JIMMA UNIVERSITY
JIMMA INSTITUTE OF TECHNOLOGY
SCHOOL OF CHEMICAL ENGINEERING

SYNTHESIS AND CHARACTERIZATION OF NANOCELLULOSE FROM BAMBOO FOR NANOCOMPOSITE APPLICATION

BY

KAFI MOHAMED

A Thesis Submitted to Jimma University, Jimma Institute of Technology, School of Chemical Engineering in Partial Fulfillment of the Requirement for the Degree of Masters of Science in Chemical Engineering (Process Engineering).

November 2021

Jimma, Ethiopia

JIMMA UNIVERSITY

JIMMA INSTITUTE OF TECHNOLOGY

SCHOOL OF CHEMICAL ENGINEERING

Synthesis and Characterization of Nano-cellulose from Bamboo for Nanocomposite Application

By

Kafi Mohamed

Advisor: Ermias Girma (Assistant Professor)

Co-advisor: Temesgen Abeto (MSc)

A Thesis Submitted to Jimma University, Jimma Institute of Technology, School of Chemical Engineering in Partial Fulfillment of the Requirement for the Degree of Masters of Science in Chemical Engineering (Process Engineering).

November 2021

Jimma, Ethiopia

APPROVAL PAGE

This is to certify that the thesis prepared by **Kafi Mohamed Hamed** entitled ***"Synthesis and Characterization of Nanocellulose from Bamboo for Nanocomposite Application"*** and submitted as partial fulfillment for the award of the Degree of Master of Science in Chemical Engineering (Process engineering) complies with the regulations of the university and meets the accepted standards concerning the originality, content, and quality.

Approved and signed by examining board

Abdisa Jabesa (PhD) ______ ______ 30/12/2021
External examiner Signature Date

Edo Begna (PhD) ______ ______ 04/01/2022
Internal Examiner Signature Date

Ketema Beyecha (MSc.) ______ ______
Chairperson Signature Date

Ermias Girma (Asst. Prof) ______ ______
School Dean Signature Date

DECLARATION

I, hereby declare that the thesis entitled *"**Synthesis and Characterization of Nanocellulose from Bamboo for Nanocomposite Application**"* is my original work, with the guidance of my advisors, and it has not been presented for a degree in Jimma University or any other University and all sources of materials that used for the research have been duly acknowledged.

Submitted by:

Kafi Mohamed Hamed

Signature Date

Approved by:

Ermias Girma (Assistant Professor)

Main-advisor Signature Date

Temesgen Abeto (MSc.)

Co-Advisor Signature Date

Ketema Beyecha (MSc.)

Chair, Process Engineering Signature Date

ACKNOWLEDGMENT

My first thanks go to Allah the most gracious and merciful who has given me the courage and strength to proceed with my thesis.

I would like to express my deep and sincere gratitude, to my advisor Mr. Ermias Girma and Co-advisor Mr. Temesgen Abeto for their critical comments, advice, guidance, and above all, I am highly thanks for your timely, detailed, and untiring corrections that have been valuable to me during this research studies. I am much indebted and heartfelt thanks to Ms. Ebise Getacho, Mr.Teshale Tadesse, and Mr. Mohammed Kedir for their great support, helpful suggestion, and ideas for my research studies success.

I would like to encompass my appreciation to all administrative and laboratory technicians from Jimma Institute of Technology, School of Chemical Engineering Laboratory technicians especially Mr.Dafar Getahun and Mr.Worku Soresa, School of Materials Science and Engineering especially Mr. Sultan Mulisa, Mr. Endarias Adane, and Mr. Tsegaye Markos as well as School of Civil and Environmental Engineering especially Mr. Mohamednur Alemu and Mr. Firomsa Bidira for their good cooperation and tirelessly working with me during laboratory work and characterization.

Finally, I would like to thank my wife Ms. Medina Ayalew, and my family for their amazing moral and economical support, advice, and encouragement in all my effective success.

ABSTRACT

Recently, nanocomposite has played an important role by giving numerous applications that may solve real-world problems and help society. The development of environmentally sustainable materials composed of lignocelluloses biomass, which comprises cellulose, hemicelluloses, and lignin has attracted a lot of interest because of petroleum-based resources and their accompanying environmental issues. Nonrenewable polymeric materials, such as plastics, are not biodegradable when discarded, posing an environmental hazard. As a result, there is a demand for ecologically friendly and resource-efficient goods such as bio-composites. The purpose of this work was to assess the potential usefulness of bamboo for the manufacture of nano-biocomposites using polyvinyl alcohol as a reinforcing ingredient. The nanocellulose particles were synthesized from the high-land bamboo plant species (Arundinaria Alpina) using acid hydrolysis. The chemical composition, particle size, and crystallinity of the particles were studied using Fourier transform infrared spectroscopy, dynamic light scattering, and X ray diffraction. The influence of reaction time, temperature, and acid concentration on nanocellulose yield was optimized by using Respon se Surface Methodology with corresponding results of 60 minute, 40℃, and 61.404wt% respectiv ely and the nanocellulose yield obtained at optimum parameter was 43.155%. The functional gro up structure of the nanocellulose could be confirmed by analyses of Fourier transform infrared spectroscopy. The average particle size was 106.5 nm with a poly-dispersity index of 0.0632. The crystallinity index of bamboo, nanocellulose and average crystallite size was 44.60%, 74.0% and, 35.20nm, respectively. Following that, the Nanocellulose/Polyvinyl alcohol composite was prepared using the solvent casting method, and the thermal stability of the composite was analyz ed using Thermogravimetry analyzer of PVA reinforced with 6wt% NC shows a high weight loss of 80.56 % of its total weight occurred at a temperature range between 290.782 °C to 380.207°C as well as water absorption capacity and mechanical properties such as tensile strength and elongation at break were studied. Different ratios of nanocellulose loading were used to see the effects of nanocellulose loading on the properties of polyvinyl alcohol. Therefore, according to the findings, bamboo is the alternative lignocellulosic material for generating nanocellulose for nanocomposite applications.

Keywords: Bamboo, cellulose, Nanocellulose, Nanocomposite, Synthesis, Characterization

TABLES OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

ACRONOMY

AGU	An-hydro-glyco-pyranose units
ANOVA	Analysis of Variance
ASTM	American Standard of Testing Material
BC	Bacterial Cellulose
CCD	Central Composite Design
CMC	Commercial Micro-Cellulose Crystalline
CNC	Cellulose Nano Crystals
CI	Crystallinity Index
DLS	Dynamic Light Scattering
DOE	Design of Experiment
DP	Degree of polymerization
EB	Elongation at break
FTIR	Fourier Transform Infrared Spectroscopy
MFC	Micro-fibrillated cellulose
MCC	Micro crystalline cellulose
NC	Nano-cellulose
PCL	Polycaprolactone
PEG	Polyethylene glycol
PLA	Poly Lactic Acid
PVA	Polyvinyl Alcohol
RSM	Response Surface Methodology
SEM	Scanning Electron microscopy
TAPPI	Technical Association of the Pulp and Paper Industry
TS	Tensile Strength
TGA	Thermogravimetry analysis
WAC	Water Absorption Capacity
XRD	X-ray Diffraction
Y	Yield

1. INTRODUCTION

1.1. Background

Nanotechnology is a discipline of science and technology concerned with the understanding and management of matter or an object with dimensions ranging from 1-100 nanometers (Thamer et al.,2018). It encompasses interdisciplinary subjects such as science, health, engineering, technology, entails photographing, describing, modeling, and altering features of materials at this length scale (Fall, 2013). It has applications in medicine, electronics, transportation, energy, and the environment. Rather than working at ever lower dimensions, studying at the nanoscale allows scientists to take advantage of the unique physical, chemical, mechanical, and optical properties of materials that occur naturally at that scale. Nanotechnology, as a result, is a multidisciplinary field that encompasses nanochemistry, nanophysics,nanomaterials, nanoelectronics, and nanobionics (L.Tan, 2009).

Lignocellulosic materials are the most prevalent biomass found in almost all plant-derived materials, from wood and grass to agricultural residues and municipal solid wastes, and can be used to replace petroleum-based resources and provide sustainability (Kalia et al., 2011).

Lignocellulosic biomass is a complex biopolymer made up of cellulose, hemicellulose, and lignin-derived from agricultural wastes or by-products. It is plentiful and is the world's largest renewable resource. It also has the advantage of being widely available, with significant quantities in various parts of the globe. It includes more oxygen and less hydrogen and carbon than petroleum-based resources. Lignocellulosic bio-refineries may be able to produce a wider range of products than petroleum-based refineries due to this variance in composition. However, handling lignocellulose biomass necessitates a broader range of processing techniques (Furukawa, 2013). The usage of renewable energy resources is becoming more important in human daily life, due to environmental friend and regeneration to replace nonrenewable resources (Faria et al., 2015).

Different processing technologies such as solvent casting, melt blending, and in-situ polymerization was used to create nanocellulose-based nanocomposites. The impacts of processing technology on the mechanical properties of nanocomposites were given diverse percolating structures by modifying the system's processing rate and viscosity (Kong et al., 2015). The manufacture of the nanocomposite was done using the solvent casting technique, which was based on the processing technology used (Dong et al., 2015). Because of its simplicity, casting-evaporation or solvent casting is the most often used process in laboratories to transport cellulose fillers into the polymeric matrix. This process comprises pouring a mixture of nanocellulose suspension and polymer solution into polypropylene dishes and letting the solvent evaporate at the proper temperature (Kong et al., 2015). Water or another organic solvent can be used as the medium.

Nanocellulose (NCs) is a natural substance that is derived from native cellulose and has at least one dimension in the nanoscale range (Bhat et al., 2017). It is derived from the plant cell wall and other parts which have desirable features such as high strength, outstanding stiffness, and a large surface area (Abitbol et al., 2016). Furthermore, due to its structure, nanocellulose contains a large number of hydroxyl groups that can be used for surface modification (Phanthong, Reubroycharoen, Hao, & Xu, 2018). Nanocellulose synthesized from cellulosic sources is becoming an important topic of current research since it offers a unique mix of high physical qua lities capable of manufacturing a wide range of high-value goods with minimum environmental impact (Ramos et al., 2016).

Cellulose is a hydrophilic biopolymer made up of 1,4 bonded an-hydro-glucose units with alcoholic hydroxyl groups in a linear macromolecular chain. Numerous hydroxyl groups on the glucose ring enable widespread intramolecular and intermolecular hydrogen interactions, which bind the molecules together tightly, culminating in the formation of insoluble crystalline cellulose micro-fibrils. Indeed, hydrogen bonding inside and between cellulose chains is linked to the material's unique qualities, including extreme strength, durability, crystallinity, stiffness, and biocompatibility (Zhao et al.,2013). Furthermore, these hydroxyl groups impart hydrophilicity to all-natural fibers, and their moisture content approximated 8–12.6%. Though the crystalline cellulose structure is the same in all-natural fibers, the degree of polymerization differs (Khan et al., 2020).

Bamboo is a naturally renewable material that may be found in abundance in most tropical areas. It is a natural fiber plant that has gotten a lot of press in recent years as a sustainable resource for a variety of applications including building construction, housing, flooring, alternative fillers, automobiles, and furniture (Anuar et al., 2017). The world's bamboo species number is believed to be 1500, covering approximately 14 million hectares of land. Among them, Africa has over 40 species spread across over 1.5 million hectares of land, with two of these species found in Ethiopia (Khalil et al., 2012).

Kassahun Embaye (2000) estimates the amount of Ethiopia's natural bamboo coverage, it is approximately 1 million hectares, accounting for approximately 67% of African bamboo forest cover and 7% of the global total. Bamboo has recently been identified as an essential plant fiber with significant potential for usage in the polymer composite sector.

Bamboo contains 60% cellulose and a high level of lignin (which is quite tiny), and these properties have made bamboo fiber the primary attraction for reinforcement in a range of matrices (Khalil et al., 2012). Researchers have devised several ways for extracting bamboo fiber for composite reinforcement and have explored the changes that occur in the fine structure of its fiber as a result of treatment with various concentrations of an alkali solution (Das & Chakraborty, 2006).

Polyvinyl alcohol (PVA) has been used in numerous research studies to create blends and composites incorporating chitosan, starch, or lignocellulosic fillers. This is due to its perfect natural attributes, which include the capacity to chemically bond with cellulose, is soluble in water, easy to process, biodegradable, biocompatible, non-toxic, quickly created, and possessing hydrophilic capabilities (Slavutsky&Bertuzzi, 2014). When cellulosic materials are utilized as fillers, the biodegradability of PVA allows for the development of green nanocomposites (Fahma et al., 2017).

1.2. Statement of the problem

Nanotechnology has recently played an essential role in the contemporary period by providing a wide range of applications, such as industrial product packaging, electrical and electronics technologies, construction and building assistance, health and biomedical service, chemical, and food industries that can solve real-world problems and benefit society (Halib et al., 2017; Lam et al., 2012; Filler & Othman, 2016). The scarcity caused by the high cost of non-renewable products currently circulating in the industry is one of the major factors driving to search alternative and viable materials that are sustainable, renewable, and environmentally acceptable materials that can either partially or completely replace synthetic products (George, 2015; Kaur et al., 2017). Because of its remarkable environmentally favorable features, lignocellulose biomass such as corncob, water hyacinth, bamboo, and bagasse (Thakur et al., 2020; Mandal & Chakrabarty, 2011), is a source of natural fiber that can substitute petroleum-based polymers. Most current industrial items are mostly unfriendly to the environment when used non-renewable and the majority of them are not easily degradable, posing a substantial environmental threat that affects society's health (Phanthong et al., 2018). Ethiopia has a dense bamboo forest and unlike in other countries, bamboo use in Ethiopia has been customary and limited to hut construction, fencing to a lesser extent, the production of handicrafts, furniture, baskets, beehives, firewood, fodder, house utensils, various artifacts, and walking sticks (Kindu, 2016 ; Embaye, 2004).

It has recently been combined with cement or adhesives to create much tougher and more aesthetically beautiful materials that are suitable with current lifestyles (Sharma et al., 2019). Despite the abundance of naturally degradable bamboo goods, the country has been harmed by the usage of environmentally damaging synthetics. Extracting nanocellulose from natural biomass and incorporating it into a polymer matrix for the synthesis of degradable green polymer composite materials is one of the alternatives being studied to address these global environmental challenges. Up to date, several studies were done on synthesis of bamboo nanocellulose, but the optimization of the chosen process parameters, such as acid concentration, reaction time, and mixing temperature of the acid hydrolysis process on the yield was not significantly optimized. Therefore, extracting cellulose from the Ethiopian highland bamboo at the nanoscale, optimization of process parameters, and its characterization and incorporating it into the degradable polymer polyvinyl alcohols to produce value added nanocellulose product for nanocomposite applications was the primary goal of this study.

1.3. Objectives

1.3.1. General objective

- The general objective of this study is to synthesize and characterization of nanocellulose from Ethiopian highland bamboo *(Arundinaria Alpina)* for nanocomposites application.

1.3.2. Specific objectives

- To investigate the proximate analysis and chemical compositions of highland bamboo
- To synthesize nanocellulose from bamboo by acid hydrolysis and study its physicochemical characteristics such as functional group, crystallinity, and particle size.
- To investigate the optimum conditions of acid hydrolysis parameters of nanocellulose synthesis such as temperature, reaction time, and acid concentrations.
- To produce PVA- Nanocellulose composite film and its characterization such as water absorption capacity, mechanical properties, and thermal stability.

1.5. Significance of the study

Nano cellulose is essential for determining the performance of reinforcing elements in polymer green composites due to their large surface area and small-scale dimensions. Because of their high content of cellulose and outstanding mechanical properties, they could be a feasible option for glass fiber composites in the burgeoning advanced composites market. By incorporating nanocellulose generated from bamboo into PVA, this study increases its properties. Each year, humans harvest a considerable amount of bamboo. However, unlike in other nations, bamboo utilization in Ethiopia has been customary and mostly limited to local materials synthesis (Kassahun Embaye, 2004). It is, nonetheless, a good source for employing it as a reinforcing material in composite construction. As a result, this research aided in refining the properties of PVA, resulting in environmental protection, as well as allowing researchers to use this very inexpensive and locally available material for additional scientific inquiries.

1.6. Scope of the study

The major goal of this study is to make nano-cellulose from bamboo and use it to reinforce with polyvinyl alcohols to synthesize nanocomposites by utilizing solvent casting methods. Bamboo chips, which are largely sourced from bamboo culms, were treated with a variety of chemicals to make nanocellulose. In general, bamboo preparation, as well as cellulose isolation and purification came first. The optimization of parameters for acid hydrolysis conditions such as temperature, reaction time, and acid concentration was also carried out by response surface methodology through design expert software. The physicochemical properties of synthesized nanocellulose were experimentally characterized by using Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), and dynamic light scattering (DLS) respectively. Finally, the nanocomposites film was fabricated by solvent casting methods and its characterization such as water absorption capacity, mechanical properties, and thermal stability performance, was investigated.

2. LITERATURE REVIEW

2.1. Nanotechnology

The word nanotechnology is derived from two words: the Greek numerical prefix Nano, which means dwarf and refers to a billionth or exceedingly small, and the word technology. As a result, nanotechnology or nano-scaled technology is commonly defined as the study of matter at a size less than 100 nm (Fall, 2013). It is now widely regarded as one of the most promising areas of technological progress in the twenty-first century. With the advancement of nanotechnology and the recent concern about environmental issues, greater emphasis has been placed on the use of bio-based materials. Natural fibers have attracted much greater attention in this area due to their promising qualities such as biodegradability, renewability, and low cost (Davoud pour et al., 2013). The most important attribute of nanoparticles is their surface area to the volume aspect ratio, which allows them to easily interact with other particles. It has been observed that the physical and chemical properties of any substance alter as its size is reduced to the nanoscale. When compared to the bulk size of the same material, nano-sized particles exhibit differences in optical, electrical, and magnetic properties (Thamer et al., 2018).

