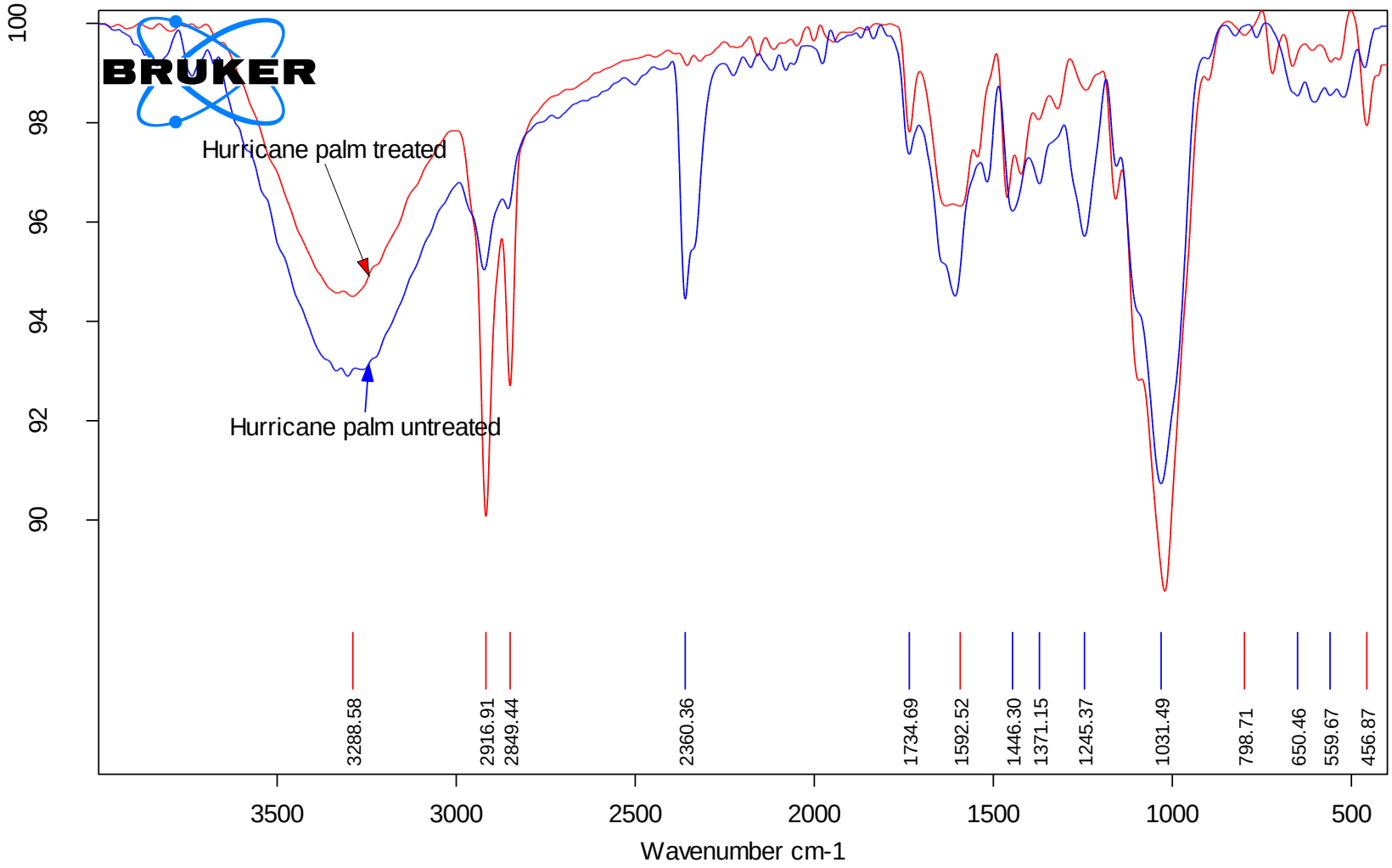

Figure 7.1c: FTIR for Hurricane Palm

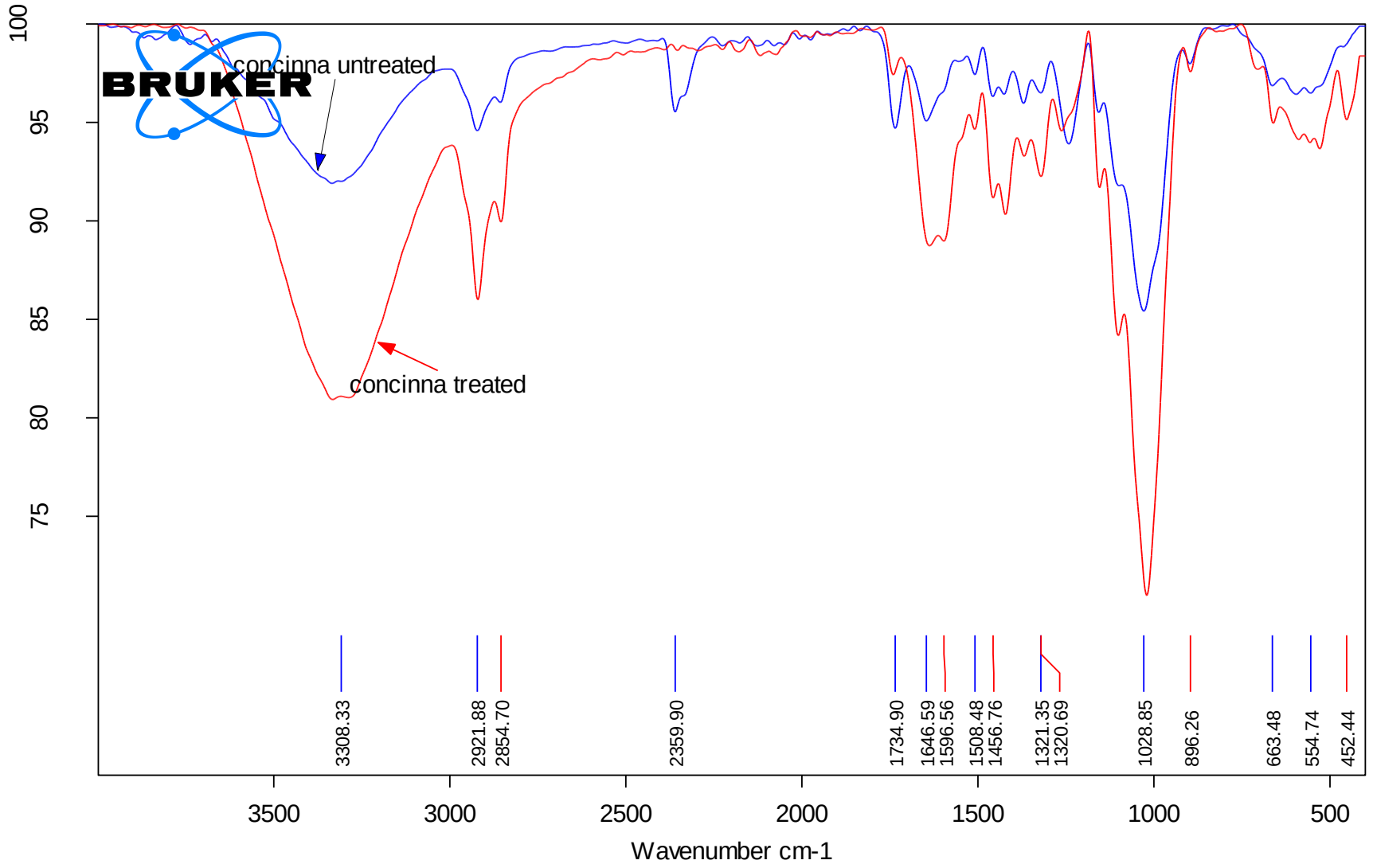

Figure 7.1d: FTIR for Dracaena concinna

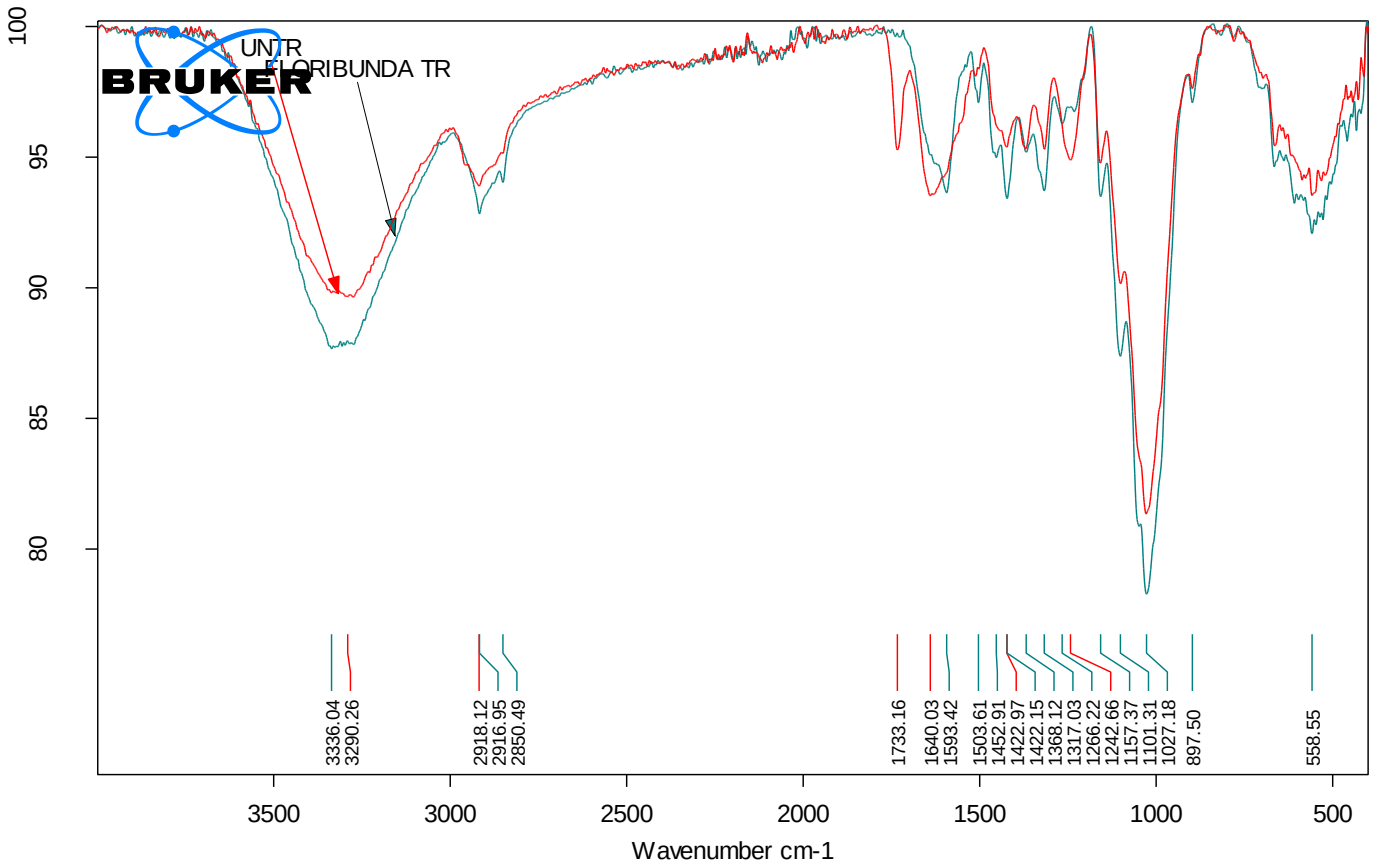

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Figure 7.1e: FTIR for Dracaena floribunda

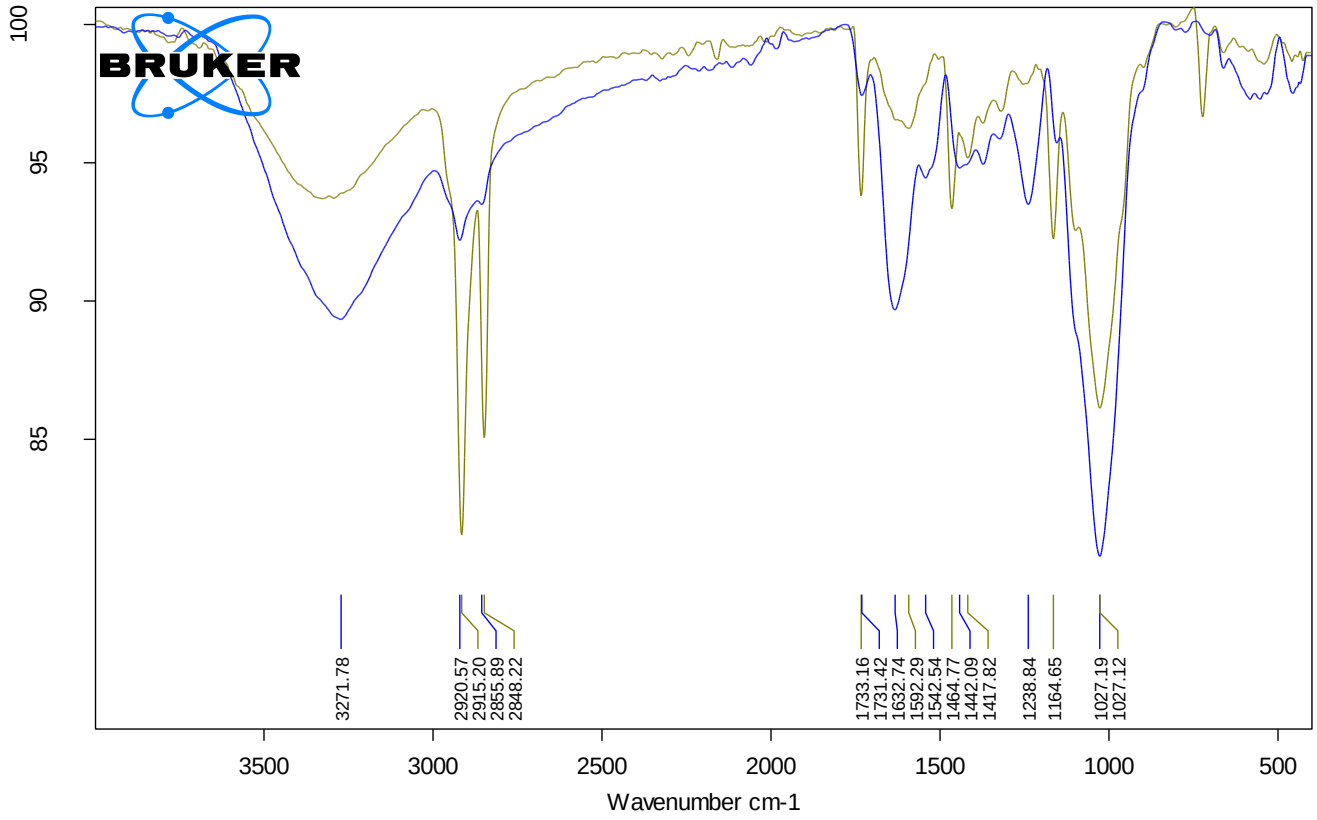

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Figure 7.1f: FTIR for Bottle Palm

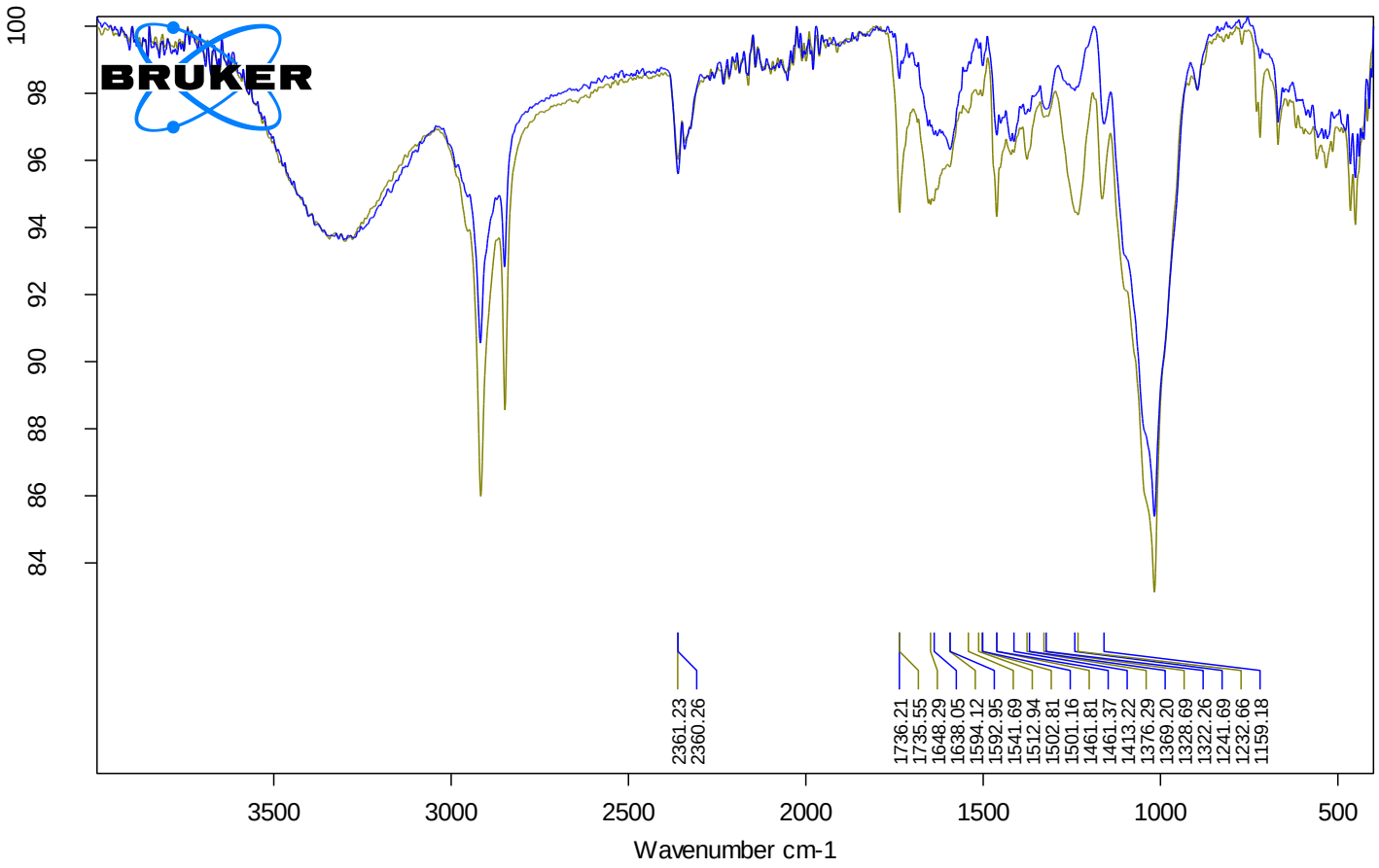

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Figure 7.1g: FTIR for Latania *loddigesii*

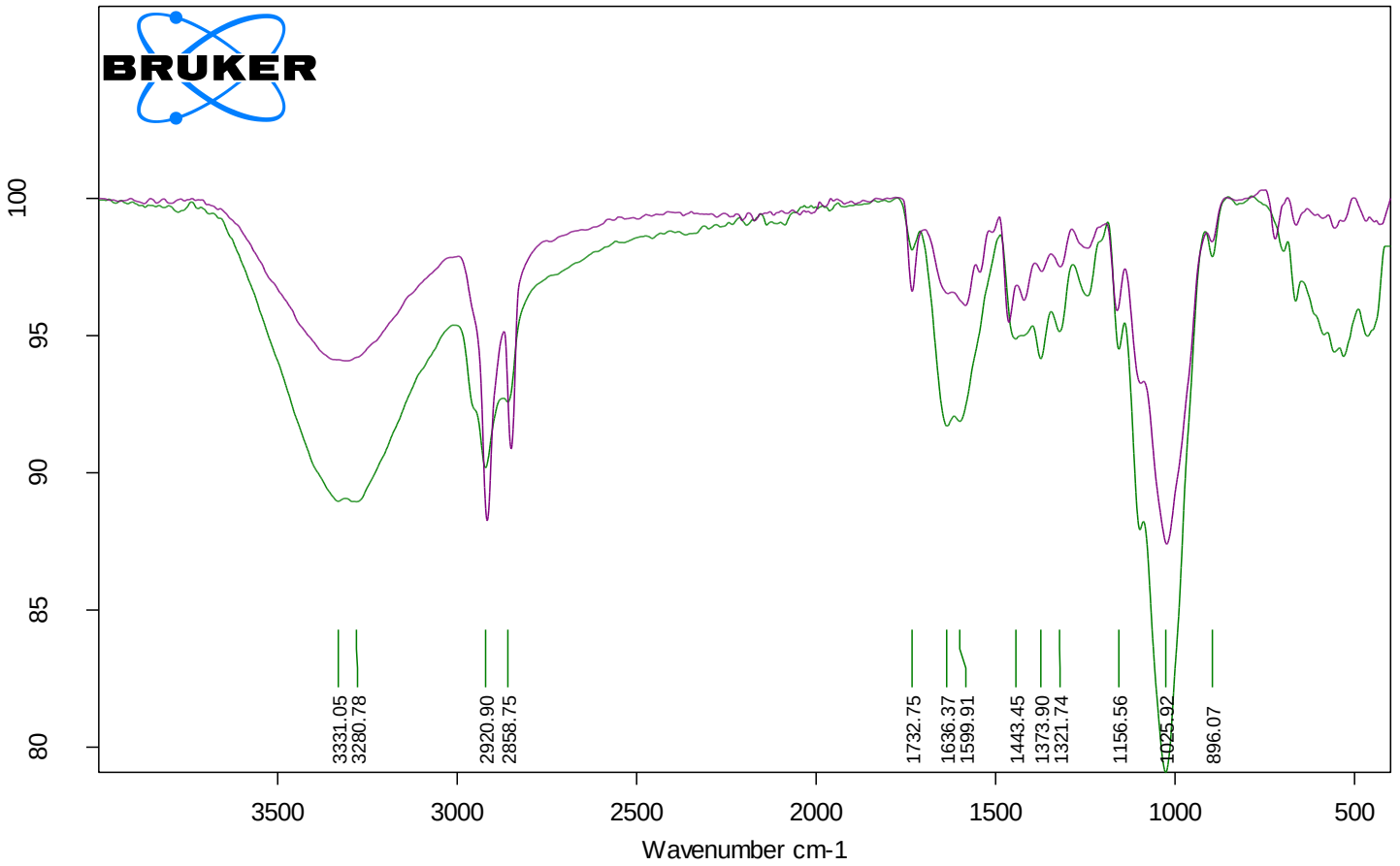

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Figure 7.1h: FTIR for Scleria *sieberi*

