

**Date Palm Midrib Fibers as an Ecofriendly Reinforcement  
in Composite Materials**

BY

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*To all my beloved family*  
*To everyone who taught and guided me*

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## LIST OF ABBREVIATIONS

|       |   |                               |
|-------|---|-------------------------------|
| DPF   | : | Date Palm Fibers              |
| $E_f$ | : | Fiber's modulus of elasticity |
| $E_m$ | : | Matrix modulus of elasticity  |
| HFB   | : | Hard Fiber Board              |
| MDF   | : | Medium Density Fiberboard     |
| OSB   | : | Orientation Stranded Board    |
| RoM   | : | Rule of Mixtures              |
| TPB   | : | Thick Particle Board          |
| vol%  | : | Volume fraction               |
| wt%   | : | Weight percentage             |

## **ABSTRACT**

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Thesis Title: Date Palm Midrib Fibers as an Ecofriendly Reinforcement in Composite Materials  
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The goal of this work is to use date palm midrib fibers as an ecofriendly substitution for synthetic fibers in composite materials and to make it competitive with the commercial wood products. Natural cellulosic fibers, such as date palm fibers, have mechanical properties comparable to synthetic fibers. Saudi Arabia is one world's leading producers of dates and thus has an exorbitant number of date palms trees whose crop residue goes largely unused. Date palms have an annual pruning process which produce about 300,000 tons/year of agricultural residue that can be used to make value-added products. Date palms' midrib core is the raw material for this work because it is one of the most available by-products, available throughout the year, and has a dense packing of fibers. The fiber-extraction process entails boiling the midribs with water to soften the lignin, followed by hammering the midribs to separate the fibers from the lignin, and then drying the isolated fibers. The matrix portion of the composite in this work is an epoxy resin. After reinforcing the matrix with the date palms midrib fibers, samples were mechanically tested to compare mechanical properties of this methodology with prior literature establishing the significance of this methodology. For the composite, the average modulus of elasticity is

in the range of 3 GPa which is the requirement for the majority of the commercial wood products. A maximum value of 9 GPa was reached for the elastic modulus. This demonstrates the potential of this extraction technique and processing route that can be achieved with improvements suggested in the future work section. The tensile strength reached as high as 40 MPa and average value of 27 MPa which also meets commercial products requirements. Tests were also performed on single fibers in order to understand their properties for modeling purposes. For single fibers, the average tensile strength and modulus of elasticity values are 333 MPa and 17 GPa, respectively. Finally, single fiber pull-out test was performed to determine the interfacial shear strength between the fibers and matrix necessary for modeling the composite material. The interfacial strength ranged between 6 – 12 MPa. Results from this effort are compared to the literature and currently available commercial products and are found to have a promising outlook.

## ملخص الرسالة

الاسم كامل: راكان عبدالمحسن نايف العبدلي  
عنوان الرسالة: ألياف جريد النخل كتدعيم طبيعي في المواد المركبة.  
التخصص: هندسة ميكانيكية  
تاريخ الدرجة العلمية: شوال 1443 هـ

الهدف من هذا العمل هو استخدام ألياف جريد النخل كبديل صديق للبيئة للألياف الصناعية في المواد المركبة وأن يكون منافساً للمنتجات والألواح الخشبية التجارية المصنعة والمستوردة. الألياف السليلوزية الطبيعية، كألياف النخيل، لها خصائص ميكانيكية منافسة للألياف الصناعية. المملكة العربية السعودية أحد أكبر منتجي التمور في العالم، حيث فيها الكثير من النخيل التي لا يستفاد من بقاياها غالباً. النخيل لها عملية تنظيف وتقليم (تكريب) سنوية تنتج قرابة الثلاث مئة ألف طن سنوياً من المخلفات الزراعية التي يمكن الاستفادة منها لصنع منتجات ذات قيمة مضافة. في هذا العمل، الألياف المستخرجة من لب الجريد هي المادة الخام التي ستننتج منها المادة المركبة، وقد اختير لب الجريد لأنه من الأجزاء المتوفرة طول العام وبكميات كبيرة وفيها كثافة ألياف عالية. عملية استخراج الألياف تقوم على غلي قطع الجريد في الماء لتطرية اللجنين وإضعاف روابطه مع الألياف السليلوزية. بعد ذلك تطرق قطع الجريد لتفتيت اللجنين وعزل الألياف واستخراجها، ثم تجفيف هذه الألياف المستخرجة. المادة الرابطة المستعملة في تجهيز المادة المركبة في هذا العمل هي الإيبوكسي (الراتنج). بعد تدعيم المادة الرابطة بالألياف، اختبرت العينات لمقارنة الطريقة المتبعة في هذا العمل مع الأبحاث الأخرى من حيث الخواص الميكانيكية وتقييم فعاليتها. متوسط نتائج اختبار الشد للمادة المركبة هي: 3 جيجاباسكال لمعامل المرونة، وهي القيمة المطلوبة لغالبية المنتجات الخشبية التجارية. أعلى قيمة لمعامل المرونة كانت 9 جيجاباسكال وهي قيمة توضح الإمكانات التي يمكن الوصول إليها بطريقة الاستخراج والمعالجة هذه مع بعض التحسينات التي ستذكر في باب العمل المستقبلي. قوة الشد وصلت إلى 40 ميجاباسكال، وقيمة متوسطة هي 27 ميجاباسكال، وهي متوافقة مع متطلبات المنتجات التجارية. بالإضافة لذلك، أجريت اختبارات على الألياف مفردة وذلك لمعرفة خصائصها لأغراض النمذجة الحسابية. وصلت القيمة المتوسطة لمعامل المرونة لليفة الواحدة إلى 17

جيجاباسكال، وقوة الشد إلى 333 ميجاباسكال. أخيراً، أجري اختبار انتزاع الليفة لتحديد قوة الترابط البيني بين الليفة والمادة الرابطة من أجل نمذجة المادة المركبة حسابياً. قوة الترابط البينية تراوحت قيمها بين 6 إلى 12 ميجاباسكال. نتائج هذا العمل واعدة ومبشرة ومنافسة للمنتجات الخشبية التجارية ومقاربة لكثير من الأبحاث الأخرى.

# CHAPTER 1

## INTRODUCTION

### 1.1. Motivation:

An Ancient Arab proverb says, “anything that is free, its benefit is clear”. Therefore, if you can turn anything that is considered waste into a valuable product there is clearly a benefit. From this proverb, the idea of this work was born. The Kingdom of Saudi Arabia has about 31 million date palms trees that have an annual pruning process which produce about 300,000 tons/year of by-products [1], [2], a.k.a. crop residue, that is generally considered waste! What if this waste could be turned into value-added products that contribute to the national income in addition to the positive environmental effect? This was the research question pursued, and the answer is at the end of this work!

### 1.2. Objectives:

The idea of using date palms fibers is not new. The literature in this field began approximately two decades ago and a majority of it uses synthetic chemicals during the extraction process[3]–[12]. This led to the identification of the first objective of this work which is to minimize the use of chemicals as much as possible in all process levels to make an eco-friendly product. The second objective is to contribute to Saudi Arabia’s national economy by using the date palm residue to produce a high quality, low-cost products that can substitute imported ones such as Orientation Stranded Board (OSB), Medium Density Fiberboard (MDF), and Plywood.

### 1.3. Methodology:

This work followed the following general methodology to make the final product:

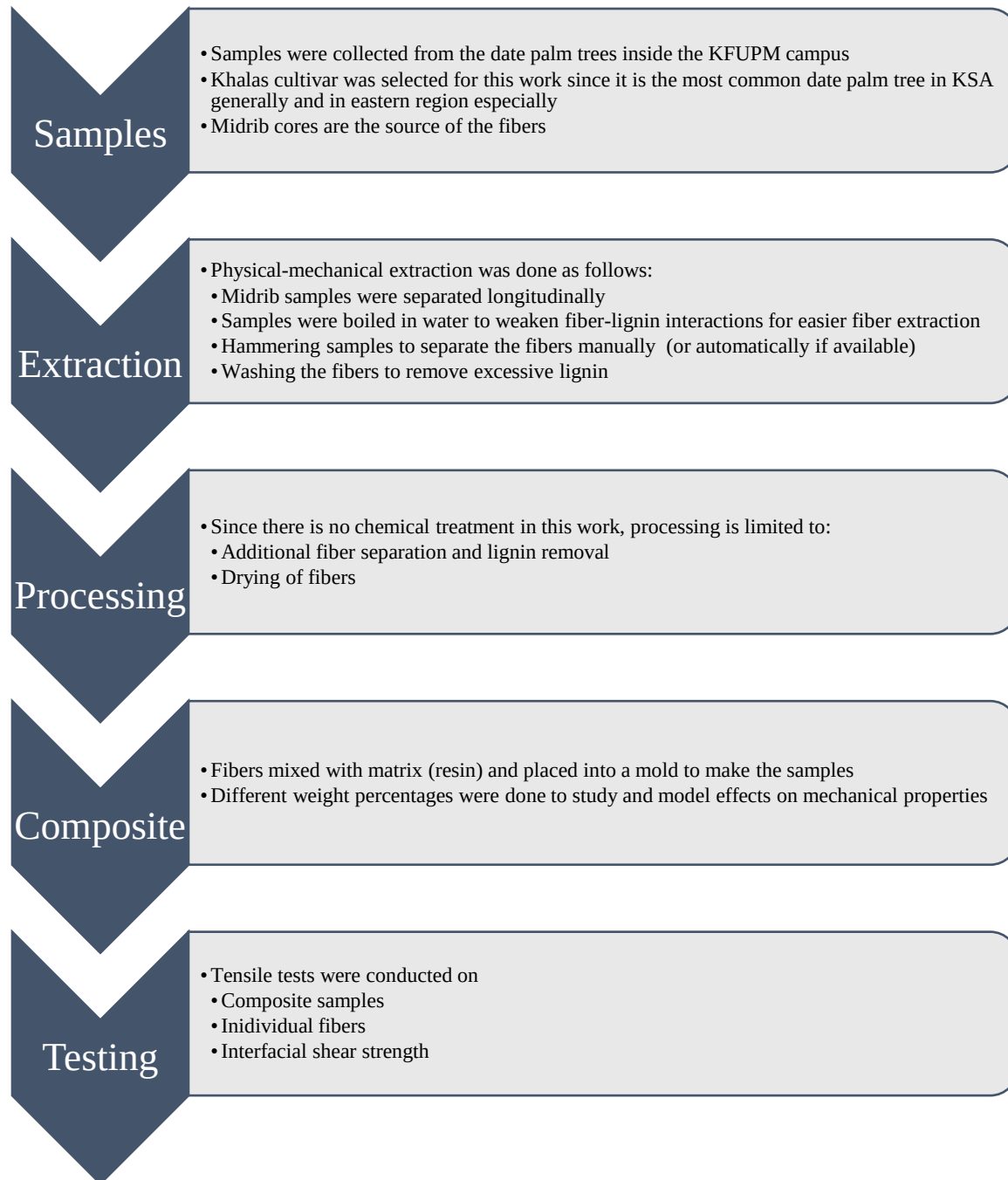


Figure 1: Methodology chart

## **CHAPTER 2**

### **BACKGROUND**

#### **2.1. Composite Materials**

Composite materials are a heterogeneous combination of two or more different materials mixed at the macro-scale in which each material keeps its integrity. The overall goal of composite material architecture is to combine advantages of each individual component such as high strength with light weight making a single material that has a high strength to weight ratio [13]–[16]. In general, composite materials are made of two main components, the reinforcement and matrix. The matrix can be considered as the housing that gives the composite its shape, which holds the reinforcements together and distributes applied load between them. Composite materials are classified based on the matrix type which is either metal, ceramic, or polymer. The reinforcement component is embedded in the matrix in the form of particles, fibers or even flakes and its role is to absorb, and resist applied stresses on the matrix to improve the composites mechanical properties such as elastic modulus and tensile strength. This gives the composite materials many variables to modify which makes this an ever-evolving field of research. Type of the matrix and fibers, fibers to matrix ratio, fibers size, and fibers orientation are all variables affecting the composite properties [4], [7]–[9], [17].

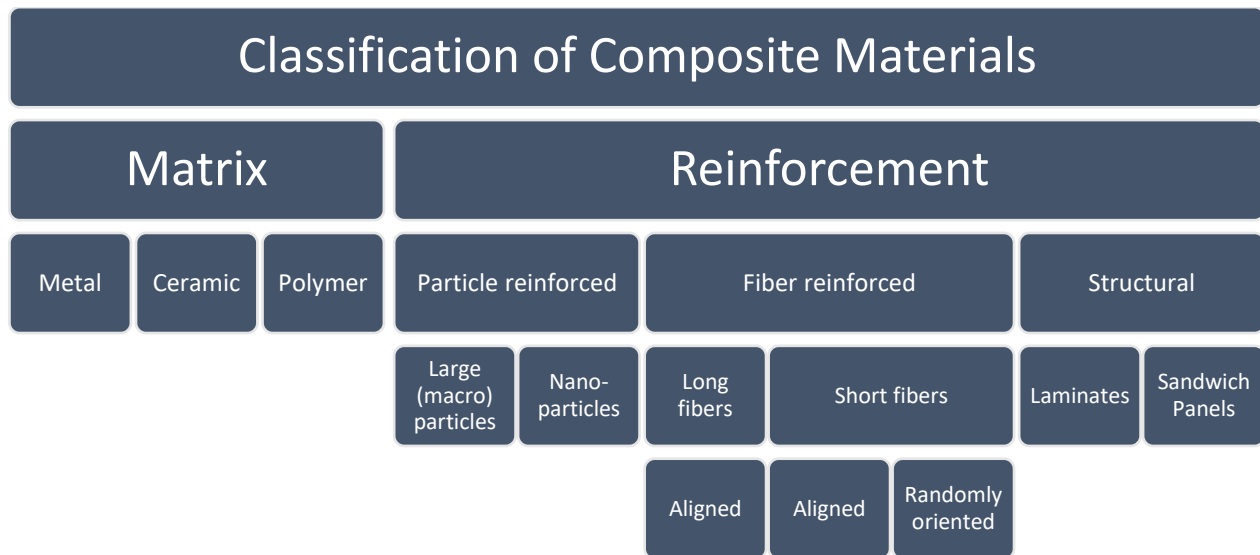


Figure 2: Classifications of Composite materials

Composite materials first emergence in recorded history was in ca. 1500 BC. This original example of a composite material was of straw reinforced clay bricks that ancients used for building. Since that time until the early 20<sup>th</sup> century, composite materials were not classified as a separate class of materials. In the early 20<sup>th</sup> century and after the development of the plastics, the need for reinforcement materials to give the plastic better mechanical properties appeared. After the introduction of fiberglass in 1935, plastic was reinforced with these fibers producing a composite with light weight and high strength that is used in variety of applications. From that time, composite materials were classified as a different type of material and developments in this field keep cause the number of materials in this class to increase at a rapid rate [18].

This work focuses on polymer based, fiber-reinforced composite materials. More specifically, the fiber phase consists of long, aligned fibers.

### 2.1.1. Theories of Composites

The main advantage of composite materials is the ability to join materials with contrasting properties that cannot be achieved by monolithic materials. With composite materials, high strength can be attained with light weights, higher toughness, and with high hardness. As mentioned before, composite materials have many factors that influence a composite's properties. Fiber-matrix interfacial bond, fibers volume fraction, length, and orientation play vital roles in the determination of a composite's properties.

The interfacial bond between fibers and matrix is the single most important factor in transmission of the applied load from the matrix to fibers. If there is no interfacial bond, there is no composite and every material will work independently. With weak interfacial bond, the load will not be transmitted to the fibers well leading to delamination between the fibers and the matrix phases. Delamination, or fiber pullout, occurs when the fibers slide in the matrix leading to voids that weaken the matrix. **Figure 3** shows a load transmitting from the matrix to the fibers and the deformation pattern associated with it when there is perfect transfer of the applied load from the matrix to the fiber, i.e. without fiber pullout [13].

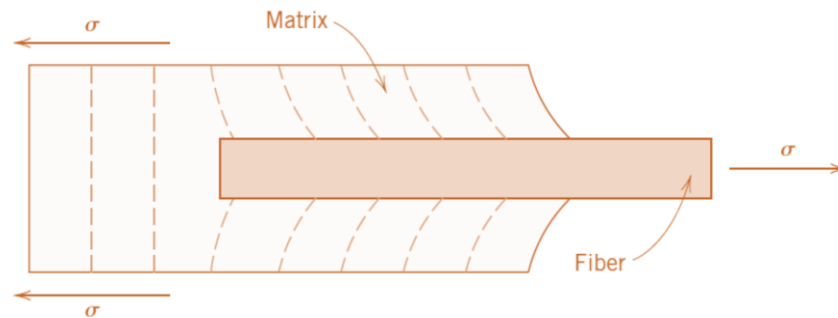


Figure 3: The deformation pattern in the matrix surrounding a fiber that is subjected to an applied tensile load [13].

This figure leads us to the second factor which is the fiber critical length. At the fiber extremes, there is less deformation which means that a critical fiber length leads to more effective load transmission. This critical length is affected by fiber tensile strength ( $\sigma_f^*$ ), fiber diameter ( $d$ ), and the fiber-matrix interfacial bond strength ( $\tau_c$ ), or the matrix shear strength, whichever is smaller. The fiber's critical length can be calculated by Kelly and Tyson equation [13], [19]:

$$l_c = \frac{\sigma_f^* d}{2\tau_c} \quad (1)$$

If the fiber is longer than the critical length, then there is better load transmission leading to better reinforcement of the overall composite. Fibers with length much longer than the critical length (about 15-times critical length) are considered as long fibers or continuous fiber composites. Shorter than this are called short fiber or discontinuous fiber composites.

Fiber volume fraction and orientation effect can be found using the “Rule of Mixtures” (RoM). RoM is a mean field theory that is used to calculate a composite's modulus of elasticity, tensile strength, and density. For the composite modulus, it should fall between upper and lower boundaries based on the direction of the applied load. If the applied load is in the direction of the fibers, then the value will be the upper bound. On the other hand, if the load is perpendicular to the fiber's direction, then the fibers reinforcement will be less effective leading to lower value of the modulus. Modulus of elasticity for continuous fibers and longitudinal loading (upper bound) can be calculated by:

$$E_{cl} = E_m V_m + E_f V_f \quad (2)$$

And for continuous fibers and transverse loading (lower bound) by:

$$E_{ct} = \frac{E_m E_f}{V_m E_f + V_f E_m} \quad (3)$$

where  $E_m$  and  $V_m$  are the elastic modulus and volume fraction of the matrix, respectively,  $E_f$  and  $V_f$  are elastic modulus and volume fraction of the fibers, respectively. The derivation of equations (2) and (3) is in the appendix (A).

Since RoM is based on volume fraction (vol%) while weight percentage (wt%) is the mixing scale used here, the conversion from wt% to vol% and vice versa are:

$$vol\%_f = \frac{wt\%_f / \rho_f}{wt\%_f / \rho_f + wt\%_m / \rho_m} \times 100 \quad (4)$$

$$wt\%_f = \frac{vol\%_f \times \rho_f}{(vol\%_f \times \rho_f) + (vol\%_m \times \rho_m)} \times 100 \quad (5)$$

where  $\rho_f$  is the density of the fibers and  $\rho_m$  is the density of the matrix. Using these definitions, the rule of mixtures can be rewritten as equation (6) for upper bound and equation (7) for lower bound:

$$E_c = E_m + (E_f - E_m) \left( \frac{1}{1 + \left( \frac{wt\%_m \times \rho_f}{wt\%_f \times \rho_m} \right)} \right) \quad (6)$$

$$E_c = \frac{E_m \times E_f}{E_f + (E_m - E_f) \left( \frac{1}{1 + \left( \frac{wt\%_m \times \rho_f}{wt\%_f \times \rho_m} \right)} \right)} \quad (7)$$

RoM in this form will be used in later sections of this thesis.

## 2.2. Natural Fibers

Natural fibers are classified based on their source and can be split into three categories, mineral, animal (or protein), and plant (or cellulose), see Figure 4. Each type has its own advantages and usages. Since this work is about using date palms fibers, the details will be about plant fibers mainly. Plant fibers are also called cellulosic fibers because cellulose is the content that gives the fiber its desired properties. Cellulose is the most common biopolymer which consists of a long carbohydrate chain. In addition to cellulose, there are hemicellulose and lignin which work as a “glue” that bonds and supports cellulose structures together. All these components are complex organics that constitute the cell walls of the plants [20]. Figure 5 and Figure 6 show the fibers structure from the macro to the micro level. In the macro scale, each fiber consists of a bundle of many vascular fibers. Then in the micro level, the wall structure of the vascular fiber consists of tiny fibrils. Each fibril consists of many cellulosic chains.

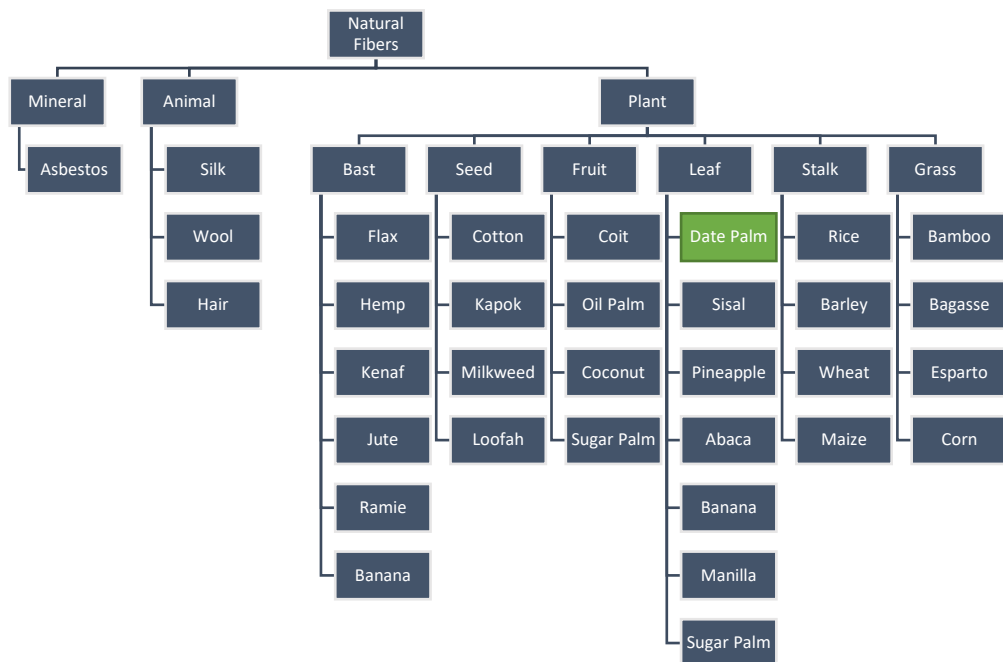


Figure 4: Types of Natural Fibers, date palms are highlighted in green adapted from [7], [9], [21].

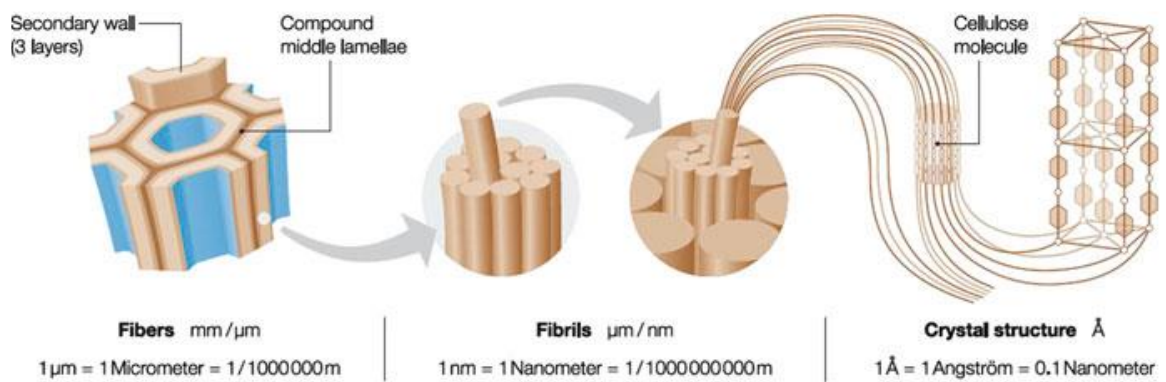


Figure 5: Representation of different fiber levels [22].

