Mechanical and Physical Characterization of Chemically Treated Corncob Fiber Reinforced Polyester Bio-composite.

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A thesis submitted in fulfilment of the requirements for the degree of Bachelor of Applied Science (Materials Technology) with Honours

FACULTY OF BIOENGINEERING AND TECHNOLOGY
UMK

2024

DECLARATION

I declare that this thesis entitled “Mechanical and Physical Characterization of Chemically Treated Corn Cob Fiber / Polyester Bio-composites” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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ACKNOWLEDGEMENT

I would like to express my gratitude to my final year thesis supervisor, Ts. Dr. Mohamad Bashree Bin Abu Bakar, who gave constructive and valuable comments, helpful advice and encouragement, help me to coordinate my thesis especially in writing this report and involved continually and convincingly pushing me through proofreading. Without his guidance and persistent help, this thesis would not have been possible.

I want to thank the many people at Universiti Malaysia Kelantan Jeli Campus for their tremendous support. Furthermore, I would also like to acknowledge with much appreciation the crucial role of laboratory's staffs. For their provision of the experimental facilities and technical assistances. My thanks go out to the panels especially in my thesis presentation that has improved my presentation skills thank to their comment and advices.

Mechanical and Physical Characterization of Chemically Treated Corn Cob Fiber Reinforced Polyester Bio-composites

ABSTRACT

The research for sustainable materials has lead top the exploration of bio-composites, such as corn cob fiber (CCCF) reinforced unsaturated polyester resin (UPR), for various application. Corn cob fiber (CCF) reinforced unsaturated polyester resin (UPR) bio-composites have been prepared using hand lay-up and compression moulding technique. To improve fiber matrix adhesion, the CCF was chemically treated with 2%, 4%, 6% and 8% alkaline solution that is sodium hydroxide (NaOH) at the same soaking time 3 hours to enhance the interfacial bonding between the fiber and matrix.. Fourier transform infrared (FTIR) was used to investigate the changes of fibers chemical constituencies and thermal behaviour after alkaline treatment. Mechanical properties such as tensile strength, tensile modulus and elongation at break of untreated and NaOH treated CCF/UPR bio-composites were also studied and compared. The incorporation of the alkaline CCF resulted in composites better tensile properties and 4% treated showed the best result. Thermal stability of the CCF/UPR bio-composites were significantly enhanced after alkaline treatment compared to those of untreated CCF/UPR bio-composites. Thus the chemical treatment on CCF improved fiber-matrix adhesion, which also contributed to the improvement of mechanical properties compared to those of untreated CCF/UPR bio-composites.

Pencirian Mekanikal dan Fizikal Serat Tongkol Jagung Dirawat Secara Kimia Diperkuatkan Poliester Bio-komposit

ABSTRAK

Penyelidikan untuk bahan lestari telah mendahului penerokaan biokomposit seperti resin polyester tak tepu bertetulang (ccf) bertetulang resin polyester (UPR) untuk pelbagai aplikasi. Bio-komposit gentian tongkol jagung (CCF) bertetulang resin poliester tak tepu (UPR) telah disediakan menggunakan teknik letak tangan dan pengacuan mampatan. Untuk meningkatkan lekatan matriks gentian, CCF telah dirawat secara kimia dengan 2%, 4%, 6% dan 8% larutan alkali (NaOH) pada masa rendaman yang sama selama 3 jam. Fourier Inframerah transformasi (FTIR) digunakan untuk menyiasat perubahan konstituen kimia gentian dan kelakuan terma selepas rawatan beralkali. Ciri-ciri mekanikal seperti kekuatan tegangan, modulus tegangan dan pemanjangan takat putus tidak dirawat dan biokomposit CCF/UPR yang dirawat NaOH turut dikaji dan dibandingkan. Penggabungan CCF beralkali menghasilkan sifat tegangan yang lebih baik komposit dan 4% yang dirawat menunjukkan hasil yang terbaik. Kestabilan terma biokomposit CCF/UPR telah dipertingkatkan dengan ketara selepas rawatan beralkali berbanding biokomposit CCF/UPR yang tidak dirawat. Oleh itu, rawatan kimia pada CCF meningkatkan lekatan matriks gentian, yang juga menyumbang kepada peningkatan sifat mekanikal berbanding dengan komposit bio CCF/UPR yang tidak dirawat.

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LIST OF ABBREVIATIONS

CCF	Corn Cob Fiber
NaOH	Sodium Hydroxide
UN	Untreated
T	Treated
UPR	Unsaturated Polyester Resin
FTIR	Fourier Transform Infrared Spectroscopy
PMCs	Polymer Matrix Composites
MMCs	Metal Matrix Composites
MPa	Megapascal
GPa	Gigapascal
FTIR	Fourier Transform Infrared Spectroscopy

LIST OF SYMBOLS

%	Percent
µm	Micrometre
MPa	Megapascal
°C	Degree Celsius
wt.%	Weight percentage
h	Hour
mm	Millimetre
g	Gram

CHAPTER 1

INTRODUCTION

1.0 Background of Study

A bio-composite is a material composed of two or more distinct substances, one of which is naturally derived, combined to yield a new material with improved performance over the individual constituent materials (Alfred Rudin & Phillip Choi, 2013). Natural composites combine plant-derived fibers with a polymeric matrix, creating bio-composites intended solely for bioengineering applications. It is applicable to any two phase composite made from continuous fiber, short fiber or particles and matrix, as long as the void content in the composite can be neglected. (Zheng-Ming Huang , 2022). In bio-composite material that consist of a matrix that is synthetic or natural polymer resin and reinforcement is natural fiber that provide biocompatibility properties (Joshi, Drzal, Mohanty & Arora, 2004). Bio-composite have several advantages compare to conventional composite such as lightweight and high strength because natural fiber used as reinforcement such as kenaf and corn cob, possess excellent specific mechanical properties. Then it improved mechanical properties because bio composite can enhance mechanical properties that include tensile strength and impact resistance. Bio-composite can use in many application across various industries among them is automotive industry because bio-composite is lightweight nature of bio composite help reduce vehicle weight and reduced emissions such as for interior component and dashboard. Then bio composite are good used in construction and building material such as roofing material and wall panels because it offer advantages like durability and thermal insulation.

The reinforcement in the composite material refer to the component that give the strength and other desired properties to the composite. It typically combined with a matrix material to create a composite structure with enhanced overall performance. The reinforcement phase can take various form including fiber and particle depending on the specific application and requirement. Fiber are commonly used form of reinforcement in composite material. Corn cob fiber that treating with sodium hydroxide (NaOH) will increased their crystallinity because the alkaline treatment can lead to an increase in the crystallinity of cellulose fibers and could improve our mechanical properties such as increased strength and stiffness. Fiber reinforcement will improve the mechanical performance of composite, making them stronger and more durable compared to the matrix material alone. The type of fiber used as reinforcement in composite including glass fiber, natural fiber and carbon fiber. The reinforcement mechanism of fiber provide distributing and transferring load within composite structure. The high tensile strength fiber help to resist applied force and prevent crack propagation, enhancing overall strength of the composite. Fiber reinforcement also have disadvantages associated with it used such as cost particularly high performance fiber like carbon fiber can be expensive compare to matrix material used in composite. Then composite material with fiber reinforcement be challenging to recycle due to the difficulties in separating and processing fiber from matrix material.