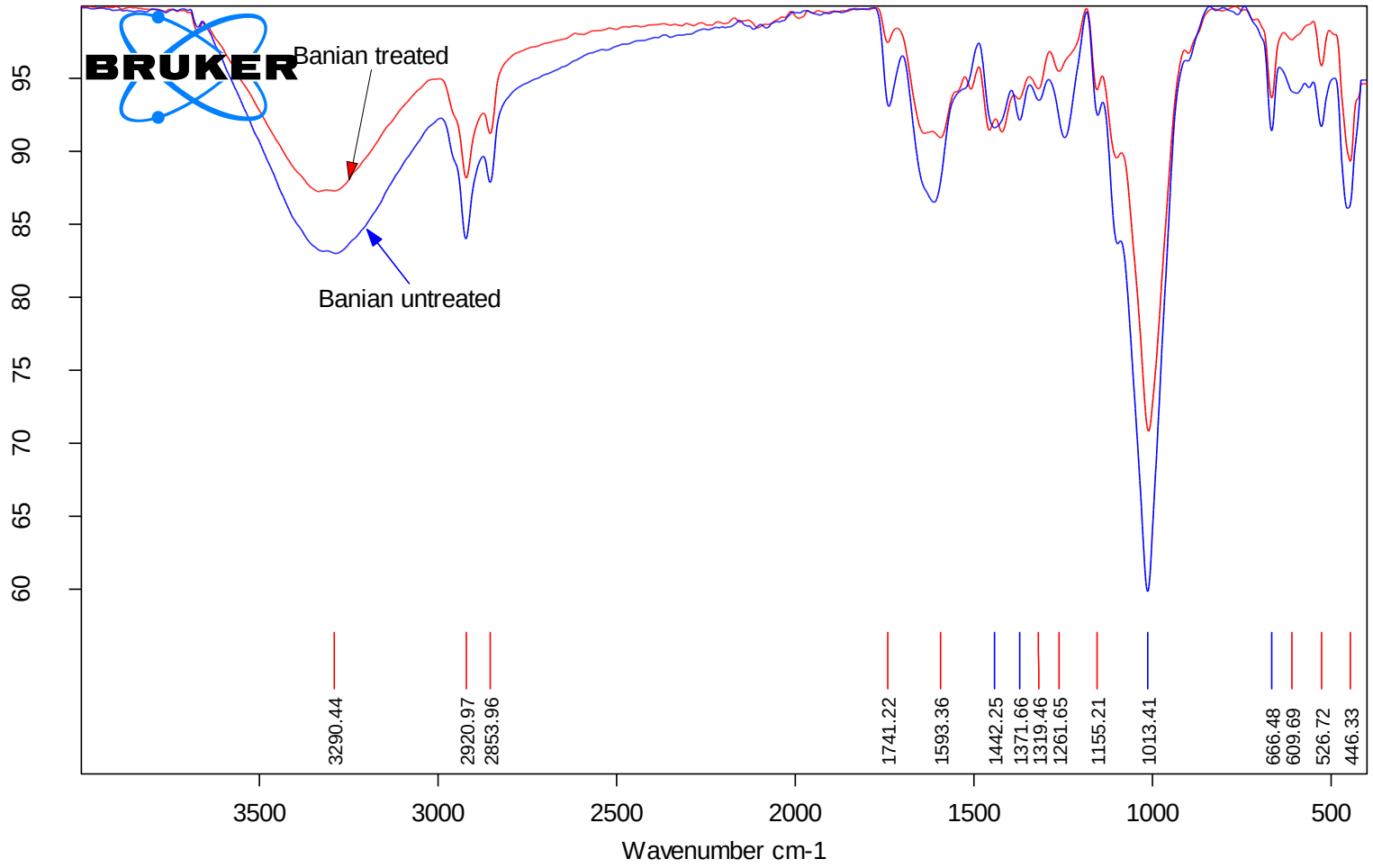

Figure 7.1i: FTIR for Banyan

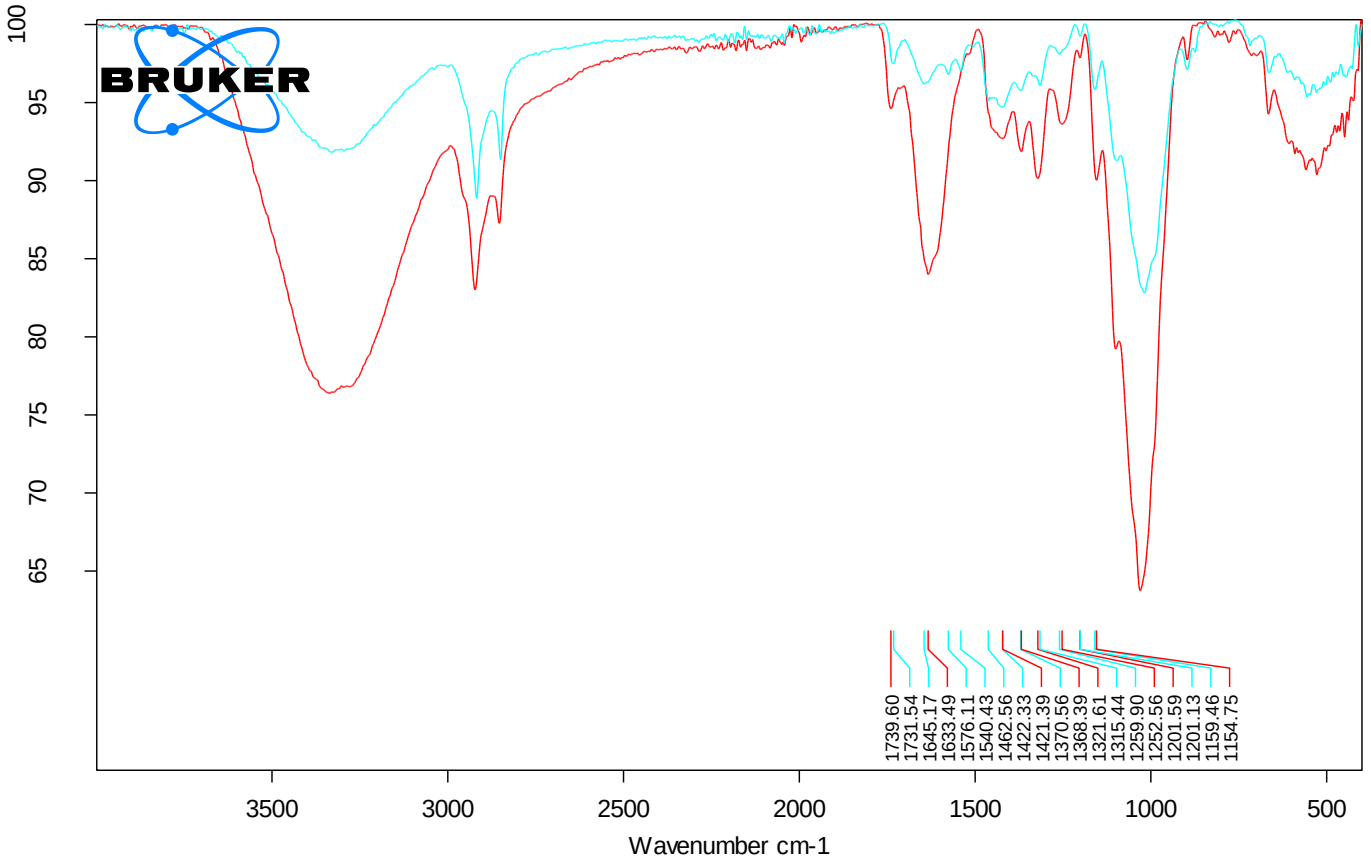

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Figure 7.1j: FTIR for Panicum *maximum*

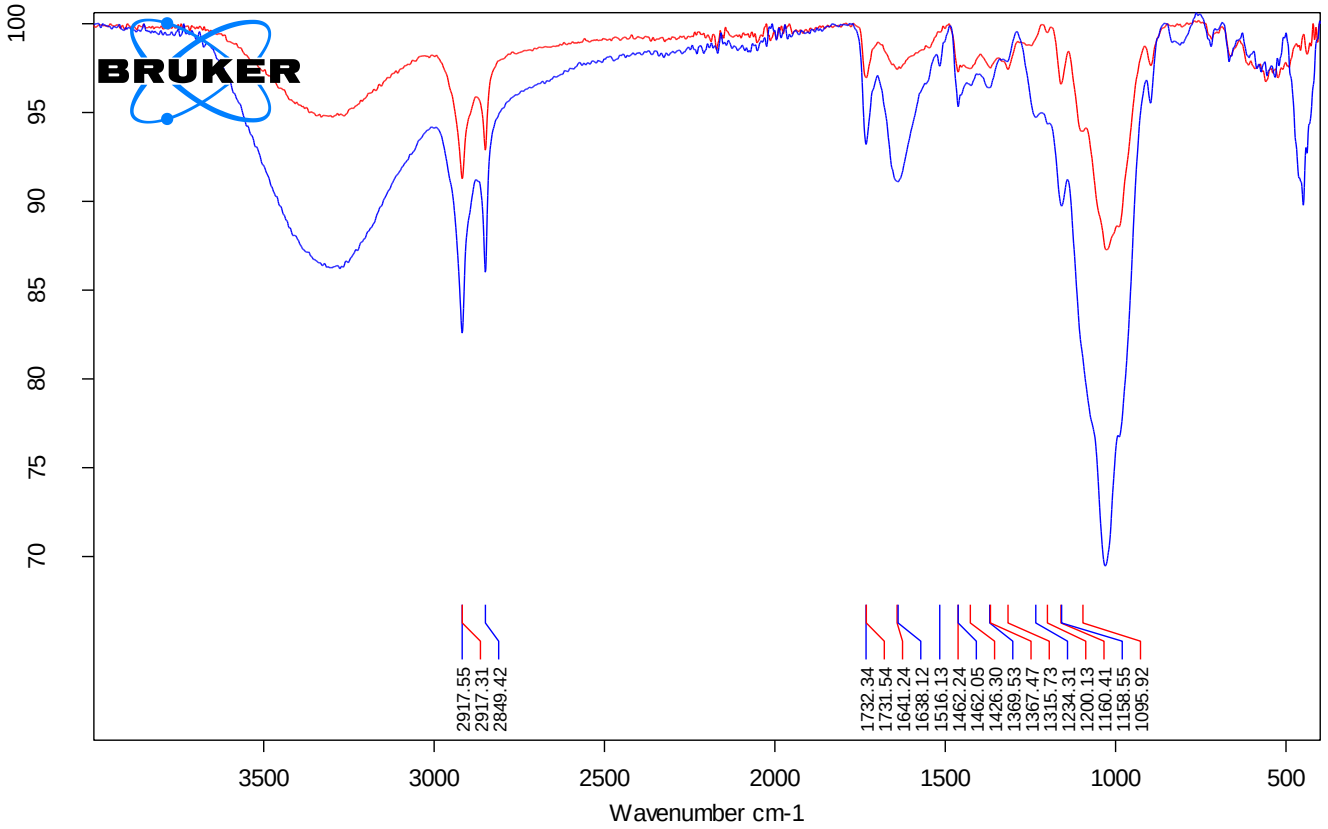

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Figure 7.1k: FTIR for *Thysanolaena latifolia* (Fatak Broom)

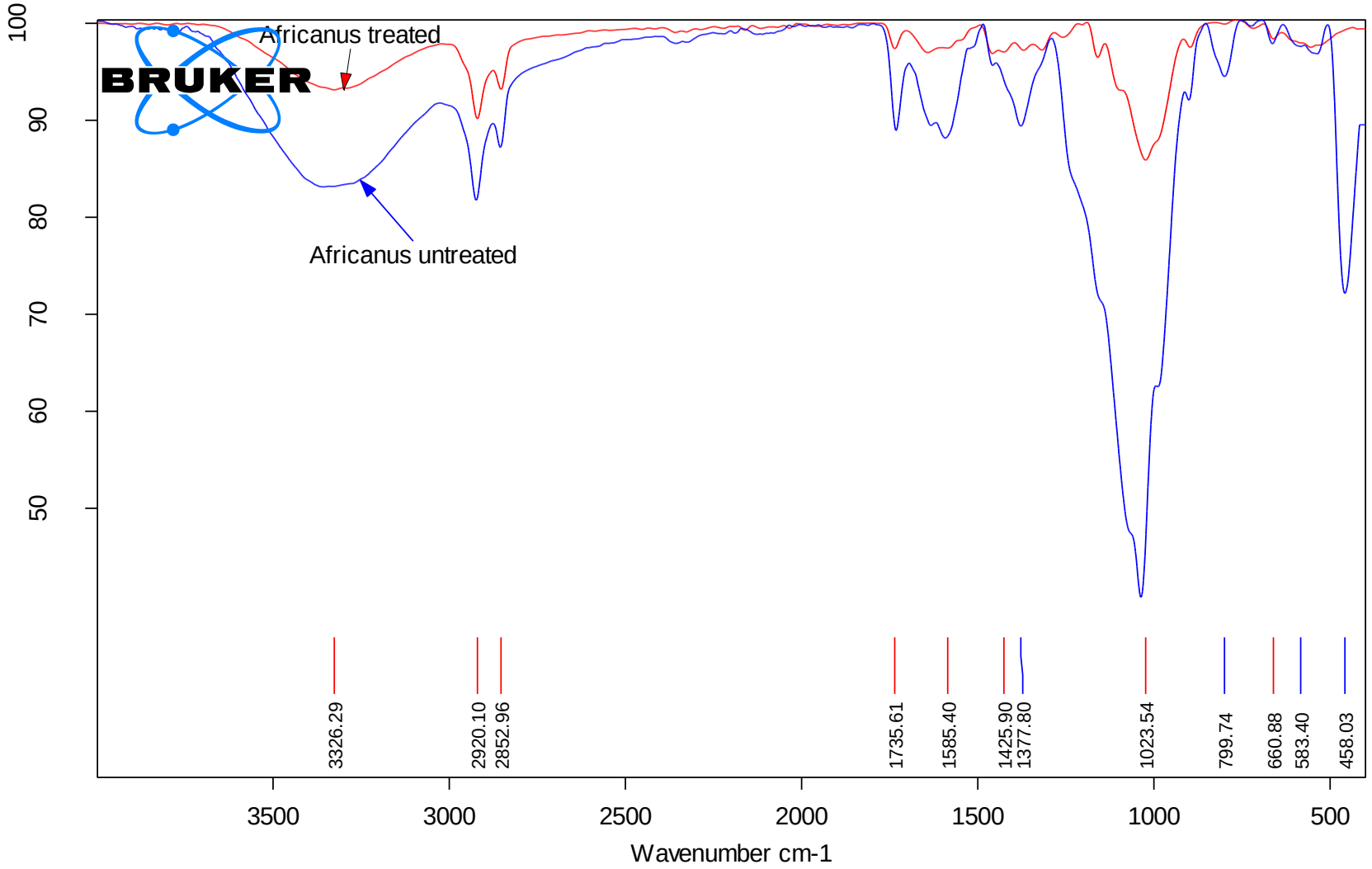

Figure 7.1l: FTIR for *Sporobolus africanus*

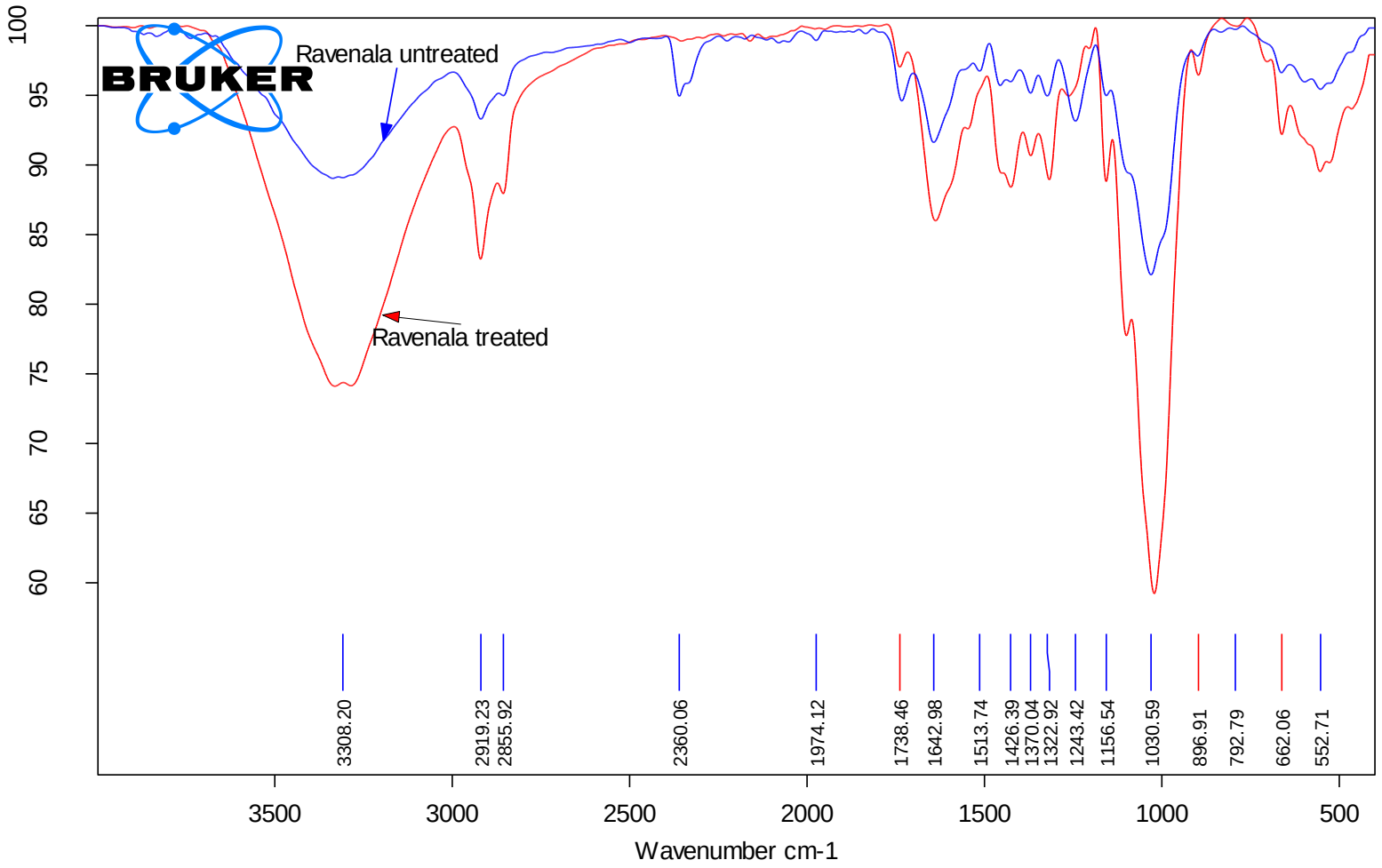

Figure 5.1m: FTIR for *Ravenala Madagascariensis*

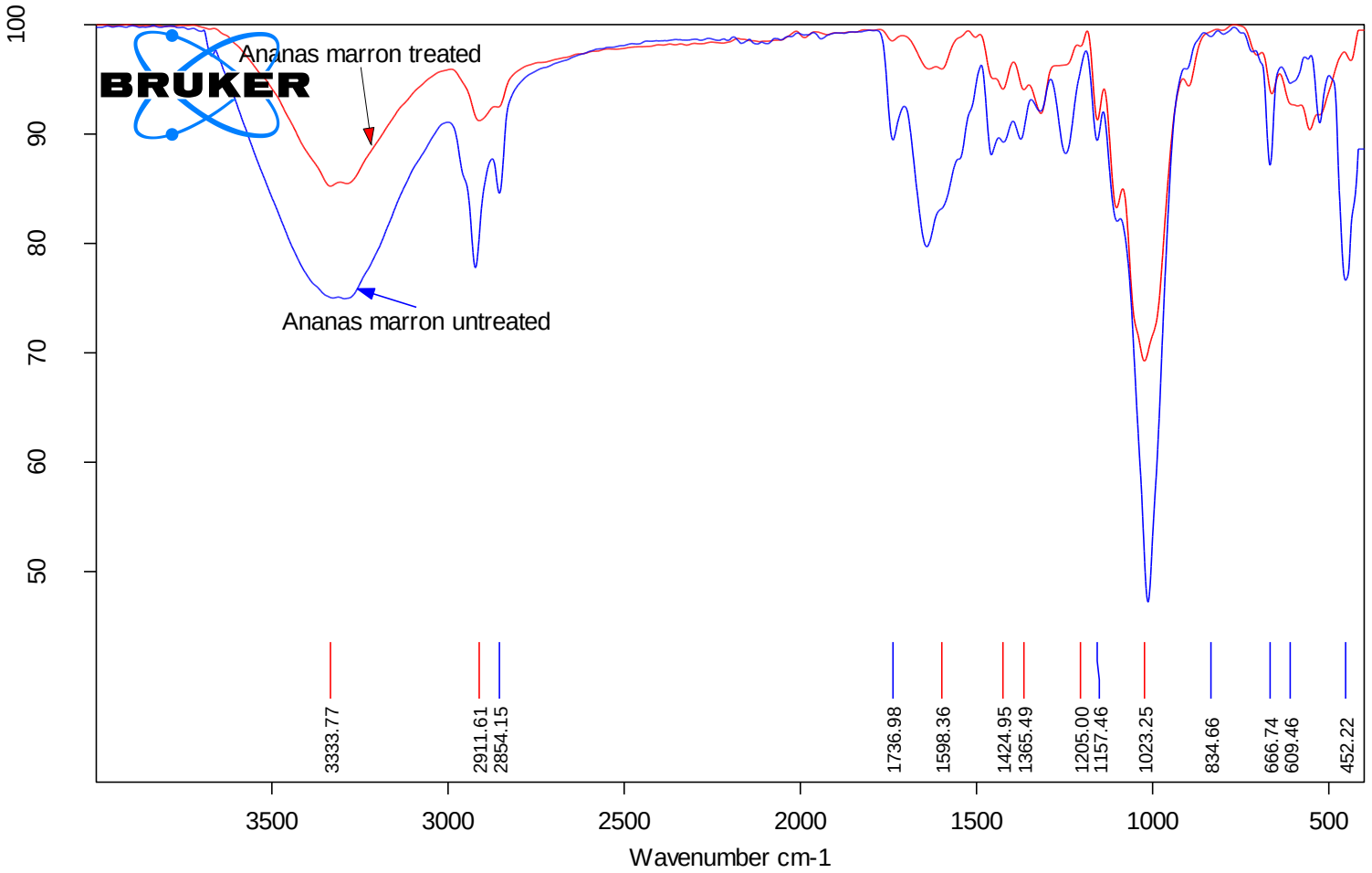

Figure 7.1n: FTIR for *Ananas bracteatus* (Ananas marron)

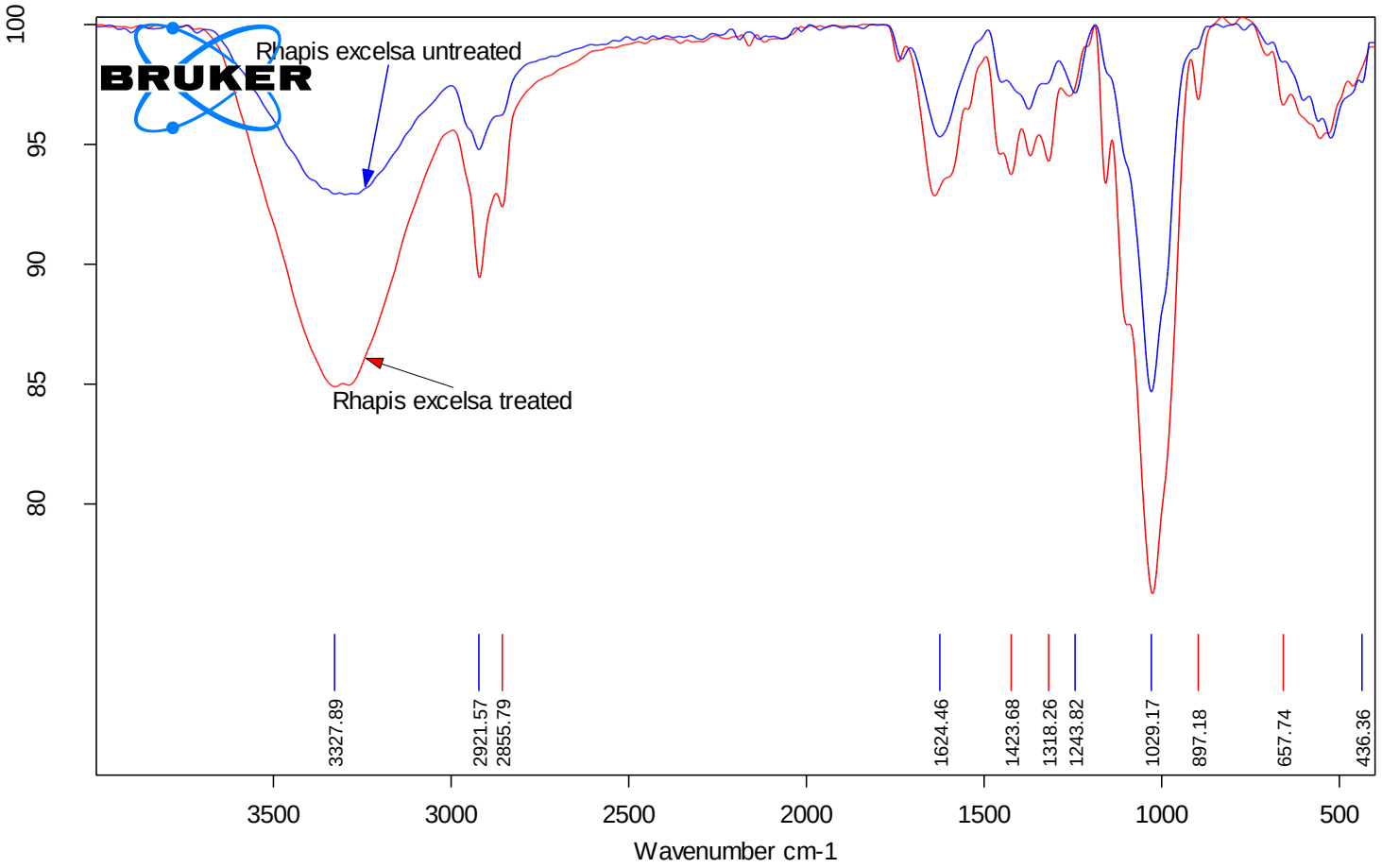

Figure 7.1o: FTIR for *Rhapis excelsa*

Table 7.2 shows a summary of the FTIR results for the alkaline treatment on the presence of different functional groups in the fifteen (15) different fibres. A comparison of the FTIR graphs for untreated and 5 % NaOH treatment for each fibre is shown in Figures 7.1a-o. The results show that in most cases, after the NaOH treatment, there is still the presence of hemicellulose, which is revealed by the presence of C=O bond in the region of 1730-1745 cm^{-1}.

The improvement of the tensile strength of all the fibres (except for Afromomum and Hurricane palm) and the FTIR results for these fibres is an indication that there has been partial removal of the hemicellulose. The peak at 1734 cm^{-1} for the Dracaena concinna treated fibres is smaller amplitude as compared to the untreated one (Figure 7.1d). This would tend to show that there has been significant removal of hemicellulose without damaging the fibre (due to fibre swelling).This is further explained by the fact that the tensile strength of the mercerized concinna fibre has increased by 42.4 % as compared to the untreated fibres. Figures 7.2a-b show the SEM micrographs of the untreated and treated concinna fibres. It can clearly be seen that there is removal of the waxy layer and partial removal of the outer layer revealing the internal cell wall of the fibre.

The peak at 1733 cm^{-1} for the untreated sample for the Dracaena floribunda is absent in the treated sample (Figure 7.1e). This could indicate the removal of hemicellulose by the 5 % NaOH for the floribunda fibres. Figures 7.3a-b show the SEM micrographs of the surface of the fibres before and after treatment with NaOH. It can be observed that the waxy layer has been removed after the alkaline treatment, and that there does not seem to be any removal of the outer layer.

The FTIR graphs for ravenala fibres (Figure 7.1m) revealed that there has been a reduction in the hemicellulose since the peak at 1738 cm^{-1} is shorter for the treated fibres as compared to

untreated fibres. This can explain the significant improvement (30.1 %) in the tensile strength of the treated fibres. Figures 7.4a-b show the SEM images of the surface morphology of the ravenala fibres before and after the alkaline treatment. It can be observed that there is a partial removal of the outer layer.