Nanotechnology is also an applied science concerned with the design, synthesis, characterization, and application of nanoscale materials and technologies. This discipline of knowledge is a subcategory of technology in colloidal science, biology, physics, chemistry, and other scientific fields, and it entails the study of phenomena and the manipulation of materials on the nanoscale. As a result of their size and structure, materials and systems frequently exhibit novel and drastically altering physical, chemical, and biological properties (Kong et al.,2000).

Green technology is a type of nanotechnology that improves the environmental sustainability of manufacturing operations. Through fast development in nanoscale science and engineering, unprecedented opportunities have been elevated to develop more cost effective and environmentally acceptable remediation processes (Fall, 2013).

The merging of nanotechnology and lignocellulosic biomass utilization is vital in sustainably meeting the needs of people for food, clothing, shelter, commerce, and the array of products and goods needed for quality of life considerations both in meeting creature comfort and ecological needs (Arora et al., 2020).

It is critically important to move forward nanotechnology involving renewable biomaterials by exploiting as an important sustainable and renewable industrial nanomaterial enabling other nanomaterials to be used in conjunction with lignocellulosic products to impart greater functionality and reducing petroleum based materials use, the environmental footprint for bproducing such materials in production sector (Jasmani et al., 2020).

The term sustainability has been used in a variety of disciplines and in numerous contexts, ranging from the concept of maximum sustainable yield in lignocellulosic biomass management to the vision of a sustainable society with a sustainable economy (Wegner & Jones, 2005).

Generally nanotechnology can solves the issue that considered with environmental sustainability, by extracting cellulose at nanoscale from lignocellulosic materials through various methods includes chemical, biological and mechanical methods for replacement target of petroleum based products in current industries inadvance.

2.2. Nano-biocomposites

Nanocomposites are composites that have at least one phase with nanoscale dimensions. Nanocomposite materials have emerged as viable alternatives to the baffling limitations of micro composites. They are also regard ed as 21st-century resources due to their design rarity and property groups that are not present in traditional composites (Dong et al., 2015). Nano-biocomposites are composite materials that contain one or more biologically produced components (Shchipunov, 2012). Commonly the reinforcing phase is made up of plant fibers found in agricultural wastes and crop residues. Vegetable oils or starches are widely employed as the matrix in bio-composites including natural fibers. It's a promising way to improve material characteristics while maintaining biodegradability, and it's gotten a lot of attention in recent years as a result of a growing awareness of and need for environmentally sustainable solutions. Composites are produced by blending two or more materials to create a one-of-a-kind set of characteristics. The arrangement of the phases relative to one other can be controlled during processing to produce the required attribute by combining two or more materials (reinforcing elements, fillers, and matrix binders) that differ in form or composition (Ching et al., 2015). It is composed of a grouping of two materials of different natures, which completes and permits us to obtain a material with a greater set of performance characteristics than the components taken independently. Mostly composite material is constituted by a matrix and a reinforcement consisting of fibers. The matrix itself comprises a resin and filler, to improve the characteristics of the resin while reducing the production cost. From a mechanical point of view, the filler-resin system behaves as a homogeneous material, and the composite is considered as being made of a matrix and reinforcement (Pandya, 2015).

Nano-biocomposites are a new class of nano-sized materials made up of a biopolymer matrix supplemented with particles with at least one dimension in the nanometer range (1-100 nm) that have significantly improved characteristics due to the nano-particles high aspect ratio and surface area (Shchipunov, 2012). It also has distinct characteristics such as low density, biodegradability, and good mechanical properties. Furthermore, it is easily modified, has a large surface area, and has a typical morphology. The reinforcement improves the mechanical performance of the composite material. The type of reinforcement matrix association is determined by the constraints imposed on high mechanical properties, good thermal stability, cost, and corrosion resistance (Fowler et al., 2006).

Natural fibers such as jute, bamboo, coir, sisal, and pineapple are known to have very strong. These fibers have special advantages in comparison to synthetic fibers in that they are abundantly available, from a renewable resource, and are degradable. But all-natural fibers are hydrophilic and have high moisture content, which leads to a poor interface between fiber and hydrophobic matrix. Several treatments are employed to improve the interface in natural fiber composites. Natural fibers are generally incompatible with hydrophobic polymer and tend to form aggregates. Therefore the surface of both (matrix and fibers) should be approximately wetted to improve the interfacial adhesion and remove any impurities. Bio-based materials, in many cases, offer weight reduction, increased functionality, and occupational health benefits (Ariadurai, 2014). Many biopolymers, such as polyvinyl alcohol (PVA), polyethylene glycol (PEG), polycaprolactone (PCL), Polylactic acid (PLA), and others, are used as reinforcement materials in nano-biocomposites (Jie Wang et al., 2019).

2.3. Cellulose and nanocellulose

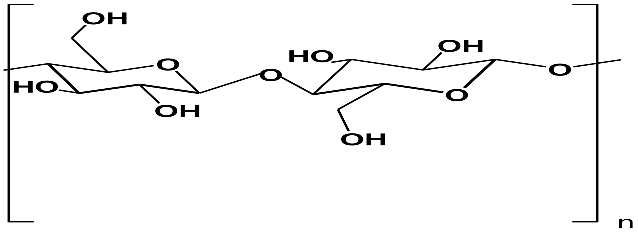
Cellulose can be considered as one of the most illimitable renewable biopolymers on the earth. It is an almost inexhaustible source of raw material to supply the growing demand for the production of environmental-friendly materials (Gan et al., 2019). It is a natural polymer classified as polysaccharide, which is most frequently found in the plant kingdom, comprising at least one-third of the vegetable material in the world and, it is present in materials such as wood, seeds, and agricultural waste (Abbate et al., 2016).

It is the most abundant component of lignocellulosic biomass, accounting for 30–50% of total biomass (Khan et al., 2020). It has a wide spectrum of outstanding biocompatibility, biological degradability, and sustainability qualities. Among these natural fibers, the most abundant biopolymer is cellulose, which can be found in a wide spectrum of natural fibers.

It is found in higher plants including wood, cotton, flax, hemp, jute, ramie, cereal straws, rice straws, and sugarcane bagasse, and it's insoluble in water and most organic solvents because of the vast network generated by multiple intermolecular and intramolecular hydrogen bonds (Zhao et al., 2013). In terms of chemistry, cellulose is a linear, stereo-regular natural homopolymer made up of -1, 4-glycosidic linkages that connect glucose units. The isomers are organized in a row and are kept together by hydrogen bonds between adjacent chains, making them more resistant to hydrolysis than starch. Mechanical characteristics, density, and biodegradability of cellulose are all influenced by the cellulosic basis and groundwork technique.

Cellulose is also the build-up of extended polymer chains with changing molecular weights and has a formula $(C_6H_{10}O_5)n$ (Wang et al., 2020).

Nanocellulose (NCs) are unique nanomaterials derived from the most abundant and almost inexhaustible natural polymer, cellulose. These naturally occurring cellulose fibers are biodegradable and renewable and hence they serve as a sustainable and environmentally friendly material for most applications. The nanocrystals are hydrophilic; however, it can be surface functionalized to meet various challenging requirements, such as the development of high-performance nanocomposites, using hydrophobic polymer matrices (George, 2015). NCs are high-aspect-ratio crystalline particles and acid hydrolysis can also produce these tiny particles in tunicates (Moon et al., 2011). The production of sulfate ester groups results in a negative particle charge, which improves the phase stability of nanocrystalline particles in an aqueous media. Nano-crystalline particles extracted from tunicates and bacterial cellulose are typically larger than NCs extracted from wood or cotton, according to studies. When nanoparticles are randomly distributed in a matrix, the surface area increases, resulting in improved stiffness, strength, and glass transition temperature. Because of its small particle size and great crystallinity, nanocellulose is often utilized as a nanofiller in several polymer products. It's applied as a barrier in the hazardous waste separation process, food wrappers that replace non-biodegradable plastics, and as a nanocomposite to enhance the polymer's mechanical, thermal, and ionic conductivity characteristics (Filler & Othman, 2016).



Source: (Phanthong et al., 2018)

Figure 2.1: Chemical structure of cellulose

Table 2.1: Chemical properties of cellulose

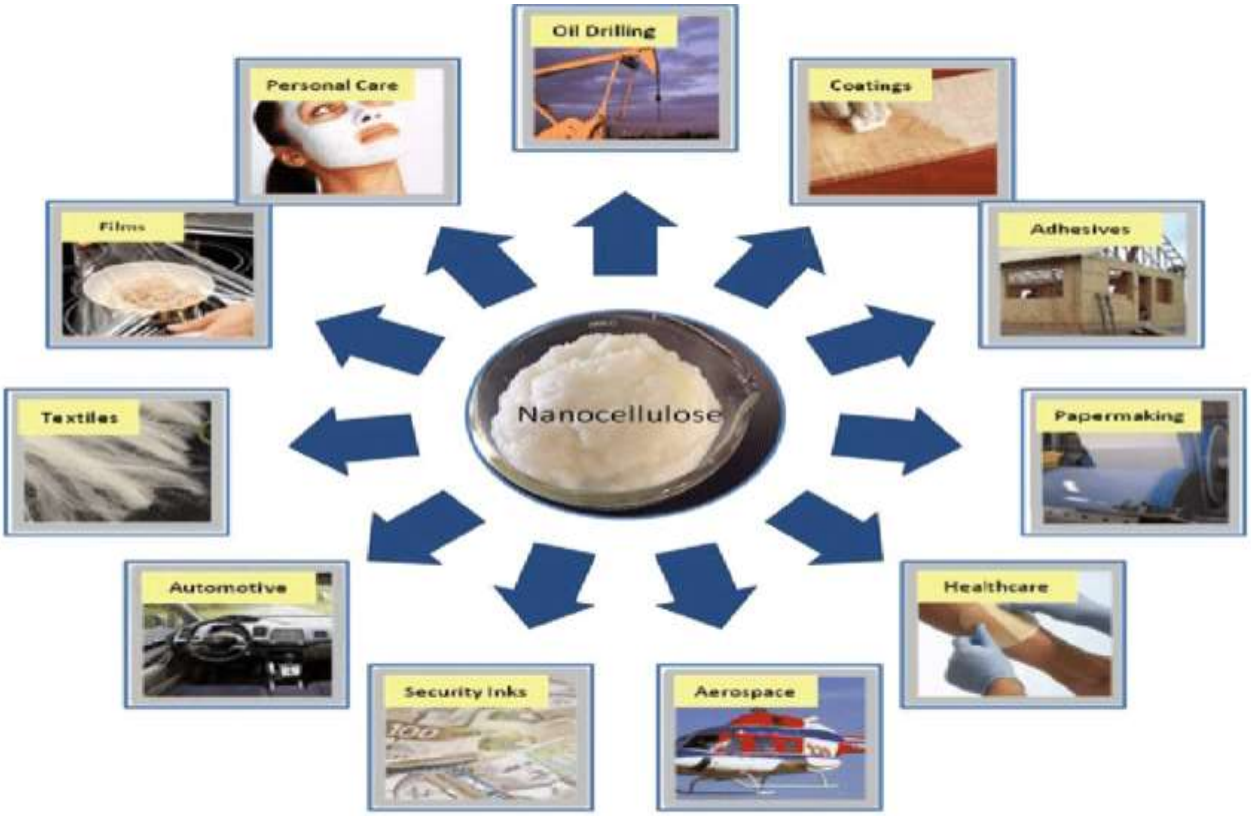
Physical and chemical properties of cellulose	
Chemical formula	$(C_6H_{10}O_5)_n$
Molar mass	162.14 g/mole
Melting point	260℃ – 270 ℃
Solubility	Insoluble
Density	1.5 g/cm3
Appearance	White powder
Odor	Odorless
Taste	Tasteless
Functional properties	Biodegradable and biocompatible

Source: (Nishiyama et al., 2003)

2.4. Application area of nanocelluloses

Nanocelluloses are a versatile nanomaterial that can be used in a variety of applications (Hossain et al., 2018) including automotive and construction industries, electronic components, enzyme immobilization, antimicrobial synthesis, sports and leisure, packaging materials, green catalysis, bio-sensing, paper production, polymer nanocomposites, and biomedical applications (Sharma et al., 2019). Packaging, automotive, aerospace, construction, furniture, biomedical, and other industries use nanocellulose-reinforced polymer matrix composites (Hossain et al., 2018). The increasing demand for lightweight materials in automotive and aerospace applications has pushed advanced materials research to new levels. Natural fiber composites are used to manufacture several components in the automotive sector (George, 2015).

Bio-composites are useful in biological applications because they are biodegradable and, more significantly, biocompatible (Phanthong, et al., 2018). One of the most important applications of bio nanocomposites in biomedicine is the regeneration of damaged tissues and the use of implants (Darder et al., 2007).

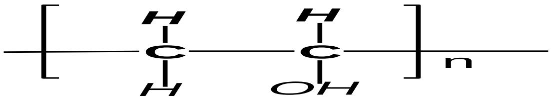


Source: (Moon et al., 2011)

Figure 2.2: Different Application areas of Nano cellulose

2.5. Polyvinyl alcohols

PVA has been used as a suitable host material to synthesize polymer nanocomposites with different fillers, as a large amount of hydroxyl groups on its carbon backbone enables the formation of hydrogen bonding with hydrogen bond acceptor atoms contained in the reinforcing phase. Hydrogen-bonding interactions provide good dispersion of nanofillers, which is a crucial factor determining the polymer nanocomposite properties (Aslam et al., 2018). It has been used in the industrial, commercial, medical, and food sectors to produce a wide range of end products, including lacquers, resins, surgical threads, and food packaging materials that come into contact with food. It is a widely used thermoplastic polymer that is non-toxic and harmless to living tissues (Cobos et al., 2020). Due to its use in cross-linked products and nano-fillers polymer, its exceptional chemical and physical properties, biocompatibility, ease of processing, biodegradability, non-toxic hydrophilic properties, and stability to temperature variation, it is being studied broadly (Fahma et al., 2017). PVAs are widely used as surface materials that should be retained on water surfaces, in a wide range of fields as films and glues. Polyvinyl alcohol for food use is an odorless and tasteless, translucent, white, or cream-colored granular powder. It is soluble in water, slightly soluble in ethanol, but insoluble in other organic solvents (Hajeeassa et al., 2018). Generally due to its outstanding chemical and physical properties, and high thermal stability, PVA can be used in broad applications in different industrial manufacturing areas rather than other plymers such as PLA (Abdullah, 2017).



Source: (Hallensleben et al., 2015)

Figure 2.3: Chemical Structure of PVA.

Table 2.2: Chemical properties of PVA

Physical and Chemical properties of PVA	
Chemical formula	$(CH_2CHOH)_n$
Density	1.19-1.31 g/cm^3
Bulk density	641kg /m^3
Melting point	200℃
Boiling point	228℃
Flashpoint	79.44℃
Solubility	Soluble in water
Appearance	White
Odor	Odorless
Specific gravity	1.3

Source: (Aslam et al., 2018)

2.6. Synthesis methods of nanocellulose

A variety of techniques have been used to synthesis nanocellulose from cellulosic materials with various yield and qualities. The most prevalent synthesis methods are acid hydrolysis, enzymatic hydrolysis, and mechanical extraction (Jabbar et al., 2016; Phanthong et al., 2018).

2.6.1. Acid hydrolysis

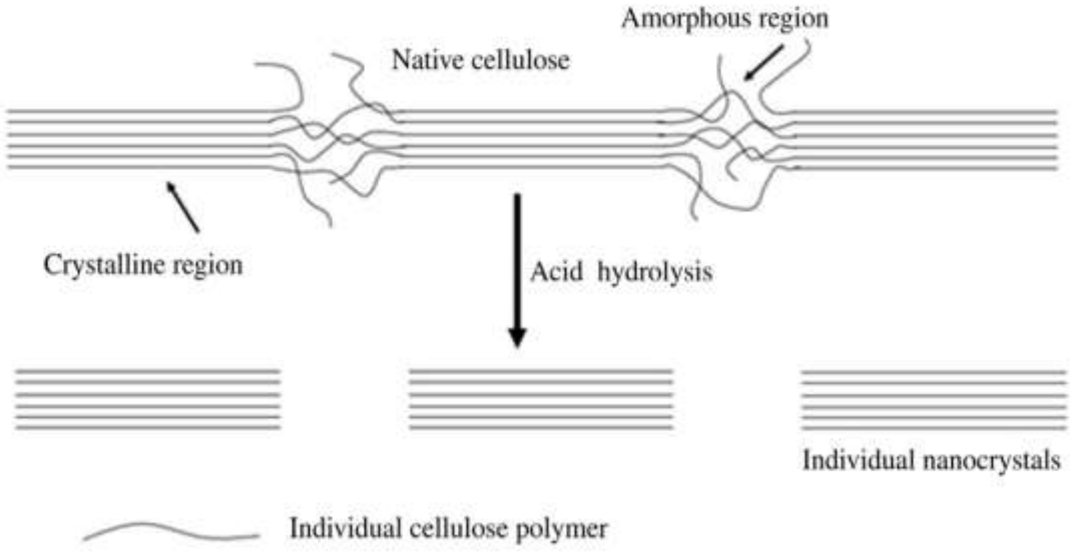
Controlled acid hydrolysis is one of the most common methods in dissolving amorphous domains of cellulose fiber and hence longitudinal cutting of the micro-fibrils. It is the process of breaking down polysaccharides in lignocellulosic materials into simple sugars using acid solutions (Mohammad et al., 2014). It is the most predominant approach for extracting nanocellulose from lignocellulosic materials among numerous chemical processes for nanocellulose synthesis. Because disordered areas of cellulose chains are rapidly dissolved by acid, ordered parts are left behind when ordered and disordered regions of cellulose chains are combined. Sulfuric acid, hydrochloric acid, phosphoric acid, hydrobromic acid, maleic acid, nitric acid, and formic acid are all mineral acids that can be utilized for this purpose (Xie et al., 2018).

Sulfuric acid is, nevertheless, the most commonly used acid for this purpose, since it produces NCs particles grafted with a sulfate ester group (Kumar et al., 2014). NCs particles have a negative electrostatic repulsion force that promotes uniform dispersion in water due to the presence of these negatively charged groups (Harmsen & Huijgen, 2010). Furthermore, the crystallinity index of Sulfuric acid was the greatest, followed by phosphoric acid, because of the esterification of the hydroxyl group by sulfate ions, nanocellulose is disseminated as a stable colloid system, and it may firmly isolate nanocellulose (Phanthong, Reubroycharoen, Hao, & Xu, 2018).