Corn cob has several potential use due to it unique properties and characteristic. Corn cob can utilized as composite material because can be incorporated as a reinforcement phase. It provides strength and other desirable properties to bio composite. Corn cob fiber (CCF) combined with plastic to create bio composite because provide a sustainable, lightweight and customized material option with improved mechanical properties and reduced environmental impact. Resin polymer are organic compound composed of long chain repeating molecular unit called monomers such as polyester and epoxy. In composite, resin polymer play a crucial role as the matrix or binder that hold the reinforcement material together. Thermoset resin is type of resin that occur the chemical reaction during curing, resulting in a permanent and irreversible solid state such as epoxy resin and polyester resin depends on specific application requirements. Unsaturated polyester resin (UPR) is the type of thermosetting polymer that is commonly used in various application but has limitation between thermosetting such as limited heat resistance compared to thermosetting resin like epoxy. The type of UPR is orthophtalic polyester resin.

Chemical treatments are often applied to natural fiber to improved their performance and compatibility with various application. Many type of chemical treatment that can use on natural fiber such as alkali treatment and bleaching. The selection of the chemical treatment for natural fiber depends on the desired properties and intended application. Chemical treatment that applied to natural fiber because can improve the physical and mechanical properties on natural fiber such as alkali treatment can increase fiber strength and surface roughness that will make suitable for reinforcement in composite material. overall that chemical treatment on the natural fiber are to improve their performance, functionality and compatibility with different application. The weakness of the natural fiber for hydrophilic that have tendency to absorb water and susceptibility to moisture. Moisture absorption will cause weaken the structure of natural fiber and their mechanical properties will may be negatively affected. Hygroscopic nature to natural fiber refer to their ability to absorb and retain the moisture from surrounding environment because it will cause expand or contract in response to change in humidity and moisture content. It can reduce this weakness by doing fiber treatments and coating. The weakness natural fiber their incompatibility with hydrophobic polymer matrices such as polyethylene that has low affinity for water and tend to repel or resist moisture. This incompatibility cause give the challenges when combined natural fiber into hydrophobic polymer matrices.

The chemical treatment that we will use is sodium hydroxide (NaOH) on natural fiber such as cotton and corn cob fiber. NaOH are used as a test reagent on the CCF to determined it reaction to alkaline condition. It will use the different concentration of the NaOH solution into the sample. The reaction of CCF to NaOH can vary depending on the factor such as the fiber composition and processing. The NaOH can use to assess the resistance of corn cob fiber to alkaline conditions. When the fiber to NaOH solution, their reaction in an alkaline environment can observed. NaOH also use in fiber dissolution because can be used to evaluate the solubility of the CCF. Do the immersing the fiber in concentrate NaOH solution, the extent to which the fiber dissolves or undergoes degradation can be assessed. NaOH is the strong alkaline and strong base with high pH. This characterization can allow the effective testing of CCF resistance to alkaline conditions. The strong alkaline of NaOH ensure that any reaction and changes can be clearly observed and assessed.

In this study, CCF reinforced UPR bio composite will be prepared by hand lay-up and the compression moulding technique. This can provide a reduction in the disposal of corn cob waste to the environment because there is no other function that can be used from the waste material. so it will be fabricate the CCF that reinforce with unsaturated polyester resin bio composite and will know about the effect of chemical treatment using NaOH on the mechanical and physical properties of the CCF.

1.2 Problem Statement

The use of synthetic fiber is widespread today, some example of synthetic fiber are nylon that use in textiles and engineering materials and polyethylene terephthalate (PET) that use in production of the bottles for beverages and food. Due to some issues, such are the use of the synthetic fiber has a harmful effect on the environment because mainly use from the petroleum source. The production of the synthetic fiber involves of use chemical such as solvent and finishing agent. These will make chemical have harmful effects on the environment. Most of the synthetic fiber are non-biodegradable that means cannot break down naturally overtime. Synthetic fiber also difficult to do recycles process due to their chemical composition and mixed fiber content. Instead of synthetic fiber, natural fiber owing several advantages corncob is abundantly available agricultural waste.

Then, the corn cob will be discarded by all and sundry who use it because not know their benefit and advantages of corn cob. So from being thrown out, we make the corn cob to do bio composite and produced the product from this corn cob waste. Corn cob also have the its own advantages to produce the good product of bio composite. The compatibility issue between the matrix UPR and the reinforcement CCF in composite material will cause the problem in interfacial adhesion. The natural fiber characteristic is hygroscopic that absorb water cause will weaken adhesion and delamination between polymer and natural fiber phase. So we will use NaOH chemical treatment to enhance the adhesion or chemical bonding networking through modification on functional group that had on the surface of natural fiber. As a result that interfacial adhesion between natural fiber and polymer matrix expected to be enhanced and gap or void that exist in interface or composite surface could be reduced.

1.3 Objective

1) To fabricated CCF reinforced UPR bio composite using hand lay-up and compression moulding technique.
2) To study the effect of NaOH chemical treatment on mechanical and physical properties of CCF reinforced UPR composite.

1.4 Scope of Study

This study is focusing on the preparation and fabrication of CCF reinforcement UPR bio composite using hand lay-up and compression moulding technique. The CCF will be prepare by drying and grinding of corn cob into powder or sawdust fiber. The CCF will be treat by NaOH chemical treatment at concentration of 0, 2, 4, 6, and 8 % for 6 hours then subsequently dry into oven for moisture removing. The CCF at different concentration of NaOH treatment will be fabricated with UPR thermosetting resin into bio composite sample using hand lay-up and compression moulding technique at 120°C. The UPR pure sample will also be prepared as a reference for comparison purpose. The compounding method with using polyester resin that weight percentage is constantly fixed while weight percentage of CCF powder is gradually change. The UPR resin pure sample will also be prepared as a reference for the comparison purpose. Besides, there are some characterization process will be done which are the mechanical and physical characterization. For mechanical characterization, the tensile test and flexural test will be conducted by Universal Testing Machine (UTM) while the physical characterization by using water absorption test.

1.5 Significance of Study

With the right weight percentage of the CCF powder reinforcement with UPR, the improvement of mechanical properties such as are elastic and tensile strength. Other than that, the use of the CCF as a reinforcing filler in polyester resin to enhance their mechanical properties. Fibrous nature of the CCF are suitable for improve their strength that can use in many application such as in automotive part in manufacturing of interior component and door panels. In addition, it will reduce the harmful problems to nature such as incineration emission and plastic landfill disposal because synthetic bio composite are hard to recycle and renewable such as from the petroleum, in other hand can reduce the dumping waste of corn cob . In addition, corn cob is natural bio composite and from renewable material that can help to solve environmental issues. The CCF with polyester resin can be used widely in various application such as in automotive manufacturing interior component for example door panels. The problem issue about of waste natural material can be reduce by utilising into bio composite material. the weakness issue between natural fiber and polymer matrix can be reduce by application of NaOH chemical treatment.