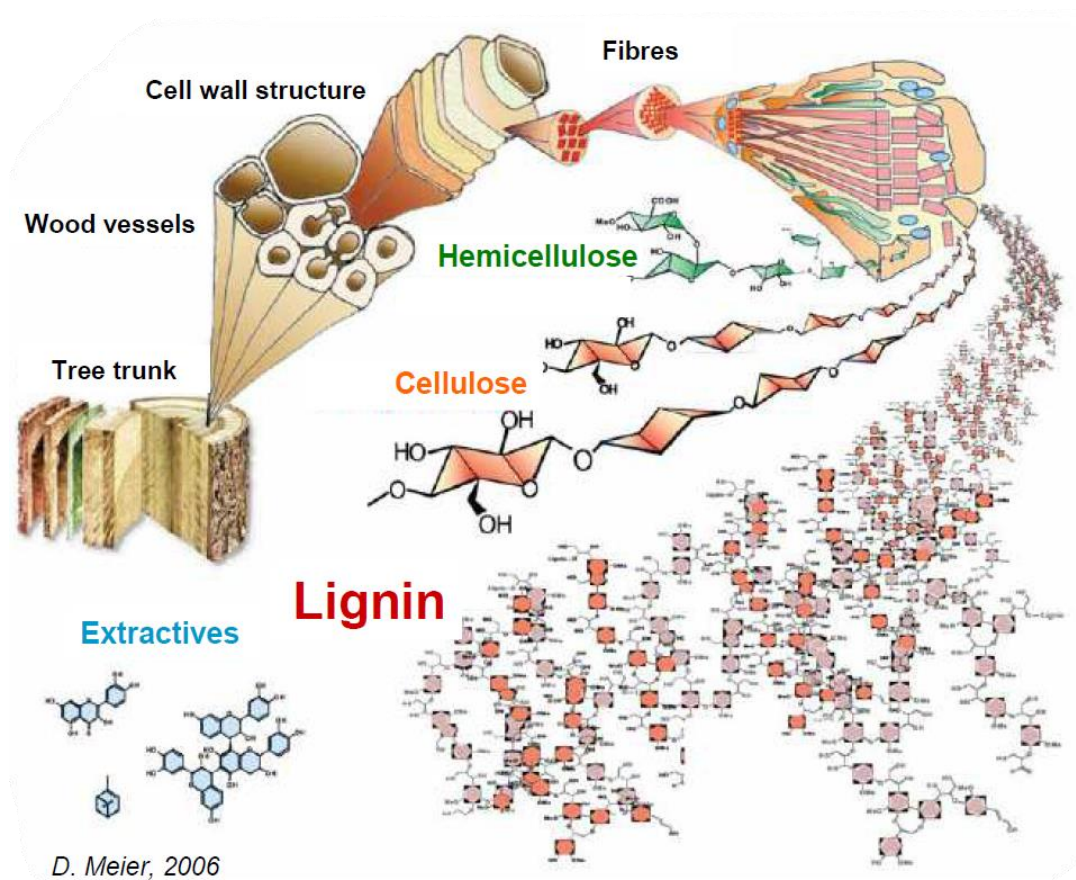


Figure 6: Plants fiber structure [© Per Hoffmann, Oskar Faix and Ralph Lehnen]

In the last two decades, the world noticed the side effect of the synthetic products on the human health and on the environment. In response, there has been a shift to products with less side effects. There are many approaches to reduce side effects; one of which is by using natural fibers to make composite materials that have properties comparable to the synthetic ones. This approach joins the advantages of composite materials with the environmental friendliness of the natural fibers [20].

There are diverse types of natural fibers, and researchers are working to utilize natural fibers to create value-added composites [19]–[31]. The best economical and ecofriendly approach is by utilizing fruiting plants' by-products to extract fibers. In this way, the plant can be used for its fruits and its residue for value-added products. The crop residue is used in a consumer product rather than disposing of or burning them, which affects the environment, and the lands are not consumed by fruitless plants. In addition to the reduction in carbon content in the atmosphere due to the plant's consumption of CO<sub>2</sub> [33], [34]. Therefore, growing and utilizing fruiting plants' by-products gathers all these benefits.

Advantages of using natural fibers approach are:

- Ecofriendly and biodegradable
- Renewable and sustainable
- Widely available as by-product with low cost
- Low density
- Good specific mechanical properties

On the other hand, using natural fibers have some disadvantages such as:

- Less control on the mechanical properties
- Production of some specific natural fibers is less than the demand
- Moisture absorption affinity

To overcome these drawbacks, researchers are utilizing all available natural fibers, treating them to improve their properties, and making the available fibers meet the demand. The major drawback is the hydrophilic nature of the natural fibers which limit their applications. Moisture absorption results in reduction of interfacial adhesion between the fibers and matrix which levels down the mechanical properties. The figure below is a schematic illustration of the debonding process due to moisture absorption [21], [22].

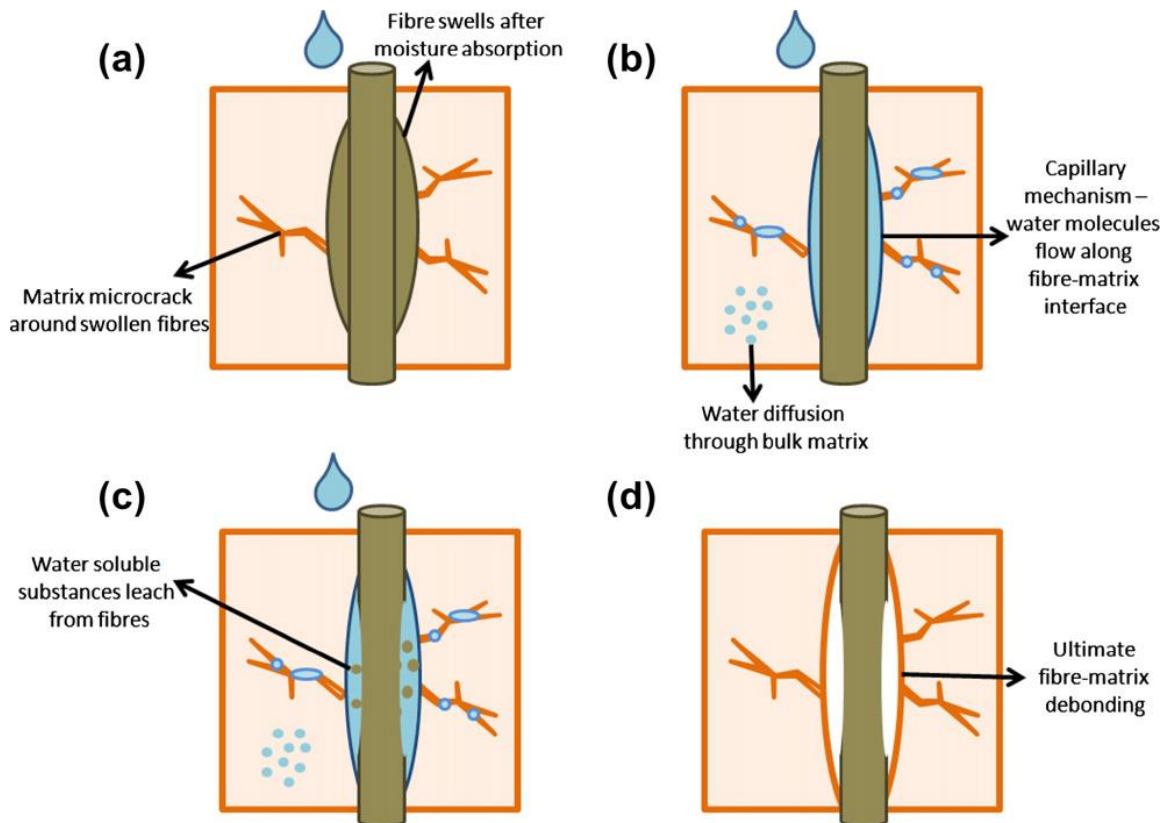


Figure 7: Fiber debonding mechanism due to moisture absorption [21].

### 2.2.1. Date Palm Fibers

Here in Saudi Arabia, date palms are the best example of this approach. The number of date palms in Saudi Arabia in 2019 was 31,234,155 date palm trees as per Saudi General Authority for Statistics [1]. From all these date palms, Khalas cultivar is the most planted palm tree with 7,903,510 Khalas palm trees representing 25% of all the date palms in Saudi Arabia [1].

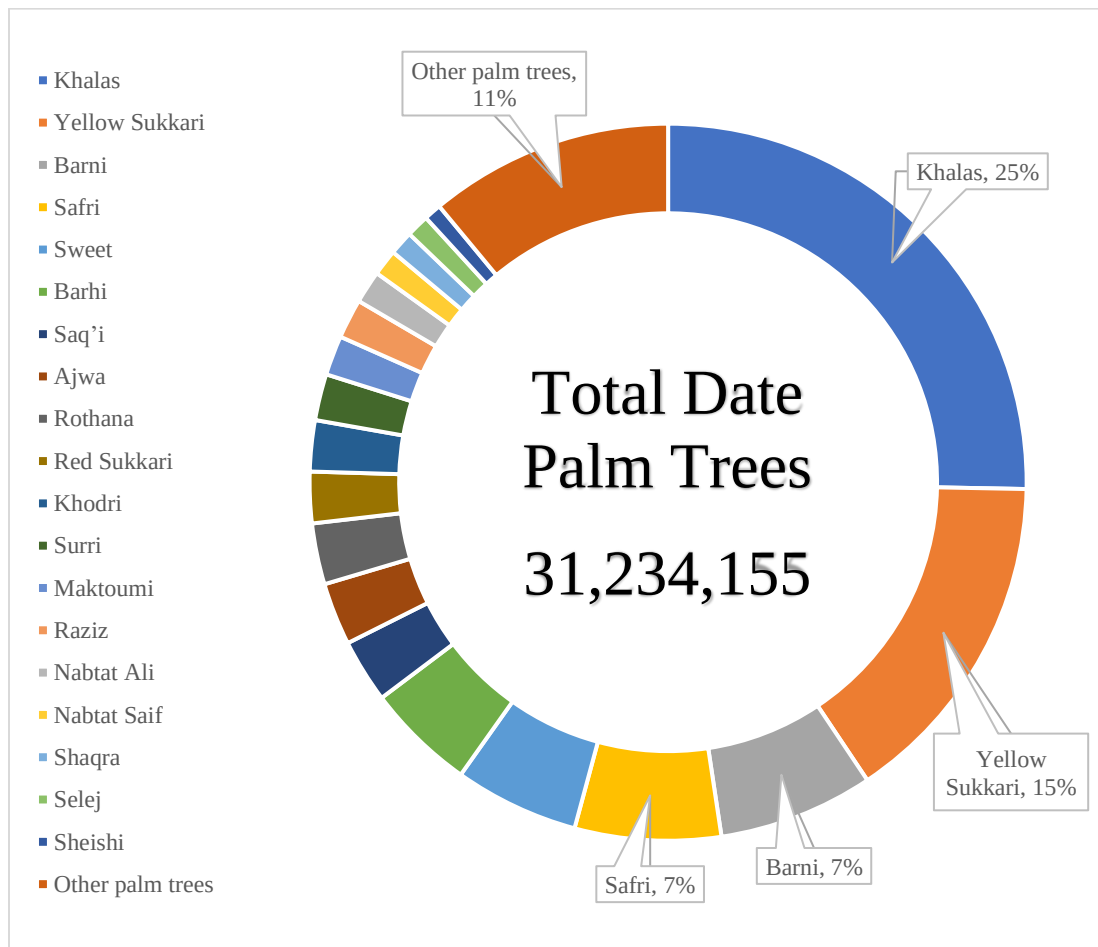


Figure 8: Date Palm trees total numbers and percentages of the most common cultivars adapted from [1]

These date palm trees have an annual pruning process which produces several tons of by-products. As per a report from Saleh Al-Rajhi endowment, the average weight of the pruning process per date palm tree is 10 kg, so the total weight of the by-products of all the date palms in Saudi Arabia is about 300,000 tons/year [2]. For this work, only the date palm midrib core fibers are used since it is available all over the year and almost always considered as waste. As per the previous report, the average weight of the midrib is 0.3 kg and the average number of midribs per tree is eight midribs. This produces about 72,000 tons/year of midribs only. This amount of agricultural residue is an enormous potential for valuable products instead of considering it as agricultural wastes.

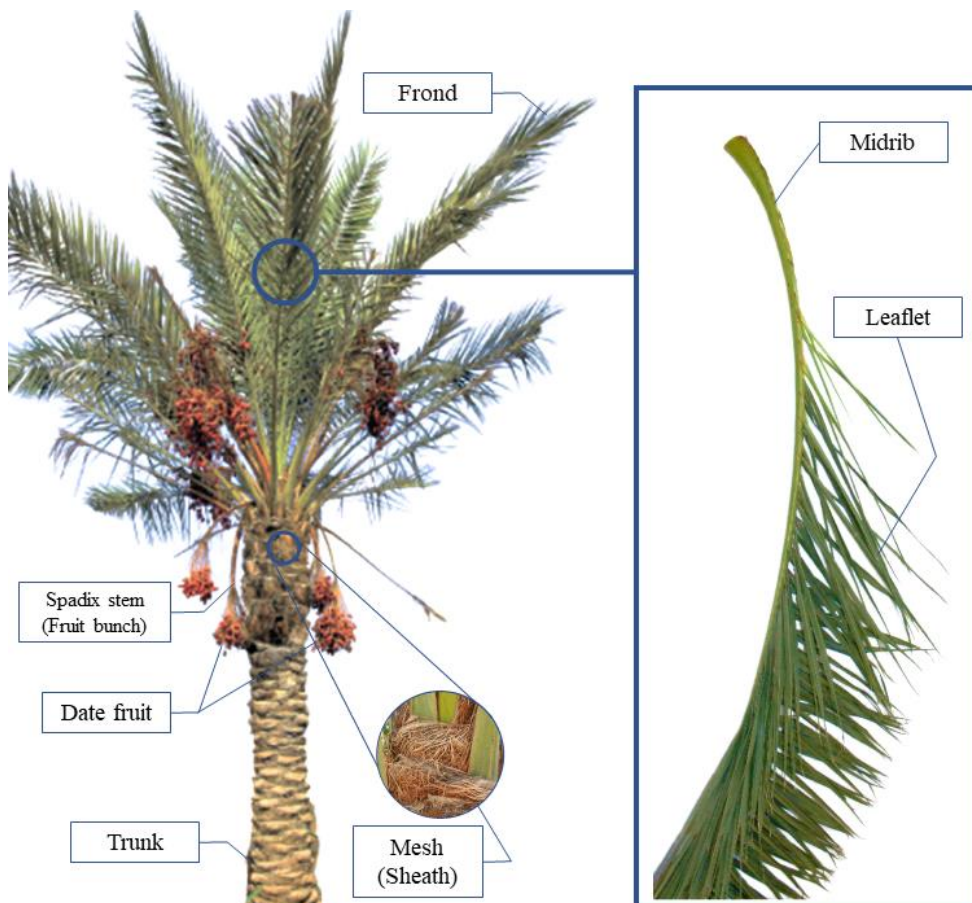


Figure 9: Date palm parts

## CHAPTER 3

### Literature Review:

In the last two decades, the world started to look towards green materials that solve issues created by synthetic materials. Combining this approach with the need for high performance and special property materials led to the use of natural fibers as a reinforcement in composite materials. This fulfills needs in the most economically and efficiently way. Over this period, copious amounts of scientific research have been published supporting this approach and proving the ability of natural fibers to substitute synthetic ones with comparable properties.

Date palms caught scientists' interest because it is a fruiting plant that has large by-product with high cellulosic fibers content. Because of that, many research studies have been done that utilize these fibers.

❖ Extraction methods for date palm fibers are classified into 4 categories [35]:

- **Physical extraction:**

This process includes pressure explosion, ultrasound-based, and microwave-based processes for fiber extraction.

- **Mechanical extraction:**

It is either done manually by knives or hammers, or by machines such as decorticator, hammermill, rolling, etc.

- **Chemical extraction:**

Extraction of fibers is achieved by alkaline solutions such as sodium-hydroxide (NaOH), calcium-hydroxide (KOH), etc.

- **Biological extraction:**

This process is known as retting, and it was used centuries ago. It relies on the long-time microorganism's activities to extract the fibers. There are three types of retting process. The first one is by lying the plant parts on the ground subjecting them to the climate conditions. The second type is similar to the first one, but the parts are immersed in water. The third type is a modern process which is achieved by using enzymes to separate the fibers.

A combination of these extraction processes is usually used. Biological extraction is a time-consuming process, except for enzymatic retting, so it is rarely used these days with the presence of the other processes which have faster extraction time. From the literature, the most common extraction methods are the mechanical and the chemical or their combination. Midani *et al* [12], in their work they used a combination of chemical and mechanical extraction. They cut the date palm midrib into 30cm long samples then immersed those samples in alkaline solution (NaOH) for different durations under different temperatures. After that, samples were squeezed by rolling machine to separate the fibers. Saadaoui *et al* [36], used a hammermill with 3 mm diameter mesh to produce the fibers. AlMaadeed *et al* [37], used manual mechanical extraction in addition to chemical processes. Chikhi *et al* [38], they used mechanical crusher to produce small fibers. Yousif *et al* [10], used date palm mesh (sheath) in their work which exists in fiber form. The extraction method they used was manual and chemical. As per Elseify *et al* review paper

[39], it can be seen that the majority of the used extraction methods are mechanical and chemical.

❖ Second part of this literature review is about the physical, chemical, morphological, and mechanical properties of the date palm fibers.

- **Physical properties of date palm fibers:**

Based on the review papers by Elseify *et al* [7], [39], it was noticed that the extraction method affects the physical properties of the fibers. Fibers extracted mechanically have shorter length and higher diameter leading to lower aspect ratio values. The table below shows the ranges of physical properties from the review papers based on the extraction process.

Table 1: Physical properties of date palm fibers [7], [39].

|                                            | <b>Mechanical</b> | <b>Chemical</b> |
|--------------------------------------------|-------------------|-----------------|
| <b>Length (mm)</b>                         | 1.1 – 1.69        | 1.01 – 300      |
| <b>Diameter (μm)</b>                       | 12.79 – 510       | 10 – 100        |
| <b>Aspect ratio</b>                        | 66.3 - 95         | 45 – 3000       |
| <b>Bulk density (g/cm<sup>3</sup>)</b>     | 0.13 – 0.3        |                 |
| <b>Absolute density (g/cm<sup>3</sup>)</b> | 1.178 – 1.324     |                 |

- **Chemical properties of date palm fibers:**

Natural fibers generally consist of cellulose, hemicellulose, and lignin as was discussed in Chapter 2. Cellulosic fibers are targeted due to their mechanical properties. Generally, as the cellulose content in the fibers increase, the mechanical

properties improve. On the other hand, hemicellulose and lignin are considered as natural polymers that hold the cellulosic fibers together and must be removed in order to free the cellulosic fibers. The content percentage of each component is important to determine the fiber purity and mechanical properties. From previous review papers [7], [39], it was noticed that midrib and mesh have the highest cellulosic fiber content. Based on the date palm cultivar, cellulose content for midrib ranges from 41.5 to 69.3% [39]. Also, it was noticed that there is a variation in composites between date palm cultivars.

- **Morphology of date palm fibers:**

From literature in the previous review papers [7], [9], it was noted that date palm fibers have a vascular bundle shape. However, there is variation in the shape of the fibers based on which part of the date palm the fibers were extracted. These vascular bundle fibers are embedded in the lignin and consist of smaller micro-fibers (~12  $\mu\text{m}$  in diameter) held together by the hemicellulose. **Figure 10** from Midani *et al* work [12] represents the morphology of the midrib fibers. It shows the cross section of the midrib fibers and the vascular nature of the fibers. The extraction method plays significant role in the morphology and the surface smoothness of the fibers. Also, it was noticed that the chemical treatment affects fibers morphology by removing impurities and remaining lignin.

- **Mechanical properties of date palm fibers:**

Based on review papers [7], [39], it was observed that the extraction process and the chemical treatment has major influence on the mechanical properties of the fibers same as the morphology of the fibers. The standard for testing a single fiber

is ASTM D-3822 [40]. Unfortunately, there are no reliable sources to compare all the research results with since researchers do not mention their testing method [39]. However, the results of the mechanical tests vary based on the source of the fibers (which part of the date palm tree), extraction method, and treatment. **Table 2** shows mechanical properties of different types of date palm fibers [7], [39]. More literatures are used for comparison in the results chapter. These values show a high potential of the date palm fibers to be used as a reinforcement for the polymeric materials to be used in different applications.

*Table 2: Mechanical properties of different types of date palm fibers [7], [39].*

| <b>Fiber Type</b>  | <b>Treatment</b> | <b>Tensile strength<br/>(MPa)</b> | <b>Modulus of<br/>Elasticity<br/>(GPa)</b> |
|--------------------|------------------|-----------------------------------|--------------------------------------------|
| <b>Midrib</b>      | Untreated        | 11.4 – 208                        | 10 – 30                                    |
|                    | NaOH             | 105 – 452.8                       | 1.59 – 10.5                                |
| <b>Spadix stem</b> | Untreated        | 117                               | -----                                      |
|                    | NaOH             | 680 – 850                         | 13 – 150                                   |
| <b>Leaflet</b>     | Untreated        | 32.43 – 225                       | 4.6 – 5.2                                  |
|                    | NaOH             | 33.33 – 1246.7                    | 5.5 – 26.1                                 |
| <b>Mesh</b>        | Untreated        | 233 – 5359                        | 0.178 – 7.15                               |
|                    | NaOH             | 215.8 – 7073                      | 0.2626 – 11                                |

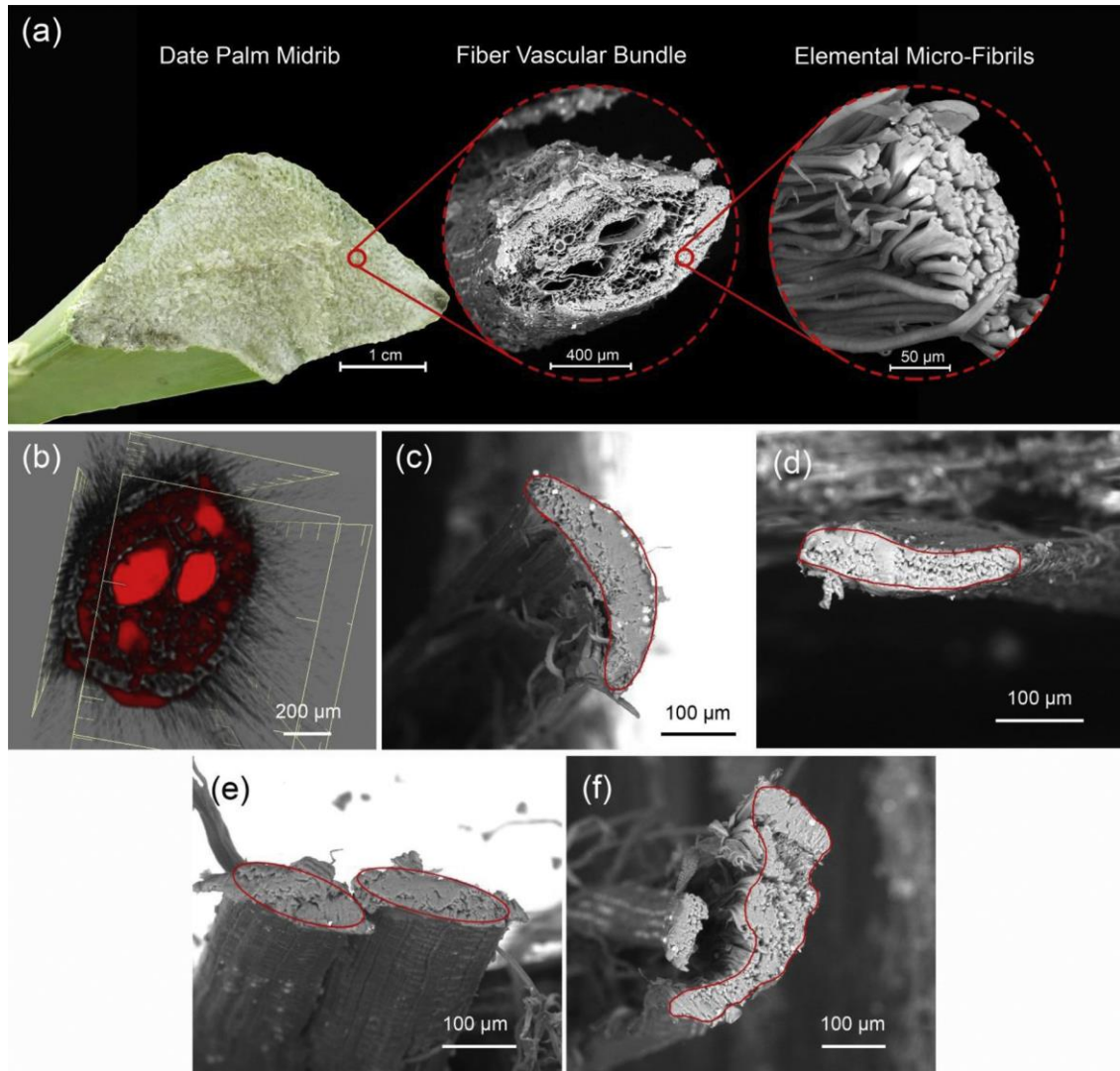


Figure 10: This figure from [12] shows that; (a) fibers of the date palm midrib, (b) scan of fibers vascular bundle showing the hollows in the fiber (c) "C" cross section fibers, (d) flaky cross section fibers, (e) elliptical cross section fibers, and (f) irregular cross section fibers.

## CHAPTER 4

### EXPERIMENTAL WORK

In this chapter, all the processes from the midrib collection until the composite testing are going to be discussed. The following graph summarized the experimental process.



Figure 11: Summary of the experimental work

#### 4.1. Materials

Based on the details in Chapters 2 and 3, Khalas cultivar were selected to extract the fibers from its midrib core. Khalas fronds were collected from date palm trees inside King Fahd University of Petroleum and Minerals (KFUPM) campus. Frond leaflets were cut out and only the midribs were used and cut to pieces of 10 cm long for the extraction process.