The materials with higher degrees of crystallinity are produced based on the kinetic difference between amorphous and crystalline areas. It is dissolving amorphous domains of cellulose fiber by longitudinal cutting of the micro-fibrils (Bhat et al., 2017). The acid hydrolysis of cellulose causes a rapid drop in its degree of polymerization, culminating in the formation of nano-sized particles by reducing the size of the cellulose chains (Bondeson et al., 2006).

Lignin and hemicelluloses are extracted from the fiber using thermal, chemical, biological, physical, or a mix of these processes, depending on the degree of separation required and the function for which they are intended (Abbate et al., 2016). The lignocellulosic material is first alkalized and bleached with bleaching agents to eliminate impurities and lignin, and hydrolyzed to produce lignin-free hollo-cellulose.

To release NCs, pure cellulosic materials must be under controlled temperature, time, agitation, and acid to cellulose ratio conditions. The NCs dispersion in a strong acid is diluted with water and washed using successive centrifugations, neutralization or dialysis with distilled water is performed to remove free acid from the dispersion. The acid wastewater generated from the washing process during neutralization of the pH value of nanocellulose suspension was the main drawback of acid hydrolysis methods (Bhat et al., 2017).



Source: (Bhat et al., 2017)

Figure 2.4: Nanocellulose synthesis by acid hydrolysis

2.6.2. Enzymatic hydrolysis

Enzymatic hydrolysis is a biological treatment method that employs enzymes since, in nature, cellulose is not degraded by a single enzyme, but by a set of cellulases. The process of digesting or modifying cellulose fibers under mild conditions. Cellulases hydrolyze the β-1, 4-D glycosidic bonds of the glucose units to cut the cellulose molecules into shorter polysaccharides. The biological treatment uses less energy, is more environmentally friendly, and produces higher yields and selectivity. To decompose lignin and hemicelluloses from lignocellulosic material, microorganisms such as brown, white, and soft rot fungus and bacteria are utilized (Bhat et al., 2017).

However, because enzymes and other microorganisms are utilized, biological treatment takes a long time and is costly. To make cellulose nanoparticles, biological hydrolysis must always be combined with additional processes such as mechanical shearing and high-pressure homogenization, resulting in controlled fibrillation down to the nanoscale (Mohammad et al., 2014).

2.6.3. Mechanical treatment processes

The mechanical treatment procedure synthesis of cellulose fibrils by cleaving the cellulose fibers in the longitudinal axis with a high shear force, resulting in nanofibrillated cellulose (Bhat et al., 2017). Mechanical methods have also been extensively researched for the fabrication of nanoscale cellulose particles, either as part of a fabrication process that includes a combination of acid hydrolytic, oxidative, and enzymatic treatment or as a direct method (Anuar et al., 2017).

High-pressure homogenization, ultra-sonication, and ball milling are the most commonly utilized mechanical processes. The mechanical procedure, on the other hand, has a major disadvantage since, it consumes a lot of energy (Phanthong et al., 2018).

2.7. Factors that affect the nanocellulose yield

During nanocellulose extraction by acid hydrolysis, various parameters affect the yields. The most common parameters are reaction time, temperature, and acid concentration (Ioelovich, 2012; Nilsson, 2017).

2.7.1. Reaction time

The most important factor in acid hydrolysis for nanocellulose synthesis is reaction time. The hydrolysis was carried out based on the time accustomed for the accomplishment of the process. Incomplete removal of amorphous fractions can occur if the hydrolysis time is insufficient, resulting in a drop in crystallinity and a change in particle shape. Similarly, increasing the reaction time beyond a certain might cause depolymerization of nanocellulose, which reduces its aspect ratio and can even result in the formation of a much smaller spherical particle. The treatment time can range from 30- 60 minutes with vigorous and constant stirring (Alves et al., 2013).

2.7.2. Acid concentration

The synthesis of nanocellulose from lignocellulosic materials depend on the concentration of acid used during processing. The crystallinity of cellulose nanocrystals is affected by the preparation technique, pretreatment, and the type of acid.

Although acid hydrolysis is widely used for the production of NCs, certain problems must be overcome, such as high consumption of energy and chemicals, acidic corrosion of equipment, and health and environmental hazards.

Recently, several studies have focused on hydrolysis parameter optimization, corrosion prevention, and waste reduction of nanocellulose extraction using sulfuric acid (Ahmad et al., 2017). Typically the concentration of sulfuric acid in hydrolysis reactions to obtain nanocellulose is around 65wt. %. from Lignocellulosic biomass (Ioelovich, 2012). Not only sulfuric acid but also hydrochloric acid in less amount was used in some studies (Xie, 2018).

When the acidic solution of cellulose is diluted with water, the soluble cellulose is regenerated and precipitated in a form of amorphous flocks. After the fragmentation of these collects in the water medium, the low-molecular amorphous particles are isolated. Another problem is the accelerated hydrolysis of cellulose macromolecules in the acidic solution that leads to a decrease in the yield of nanoparticles. So, nanoparticles prepared by hydrolysis of cellulose with highly concentrated acids (> 63 wt. %) were amorphous. Thus, more exhaustive studies are required to determine optimal conditions (Ioelovich, 2012).

2.7.3. Reaction temperature

The temperature of the mixing process has a significant influence on the yield and other properties of the nanocellulose. During nanocellulose synthesis, the temperature is accustomed for the conversion of cellulose to nanocellulose by supplying heat to break the amorphous parts of the sample. If the acid treatment temperature is less than 30°C, the hydrolysis process will take a long time. If the hydrolysis temperature is higher than 60°C, however, hydration and carbonization cause the cellulose particles to darken (Saba et al., 2017). As a result, the ideal temperature for acid treatment is between 45-60 ℃ (Dong et al., 2015).

2.8. Preparation of nanocellulose/PVA composites

2.8.1. Solvent casting methods

The solvent casting process, in which the solvent is evaporated from the film after dispersion, is one of the most common means of creating nanocomposite films. Nanocellulose-based fillers must not form aggregates and must be homogeneously disseminated in polymer matrixes, to utilize its advantage as an effective reinforcement to generate high-strength polymer composites (Jara et al., 2020). The fundamental benefit of employing an aqueous media for processing is that the micro-particle dispersion condition is conserved.

The aqueous solution can be combined with the aqueous suspension of cellulose micro-particles once the hydro-soluble polymer has been dissolved. To achieve a solid micro-particle film, the resulting mixture is usually cast and evaporated.

The standard solvent is hot distilled water, and the cellulose is initially dispersed in the standard casting solution of hot water before being ultra-sonicated. For chemically modified cellulose, glycerol is used as a dispersion agent.

The polymer solution is made by dissolving it in hot water and mixing it with cellulose suspension for polymer cellulose suspension reinforcement. The solution was cast onto a glass petri dish and oven-dried or dried at decreased pressure inside a vacuum oven to remove the solvent after manually mixing for the desired period at a steady speed. This method was chosen for this study because it is simple and can be done manually at a lab-scale without any involvement of specialized equipment or automated devices (Dufresne, 2015). The main disadvantages of this approach include the potential for harmful solvent retention inside the polymer matrix, the denaturation of proteins and other molecules introduced into the polymer matrix due to solvent use, and the end product's form limitations (Chu & Liu, 2008).

2.8.2. Melt blending methods

Melt blending of nanocomposite fabrication is also a common method. Cellulose composites with and without the use of solvents by melt blending are generated by using a batch mixer through a combination of plasticizer and polyvinyl alcohols. This was monitored by the addition of cellulose and mixing. After adding the compatibilizer, the molten composites were put into the molder and crushed between two plates or a hydraulic press before being cooled to form the film. Melt blending shows higher gas barrier performance compared to solvent casting (Dong et al., 2015).

2.8.3. In situ polymerization methods

Due to the highly toxic solvents used to dissolve polymers, this method is primarily used for polymers that cannot be produced economically or safely utilizing solution methods. It enhances good nanoparticle dispersion and distribution in the polymer matrix.

The first is associated with the process's cost, which may necessitate certain modifications compared to standard polymer synthesis. It's also important to find the best catalyst for the work. The apparatus used for polymerization without nanoparticles can be the same (Shchipunov, 2012).

Intercalation is the reversible inclusion or insertion of a molecule or ion into materials with layered structures. It expands the vander waals gap between sheets which requires energy.

Usually, this energy is supplied by charge transfer between the guest and host solid. Exfoliation is an extreme case of intercalation, where the complete separation of the layers of the materials happens. Typically aggressive conditions are required involving highly polar solvents and aggressive reagents (Ines et al., 2014).

2.9. Bamboo Plant

Bamboo is a naturally occurring composite biological plant material that grows abundantly in tropical climates. It is classified as a composite material. Since it is made up of cellulose fibers agglomerated in a matrix of hemicelluloses and lignin as an encrusting ingredient. Cellulose fibers run the length of the bamboo, providing maximal tensile flexural strength and rigidity in that direction. It is commonly understood that ecological materials meet key requirements such as pollution avoidance and cost reduction. The use of environmentally friendly agricultural byproducts such as rice husk, coconut fibers, sisal, and bamboo to reduce energy consumption, conserve nonrenewable natural resources, reduce pollution, and maintain a healthy environment (Lugt & Dobbelsteen, 2005).

Bamboo is the heart of these materials that provide these benefits. It is estimated that there are 60 to 90 genera of bamboo, with roughly 1100 to 1500 species, and that there are approximately 600 different botanical species of bamboo in the world (S. George, 2007). Bamboo plants are highly sturdy in their longitudinal direction due to strong fiber bundles penetration. The thickness and strength of bamboo, on the other hand, decrease from the base to the top of the bamboo culm (Lugt & Dobbelsteen, 2005).Various researchers conduct experiments on the mechanical properties of various kinds of bamboo fibers as a function of age, moisture content, density, and others (Osorio et al., 2018).

Bamboo plants are classified as highland or lowland according to their geographical location in Ethiopia. The solid culm of lowland bamboo is not preferred for fiber split extraction. The lowland bamboo plant culms are distinguished by a large diameter culm, a thick internal wall, a low amount of fiber, and a high fiber strength, whereas the highland bamboo plant culms are distinguished by a small culm diameter, a thin wall thickness, a dense amount of fiber, and a high fiber strength. Bamboo species from Ethiopia's highlands were chosen for this study due to their richness, highly dense fibers found along the culm, and naturally thin wall thickness (Kindu, 2016).

2.10. Bamboo in Ethiopia

In contrasting to other countries, bamboo use in Ethiopia has been customary and has primarily been limited to hut construction, fencing, and, to a lesser extent, the production of handicrafts, furniture, containers for water transport and storage, baskets, beehives, firewood, fodder, house utensils, various artifacts, and walking sticks (Kassahun Embaye et al., 2004).

Figure 2.5: Highland Bamboo *Arundinaria Alpina*

Table 2.3: Major highland bamboo areas in Ethiopia

No	Bamboo Area	Region	Natural Stand (Ha)	Plantation (Ha)	Total (Ha)
1	Injibara	Amhara	30	2,350	2,380
2	Agaro	Oromia	-	1,500	1,500
3	Bale Mountains	Oromia	56,851	-	56,851
4	Shenen/Jibat	Oromia	1,774	2,561	4,335
5	Gera	Oromia	36,000	1,250	37,250
6	Bore/Hagereselam	Oromia	-	2,460	2,460
7	Chencha/Arbaminch	South	2,460	3,250	5,710
8	Indibir/Jembero	South	-	1,850	1,850
9	Jimma/Ameya	Oromia/South	-	900	900
10	Mizan Teferi/Kulish	South	-	1,850	1,850
11	Debresina/Wofwasha	Amhara	35	-	35
12	Wushwush/Bonga	South	-	1,120	1,120
13	Bonga/Ameya	South	7,997	-	7,997
14	Masha	South	18,652	-	18,652
15	Munesa/Shashemene	Oromia	4,183	-	4,183
Total	- -------	--------	127,982	19,091	147,073

Source : (*Socio-economic case study of the bamboo sector in Ethiopia : an analysis of the production-to-consumption system*, 2000).

2.11. Ligno-cellulosic content of bamboo fiber

Lignin, hemicellulose, and cellulose are the three primary polymers found in the cell walls of lignocellulosic biomass. However, because of differences in species, types, and sources of their biomass, the composition and amount of these components vary. Its structure is a composite material composed of long and aligned cellulose fibers embedded in a ligneous matrix (lignin and hemicellulose). Bamboo's chemical ingredients are main cellulose, hemicellulose, and lignin (Design et al., 1992).

2.11.1. Cellulose

Cellulose is the main component in lignocellulosic biomass, accounting for around 35–50% of the total. It is made up of a linear homo-polysaccharide comprising β-1, 4-linked anhydrous-D-glucose units, and cellobiose repeating unit. The anhydro-glucose unit, a monomer of cellobiose, is made up of three hydroxyl groups that create strong hydrogen bonds with adjacent glucose units in the same chain and with distinct chains, respectively, forming intramolecular and intermolecular hydrogen bonding networks (Xu, et al., 2018). It is found in a variety of natural fibers, including cotton, flax, hemp, jute, and sisal, among others. This natural polymer is found in one-third of plant tissues and can be replenished through photosynthesis (Mora, 2008). It is a basic structural unit of many plants, making it a nearly limitless source of raw material; in nature, celluloses, along with hemicellulose, serve to physically strengthen plant structures (J. Xie, 2018).

2.11.2. Hemicellulose

In lignocellulosic biomass, hemicellulose accounts for 20–35% of the total. Hemicellulose is a heteropolymer made up of short, linear, and branching chains of several types of monomers including pentose and hexoses. Xylans and glucomannans are two common forms of hemicellulose. Hardwood contains a lot of xylans, whereas softwood has a lot of glucomannans. Through hydrogen bonding and Vanderwal interactions, hemicellulose binds to cellulose fibrils. Moreover, it forms cross-links with lignin. The strength of the plant cell wall structure is linked to the embedding of hemicellulose with cellulose and lignin.

Hemicellulose can be hydrolyzed in mild conditions with acid, alkali, or enzymes to generate fuel ethanol and valuable compounds from its oligomers or monomers, which can be used in food, cosmetics, mining, and other sectors. It is mostly amorphous and has a highly branching random structure (Mora, 2008).

2.11.3. Lignin

Lignin makes up approximately 10%–25% of dry lignocellulosic biomass by weight. It acts as a binding agent in plant cell walls, holding cellulose and hemicellulose together. It provides stiffness, compressive strength, decay resistance, and water impermeability to plant cell walls through its binding activity. Lignin is a cross-linked amorphous copolymer made up of three distinct phenyl propane monomers, namely p-coumarin, coniferyl, and sinapyl alcohols, which have zero, one, and two methoxyl groups respectively. The ratio of these three major monomer units varies depending on the lignocellulosic biomass type and sources.

Recent research has focused on isolating lignin from lignocellulosic biomass nd depolymerizing it for the manufacture of biofuels and compounds from natural resources. Furthermore, lignin-based carbon compounds are used for catalysis, energy storage, and pollutant removal, all of which are promising developments (Xu, et al., 2018). It is completely amorphous and hydrophobic, and it is responsible for plant stiffness. It is colorless or pale yellow, but when exposed to acid or alkali, the color changes to brown (Kalia et al., 2011).

Table 2.4. Chemical composition of common lignocellulosic materials

Lignocellulosic materials	Composition (%)			References
	Cellulose	Lignin	Hemicellulose	
Corn Stover	40-45	22-28	18-22	(Costa et al., 2015)
Hamulus japonicas	44	23	20	(Y. Jiang et al., 2017)
Sugarcane bagasse	45	30	20-22	(Oliveira et al., 2016)
Arecanut husk fiber	34.18	20.83	31.6	(George, 2015)
Coconut fiber	31–32	25–26	33–37	(Mugo, 2020)
Barley straw	33-40	20-30	8-17	(Lee et al., 2014)
Corncobs	45	35	15	(Abbate et al., 2016)
Empty fruit bunch	41	24	21.2	(Abdel-Halim,2014)
Switch grass	45	32	12	(Yoon et al., 2012)
Nut shells	25-30	25-30	30-40	(Rivai et al., 2021)
Wheat straw	30	50	15	(Sirisomboon, 2017)
Pineapple leaf	35-40	20-25	25-30	(Osorio et al., 2018) (Nilsson, 2017)
Bamboo	50-55	20-30	15-20	This study

2.12. Challenges with the synthesis of nanocellulose

Recent developments in sourcing materials from renewable natural resources are likely to continue due to changing consumer perceptions, increasing ecological considerations, and technological advances. This has also increased from the standpoint of nanocellulose and composites, as nanocellulose has an interesting application for polymer matrices, additives, and reinforcement in the production of various composite materials (Lani et al., 2014).

This nanocellulose has the potential to provide benefits in specific properties such as lower density, renewability, high strength, biodegradability, transparency, and other properties that are not possible with other materials. However, the utilization of nanocellulose as reinforcement in the fabrication of various composite materials is still in its early stages (Moubarik et al., 2013).

The potential of nanocellulose reinforcing has not yet been completely exploited due to scaling problems in industrial manufacturing methods. This is because only a small amount of nanocellulose is available for large-scale research, industrial manufacturing processes, and development initiatives. Recent commercial and government pilot and plant-scale nanocellulose production has been reported, which will likely improve the situation (Yoon et al., 2012). As a result, the synthesis of nanocellulose has piqued the interest of various researchers interested in the production of composite materials. While much recent great development has been made in nanocellulose synthesis and reinforcing in polymeric materials, there are still several obstacles in terms of efficiency and cost-effective manufacture to be employed as reinforcement.

As a result, more efficient and effective methods for generating nanocellulose with ideal properties must be developed, which will necessitate future study to solve challenges connected to their hydrophilic nature for widespread deployment (Moubarik et al., 2013).