CHAPTER 2

LITERATURE REVIEW

2.1 Composite Material

2.1.1 Introduction

Composite is the material that form from two or more different own constituents with the mechanical properties or chemical properties that are different. This constituents known as reinforcement and the matrix that combined together for create material that have the enhanced of their properties or unique that not have in the individual components alone. A composite material is a material that has two or more different material that consist of structure of reinforcement and the matrix that is integrated into enhancing the properties of the composite (Bouhfid et al., 2018; İşmal & Paul, 2017). Composite have the good own properties to be widely used in industry among them are superior strength, stiffness and lightweight nature. Composite features and properties are quite different from those of conventional material due to the combination of two material and also may contain fillers or additives which could lead to a better utilization in various application (automobile and construction) and some improvements can be done as desired (Yıldızhan et al., 2018). It is used as an alternative material to conventional material because of its improved properties such as electrical, thermal, and mechanical properties (Bouhfid et al., 2018). The reinforcement component composite that consist the material with high strength and it form of fiber, particles or flakes. The matrix act as fasteners to hold the reinforcement together and transfer their pressure between the reinforcement elements. Matrix material will make the composite be durability, toughness and resistance to environmental factors. The final composite material will show its properties depend on some factors, for instance, the geometry of the reinforcement (distribution size, shape), matrix materials, and the creation method. Hence, it is understood that the selection of those factors is important in determining and producing high performance products (Arumugaprabu, Ko, Uthayakumar, & Deepak Joel Johnson, 2018).

2.1.2 Bio-composite

Bio-composite is the type of composite that combined natural fiber or particles that obtained from the renewable source as the reinforcement component. Bio-composite material is obtained with the utilize of natural fiber (reinforcement) or resin (matrix) rather than synthesized fiber or resin (Yıldızhan et al., 2018). Normally the natural fiber or particles are combined with biodegradable polymer matrix or resin bio-based to produce composite material that sustainable and environmentally. Bio-composites are the special type of composite materials formed by polymers matrix and a reinforcement of different natural fiber (Trivedi, Alok Kumar, 2023). By using bio-composite material will give more benefit including reduce environmental impact, low carbon footprint and potential for end-of-life recyclability or biodegradability. natural fiber are relatively long and have a high percentage of cellulose, which offers high tensile strength and crystallinity (Trivedi, Alok Kumar, 2023). Moreover, bio-composite of fiber reinforced polymer matrix got an impressive consideration in various automotive application which are in the basic segment, construction, and many other application (Sassoni et al., 2014; Shalwan & Yousif, 2013). This is because of the excellent properties provided by natural fiber better than synthetic fiber as the reinforcement in polymer matrix composite in terms of mechanical properties (flexural and tensile modulus), renewable, adaptable during handling and lighter in weight (Shalwan & Yousif, 2013). The challenges that occur is to ensuring the quality of fiber are consistent, optimize process technique and addressing potential limitations in mechanical properties and moisture absorption compared to traditional composite. Natural fibre composite are the composite material which at least reinforcing fibres are derived from renewable resources like plant (corncob and pineapple leaf). For the fiber, it could be get from natural source such as recover cellulose fiber and crops (flax and cotton). Bio-composite has advantages due to natural fiber contents which are the production are canter to low cost compared to the synthetic fiber it can be renewed which is environmentally safe and utilized in many application due to its good properties (Yıldızhan, Çalık, Özcanlı, & Serin, 2018). Natural fibre composite become as a filler for reduce the cost because many natural fibre waste not be used but just throw it away. Instead of it going to waste, I will create from that waste material corn cob fibre as a filler, so that it becomes a good and usable product. Bio-composite has good advantages due to natural fiber content which are the production are center to low cost compared to synthetics fiber, it can be renewed which is environmentally safe and utilized in many applications due to it good properties (Yıldızhan, Çalık, Özcanlı, & Serin, 2018). Natural fiber also have low-density properties that can lead to high specific stiffness and strength.

2.2 Reinforcement Material

2.2.1 Introduction

Reinforcement material is the material that used to be added to matrix materials for upgrading purpose of physical properties and other properties of the final composite materials. There are have synthetic fiber and natural fiber which are the type of reinforcement material that typically utilized by the researchers (Arumugaprabu et al., 2018). All these reinforcement has own advantages and disadvantages which synthetic fiber such as Kevlar and glass that provide great properties like great wear resistance, high in stiffness and strength but it also high cost, has poor biodegradability and recyclability which leading to the limitation in applications (Srinivas, Lakshumu Naidu, & Raju Bahubalendruni, 2017). The application by using synthetic fiber is in packaging material such as bag and films because it have good in strength , flexibility and moisture.

2.2.3 Natural Fiber

While for natural fiber such as kenaf and corn cob that has good properties which are has low weight, low cost, great mechanical properties and come from abundant sources and renewable resources (Shalwan & Yousif, 2013). But it cannot bear with high loading which causing a limitation in heavy load application (Srinivas et al., 2017). To compare, the natural fiber is viewed as an environmentally friendly reinforcement material with great properties (Mohammed, Ansari, Pua, Jawaid, & Islam, 2015). Based on previous study, the hybridization of epoxy and vinyl ester may be presumed as a decent method in improving the toughness of thermoset resin without decreasing the strength (Turcsan & Meszaros, 2017).

2.2.4 Corn Cob Fiber

The effectives by using corn cob fiber as reinforcement in the composite material that has been previously demonstrated by the author who tested their mechanical properties in flexion and tension (Luo et al., 2017; Panthapulakkal and Sain, 2007 ; Luo et al.,2014; Garadimani et al., 2015). Corncob are waste resource utilization today that currently research work focuses on utilizing post-consumer corncob waste by explore it potential as a filler or additive in composite. The agriculture of the corn products are produced widespread in this country. Corn utilization are currently diverse, ranging from food to biofuels. Utilization of the waste corn cob is limited when the content of hemicellulose and fiber in corn cob is quite high. Corn cob fiber is the natural fiber that extracted from the inner core of corn cob which is at the cylindrical central part of corn ears. In the corn cob that mainly contain cellulose, hemicellulose and lignin. Corn cob fiber can renewable and biodegradable that also consider a waste product of the agricultural industry. Corn cob fiber are best way use as reinforcement in composite materials because it has the potential to serve as a natural reinforcement in composite material. it can be combined into the matrices polymer to increase their own mechanical properties such as tensile strength and stiffness of the resulting composites. This application that use corn cob fiber is often used in green house and container gardening applications. By using the waste material from the corn can make cost effective because corncob fiber is often an economically viable option compared to the synthetic alternative.

2.3 Matrix Material

2.3.1 Introduction

Matrix material is the composite that refers to the continuous phase and binds the reinforcement. It will give the good structural and hold it the reinforcement together and form a cohesive composite structure. The type of matrix material is polymer, ceramic, metal or combination of this material. In composite material phase consist the reinforcing and matrix, for the matrix phase is the polymer utilized and for reinforcing phase is fiber, particles utilize (Arun Kumar Sharma. 2020). In matrix material that has divide into two categories that is thermoplastic and thermoset. Matrix material will give cohesion and structural integrity for the composite.

2.3.2 Thermoset Polymer

The polymer of thermoset are cannot to be melted or reshaped. This physical structure will protect them from being soft when heat. Thermoset polymer will give high heat resistances to heat and make excellent in thermal stability. This structure prevents the material of thermoset material from soft allow to withstand and not loss of properties and deformation. This thermoset polymer have polymer matrix that consist epoxy resin, polyester resin and others. Matrix polymer have cohesion, transfer load between reinforcement to determine the overall of the mechanical and physical bio composite.