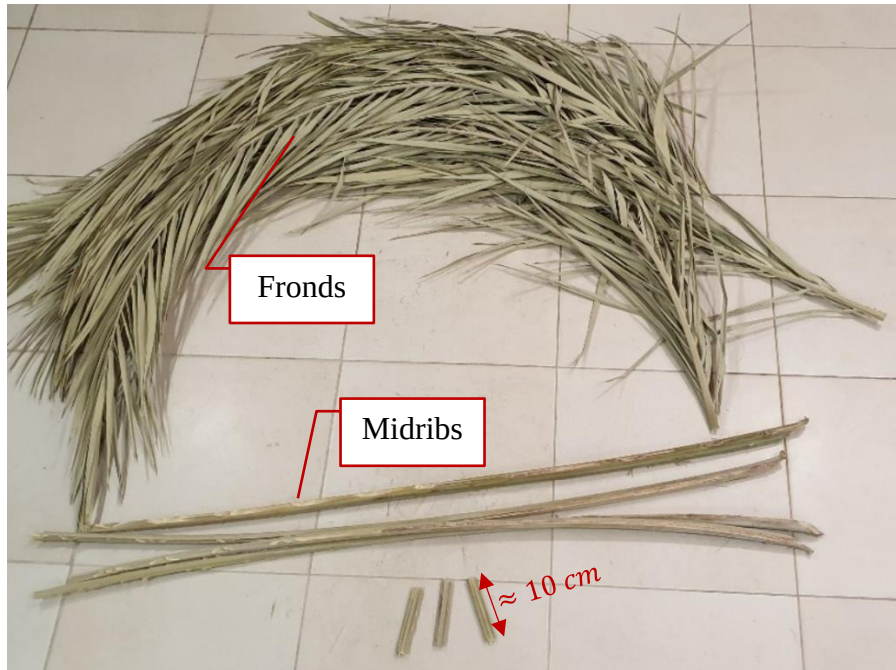


Figure 12: Khalas midrib cut out

#### 4.2. Fiber Extraction Process

The natural fiber extraction process is crucial and plays the main role in the resultant properties of the fibers. It affects the physical, chemical, morphological, and mechanical properties. The criterion of the good extraction process is the one that produces clean cellulosic fibers without damaging their structure. The easiest and most practical way to achieve this criterion is by using a combination of processes. The most common extraction processes combination in the literatures is chemical-mechanical combination because it is a quick process, and it is easier to remove unwanted components by chemical dissolving. This process showed excellent results that make it the first choice for the scientist in the field of natural fibers. The drawback of this process is the use of hazardous synthetic chemicals that are not eco-friendly in contrast with the main goal of the natural fibers' usage.

For that reason, in this work a physical-mechanical extraction process was implemented excluding any chemical process. There are different physical extraction processes one of which is the hydrothermal treatment which involves boiling the natural fiber source with water to weaken the bonds between cellulosic fibers, hemicellulose, and lignin. In addition to the removal of unwanted components that dissolve in water which affect the fiber-matrix interfacial strength. As per the available literatures, only Boumediri et al [41] who used the same principal of boiling the fibers source with water. In their work they used autoclave to boil the water while in this work a microwave was used for boiling. Microwave has an effect on the fibers extraction and it was studied in the literatures [42], [43], but the result of this effect was not part of this work's scope. The main advantage of this process is that it coincides with the goal of using natural fibers as a substitution for the synthetic fibers for more sustainability and environmental protection. The drawback of this process is the difficulty of extracting high purity cellulosic fibers as compared with the chemical process. Despite this difficulty, this process was used to be coincide with the goal of sustainable eco-friendly products and to study the results of this process.

The process of extracting natural fibers by sinking the fibers source in water is known as retting and it is an old process that consumes time. By using heat, the several weeks or months are shortened to few hours or minutes. Extraction process steps are:

1. Starting with breaking the midrib pieces longitudinally to increase the area of water exposure which significantly affect the process and reduce the time required.



*Figure 13: Broken midribs*

2. Washing midrib pieces with tap water to remove any impurities.
3. Immersing the midribs in hot water and boiling them for enough time until they wet, soften, and get saturated by the absorbed water. The required time depends on the size of the midribs and their exposure to water. Thinner midribs lead to higher area to volume ratio meaning higher contact area with water resulting in shorter time to be saturated with absorbed water. In this work boiling time ranged between 30 to 45 minutes using microwave for heating. The extraction was conducted at sea level under ambient pressure which means the boiling temperature of water is 100°C. Higher extraction temperature is assumed to give shorter time and better results, but as initial step it was suggested to start at ambient pressure. To increase the temperature of the extraction, steam boiling of midribs or boiling them under pressure can be used.
4. After the bonds between cellulosic fibers and the other components are weakened, mechanical extraction is undertaken. There are different mechanical processes such as hammering, rolling, scraping, etc. In this work manual hammering using a rubber mallet was used to avoid damaging the fibers. The best practice was to have a multiple hammering rounds. First round removes the outer bark of the midrib,

then the second round separates the fibers from the unwanted components (lignin and hemicellulose). The final round is to farther remove of unwanted components and to reduce fibers diameter by hammering and rubbing between hands. Between each round, fibers are washed with water to remove any contaminants.

5. Finally, fibers are dried using the microwave to reduce the drying time. Further processes can be done, if the fibers are not extracted well by the previous steps, by rubbing the dry fibers between hands for farther removal of the contaminants.



*Figure 14: Fibers' extraction levels and the extraction residues*

After these steps are completed and enough fibers are extracted, the 10 cm fibers are ready to be used as a reinforcement with the epoxy resin

#### **4.3. Composite Process**

To make the composite, epoxy resin was used since it is one of the most frequently used composite matrix in the literature making it easier to compare the results of this work. ENVIROTEX LITE<sup>®</sup> resin from Environmental Technology, Inc. (ETI) was used in this work as the matrix. The resin properties are:

Table 3: Resin properties

| Brand name        | Color | Mixing ratio<br>(By Volume) | Density<br>(g/cm <sup>3</sup> )                      | Tensile strength<br>(MPa) | Tensile modulus<br>(MPa) |
|-------------------|-------|-----------------------------|------------------------------------------------------|---------------------------|--------------------------|
| ENVIROTEX<br>LITE | Clear | 1:1                         | Resin = 1.136<br>Hardener = 0.960<br>Average = 1.048 | 21.43                     | 981                      |

Four sets of composite samples were made with different weight percentage (wt%), which are 10wt%, 15wt%, 17wt%, and 24wt%. The steps to make the composite material that are made of date palm midrib core fibers and epoxy resin are:

1. Measure the extracted fibers mass and then calculate the expected total mass based on the desired weight percentage (wt%). The calculations are:

$$Total\ weight = fibers\ weight \div wt\%$$

$$Matrix\ weight = Total\ weight - fibers\ weight$$

$$Mixing\ volume\ of\ the\ resin\ and\ hardener = \frac{Matrix\ weight}{Matrix\ density} \div 2$$

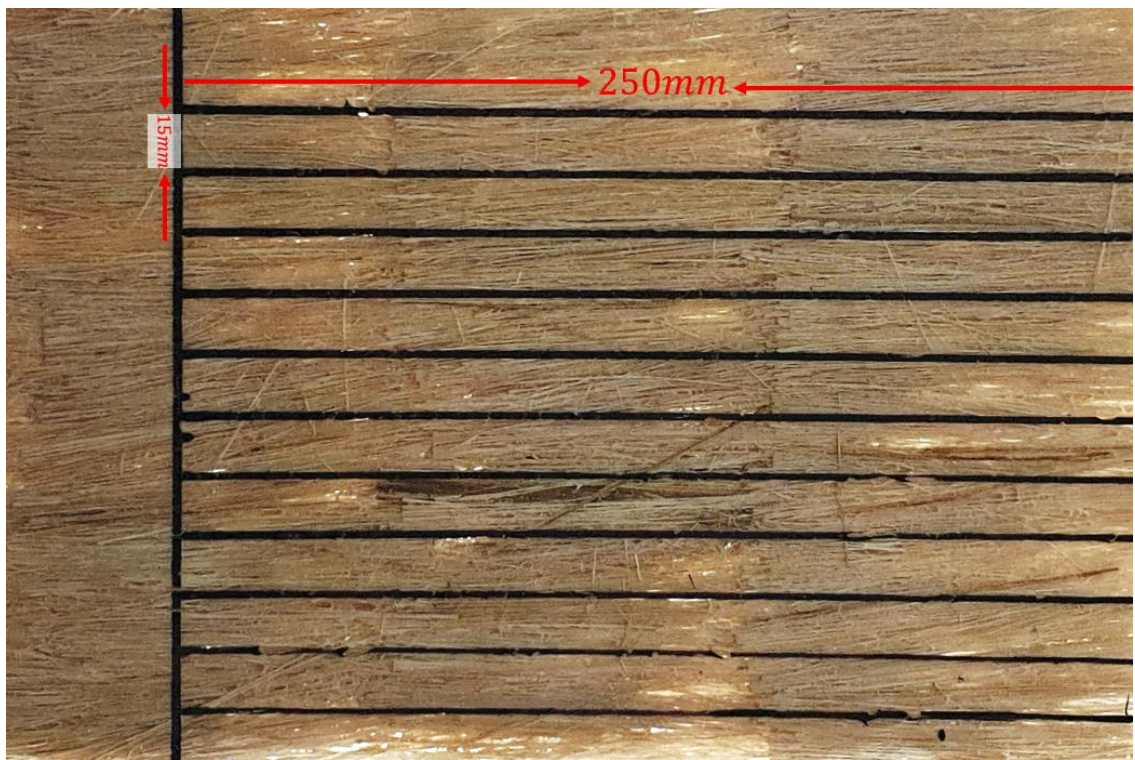
2. Laying out the extracted fibers in one direction (longitudinally) layer by layer in the mold for better distribution and overlapping of the fibers. It is difficult to handle this kind of fibers to get the best distribution because they are long, mesh together, and are non-woven.
3. After laying out the fibers, the resin is mixed based on the calculated volume for each component (resin and hardener) by a 1:1 volumetric ratio. Manufacturer mixing instruction should be followed carefully to get the resin to cure correctly.
4. Pouring the resin on the laid fibers evenly. Then, using a roller to roll the mixture to ensure good fiber-resin mixing and for more even distribution of fibers.

5. In this work a closed mold method is used, so after the previous steps the mold is closed and compressed by adding weight on top of the mold cover. In the first samples (10, 15, and 17 wt%) the weight was about 6 kg and the last sample (24wt%) the weight was 45 kg. Also, a vacuum bag was used with the last two samples (17 and 24 wt%). Higher weight and a vacuum bag were used with the higher wt% samples because it becomes more difficult to make a composite with high fiber average wt% without high levels of compression.
6. Leaving the composite until the resin is completely cure. In case there are any voids on the surface or uncovered fibers with the resin in the composite, extra thin layer of resin is used to fix those defects. This fixing process works well only if the resin layers are bonding to each other strongly same as this type of resin.



*Figure 15: Date palm fibers laid down longitudinally and mixed with the resin in the aluminum mold*

Finally, date palm fibers composite samples were prepared for tensile test by cutting the large composite board into rectangular samples according to ASTM D-3039 [44] which is the “Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials”. The recommended length and width from the standard are 250mm and 15mm, respectively. Sample thickness required is “as needed” as per the standard.



*Figure 16: Composite tensile test samples cutting dimensions lines*

## CHAPTER 5

### TESTING

In this chapter, the details of the testing machine and the three mechanical tests performed in this work are discussed. The discussion will include the standards used, the tests setups, and calculations.

#### 5.1. Tensile Machine

In this work, an INSTRON<sup>®</sup> universal tensile machine was used. Model 3367 is equipped with a load cell whose capacity is 30 kN, catalog number of 2530-444. This machine is connected to Bluehill<sup>®</sup> software version 4.18.26565. The machine measures the time, force, and displacement. The software does the required calculations automatically based on pre-defined algorithms, which will be discussed for each test. Specifically, the modulus of elasticity, stress, and strain are used. For the tensile test, an INSTRON<sup>®</sup> extensometer was used for accurate strain measurement. Its catalog number is 2620-601 which has a gage length of 12.5 mm and travel of  $\pm 5$  mm.

To be consistent with the software terminology, **specimen** means the individual testing coupon, and **sample** means the group of specimens (batch), also it refers to the composite board before it was cut to specimens.

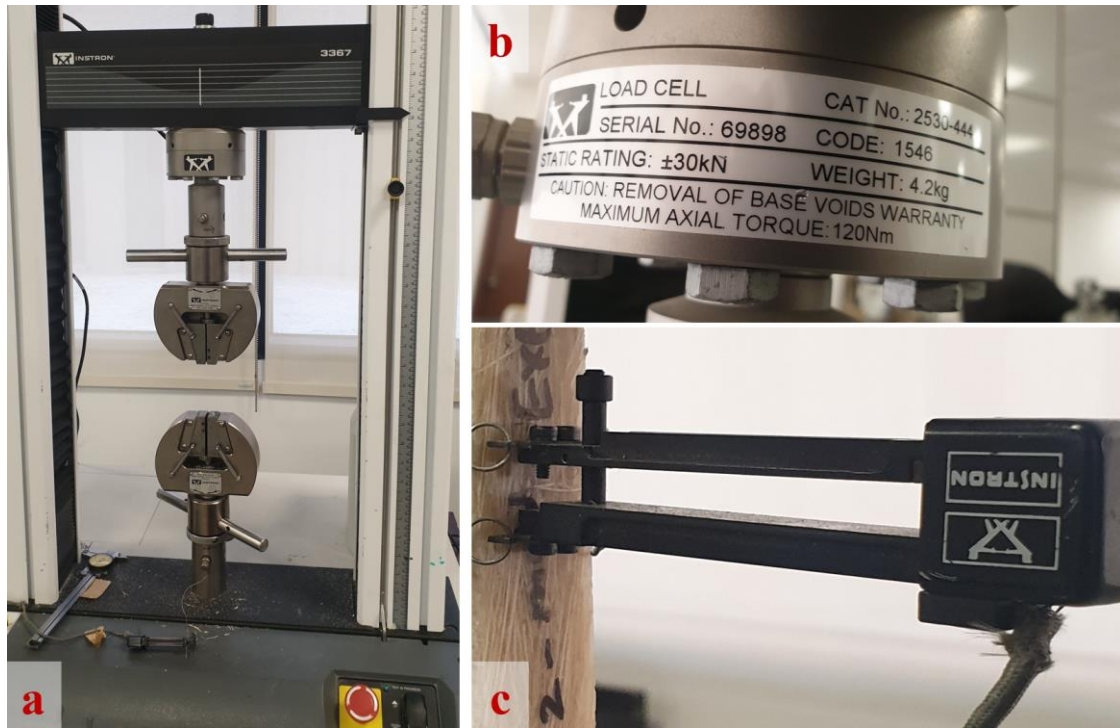


Figure 17: Tensile testing apparatus used for this thesis: (a) INSTRON® universal tensile machine, (b) load cell properties, and (c) INSTRON® extensometer

## 5.2. Single Fiber Tensile Test

This test is to determine the mechanical properties of the single fiber, and it was performed according to ASTM D3822 [40] which is “Standard Test Method for Tensile Properties of Single Textile Fibers”. According to the standard, the single fiber should be enclosed in a paper frame with gage length of 10 mm. The frame dimensions are modified without affecting the gage length for the ease of preparation and best fitting in the tensile machine jaws. The frame length is 50 mm, and the width is 30 mm with central gage of 10 mm. The fiber was glued between two paper frames ensuring good fiber alignment. After gripping

the fiber in the tensile machine, the frame sides are cut. In this test, ten specimens were tested. The test setup was:

- Test Rate = 0.5 mm/min
- Preload = 0 N
- Fibers average diameter = 0.4 mm

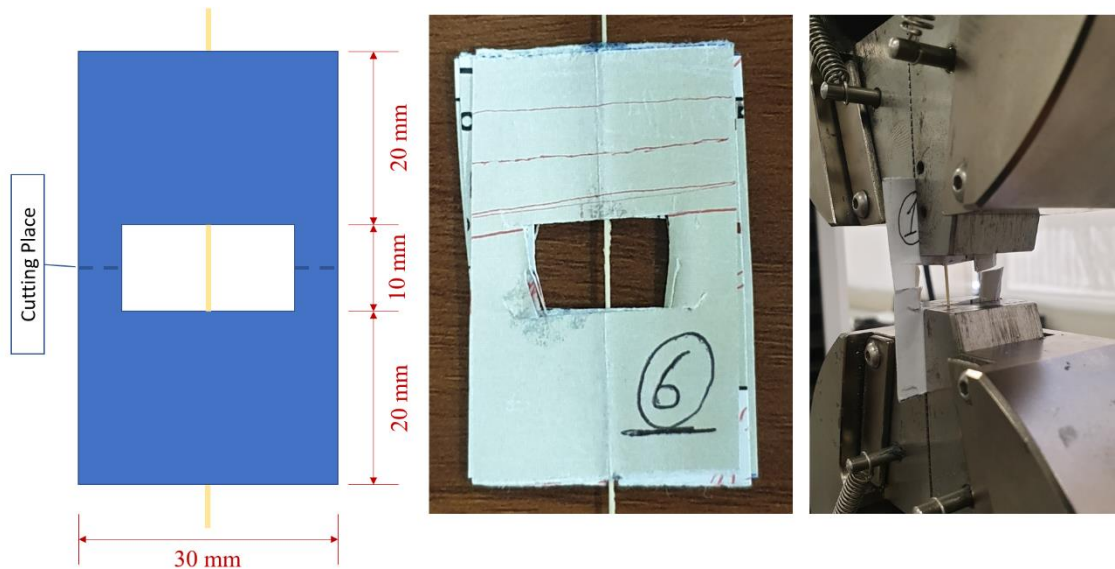


Figure 18: Single fiber tensile test specimen

### 5.3. Interfacial Shear Strength Test

This test is to evaluate the shear strength between the fiber and the matrix which is how well the fibers, and the matrix are bonded together. As the interfacial shear strength increases, the mechanical properties of the composite improve and the fiber critical length decreases. There are different tests to evaluate the interfacial shear strength between the

fiber and the matrix one of which is the fiber pull-out test [3], [45]–[49]. In this test, portion of the fiber is embedded in the matrix, and then it is pulled-out until the fiber detaches from the matrix. The advantage of this test is that it is easy to prepare, does not require advanced testing tools, and gives a reasonable result for the interfacial strength measurement. On the other hand, it does not represent the real behavior since not all the fiber is embedded in the matrix, i.e., only the local properties are tested.

Preparation and testing of the specimens are relatively easy and direct using the tensile machine. First, embedding a short portion of the fiber in the epoxy resin. The thickness of the resin must be small ( $\approx l \leq 2mm$ ) to ensure that the interfacial shear will reach its maximum, so it pulls out of the matrix before the fiber reaches its ultimate tensile strength and breaks. As can be seen in [Figure 19](#), the unembedded fibers are marked with black color to notice the fiber pull-out either by the appearance of the embedded unmarked fiber or the change of the graph slope. After the fiber is pulled out, the maximum force was used to calculate the interfacial strength by:

$$\tau = \frac{F_{max}}{\pi DL} \quad (8)$$

Where:

$F_{max}$ : Maximum tensile force (N)

$D$ : Fiber's diameter (mm)

$L$ : Length of the embedded fiber (mm)

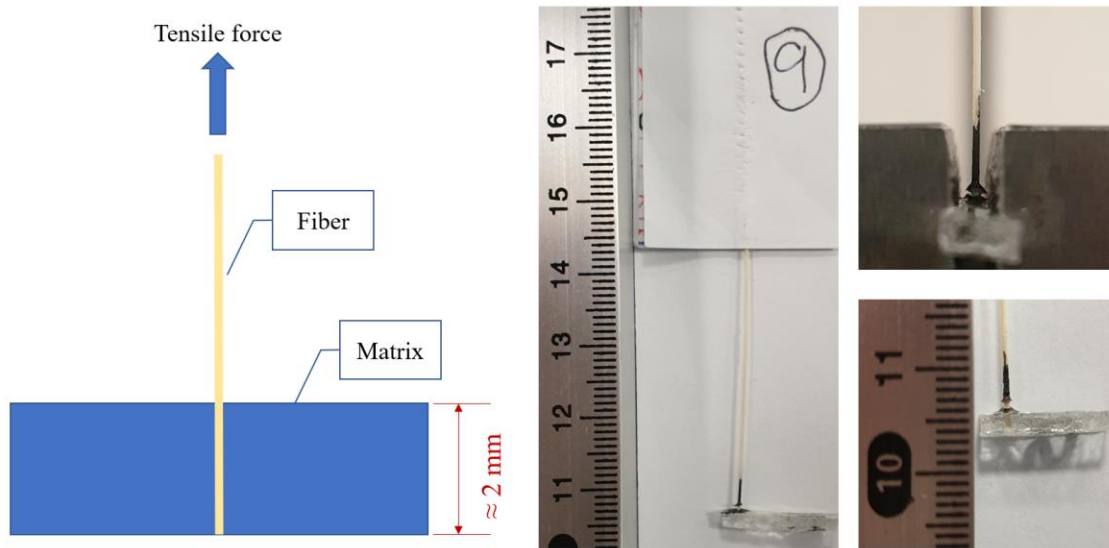


Figure 19: Fiber pull-out test specimen

#### 5.4. Composite Tensile Test

After the four composite samples were cut into rectangular specimens with dimensions according to ASTM D-3039 [44], the thickness and width of the specimens were measured using Mitutoyo digital micrometer of 0.001 mm resolution. Variation in these two dimensions is due to the preparation and cutting processes which affect the thickness and width, respectively. The length of the specimens is constant with 250 mm and the variation is negligible. The average of all measurements of the sample was assigned as the dimension of each specimen.

After taking the measurements, the average dimensions were inserted to the software, the extensometer was attached to the specimen. By this the specimen was prepared for the test.

The test setup was:

- Preload = 1 N
- Loading rate = 2 mm/min
- Auto-balance = ON for displacement
- Extensometer removal @ 0.4%

When using the extensometer, the software takes the gage length as the extensometer gage length which is 12.5 mm in our case. To avoid any damage to the extensometer since it is a sensitive device, it is removed from the specimen before it reaches its maximum travel length or before the specimen fracture. In our case, the extensometer removal was at 0.4% strain. After extensometer removal, the software shifts the displacement measurement to the displacement of the crosshead of the machine. To ensure consistency in the readings, the software corrects and normalizes the crosshead displacement readings using this algorithm:

$$D_c = (D_x - D_{rp}) \times \left( \frac{L_{EXT}}{L_o} \right) + EXT_{rp} \quad (9)$$

Where:

$D_c$ : Corrected displacement

$D_x$ : Displacement reading of the crosshead at certain point

$D_{rp}$ : Removal point displacement of the crosshead at last data point before removal

$L_{EXT}$ : Extensometer gage length

$L_o$ : specimen gage length

$EXT_{rp}$ : Removal point displacement of the extensometer at last data point before removal

After the tensile test finishes, due to the fracture of the specimens, the software will display all the needed measurements and calculation. For the strain, it is measured by the extensometer and by the corrected crosshead displacement after extensometer removal using the engineering strain formula:

$$\varepsilon = \frac{\Delta L}{L_o} \quad (10)$$

Where:

$\varepsilon$ : Strain (*mm/mm*)

$\Delta L$ : Displacement (*final length – initial length*) (*mm*)

$L_o$ : Initial length (*mm*)

The engineering stress is calculated using the measured load from the load cell and the inserted average dimensions of the specimens:

$$\sigma = \frac{F}{A} \quad (11)$$

Where:

$\sigma$ : Stress (*MPa*)

$F$ : Applied force (*N*)

$A$ : Cross sectional area of the specimen (*thickness × width*) (*mm<sup>2</sup>*)

For the modulus of elasticity, the software has different methods of calculation. Young's modulus and the automatic Young's modulus are two ways of calculating modulus of elasticity. Their algorithms are almost the same, but they differ in specifying the initial and final data points to calculate the modulus. As their name implies, the automatic Young's method specifies the initial point automatically as the 2% of the maximum force and the

final point as the yield point or the maximum force. In contrast, the Young's Modulus method gives the operator the ability to specify the initial and the final data points to calculate the modulus. In this thesis and since we are using extensometer, it is better to use the Young's Modulus method rather than the automatic one because the reading of the extensometer is more accurate than the crosshead's displacement and therefore it was desired to restrict the elastic modulus calculations to the extensometer reading range. Since the extensometer removal is at 0.4% strain, the selected initial value was 0.05% to reduce the initial variation of the measurement and the final value was 0.35% to be away of any variation that may be associated with the extensometer's removal. After that, the software divides the selected range into number of regions specified by the operator which were in our case six regions divided on the strain axis. Then, the software applies the least squares fit on all the data points of each region, calculates the sum of the slopes of each adjacent region to find the pair of regions with the highest sum of slopes. Finally, the modulus is assigned to the region with the highest slope of this pair of regions. **Figure 20** is an example of stress-strain graph divided into eight regions on the strain axis from the help section of the Bluehill<sup>®</sup> software.

At least seven specimens of each sample were tested and the average of all the mechanical properties is considered as representative of that sample's wt%. Test results are in the next chapter.

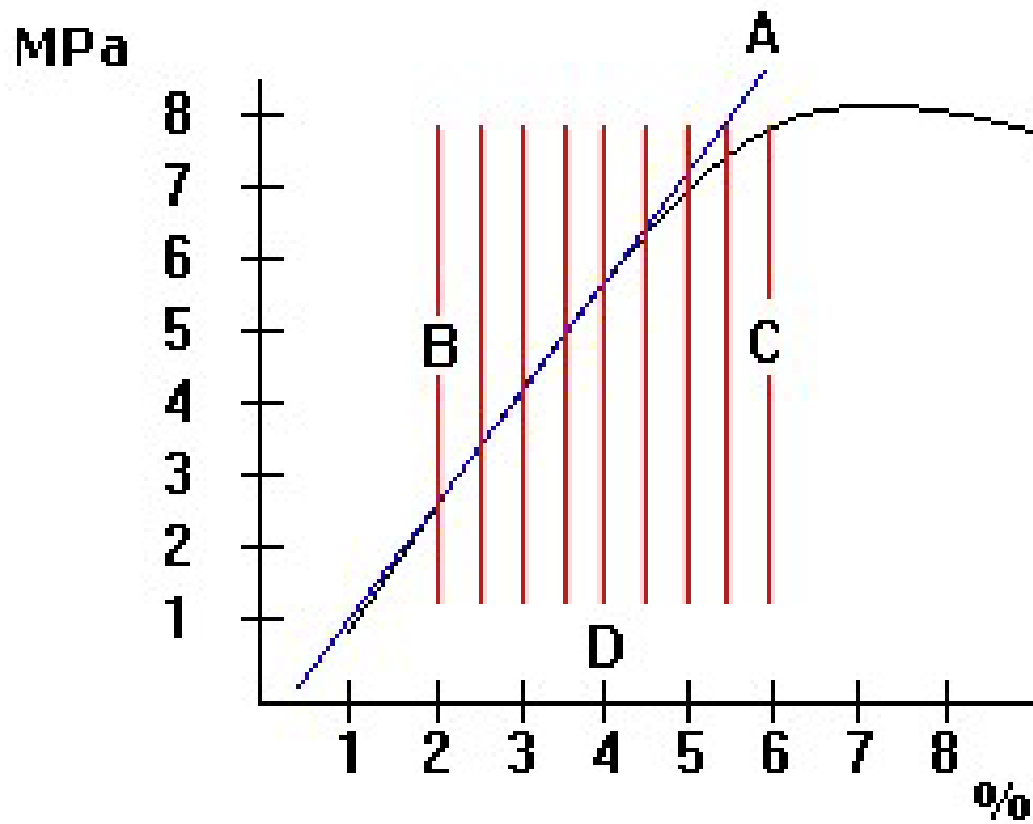


Figure 20: schematic illustration of young's modulus calculation algorithm, A is the modulus line, B is the initial point at 2%, C is the final point at 6%, and D is the pair with highest sum of slopes. [Bluehill® software help section]

## CHAPTER 6

### RESULTS & ANALYSIS

In this chapter, tests results are shown, analyzed, and compared with literature and commercial products.