Acceptable dispersion of nanocellulose as reinforcement into a range of matrices is required for future acceptable applications to be identified, explored, shown, understood, and addressed for incorporation into different materials. The majority of these investigations used nanofillers such as nano silicon dioxide, montmorillonite clay, sodium montmorillonite clay, and poly(methyl methacrylate-co-acrylamide), cellulose, and micro cellulose (Yoon et al., 2012). To overcome these constraints, this study was chosen to extract nanocellulose from bamboo and reinforce it in polymer to improve the qualities of PVA reinforced with nanocellulose.

Furthermore, the enormous generation of unused bamboo from processing enterprises in Ethiopia that discard it for the environment has drawn this thesis to turn this underused Lignocellulosic material into a value-added product. This massive production of bamboo, as well as its high cellulose content, has inspired this research to create nanocellulose for use as a reinforcement in composite materials (Khalil et al., 2012). Additionally, the use of nanocellulose has gained a lot of attention due to its nano-dimension, biodegradability, abundance, high strength, renewability, and uniform dispersion in the matrix, making it a good candidate for the preparation of polymer nanocomposites and opening up a wide range of possible discoveries (Plengnok & Jarukumjorn, 2020).

2.13. Future developments in bamboo fiber-reinforced composites in advanced technology

The current industrial progress toward eco-efficiently of industrial products and manufacturing processes holds the key to a sustainable tomorrow for future generations. High performance, biodegradable materials, and renewable plant materials can create a new platform for sustainable and eco-efficient advanced technology products, competing with synthetic/petroleum-based products that currently dominate the market, even though natural petroleum feedstock is diminishing (Kumar et al., 2014).

Natural fibers and bio-composites derived from natural sources are incorporated into sustainable, eco-friendly, and well-designed industrial products that can eventually replace the dominance of petroleum-based products. Bamboo fiber comes from a plant that is noted for its renewability in terms of rapid growth and superior mechanical characteristics. The use of bamboo fiber for the manufacturing of bio-composites employing advanced technologies transforms future generations. Bamboo fiber products that are carefully conceived and constructed can contribute to a new revolution in the conservation of our natural resources (Khalil et al., 2012). As a result of this case, bamboo fibers can be used for advanced and engineered product creation for a variety of purposes.

Table 2.5: Summary of the common literature on process conditions of Nano cellulose

Lignocellulosic materials	Chemicals and process conditions used			Cr (%)	Y (%)	References
	Alkaline treatment	Bleaching	Acid hydrolysis			
Corncob	3wt.%NaOH solution for 3 h 100 ℃	$NaClO_2$ (0.02wt.%) at 80℃/ 2h	H_2SO_4(64wt.%) at 45 ℃, for 60min	83.7	41.5	(Dong et al., 2014)
Sugar bagasse	5%(w/v) NaOH at 80 ℃ for 1 h	3.3%(w/v) $NaClO_2$ 90 ℃/ 1 hr.	64%(w/w) H_2SO_4 at 45℃/60min	56.7	30	(Mandal, 2011).
Rice straw	600mL 5% KOH at room temp for 90 min	1.4%acidified $NaClO_2$(1000mL), at 70℃ for 5h	64-65wt.% H_2SO_4 at an 8.75 ml/g acid tocellulose ratio at a temp 45℃ for 45 min	61.8	45	(Lu,2012)
Palm Oil Empty Fruit	17.5 %(w/v) (NaOH)solution for 2 hours at 45℃.	1.7wt% $NaClO_2$ in water, mixtures of 27 g of NaOH & 75 mL acetic acid in 1 L of distilled water at 80℃/4h.	64wt.% H_2SO_4 solution under strong agitation at 45℃ for 45 min	73.63	50	(Marcos et al., 2014)
Pineapple leaf	2% (w/w) NaOH for 4h at 100℃ under mechanical stirring	1.7wt.% $NaClO_2$in water, mixtures of 27 g of NaOH and 75 mL glacial acetic acid in 1L of distilled water at 80℃ for 4 h	H_2SO_4 64%(w/w) at 45 ℃ for 60 min under constant stirring	73.63	50	(Marcos et al., 2013)
Corn Stover	2.0%NaOH solution at 80 °C for 4 h	1.7% $NaClO_2$ and buffer solution(CH3COOH/ NaOH) 1:1 (w/ v) at 60 °C for 24h	H_2SO_4 at 60 %(v/v) for 50 °C for 20 min	55.04	64	(Costa et al., 2015)

3. METHODOLOGY

3.1. Materials and Methods

3.1.1. Materials

Highland bamboo species used for this study were collected from Jimma Agricultural Research Center. After collection, packed in polythene bags and transported to the chemical engineering laboratory at Jimma Institute of Technology.

3.1.2. Chemicals and reagents

The chemical in Table 3.1, were used for this study in laboratory experiments. All chemicals are with their analytical grade and are used exactly as received. The experiments were carried out at Jimma Institute of Technology School of Chemical Engineering, School of Materials Science and Engineering, and Addis Ababa Science and Technology University Department of Chemical Engineering (Addis Ababa).

Table 3.1: Major Chemicals and reagents used in the experiment

Chemicals	Sources
Sodium hydroxide (97%)	Cherkos marketing center A.H plc.
Sodium hypochlorite (80%)	East Africa detergent factory
Sulfuric acid (98%)	Neway chemical and laboratory equipment plc
Glacial acetic acid (96%)	Neway chemical and laboratory equipment plc
Glycerol (92%),	Raychem industry and trading plc
Chloroform (99%)	Neway chemical and laboratory equipment plc.
Polyvinyl Alcohol (95%)	Alkane plc. Addis Ababa Ethiopia

3.1.3. Equipment and instruments

Centrifuge, Oven, Aluminum foil, Grinder, Heater with stirrer, Cooler, vacuum filter, petri dish, furnace, refrigerator, water bath, pH meter, sample collector, measuring cylinders, analytical balance, and evaporating dish were all employed in this investigation. Fourier transform infrared spectroscopy (FTIR),Dynamic light scattering (DLS), X-ray diffraction(XRD), Thermogravimetry analyzer (TGA) and tensile testing machine were employed for characterization.

3.2. Synthesis process of nanocellulose

3.2.1. Sample preparation

The collected highland bamboo was cleaned and sun-dried for five days and then oven-dried for 24 h at 105 °C until a constant weight was obtained. The dried bamboo was ground into powdered form by using a mortar and sieved to the proper size for the next process.

3.2.2. Purification processes

The purification of bamboo was the pretreatment for removing unwanted materials to obtain pure cellulose pulp. The bamboo chips was washed with 2wt% NaOH in distilled water, to remove lignin and other contaminants. This was accomplished by combining 100g of bamboo sawdust with 2wt% NaOH in 1 liter of distilled water. In a water bath at 90°C and 1000rpm stirring speed, the solution was manually and continuously swirled for four hours. The suspension was filtered through a vacuum filter and rinsed with distilled water numerous times. After four washes, the purified cellulose product was air-dried before being subjected to the bleaching process to lighten the color (Jabbar et al., 2016).

3.2.3. Bleaching and cellulose synthesis processes

Cellulose was synthesized according to the methodology proposed by Mandal and Chakrabarty (2011) with a few modifications. The bleaching procedure was utilized to remove lignin from bamboo powder using acidified sodium chlorite ($NaClO_2$). Briefly, one liter of distilled water was treated with 1.7% sodium chlorite ($NaClO_2$) and adding 75ml of acetic acid glacial with 27g of sodium hydroxide. The solution was handled in a water bath at 70°C with continuous mechanical stirring at a speed of 1500rpm. In this research, the bamboo fiber to acidified solution ratio was (1:50 w/v) (Mandal & Chakrabarty, 2011). The bamboo fiber was then cooked in the $NaClO_2$ and NaOH solution for 4 hours at 70 °C with continuous mechanical stirring at 1500rpm. The bleaching process was performed three times until the bamboo fiber's color became white. The suspension was filtered using a vacuum filter and the solid was washed with distilled water repeatedly until the yellow tint was gone, then air-dried.

3.2.4. Nanocellulose synthesis

The nanocellulose was synthesized according to the method described in Mandal, and W. Chakrabarty (2011) with slight modification. The bleached bamboo sample was then subjected to acid hydrolysis to remove the amorphous area of the cellulose to obtain crystalline nanocellulose.

10g of purified and bleached cellulose was hydrolyzed with different concentrations of (45, 55, and 65 wt. %) H_2SO_4 solution at a ratio of 1:10 (w/v) at the temperature of (40, 50 and 60°C) for reaction time (30, 45 and 60 minutes) under continuous mechanical stirring (Mandal & Chakrabarty, 2011). To quench the hydrolysis reaction, ten times the amount of distilled water (1000ml) was added to the reaction mixture soon after the termination time. The diluted suspensions were cooled to room temperature and centrifuged at 6000 rpm for 15 minutes to eliminate acidity. The cellulose was rinsed again with distilled water and centrifuged again. When the supernatant became turbid or the suspension of pH exceeded 5, washing and centrifuging operation was terminated and the remaining acid was removed using the neutralization process. Finally, the yield of nanocellulose was determined by the equation (3.1).

$$\text{Yield} = \frac{W_2}{W_1} \times 100 \qquad (3.1)$$

Where W_2 and W_1 were the weight of cellulose after hydrolysis and before hydrolysis respectively in (g).

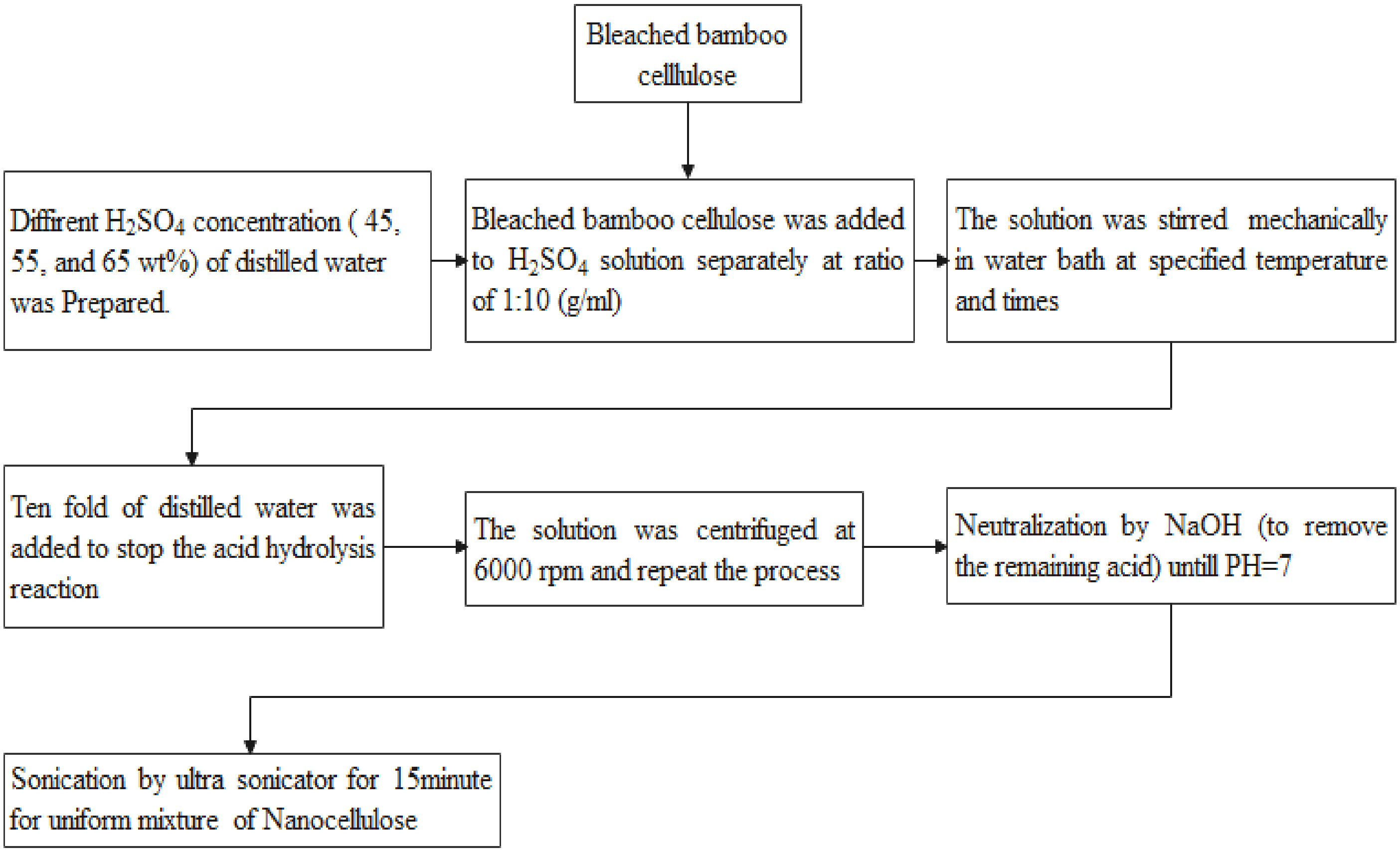


Figure 3.1: Block flow diagram of Nanocellulose synthesis from bamboo

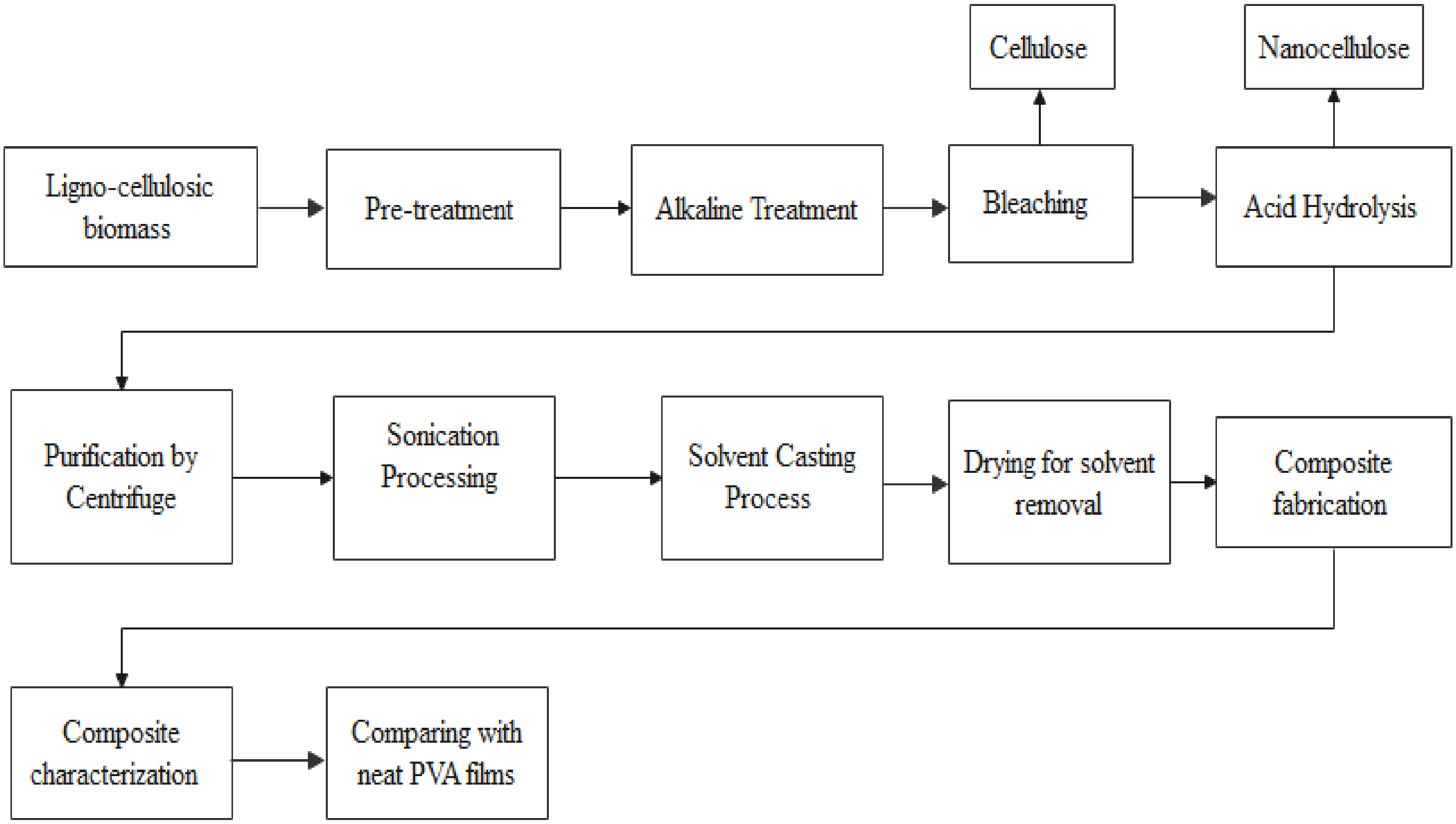


Figure 3.2: Nanocomposite processing from lignocellulosic biomass

3.3. Proximate analysis of bamboo

3.3.1. Determination of moisture content

Oven drying methods are used to determine the moisture content of bamboo by specified temperature of 105°C for 2hours until a constant weight was obtained by conforming to ASTM D1037 (1991) and calculated by equation (3.2).

$$\text{Moisture content} = \frac{M_i - M_f}{M_i} \times 100\ \% \qquad (3.2)$$

Where , M_i is initial mass before drying and M_f is the final mass after dried (g).

3.3.2. Determination of ash content

Ash content was determined based on standard methods from the American Society for Testing and Materials (ASTM D2017) (1998). This was done by measuring a fixed amount of bamboo powder and burned in a muffle furnace at 600-650°C for 2 hours until complete ash was achieved. Finally, the ash content was calculated by equation (3.3).

$$\text{Ash content} = \frac{M_a - M_c}{M_b - M_c} \times 100\ \% \qquad (3.3)$$

Where, M_a is the mass after complete ash (gram), and crucible M_b (gis the mass before ash and crucible (g) and M_c is the mass of the empty crucible (g).

3.3.3. Determination of volatile matter content

The volatile matter of bamboo powder was calculated by taking 5g of bamboo samples of the fiber was burnt in a crucible at a temperature of 650 °C for 10 minutes and permitted to cool in a desiccator (Nilsson, 2017). Based on a dry basis the volatile matter content was calculated by using equation (3.4).

$$\text{Volatile content} = \frac{M_i - M_f}{M_i} \times 100\ \% \qquad (3.4)$$

Where Mi and M_f were the initial and final mass of the sample (g), respectively.