2.3.3 Unsaturated Polyester Resin (UPR)

In the context of CCF composite with polyester resin, the matrix material is unsaturated polyester resin. UPR is the thermosetting polymer. UPR is suitable for composite incorporating natural fiber like CCF because it compatibility with hydrophilic nature of these fiber. Thermosetting plastic do not soften but decompose upon heating. They cannot be reshaped once they are solidified (Sreekumar & Thomas, 2008). UPR represent an interesting category among other commercially available resins due to their outstanding mechanical properties, corrosion resistance and lightness (Bartoli, Rossi & Frediani , 2019). UPR also represent the largest market among other resin. Polyester resin are widely use in various industries and application due to versatility, ease of use and desirable properties. Polyester resin are economical resin system that used in engineering application, but it limited to use for the high performance composite. It can be produce for a large variety of properties from soft and ductile to hard and brittle (Balaguru et al., 2009). Methyl ethyl ketone peroxide (MEKP) is used as curing agent and cobalt naphthenate as a catalyst for making the product of natural composite. The polyester resin are the good in mechanical properties as well as corrosion properties and low weight. This are less costly compared to the epoxy resin. Polyester resin are principally used for the electrical, chemical, marine and automotive application (Kar, 2017; Mallick., 2007). Polyester resin are unsaturated and it present about 75% of the total resin used in the composite industry.

2.4 Chemical Treatment

2.4.1 Introduction

Chemical treatment is the process that use the chemical substances into the material or the surface for change their properties and improve their performance. The chemical treatment can use on various of material such as metal, polymer and composite. Chemical treatment can treat on the material waste to repair their characteristic that is reducing their porosity (Catarina Farinha, Maria Do Veiga, 2021). It also can improve their properties and make it good in various application.

2.4.2 Sodium Hydroxide

Chemical treatment can use to natural fiber are alkali treatment such as using sodium hydroxide (NaOH) are commonly used to natural fiber like CCF. This treatment is submerge the fiber in alkaline solution to remove impurities and other component. This chemical treatment can improve the strength of fiber and improve dye-uptake. The reaction of CCF to sodium NaOH can vary depending on the factor such as the fiber composition and processing. The NaOH can use to assess the resistance of corn cob fiber to alkaline conditions. When the fiber to sodium hydroxide solution, their reaction in an alkaline environment can observed. NaOH also use in fiber dissolution because can be used to evaluate the solubility of the CCF. Do the immersing the fiber in concentrate sodium hydroxide solution, the extent to which the fiber dissolves or undergoes degradation can be assessed.

2.4.3 Effect of Chemical Treatment on Mechanical and Physical of Polymer Thermoset Unsaturated Polyester Resin (UPR)

CCF are use as reinforcement for UPR that are prepare using NaOH for chemical treatment reaction. The mechanical and morphological properties of composite has been studied the effect of fiber surface treatment (Nurul Munirah Abdullah & Ishak Ahmad. 2012). Chemical treatment effect the strength, compatibility and adhesion of the natural fiber matrix. In this study, natural fiber are chemically modified (Saroj et al., 2022). The result show that treated fiber has been show the better physical properties than untreated, than untreated fiber has better tensile strength that the treated fiber and the fiber that treated to NaOH has the highest tensile strength (Prashantha Acharya, Dayananda Pai, GT. Mahesha , 2023). The effect on mechanical properties is the adhesion will increase between unsaturated polyester resin and other material such as reinforcement fiber. This treatment will increase the bonding strength and interfacial adhesion that give the good performance of mechanical in composite material. For the effect of chemical treatment in physical of polymer thermoset (UPR) shows in toughness and impact resistances such as add the toughening agent through chemical treatment can increase the ability of resin to absorb energy during impact and deformation and reduce of brittle fracture.

CHAPTER 3

MATERIALS AND METHODS

3.0 Materials

The materials that were are used in this study the corn cob fiber (CCF) used as a reinforcement of this research supplied from corn field at Tumpat, Kelantan. The polymer matrix use unsaturated polyester resin (UPR). The hardener material that used is methyl ethyl peroxide (MEKP) and Cobalt Naphthenate (CN) were are used together as respectively. All measurements were taken according to the size of the CCF for 3 characterization to be carried out. The mechanical properties such as strength and modulus of tensile and flexural specimens and thermal properties such as mass changes and char residues of untreated and treated composites should be pointed respectively. The chemical treatment used is sodium hydroxide (NaOH).

Sample preparation were divided into two different group that is untreated and treated CCF. Before the composite sample were fabricated using hot compress technique, chemical treatment was used followed different soaking times.

Table 3.1 shows the type of material and their function.

No.	Materials	Purpose
1.	Corn cob fiber	As a filler
2.	NaOH	Chemical Treatment
3.	Polyester resin	Polymer

Table 3.2 show the type of equipment and their function.

No.	Equipment	Purpose
1.	Grinding machine	To crush into fine particles
2.	Mold sheet plate	Mold
3.	Spatula	To mix solution of polyester resin + MEKP + corn cob fiber powder
4.	Scale	Weigh the weight of the material and sample.
5.	Micron size filter	To filter corncob fiber powder up to a size of 63 micron.
6.	Plastic cup	Stir the solution in a plastic cup.
7.	Electronic Digital Caliper	To measure length, width, and height.
8.	Cutter	Cut the sample into the parts we want.
9.	Universal Testing Machines (UTM)	For run tensile flexural test
10.	Hydraulic Molding Press (GT-7014-H)	To speed up the plastic hardening process.

3.1 Sample Preparation

Corn cob fiber were collected the nearest from our rental house as there were several food shops.

Figure 3.1: Corn Cob Fiber.

The corn cob has been wash to remove any potentially hazardous dust that may present in corn cob using distilled water. This will ensure that the corn cob are clean. Then, cut the corn cob using the sharp knife and cutter machine to the smallest size. It will put through the drying process that will last for 24 hours and will carry out in oven at the temperature of 120°C. This step are to remove any moisture that had in the corn cob during cleaning proses and existing. After that, the corn cob will be ground using the grinder until it reaches a small particle size and become the powder.

The CCF powder will do chemical treatment using the NAOH with different concentration that is 0, 2, 4, 6, and 8% for 6 hours. Then dry again the CCF power into the oven for 24 hours in the temperature 120°C because to remove this solution and moisture.

Table 3.3 shows the composites formulations.

Sample	Chemical treatment concentration NaOH (%)
S1	0
S2	2
S3	4
S4	6
S5	8

The UPR are serve as a matrix that filled with the CCF as natural fiber and the reinforcement. The CCF will use in different percent of the treated with alkaline soluition (NaOH) that is 2%, 4%, 6% and 8% and the same weight that use is 20grams.. The compound will be mix it. After the UPR and CCF is mixed thoroughly, add the 3% of MEKP and 3 drop of CN and mixed together. The compound composite will poured and spread into 150mm (length) x 150mm (width) x 3mm (thickness). This mixture will hot-pressed at the temperature of 110°C. heating pressure of 8 MPa and 8 minutes of heating. The sample will be cooled for 5 minutes. After 24 hours, the sample are blend and will do cutting process for the characterization.

3.2 Characterization of Composite

3.2.1 FTIR

Fourier Transform Infrared Spectroscopy (FTIR) are used for analysis of infrared radiation with the matter and can identify the chemical composition of the composite sample that present in sample. FTIR involved to high sensitive and provide accurate. Infrared spectra of each samples was obtained in the range of 4000-400 cm-1 using FTIR spectrophotometer from NICOLET iZ10. FTIR was employed to determine functional groups and its molecular bond presented in CCF by effect of chemical treatment NaOH. NICOLET iZ10 measured the infrared spectra of each samples at the room temperatures using KBr pellet.

For carried out the test, 20mg of CCF samples analysed in this studied was cut using the band saw machine for FTIR spectroscopy. A hydraulic pressure was applied onto mixture sample at two bar for 2 minutes by 3mm disc. The hydraulic pressure causes the removal of moisture inside spectroscopy. IR spectrum band were obtained when laser of infrared projected onto the mixture pallet. All the information obtained was characterized according to ASTM E168-06 and ASTM E1252-98 standards used by Bakri and Jayamani (2016).