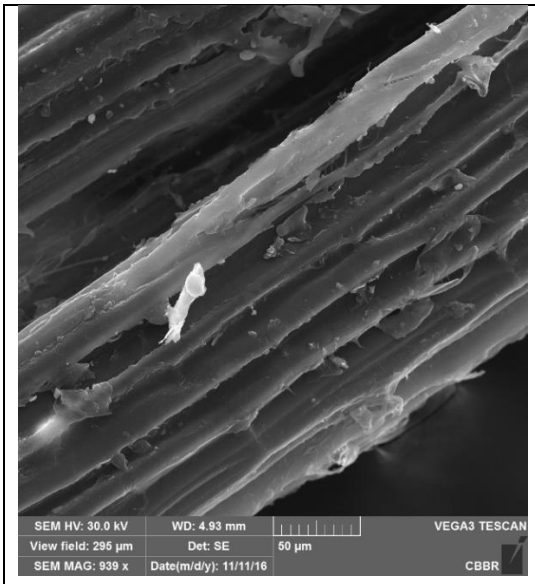

Figure 7.2a: Dracaena concinna untreated fibre at 939X

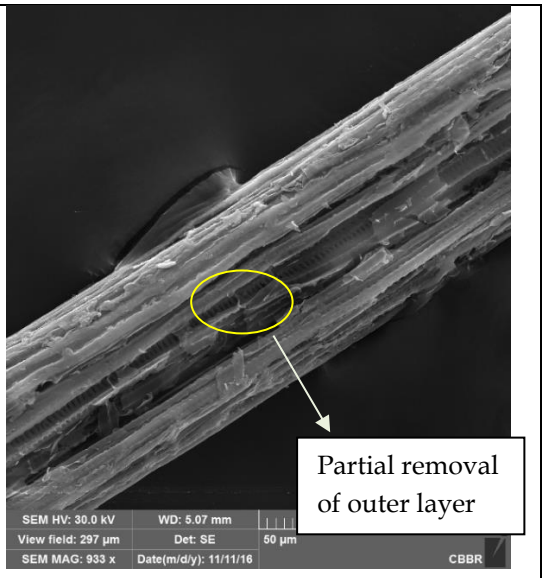

Figure 7.2b: Dracaena concinna treated fibre at 933X

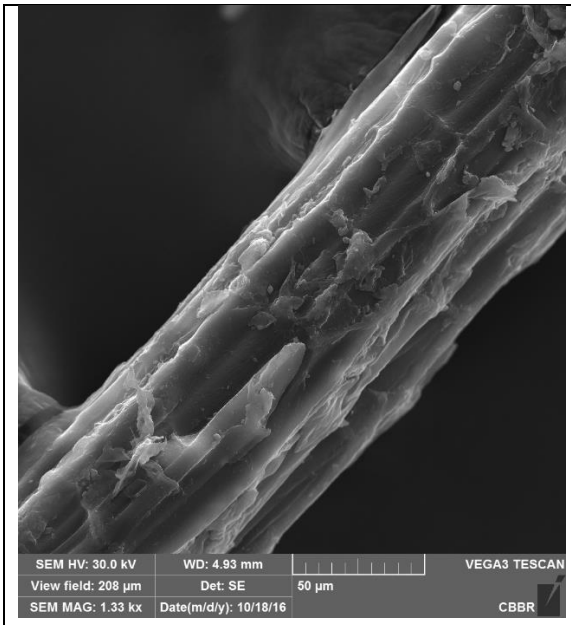

Figure 7.3a: Dracaena floribunda untreated fibre at 1330X

Figure 7.3b: Dracaena floribunda treated fibre at 1340X

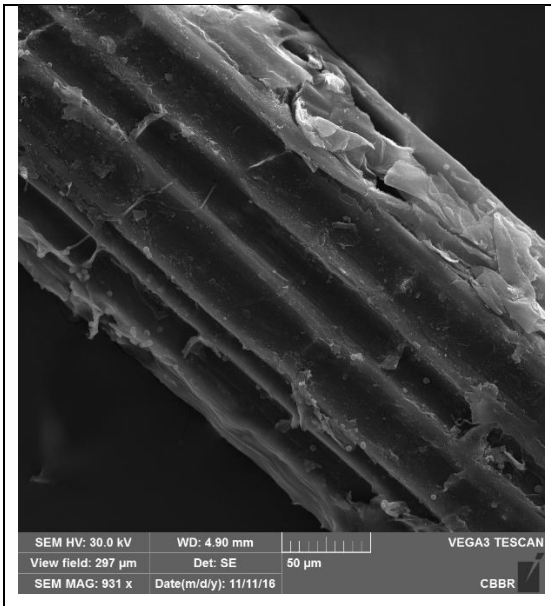

Figure 7.4a: Ravenala untreated fibre at 931X

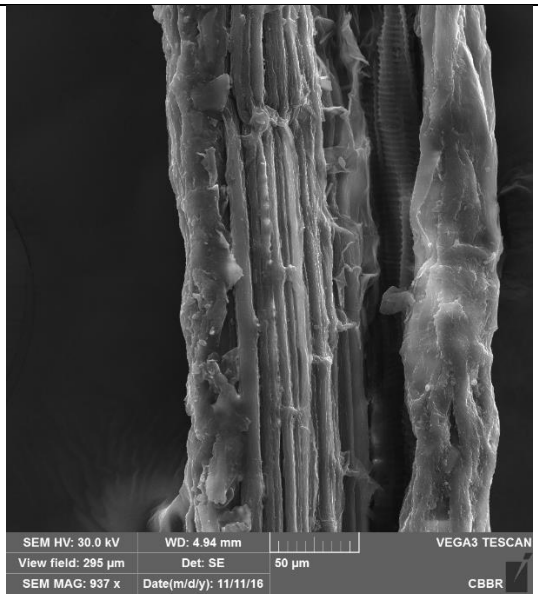

Figure 7.4b: Ravenala treated fibre at 937X

Figure 7.1f shows a significant increase in the amplitude of the 1730 cm^{-1} peak for the Bottle palm from untreated to treated condition. This is accompanied by a significant increase (92.2 %) of the tensile strength of the treated fibres. Figure 7.5b shows the surface of the treated fibre without the waxy layer as well as partial removal of the outer layer.

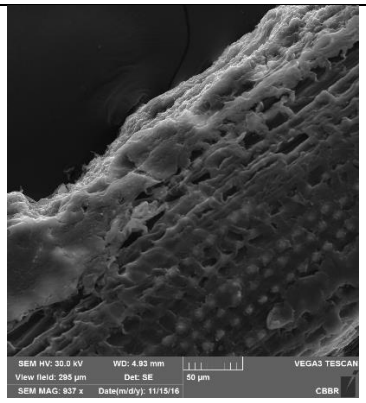

Figure 7.5a: Bottle Palm untreated fibre at 937X

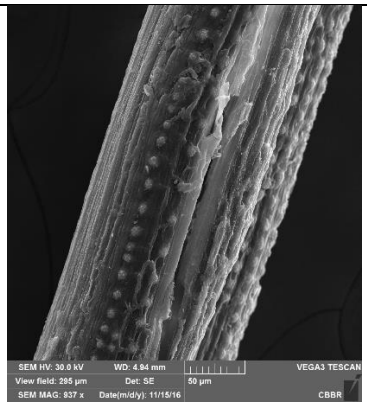

Figure 7.5b: Bottle Palm treated fibre at 937X

Figure 7.1g also shows a significant reduction in the amplitude of the 1736 cm^{-1} peak for the treated Latania fibres, which indicates that there has been partial removal of hemicellulose. This reduction in the non-cellulosic constituents in the fibres is reflected in an increase of about 61 % in the tensile strength. The SEM pictures as shown in Figures 7.6a-b show that there has been significant removal of the waxy outer layer of the fibre following the mercerization process with the 5 % NaOH.

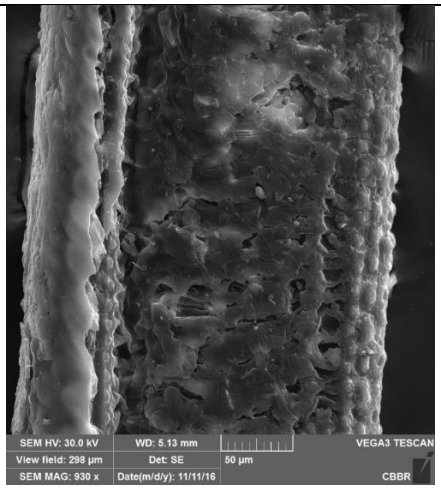

Figure 7.6a: Latania untreated fibre at 930X

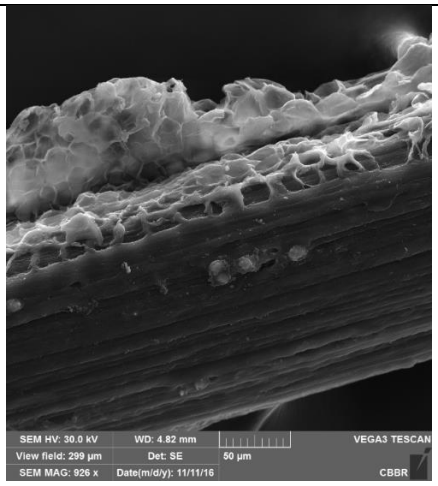

Figure 7.6b: Latania treated fibre at 926X

The peak at 1731 cm^{-1} for Palmiste Rouge (Figure 7.1a) for the treated fibre is of lower intensity as compared to the untreated one, indicating that there has been partial removal of the hemicellulose. The SEM images of Figures 7.7a-b show the removal of the waxy and outer layers after the alkaline treatment.

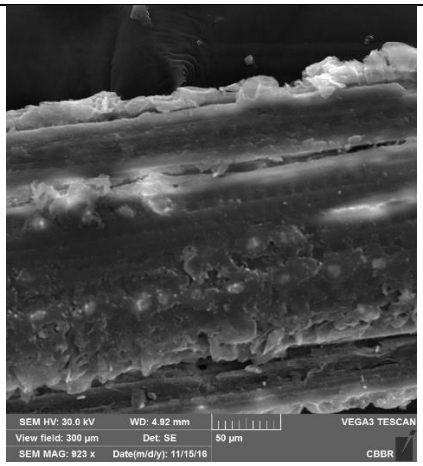

Figure 7.7a: Palmiste Rouge untreated fibre at 923X

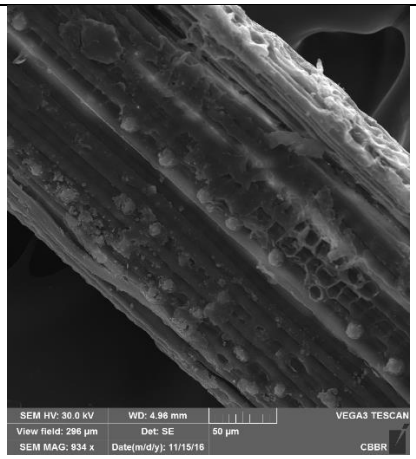

Figure 7.7b: Palmiste Rouge treated fibre at 934X

Figure 7.1k show that the peak at 1731 cm^{-1} is smaller in amplitude for the treated Latifolia (Fatak) fibre as compared to the untreated fibre. Again this indicates the partial removal of hemicellulose. Figures 7.8a-b show a significant removal of the waxy outer layer.

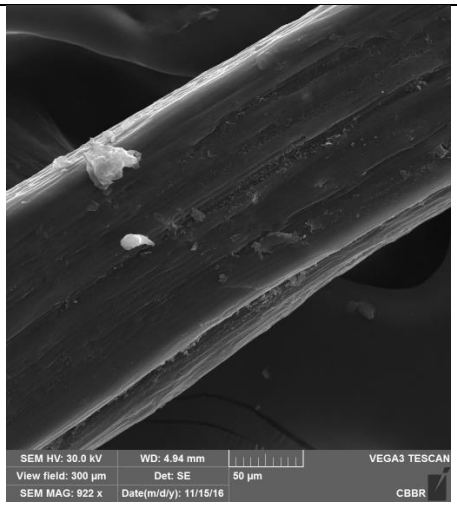

Figure 7.8a: Latifolia untreated fibre at 922X

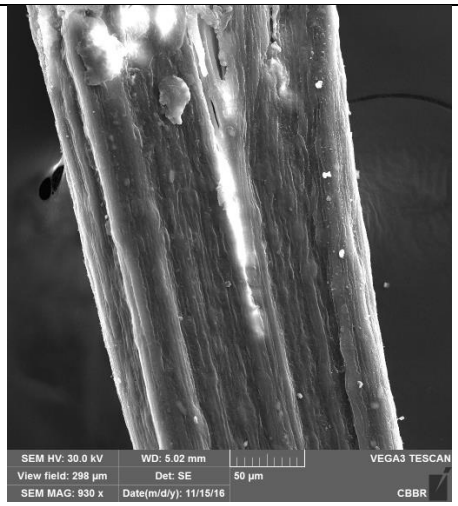

Figure 7.8b: Latifolia treated fibre at 930X

The FTIR graphs of Figure 7.1o for Rhapis excels (Zon) show that there is a peak at *around* 1602 cm^{-1}. The SEM images of Figures 7.9a-b show a significant removal of the outer layer revealing the internal fibre bundles.

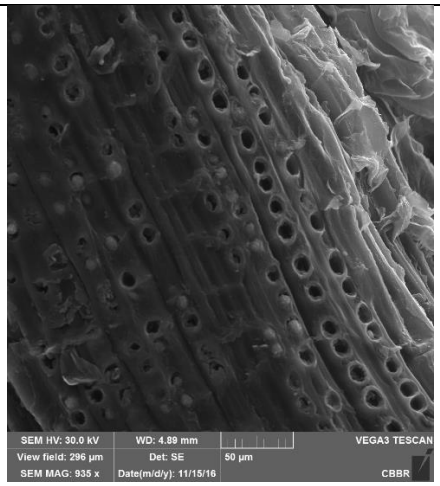

Figure 7.9a: Rhapis excelsa untreated fibre at 935X

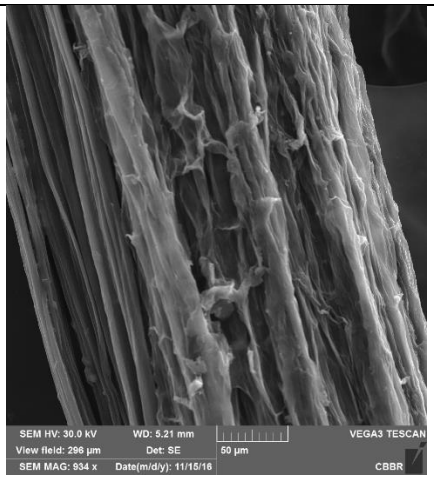

Figure 7.9b: Rhapis excelsa treated fibre at 934X

Figure 5.1j shows the FTIR graphs for Panicum maximum where the peak at about 1732 cm^{-1} for the treated fibre is smaller in amplitude as compared to the peak at 1739 cm^{-1} for the untreated fibre. Figures 5.10a-b shows that there is significant removal of the outer layer revealing the internal fibre bundles.

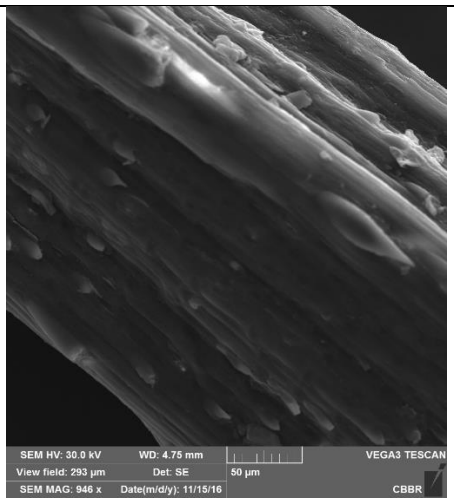

Figure 7.10a: Panicum maximum untreated fibre at 946X

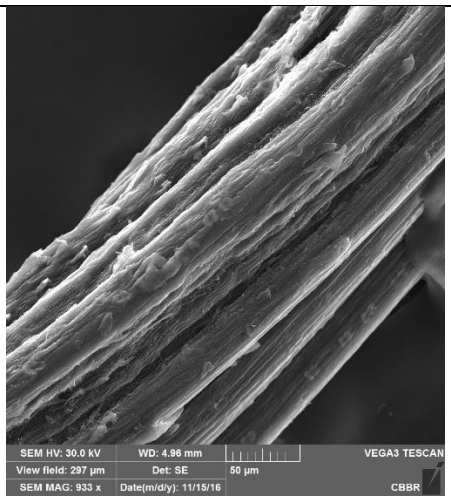

Figure 7.10b: Panicum maximum treated fibre at 933X

Similar to many of the previous fibres, figure 7.1n reveals that there is a significant reduction in the amplitude of the hemicellulose peak of 1736 cm^{-1} for Ananas Bractaetus. Figures 7.11a-b show the SEM images of the surface morphology.

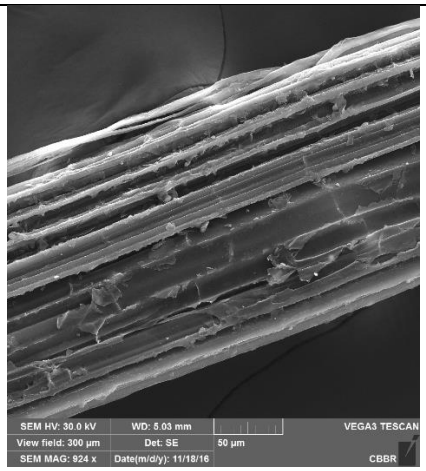

Figure 7.11a: Ananas Bractaetus untreated fibre at 924X

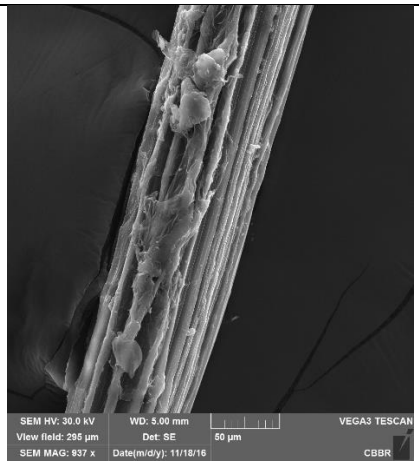

Figure 7.11b: Ananas Bractaetus treated fibre at 933X

For the scleria fibre Figure 7.1h reveals a larger amplitude peak at about 1732 cm^{-1} as compared to the untreated Scleria fibe. The SEM images of Figures 7.12a-b seem to reveal that there has been partial removal of the waxy outer layer.

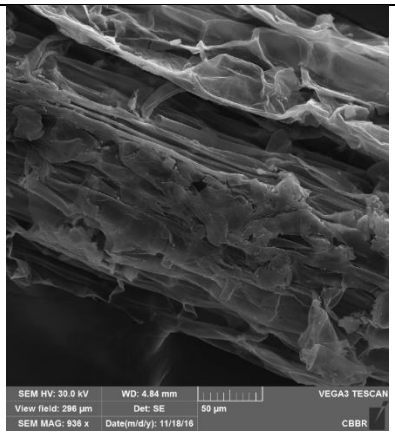

Figure 7.12a: Scleria untreated fibre at 936X

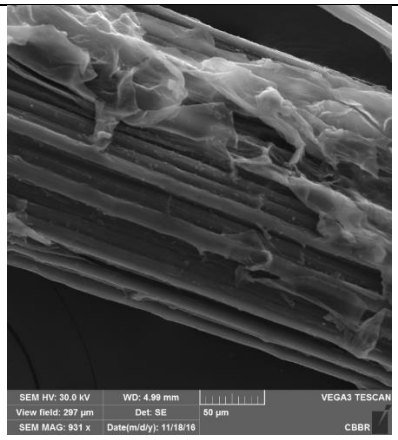

Figure 7.12b: Scleria treated fibre at 931X

For the Banyan fibre, there is also a reduction in the amplitude of the hemicellulose peak at 1741 cm^{-1} (Figure 7.1i). There is a removal of the outer layer under the action of the NaOH (Figures 7.13a-b).

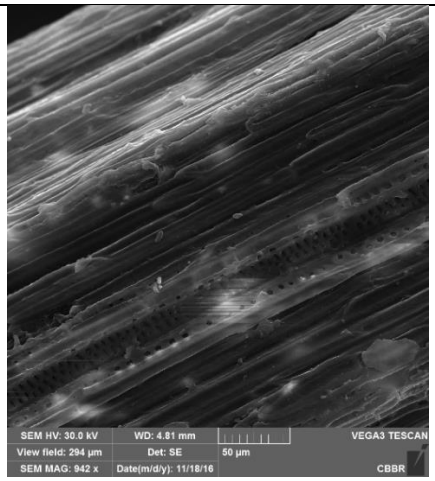

Figure 7.13a: Banyan untreated fibre at 942X

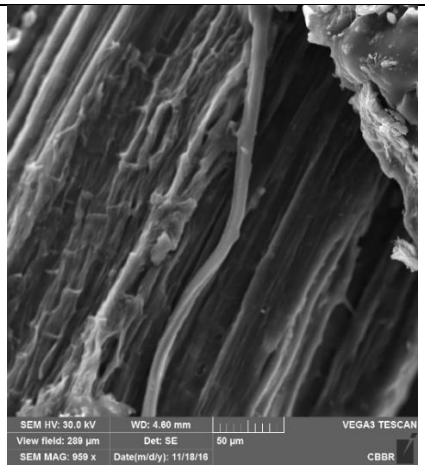

Figure 7.13b: Banyan treated fibre at 959X

Figure 7.1c reveals that there is a peak at 1734 cm^{-1} for both the untreated and treated Hurricane palm fibres. The SEM images of Figures 7.14a-b reveal the removal of the outer layer.

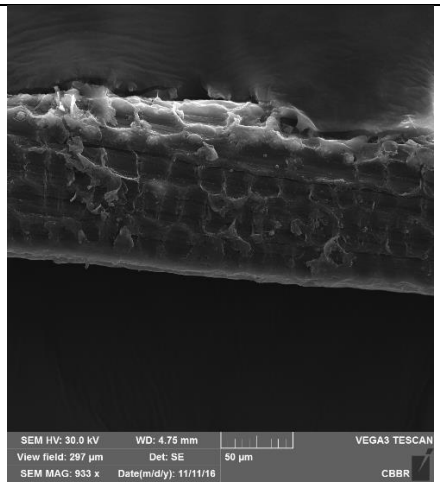

Figure 7.14a: Hurricane palm untreated fibre at 933X

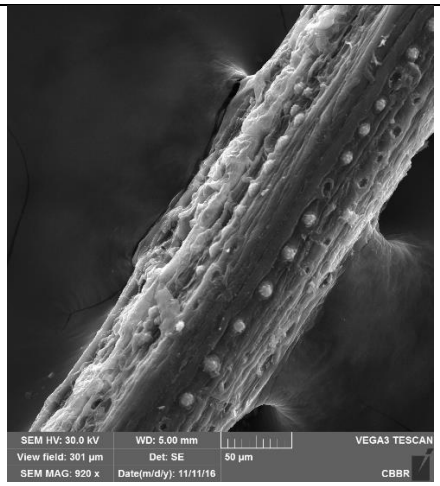

Figure 7.14b: Hurricane palm treated fibre at 920X

From Figure 7.1b, it can be noted that there is a peak at 1737 cm^{-1} for the untreated Afromomum fibre but no peak for the treated fibre. Figures 7.15a-b show that the waxy outer layer has been significantly removed from the surface of the fibre.

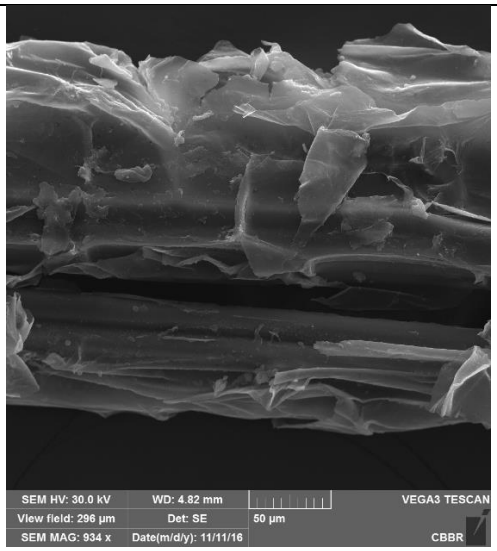

Figure 7.15a: Afromomum untreated fibre at 934X

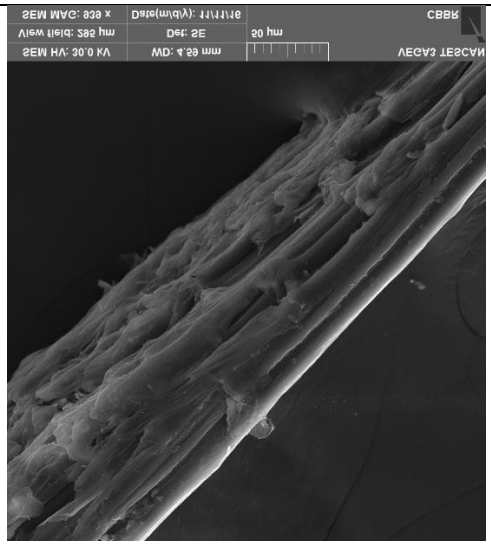

Figure 7.15b: Afromomum treated fibre at 939X

Figure 7.1l reveals a peak of smaller amplitude at 1735 cm^{-1} for the treated Sporobolus Africanus fibre as compared to the untreated one. This is a similar phenomenon as for Banyan and other fibres. Figures 7.16a-b show the removal of the outer layer revealing the internal fibre bundle.