#### 6.1. Single Fiber Tensile Test Results

After the specimens were prepared according to ASTM D3822 [40], the test was conducted, and the results are:

Table 4: Single fiber tensile test results

|             | Force at<br>Maximum<br>Load | Tensile<br>Strength | Elongation | Yield<br>Stress | Young's<br>Modulus |
|-------------|-----------------------------|---------------------|------------|-----------------|--------------------|
|             | (N)                         | (MPa)               | (%)        | (MPa)           | (MPa)              |
| <b>1</b>    | 42.6                        | 294                 | 1.69       | 258             | 20700              |
| <b>2</b>    | 30.5                        | 210                 | 2.12       | 196             | 11800              |
| <b>3</b>    | 55.4                        | 382                 | 2.76       | 323             | 18900              |
| <b>4</b>    | 58.0                        | 400                 | 1.92       | 370             | 18100              |
| <b>5</b>    | 57.4                        | 395                 | 2.17       | 327             | 20400              |
| <b>6</b>    | 63.2                        | 435                 | 2.55       | 327             | 22000              |
| <b>7</b>    | 57.5                        | 396                 | 2.50       | 279             | 20600              |
| <b>8</b>    | 26.6                        | 183                 | 2.91       | 135             | 8600               |
| <b>9</b>    | 51.6                        | 355                 | 2.71       | 261             | 15700              |
| <b>10</b>   | 40.4                        | 278                 | 2.23       | 218             | 14800              |
| <b>Min</b>  | 26.6                        | 183                 | 1.69       | 135             | 8600               |
| <b>Mean</b> | 48.3                        | 333                 | 2.36       | 269             | 17200              |
| <b>Max</b>  | 63.2                        | 435                 | 2.91       | 370             | 22000              |
| <b>CV</b>   | 26.1                        | 26.1                | 16.8       | 26.5            | 39.4               |
| <b>SD</b>   | 12.6                        | 86.8                | 0.39       | 71.4            | 6180               |

The variation in the single fiber tensile results is high as the variation in the literatures. This variation is because of fibers' type, surface defects, and treatments. The following table is a comparison between this work and the other available literatures with a restriction to the midrib fibers with a quick comparison with the mesh properties:

Table 5: Comparison of single fiber tensile test results with literatures

| Fibers' type                             | Chemical treatment | Tensile strength (MPa) | Modulus of elasticity (GPa) | Reference |
|------------------------------------------|--------------------|------------------------|-----------------------------|-----------|
| <b>Midrib</b>                            | Un-treated         | 183 – 435              | 8.6 – 22                    | This work |
| <b>Midrib</b>                            | Un-treated         | 129 – 530              | 5.88 – 21.9                 | [41]      |
| <b>Midrib wall</b><br><b>Midrib core</b> | Un-treated         | 116 – 208<br>71 – 100  | 10 – 30                     | [7], [50] |
| <b>Midrib</b>                            | Un-treated         | 118                    | 4.4                         | [12]      |
|                                          | NaOH               | 164 – 453              | 2.3 – 16.1                  |           |
| <b>Mesh</b>                              | Un-treated         | 200 – 275              | 0.8 – 0.845                 | [5]       |
|                                          | HCl                | 105                    | 3.23                        |           |
|                                          | NaOH               | 450 – 845              | 70 – 160                    |           |
| <b>Mesh</b>                              | Un-treated         | 60 – 115               | 1.4 – 2.6                   | [51]      |
|                                          | NaOH               | 80 – 150               | 1.4 – 3.1                   |           |
| <b>Mesh</b>                              | Un-treated         | 600 – 1300             |                             | [6]       |
|                                          | NaOH               | 700 – 1550             |                             |           |
|                                          | Silane             | 850 – 1800             |                             |           |
|                                          | NaOH+ Silane       | 1000 – 2000            |                             |           |

This short comparison shows that the single fiber testing results of the water treated fibers from this work and other literatures are competitive with the chemical treated DPF. Meaning that it is possible to have good properties fibers with almost zero synthetic chemicals.

## 6.2. Interfacial Strength Test Results

Ten samples were made for this test. Three of them broke before they pull-out the matrix, so seven specimens passed the test, and their results are:

Table 6: Interfacial strength test results

|      | Force at<br>Maximum Load | Diameter | Embedded Fiber<br>Length | Interfacial<br>strength |
|------|--------------------------|----------|--------------------------|-------------------------|
|      | (N)                      | (mm)     | (mm)                     | (MPa)                   |
| 1    | 41.5                     | 0.489    | 2.58                     | 10.5                    |
| 2    | 56.6                     | 0.440    | 4.39                     | 9.33                    |
| 5    | 43.1                     | 0.464    | 2.93                     | 10.1                    |
| 6    | 42.4                     | 0.506    | 4.04                     | 6.61                    |
| 7    | 31.8                     | 0.417    | 2.81                     | 8.65                    |
| 9    | 16.8                     | 0.360    | 2.48                     | 5.99                    |
| 10   | 54.1                     | 0.313    | 4.24                     | 13.0                    |
| Min  | 16.8                     | 0.313    | 2.48                     | 5.99                    |
| Mean | 40.9                     | 0.427    | 3.35                     | 9.16                    |
| Max  | 56.6                     | 0.506    | 4.39                     | 13.0                    |
| CV   | 33.0                     | 16.3     | 24.8                     | 26.0                    |
| SD   | 13.5                     | 0.070    | 0.833                    | 2.38                    |

By the knowledge of the fiber's ultimate tensile strength from the previous test, and the interfacial strength from this test, the critical length of the DPF extracted by this method can be calculated using the critical length equation from chapter 2. Assuming that the shear strength of the matrix is higher than the interfacial strength, taking the mean value of the fiber tensile strength, and the fiber's diameter as 0.4 mm, this results in:

$$l_c = \frac{\sigma_f^* d}{2\tau_c} = \frac{333 \times 0.4}{2 \times 9.16} \approx 7.3 \text{ mm}$$

In this work, the fibers length is 100 mm which is about 14 times the critical length and that is considered as continues fibers. The continues fibers gives the best load transformation in the composite. This is also a factor that makes the composite results

competitive with the other literatures since most of them used short fibers. Below is a table comparing the interfacial strength of this work with other literatures:

Table 7: Comparison of interfacial strength test results with literatures

| Fiber's type             | Matrix type           | Test type     | Chemical treatment                              | Interfacial strength (MPa)               | Reference |
|--------------------------|-----------------------|---------------|-------------------------------------------------|------------------------------------------|-----------|
| <b>Midrib</b>            | Epoxy                 | Pull-out      | Un-treated                                      | 5.99 – 12.96                             | This work |
| <b>Fronid (petioles)</b> | Epoxy                 | Pull-out      | Un-treated<br>NaOH                              | 12.5<br>15 – 25                          | [3]       |
| <b>Mesh</b>              | Epoxy                 | Fragmentation | Un-treated<br>NaOH                              | 3.5 – 10<br>4 – 10.5                     | [51]      |
| <b>Mesh</b>              | Epoxy<br>polyurethane | Pull-out      | NaOH+Silane                                     | 0.31 – 0.42<br>0.3 – 0.42                | [45]      |
| <b>Mesh</b>              | polyester             | Pull-out      | Un-treated<br>NaOH<br>Silane<br>NaOH+<br>Silane | 10 – 13<br>13 – 16<br>14 – 17<br>17 – 19 | [6]       |
| <b>Mesh</b>              | Epoxy                 | Pull-out      | NaOH                                            | 2.25 – 4.6                               | [10]      |

These literatures showed how the chemical treatment improved the interfacial strength and this is attributed to the removal of fibers surface impurities and wax layers which reduces the adhesion between the fiber and the matrix. Unfortunately, there was not enough time to characterize the fibers' surface extracted and treated by water boiling to quantify the impurities on the surface and study their effect. However, the interfacial strength of this work even if it is not the best but it is close to the treated ones and have the opportunity to be improved in the future work.

### 6.3. Composite Tensile Test Results

In this work, four composite samples were made with different average wt% of fibers which are 10 wt%, 15 wt%, 17 wt%, and 24 wt%. Their tensile test results are:

Table 8: 10wt% Sample Tensile Test Results

| 10wt% |                       |                  |            |              |                       |
|-------|-----------------------|------------------|------------|--------------|-----------------------|
|       | Force at Maximum Load | Tensile Strength | Elongation | Yield Stress | Modulus of Elasticity |
|       | (kN)                  | (MPa)            | (%)        | (MPa)        | (MPa)                 |
| 1     | 1.45                  | 19.4             | 1.67       | 18.3         | 1720                  |
| 2     | 1.27                  | 16.9             | 1.65       | 13.4         | 1930                  |
| 3     | 1.62                  | 21.6             | 1.66       | 21.6         | 1840                  |
| 4     | 1.81                  | 24.2             | 1.57       | 23.9         | 2130                  |
| 5     | 1.97                  | 26.2             | 1.44       | 25.9         | 2320                  |
| 6     | 1.52                  | 20.3             | 1.27       | 17.3         | 2460                  |
| 7     | 2.10                  | 28.0             | 1.43       | 21.6         | 2640                  |
| 8     | 1.78                  | 23.8             | 1.47       | 23.8         | 1890                  |
| 9     | 1.77                  | 23.6             | 1.46       | 18.0         | 2410                  |
| 10    | 1.66                  | 22.1             | 1.85       | 15.7         | 2450                  |
| Min   | 1.27                  | 16.9             | 1.27       | 13.4         | 1720                  |
| Mean  | 1.70                  | 22.6             | 1.55       | 19.9         | 2180                  |
| Max   | 2.10                  | 28.0             | 1.85       | 25.9         | 2640                  |
| CV    | 14.5                  | 14.5             | 10.7       | 20.3         | 14.6                  |
| SD    | 0.246                 | 3.28             | 0.165      | 4.04         | 318                   |

Table 9: 15wt% Sample Tensile Test Results

| 15wt% |                       |                  |            |              |                       |
|-------|-----------------------|------------------|------------|--------------|-----------------------|
|       | Force at Maximum Load | Tensile Strength | Elongation | Yield Stress | Modulus of Elasticity |
|       | (kN)                  | (MPa)            | (%)        | (MPa)        | (MPa)                 |
| 1     | 1.73                  | 26.3             | 1.24       | 22.9         | 2780                  |
| 2     | 1.94                  | 29.5             | 1.23       | 23.4         | 2660                  |
| 3     | 1.71                  | 26.0             | 1.21       | 21.3         | 3310                  |
| 4     | 1.69                  | 25.8             | 1.22       | 25.3         | 2530                  |
| 5     | 2.28                  | 34.7             | 1.33       | 25.6         | 4070                  |
| 6     | 2.11                  | 32.2             | 1.41       | 32.0         | 2730                  |
| 7     | 1.41                  | 21.4             | 0.960      | 20.9         | 2830                  |
| Min   | 1.41                  | 21.4             | 0.960      | 20.9         | 2530                  |
| Mean  | 1.84                  | 28.0             | 1.23       | 24.5         | 2990                  |
| Max   | 2.28                  | 34.7             | 1.41       | 32.0         | 4070                  |
| CV    | 15.9                  | 15.9             | 11.3       | 15.4         | 17.9                  |
| SD    | 0.292                 | 4.45             | 0.139      | 3.77         | 536                   |

Table 10: 17wt% Sample Tensile Test Results

| 17wt%       |                       |                  |            |              |                       |
|-------------|-----------------------|------------------|------------|--------------|-----------------------|
|             | Force at Maximum Load | Tensile Strength | Elongation | Yield Stress | Modulus of Elasticity |
|             | (kN)                  | (MPa)            | (%)        | (MPa)        | (MPa)                 |
| <b>1</b>    | 1.35                  | 20.6             | 1.19       | 20.5         | 2060                  |
| <b>2</b>    | 1.27                  | 19.3             | 0.960      | 18.2         | 2820                  |
| <b>3</b>    | 1.44                  | 21.9             | 1.36       | 18.0         | 2420                  |
| <b>4</b>    | 1.54                  | 23.4             | 0.970      | 20.5         | 3840                  |
| <b>5</b>    | 1.86                  | 28.3             | 1.42       | 19.7         | 3930                  |
| <b>6</b>    | 1.69                  | 25.7             | 1.32       | 19.8         | 3390                  |
| <b>7</b>    | 2.40                  | 36.5             | 1.75       | 23.5         | 3200                  |
| <b>8</b>    | 1.82                  | 27.8             | 0.300      | 17.2         | 9520                  |
| <b>9</b>    | 2.45                  | 37.3             | 1.63       | 23.7         | 3680                  |
| <b>10</b>   | 2.00                  | 30.4             | 1.73       | 20.9         | 2760                  |
| <b>Min</b>  | 1.27                  | 19.3             | 0.300      | 17.2         | 2060                  |
| <b>Mean</b> | 1.78                  | 27.1             | 1.26       | 20.2         | 3120                  |
| <b>Max</b>  | 2.45                  | 37.3             | 1.75       | 23.7         | 9520                  |
| <b>CV</b>   | 23.0                  | 23.0             | 34.8       | 10.7         | 20.9                  |
| <b>SD</b>   | 0.410                 | 6.25             | 0.439      | 2.17         | 652                   |

Table 11: 24wt% Sample Tensile Test Results

| 24wt%       |                       |                  |            |              |                       |
|-------------|-----------------------|------------------|------------|--------------|-----------------------|
|             | Force at Maximum Load | Tensile Strength | Elongation | Yield Stress | Modulus of Elasticity |
|             | (kN)                  | (MPa)            | (%)        | (MPa)        | (MPa)                 |
| <b>1</b>    | 1.56                  | 23.9             | 1.36       | 22.6         | 1540                  |
| <b>2</b>    | 1.88                  | 28.9             | 1.19       | 27.1         | 2530                  |
| <b>3</b>    | 1.82                  | 27.9             | 1.50       | 24.9         | 1930                  |
| <b>4</b>    | 1.95                  | 29.9             | 1.22       | 21.4         | 3610                  |
| <b>5</b>    | 2.10                  | 32.2             | 1.39       | 31.8         | 2830                  |
| <b>6</b>    | 1.46                  | 22.5             | 1.01       | 19.7         | 2470                  |
| <b>7</b>    | 1.74                  | 26.8             | 1.08       | 20.0         | 3820                  |
| <b>8</b>    | 2.03                  | 31.2             | 1.24       | 26.8         | 2490                  |
| <b>9</b>    | 2.85                  | 43.8             | 1.24       | 28.5         | 5540                  |
| <b>10</b>   | 2.58                  | 39.7             | 1.25       | 36.8         | 3390                  |
| <b>Min</b>  | 1.46                  | 22.5             | 1.01       | 19.7         | 1540                  |
| <b>Mean</b> | 2.00                  | 30.7             | 1.25       | 25.9         | 3020                  |
| <b>Max</b>  | 2.85                  | 43.8             | 1.50       | 36.8         | 5540                  |
| <b>CV</b>   | 21.6                  | 21.6             | 11.5       | 21.0         | 37.7                  |
| <b>SD</b>   | 0.431                 | 6.63             | 0.144      | 5.46         | 1140                  |

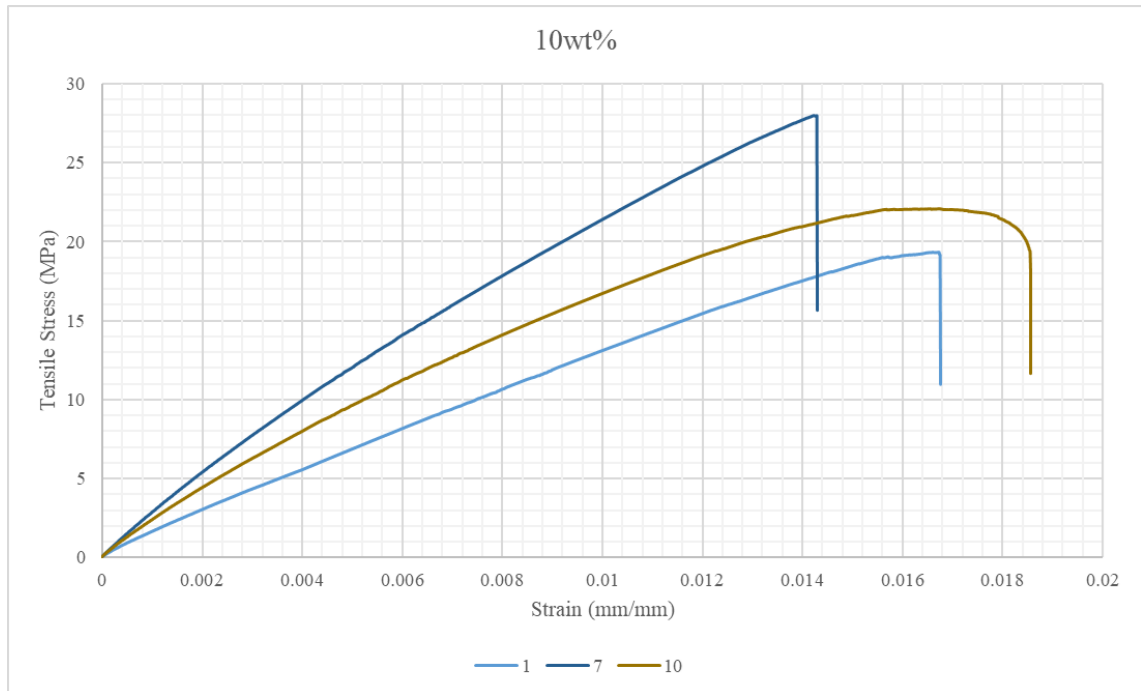


Figure 21: Composite tensile test graph for 10wt%

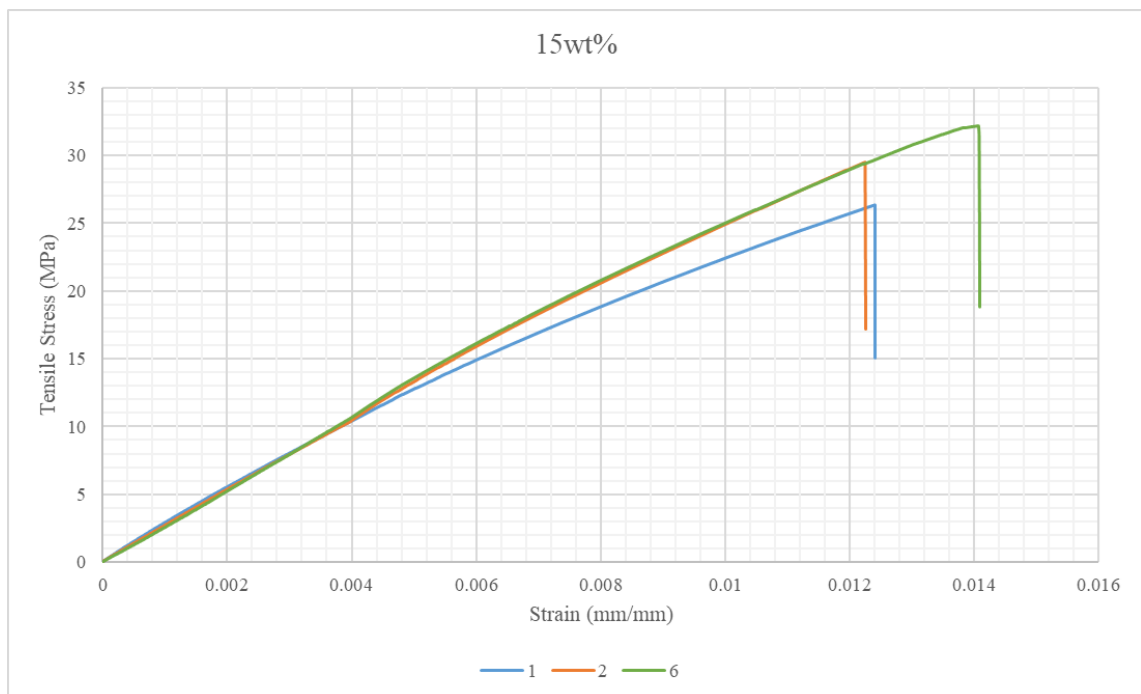


Figure 22: Composite tensile test graph for 15wt%

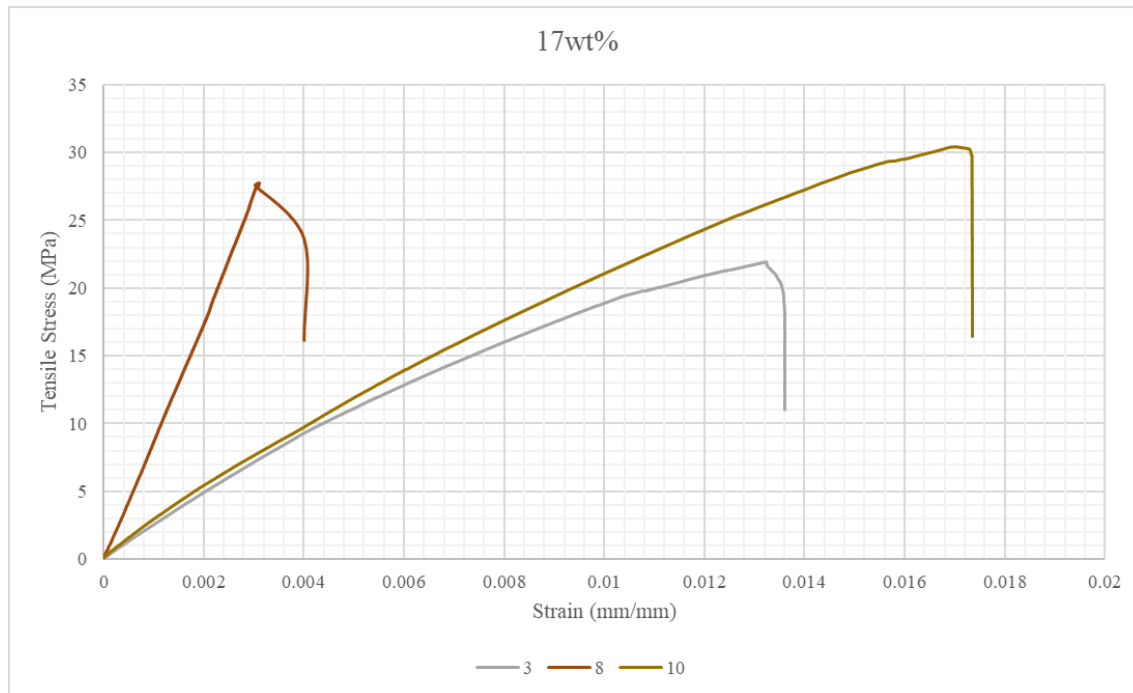


Figure 23: Composite tensile test graph for 17wt%

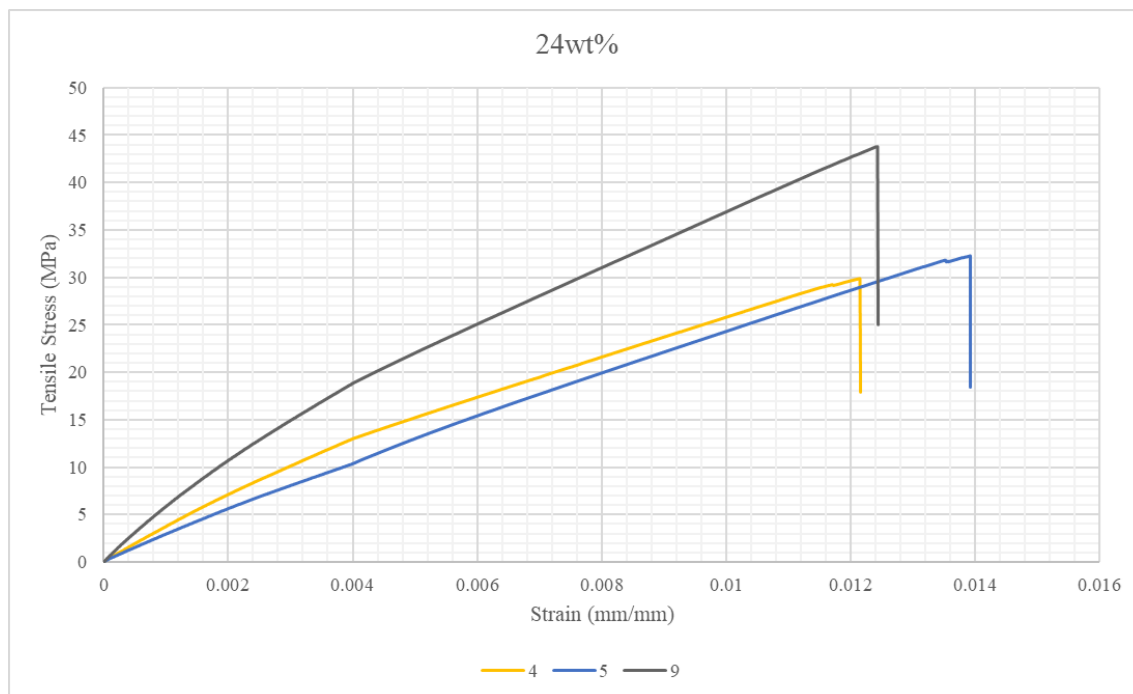


Figure 24: Composite tensile test graph for 24wt%

The previous tables show the data of all the specimens while the graphs are representative with three random specimens of each sample. Due to the extensometer removal, there is going to be a large variation in the graph's lines because of changing the strain measurement device. The software use [equation 8](#) which was discussed in Chapter 5 to correct the crosshead reading to match with the extensometer reading. Since the stress is related to the load cell reading and the specimen dimensions, and the modulus is calculated before the extensometer removal, the error due to this correction is in the elongation values only.