3.2.2 Tensile Test

The tensile test is to determine the mechanical properties under tensile loading. This test are to measure that reaction to this applied force under tension. In this tensile test can measured of ultimate tensile strength (UTS) that maximum load that the material can accept from the material before it breaks. It will represent the maximum strength under tensile loading. The test was performed conforming to ASTM D 5083 procedure using universal testing machine, tested at cross head speed of 5mm/min and carried out in laboratory environment until tensile specimen failed with load of 50kN at the room temperature. Test specimen were prepared by cutting from CCF in rectangular form using band saw machine. Before the test, the seen surface of specimens were cleaned by mean of specimens were in rectangular form (150mm x 25mm x 3mm) depicted in Figure 3.4.

All the statistical significant result were obtained as the average value and the standard deviation of five test specimens of untreated and treated KFM-UPE composites after the test. Tensile strength, elongation at break and tensile modulus were recorded for all samples from stress-strain curves. The tensile strength elongation at break and tensile modulus were measured using the following formula.

$$\text{Tensile strength} = \frac{\text{maximum load force (F)}}{\text{Surface area (A)}}$$

$$\text{Tensile modulus} = \text{stress / strain}$$

$$\text{Elongation break} = \frac{l - l_o}{l_o} \times 100\%$$

l_o = Original length of test piece
l = Length of test piece at break

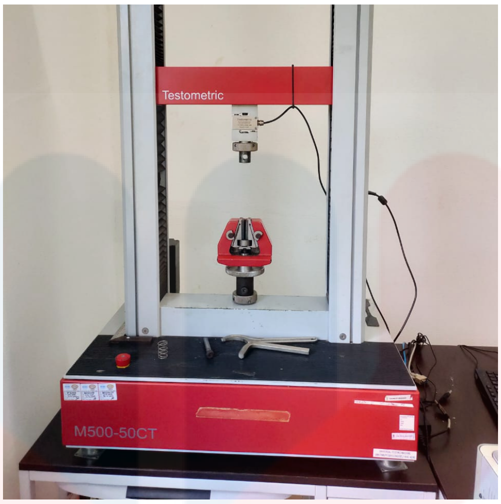

Figure 3.2: Universal Testing Machine (UTM)

3.3.3 Water Absorption Test

The water absorption test measures the amount of the water absorbed by the material sample over a period. The weight is measure before and after the specimen has been submerged. The difference in weight is used to calculate the percentage of water absorbed by the material, using the formula:

Water absorption = [(weight after – weight before)/weight before] x 100%

The thickness swelling test, conducted after the water absorption test, measures the percentage of swelling or expansion that occurs in the material composite upon contact with liquid. This swelling has implication for the physical, mechanical and chemical properties of the product. It is necessary to measure the initial thickness before conducting the water absorption test and then measure the final thickness after completing the test. The percentage of thickness swelling can be calculated using this formula:

Percentage of thickness swelling : (thickness after swelling - thickness before swelling)/(thickness before swelling)100%

CHAPTER 4

RESULT AND DISCUSSION

4.1 Fourier Transform Infrared Spectroscopy Analysis

4.1.1 FTIR Analysis of Corn Cob Fiber

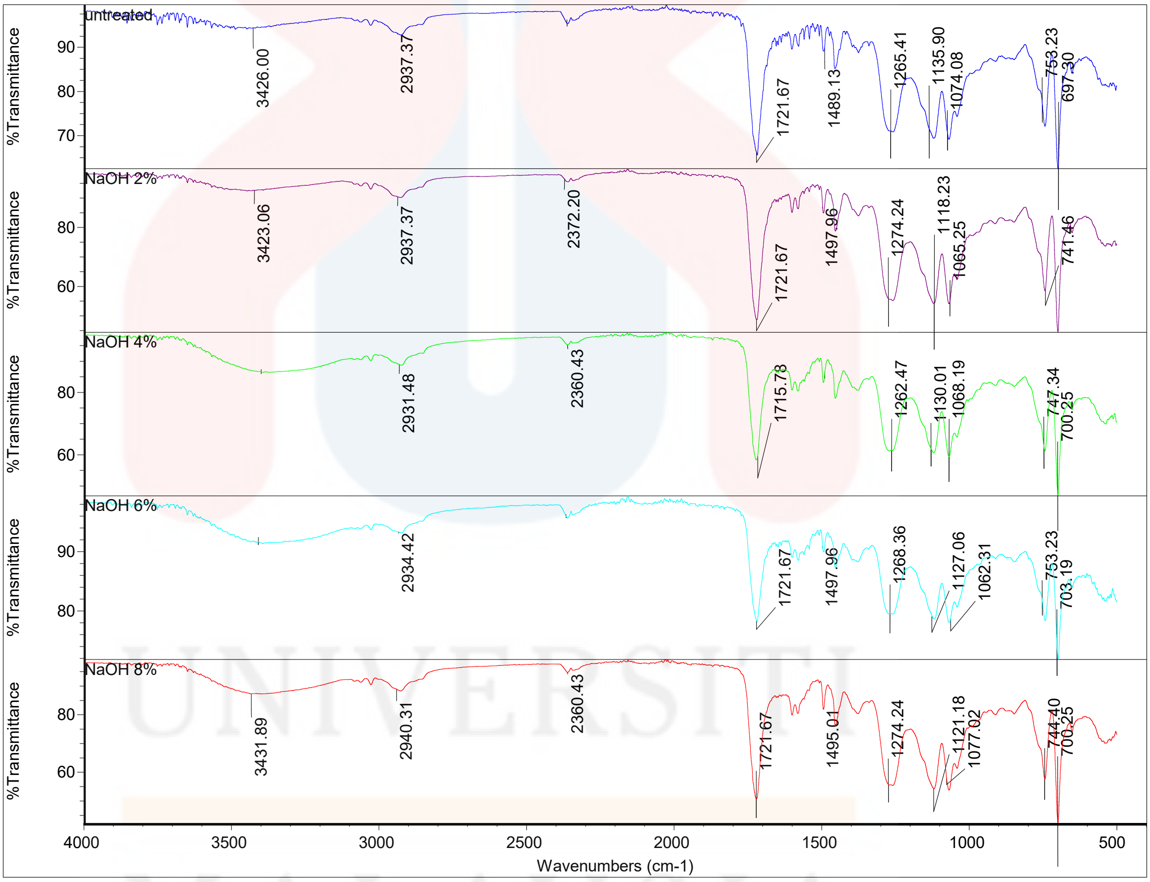

Figure 4.1 : FTIR spectra of untreated and NaOH-treated CCF at concentration of 2%, 4%, 6% and 8%.

Figure 4.1 illustrates the FTIR spectra comparison between untreated and chemically treated corn cob fiber (CCF) with NaOH concentration of 2%, 4%, 6% and 8%. A broad absorption band at 3431.89 cm^{-1} is due to O-H stretching vibrations and

H-bonded of alcohol and phenol as the functional group. The region 3500 – 2500 cm^{-1} for the untreated and treated CCF are related to OH and CH_2 groups. The peaks located at the range 2940 cm^{-1} for the all sample are attributed to CH and CH_2 groups, respectively (Khalil *et al.,* 2013). The band at range 2372 cm-1 correspond to H-C=O and C-H stretch vibration that contains a functional group of aldehydes. As for chemical treatment CCF, the 3431 cm^{-1} band assigned to the OH group decreased after this peak (Punyamurthy *et al.,* 2012). This disappearance is a consequence of the hemicellulose are removed from the fiber and the formation of ionic carboxylates in the incompletely extracted samples, in which instances the corresponding peak appears at lower frequencies (2940 cm-1).