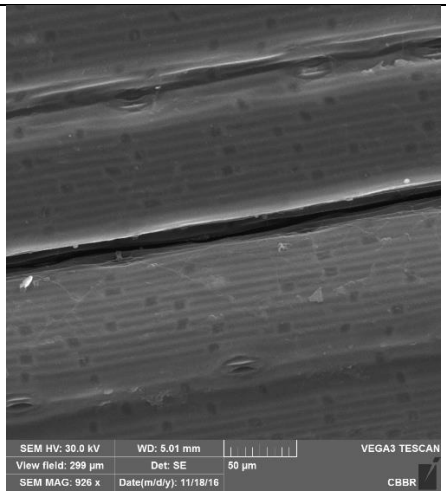

Figure 7.16a: Sporobolus Africanus untreated fibre at 926X

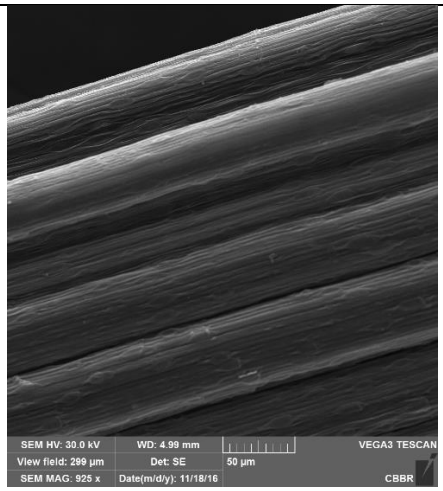

Figure 7.16b: Sporobolus Africanus treated fibre at 925X

Table 7.2: Summary of bands obtained for the different chemical treatments

	Condition: Untreated and 5 % NaOH Treatment	**Presence of C=O bond; Hemicellulose & Pectin** (~ 1730 **cm^{-1}**)	**Presence of lignin** (C=C bond) (~ 1602 **cm^{-1}**)	**Presence of lignin** (C=C bond) (~ 1505 **cm^{-1}**)	**Presence of lignin** (C-O bond) (~ 1239-1245 **cm^{-1}**)	**OH Bending of the adsorbed water** (~ 1649-1641 **cm^{-1}**)
Palmiste rouge	Untreated	1733	1604	1507	1242	1636
	Treated	1735	1591	1504	1244	1636
Afromomum	Untreated	1737	-	-	1249	1631
	Treated	1742	1604	1540	1262	1645
Ravenala	Untreated	1733	-	1508	1242	1647
	Treated	1739	-	1542	1262	1634

Table 7.2: Summary of bands obtained for the different chemical treatments (*continued*)

	Condition: Untreated and 5 % NaOH Treatment	**Presence of C=O bond; Hemicellulose & Pectin** (~ 1730 **cm^{-1}**)	**Presence of lignin** (C=C bond) (~ 1602 **cm^{-1}**)	**Presence of lignin** (C=C bond) (~ 1505 **cm^{-1}**)	**Presence of lignin** (C-O bond) (~ 1239-1245 **cm^{-1}**)	**OH Bending of the adsorbed water** (~ 1649-1641 **cm^{-1}**)
Hurricane Palm	Untreated	1733	1616	1508	1249	1653
	Treated	1736	1597	1518	1235	-
Dracaena Concinna	Untreated	1733	1595	1507	1239	1647
	Treated	1741	1595	1508	1262	1640
Dracaena Floribunda	Untreated	1733	1618	1508	1246	1652
	Treated	-	1592	1508	-	1648
Bottle Palm	Untreated	1736	1588	1529	1253	1638/1655
	Treated	1737	1593	1505	1246	1644

Table 7.2: Summary of bands obtained for the different chemical treatments (*continued*)

	Condition: Untreated and 5 % NaOH Treatment	**Presence of C=O bond; Hemicellulose & Pectin** (~ 1730 **cm^{-1}**)	**Presence of lignin** (C=C bond) (~ 1602 **cm^{-1}**)	**Presence of lignin** (C=C bond) (~ 1505 **cm^{-1}**)	**Presence of lignin** (C-O bond) (~ 1239-1245 **cm^{-1}**)	**OH Bending of the adsorbed water** (~ 1649-1641 **cm^{-1}**)
Latania	Untreated	1735	1594	1502	1232	1648
	Treated	1736	1592	1501	1241	1638
Scleria	Untreated	1738	1605	1501	1246	1629
	Treated	1736	-	1542	1248	1639
Banian	Untreated	1737	-	1550	1249	1639
	Treated	1736	-	1577	1248	1638
Latifolia (Fatak)	Untreated	1732	1516	-	1234	1638
	Treated	1732	-	1501	1247	1631/1641

Table 7.2: Summary of bands obtained for the different chemical treatments (*continued*)

	Condition: Untreated and 5 % NaOH Treatment	**Presence of C=O bond; Hemicellulose & Pectin** (~ 1730 **cm^{-1}**)	**Presence of lignin** (C=C bond) (~ 1602 **cm^{-1}**)	**Presence of lignin** (C=C bond) (~ 1505 **cm^{-1}**)	**Presence of lignin** (C-O bond) (~ 1239-1245 **cm^{-1}**)	**OH Bending of the adsorbed water** (~ 1649-1641 **cm^{-1}**)
Africanus	Untreated	1734	1601	1509	-	1655
	Treated	1736	1583	1547	1259	1642
Rhapis excels	Untreated	1731	-	-	1244	1633
	Treated	1765	-	1503	1275	1632
Ananas Marron	Untreated	1738	-	1543	1246	1642
	Treated	1741	1594	1550	1261	1639
Panicum	Untreated	1739	1633	1516	1251	1633
	Treated	1731	-	1543/1578	1202/1259	1641

7.2 Analysis of the peak area for hemicellulose

In order to better understand the change in the hemicellulose peak at around 1730 cm^{-1}, an analysis of the peak area at this wavenumber has been conducted for selected species. Table 7.3 shows the results of the peak area for the untreated and treated fibre for each plant species.

Table 7.3

	Peak area at ~1730 cm^{-1}	
Plant specie	**Untreated fibre**	**Treated fibre**
Ravenala	1.149	0.490
Dracaena concinna	1.093	0.323
Dracaena floribunda	0.768	0 (No peak)
Latania	0.907	0.161
Latifolia	1.171	0.467
Afromomum	0	0
Hurricane palm	0.454	0.345
Scleria sieberi	0.296	0.455
Bottle palm	0.519	0.741
Palmiste Rouge	1.241	0.204
Banyan	1.347	0.374

The results have shown that there is a general decrease, more than 50 %, in the hemicellulose peak area after the chemical treatment with NaOH, except for Scleria sieberi and Bottle palm. For Afromomum no proper peak could be observed in both the untreated and treated samples. The decrease in the peak area confirms the observation of the FTIR graphs that there is a reduction of the amplitude of the peak at about 1730 cm^{-1} when the sample is treated with NaOH solution. Thus it can be deduced that for most of the treated samples (as mentioned above), the alkaline treatment is effective in significantly removing the hemicellulose content. However, the optimum alkaline treatment to remove the hemicellulose whilst maximising the tensile strength for each specie still remains to be determined.

A reduction of the amplitude of the peak at about 1730 cm^{-1} is noted when the sample is treated with NaOH solution.

Figure 7.2 shows the FTIR of five different fibres which could not be chemically treated.

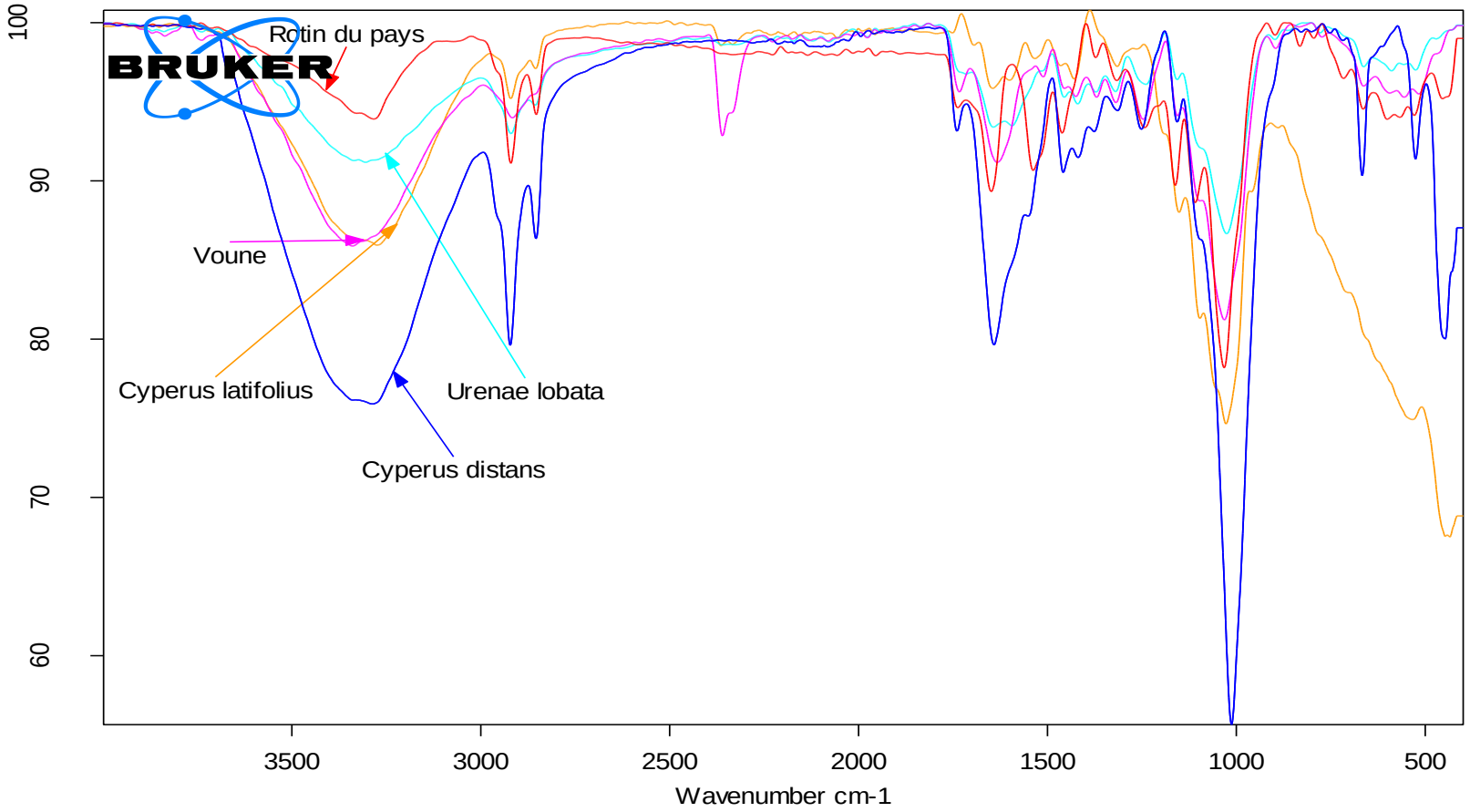

Figure 7.2: FTIR for untreated fibres

7.3 References

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Sun, J. X., Sun, X. F., Zhao, H., & Sun, R. C. (2004). Isolation and characterization of cellulose from sugarcane bagasse. *Polymer Degradation and Stability*, *84*(2), 331-339.

CHAPTER 8: THERMAL ANALYSIS

8.1 Palmiste rouge

8.1.1 DSC analysis

Figure 1 of Appendix 1 shows the result of the DSC analysis for the untreated and treated Palmiste rouge fibres. When comparing the DSC graph of untreated fibre with that of treated fibre, it can be seen that there is a significant shift in the first endothermic peak from 66.8 °C to 84.7 °C. This first peak is associated with the loss of water due to evaporation. However, after the alkaline treatment, if there has been partial removal of hemicellulose, then the cellulose might have adsorbed moisture. Thus a higher temperature is required to remove this adsorbed water, and this explains the higher temperature of the first peak for the treated fibre (84.7 °C). Furthermore, the second peak of the untreated fibre, which occurred at 280.9 °C is flatter in the treated sample, and the temperature of the second peak is now 293 °C. This second peak seems to represent the presence of hemicellulose which has been partly removed by the alkaline treatment, and this explains why the second peak is flatter in the treated sample. Finally the third peak is shifted from 346.6 °C for the untreated sample to 351.3 °C for the treated sample. This can be explained by the fact the removal of the hemicellulose has caused a rearrangement and reorientation of the cellulose crystals, and this yield a more compact crystal structure (Mandal and Chakrabarty, 2011).

8.2 Afromomum Fibre

8.2.1 DSC analysis

The first peak of the treated sample has a much lower temperature (69.2 °C) as compared to the untreated sample (94.6 °C). This observation is completely different from the Palmiste Rouge fibres. Moreover, the second peak of the untreated fibre is at 167.1 °C but there is no corresponding peak for the treated sample. Finally the third peak of the untreated fibre occurred at 333.2 °C but that from the treated fibre is at 332.5 °C and much flatter. The absence of a corresponding second peak for the treated fibre might well indicate the complete removal of hemicellulose, and the flatter peak at 332.5 °C might indicate a lesser amorphous cellulosic content. Thus the alkaline treatment could well have transformed part of the amorphous cellulose into a more crystalline structure.

8.2.2 TGA analysis

Figure 1 of Appendix 2 shows that the total mass loss during the degradation stage is 81.75 % and the temperature of the onset degradation is 264.9 °C for the untreated sample. On the other hand for the treated sample (Figure 2 Appendix 2) shows that the mass loss for the main degradation phase is 73.57 %, and the temperature of the onset of degradation is 305.3 °C. The much higher onset degradation temperature might well confirm the removal of hemicellulose, which thus cause a rearrangement and reorientation of the cellulose crystals. The higher mass loss for the untreated sample leads to a smaller mass of residue, 1.69 % as compared to 12.9 % for the treated sample. This higher residual mass for the alkaline treated sample can be explained by the presence of a greater amount of crystalline cellulose I having parallel orientation whereas the untreated sample had a higher percentage of the less crystalline cellulose II (having antiparallel orientation) and amorphous cellulose (Mandal and Chakrabarty, 2011).

8.3 Hurricane palm fibre

8.3.1 DSC analysis

The DSC graph for the treated sample (Figure 3) showed the same trend as the afromomum fibres for the first endothermic peak since the peak occurred at 90.1 °C whereas the first peak occurred at 93.5 °C for the untreated sample. The second peak, which can be attributed to the presence of hemicellulose, is observed in both graphs showing that the alkaline treatment has not effectively removed the non-cellulosic content. The slight shift in the second peak (from 285.7 °C for untreated and 287 °C for treated) could possibly be due to a small removal of hemicellulose. Furthermore, the third peak, which can be attributed to the amorphous cellulose has shifted from 347.3 °C to 355.2 °C. This shift could be due to a small rearrangement of the amorphous cellulose due to the partial removal of hemicellulose.

8.3.2 TGA analysis

Figures 3 & 4 of Appendix 2 shows that the temperature of the onset of degradation for the untreated sample is higher (294.4 °C) than that for the treated sample (280.1 °C), and the mass loss during the degradation stage is also lower for the untreated sample (75.82 %) as compared to the treated sample (82.12 %). The amount of residue is thus more for the untreated sample (12.37 %) than the treated sample (3.16 %). This could possibly be due to the presence of a higher percentage of crystalline cellulose I in the untreated sample. The alkaline treatment could possibly have degraded the cellulose I which can explain the lower temperature of the onset of degradation as well as a lower residual mass.

8.4 Dracaena concinna

8.4.1 DSC analysis

Figure 4 of Appendix 1 show that the first endothermic peak for both untreated and treated samples occurred almost at the same temperature (84.2 °C for untreated and 83.8 °C for treated sample). However, the second peak which occurred at 288.4 °C for the untreated sample is not present for the treated sample. On the other hand the third peak occurring at 334.3 °C for the untreated sample also occurred for the treated sample at 332.2 °C.

8.4.2 TGA analysis

Figures 5 & 6 of Appendix 2 shows that the temperature of the onset of degradation for the untreated sample is higher (286.9 °C) than that for the treated sample (279.6 °C), and the mass loss during the degradation stage is higher for the untreated sample (76.01 %) as compared to the treated sample (71.43 %). The amount of residue is thus more for the treated sample (14.26 %) than the untreated sample (10.51 %).

Based on the FTIR, DSC and TGA results, there is sufficient indication that there is partial removal of hemicellulose, and possibly the presence of higher percentage of crystalline cellulose I in the treated fibre as compared to the untreated fibre.

8.5 Dracaena floribunda

8.5.1 DSC analysis

Figure 5 of Appendix 1 show that the first peak for the treated fibre is at a higher temperature of 73 °C as compared to the untreated sample, 69.8 °C. The second peak of the untreated sample at 279.7 °C could be due to the presence of hemicellulose, but there is no corresponding peak in the treated sample. The third peak occurred at 340.5 °C for the untreated sample, which could indicate cellulose, and the corresponding peak for the treated sample occurred at 346.9 °C.

8.5.2 TGA analysis

The temperature of onset of degradation for the untreated sample occurred at 290.4 °C and at 301.6 °C for the treated sample (Figures 7 & 8). There is not much difference in the mass loss; 75.36 % for the treated sample, and 76.6 % for the untreated sample. The residual mass is slightly higher for the treated sample 16.58 % as compared to that for the untreated sample, 12.91 %.

The DSC and TGA results tend to show that there is a much better thermal stability for the treated sample. This could be due to the partial removal of hemicellulose, and also a higher cellulose I in the residual mass as compared to the untreated sample.

8.6 DSC analysis of Bottle palm

Figure 6 of appendix 1 shows that there is a major shift in the first endothermic peak for the treated sample (85.1 °C) as compared to the untreated sample (68.6 °C). This could be due to both the surface moisture and adsorbed moisture due to the removal of non-cellulosic content. There is absence of a corresponding second peak in the treated sample as compared to the untreated sample (287.8 °C). This could be due to partial removal of non-cellulosic content such as hemicellulose. However, both samples have a peak at 342.1 °C.

8.7 DSC analysis of Latania *loddigesii*

The first endothermic peak for both untreated and treated samples (Figure 7) occurred at almost the same temperature (72 °C for untreated and 71.7°C). However, the second peak occurred at 303.5 °C for untreated sample whilst occurring at 278.3 °C for the treated sample. This shows that there is still presence of hemicellulose in the alkaline treated sample. The third peak occurred at 338.5 °C for the untreated sample, and occurred at 353.5 °C (more pronounced) for the alkaline treated sample. This result tends to show that there is better thermal stability after the alkaline treatment.

8.8 DSC analysis of Scleria *sieberi*

Figure 8 of Appendix 1 shows that there is a major shift in the first endothermic peak for the treated sample (84 °C) as compared to the untreated sample (73 °C). This could be due to both the surface moisture and adsorbed moisture due to the removal of non-cellulosic content. The second peak occurred at 318.2 °C for the untreated sample, and at 339.7 °C for the treated sample. This shows better thermal stability for the alkaline treatment sample.

8.9 DSC analysis of Banyan

Figure 9 of Appendix 1 shows that there is a shift in the first endothermic peak for the treated sample (84 °C) as compared to the untreated sample (78.5 °C). This could be due to both the surface moisture and adsorbed moisture due to the removal of non-cellulosic content. The second peak occurred at 306.4 °C for the untreated sample, and at 361.5 °C for the treated sample. This shows better thermal stability for the alkaline treatment sample of the banyan fibres.

8.10 DSC analysis of Panicum maximum

Figure 10 of Appendix 1 shows that there is a slight shift in the first endothermic peak for the treated sample (72.3 °C) as compared to the untreated sample (67.8 °C). This could be due to the evaporation of the surface moisture. There is absence of a corresponding second peak in the treated sample as compared to the untreated sample (284.9 °C). This could be due to partial removal of non-cellulosic content such as hemicellulose. The third peak occurred at 344.4 °C for the untreated sample, and occurred at 342.7 °C for the alkaline treated sample. There does not seem to be any major improvement in the thermal stability after the alkaline treatment.

8.11 DSC analysis of Thysanolaena Latifolia (Fatak)

The first endothermic peak for both untreated and treated samples (Figure 11) occurred at almost the same temperature (78.5 °C for untreated and 76.8°C). However, the second peak occurred at 275.4 °C for untreated sample whilst occurring at 290.5 °C for the treated sample. This shows that there is still presence of hemicellulose in the alkaline treated sample. The third peak occurred at 335.8 °C for the untreated sample, and occurred at 347.9 °C for the alkaline treated sample. This result tends to show that there is better thermal stability after the alkaline treatment.

8.12 DSC analysis of Sporobolus africanus

The first endothermic peak occurred at 73.6 °C for the untreated sample (Figure 12), and at 77.2 °C for the treated sample. Moreover, the second peak occurred at 265.7 °C for untreated sample whilst occurring at 284.6 °C for the treated sample. This shows that there is still presence of hemicellulose in the alkaline treated sample. The third peak occurred at 340.8 °C for the untreated sample, and occurred at 344.9 °C for the alkaline treated sample. This result tends to show that there is slightly better thermal stability after the alkaline treatment.

8.13 Ravenala Madagascariensis

8.13.1 DSC analysis

Figure 13 of Appendix 1 shows that there is a major shift in the first endothermic peak for the untreated sample (58.3 °C) as compared to the treated sample (95.2 °C). This could be due to both the surface moisture and adsorbed moisture due to the removal of non-cellulosic content. There is absence of a corresponding second peak in the treated sample as compared to the untreated sample (291.8 °C). This could be due to partial removal of non-cellulosic content such as hemicellulose. The third peak occurred at 339.1 °C for the untreated sample, and occurred at 337.3 °C for the alkaline treated sample.

8.13.2 TGA analysis

Figures 9 & 10 of Appendix 2 shows that the temperature of the onset of degradation for the treated sample is higher (318.2 °C) than that for the untreated sample (271 °C), and the mass loss during the degradation stage is slightly lower for the untreated sample (85.17 %) as compared to the treated sample (86.81 %). The amount of residue is more for the treated sample (2.11 %) than the untreated sample (0.15 %). The much higher onset degradation temperature of the treated sample might well confirm the partial removal of hemicellulose, which thus cause a rearrangement and reorientation of the cellulose crystals. The higher residual mass for the alkaline treated sample can be explained by the presence of a greater amount of crystalline cellulose I.

8.14 DSC analysis of Ananas Bracteatus

Figure 14 of Appendix 1 shows that the first endothermic peak for the untreated sample (79.2°C) is slightly higher than that for the treated sample (74.8 °C). There is absence of a corresponding second peak in the treated sample as compared to the untreated sample (289.7 °C). This shows better thermal stability for the alkaline treatment sample of the banyan fibres. The third peak occurred at 329.8 °C for the untreated sample, and occurred at 332.9 °C for the alkaline treated sample. There does not seem to be any major improvement in the thermal stability after the alkaline treatment.

8.15 DSC analysis of Rhapis excels (Zon)

Figure 15 of Appendix 1 shows that there is a major shift in the first endothermic peak for the treated sample (79.3 °C) as compared to the untreated sample (69 °C). This could be due to both the surface moisture and adsorbed moisture due to the removal of non-cellulosic content. The second peak occurred at 278.4 °C for the untreated sample, and at 272.2 °C for the treated sample. This shows better thermal stability for the alkaline treatment sample. The third peak occurred at 326.4 °C for the untreated sample, and occurred at 351 °C for the alkaline treated sample. This shows a better thermal stability.

CHAPTER 9: CONCLUSION & RECOMMENDATIONS

9.1 Conclusion

Based on the results of the tensile strength the following fibres have good potential for use as reinforcing materials in bio-composite applications: Ravenala Madagascariensis, Dracaena floribunda, Latania loddigesii, Dracaena concinna, and Thysanolaena latifolia. This is because these fibres have relatively high tensile strength in the treated state, that is, above, 150 MPa. The first four fibres, mentioned in Section 6.2, have a tensile strength greater than 200 MPa, even though that their respective optimum chemical treatments have not been identified, showing that there potential for identification of stronger fibres from the group.

The FTIR analysis has indeed shown that there has been a reduction in the amount of hemicellulose in the chemically treated fibres for Ravenala Madagascariensis, Latania loddigesii, Dracaena concinna, and Thysanolaena latifolia. However, it seems that there has not been any removal of hemicellulose for Dracaena floribunda.

The DSC and TGA analyses tend to show that there is better thermal stability of the chemically treated fibres as compared to that of the untreated fibres for all the five above mentioned species.

The results of the tensile strength of the bio-composite manufactured with Ravenala Madagascariensis, Dracaena floribunda, Dracaena concinna, and Thysanolaena latifolia, have shown a significantly lower strength as compared to the strength of the resin alone. This has been explained due to poor wettability of the fibres to the resin, presence of air bubbles that induce weak places in the composite. Moreover, the optimum length of fibre leading to improved mechanical strength has not been defined.

Table 9.1 shows the fibre yield for the Ravenala Madagascariensis, Dracaena floribunda, and Dracaena concinna fibres. It can be seen that on average the yield of the dry fibre to the dry leaf is 20 % but a higher yield for Dracaena floribunda at 25 %.

Table9. 1 Result of yield extraction.

	Mass [g]				Yield (%)	
	Green leaf	Dry leaf	Extracted fibre	Dry fibre	Yield 1	Yield 2
Ravenala	562.1	114	92	22	3.91	19.30
Concinna	55.82	8.9	3.85	1.9	3.40	21.35
Floribunda	42.48	9.7	4.8	2.5	5.89	25.77

Note: **Yield 1:** Ratio of dry fibre to green leaf, **Yield 2:** Ratio of dry fibre to dry leaf.