3.3.4. Determination of fixed carbon content

The fixed carbon of the fiber was calculated by subtracting the sum of moisture content (%), ash content (%) and volatile matter (%) from 100 based on a dry basis matter. The fixed carbon is defined as the residue left after removing the volatile matter and the ash from the substance.

$$\text{Fixed Carbon \%} = 100 - (\text{moisture content} + \text{ash content} + \text{volatile matter}) \qquad (3.5)$$

3.4. Component characterization of analysis of bamboo

3.4.1. Determination of Hemicellulose content

One gram of extracted dried bamboo was transferred into a 250 mL flask and then 150 mL of 0.5 moles NaOH was added. The mixture was boiled for 3.00 h with distilled water. It was filtered after cooling through vacuum filtration and washed until neutral pH. The residue was dried to a constant weight at 105℃ in an oven and the amount was calculated using the equation (3.6).

$$\text{Hemicellulose content} = M_a - M_b \qquad (3.6)$$

Where M_a and M_b, were initial and final mass of sample (g) respectively.

3.4.2. Determination of Lignin

The lignin content was determined using TAPPI norm T222 om-88. In brief, 1 g of bamboo powder was mixed with 15 mL of H_2SO_4 and left at room temperature for 2 hours.The mixture was then boiled for 3hours before centrifugation to extract the insoluble lignin, and 500 mL of distilled water was added (Song et al., 2019). The obtained lignin was oven-dried and weighed. The amount of lignin was calculated using equation (3.7).

$$\text{Lignin content (\%)} = \frac{M_f}{M_i} \times 100\% \qquad (3.7)$$

Where M_i and M_f were initial and final mass of sample (g) respectively.

3.4.3. Determination of Cellulose content

The cellulose content was calculated by subtracting the sum of hemicellulose and lignin from a 100 and taking into account the other lignocellulose constituents constants (Ayeni et al., 2015).

$$\text{Cellulose \%} = 100 - (\text{Hemicellulose} + \text{lignin}) \qquad (3.8)$$

3.5. Characterization of the nanocellulose

3.5.1. Particle size

The experiment analysis was performed by dynamic light scattering (DLS) utilizing the Zetasizer Nano instrument (Malvern, UK) at Addis Ababa Science and Technology University (Ethiopia). The nanocellulose dispersion in water was measured with 0.1wt. % concentration, 0.8872 CP viscosities, 344 record numbers, 0.01 material absorption, 1.59 material refractive index, and 1.330 dispersion reflective indices. DLS measurements provide the mean particle size (Z-average) and particle size distribution (Poly-dispersity index, POI) in suspension. The techniques use light scattering by diffusing particles that move in Brownian motion, and the analysis is based on the particle diffusion rate in the fluid. Because of the non-spherical properties of nanocellulose, particle size was determined by correlating the diffusion coefficient of the particles in dispersion using the Stokes-Einstein relation (hydrodynamic diameter) that the particles in dispersion undergo Brownian motion (Ramos et al., 2016).

3.5.2. Crystallinity

XRD analysis was performed to predict the crystallinity index value of the bamboo powder and isolated nanocellulose samples for a better understanding of the crystalline and amorphous regions of the product**.** The experimental analysis was conducted at Jimma Institute of Technology, School of Material Science and Engineering (, Ethiopia). DW-XRD-Y7000 instrument was used with Cu Kα radiation beam operated at 30kv and 25mA at 25℃. The scanning range was 2θ = 10 to 70 degrees at a scanning speed of 0.03°/s.

Mechanical characteristics of nanofiber improved as crystallinity increased. To conduct the XRD analysis the samples need to be dried in advance, which was affect the results as the structure changes upon drying. The diffraction peaks are a clear indication of the highest degree of crystallinity in the fiber structure (Lani et al., 2014).

It was calculated as:

$$\text{Crystallinity} = \frac{I_{200} - I_{am}}{I_{200}} \times 100\% \qquad (3.9)$$

Where, I_{200} is the height of the 200 peak, which represents both crystalline and amorphous material and I_{am} representing the amorphous.

The crystallite sizes of the lattice planes were estimated by using the Scherer equation (Mandal & Chakrabarty, 2011).

$$D = \frac{K\lambda}{\beta \mathrm{Cos}\theta} \qquad (3.10)$$

Where

D is crystallite size

K is Scherer constant (shape factor) = 0.9

λ , is wavelength = 0.154 nm

β = Full width at half maximum (FWHM) in radians.

3.5.3. Functional group analysis

Fourier transform infrared spectroscopy (FTIR) analysis was used to determine the chemical composition of raw bamboo and nanocellulose suspension. FTIR spectra were acquired at Jimma Institute of Technology’s, School of Material Science and Engineering using a Perkin Elmer FTIR spectrometer (Perkin Elmer spectrum Two, China) (Ethiopia). The measurement was used to compare the state of nanocellulose to that of raw bamboo, as well as to investigate the removal efficiency of other components (lignin and hemicellulose) during pretreatment steps with acidified sodium chlorite and NaOH solution. The examination was performed in transmittance mode at a wave number range of 4000–400 cm^{-1} using potassium bromide, since KBr, has a transmittance of 100 % in this range rather than others ionic cyrstals (Kandhola et al., 2020).

3.6. Statistical analysis of the experimental data

Experimental data analysis was done using Design-Expert software version 11.1.2.06 (STAT-EASE Inc., Minneapolis, USA).

The experimental design selected for this study is response surface methodology, three factors, central composite design (CCD), three levels, and the response variable measured was the yield (Y%) of nanocellulose (Mefteh et al., 2019). The effect of reaction time, temperature and acid concentration were analyzed as they are the three factors that mostly affect the nanocellulose yield by comparing with the other factors (Beyan et al., 2021).

RSM is a set of mathematical and statistical approaches for modeling and analysis in situations where multiple variables influence the desired result. It is used in such a way that, it determines the factor levels that simultaneously satisfy a set of the desired specification; determine the optimum combination of factors that yield the desired response and describes the response near the optimum; determine how a specific response is affected by changes in the level of the factors over the specified levels of interest and find conditions for process stability.

Therefore, it is considered an appropriate approach to optimize a process with one or more responses. Multiple regression equations, which may be used to estimate the expected values of the performance level for any factor levels, express the relationship between the factors and the performance measures (Carley, 2014). For this study, a second-order model is utilized to find a suitable prediction for the functional relationship between independent variables and the response. The selected parameters were based on a preliminary study (lin et al. 2019).

Table 3.2: Levels of three independent variables

Variables	Factor coding	Units	Low level	Center	High level	-α	+α
Temperature	A	°C	40	50	60	30	70
Time	B	Minutes	30	45	60	15	75
H_2SO_4 Con	C	(Wt. %/v)	45	55	65	35	75

Using the equation (3.11), the total number of experiments (N) was calculated. Eight factorial points, six axial points, and six center points were used in the studies as three separate process variables.

$$N = 2^n + 2n + n_c = 2^3 + 2*3 + 6 = 20 \qquad (3.11)$$

Where **N** is the total number of experiments required, **n** is the number of variables and **n_c** is the number of replicates. So, the central composite design was performed to investigate the effect of these parameters. The second-order polynomial equation in its generic form is:

$$Y = \beta_o + \sum \beta_i x_i + \sum \beta_{ii} x^2 + \sum \beta_{ij} x_i x_j \qquad (3.12)$$

Where:

Y is the predicted response, X_i and X_j are independent factors, β_0 is the model intercept, β_i is the linear coefficient, β_{ii} is the quadratic coefficient and β_{ij} is the interaction coefficient.

3.7. Synthesis of nanocellulose/PVA composite

The composites were synthesized by varying the nanocellulose loading ratio and reinforcing with polyvinyl alcohol, and the change in PVA properties after reinforcing with nanocellulose. Polyvinyl alcohol with a degree of hydrolysis of 90% and a molecular weight of 88 kg/mole with 96.7% purity for use as a matrix in the preparation of the composite. Glycerol was used on the surface of the Petri dish to prevent the film from sticking to it during drying. Four different composites were prepared by using the nanocellulose suspension (2 %, 4%, 6%, and 8 %) with different ratios. The solvent casting method was used to create the nanocellulose−PVA composite films. The aqueous solution of PVA was prepared by dissolving 5g of PVA powder in distilled water at 80°C and keeping it in a water bath for 2 hours with mechanical stirring (500 rpm) until the PVA was fully dissolved and a clear jelly-like solution formed. The solution was cooled to room temperature while stirring, and different amounts of nanocellulose suspensions (2 %, 4%, 6%, and 8%) were added to the PVA solution while stirring continuously until the solution became viscous. using a sonicator in water for 15 minutes to obtain a homogeneous solution and to avoid agglomeration of nanocellulose in the PVA matrix. Then, different films were cast on the petri dish based on their perspective ratio and left for 3 days to dry. The films were then stored in plastic bags for further characterization and comparison (Aslam et al., 2018).

Table 3.3: Synthesis ratio for the preparation of Nanocomposite films

Ratios	Amount of PVA (%)	Amount of NC Used (%)	Glycerol (ml)
PVA/0%NC	100	0	3
PVA/2% NC	98	2	3
PVA/4% NC	96	4	3
PVA/6% NC	94	6	3
PVA/8% NC	92	8	3

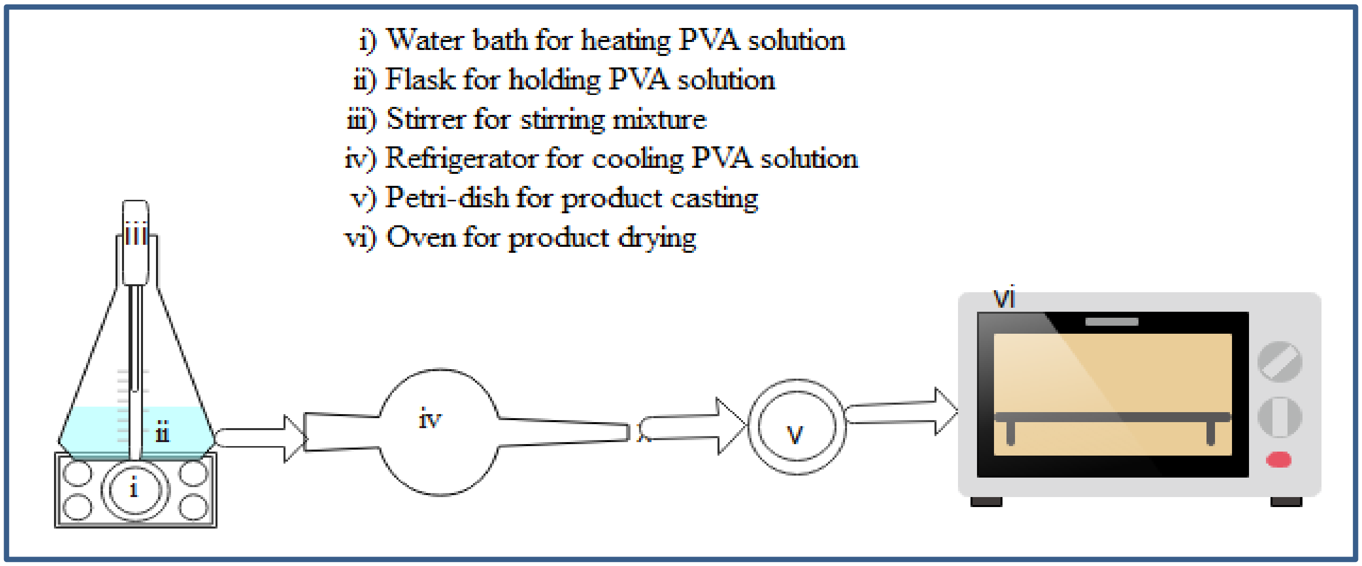


Figure 3.3: Experimental set up for PVA/NC composite film

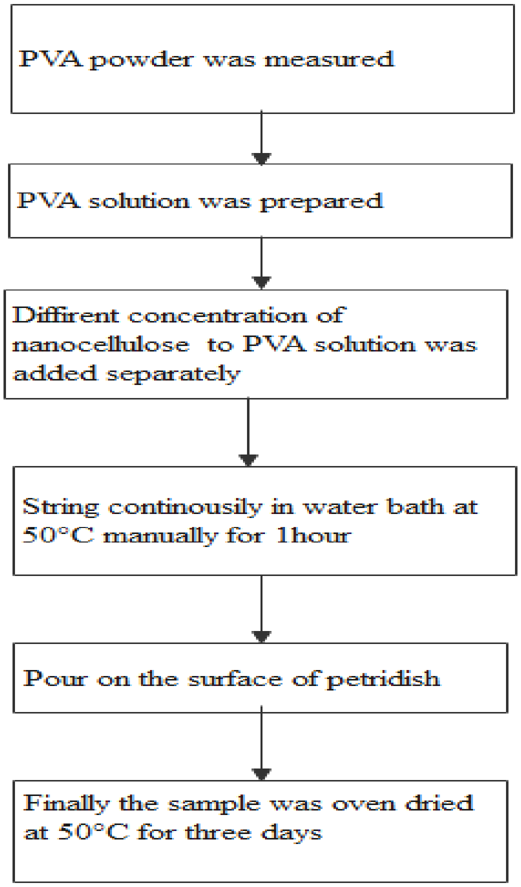


Figure 3.4: Preparation procedure for PVA/NC composite film

3.8. Characterization of nanocellulose/PVA composite

3.8.1. Water absorption test

The characterization was accustomed to investigating the effectiveness of the synthesized nanocomposite water absorption capacity. This was done by cutting the composites with sharp materials and oven-dried at 105°C for 24 hrs and then cooling them at room temperature (Yoon et al., 2012). Finally, after the sample was cooled, the water absorption was calculated based on the equation (3.13).

$$\text{Water absorption } = \frac{M_a - M_b}{M_b} \times 100\% \tag{3.13}$$

Where M_a and M_b are the mass of wet samples and the initial samples respectively (g).

3.8.2. Thermal stability performance

Thermogravimetry analysis (TGA) continually monitors a sample's weight as a function of temperature. It is also regarded as a valuable tool for investigating the heat stability of polymer nanocomposite (Salh & Raswl, 2018). The fibers are chopped into fine portions and evaluated using an aluminum pan at a temperature range of room temperature to 700°C under an inert environment of nitrogen at a flow rate of 100ml/min and a heating rate of 10°C/min to prevent any thermo-oxidative degradation (Mora, 2008). The experimental analysis was done at the Bahirdar institute of Technology (Ethiopia).

3.8.3. Mechanical tests

The mechanical properties of nanocomposite were investigated as a result of the interaction between nanocellulose and the PVA matrix. The increases in mechanical qualities, such as tensile strength and elongation at break, were obtained by mechanical testing concerning reinforcement, which was carried out using mechanical testing machines that follow conventional procedures (Lani et al., 2014). It was calculated as:

$$\text{Tensile strength of the film} = \frac{\text{Maximum breaking load applied}}{\text{Cross-sectional area of the film}} \tag{3.14}$$

$$\text{Elongation at break} = \frac{(\text{Final length at break} - \text{initial length})}{\text{Initial length}} \tag{3.15}$$

4. RESULT AND DISCUSSION

4.1. Proximate analysis of highland bamboo

Based on the results of oven drying at 105°C for 2 hours, the moisture content of the bamboo sample was calculated. Finally, using equation 3.2, the moisture content was 8.5%, this result was comparable to a prior literature (Sirisomboon, 2017). The ash content of the bamboo samples was determined using the standard procedure specified in section (3.3), and the results were 2.25%, which was comparable to previously published data (Ayeni et al., 2015). Ash refers to the inorganic residue remaining after either ignition or complete oxidation of organic matter in Lignocellulosic biomass. The higher ash content may decrease the cellulose amount that reduces the yield of nanocelulose. The bamboo's volatile content was determined using equation (3.4), and the value obtained was 73.5%, which was acceptable to earlier research (Posom & Sirisomboon, 2017). Finally, based on equation (3.5), the fixed carbon content of bamboo fiber was estimated and the result was 16.5 %. From the different works of literature, it is reported that the fixed carbon content of bamboo was 10% to 18% (Sirisomboon, 2017). Therefore based on this analysis, Ethiopian highlands bamboo was the alternative raw materials for nanocellulose synthesis.

4.2. Component characterization of highland bamboo

The hemicellulose content of bamboo fiber was calculated based on equation (3.6) and yields 19.5%, from previously founded studies hemicellulose covers (15 to 20%) of the total plant component of bamboo. The lignin content of bamboo fiber was found to be 28.1 %. The lignin percentage of bamboo fiber varies between 20% and 30%, according to several studies (Mefteh et al., 2019). Synthesis of cellulose from plants with a high lignin content necessitates lengthy treatments that take more time and chemicals to remove the lignin. The amount of cellulose was calculated according to equation (3.8) and was 52.4 % (Yoon et al., 2012). The yield of nanocellulose products obtained from lignocellulosic materials was high due to the high cellulose content.

From Table 4.1, the findings of this study were discussed that, and it were found to be consistent with previous studies. The higher the ash level, the lower the cellulose content, which may reduce the yield of nanocellulose in the end. The amount of hemicellulose in a product has another effect since it absorbs more moisture, which affects the entire product. Following the removal of hemicellulose and lignin, the cellulose content was enhanced using a variety of methods.

Table 4.1: Proximate analysis and chemical compositions of high bamboo

Components	Results obtained (wt. %)	Literature (%)	References
Cellulose	52.4	30 to 55	(Yoon et al., 2012),(Khan et al., 2020) ,(Abdel-Halim,2014), (Rivai et al., 2021) and (Mugo, 2020)
Hemicellulose	19.5	15 to 20	
Lignin	28.1	20 to 30	
Ash	2.25	1.1 to 2.7	(Sirisomboon, 2017), (Osorio et al., 2018) and (Nilsson, 2017) (Ayeni et al., 2015)
Moisture	8.5	7.1 to 10.2	
Volatility	73.5	70 to 80	
Fixed carbon	16.5	10 to 18	

4.3. Synthesis of nanocellulose from bamboo

Purification, alkali treatment, and bleaching processes were used to isolate cellulose with white color. The color varied as the process progressed. The residues filtered at the beginning of the treatment were dark black, but after delignification, they were dark yellow. After the bleaching cycle was repeated, the product turned to white, and the residual water turned a light yellow color and at the initial stage, they're almost totally gone. The presence of lignin and hemicellulose in the fiber created the yellow tint. The absence of those colors implies that the series of therapy actions were effective.