The absorption band at 2360 cm^{-1} is due to aldehydes group (H-C=O and C-H) stretching and vibration of acetyl groups of hemicellulose, but this band can no longer be observed in chemical treated CCF. This peaks characterizes the hemicellulos, pectin and waxes present in the fibers before treatment. However, the intensity of the absorption band at around 2360 cm^{-1} which corresponding to H-C=O and C-H stretching is found to depend on chemical treatment. The H-C=0 and C-H stretching in chemical treatment CCF are significantly higher than that of untreated CCF. This is due to process between the hydroxyl (OH) group of CCF and the sodium group with sodium hydroxide. In chemical treated CCF curve the intensity H-C=0 and C-H stretching were less intense due to the removal of non-cellulosic impurities such as pectin and hemicellulose from the surface by absence of carbohydrates (Dwivedi & Mehta, 2011).

The large peak observed at the range 1721 cm^{-1} for the untreated and treated CCF can be associated to the presence of lignin, assigned to a, β-unsaturated esters vibration of C=O stretching in lignin and are confirmed by the peak at around 700 cm^{-1}. The spectrum of untreated CCF is distinguishable from that of treated CCF, by observed decreased at 1497 cm^{-1} and 741 cm^{-1} are due to the plane CH_2 symmetric bending of the assigned to the symmetric ring vibration contribution. It observed that the peak at 1274 cm^{-1} disappears in chemical treatment CCF. Chemical treatments removes the waxy epidermal tissue, adhesive pectin and hemicellulose that bind fiber bundles to each other. It can thus be summarized that the NaOH chemical treatment remove most of the lignin and hemicellulose component which help to improves the mechanical properties of CCF-UPE composites that the alkaline treatment.

The peak located at 1489.13 cm^{-1} appeared to decrease as soaking time increased. The intensity of transmission peak at 1489.13 cm^{-1} assigned to the C-O stretching vibration of the acetyl group in lignin component, separated into two smaller peaks at 1500 cm^{-1} and 1460 cm^{-1}. The first one corresponds to vibrations in the guaiacol structure of lignin and the second to the scraggly structure (Fengel & Wegener, 1984). The change of intensities at this peak after treatment indicates the removal lignin after alkaline treatment. The peak at 697.30 cm^{-1} reflects the carbohydrate backbone of cellulose and the C-C ring breathing. The absorption band at 1489.13 cm^{-1} is owing to CH2 bending in lignin whereas the broad peak at 697.30 cm^{-1} is due to C-O stretching bond structure from the functional group alcohol (cellulose, hemicellulose and lignin). As for untreated kenaf fibers, the small peak observed at last cm^{-1} as compared with treated fibers. The result obtained such that disappearing of smell from the kenaf fibers similar to Garside and Wyeth (2013).

4.2 Mechanical Properties of KFM-UPE Composites

4.2.1 Tensile Strength

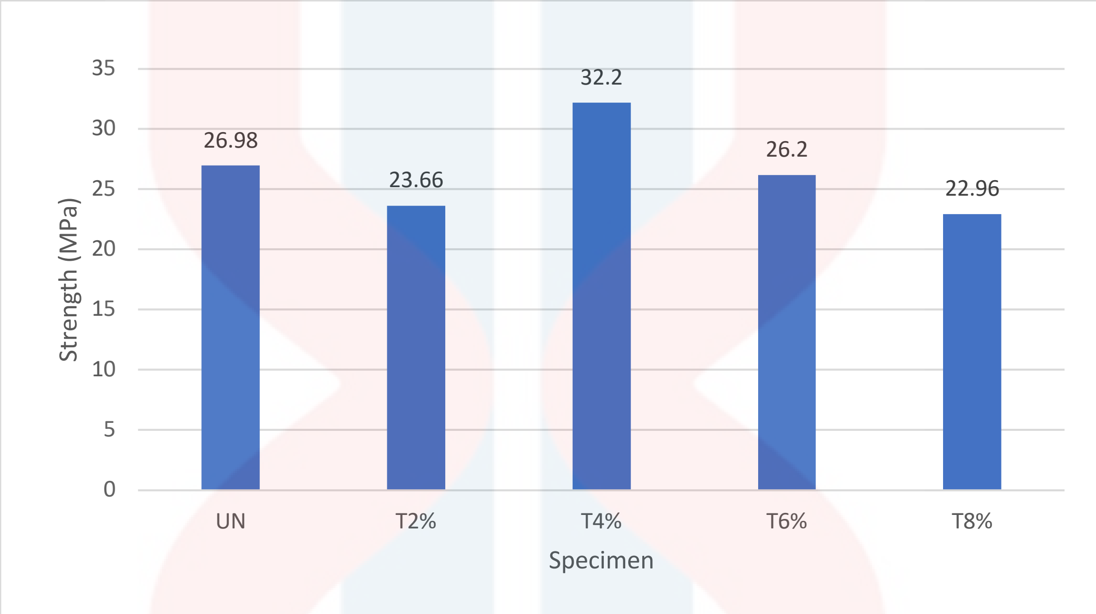

Figure 4.2 Tensile strength for untreated and NaOH-treated CCF/UPR composites.

Figure 4.2 displays the average tensile strength measured for five specimens encompassing both untreated and alkaline-treated CCF/UPR composites. The tensile strength was highest for composites treated with a 4% NaOH solution (32.30 MPa) and decreased for other treatments. Then the second highest value of the strength is that controlled sample that is untreated CCF/UPR composite (26.98 MPa). Subsequently, composites treated with 6% and 2% aNaOH solutions showed further decreased tensile strength of 26.20 MPa and 23.66 MPa, respectively. The lowest tensile strength was observed in composites treated with 8% NaOH solution at 22.96 MPa.

The tensile strength of composites depicted in Figure 4.2 revealed that there has been marked rise by the increasing of immersion time at the same fiber content which was consistent with the finding of Edeerozey *et al.* (2007). Tensile strength of treated 4% NaOH solution CCF/UPR composite demonstrated the most outstanding result compared to others sample formulation. It was found that the increasing in tensile strength T4% was 19.35% higher than UN respectively. The tensile strength of the T4% treatment was 36.09%, 22.9% and 40.24% higher than that of the T2%, T6%, and T8% treatments respectively. This study indicated that the interface between fiber and matrix was related to the load share from the matrix to the fiber, which contributed to the tensile strength. For good interfacial adhesion, the value of tensile strength of untreated UPR was high (Rokbi *et al.* 2011). The present finding also suggested that when MEKP was added to enhance the fiber-matrix adhesion, the loading force can transfer from low strength matrix to high strength fiber. The increase in tensile properties for 4% NaOH-treated fiber composite was due to greater fiber-matrix interaction and physical bonding, because physical bonding also increased after alkali treatment due to the dipolar interaction between fiber and matrix (Rout *et al.* 2001). Salmah *et al.* (2013) agreed that the increase of tensile strength due to removal of cementing materials and increased surface roughness.

The ultimate tensile strength is a maximum stress value obtained on a stress strain curve by dividing the maximum load on a material by the initial cross section of the test specimen. When reaching the Ultimate Tensile Strength (UTS), the specimen tends to resulting change in the length beyond while the cross-section of the specimens becomes thinner. Yield strength is the maximum stress that can be applied without permanent deformation of the composites in this study while elasticity concerns the below the point relationship between stress and strain for non-permanent deformation.