9.2 Recommendations

The following are the main recommendations:

a. Perform detailed investigation for each of the five above-mentioned fibres in order to determine the optimum NaOH treatment which will yield the fibre with the highest mechanical strength.

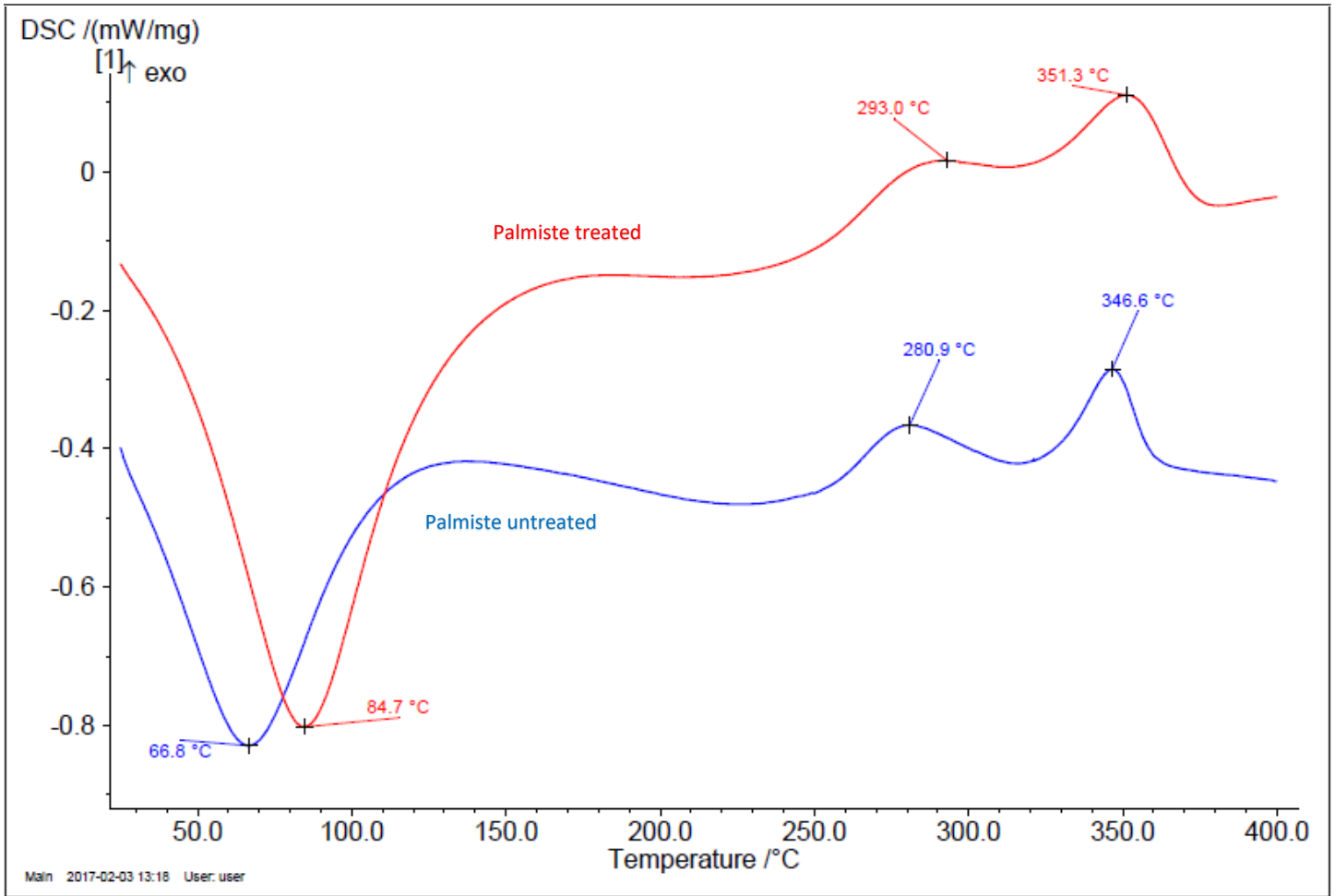
b. Perform an investigation for each of the five above-mentioned fibres in order to determine whether better mechanical strength can be obtained with other chemical substances such as sodium bicarbonate. Investigate the possibility of using other means of environmental-friendly fibre-treatment methods such as enzyme treatments.

c. Perform an investigation for each of the five above-mentioned fibres in order to determine whether better mechanical strength can be obtained with naturally occurring enzymes, obtained from agro wastes.

d. Investigate the effect of fibre orientation, fibre to resin ratio, and fibre length on the mechanical strength of the bio-composites.

e. Develop non-woven mat for each of the five above-mentioned fibres, which can be used as reinforcing material in bio-composites.

APPENDIX 1



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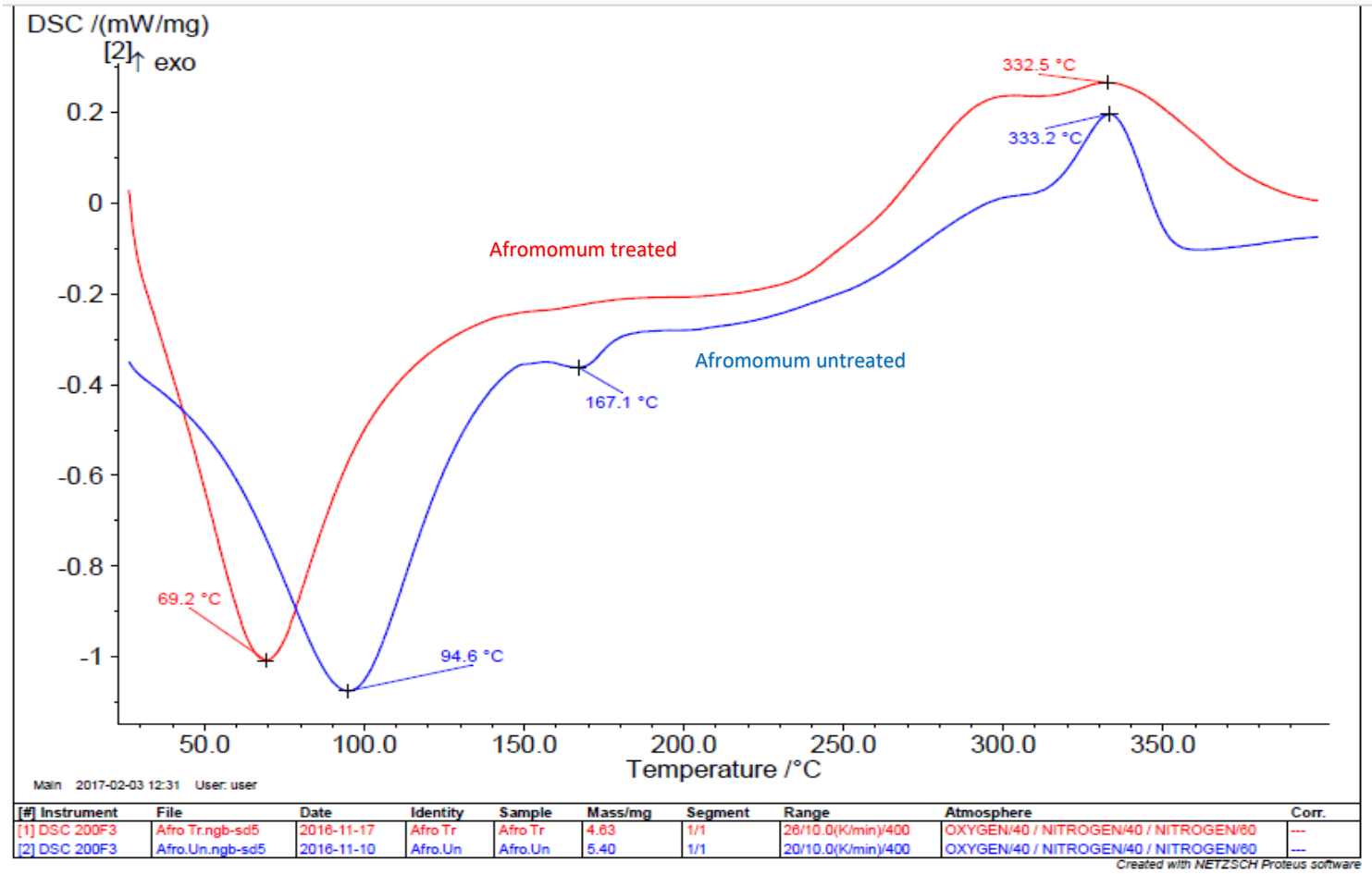
[1] DSC 200F3	Palmiste tr.ngb-sd5	2016-11-18	Palmiste tr	Palmiste tr	7.52	1/1	15/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---
[2] DSC 200F3	Palmiste un.ngb-sd5	2016-11-18	Palmiste un	Palmiste un	6.53	1/1	22/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

Figure 1: DSC for Palmiste Rouge

APPENDIX 1



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[1] DSC 200F3	Afro Tr.ngb-sd5	2016-11-17	Afro Tr	Afro Tr	4.63	1/1	26/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---
[2] DSC 200F3	Afro.Un.ngb-sd5	2016-11-10	Afro.Un	Afro.Un	5.40	1/1	20/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

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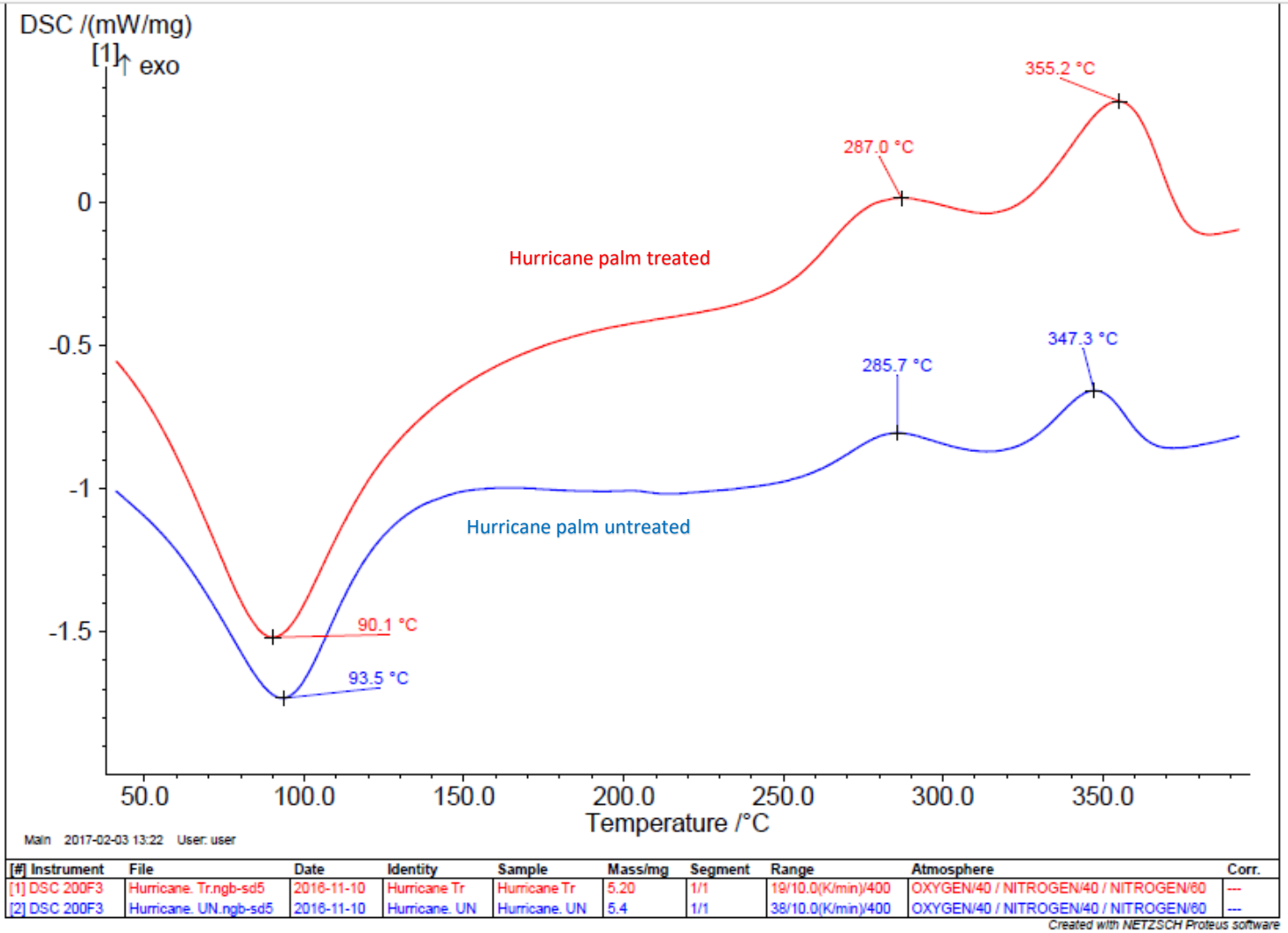
Figure 2: DSC for Afromomum

APPENDIX 1



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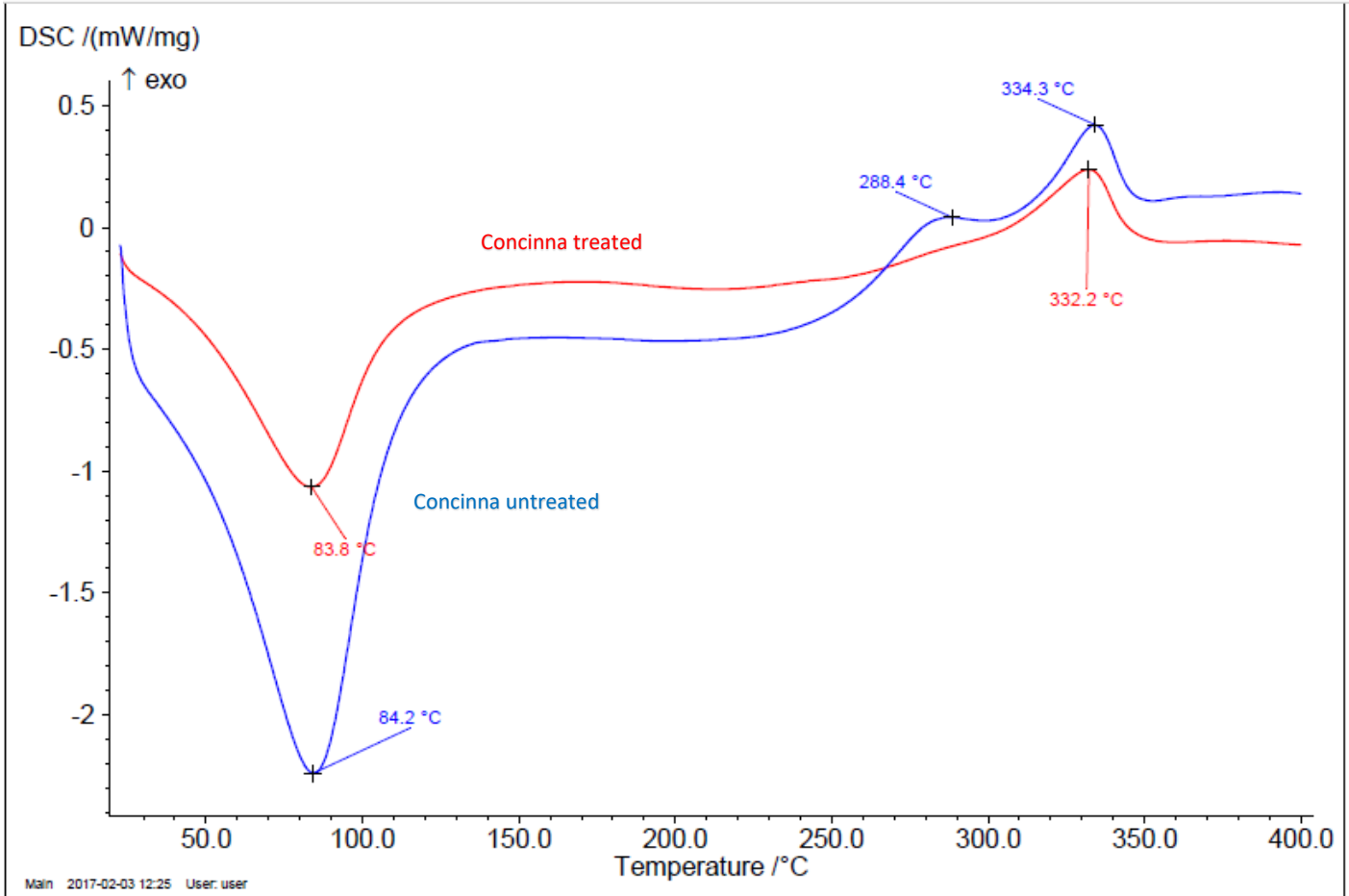
[1] DSC 200F3	Hurricane. Tr.ngb-sd5	2016-11-10	Hurricane Tr	Hurricane Tr	5.20	1/1	19/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---
[2] DSC 200F3	Hurricane. UN.ngb-sd5	2016-11-10	Hurricane. UN	Hurricane. UN	5.4	1/1	38/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

Figure 3: DSC for Hurricane palm

APPENDIX 1



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[2] DSC 200F3	DC1.ngb-sd5	2016-09-15	DC1	DC1	5	1/1	22/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

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Figure 4: DSC for Dracaena concinna

APPENDIX 1

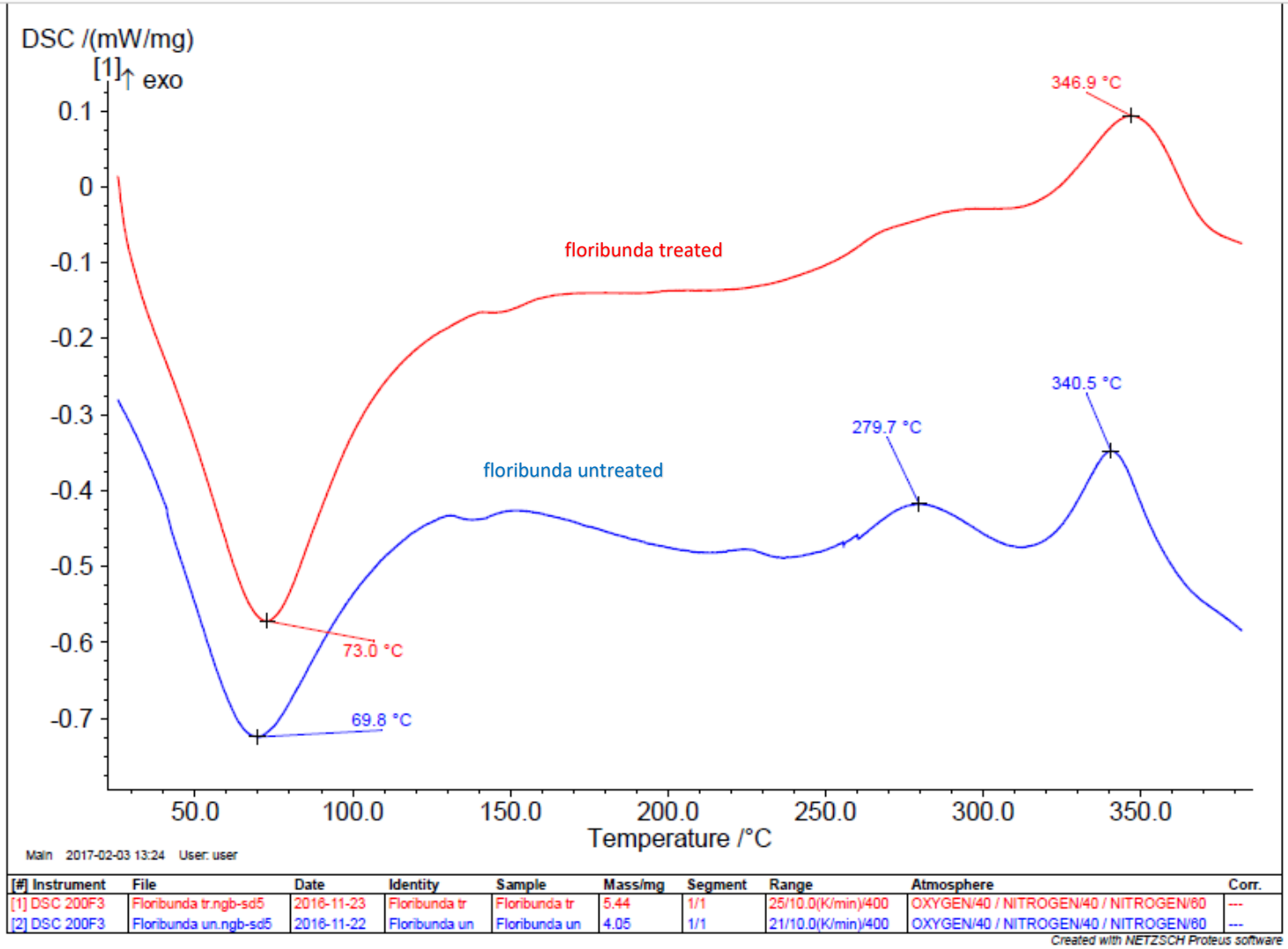
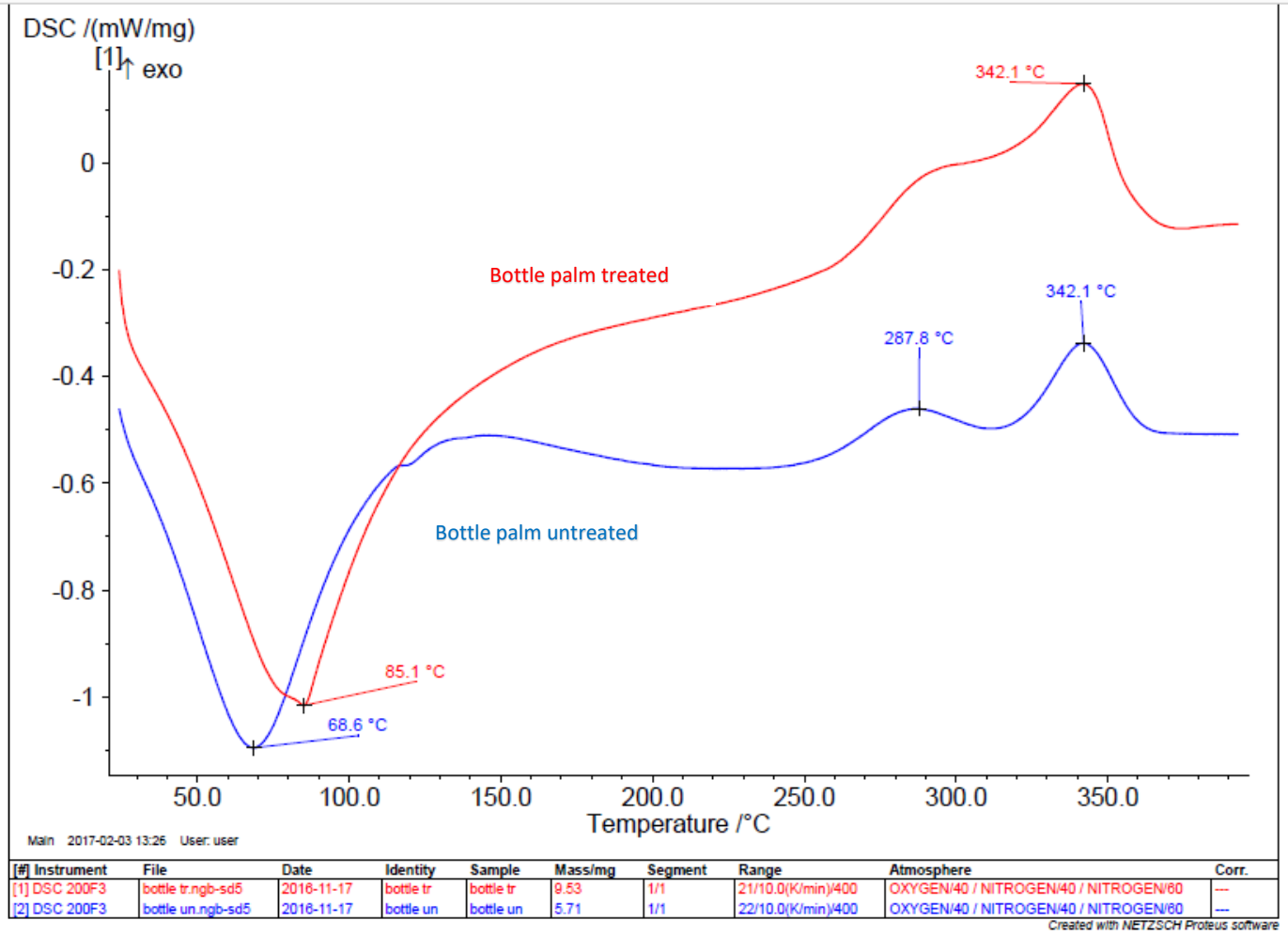


Figure 5: DSC for Dracaena floribunda

APPENDIX 1



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[2] DSC 200F3	bottle un.ngb-sd5	2016-11-17	bottle un	bottle un	5.71	1/1	22/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

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Figure 6: DSC for Bottle palm