In these experiments, there are two main sources of variation. The first source is the variation within the sample which is known as error in statistics. This variation is due to errors associated with the measurements or sample preparation. The second source is the variation between the samples which is known as factor or treatment in statistics. This variation represents the effect of the factor used which is the fibers wt% in our case.

Starting with the variation within the sample with the best example of 17wt% sample. Specimen 8 in this sample has an up normal modulus value compared with the other specimens which is 9520 MPa. To find the reason behind this high value, a general look at all samples results showed that all of them have the highest modulus value within the last specimens that are in middle of the sample as can be seen in [Figure 25](#) and [Figure 26](#). Since the specimens numbering starts from the edge of the composite board, that means the specimens in the middle of the composite board have higher modulus values. By looking at the composite board, this variation between the specimens within the sample can be attributed to the variation in fibers concentration and uneven distribution of the fibers

through the composite board. This leads to the assumption that not all the specimens have the same wt% and they are different from the overall wt% of the composite board.

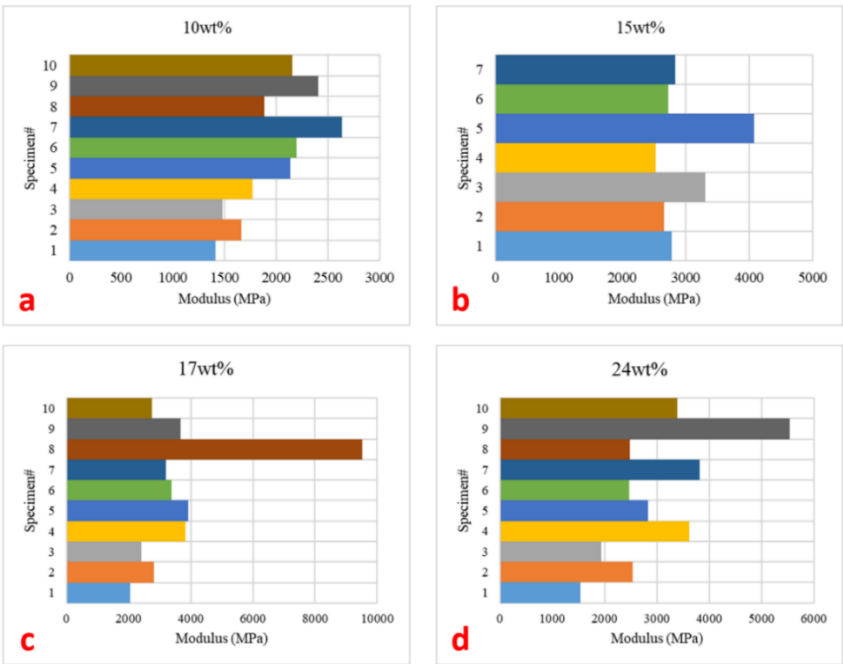


Figure 25: The trend of modulus variation through the samples

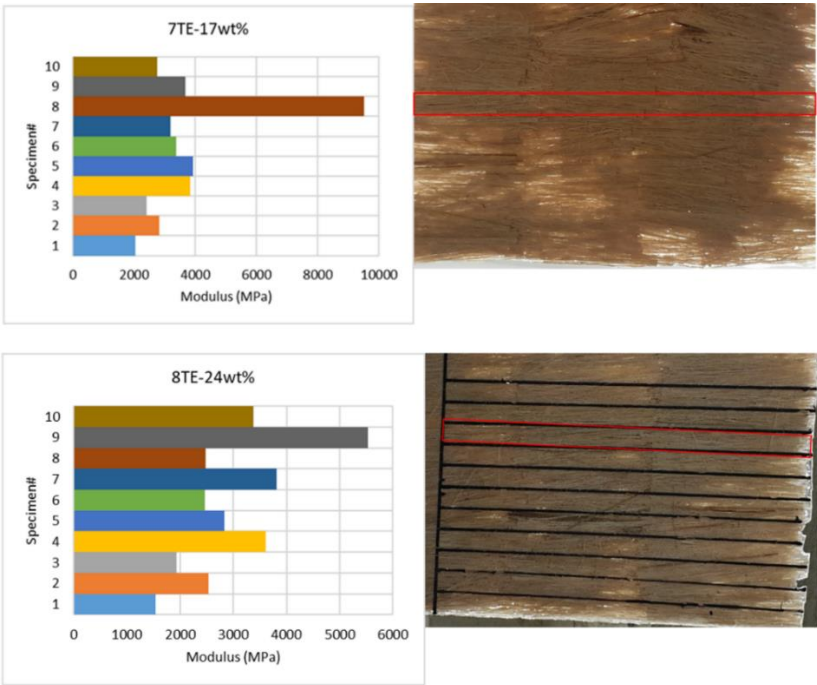


Figure 26: The modulus values of the sample and the corresponding place of the specimen with highest modulus

The assumption of wt% variation can be proven by the variation of the fibers' concentration through the sample, but for more verification and clarification, rule of mixture was used to see if the experimental results are enclosed within the theoretical boundaries or not. Using the mean values of the matrix modulus and the fibers modulus (which is going to be discussed in the next topic) with the rule of mixture equations from Chapter 2, the theoretical boundaries can be plotted. The mean value of the fibers' modulus is 17200 MPa and the resin mean modulus is 558 MPa.

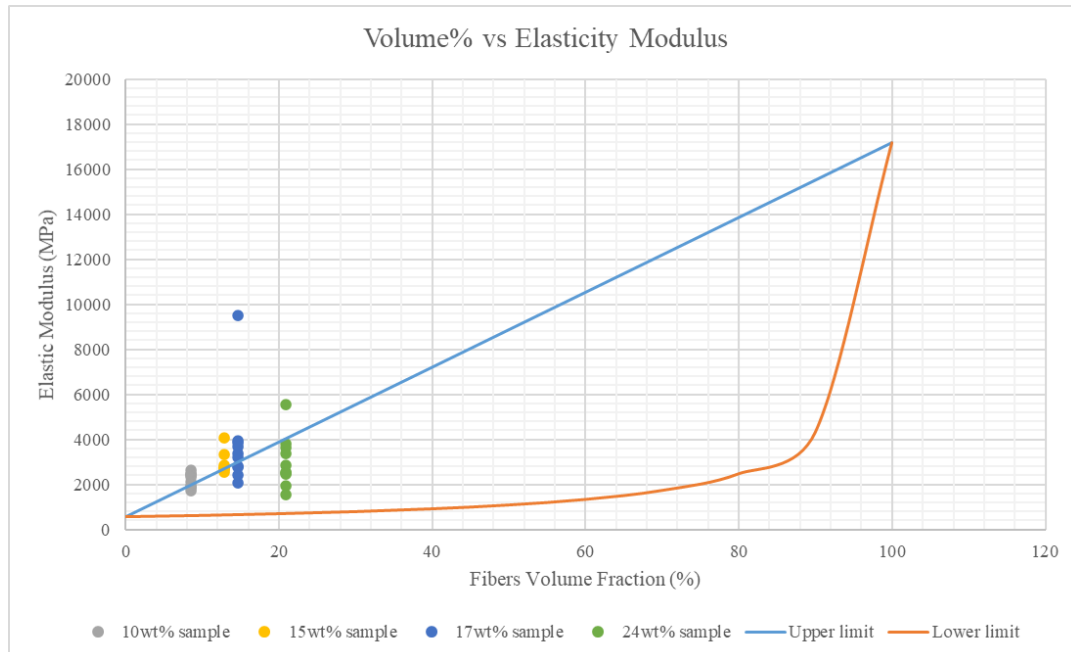


Figure 27: Samples modulus of elasticity data with the rule of mixture boundaries (using mean values of  $E_f$  &  $E_m$ )

From Figure 27 there are many data points are higher than the upper limit. For these data points there are two approaches, either their wt% is higher than the sample wt% or the boundaries values are less than the real values. Thus, changing the boundaries values to the maximum values, which are  $E_f = 22000$  MPa and  $E_m = 981$  MPa (by the manufacturer),

will enclose more data point, but still there are some points are higher than the upper limit **Figure 28**. This approach is not realistic since it is impossible that all the fibers have the maximum modulus, and the maximum experimental value of the resin modulus was 685 MPa.

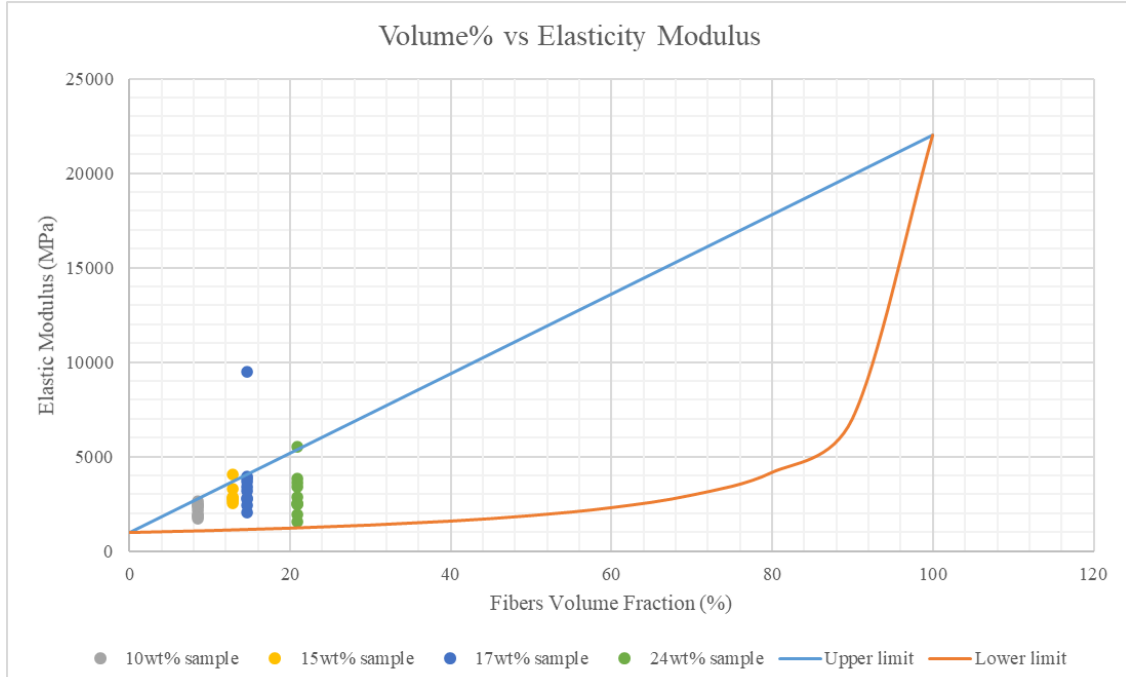


Figure 28: Samples modulus of elasticity data with the rule of mixture boundaries (using Maximum values of  $E_f$  &  $E_m$ )

Using the mean values for the boundaries and changing the wt% of the specimens that are above the upper boundary with their correct values is the best solution to enclose all the values in addition it verifies wt% variation assumption. To change the values above the upper limit to the closest wt% that makes them on the upper limit line, we need to solve for volume in the RoM upper limit equation to get the exact vol% by:

$$V_f = \frac{E_{cl} - E_m}{E_f - E_m} \quad (12)$$

This change result is shown in **Table 12** as a theoretical value which lay on the upper limit and the experimental value should not be less than this theoretical value to be enclosed within the limits.

By changing the wt%, specimen 8 from 17wt% sample should have at least 53.95 vol% which is equivalent to 58.29 wt%. This is a very high wt% in the natural fibers composite field. For more investigation, specimens of each sample were analyzed to find the exact vol% of each specimen. Using ImageJ software, the fibers area fraction was analyzed which can be representative to the volume fraction (see appendix (B) for the analysis images). It is not the best way to find the composite wt% and its results are not very accurate, but it is one of the used methods in the composite materials field.

The error sources in this method are:

- Specimen's surface clarity

The specimen needs long preparation process to get clear surface. However, the fibers impurities also affect the clarity of the surface and reduces the contrast between the fibers and the resin.

- Fibers/matrix identification

Because of the low contrast between the fibers/matrix in some images, it is difficult to select the accurate area of the fibers especially with the higher wt% samples.

- Bias potential

There is a bias potential since all the analysis is based on the user selections and preferences.

The effect of these error sources was reduced by:

- Using both transmitted light and reflected light techniques

This was done with the specimens that have extreme values to double check their vol%. For the transmitted light, two light sources were used on both sides of the specimen to illuminate the clear resin leaving the fibers as dark spots and vice versa with the reflection method.

- Image processing was done before the analysis

To reduce the bias potential, the images were processed until an acceptable contrast between the fibers/matrix was achieved. Then, the analysis was done and the resultant vol% is accepted with less bias.

The results of the analyzed specimens compared with the theoretical vol% are in [Table 12](#). By plotting these values, it can be seen in [Figure 29](#) that there is a general increase trend of the mechanical properties as the vol% increases (the dashed line). The variation from the increasing trend can be attributed to the variation in the interfacial strength, and the defects of the composite. Using ImageJ vol% analysis results with the RoM, all the data points are enclosed within the RoM boundaries with more realistic scattering which supports the assumption of wt% variation [Figure 30](#). In [Figure 30](#), the data points direct towards the lower bound of the RoM which can be attributed to the difficulties of avoiding the voids defects with the increase of the fibers wt%.

Table 12: ImageJ vol% analysis results

| Specimen        | ImageJ vol% | Theoretical vol% | Elastic modulus |
|-----------------|-------------|------------------|-----------------|
| <b>10wt%-1</b>  | 13.06       | 7.00             | 1720            |
| <b>10wt%-2</b>  | 16.44       | 8.22             | 1930            |
| <b>10wt%-3</b>  | 14.12       | 7.68             | 1840            |
| <b>10wt%-4</b>  | 24.48       | 9.47             | 2130            |
| <b>10wt%-5</b>  | 14.01       | 10.59            | 2320            |
| <b>10wt%-6</b>  | 14.02       | 11.45            | 2460            |
| <b>10wt%-7</b>  | 22.17       | 12.49            | 2640            |
| <b>10wt%-8</b>  | 11.53       | 7.98             | 1890            |
| <b>10wt%-9</b>  | 12.36       | 11.10            | 2410            |
| <b>10wt%-10</b> | 14.11       | 11.36            | 2450            |
|                 |             |                  |                 |
| <b>15wt%-10</b> | 20.05       | 13.38            | 2780            |
| <b>15wt%-11</b> | 20.59       | 12.64            | 2660            |
| <b>15wt%-12</b> | 18.44       | 16.53            | 3310            |
| <b>15wt%-13</b> | 38.40       | 11.83            | 2530            |
| <b>15wt%-14</b> | 28.62       | 21.13            | 4070            |
| <b>15wt%-15</b> | 24.03       | 13.04            | 2730            |
| <b>15wt%-16</b> | 17.02       | 13.66            | 2830            |
|                 |             |                  |                 |
| <b>17wt%-1</b>  | 43.23       | 9.02             | 2060            |
| <b>17wt%-2</b>  | 48.77       | 13.58            | 2820            |
| <b>17wt%-3</b>  | 35.59       | 11.21            | 2420            |
| <b>17wt%-4</b>  | 21.02       | 19.70            | 3840            |
| <b>17wt%-5</b>  | 26.43       | 20.25            | 3930            |
| <b>17wt%-6</b>  | 31.96       | 17.02            | 3390            |
| <b>17wt%-7</b>  | 36.41       | 15.88            | 3200            |
| <b>17wt%-8</b>  | 55.95       | 53.83            | 9520            |
| <b>17wt%-9</b>  | 53.51       | 18.75            | 3680            |
| <b>17wt%-10</b> | 51.23       | 13.21            | 2760            |
|                 |             |                  |                 |
| <b>24wt%-1</b>  | 40.19       | 5.89             | 1540            |
| <b>24wt%-2</b>  | 53.81       | 11.87            | 2530            |
| <b>24wt%-3</b>  | 50.57       | 8.26             | 1930            |
| <b>24wt%-4</b>  | 50.54       | 18.36            | 3610            |
| <b>24wt%-5</b>  | 52.42       | 13.67            | 2830            |
| <b>24wt%-6</b>  | 43.70       | 11.50            | 2470            |
| <b>24wt%-7</b>  | 47.03       | 19.61            | 3820            |
| <b>24wt%-8</b>  | 36.91       | 11.59            | 2490            |
| <b>24wt%-9</b>  | 41.49       | 29.93            | 5540            |
| <b>24wt%-10</b> | 53.63       | 16.99            | 3390            |

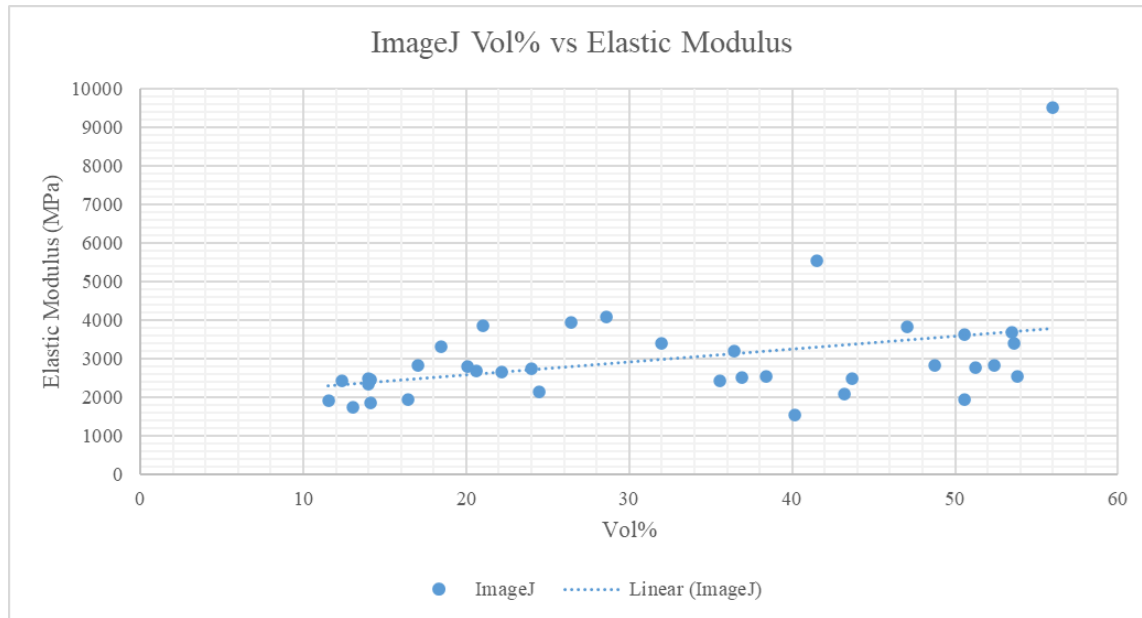


Figure 29: ImageJ analysis of vol%

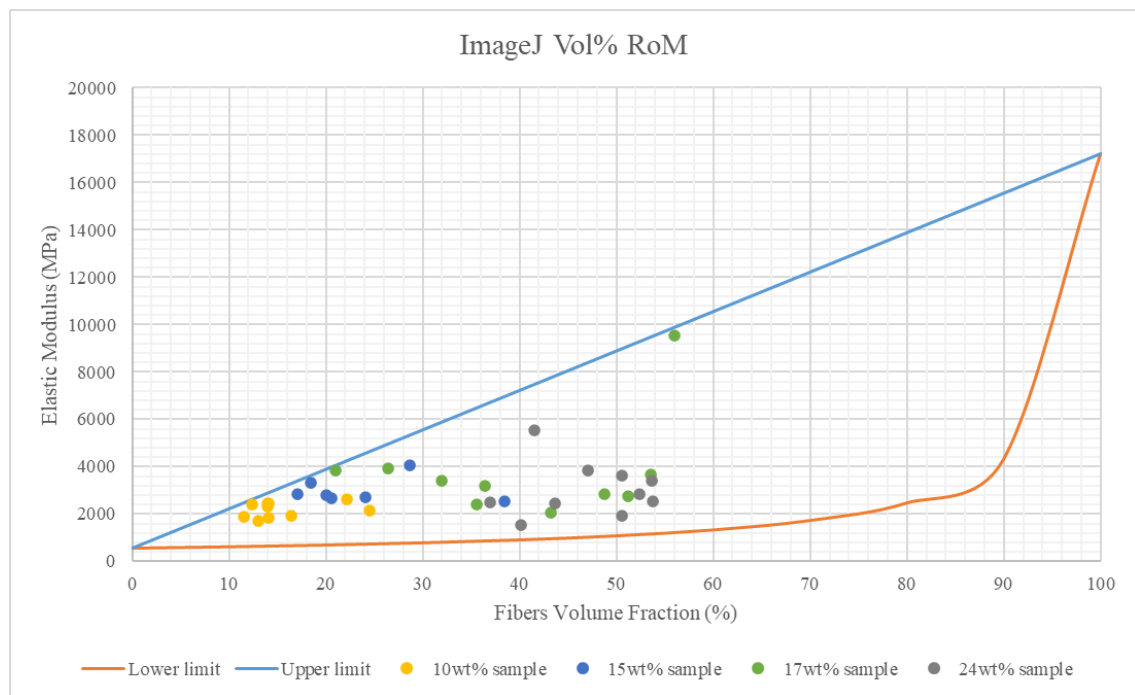


Figure 30: Samples modulus of elasticity data with the rule of mixture boundaries (ImageJ vol%)

Theoretically, the composite fails, in general, when it reaches the fibers' failure strain because the load is held by the fibers, and when the fibers fracture, the matrix will fail directly since its strength is lower than the fibers strength **Figure 31**.

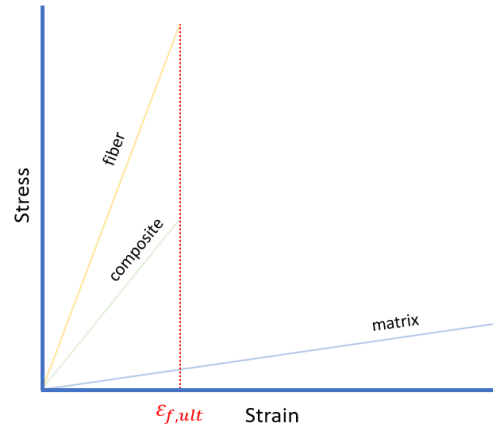


Figure 31: Theoretical failure point of composites

By comparing this work composite specimens' failure strain with the fibers' ones, the composite failure strain is lower than the lower range of the fibers' failure strain **Figure 32**.

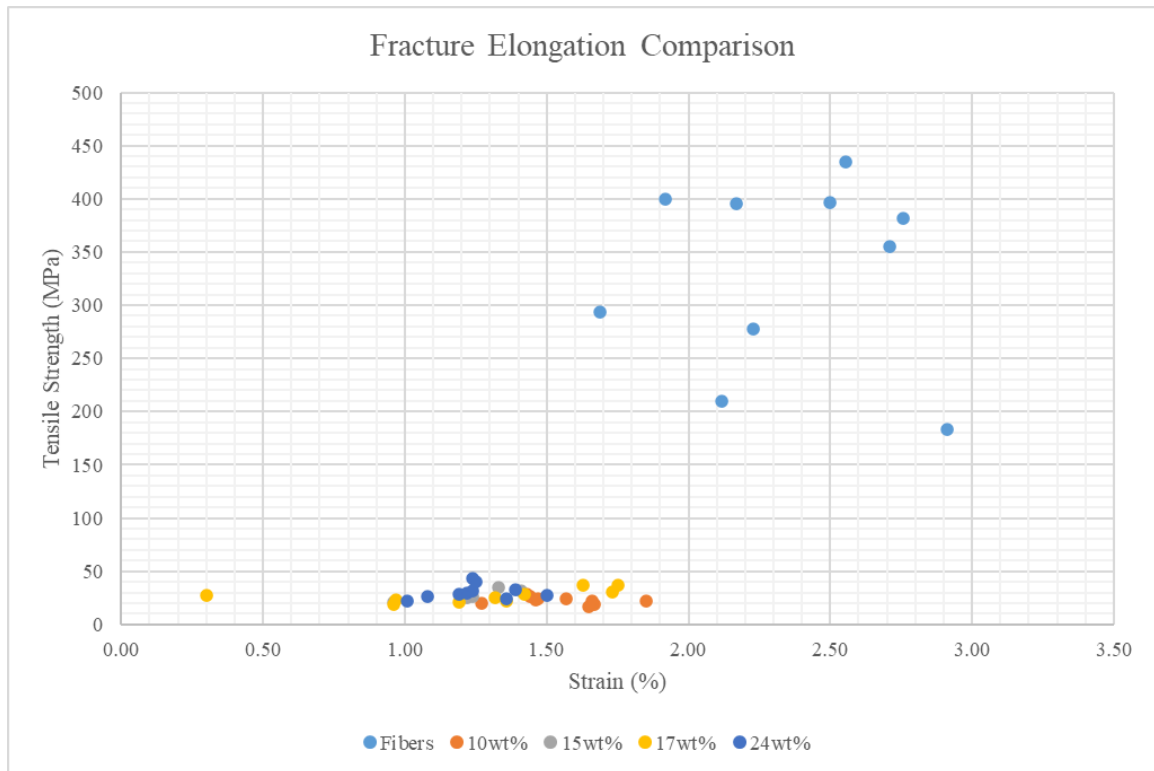


Figure 32: Comparison between specimen's failure strain and fiber's failure strain

Also, it can be noticed from **Figure 32** that the 10wt% sample is the closest to the fibers' fracture strain while the 24wt% is the furthest one. Since all the samples were made with the same treatment and procedure, so the interfacial strength effect can be excluded, and it can be attributed to the samples' defects that associated with the higher wt% samples. These defects are mainly voids that works as stress concentration and riser. These stress risers increase the applied stress locally at the void's sides, and when the local load reaches the ultimate strength of the matrix, a crack starts to grow through the specimen which leads to a failure at lower tensile strength.