Figure 4.1: Bleached and dried bamboo cellulose

The nanocellulose suspension was made from the obtained cellulose using sulfuric acid hydrolysis. The product was in the form of a suspension and was lighter in color than cellulose. A strong acid can dissolve the amorphous sections of fiber cellulose. Some variables influence the yield of nanocellulose. The reaction time, acid concentration, and reaction temperature are the parameters in question (Nilsson, 2017). Acid can spread through the fibers and dissolve the amorphous component faster than the crystalline component. Because dilute acids have a limited ability to dissolve amorphous, the suspensions were left with the amorphous components when the acid concentration was low. Since the acid dissolves the crystalline portion of the cellulose as it diffuses through the fibers, too long reaction times result in lower yields of nanocellulose suspensions (H. Xie et al., 2018).

As a result of the excessively short reaction periods, micro cellulose with greater diameters and amorphous sections was generated. The yield of nanocellulose is also influenced by the temperature of the reaction. Higher temperatures resulted in unpredictable and difficult reactions, and the suspension's color shifted from white to black in just a few minutes. The reaction times were too long at lower temperatures to produce nanocellulose suspensions. As a consequence of using design expert software and a response surface technique, the nanocellulose extraction was carried out at the ideal temperature, reaction duration, and acid concentration. Finally, using the experimental results, the yield of dried nanocellulose was calculated.

4.4. Statistical analysis of the experimental results

4.4.1. Experimental data

Following the completion of the laboratory experiments, the yield of nanocellulose was estimated and examined using design expert 11 software and discussed in Table 4.2.

Table 4.2: CCD matrix of independent variables used in RSM

Run Order	Standard Order	Temperature (°C)	Reaction Time (minute)	Sulfuric Acid Con (wt. %)	Yield (%) Actual	Yield (%) Predicted
1	7	40	60	65	43.40	42.90
2	20	50	45	55	40.01	40.77
3	16	50	45	55	42.50	40.77
4	2	60	30	45	41.90	42.62
5	17	50	45	55	41.25	40.77
6	14	50	45	75	28.50	28.56
7	15	50	45	55	40.50	40.77
8	6	60	30	65	27.75	28.91
9	12	50	75	55	37.90	38.40
10	4	60	60	45	39.10	39.82
11	3	40	60	45	38.80	37.86
12	13	50	45	35	37.50	37.22
13	11	50	15	55	36.50	35.79
14	18	50	45	55	40.10	40.77
15	9	30	45	55	37.01	37.96
16	8	60	60	65	27.50	27.44
17	19	50	45	55	40.50	40.77
18	1	40	30	45	33.50	33.77
19	10	70	45	55	32.50	31.34
20	5	40	30	65	38.00	37.49

4.4.2. Model summary statistics

The model suggested by software for this work was the Quadratic model. The application of RSM gives an empirical relationship between the response function and the independent variables.

Table 4.3: A suggested model for the design.

Source	Std. Dev.	R^2	Adjusted R^2	Predicted R^2	PRESS	
Linear	4.47	0.2818	0.1472	-0.1609	517.59	
2FI	3.33	0.6773	0.5284	0.3904	271.81	
Quadratic	**1.06**	**0.9746**	**0.9517**	**0.8581**	**63.27**	**Suggested**
Cubic	0.8714	0.9898	0.9676	0.9077	41.16	Aliased

4.4.3. Analysis of variance

The ANOVA results showed a perfect fit of the quadratic regression model (F-value of 42.61) ($p< 0.0001$) highly significant and there is only a 0.01% probability that the F value could occur due to noise. P-values less than 0.0500 indicate model terms are significant.

In this case, A, B, C, AB, AC, A^2, B^2, C^2 are significant model terms while BC is insignificant to the response. Values greater than 0.0500 indicate the model terms are not significant. The p-value of "Lack of Fit" for nanocellulose was found to be insignificant ($p > 0.3131$), thus indicating that the model was accurate for predicting the response. The results revealed that concentration has the most significant effect on nanocellulose synthesis.

Table 4.4: Analysis of variance (ANOVA) for Response Surface Quadratic Model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	434.53	9	48.28	42.61	< 0.0001	significant
A-temperature	43.79	1	43.79	38.64	< 0.0001	
B-reaction time	6.83	1	6.83	6.02	0.0340	
C-concentration	75.04	1	75.04	66.22	< 0.0001	
AB	23.63	1	23.63	20.85	0.0010	
AC	151.82	1	151.82	133.97	< 0.0001	
BC	0.8778	1	0.8778	0.7746	0.3994	
A^2	58.99	1	58.99	52.05	< 0.0001	
B^2	21.30	1	21.30	18.80	0.0015	
C^2	97.62	1	97.62	86.15	< 0.0001	
Residual	11.33	10	1.13			
Lack of Fit	6.95	5	1.39	1.58	0.3131	not significant
Pure Error	4.39	5	0.8772			
Cor Total	445.86	19				

4.4.4. Model adequacy

The correlation between experimental data and predicted responses was quantitatively estimated by regression coefficients R^2. The obtained values of R^2 and adjusted R^2 were 0.9746 and 0.9517 respectively, showing there is good agreement between the predicted values and experimental ones. The values of R^2 should be between 1 and 0. The R^2 value is closer to 1.0 demonstrating that the regression model equation provides a very accurate description of the experimental data, in which all the points are very close to the line of a perfect fit.

The same to that, in this study R^2 obtained, was 0.9746, which was close to1. The values of R^2 almost equal to 1.0 were indicated that a high correlation between the experimental and predicted data. The predicted R^2 of 0.8581 is in reasonable agreement with the adjusted R^2 of 0.9517 which means the difference is less than 0.2. Adequate precision measures the signal-to-noise ratio and a ratio greater than 4 is desirable. The ratio of 20.5368 indicates an adequate signal. The model can be used to navigate the design space.

Table 4.5: Model statics of the design

Std. Dev.	Mean	C.V. %	R^2	Adjusted R^2	Predicted R^2	Adeq Precision
1.06	37.24	2.86	0.9746	0.9517	0.8581	20.5368

From Figure 4.2, the normal probability plot indicates that the residuals are followed by the normal % probability distribution.

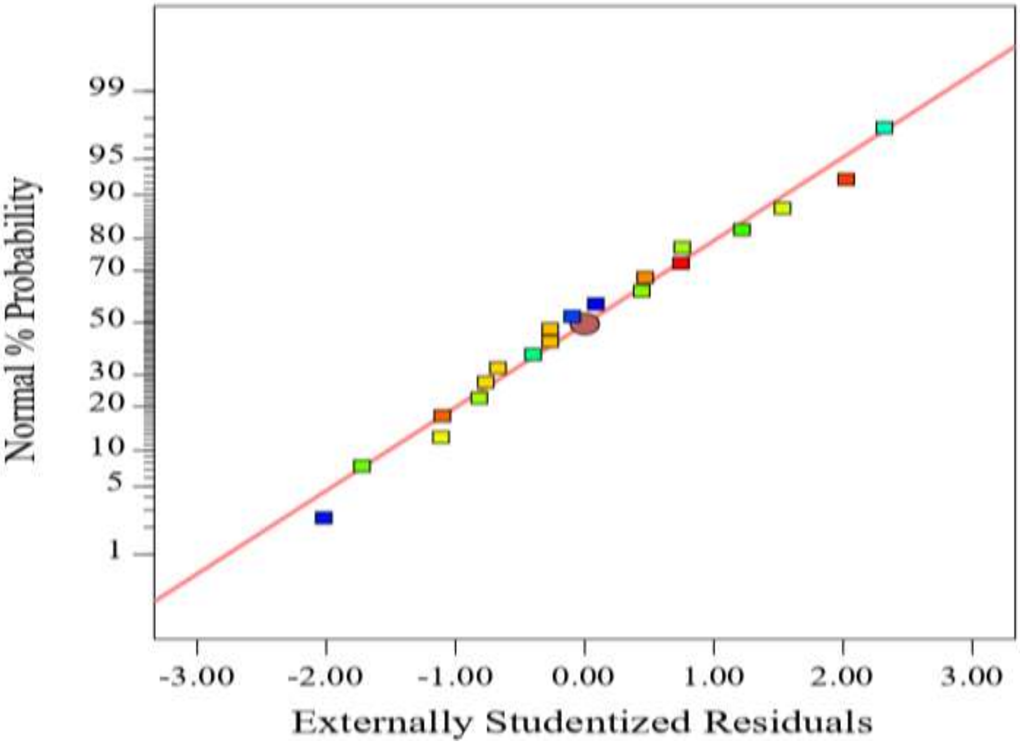


Figure 4.2: Normal plots of residuals for nanocellulose yield

If the model is accurate and the assumption is fulfilled, the residuals should be structured in fewer particular, they should be unrelated to any additional variable includes the predicted response. A plot of the residuals versus the increasing predicted response values test, the assumption of constant variance. The plot shows random scatter which justifies no need for any alteration to minimize personal error.

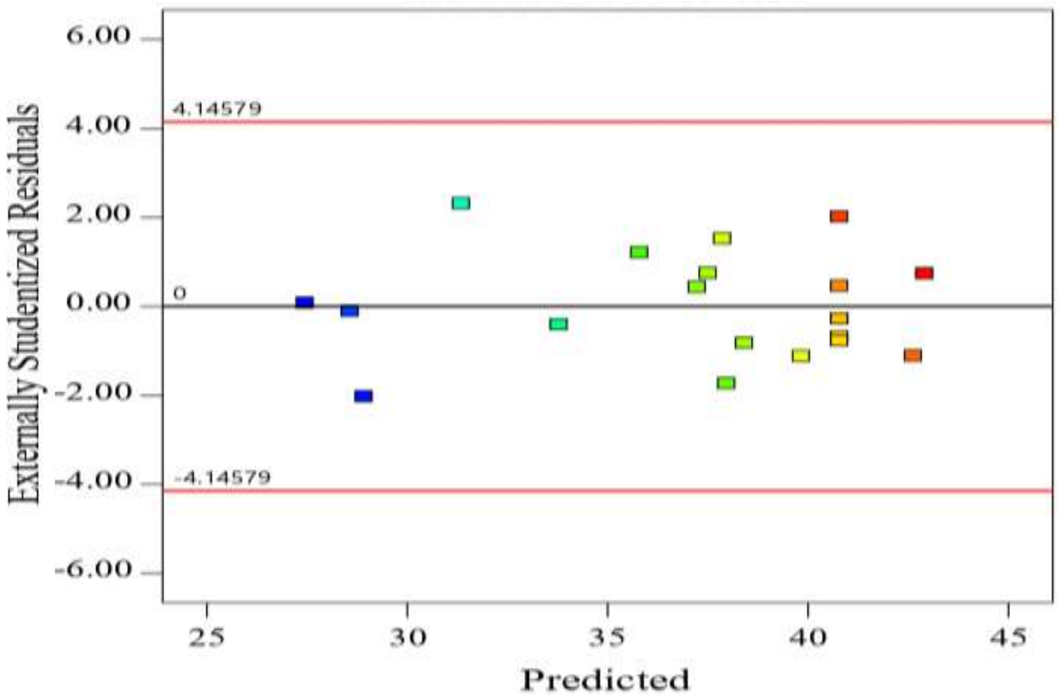


Figure 4.3: Plot of predicted versus residuals for the yield.

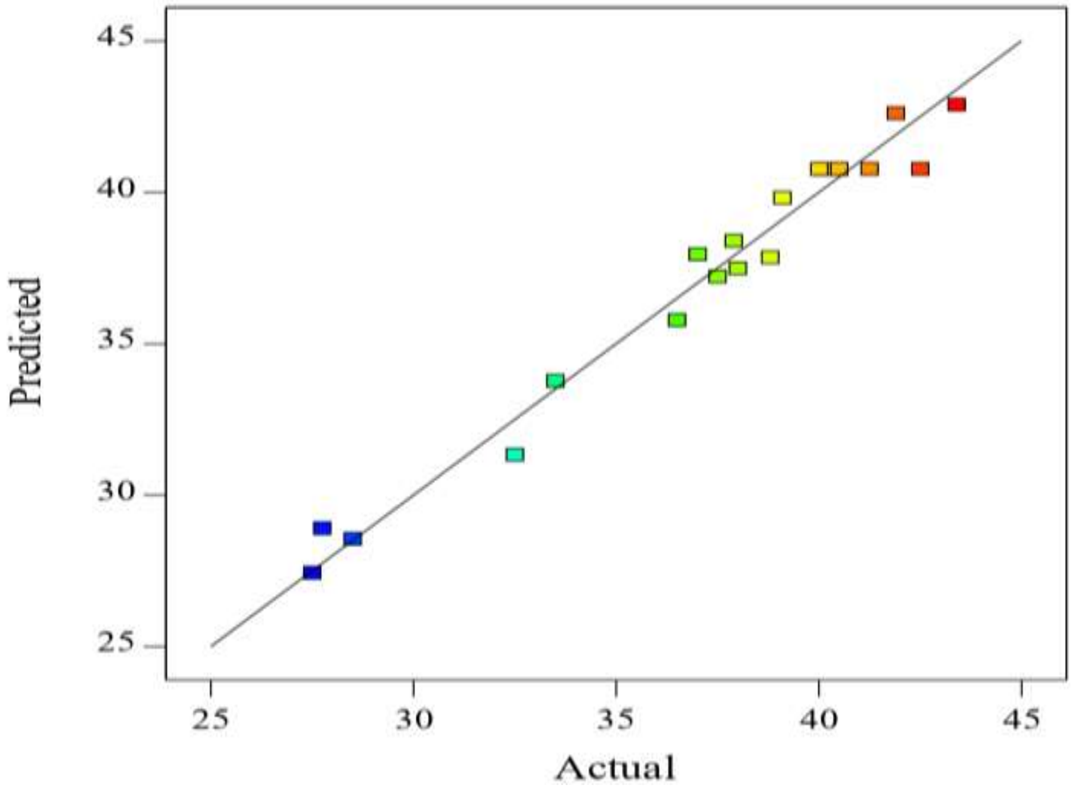


Figure 4.4: Plot of actual yield versus the predicted yield of nanocellulose

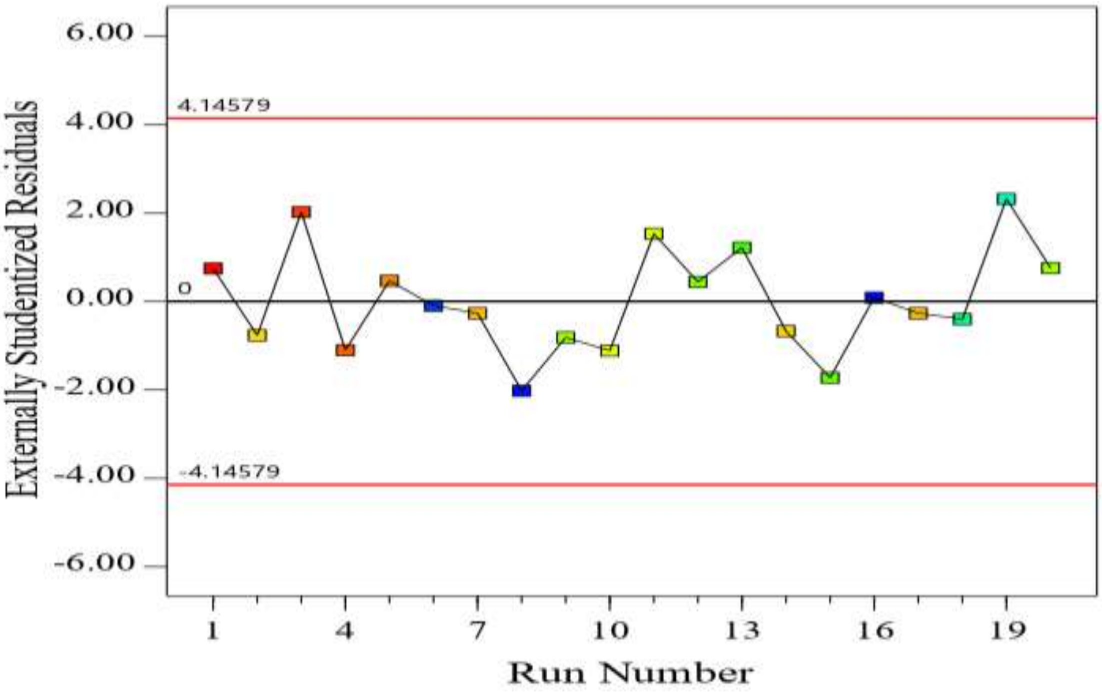


Figure 4.5: Plot of residual versus the run number of nanocellulose

4.4.6. Development of a model equation

The model equation that correlates the response to the acid hydrolysis process variables in terms of independent variables were obtained. From the model summary statistic section, a quadratic model is selected from other models because it has a high accuracy of fitting the data.

$$Y = +40.77 - 1.65*A + 0.6531*B - 2.17*C - 1.72AB - 4.36*AC - 1.53*A^2 - 0.9205*B^2 - 1.97*C^2 \qquad (4.1)$$

Where Y is yield, A is temperature, B is reaction time and C is acid concentration.

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

4.4.7. Effects of the significant variable’s on the nanocellulose yield

Sulfuric acid concentration, hydrolysis temperature, and reaction time all affected nanocellulose yield.

4.4.7.1. Effect of acid concentration on yield of nanocellulose

Figure 4.6 depicts the influence of acid concentration on nanocellulose yield. The acid concentration was inversely proportional to nanocellulose yield and indicated that increasing the value of acidic concentration would result in decreasing the yield (Eq.4.1).This is due to acid penetration into the amorphous component of cellulose, which improves cellulose conversion to NCs. The yield gradually decreases as the acid concentration of 55 wt.% and subsequently decreases as the acid concentration exceeded 55%wt, because of the cellulose over-degradation to undesirable compounds.