APPENDIX 1

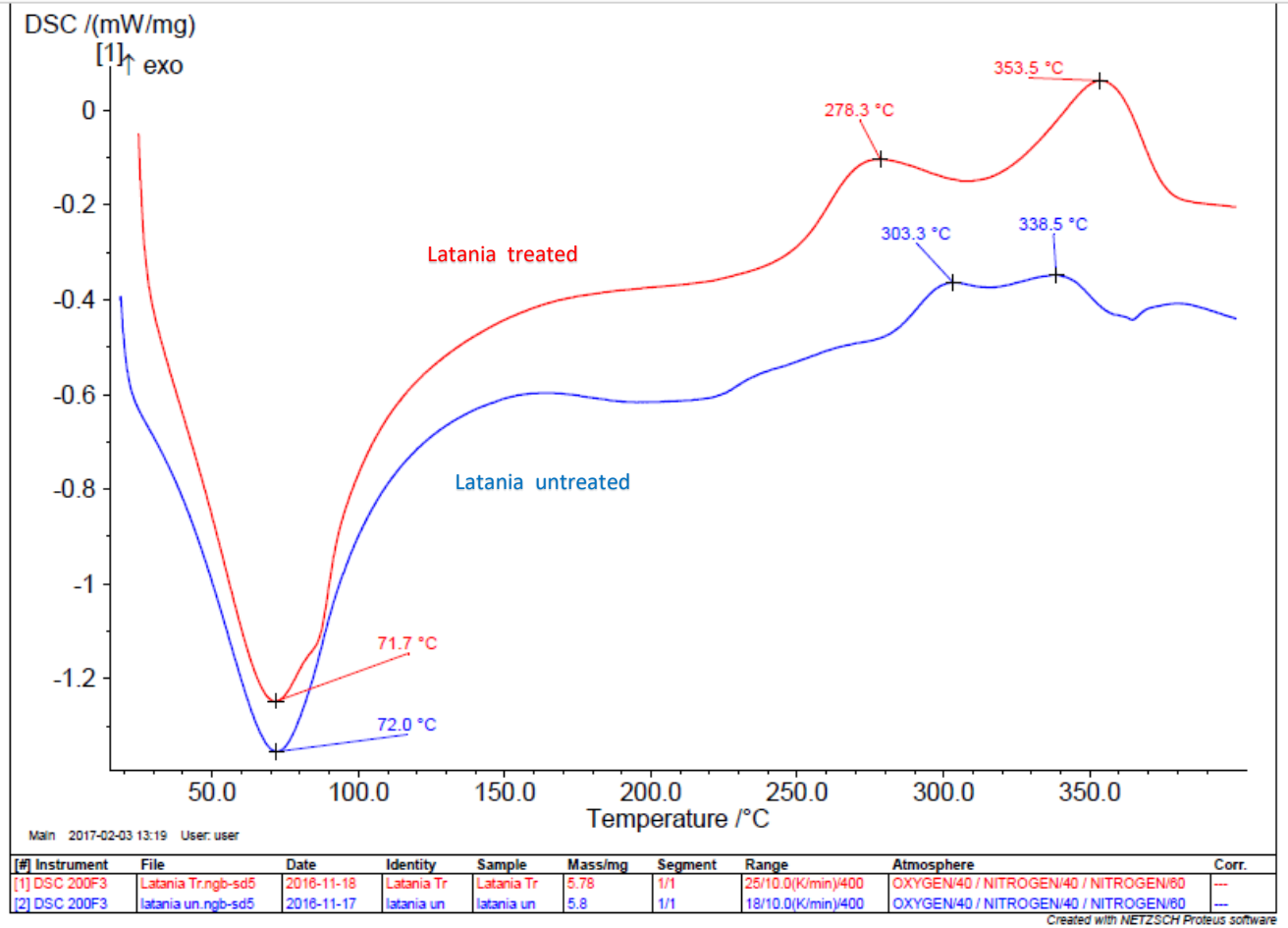


Figure 7: DSC for Latania

APPENDIX 1

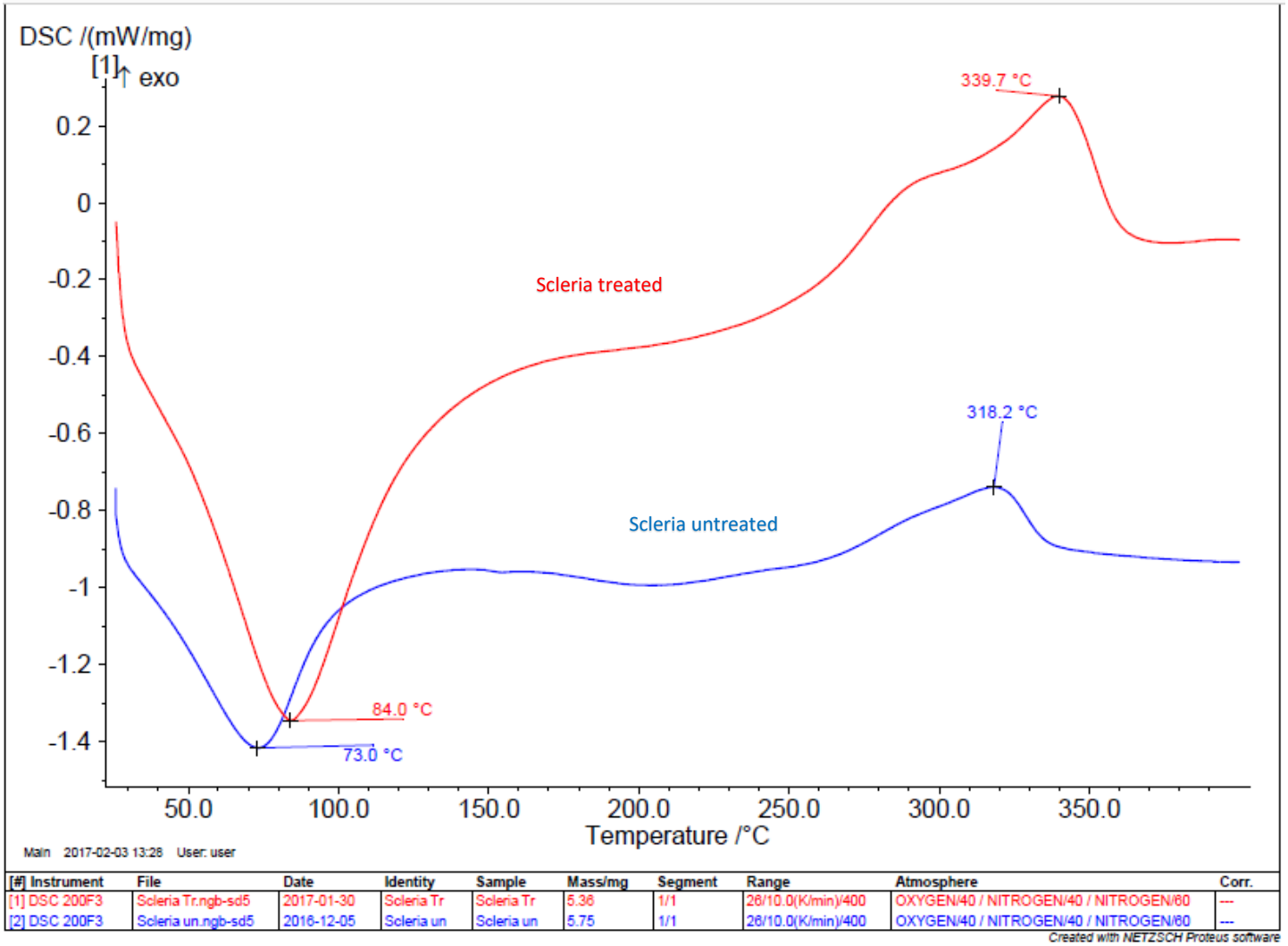


Figure 8: DSC for Scleria

APPENDIX 1

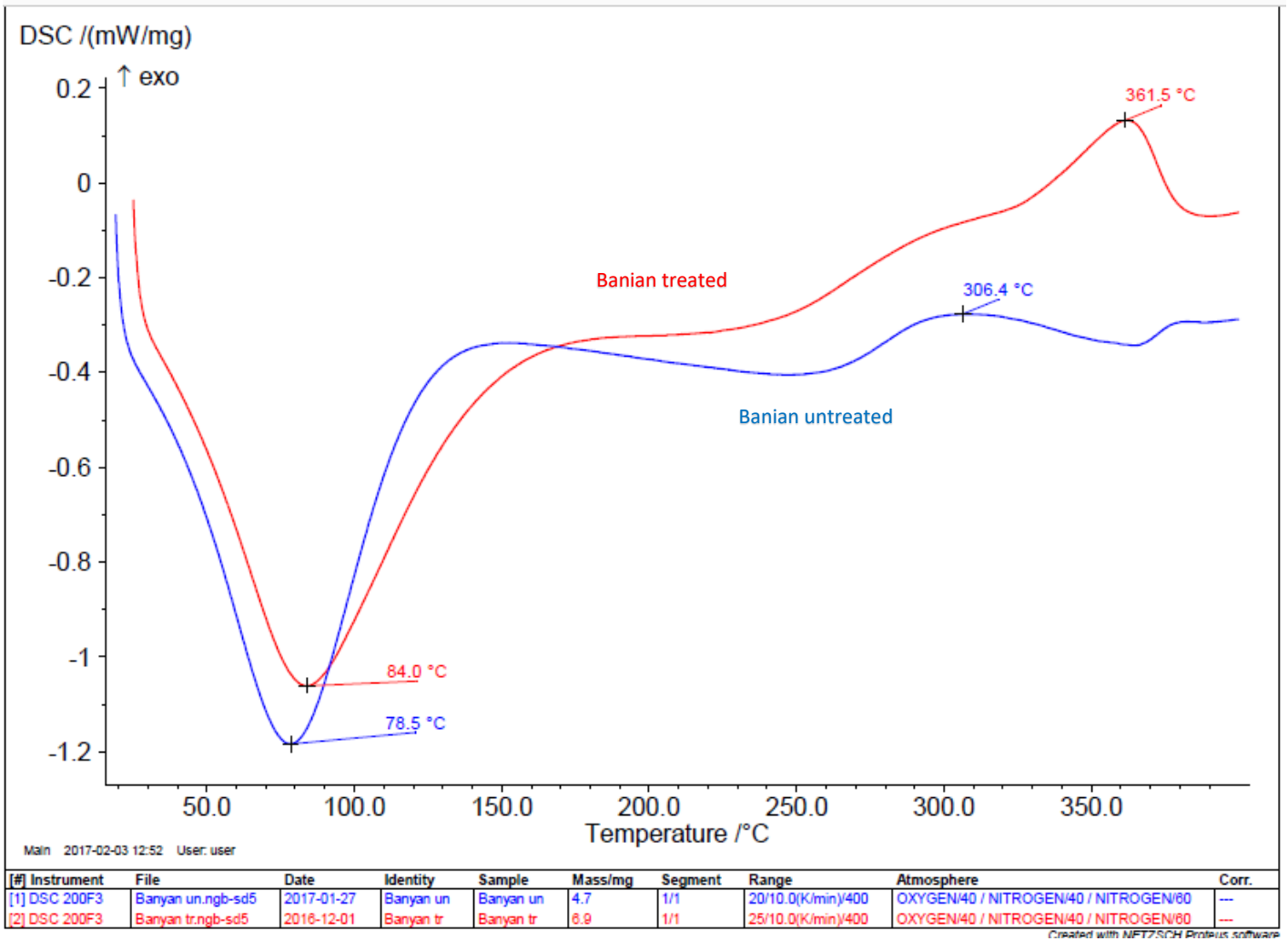
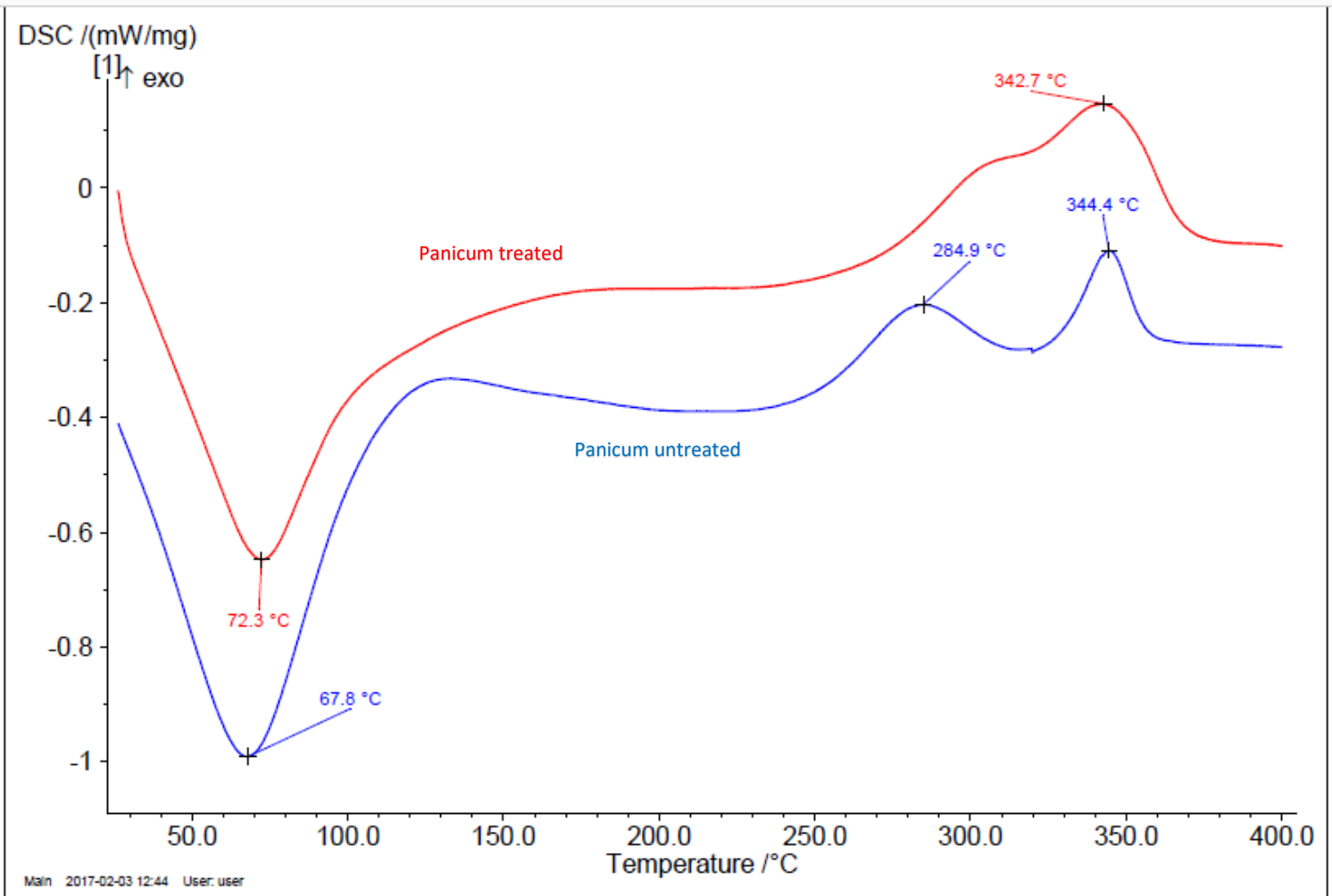


Figure 9: DSC for Banyan

APPENDIX 1



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[2] DSC 200F3	Panicum un.ngb-sd5	2016-11-18	Panicum un	Panicum un	4.04	1/1	16/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

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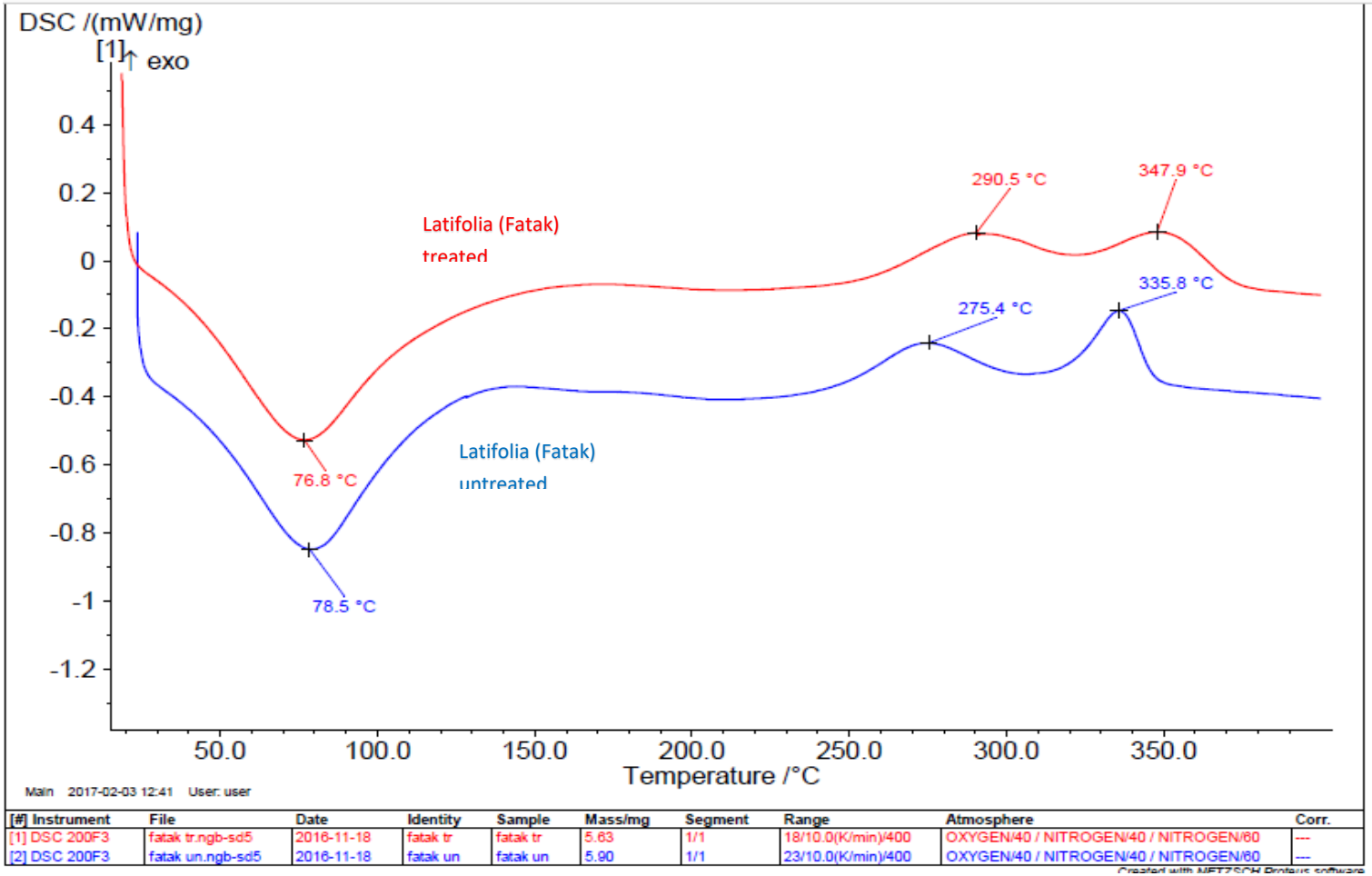
Figure 10: DSC for Panicum maximum

APPENDIX 1



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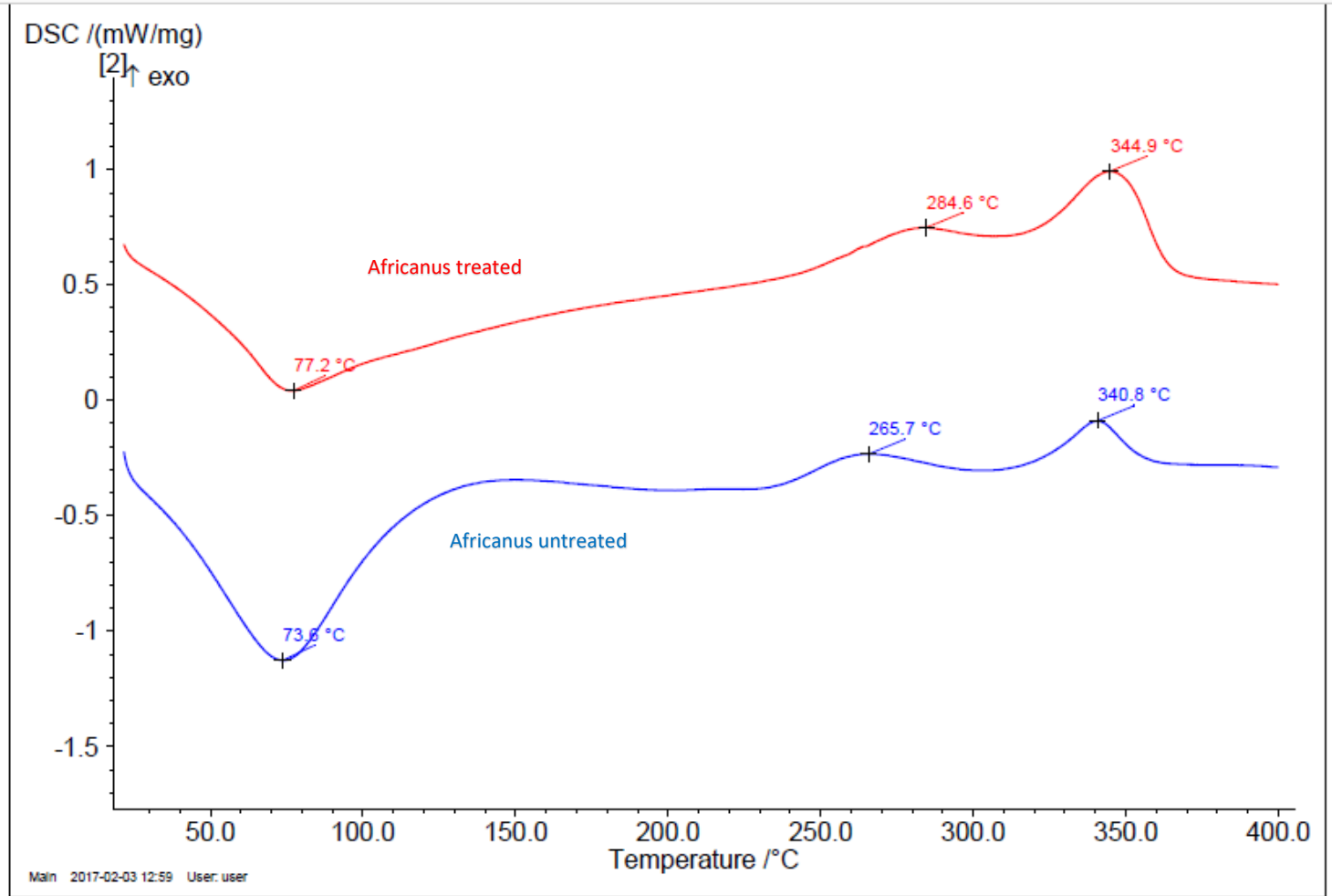
[2] DSC 200F3	fatak un.ngb-sd5	2016-11-18	fatak un	fatak un	5.90	1/1	23/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

Figure 11: DSC for Latifolia (Fatak)

APPENDIX 1



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[2] DSC 200F3	Africanus Un.ngb-sd5	2017-01-26	Africanus Un	Africanus Un	4.38	1/1	19/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

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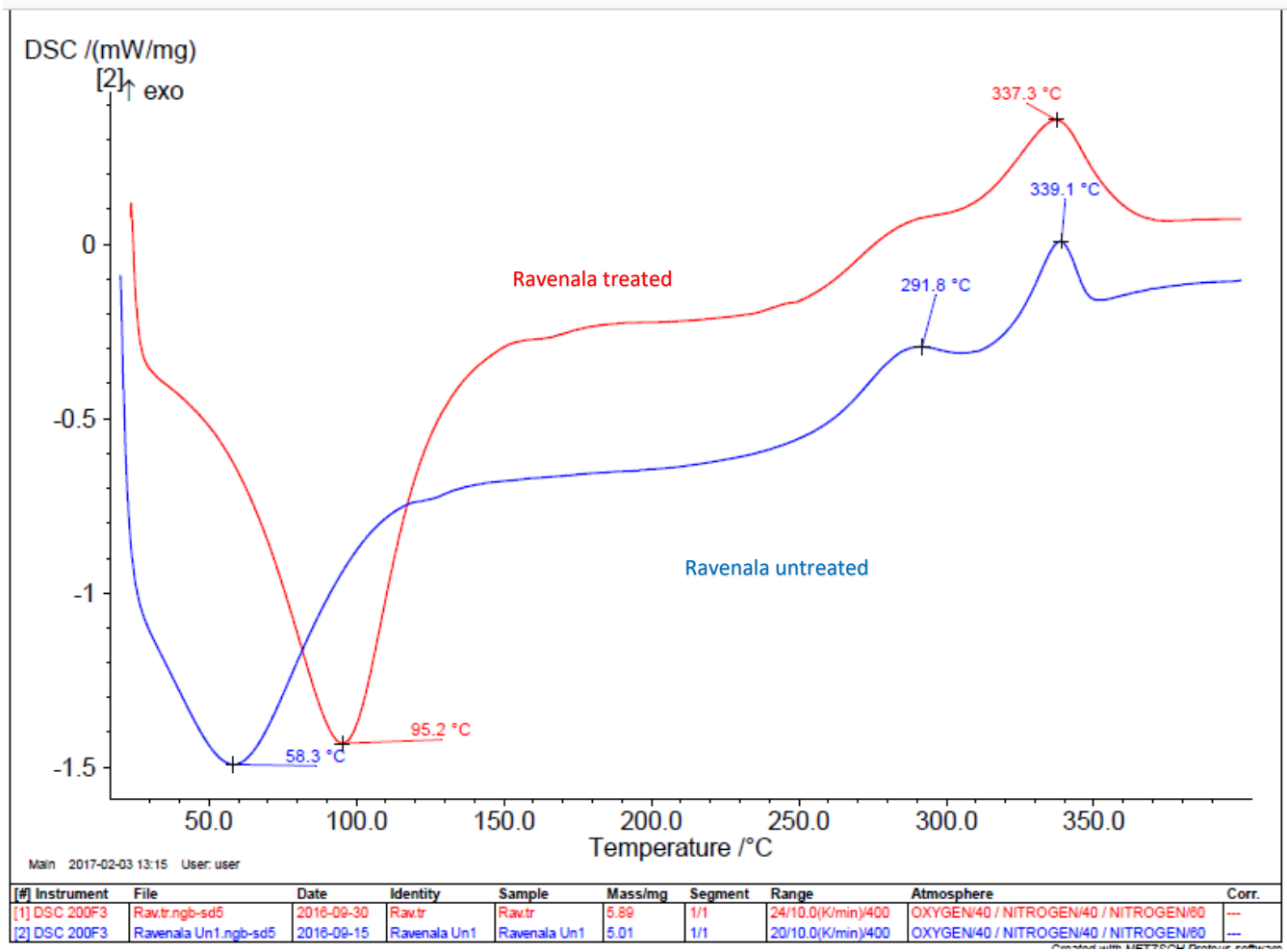
Figure 12: DSC for Sporobolus africanus