4.2.2 Tensile Modulus

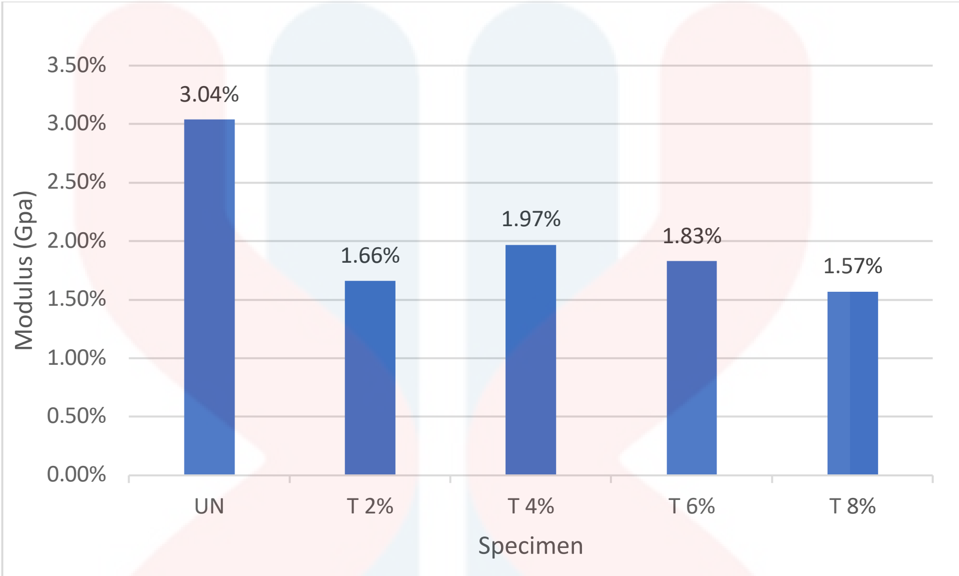

Figure 4.3 Tensile modulus for untreated and NaOH-treated CCF/UPR composites.

As illustrated in Figure 4.3 , the measured average tensile modulus of three specimens involved untreated and treated alkaline treated CCF-UPR composites. The tensile modulus varied with treatment condition, it was highest for composites treated with 6% NaOH for 3 hours (1.37 GPa) and lowest for those treated with 8% NaOH (0.88 GPa). Untreated composites showed a modulus of 1.17 GPa highlighting the impact of NaOH concentration on the mechanical properties.

Figure 4.3 presented the tensile modulus of CCF-UPR composites with untreated and treated 3 hours of the alkaline solution with different concentration 2% , 4%, 6% and 8% NaOH. It can be observed that the tensile modulus decreases with an increase in immersion time, holding fiber content constant. Tensile modulus of T4% was 17.8%, 23.5% and 35.3% higher than T2%, T6% and T8% respectively. The tensile modulus of UN demonstrated a modulus increase of almost 14.6%, 16.1%, 2% and 35.8% than that T2%, T4%, T6% and T8% respectively.

In consideration of the effect of alkaline treatment on the tensile modulus of CCF-UPR composites with 3 hours, the result obtained the tensile modulus of composites in 3 hours of treatment is 14.6% higher than that of untreated CCF-UPR composites. Due to better stiffness, values of CCF-UPR composites are much higher than untreated alkaline solution. For CCF-UPR composites, the findings suggested that the tensile modulus must not be higher or equivalent to untreated CCF-UPR composites. In contrast, this higher value of tensile modulus was influenced by the fibers that support the stress from the matrix. The presence of CCF in UPR effectively enhanced the tensile modulus of UPR which was double greater than that of untreated CCF-UPR composites. According to Liu *et al.* (2009), the tensile modulus of a NFPC depended on the modulus of the fiber and the matrix. Consistent with findings by Edeerozey *et al.* (2007), the present finding found that the treated 6% alkaline solution (NaOH) with 3 hours immersion time was the best treatment condition in terms of tensile properties.

4.2.3 Tensile Elongation at Break

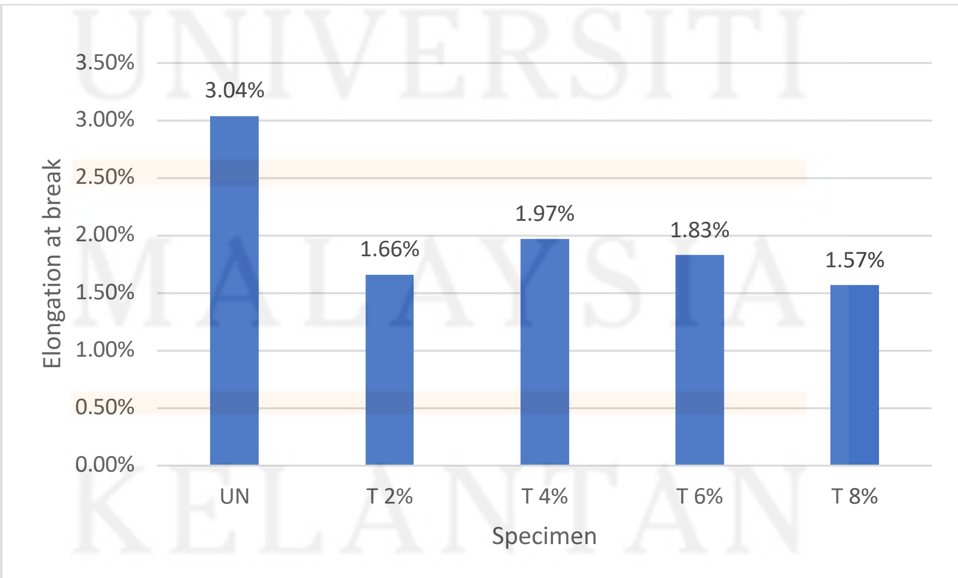

Figure 4.5 Tensile elongation at break for untreated and treated CCF-UPR composites.

Figure 4.5 shows the measured average tensile elongation at break of four specimens involved untreated alkaline treatment CCF-UPR composites. The tensile elongation at break decreased from untreated composites with highest (3.04%) elongation at break , treated 4% NaOH composites (1.97%), T6% NaOH composite (1.83%), T2% NaOH composite (1.66%) and while treated composite show in treated 8% of composites the least (1.51%).

The result revealed that the increasing of immersion time at the same fiber content decreases the elongation break of the composite because CCF and UPR are rather stiff and brittle. Thus the composites had lower elasticity than neat UPR can be expected. The breakage of the composite material started from the interface between two phases. When the immersion concentration increased, the breakage of the composites material increased. The percent elongation at break of the composites ended over than that of the matrix. This affected composites because of low fracture strain and the poor adhesion between the matrix and the fibers (Chumsamrong *et al.,* 2005).

Elongation at break of CCF happened higher than of UPR can explain the fracture of UPR before tensile fracture of CCF. Salmah *et al.* (2013) agreed well with the results obtained from the elongation at break of those treated composited if fewer than untreated composites exhibited high modulus of elasticity through the enhanced fiber wettability by the surface treatment of CCF using 4% NaOH solution. Furthermore, the decrease in essential elongation at break was due to loading of CCF can eliminate structural integrity of UPR. Treated composites of high concentration NaOH solution produced fast brakeage can mean improved fiber-matrix adhesion compared to untreated composites. Another reason for the result obtained was the progressive decrease on the elongation at break can be attributed to a reduction in deformability of the rigid interface between fibers and matrix.