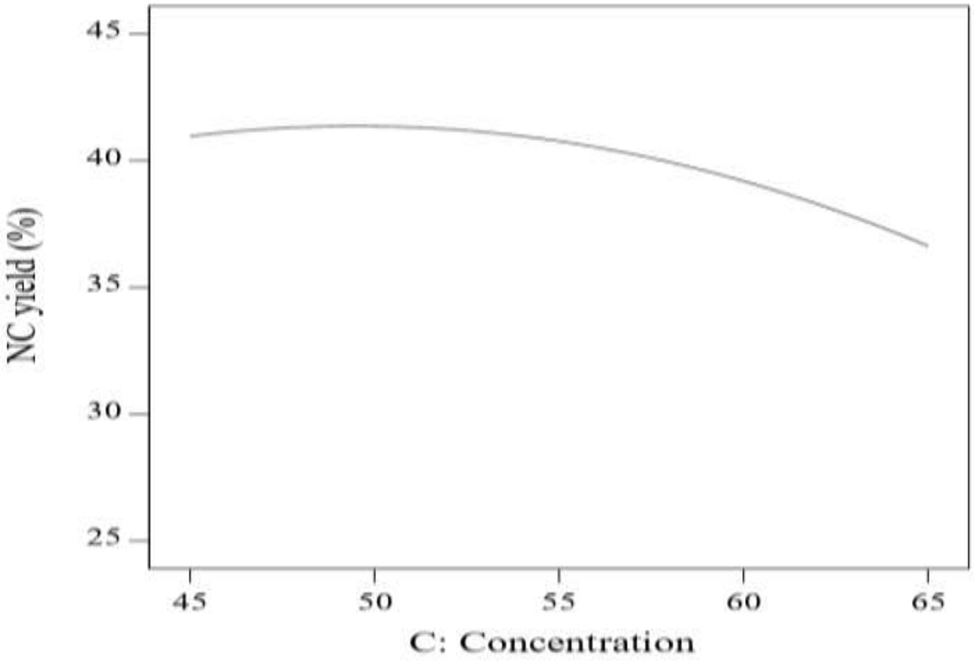


Figure 4.6: Effect of acid concentration on the yield of NCs

4.4.7.2. Effects of temperature on yield of nanocellulose

As indicated in Figure 4.7, the yield was higher at lower temperatures and decreases with increasing temperatures, because increasing the temperature increases the hydrolysis process at the start of the reaction.

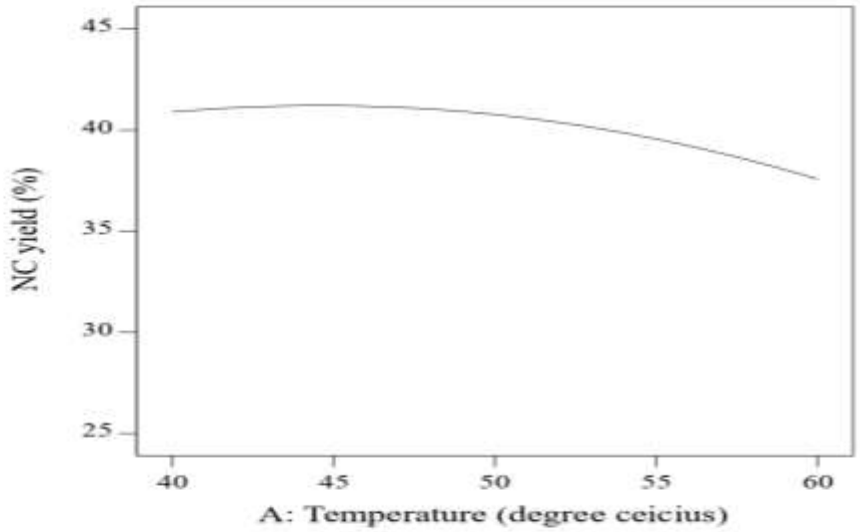


Figure 4.7: Effects of temperature on yield of NCs

4.4.7.3. Effect of hydrolysis time on the yield of nanocellulose

Figure 4.8 depicts the influence of hydrolysis time on nanocellulose yield and it was discovered that the minimum value of yield was attained at the lowest level of time and rose with increasing until optimum values were achieved. As the running time increases, the cellulose degrades further, resulting in a decrease in NC yield.

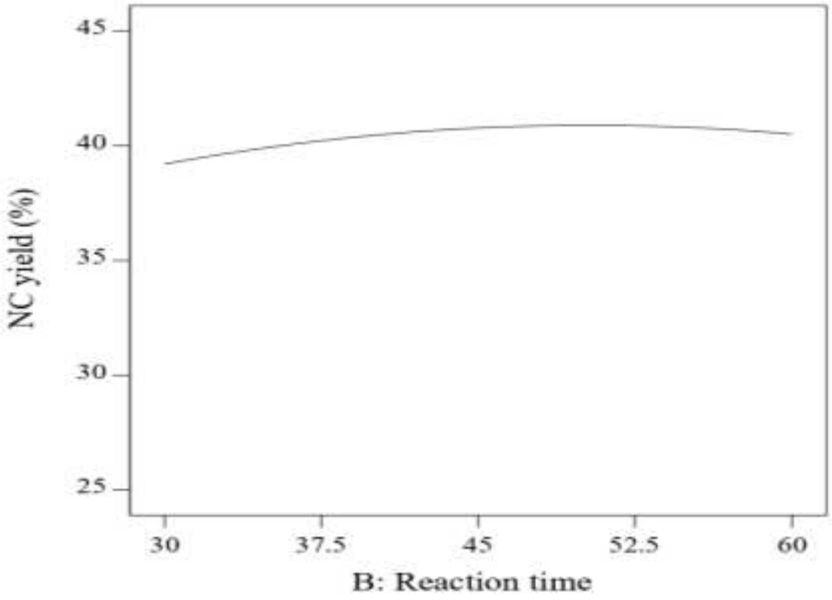


Figure 4.8: Effect of hydrolysis time on the yield of NCs

4.4.8. The interaction effects of significant factors on nanocellulose yield

4.4.8.1. Effects of temperature and acid concentration on the yield of nanocellulose

Figure 4.9 depicts the effect of temperature and acid concentration on the yield of nanocellulose at a constant hydrolysis period of 45 minutes. According to equation (4.1), the interaction between temperature and acid concentration has a negative effect (-4.36) on NCs yield after its optimal point. It was discovered that at higher temperatures, a smaller value of acid concentration yields a higher yield, which decreases with rising temperature. The fluctuation of acid concentration, on the other hand, has no effect at higher temperatures. It was also revealed that the influence of acid concentration change on yield was greater at the lower temperature than at the higher temperature. Even though both process variables influence nanocellulose production, acid concentration has a greater impact than temperature. Similar works was confirmed by (Gallardo-Sánchez et al., 2021).

4.4.8.2. Effect of temperature and time on the yield of NCs

Figure 4.9 shows the influence of temperature and time on the yield of nanocellulose when the acid content was fixed. Generally, the influence of temperature and time on the yield of nanocellulose when the acid content was fixed at 55wt%, larger yields of nanocellulose were achieved at lower temperatures and longer hydrolysis times. By maintaining the acid concentration constant, the graph revealed an inversely proportionate relationship between reaction time and mixing temperature on the yield of nanocellulose (Eq.4.1) and similar works confirmed by (Gallardo-Sánchez et al., 2021).

Mixing temperature, reaction time, and Sulfuric acid concentration were the significant parameters on the synthesis of NCs during the acid hydrolysis process. Reaction time affects various NCs characterization such as crystallinity index, size, and most of all, the yield. This is due to as reaction time increases, some hydrolyze crystalline surfaces will, in turn, decrease the yield and as time is lower, the polymerization rate will be high due to undispersed fiber, and also the obtained nanocellulose will have an amorphous structure and larger diameter. According to different previous studied data, reaction time varies from 30 min to 4 hours (Julie Chandra et al., 2016). The concentration of sulfuric acid has a potential effect on synthesizing of NCs. Sulfuric acid concentration can range from 40 to 60 wt. %, and it has been widely used for the isolation of nanocellulose from residues of agriculture. The temperature can range from 25 to 70°C. At low temperatures, most of the process reactions take a long time to get the desired yield and at high temperatures, the reaction is hard to control.

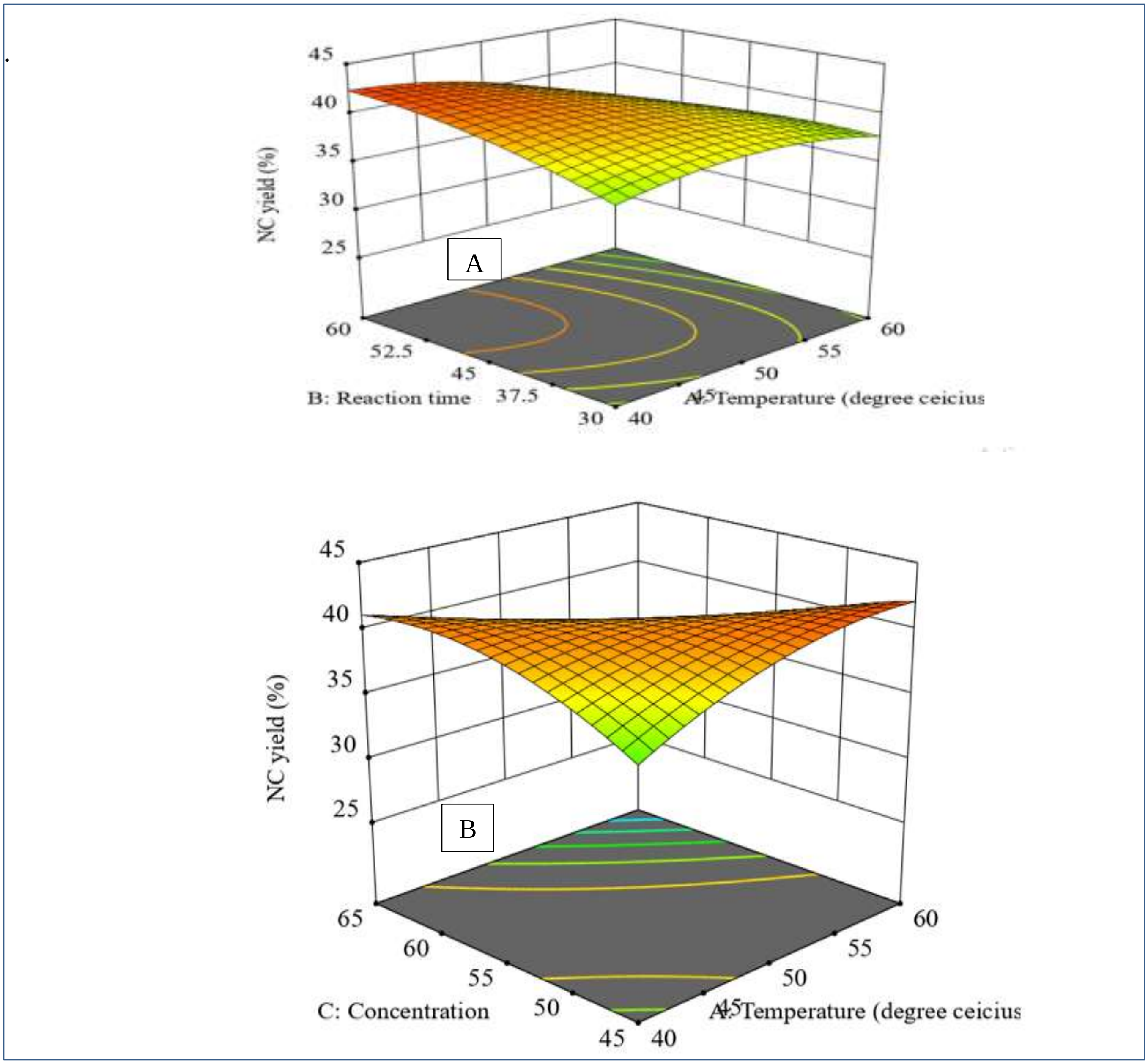


Figure 4.9: A) Interaction effect of temperature and time on the yield of NCs and B) Interaction effects of temperature and acid concentration on the yield of NCs.

4.4.10. Optimization of process parameters for acid hydrolysis

According to the optimization principle, to maximize the economic benefit by minimizing process cost, the process variables should be set to their value in range as much as possible, and the response variable nanocellulose yield was set to maximum.

To optimize the effects of process parameters (acid concentration, reaction temperature, and hydrolysis time) on the nanocelulose yield, a multiple response method called desirability function was used.

Table 4.6: Constraints for the factors and responses in numerical optimization

Parameters	Goal	Lower Limit	Upper Limit
Temperature	in range	40	60
Time	in range	30	60
Acid concentration	in range	45	65
NCs Yield	maximize	27.5	43.4

The maximum yield of nanocellulose 43.4% was obtained at the interaction parameter of temperature (40°C), time (60min), and acid concentration (65 wt. %), as shown in Table 4.6. This demonstrates that the predicted values of the NC yield agreed with the experimental results (Santos et al., 2013). The yield of nanocellulose was optimized to 43.155 % using the response optimization technique used in this study, at a desirable parameter interaction of acid concentration (61.404 wt. %), reaction time (60.00 min), and temperature (40°C).

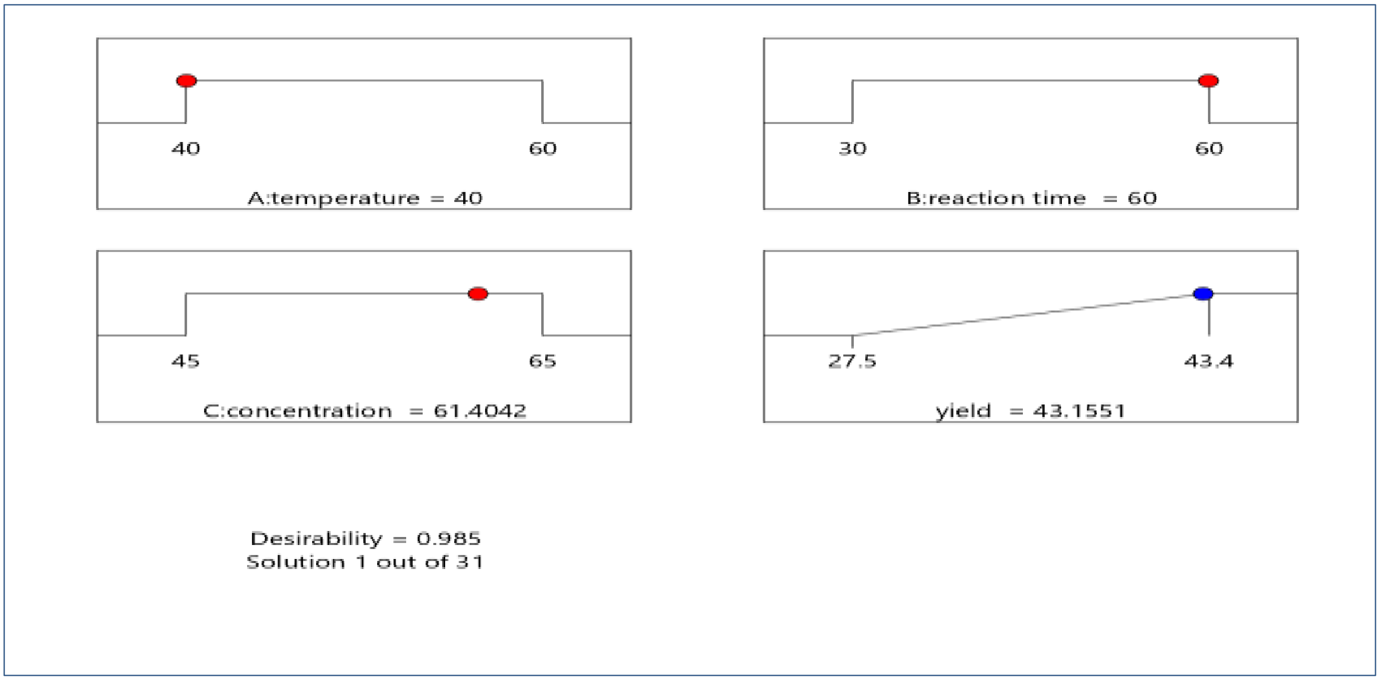


Figure 4.10: Ramps numerical optimization of parameters and response

4.4.11. Model Validation

The optimum points were acid concentration (61.404 wt. %), reaction time (60.00 min), and temperature (40°C). Quadruplicate experiments were conducted at the above specified optimum process conditions predicted by the model to validate the optimum conditions predicted by the response surface methodology model results. At this condition, the mean percentage of yield obtained was 42.75% as shown in Table 4.7 and which was then related to the data obtained from optimization analysis using the desirability function.

Table 4.7: Result of optimization and model validation

Number	Concentration (Wt. %)	Temperature (℃)	Time (min)	Yield (%)
Predicated value	61.404	40.00	60.00	43.15
Experimental value	61.404	40.00	60.00	42.75

The mean yield percentage obtained by quadruplicate the experiments was 42.75, which was not significantly different from the predicted value of 43.15%. The optimal conditions for percent yield are acid concentration (61.404 wt. %), reaction time (60.00min), and temperature (40°C). As a result, the model was valid and capable of predicting the maximum nanocellulose yield, indicating that numerical optimization can be considered an optimal value because the predicted value was close enough to the experimental value.

4.5. Characterization of nanocellulose

The physicochemical characterization of nanocellulose suspension was carried out at optimized conditions to verify the quality.

4.5.1. Functional group analysis

FTIR spectroscopy was utilized to show that lignin and hemicellulose were reduced during the cellulose isolation process by analyzing its functional groups. The FTIR results for bamboo fibers and nanocellulose are shown in figure 4.11. The appearance of O–H and C–H stretching vibration peaks 3405cm^{-1} and 2900 cm^{-1} respectively, and represents the cellulose content in both samples and the hydrophilic tendency of chips. The peak of O–H stretching vibration in the NCs has stronger transmittance compared to bamboo samples, which became narrower after the various treatments due to the removal of some of the amorphous components, with bleaching and acid hydrolysis procedures.