APPENDIX 1



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[2] DSC 200F3	Ravenala Un1.ngb-sd5	2016-09-15	Ravenala Un1	Ravenala Un1	5.01	1/1	20/10.0(K/min)/400	OXYGEN/40 / NITROGEN/40 / NITROGEN/60	---

Figure 13: DSC for Ravenala

APPENDIX 1

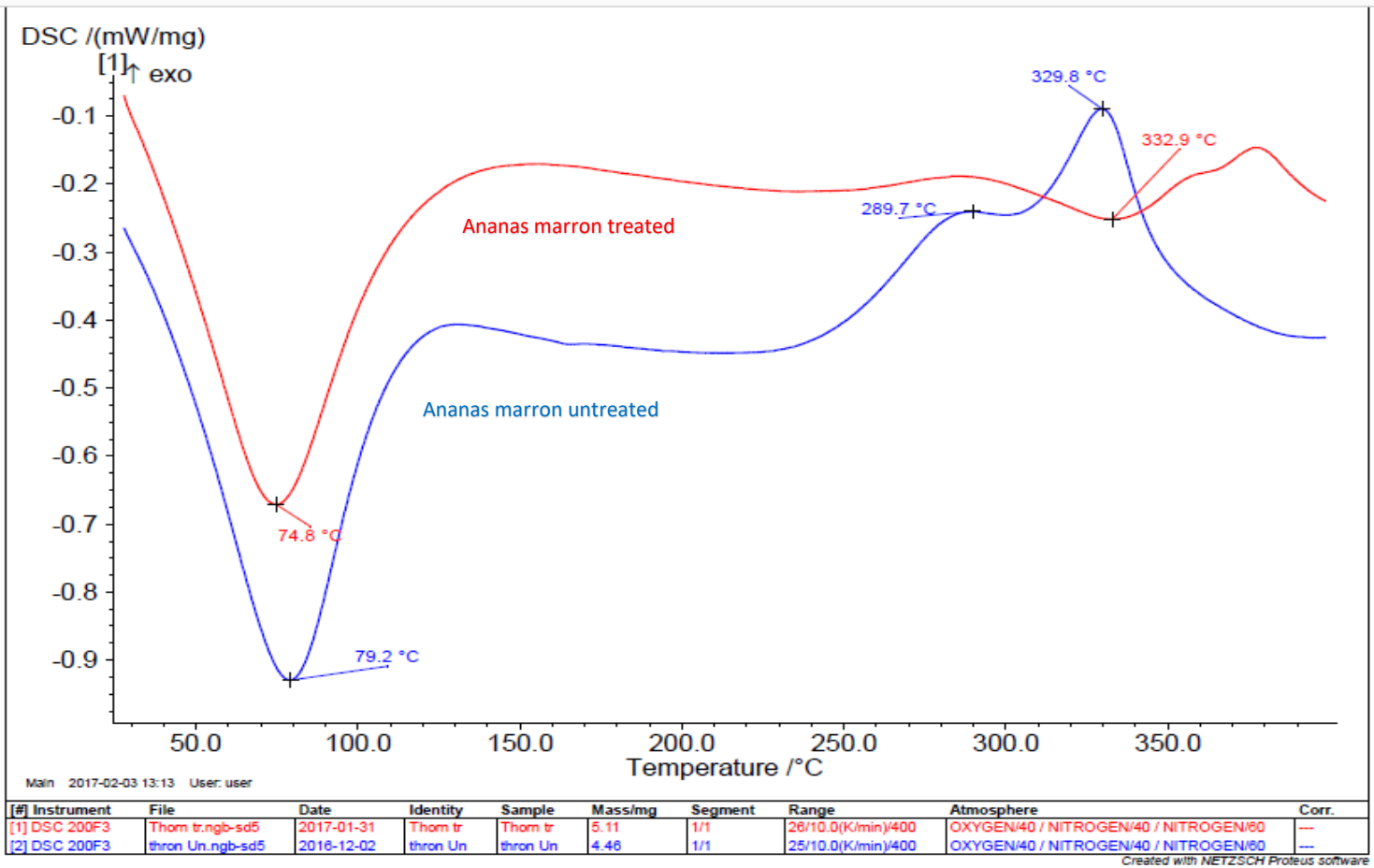


Figure 14: DSC for Ananas Bracteatus

APPENDIX 1

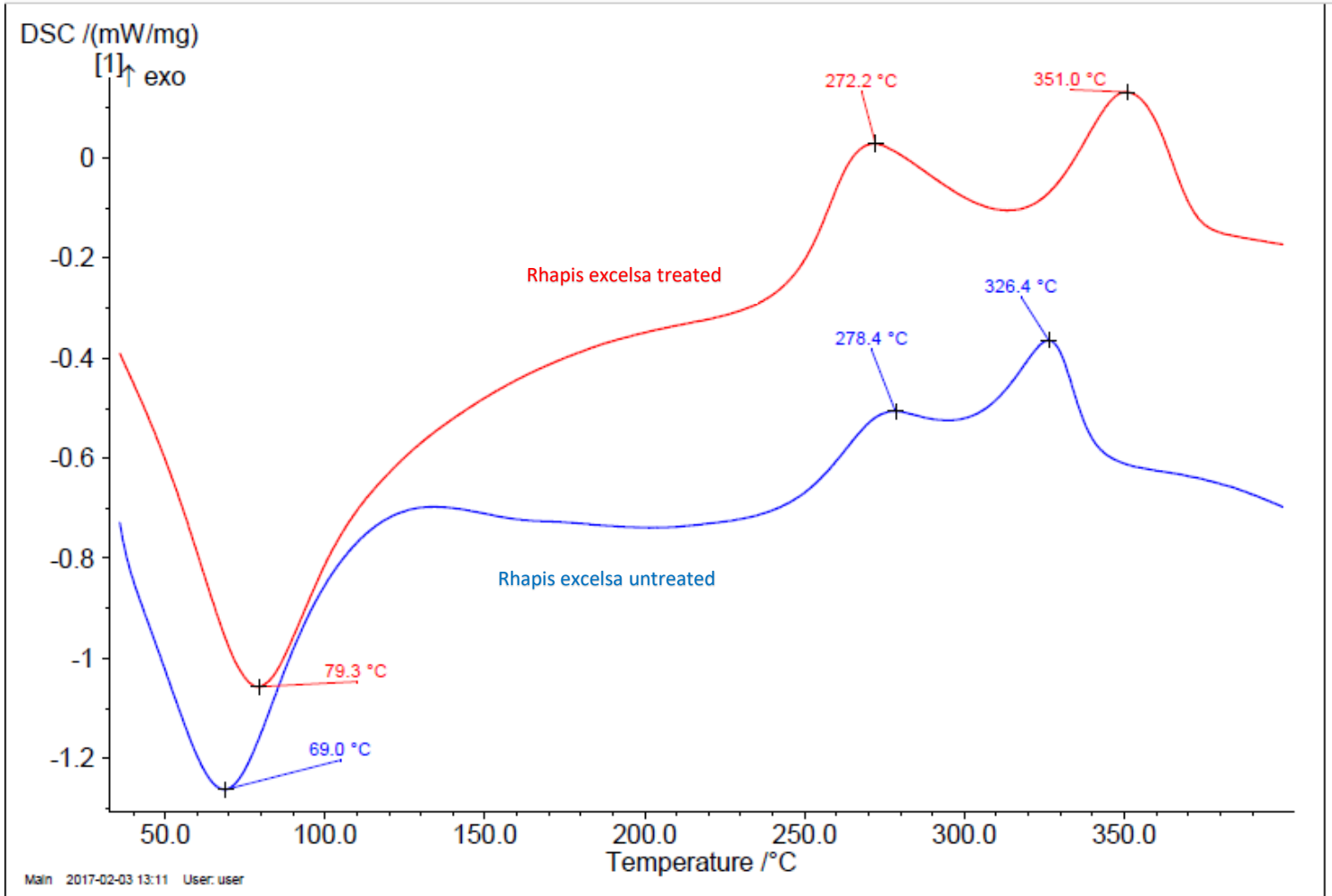
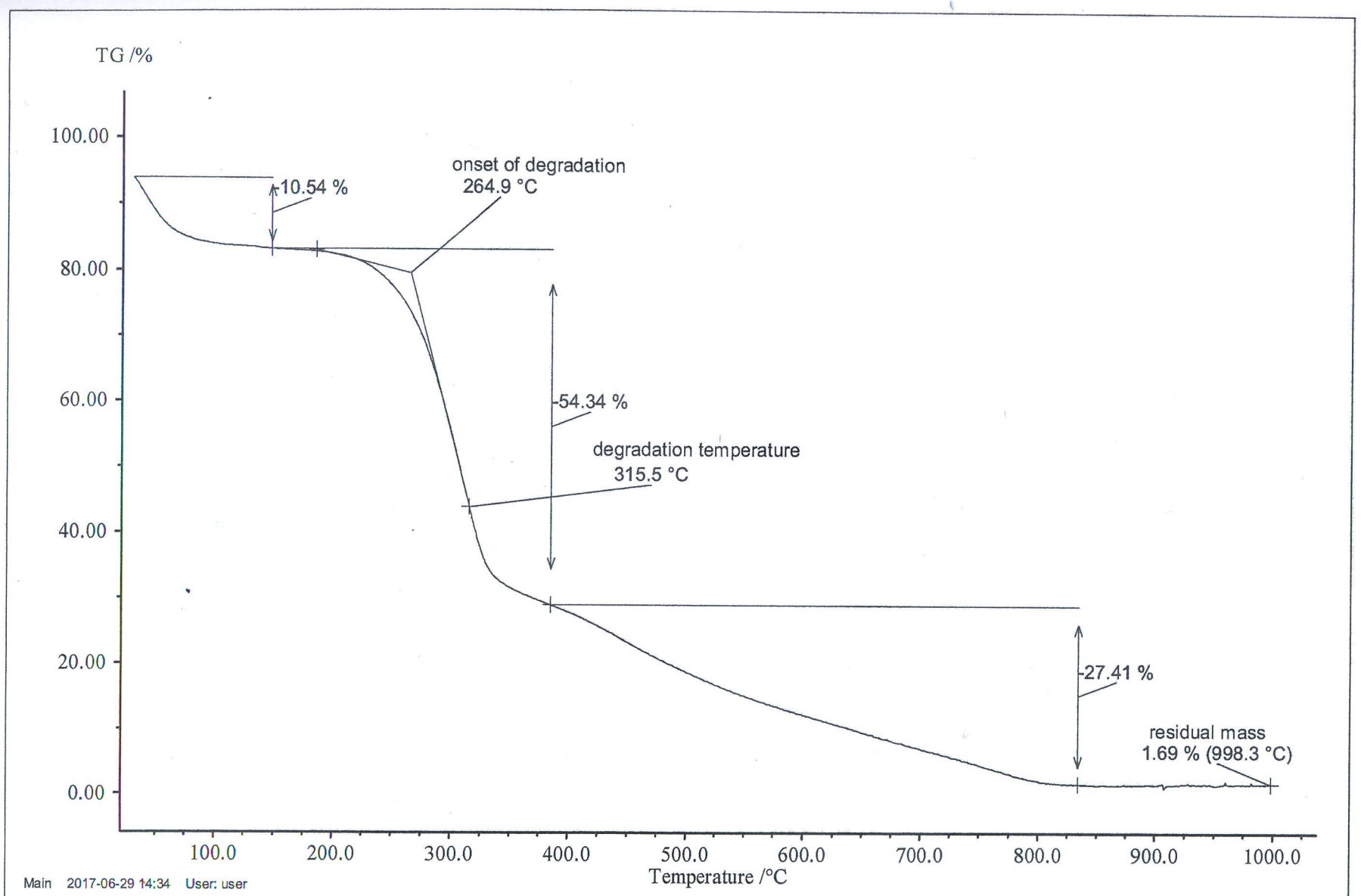


Figure 15: DSC for Rhapis excels (Zon)



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Project :		**Sample :**	Afromomum augusti folium	**Sample car./TC :**	TG 209F3 standard/P	**Atmosphere :**	-- / N2 / N2
Identity :	untreated	**Material :**		**Sample mass :**	4.7181 mg	**Corr/m. range :**	520/2000 mg
Date/time :	6/21/2017 9:41:07 AM	**Correction file :**	baseline 10K 4.7.14.ngb-bt6	**Mode/type of meas. :**	TG/sample with correction		
Laboratory :	Characterisation Lab	**Temp. calib. file :**	TCALZERO.TCX	**Segments :**	1/1		
Operator :		**Range :**	26/10.0(K/min)/1000	**Crucible :**	Al2O3		

Figure 1: TGA for untreated Afromomum

Created with NETZSCH Proteus software

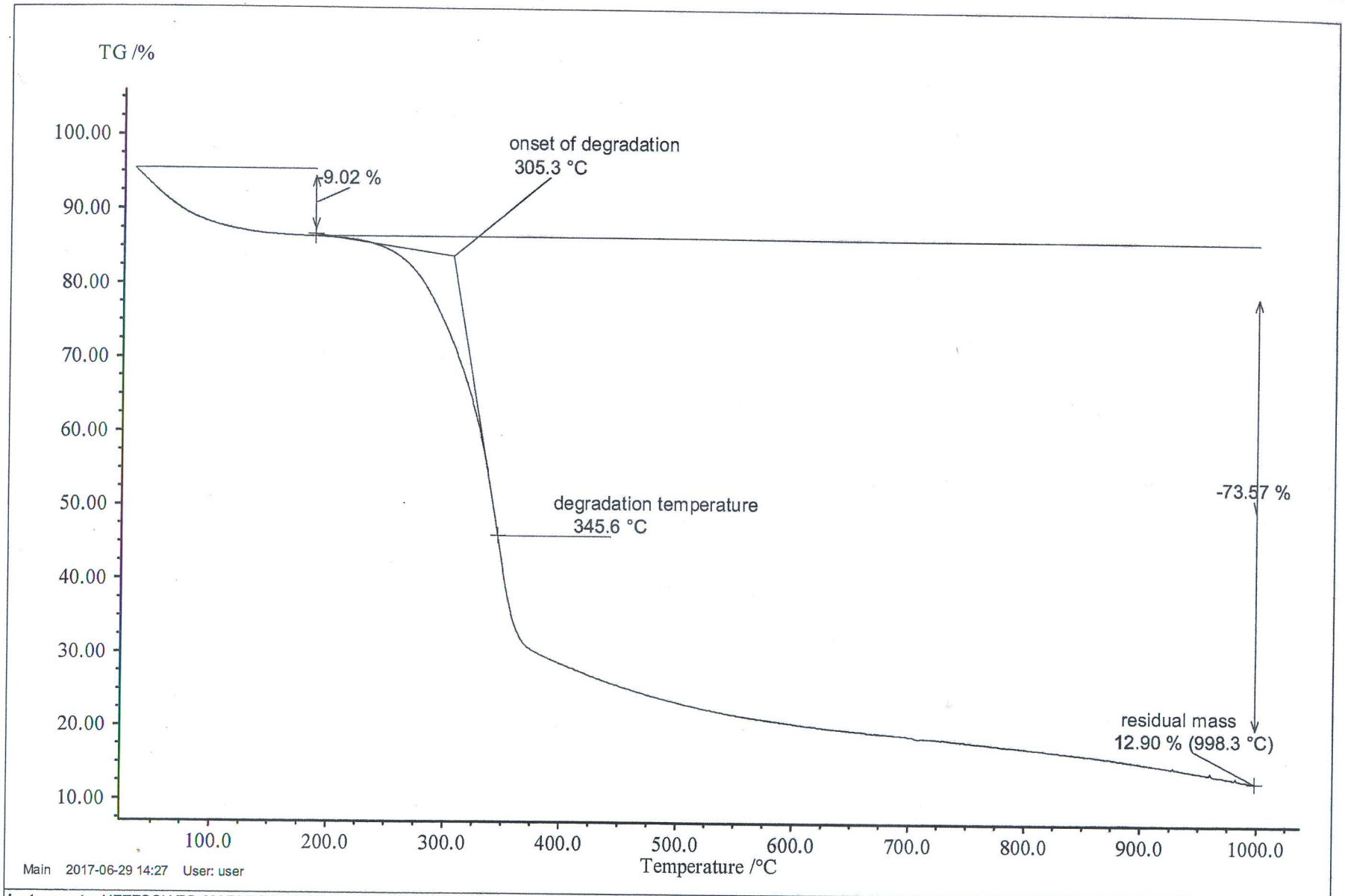
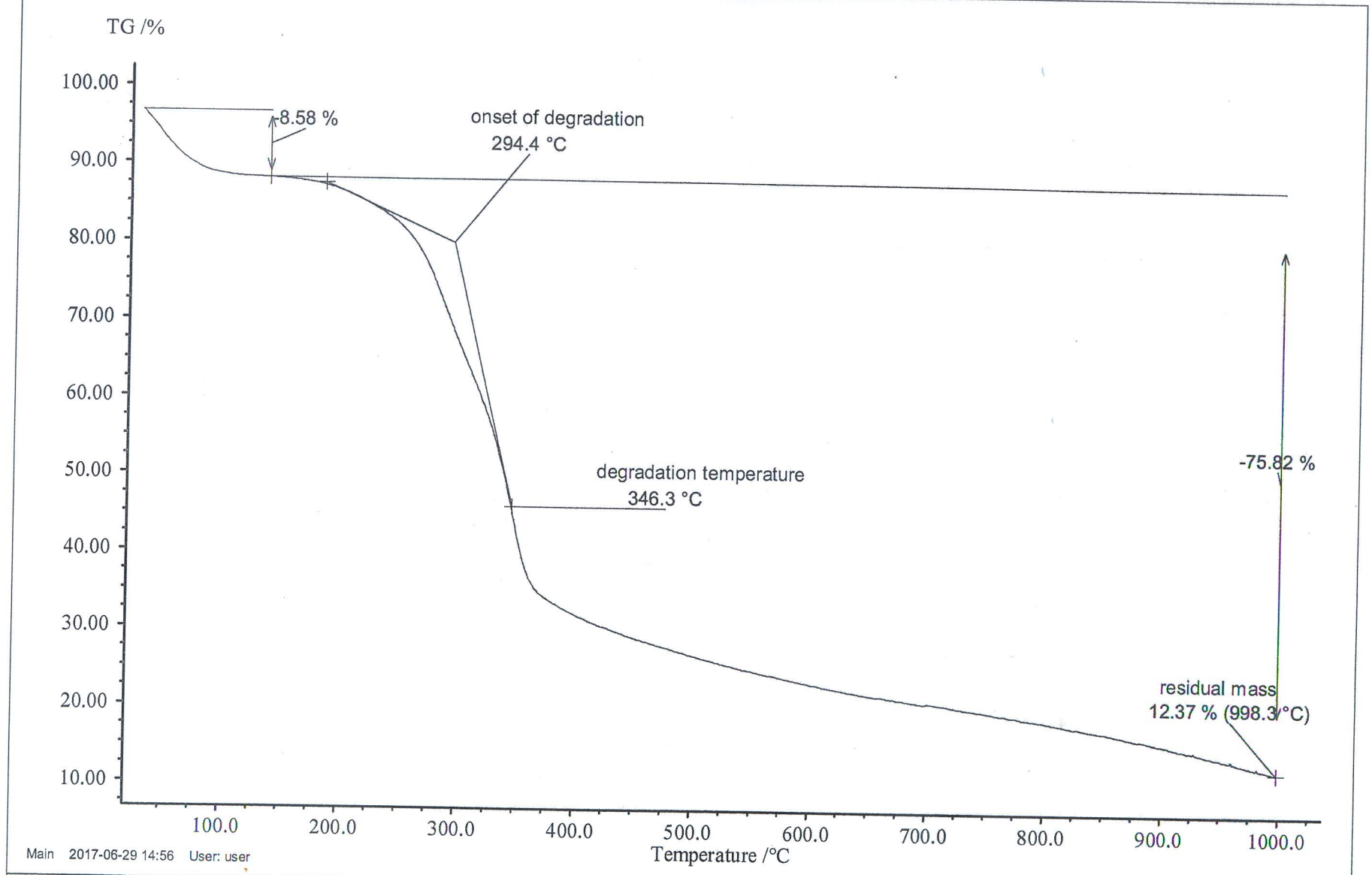


Figure 2: TGA for treated Afromomum



Instrument :	NETZSCH TG 209F3 TGA209F3A-0254-L	**File :**	C:\NETZSCH\Proteus\data5\14-2-12 TG\Engineering\Hurricane Palm Untreated .ngb-dt6				
Project :		**Sample :**	Hurricane Palm Untreated	**Sample car./TC :**	TG 209F3 standard/P	**Atmosphere :**	-- / N2 / N2
Identity :	Hurricane Palm Untreated	**Material :**		**Sample mass :**	7.06 mg	**Corr/m. range :**	520/2000 mg
Date/time :	6/20/2017 9:41:51 AM	**Correction file :**	baseline 10K 4.7.14.ngb-bt6	**Mode/type of meas. :**	TG/sample with correction		
Laboratory :	Characterisation Lab	**Temp. calib. file :**	TCALZERO.TCX	**Segments :**	1/1		
Operator :		**Range :**	26/10.0(K/min)/1000	**Crucible :**	Al2O3		