There are two sources of voids in the composite samples. First, the small-scale gas bubbles resulted from the resin and hardener mixing. These bubbles are less effective since they are very small, most of them are on the upper surface, and their quantity was low. Second, the large-scale voids which is due to the bridging of the fibers and the less resin quantity as the wt% increase. The fibers bridging is due to the large fibers' diameter and length, in which, if there is a gap between two fibers bundles, they work as two bridges lifting the mold cover, since the pressure on the cover and the resin quantity are not high resulting in large voids. These large-scale voids were fixed by adding a surface layer of resin to fill these voids. The problem with 24 wt% sample was the internal voids between the fibers' layers

**Figure 33!**



Figure 33: (a) Top view of the large-scale voids, (b) side view of the internal voids between layers

Further support of the assumption that the main reason is the voids defect not the interfacial strength is by comparing the load required for fiber fracture and the load required for fiber debonding. Also, to count for the worst scenario, the lowest interfacial strength and the highest tensile strength values are going to be used for the comparison. Fiber fracture load ( $F=\sigma\pi r^2$ ) with  $\sigma = 435 \text{ MPa}$  and  $r = 0.2 \text{ mm}$ , the load required for fiber's fracture is 54.7 MPa. Fiber debonding load ( $F=2\sigma\pi r l_c$ ) with  $\sigma = 5.99 \text{ MPa}$  and  $l_c=7.3 \text{ mm}$ , the load required for fiber's debonding is 54.9 MPa. So, even with the worst scenario, the fibers are going to fracture before the debonding from the matrix. However, most of the samples showed a fiber pull-out after their fracture. **Figure 34** shows the variation in fiber pull-out between the specimen after the fracture. They range from the complete brittle fracture to the brittle fracture with long fiber pull-out. This pull-out can be attributed in the first place to the crack growth that accelerate the debonding between the fibers and the matrix. The

effect of the crack growth in the composites can be described by **Figure 35**. In the second place to the impurities that reduce the interfacial strength.

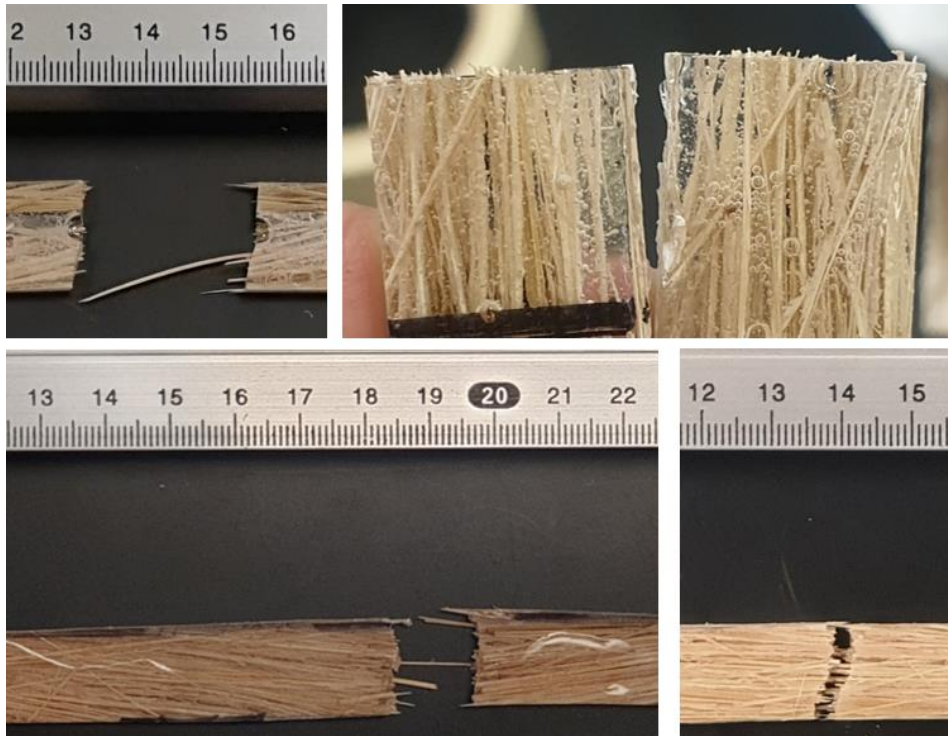


Figure 34: Samples of the test specimens showing the variation in the fiber pull-out after the fracture

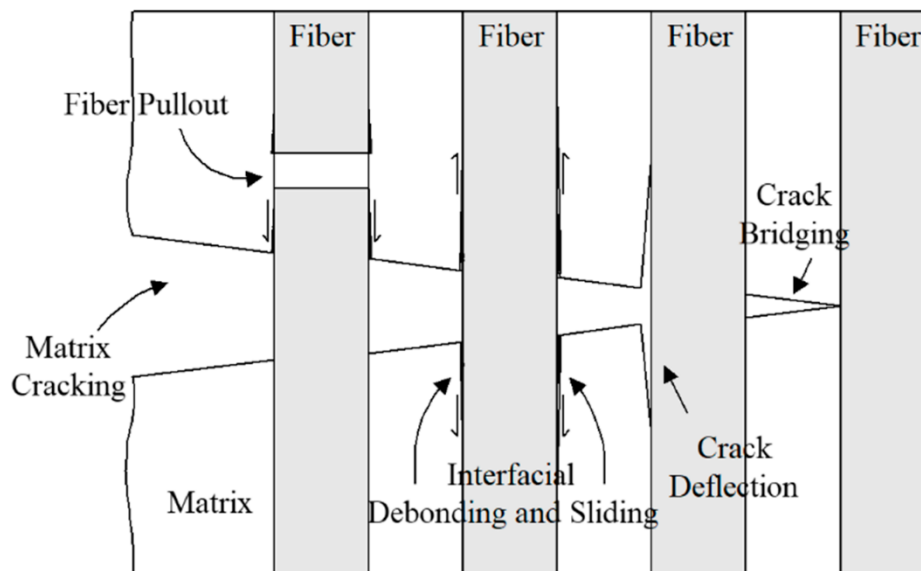


Figure 35: The effect of crack growth in the composite materials [52]

Comparing the experimental values of the ultimate tensile strength with the RoM model shows that the ultimate tensile strength data points of the samples are extremely below the theoretical limit (Figure 36). 10wt% sample is the closest and the 24wt% is too far because of the defects associated with the high wt% as described earlier. 15wt% and 17wt% have a wide range of variation because of the variation in defects concentration. For comparison, theoretical ultimate tensile strength was calculated using Hook's law by using the experimental modulus of the composite and the fiber's mean ultimate strain (2.36 %). The theoretical ultimate tensile strength points represent the values that the experimental results should be if their stress-strain curve was perfectly linear, and they reach the ultimate fiber strain.

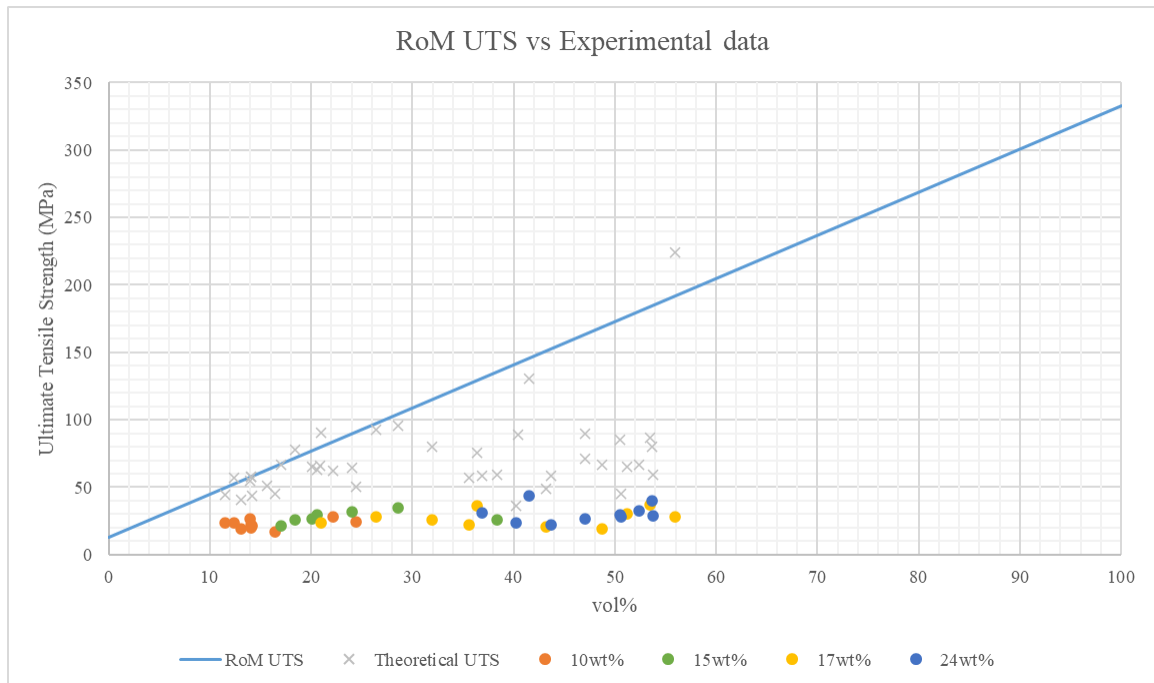


Figure 36: Comparison between the experimental data, RoM limit, and the theoretical data of the ultimate tensile test

Even though with the presence of such defects that reduces the mechanical properties, the comparison with the other literatures or with the commercial wood products, such as MDF, OSB, and plywood, shows that date palm fibers treated with water have a promising future as a composite reinforcement. In the literatures, there is a very high variation in the results and that is because of variation in the type, treatments, and testing of the date palm fibers. Anyway, literatures that used epoxy as the matrix and fronds as the fibers source, as much as available, will be considered for more consistent comparison. These conditions make the available literatures is limited, so the fibers from other sources from the date palm tree with the epoxy will be used for the comparison also. the following table shows the available literatures results:

*Table 13: Comparison with the mechanical properties of the epoxy-DPF composites*

| Fibers' type            | Matrix type | Chemical treatment         | Tensile strength (MPa)             | Modulus of elasticity (GPa)          | Reference |
|-------------------------|-------------|----------------------------|------------------------------------|--------------------------------------|-----------|
| <b>Frond (Midrib)</b>   | Epoxy       | Un-treated                 | 16.9 – 43.8                        | 1.7 – 9.5                            | This work |
| <b>Frond (petioles)</b> | Epoxy       | Un-treated<br>NaOH         | 35<br>37 – 45                      | 0.5 – 0.59                           | [3]       |
| <b>Frond</b>            | Epoxy       | Un-treated                 | 26.45                              | 1.42                                 | [53]      |
| <b>Mesh (sheath)</b>    | Epoxy       | NaOH                       | 21 – 41                            | 0.6 – 2.5                            | [4], [54] |
| <b>Mesh (sheath)</b>    | Epoxy       | HCl<br>Acetic acid<br>NaOH | 28 – 132<br>32 – 215<br>63 – 141.6 | 0.8 – 12.1<br>1.7 – 31.4<br>1.5 – 16 | [11]      |
| <b>DPF + Coir</b>       | Epoxy       | NaOH<br>HCl                | 20.45 –<br>27.22                   | 3.5 – 6.3                            | [17]      |

On the other hand, the most important is to compare this work with the commercial products to find out if the DPF composites can substitute these products. For MDF, the majority of the standards are in the range of 2 – 3 GPa for modulus of elasticity which is within the range of the DPF composite.

*Table 14: Comparison with the mechanical properties of the commercial wood products*

| Product                                | Test type | Tensile/Bending strength (MPa)  | Modulus of elasticity (GPa) | Reference |
|----------------------------------------|-----------|---------------------------------|-----------------------------|-----------|
| <b>Midrib DPF composite</b>            | Tensile   | 16.92 – 43.77                   | 1.7 – 9.5                   | This work |
| <b>MDF</b>                             | Bending   | 22 – 40                         | 1.4 – 3.3                   | [55]–[58] |
| <b>TPB<br/>MDF<br/>HFB<br/>Plywood</b> | Tensile   | 7.4 – 7.9<br>9<br>23<br>15 – 30 |                             | [59]      |
| <b>Softwood</b>                        | Tensile   |                                 | 8.3 – 13.28                 | [60]      |
| <b>OSB</b>                             | Tensile   | 3.8 – 11.15                     | 1.6 – 5.15                  | [61]      |

From this short comparison restricted to the available epoxy-DPF from the frond and the mesh fibers literatures only, fibers extracted and treated by water boiling only can have competitive results with the chemical treated fibers from the literatures and with the commercial products which can substitute also.

## **CHAPTER 7**

### **CONCLUSION**

Composite materials field is very wide with variety of factors and various applications. These applications range from high technology of aerospace and automobile application to furniture and kitchen tools. This multiple usage of composite materials is due to their dynamic properties. These materials can combine divergent properties that meets unique needs. This main advantage makes composite materials to be a solution for many technical issues and to be involved in a lot of applications.

Sustainability can also be achieved by composite materials by using renewable resources to make the composite. The scientific and technical community return toward the natural renewable resources to substitute the synthetic products with natural ones that have comparable properties. One of these renewable ecofriendly resources are the agricultural residue which can be used to make composites with suitable properties. By extracting the cellulosic fibers to be used as reinforcement, and lignin can be used as a natural resin, composites can be made from completely natural resources.

In Saudi Arabia, millions of date palm trees are pruned every year which produce hundreds of thousand tons of agricultural residue. These residue materials are a potential resource to be converted into valuable products with low costs. This motivates us to work on this project to figure out the best way of changing these residue materials to products that compete the imported products with much lower costs. One great solution is by using the natural fibers in these agricultural residues to make composite materials with properties

comparable to those made by synthetic products. With this approach, the release of carbon dioxide emissions into the environment will be averted by redirecting agricultural residues which typically burned into valuable products, and the national income will be supported by turning wastes to valuable products.

At the end of the first chapter of this project, the results are promising and are competitive with the commercial products in the market even at this first stage of the project. The goal of reducing the use of chemical as much as possible was achieved in the extraction and treatment processes by using the hydrothermal process. The remaining is to reduce the chemical in the matrix by producing a bio-resin with good mechanical properties to be reinforced with these natural fibers to produce a completely bio-composite. Finally, the date palm is a blessed tree full of benefits and we have to exploit all its potentials with zero waste.

## **Future Work:**

These promising results are exciting for more improvement to overcome the defects and limitation through this first part of this project. All the factors used should be revised and tested again. First, the extraction and treating processes can be improved by studying the effect of the boiling temperature and duration on the removal of the impurities, and water-soluble materials which reduce the interfacial strength and mechanical properties. Higher temperature and pressure can be used to remove these unwanted materials since the high temperature reduce their bond with the cellulose fibers. Second, using more fibers mass in the preparation stage of the composite and compacting these fibers well before and after the mixing with resin to insure even distribution of the fibers and consistency in the wt% of all the specimens. Characterization of the fibers extracted by this method is very important to improve the interfacial strength which in turn improve the mechanical properties of the composite. Finally, the most important is to extend the goal of this work to the matrix material by using bio-resin. It would be the best is if this bio-resin is made of the extraction process by-product which are lignin and hemicellulose. These materials are biopolymers that works as a matrix holding the cellulosic fibers and supporting them. By using these biopolymers, the waste from the date palm trees can be reduced up to zero percent and the need of synthetic and harmful chemical can be reduced.

## APPENDIX (A)

The derivation of [equation \(2\)](#), where the applied load direction is parallel to the fiber's direction (longitudinal), is based on the following assumptions:

$$\varepsilon_c = \varepsilon_m = \varepsilon_f \quad (13)$$

The first assumption ([equation \(13\)](#)) is that the strain is the same for the fibers and the matrix (isostrain). This means that a perfect bonding between the fibers and the matrix is assumed which is not the case in the real composite.

This leads to the second assumption ([equation \(14\)](#)) which is the total load applied on the composite is equal to the sum of the loads held by the fibers and the matrix.

$$F_c = F_f + F_m \quad (14)$$

From the stress equation ( $F = \sigma A$ ), [equation \(14\)](#) can be written as:

$$\sigma_c A_c = \sigma_f A_f + \sigma_m A_m \quad (15)$$

Dividing all the sides by  $A_c$ :

$$\sigma_c = \sigma_f \frac{A_f}{A_c} + \sigma_m \frac{A_m}{A_c} \quad (16)$$

Since  $A_c$  represents the total cross section area and the length is assumed to be the same, then  $\frac{A_f}{A_c}$  and  $\frac{A_m}{A_c}$  represents the fibers volume fraction  $V_f$  and the matrix volume fraction  $V_m$ , respectively. Now, [equation \(16\)](#) can be written as:

$$\sigma_c = \sigma_f V_f + \sigma_m V_m \quad (17)$$

Equation (17) can be used to predict the tensile strength of the composite if the load is in the parallel to the fiber's direction. It is considered as the upper theoretical bound.

For the elastic modulus, the first assumption and the Hook's law are used by dividing each side with the corresponding strain:

$$\frac{\sigma_c}{\varepsilon_c} = \frac{\sigma_f}{\varepsilon_f} V_f + \frac{\sigma_m}{\varepsilon_m} V_m \quad (18)$$

$$E_c = E_f V_f + E_m V_m \quad (19)$$

Equation (19) is the basic formula to predict the upper bound of the composite elastic modulus. In equation (2), it is denoted by  $E_{cl}$  where the subscript ( $l$ ) means (longitudinal) to differentiate between it and the transvers one which is denoted by  $E_{ct}$  in equation (3).

Equation (3) represent the lower bound from the RoM when the applied load is perpendicular to the fiber's direction (transvers). In this case, it is difficult to derive it in a fiber form, but a composite made of layers with the same cross section area will make the derivation easier. The derivation is based on the following assumptions:

$$\sigma_c = \sigma_m = \sigma_f \quad (20)$$

The constant stress (isostress) is due to constant cross section area of the layers and the change is only in the total length of the composite which is the sum of all constituent layers' length:

$$L_c = L_m + L_f \leftrightarrow \Delta L_c = \Delta L_m + \Delta L_f \quad (21)$$

Since the strain of each part is  $\varepsilon_c = \frac{\Delta L_c}{L_c}$ ,  $\varepsilon_m = \frac{\Delta L_m}{L_m}$ ,  $\varepsilon_f = \frac{\Delta L_f}{L_f}$  then, **equation (21)** can be written as:

$$\varepsilon_c L_c = \varepsilon_m L_m + \varepsilon_f L_f \quad (22)$$

Dividing all terms with the total length  $L_c$ :

$$\varepsilon_c = \frac{\varepsilon_m L_m + \varepsilon_f L_f}{L_c} \quad (23)$$

Since the area is constant, then the length ratios  $\frac{L_m}{L_c}$  and  $\frac{L_f}{L_c}$  are equivalent to matrix volume fraction  $V_m$  and fibers' volume fraction  $V_f$ . Also, by the Hook's law, **equation (23)** can be written as:

$$\frac{\sigma_c}{E_c} = \frac{\sigma_m}{E_m} V_m + \frac{\sigma_f}{E_f} V_f \quad (24)$$

Since all stresses are equal, they cancel each other, and the final form will be

$$E_c = \frac{E_m E_f}{E_f V_m + E_m V_f} \quad (25)$$

**Equation (25)** is the basic formula to predict the lower bound of the composite elastic modulus. In **equation (3)**, it is denoted by  $E_{ct}$  where the subscript ( $t$ ) means (transvers) to differentiate between it and the longitudinal one which is denoted by  $E_{cl}$  in **equation (2)**.

## APPENDIX (B)

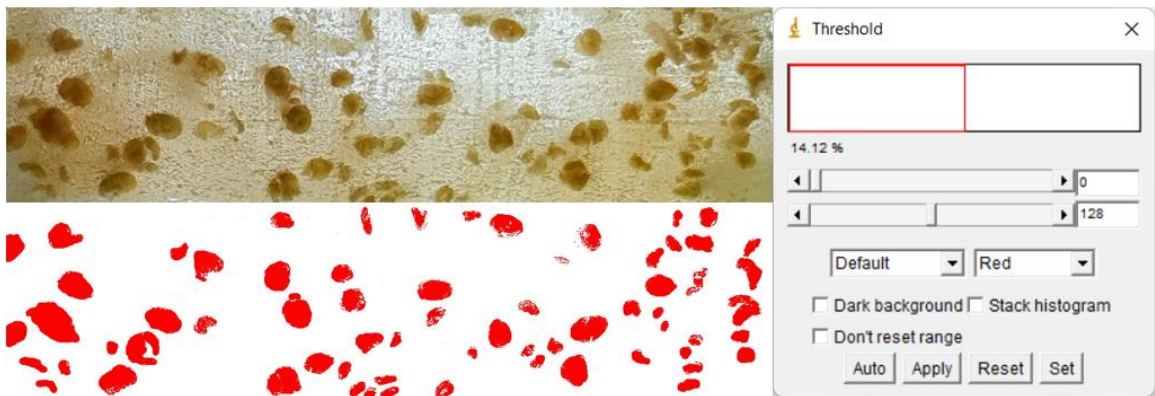
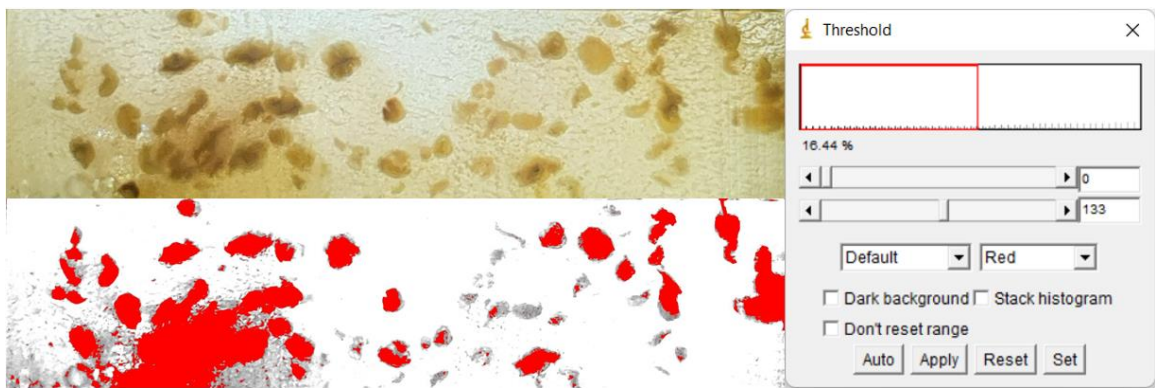
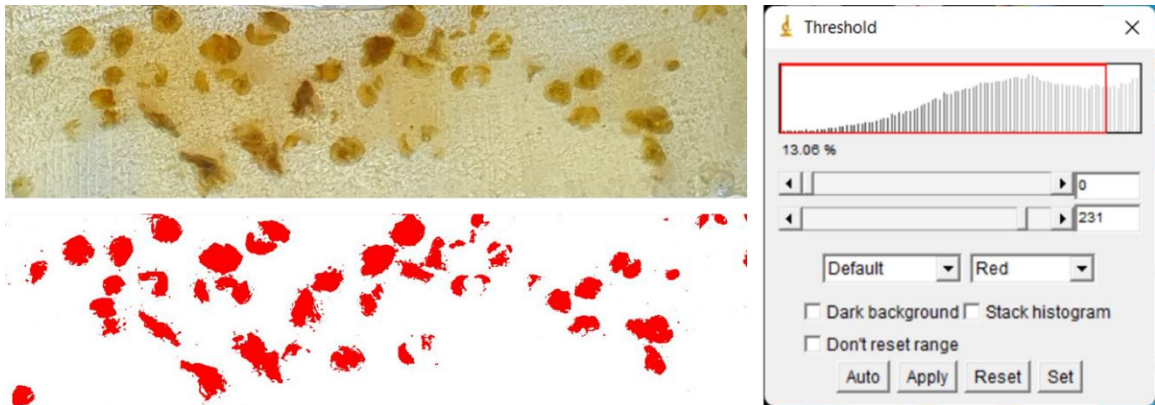
All the specimens' images are from the edge cross section except for the outlier specimens which are 17wt%-8 and 24wt%-9 their images are at the fracture surface. Images were taken by illuminating the clear resin with two light sources on the sides of the specimen so that the resin will be light, and the fibers will be dark.

ImageJ software version is Java 1.8.0\_172 (64-bit) from <https://imagej.nih.gov/ij> .

Analysis method was by:

1. Changing the image to 8-bit by Image > Type > 8-bit. This will change the image to the grayscale.
2. Preparing the image until as much as possible acceptable contrast between the resin and the fibers is achieved by the different tool that the software provides from Image > Adjust or Process tabs. In some images with high wt% or high noise, the selection tool was used to select the fibers or to erase the noise from the image manually. This step takes long time to get the most accurate result as much as possible and to reduce any chance of bias.
3. Finally, using the threshold tool to find the fibers area percentage from the prepared image by Image > Adjust > Threshold. The result shown by the software was taken without any change in the setups to eliminate the bias.