4.3 Physical Testing

4.3.1 Water Absorption Test

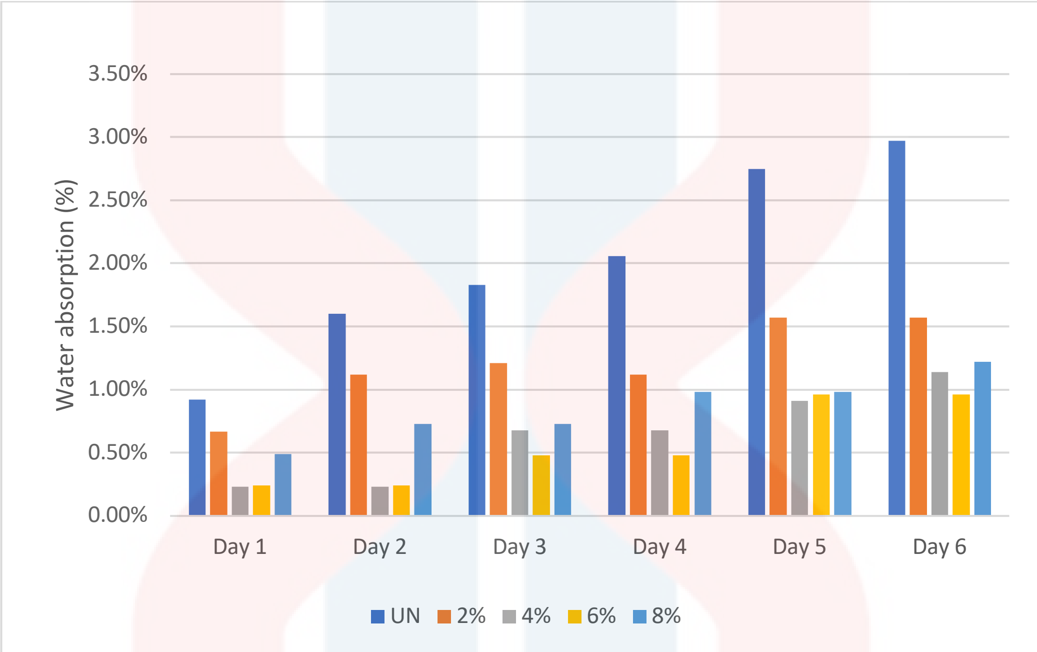

Figure 4.6 Water absorption test for untreated and NaOH-treated CCF-UPR composites

Figure 4.6 present the data from a water absorption test conducted on the untreated CCF-UPR composites and treated CCF-UPR composites with 2%, 4%, 6% and 8% of the NaOH solution over a period of 6 days. The researcher has segmented the data into 6 intervals days.

The untreated CCF-UPR composite sample exhibit the highest water absorption percentage over the entire 6 days period. On the second day, it absorb 0.92% water, followed by 1.60% on third day, 1.83% on fourth day, 2.06% on fifth day , 2.75% on sixth day and 2.97% on seventh day. The cumulative water absorption from day one to the last day is 12.13%. the untreated CCF-UPR composite sample exhibit the highest water absorption rate among the sample. The significant increase in water absorption indicates that the high concentration of CCF powder filler leads to a more porous structure, facilitating greater water infiltration. Although a higher filler content can improve certain properties, it also introduces a trade-off with increased water susceptibility.

Farhana et al., (2015) CCF powder generally has a porous structure which mean it can absorb some water. The level of water absorption depends on factor such as particle size, porosity and surface characteristic of CCF powder. The porous nature of CCF is a result of the arrangement of the fiber and the organic matrix. This porosity allow water molecules to penetrate and be absorbed to varying degrees.

The second highest water absorption rate is observed in the treated of 2% NaOH on CCF-UPR composites sample with the total absorption of 7.26% over 6 days. On the second day, it absorb 0.67% followed by 1.12% on third day, 1.21% on fourth day, 1.12% on fifth day, 1.57% on sixth day and last day 1.57% respectively.

In the additionally, the CCF-UPR composites treated with 8% of NaOH sample records the water absorption rate of 5.1% over the 6 days period. On the second day, it absorb 0.49% followed by 0.73% and 0.73% on fourth, 0.98% on the fifth and sixth and the last day is 1.22% at day 6. Water absorption rate increased with CCF-UPR composite treated with 8% NaOH compared to treated with 6% NaOH . According to Farhana *et al.,* (2015) show that moderate concentration of CCF powder filler content increase, so does the potential for water absorption.

The treated CCF-UPR composites with 6% NaOH sample exhibits the lowest water absorption rate among the sample with CCF powder filler, total 3% over 6 day respectively. On the second and third day it absorb 0.24% followed by 0.48% on the fourth and fifth day and the last that absorb were 0.96% on sixth and last day for water absorption test. This indicates that a lower concentration of CCF powder filler result in composite material with reduced susceptibility to water absorption. The limited presence of filler can contribute to a denser structure, preventing water ingress (Hiremath et al,. 2018). Next as a benchmark, the CCF-UPR composite with treated 4% NaOH sample show a 3.79% accumulated water absorption rate over 6 days with incremental absorption of 0.23% on second and third day followed with 0.68% for fourth and fifth day, 0.91% on sixth day and for the last day was 1.14% respectively.

In summary, among the sample containing CCF powder filler, the CCF-UPR composite with treated 6% NaOH demonstrates the lowest water absorption rate 3% followed to treated with 4%, 8%, 2% and untreated CCF-UPR composites.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This study aimed to fabricates CCF reinforced UPR composites using hand lay-up and compression moulding techniques. The present study was designed to determine the effect of NaOH chemical treatment on mechanical and physical properties of CCF reinforced UPR composites. The following conclusion can be drawn from the present study.

FTIR study has identify the optimum alkaline immersion time for 2%, 4%, 6% and 8% NaOH treatment to detect change of functional group in alkaline-treated on CCF. This experiment confirmed that the hemicellulose, pectin and waxes present in the fibers before treatment at 2372.20 cm-1, but this band can no longer be observed in alkali-treated corn cob fiber. The current data highlight the importance of treated CCF/UPR bio-composites have the highest tensile strength (32.2 MPa), and tensile modulus (1.37 GPa).

This study was limited by the absence of DSC experiment to study the melting temperature about the processing temperature of UPR resin when CCF/UPR bio-composites are fabricated. It is unfortunate that the study did not include new chemical treatment such as DIH-HEA and surface acetylation. Notwithstanding the relatively limited sample, this work offers valuable insight into FESEM investigation that has been confirmed by FTIR study and mechanical test.

5.2 Recommendations

These findings provide the following insight for future research:

i) The introduction of improved compounding technology and new chemical fiber pre-treatments such as DIH-HEA and surface acetylation can overcome hydrophilic character of natural fiber.

ii) Utilize computer software, such as SolidWorks to predict the properties of natural fiber plastic composites.

iii) Controlling processing parameters such, as temperature, pressure and time is required to manufacture high-quality composites. The stainless steel mold must be kept moisture-free and natural fiber must be allowed hours to dry. Repeat measurements of test specimens three to seven times to ensures accuracy and minimize the standard error, thereby making data trends more discernible.

iv) Average reading of test specimens must repeat three to seven times. Accurate reading eliminates standard error of measurement so that data trends can be nearly visible.

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APPENDIX

Corn cob fiber

Compression moulding

Tensile machine

FTIR machine