Created with NETZSCH Proteus software

Figure 3: TGA for untreated Hurricane palm

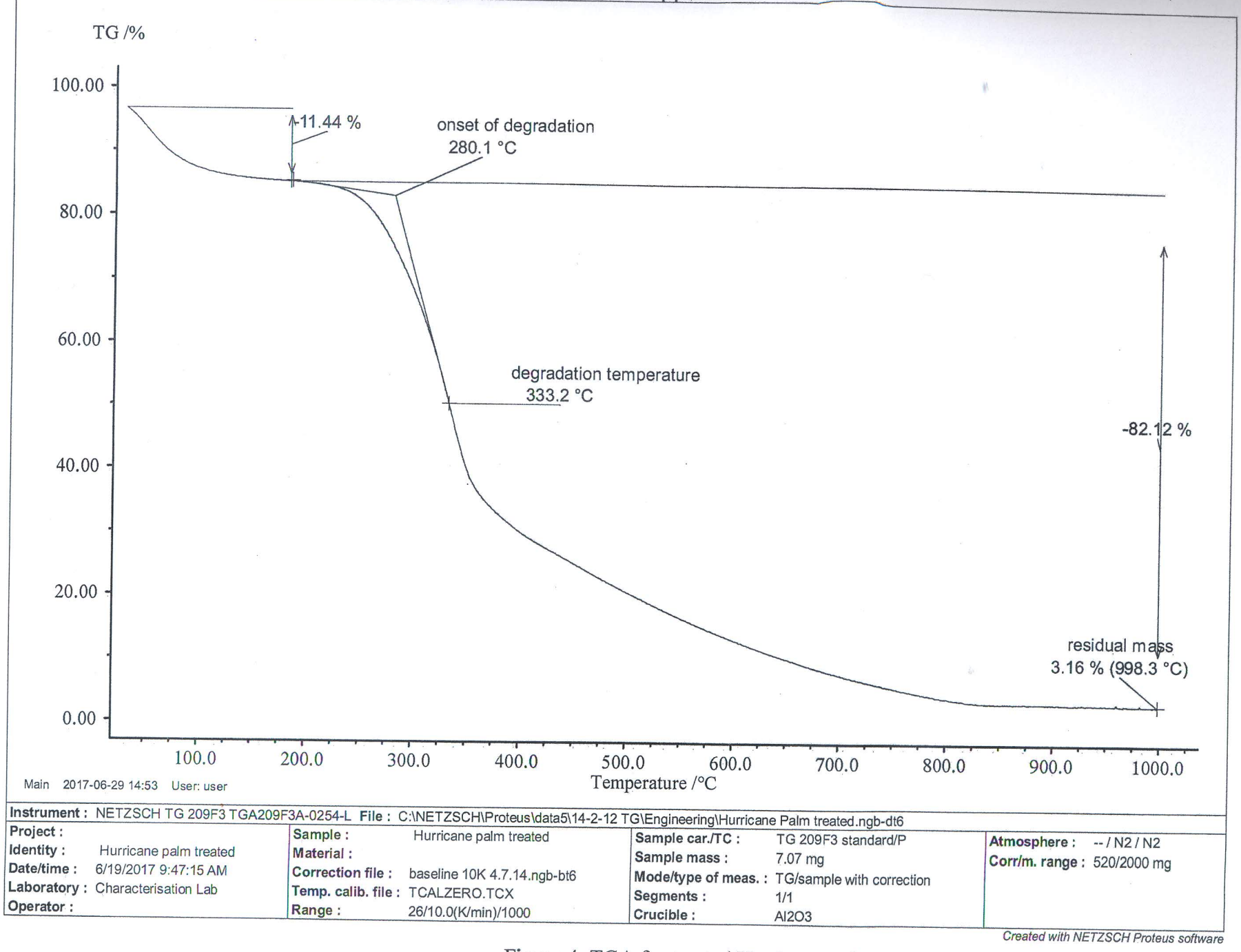


Figure 4: TGA for treated Hurricane palm

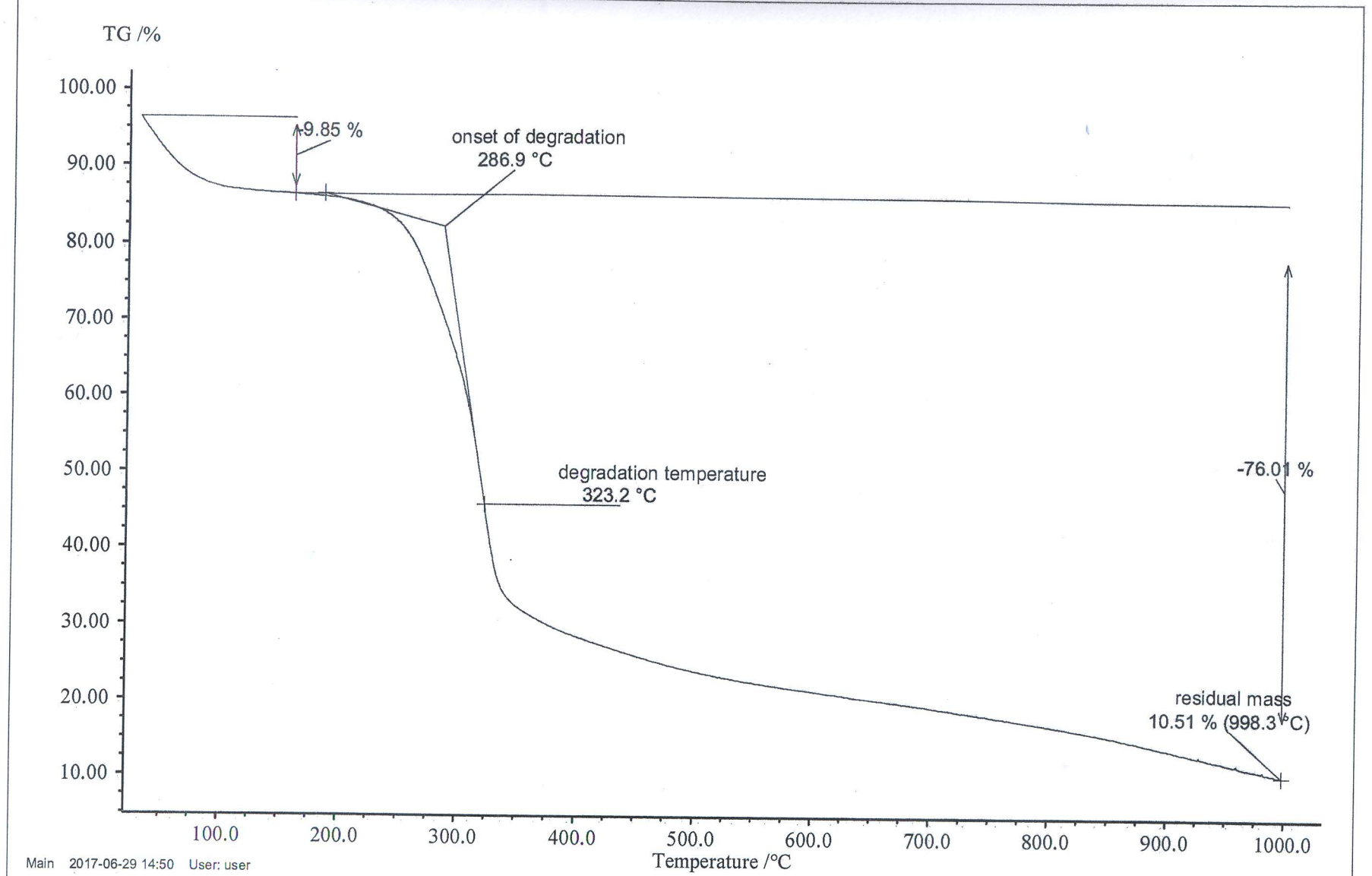
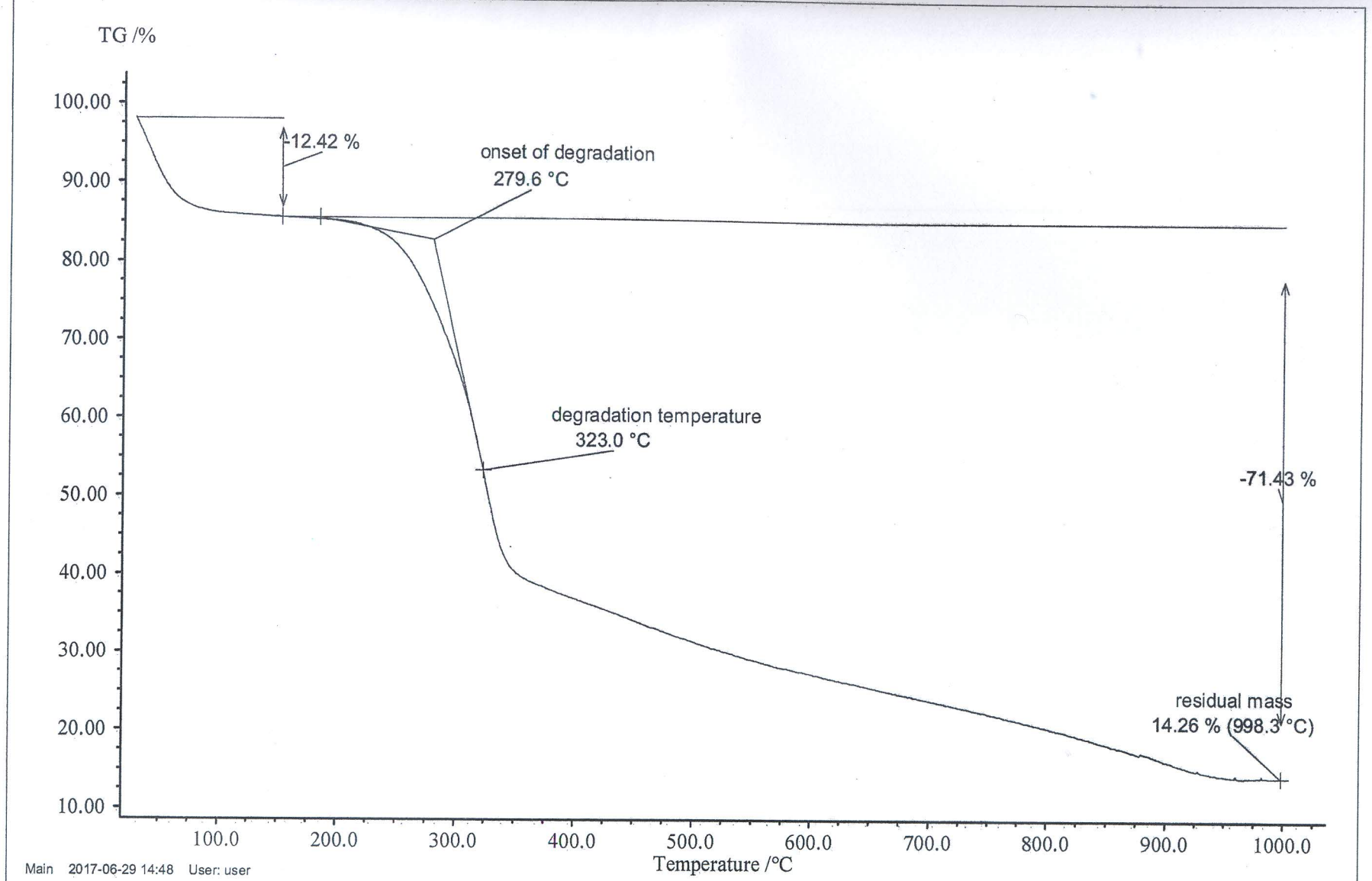


Figure 5: TGA for untreated Dracaena concinna



Instrument : NETZSCH TG 209F3 TGA209F3A-0254-L **File :** C:\NETZSCH\Proteus\data5\14-2-12 TG\Engineering\Drac. Concinna treated.ngb-dt6							
Project :		**Sample :**	Drac. Concinna treated	**Sample car./TC :**	TG 209F3 standard/P	**Atmosphere :**	-- / N2 / N2
Identity :	Drac. Concinna treated	**Material :**		**Sample mass :**	6.90 mg	**Corr/m. range :**	520/2000 mg
Date/time :	6/16/2017 11:17:24 AM	**Correction file :**	baseline 10K 4.7.14.ngb-bt6	**Mode/type of meas. :**	TG/sample with correction		
Laboratory :	Characterisation Lab	**Temp. calib. file :**	TCALZERO.TCX	**Segments :**	1/1		
Operator :		**Range :**	26/10.0(K/min)/1000	**Crucible :**	Al2O3		

Created with NETZSCH Proteus software

Figure 6: TGA for treated Dracaena concinna

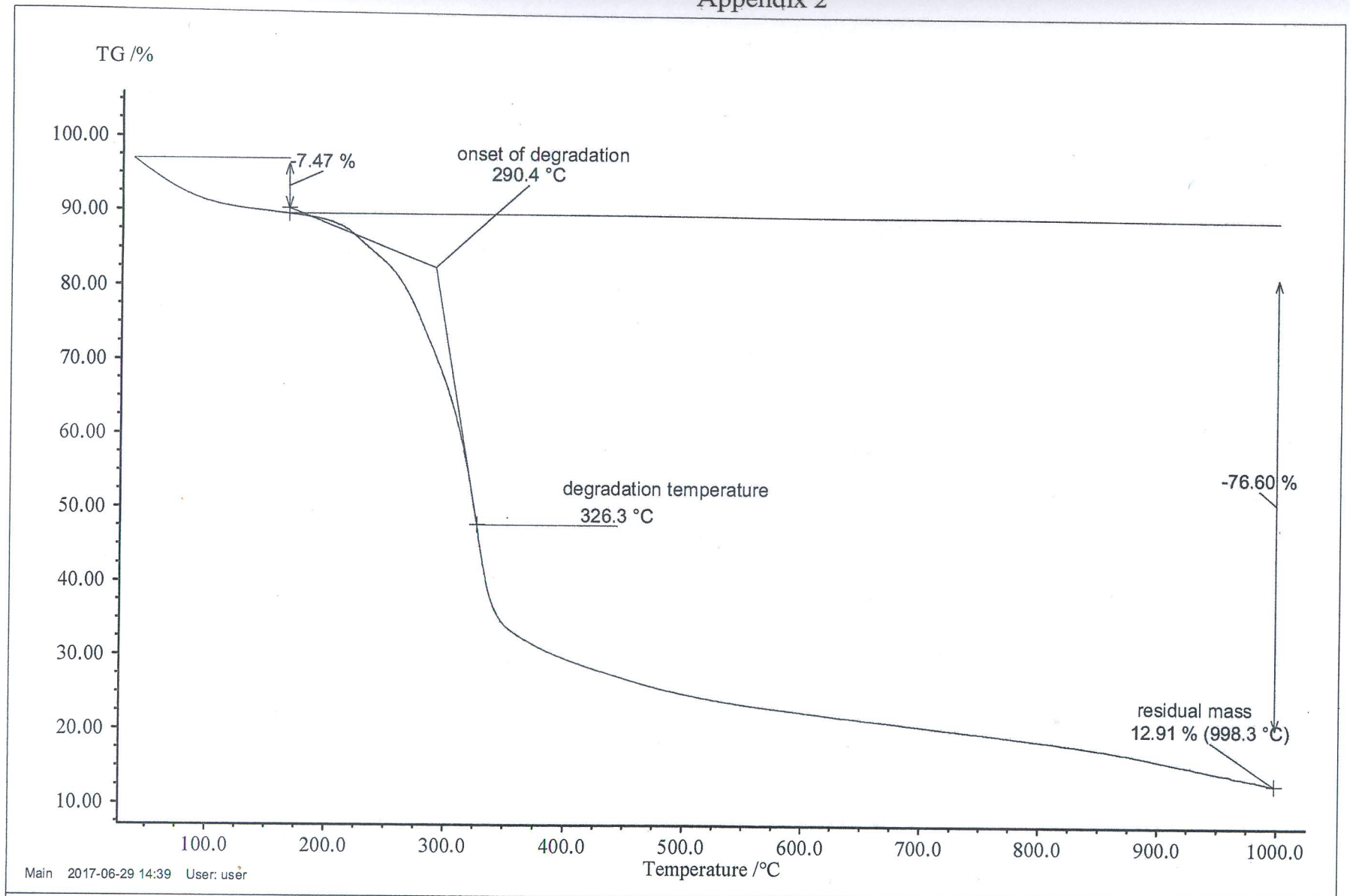


Figure 7: TGA for untreated Dracaena floribunda

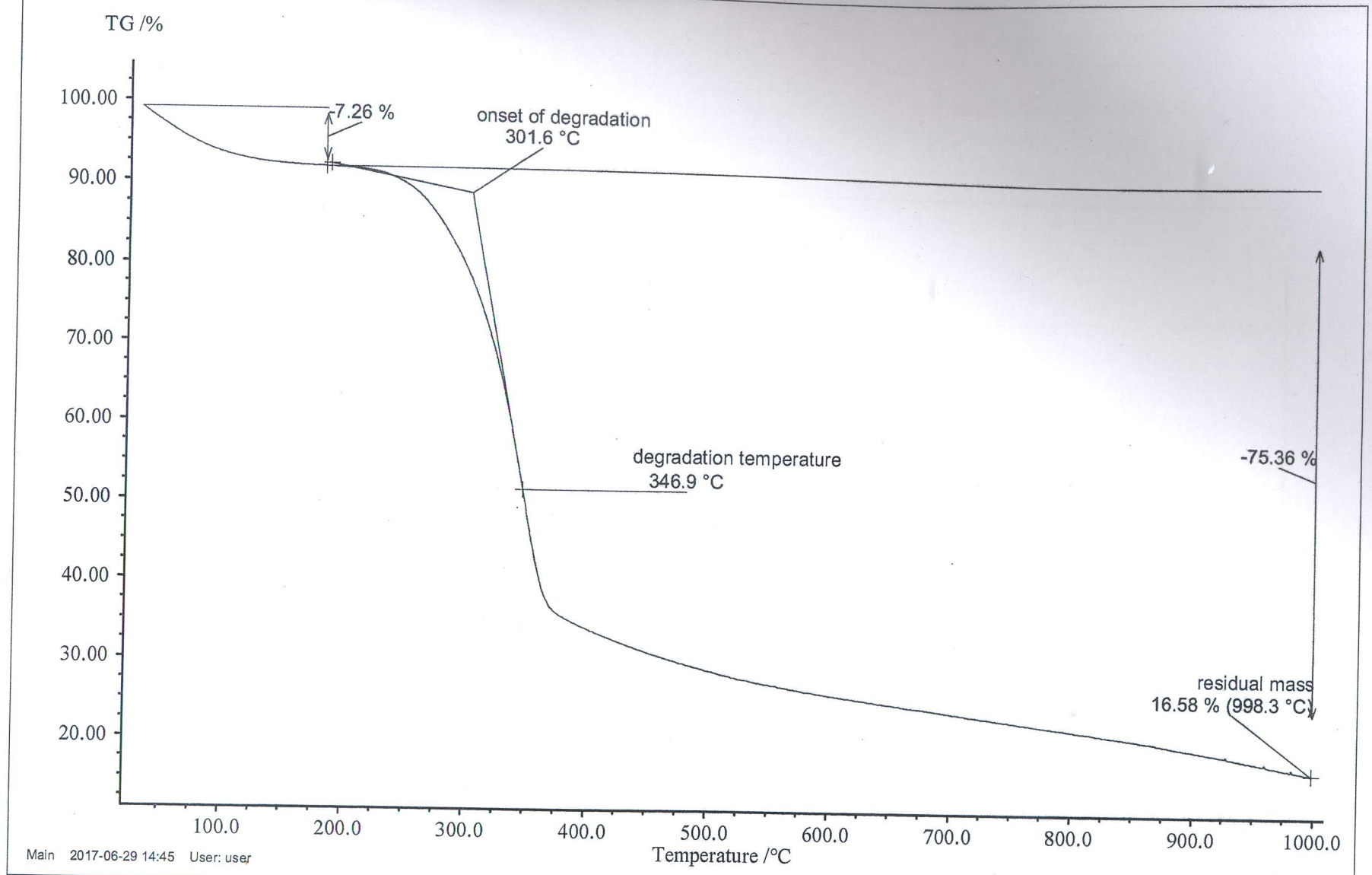


Figure 8: TGA for treated Dracaena floribunda

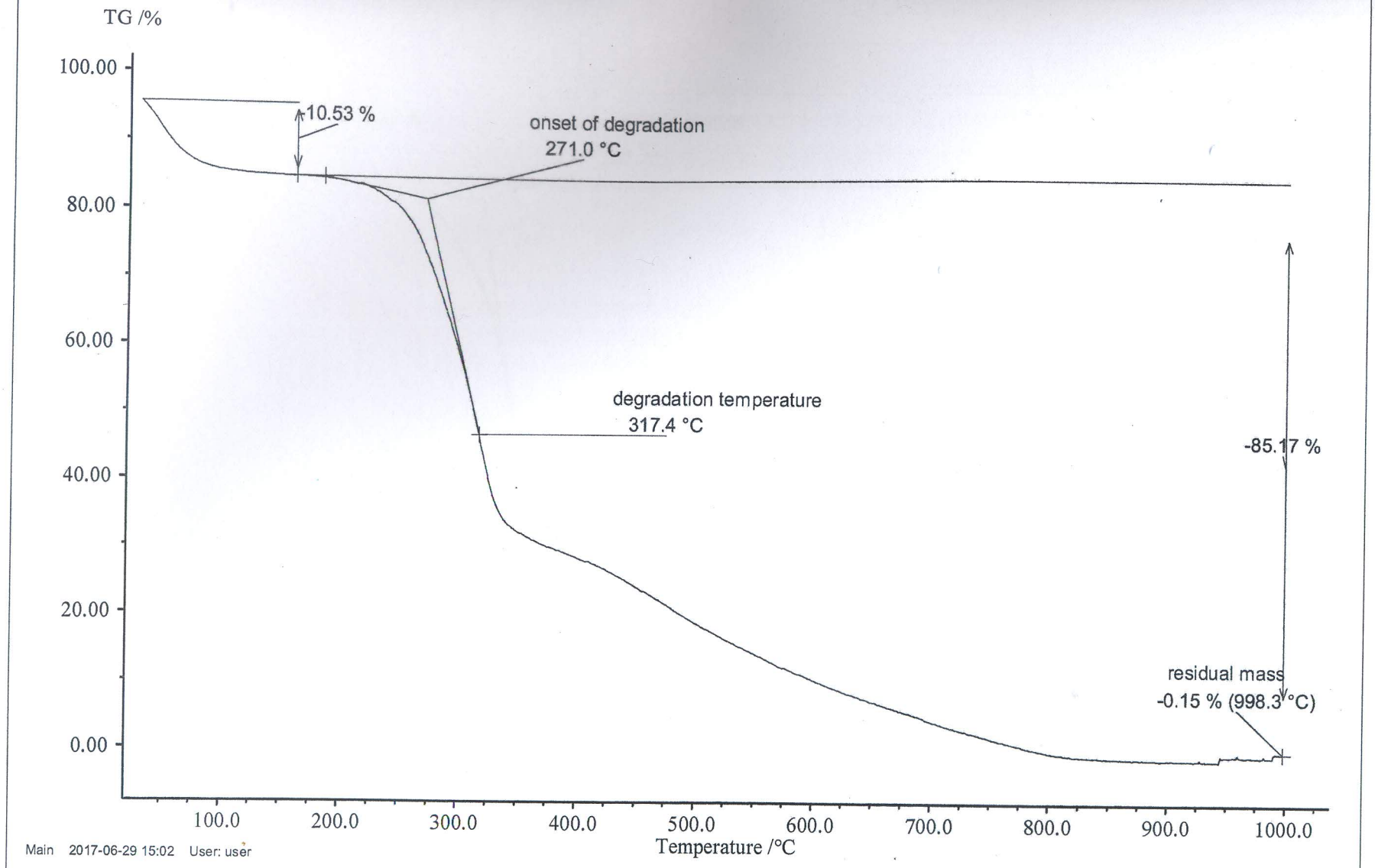
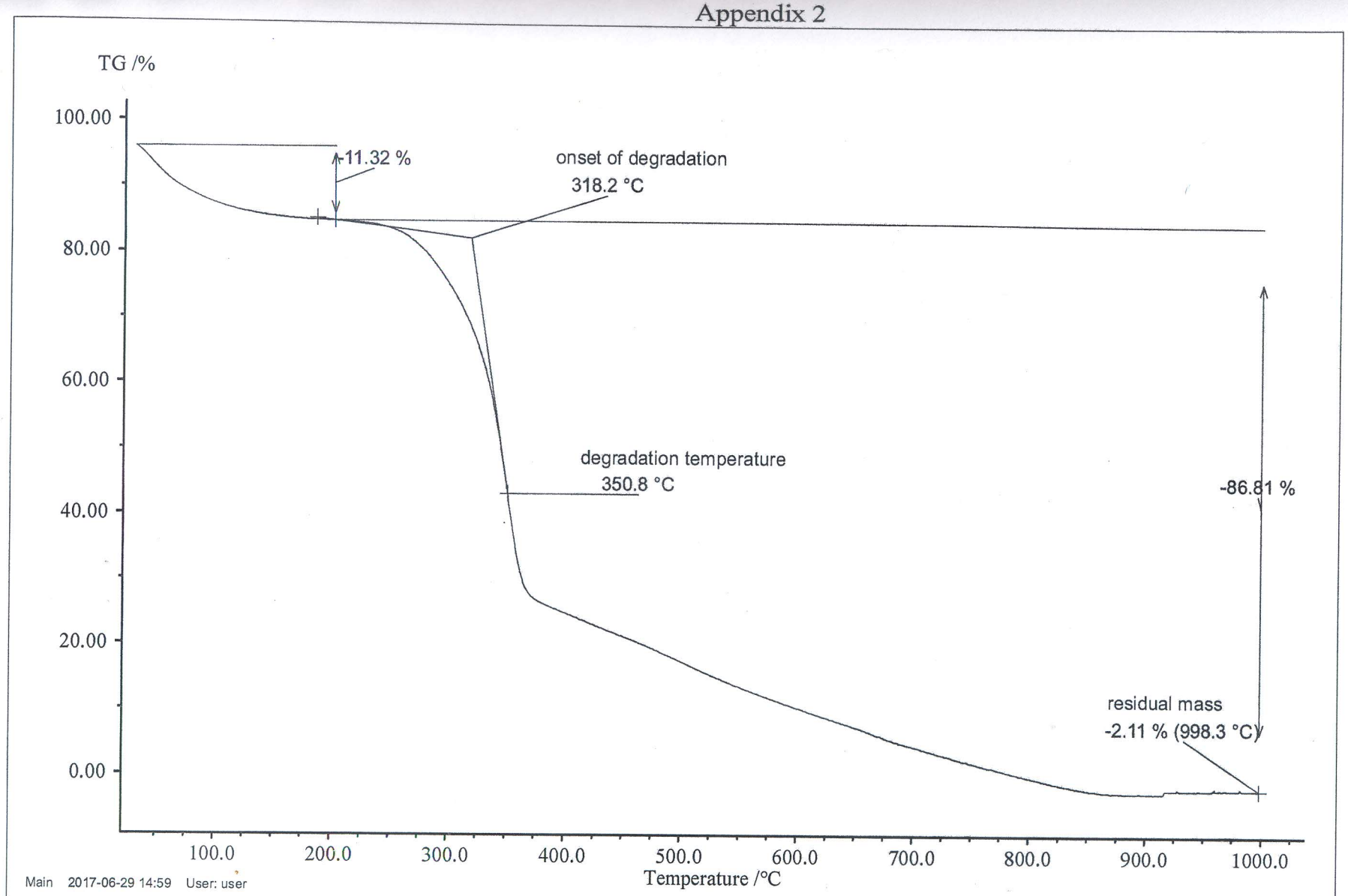


Figure 9: TGA for untreated Ravenala Madagascariensis



Instrument : NETZSCH TG 209F3 TGA209F3A-0254-L **File :** C:\NETZSCH\Proteus\data5\14-2-12 TG\Engineering\Ravenala treated.ngb-dt6							
Project :		**Sample :**	Ravenala treated	**Sample car./TC :**	TG 209F3 standard/P	**Atmosphere :**	-- / N2 / N2
Identity :	Ravenala treated	**Material :**		**Sample mass :**	7.08 mg	**Corr/m. range :**	520/2000 mg
Date/time :	6/15/2017 9:44:04 AM	**Correction file :**	baseline 10K 4.7.14.ngb-bt6	**Mode/type of meas. :**	TG/sample with correction		
Laboratory :	Characterisation Lab	**Temp. calib. file :**	TCALZERO.TCX	**Segments :**	1/1		
Operator :		**Range :**	26/10.0(K/min)/1000	**Crucible :**	Al2O3		

Created with NETZSCH Proteus software

Figure 10: TGA for treated Ravenala Madagascariensis