Following are the images of all the tested specimens for wt% analysis using ImageJ:



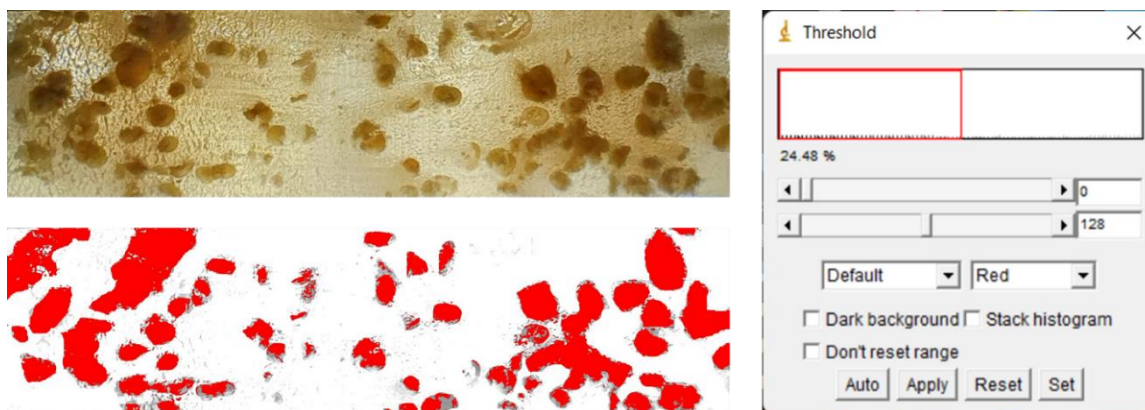


Figure 40: ImageJ analysis of specimen 10wt%-4

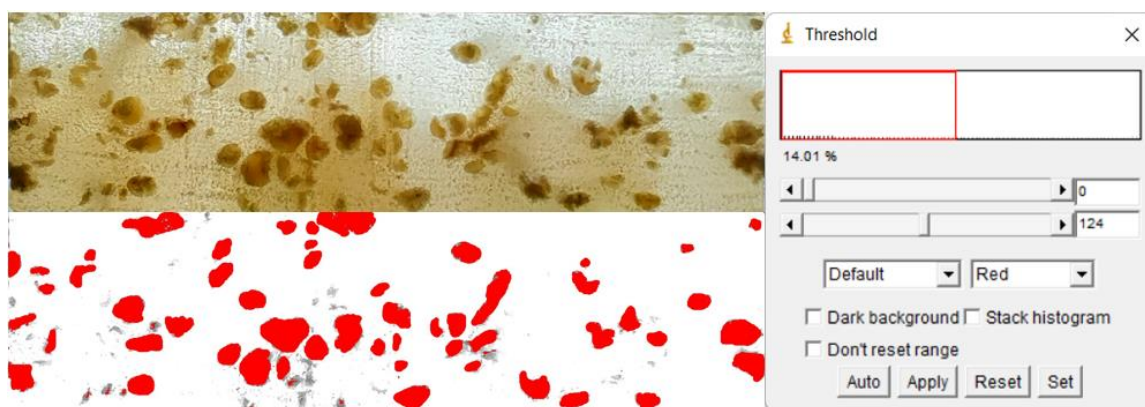


Figure 41: ImageJ analysis of specimen 10wt%-5

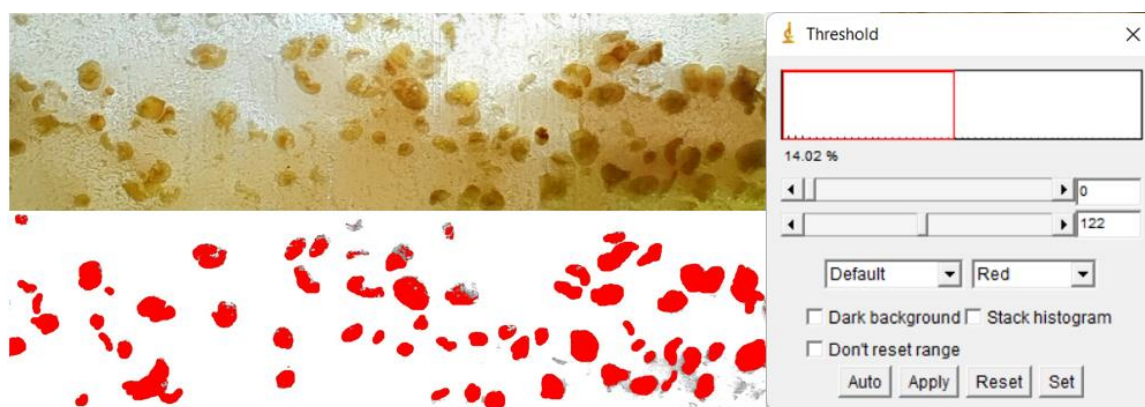


Figure 42: ImageJ analysis of specimen 10wt%-6



Figure 43: ImageJ analysis of specimen 10wt%-7

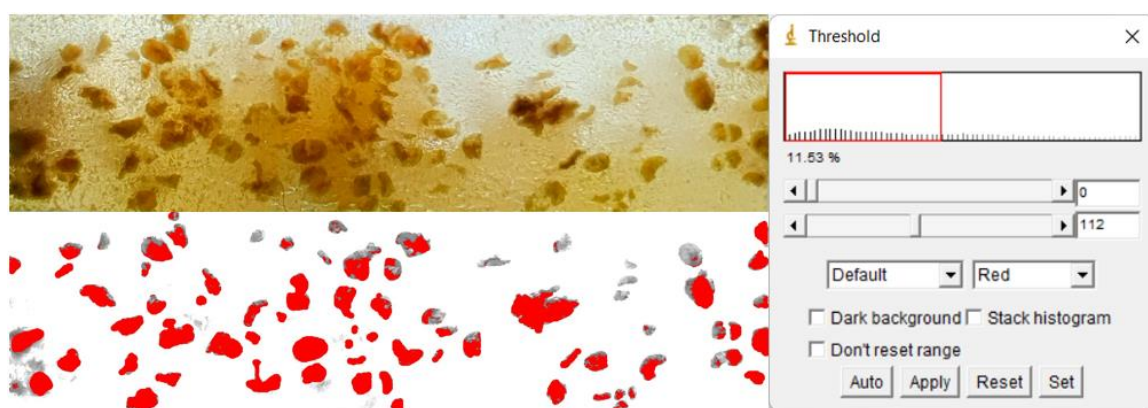


Figure 44: ImageJ analysis of specimen 10wt%-8



Figure 45: ImageJ analysis of specimen 10wt%-9

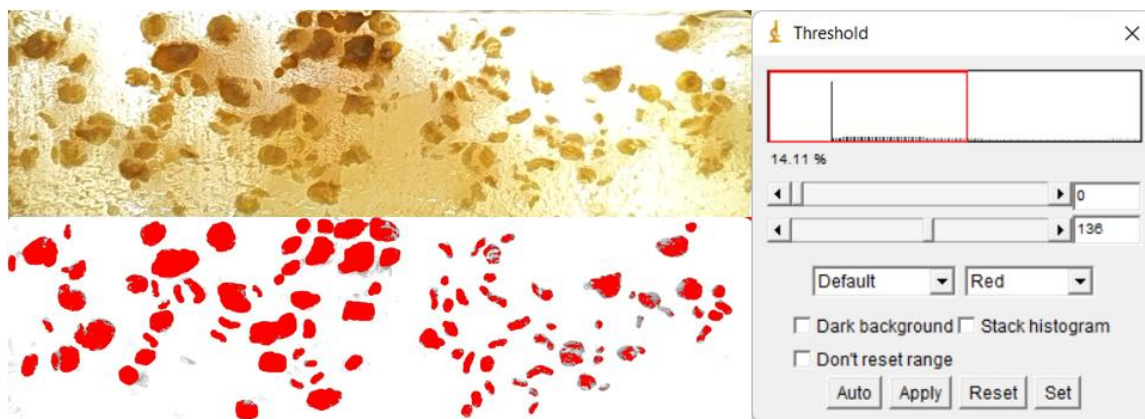


Figure 46: ImageJ analysis of specimen 10wt%-10

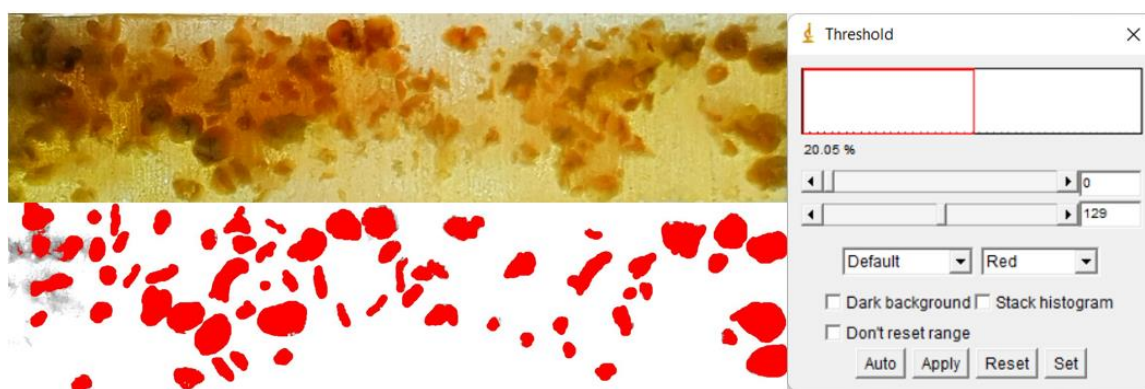


Figure 47: ImageJ analysis of specimen 15wt%-10

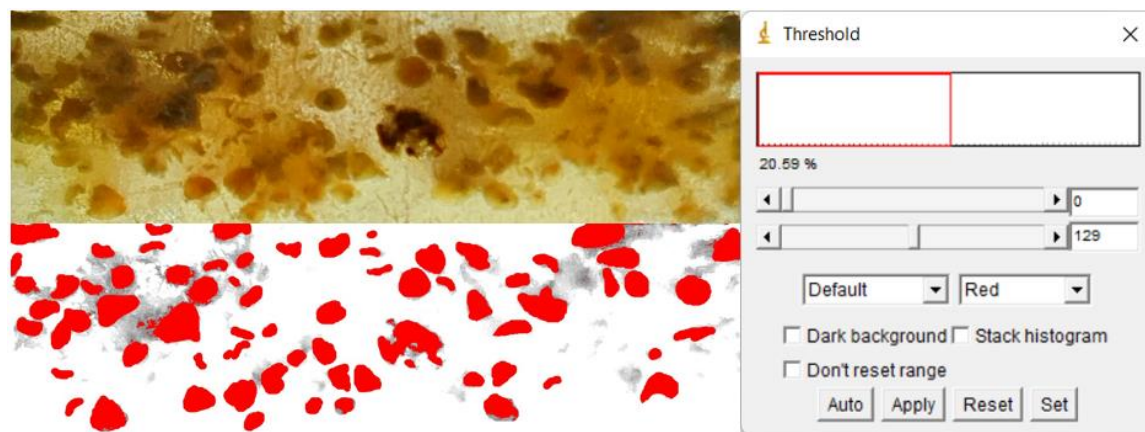


Figure 48: ImageJ analysis of specimen 15wt%-11

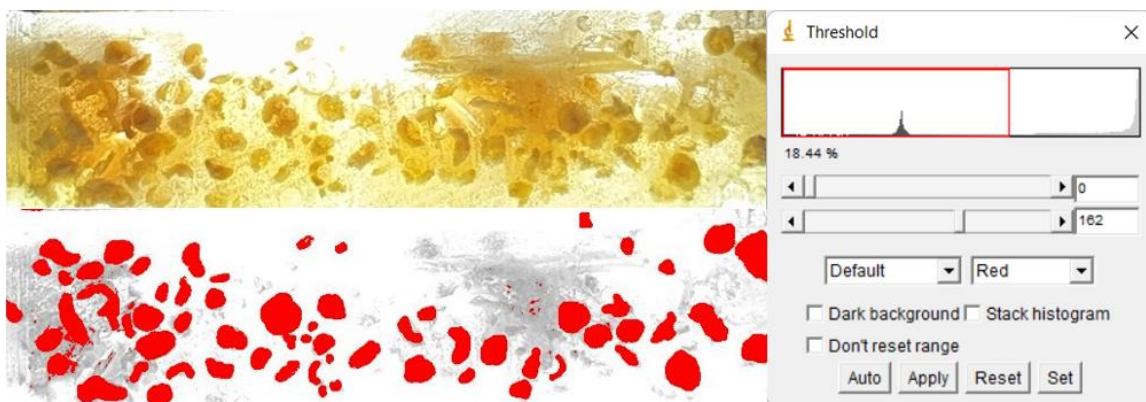


Figure 49: ImageJ analysis of specimen 15wt%-12

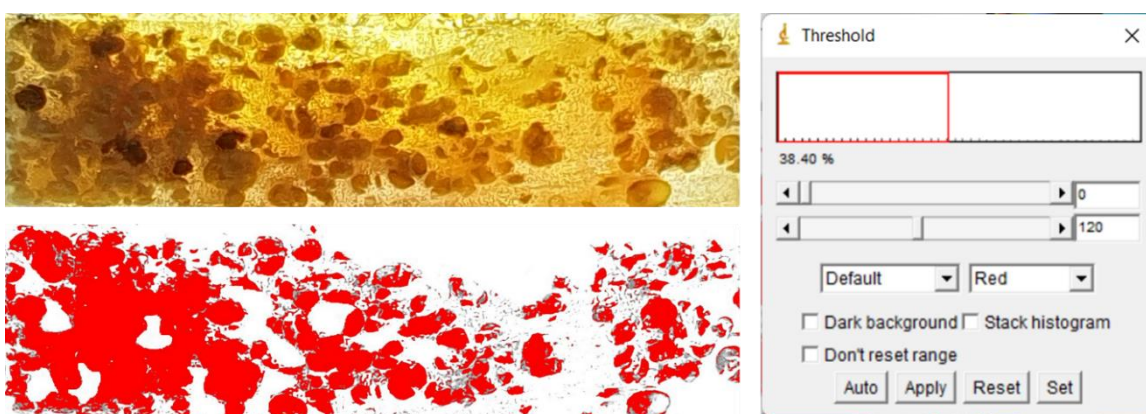


Figure 50: ImageJ analysis of specimen 15wt%-13

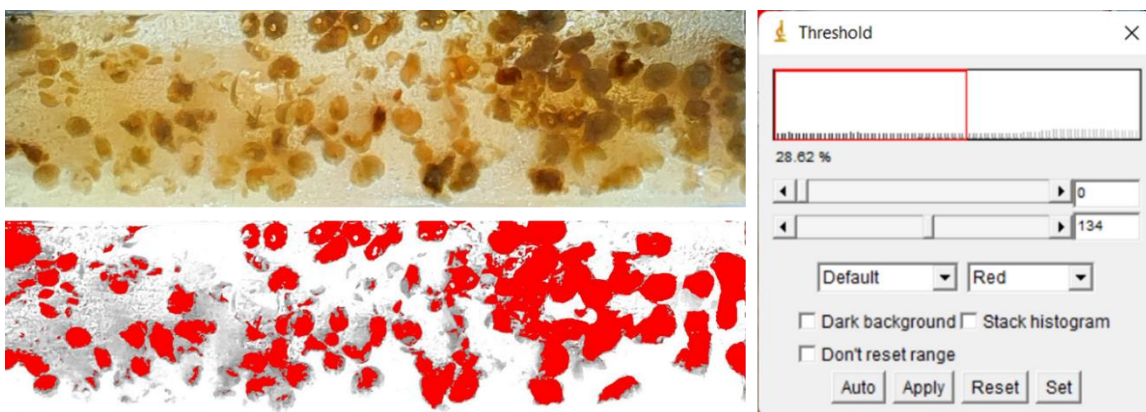


Figure 51: ImageJ analysis of specimen 15wt%-14

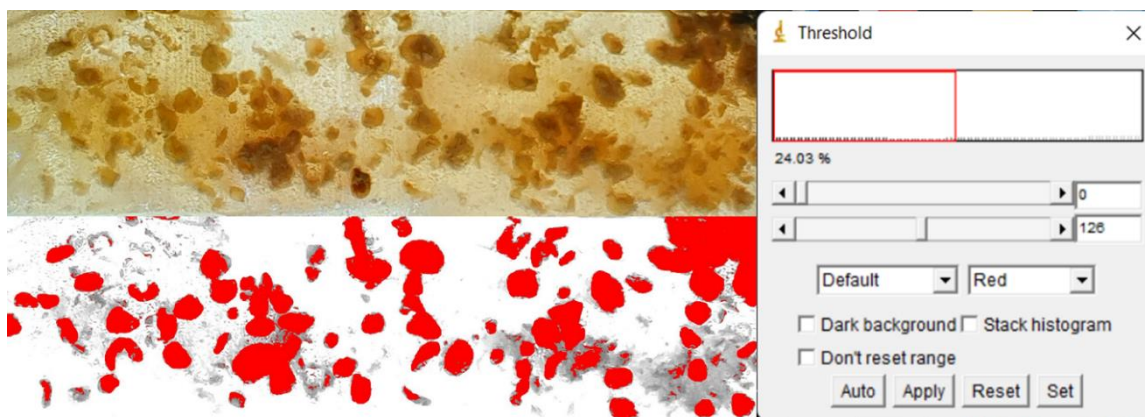


Figure 52: ImageJ analysis of specimen 15wt%-15

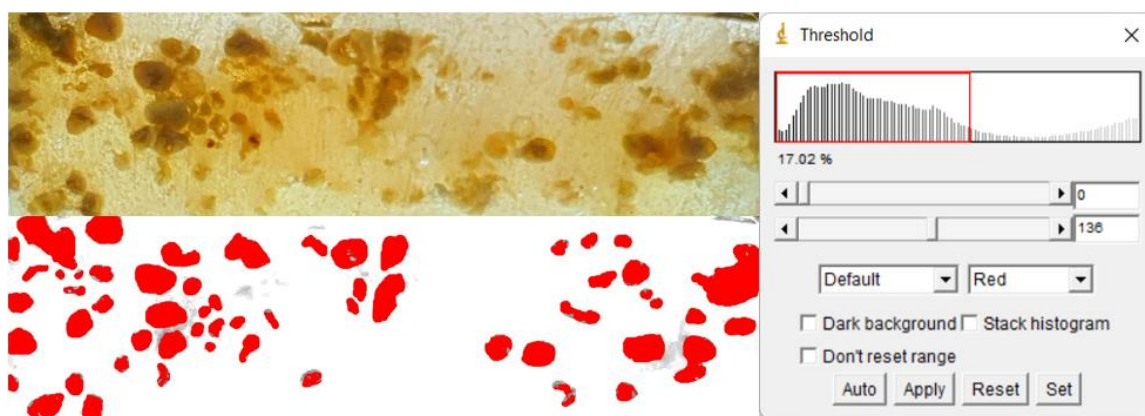
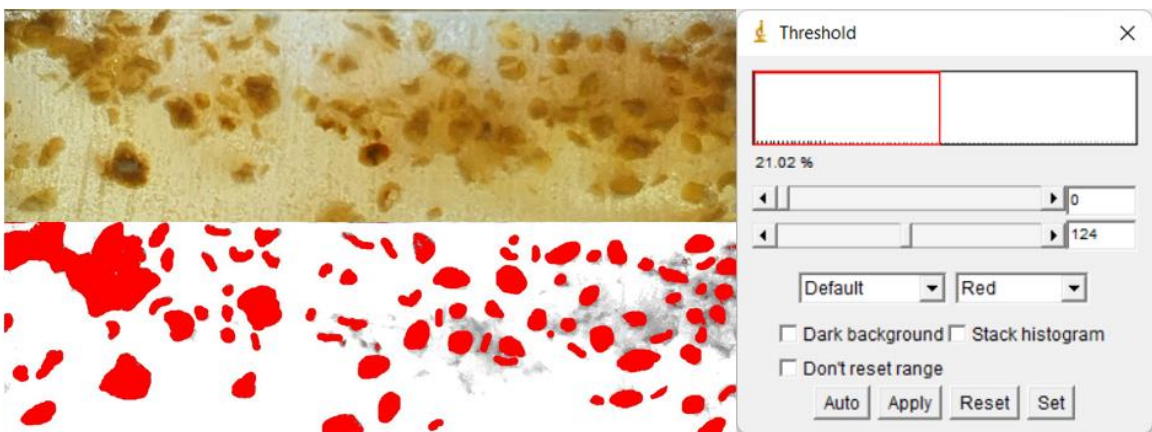
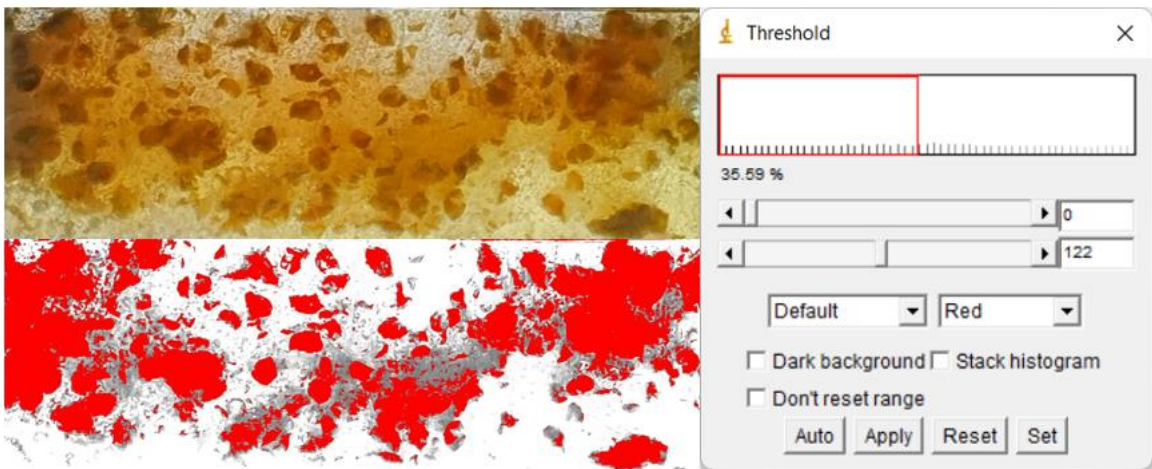
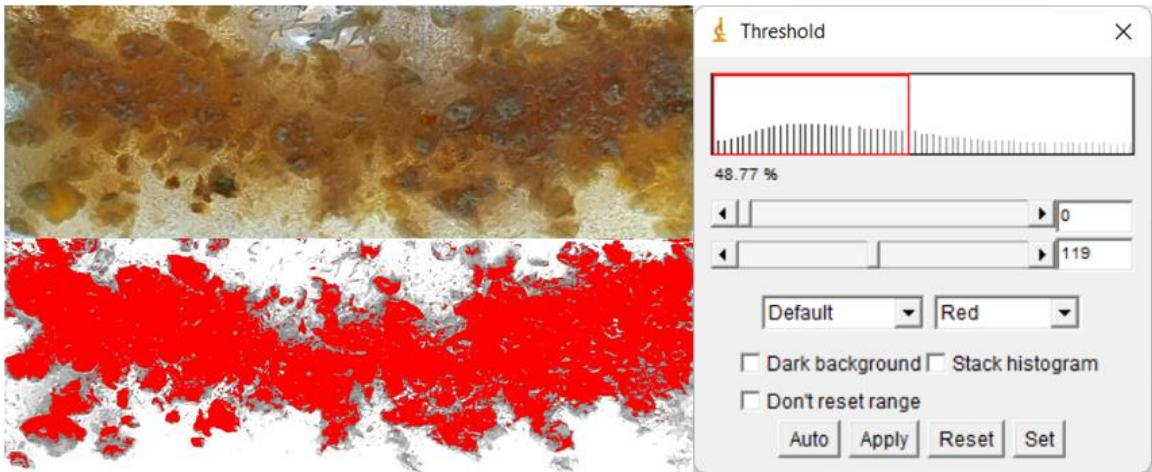


Figure 53: ImageJ analysis of specimen 15wt%-16



Figure 54: ImageJ analysis of specimen 17wt%-1



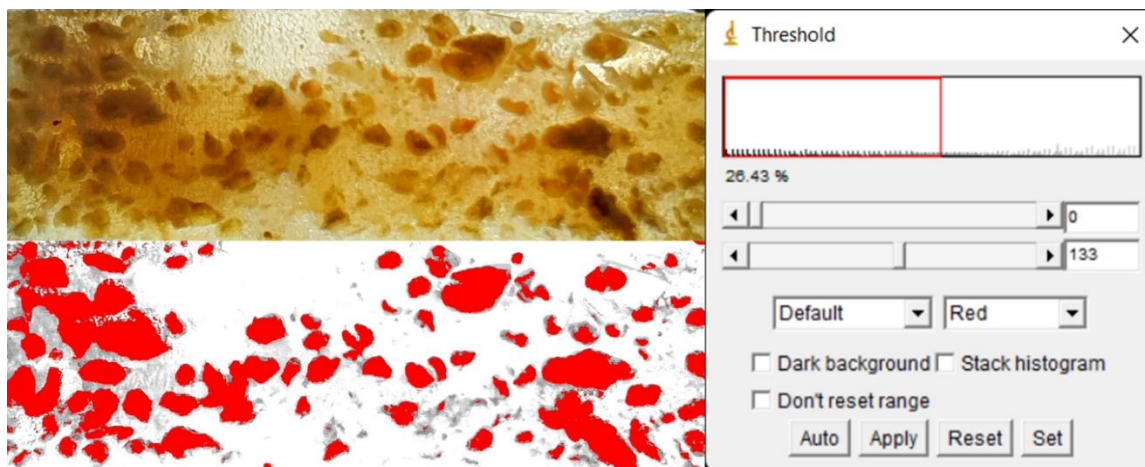


Figure 58: ImageJ analysis of specimen 17wt%-5

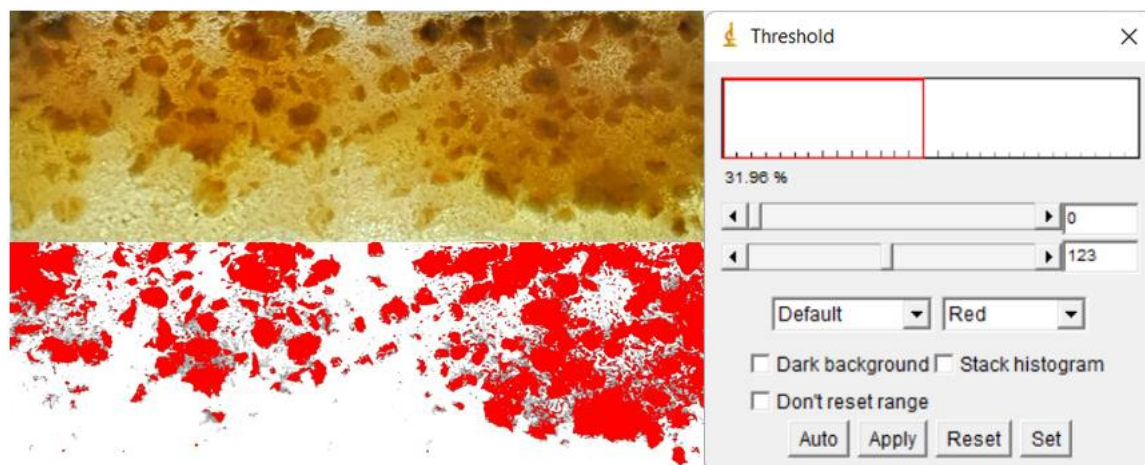


Figure 59: ImageJ analysis of specimen 17wt%-6

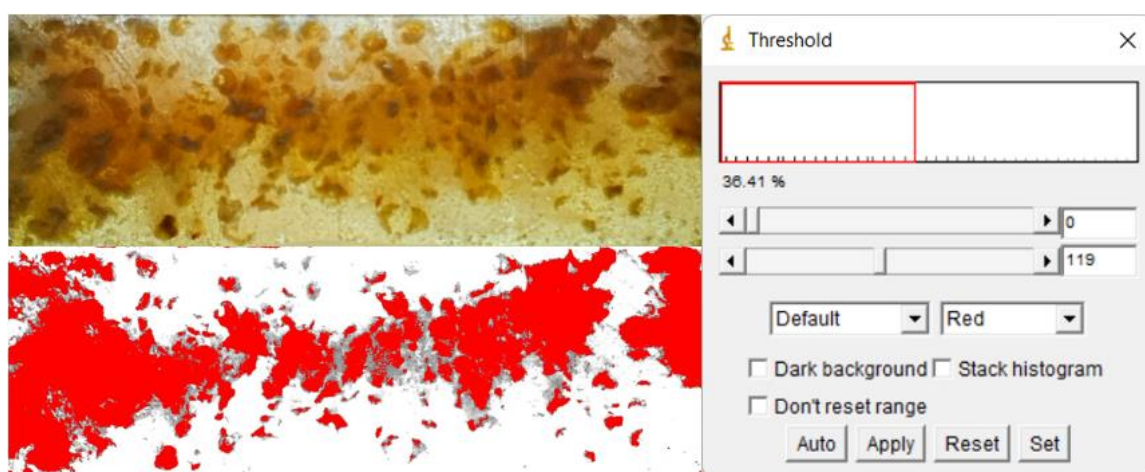


Figure 60: ImageJ analysis of specimen 17wt%-7

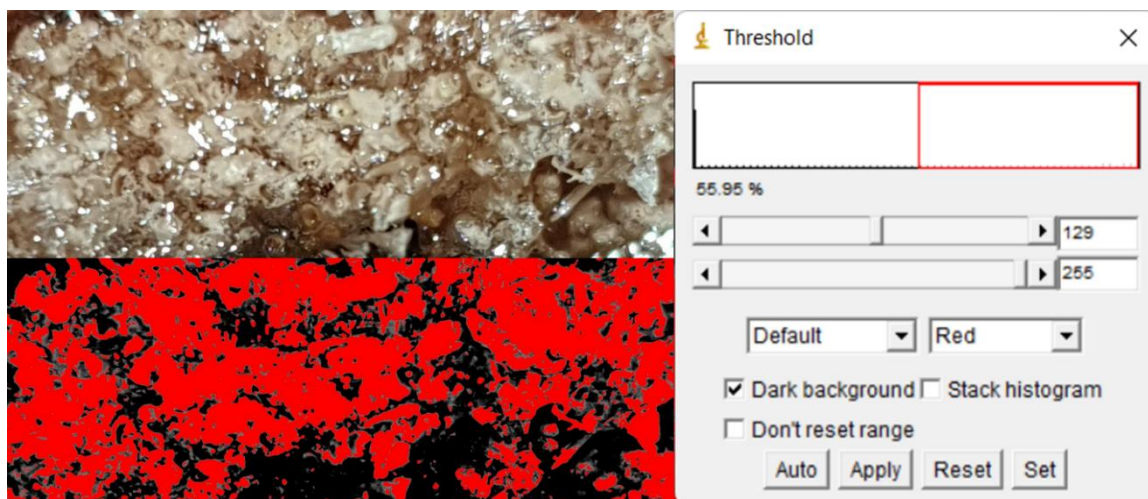


Figure 61: ImageJ analysis of specimen 17wt%-8

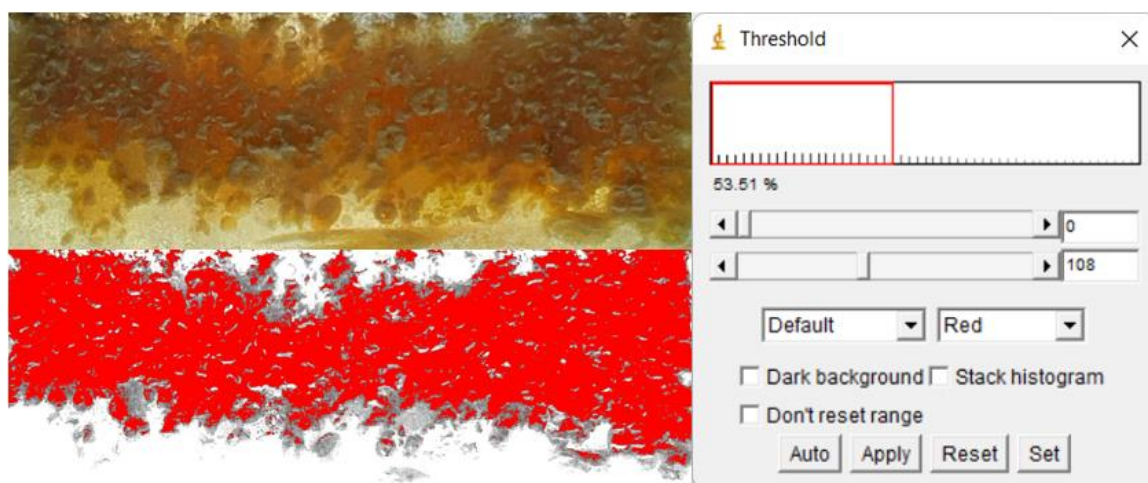


Figure 62: ImageJ analysis of specimen 17wt%-9

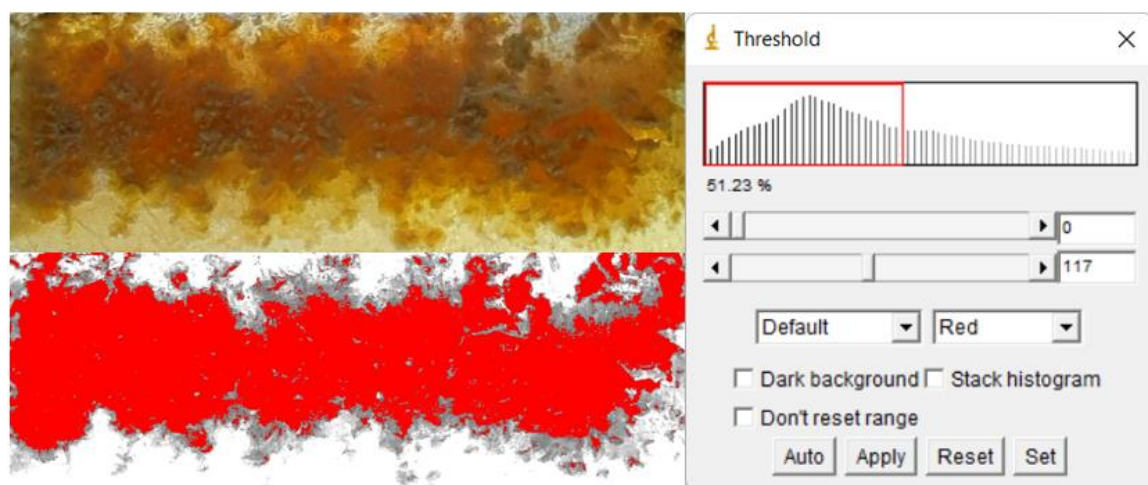


Figure 63: ImageJ analysis of specimen 17wt%-10

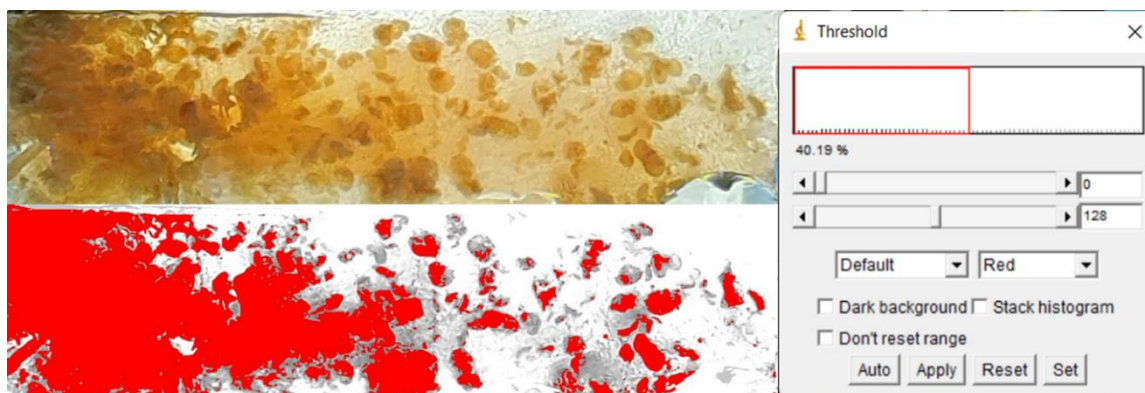


Figure 64: ImageJ analysis of specimen 24wt%-1

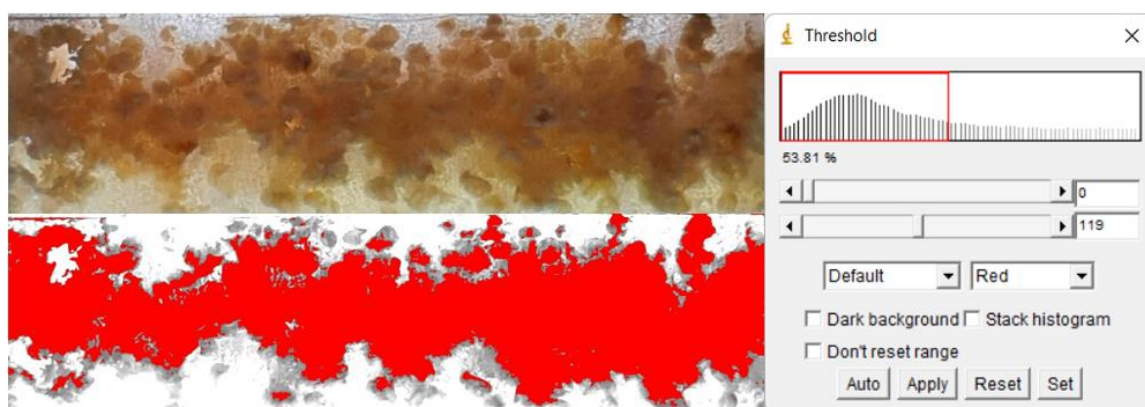


Figure 65: ImageJ analysis of specimen 24wt%-2

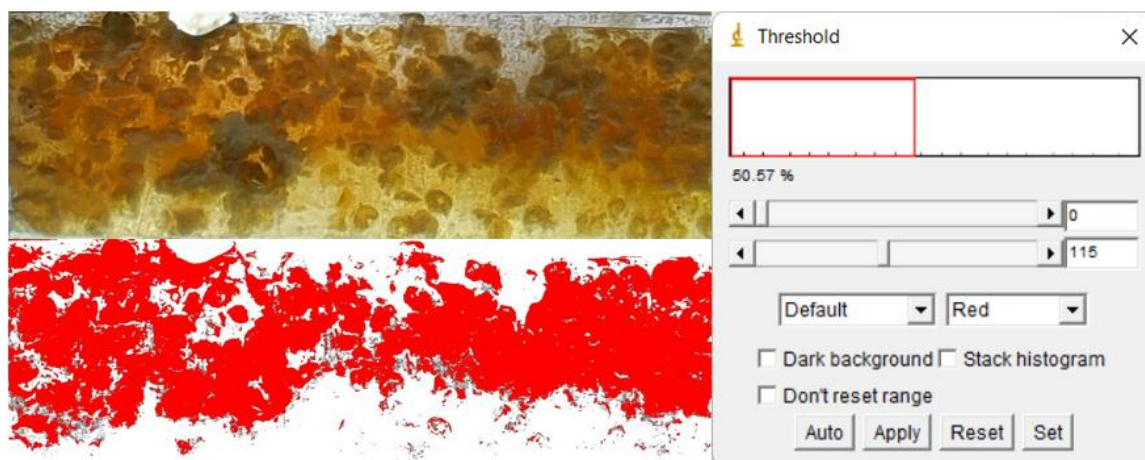


Figure 66: ImageJ analysis of specimen 24wt%-3

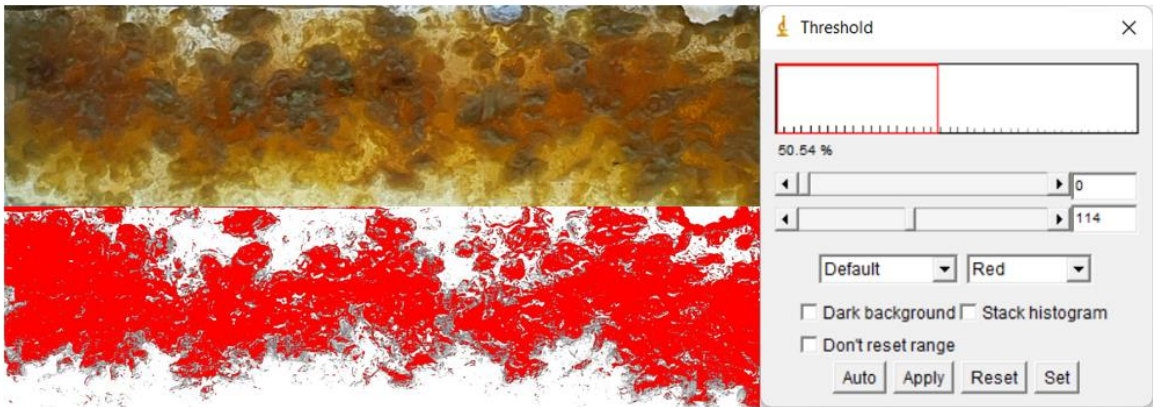


Figure 67: ImageJ analysis of specimen 24wt%-4

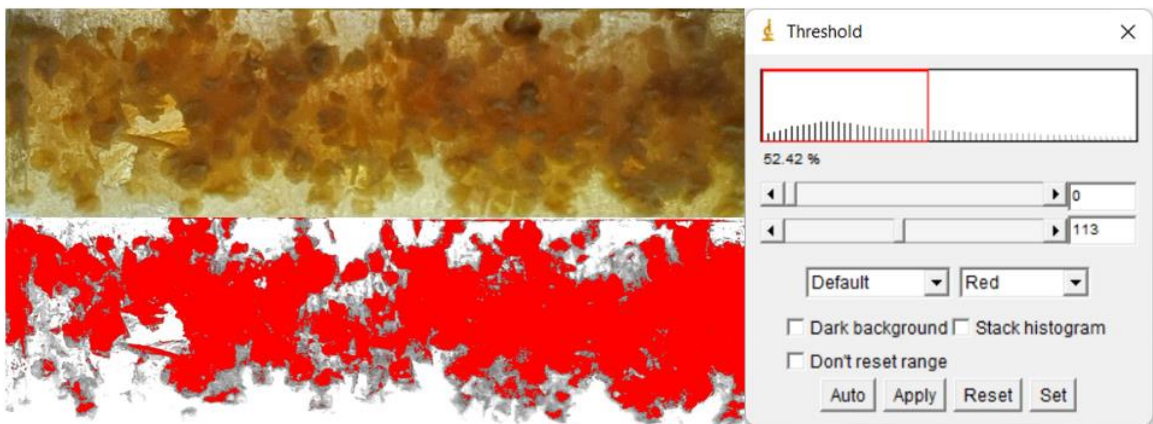


Figure 68: ImageJ analysis of specimen 24wt%-5

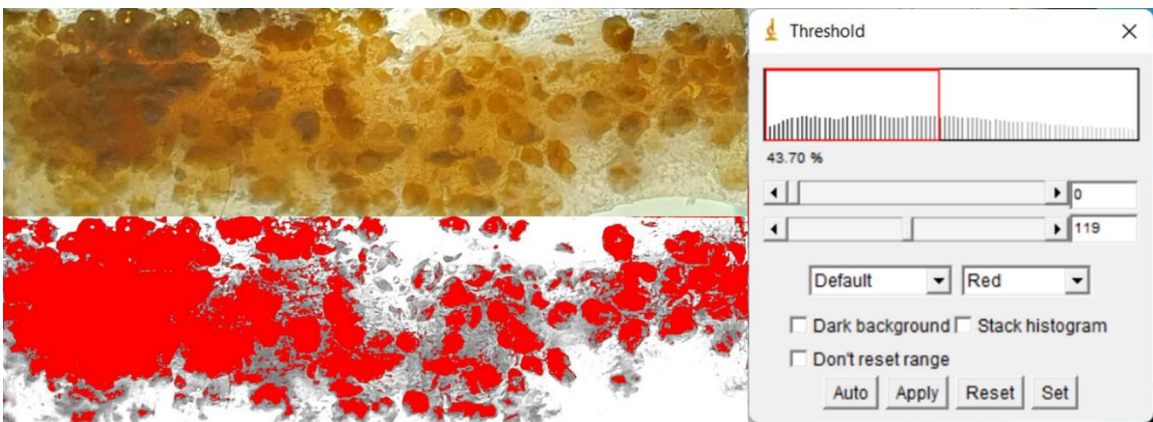


Figure 69: ImageJ analysis of specimen 24wt%-6

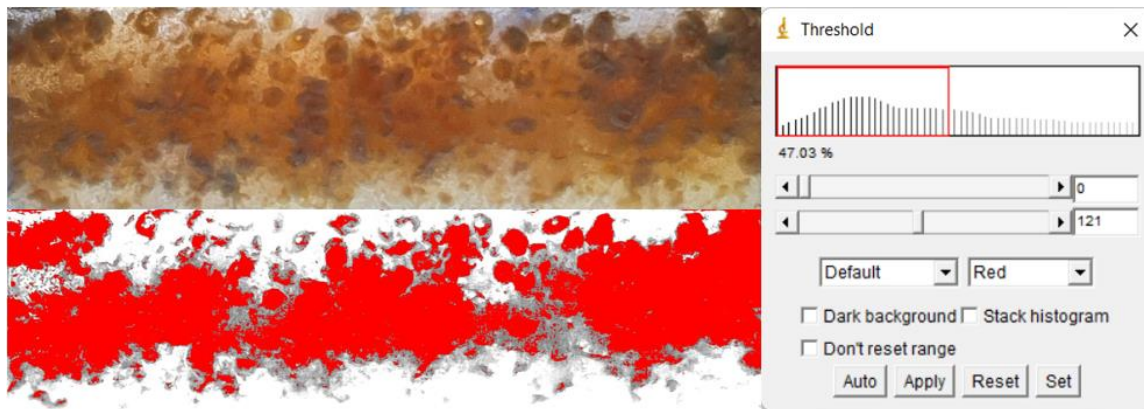


Figure 70: ImageJ analysis of specimen 24wt%-7

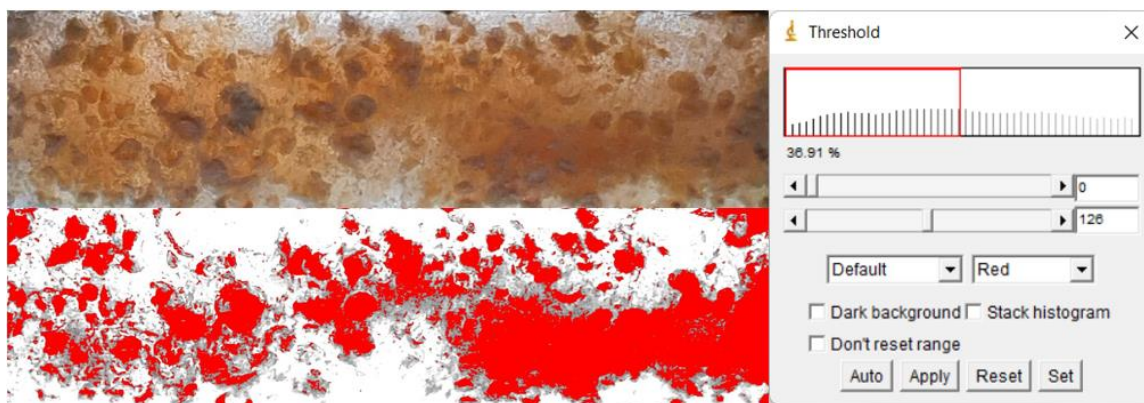


Figure 71: ImageJ analysis of specimen 24wt%-8

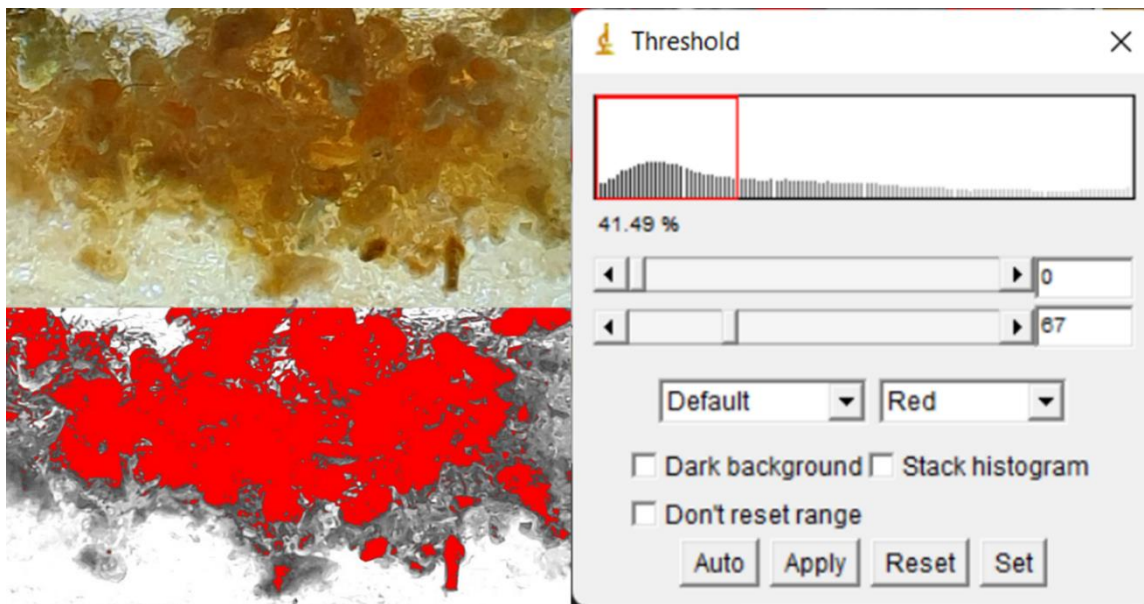


Figure 72: ImageJ analysis of specimen 24wt%-9

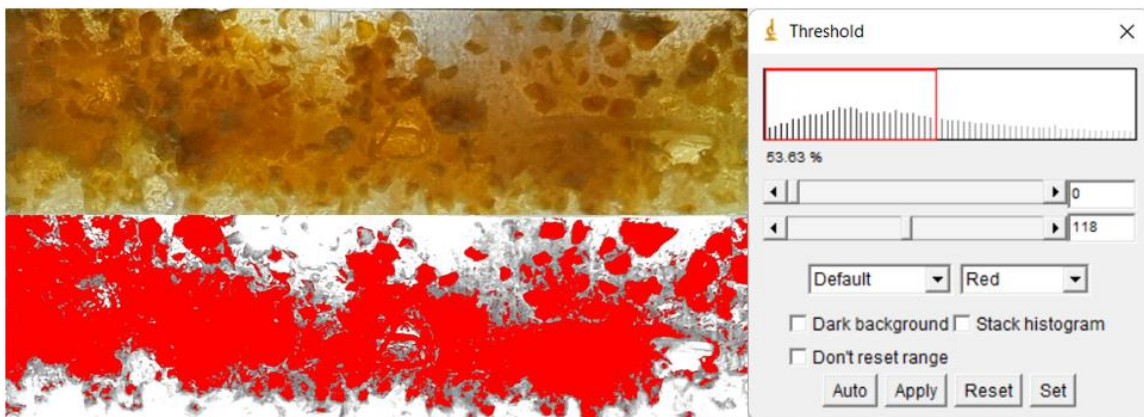


Figure 73: ImageJ analysis of specimen 24wt%-10

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