The other peak, which appears around 1728 cm^{-1}, corresponds to a C=O bond, which is normally found in ester linkages in hemicellulose and lignin (Han et al., 2016). This was reduced and nearly eliminated after the treatment process. The aromatic ring found in lignin is indicated by the peaks observed around 1600-1700 cm^{-1}.

At the wavenumber of 1644 cm^{-1}, corresponds to a C=C bond stretching of both samples was observed and indicative of the presence of lignin. The removal of some parts of the amorphous region is supported by the peak reduction of CH2 bonding at the C_6 in the cyclic chain of glucose with wavenumber 1320 cm^{-1} and the peak at 1050 cm^{-1} is associated with C-O Alkyl-substituted ether stretching vibrations. The small peak at 1108 cm^{-1} in the spectrum of nanocellulose is related to S = O vibration, due to the esterification reaction which occurred in the hydrolysis process. The peak around 900 cm^{-1} is associated with cellulosic -glycosidic linkages, which is characteristic of β-glycosidic linkages between monomers of cellulose. The FTIR spectra showed that the acid hydrolysis treatment did not affect the chemical structure of the nanocellulose synthesized from bamboo (Wijaya et al., 2019).

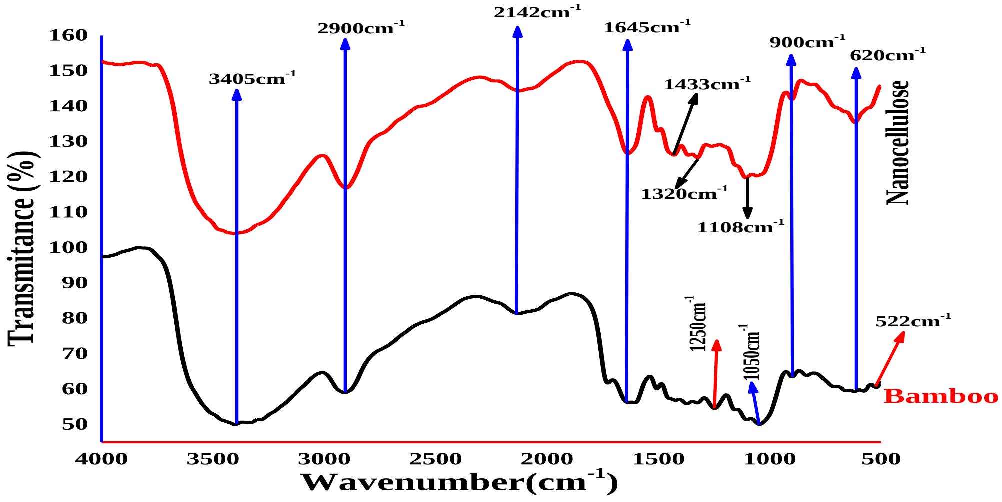


Figure 4.11: FTIR spectra for Bamboo powder and Nano-cellulose

4.5.2. Particle size

The particle size results derived by DLS were given in Figure (4.18) and Table (4.8) shows that the peak obtained was Unimodal and the particle size distribution was in a range of 60 nm to 110nm. The Z-average particle size given by the instrument was 106.5 nm and this shows that the obtained nanocellulose suspension from experimental work by acid hydrolysis of cellulose is in the nanometer scale. Z-average is the mean particle size as weighted by the intensity and hydrodynamic size of the ensemble collection of the particle measured by dynamic light scattering. The nanocellulose suspension uniformity was described by polydispersity index (PDI).

The higher poly-dispersity value shows the uniformity of the sample was less and the lower values indices the sample was uniform and free of agglomeration. Generally, samples with a PDI of less than 0.1 are mono dispersion, and the unimodal peak is produced. Poly dispersed samples with a PDI greater than 0.1 can produce bimodal or tri-modal peaks. This product got a PDI of 0.0632, which is less than 0.1, indicating that the nanocellulose suspension was uniform.

Table 4.8: Z-Average particle size from DLS

		Size (d. nm)	% Intensity	St Dev (d. nm)
Z-average (d.nm) : 106.5	Peak 1	79.64	100.0	2.595
PDI : 0.0632	Peak 2	0.000	0.0	0.000
Result quality : Good	Peak 3	0.000	0.0	0.000

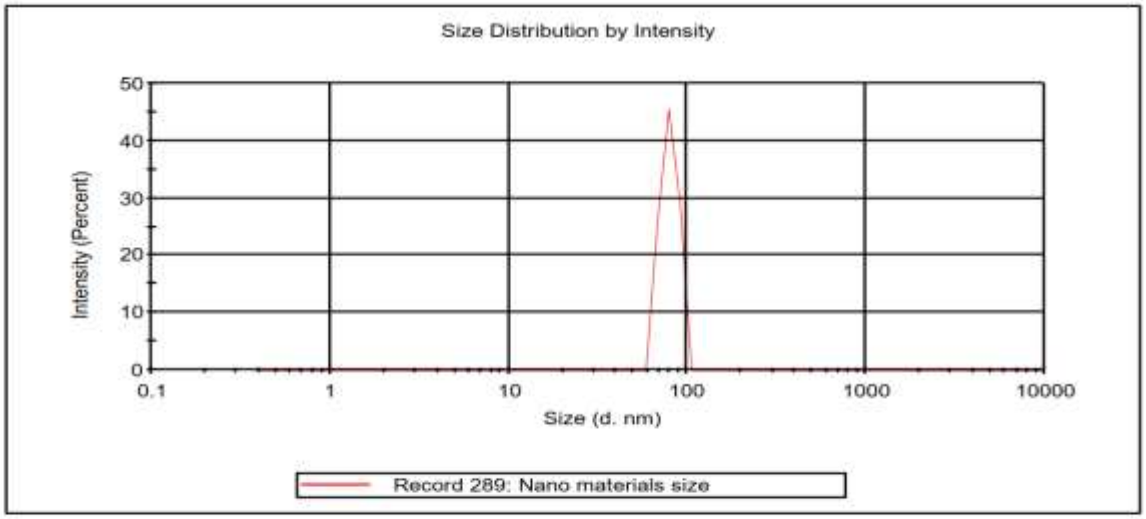


Figure 4.12: Particle size distribution

The particle size distribution, Z-average particle size, and Poly-dispersity index (PDI) of nanocellulose obtained in this work show that the product obtained was in the nanometer range and also in an acceptable range with the nanocellulose obtained by other researchers. Table 4.9 shows the average particle size reported by different researchers and compared with this study.

Table 4.9**:** Comparison of average particle size with literature

Raw material	Average particle size (nm)	Reference
Bagasse	240	(Sofla et al., 2016)
Cotton linter	179.3	(Morais et al., 2013)
Bamboo shoots	170.67	(Jara et al., 2020)
Enset fiber	66	(Beyan et al., 2021)
Commercial MCC	115	(Zhou et al., 2012)
Cotton fiber	235	(Heriberto et al., 2015)
Sisal fiber	30.9	(Mora, 2008)
Rice hulls	100	(Nascimento et al.,2016)
Corn cob	70	(Ditzel et al., 2017)
rayon fiber	348	(Dp, 2016)
Bamboo	106.5	Measured

4.5.3. Crystallinity index analysis

The crystalline index of raw bamboo and nanocellulose was determined using X-ray diffraction patterns. The crystallinity index (CrI) is the ratio of crystalline to amorphous cellulose regions (Yoon Yee et al., 2017). The interactions between hydrogen and Van der Waals forces between adjacent molecules cause nanocellulose crystallinity. X-ray diffraction (Figure 4.13) was used to see if the crystallinity of the material had changed and depicts the diffraction peak for raw bamboo, which became sharper and narrower after chemical treatments (alkaline treatment). The peak of nanocellulose, on the other hand, was sharper and narrower. This indicates that the cellulose content and degree of crystallinity were increased and that chemical treatments have an effect on cellulose crystallinity (Thakur et al.,., 2020).

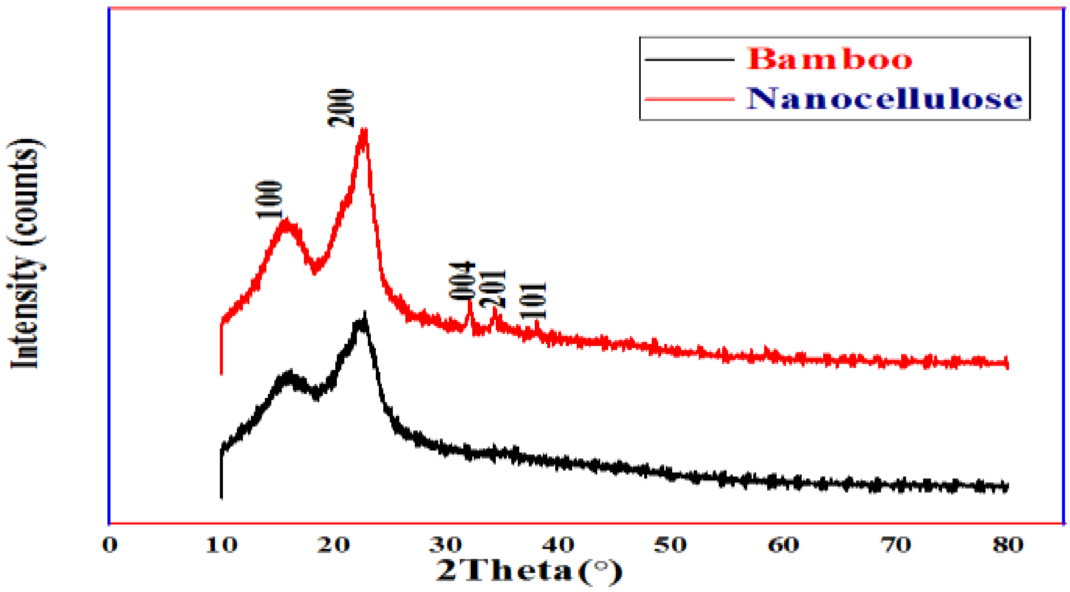


Figure 4.13: X-ray diffraction of raw bamboo and Nano-cellulose

The amorphous parts of the raw bamboo were removed by alkaline, bleaching, and acid hydrolysis treatments, as shown in Figure 4.13, giving nanocellulose a sharp and large peak. All have the particularity that they show a peak of around 2θ= 22.28 degrees. For raw bamboo and nanocellulose, the crystallinity index (CrI) determined using equation (3.11) was 44.60 and 74.07% respectively. The reduction and removal of amorphous cellulosic compounds induced by the alkali and bleaching treatments used in the purification process can explain why nanocellulose has a higher CrI value than raw bamboo. Because of the higher crystallinity of such nanostructures compared to raw fibers, the acid treatment results in narrower, sharper peaks for the nanocellulose.

Table 4.10: Crystallinity index of bamboo and nanocellulose

Samples	2θ (amorphous)(°)		2θ (002) (°)		Crystallinity index (CrI) (%)
	Degree	Intensity (I_{am})	Degree	Intensity (I_{002})	
Bamboo	18.23	957.7	22.57	1729.64	44.60
NCs	18.35	700.56	22.28	2700.73	74.07

Amorphous regions in the cellulose structure were removed and the crystallinity index is increased after alkali treatments and the removal of hemicellulose and lignin present in raw bamboo, as well as acidic hydrolysis by sulfuric acid. The crystallinity index was raised from 44.60 to 74.07% after acidic hydrolysis. The removal of hemicellulose and lignin, which exist, in amorphous regions, resulted in an increase in the crystallinity index for nanocellulose. The degree of crystallinity is affected by the raw material source, sample purification time, and hydrolysis conditions. There are 5 peaks in Figure 4.13, and the crystallite size was calculated by using the equation (3.12) for nanocellulose is shown in Table 4.11.

Table 4.11: Peak data list and average crystallite size for Nanocellulose

Peak No.	2θ (degree)	d(A)	FWHM (Counts)	Intensity (Counts)	Crystallite Size (nm)	Integrated Area	Total area
1.	15.759	5.57580	0.0787	1506	26.91	4647	1829613
2.	22.864	3.88967	0.3149	2320	17.92	4245	
3.	32.174	2.78241	0.1968	753	43.90	13302	
4.	34.290	2.61517	0.2755	630	31.50	1803174	
5.	38.117	2.36092	0.1574	484	55.78	4245	

4.6. PVA-Nanocellulose composite films

The composite films of PVA reinforced with nanocellulose of different ratios and neat PVA were presented in Figure 4.14. The films obtained were flexible and transparent.

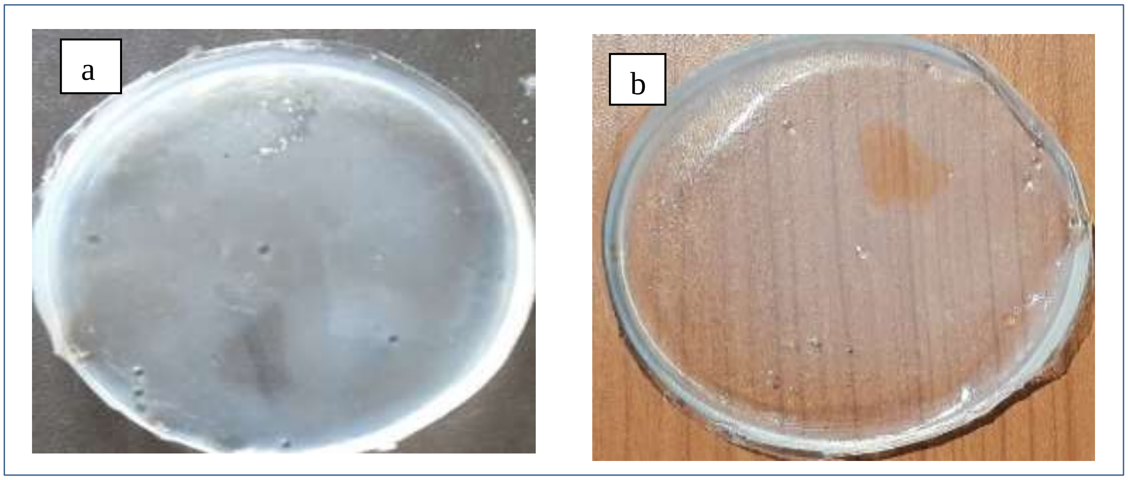


Figure 4.14: a) PVA/6%NC b) Neat PVA composite films

4.7. Characterization of PVA-nanocellulose composite films

4.7.1. Water absorption test

Based on the equation (3.13), the water absorption of the neat PVA and reinforced nanocellulose-PVA was calculated.

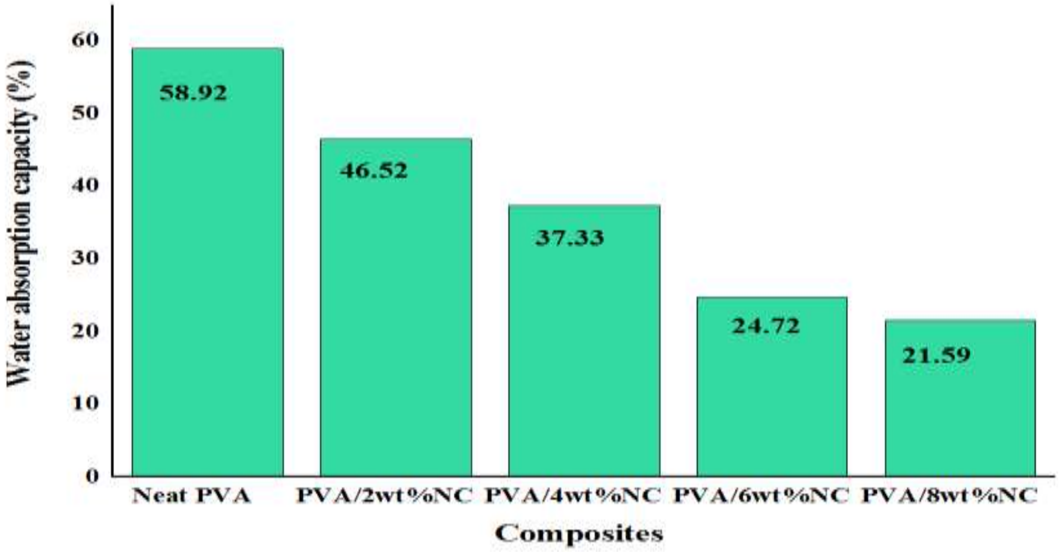


Figure 4.15: Water absorption result

Increased nanocellulose content in the PVA matrix limits water intake capacity, as shown in Figure 4.15. This could be the strong network bond formed due to nanocellulose reinforcement inhibiting water from freely entering composite molecules. The stiffness performance of polyvinyl alcohols matrix improves the weight percentages of nanocellulose in matrix increases, resulting in a decrease in water intake. The comparative analysis of the neat polyvinyl alcohols and reinforced with nanocellulose shows that reinforced PVA had lower water absorption than neat PVA due to nanocellulose reinforcement used was effectively lowering water abrasion and neat PVA absorbs more water. These variations demonstrate that nanocellulose or fiber reinforcement reduces the water absorption capacity of PVA polymers. This decrease in water absorption capacity demonstrates that polymer matrices containing nanocellulose have good water-repellent properties. The nanocellulose-reinforced films outperform the neat PVA in terms of water resistance. This effect demonstrates the efficacy of nanocellulose reinforced in a PVA polymer matrix in reducing water absorption capacity.

It can be seen that the presence of nanocellulose in the PVA matrix was able to reduce the composite's water uptake, indicating that the nanocellulose was able to increase tortuosity in the composite and reduce the composite's water absorption. Another issue is that the use of polyvinyl alcohol in food packaging applications has been limited due to its ability to absorb water. Water-absorbing packaging materials are ideal for the growth of bacteria and microorganisms. These properties have an impact on and contribute to the deterioration of food in storage. As a result, the incorporation of Nano cellulose improves the water preventability of the PVA matrix.

4.7.2. Thermal stability analysis

Thermogravimetry continually monitors a sample's weight as a function of temperature. TGA analyses were performed with a heating rate of 10°C per minute from room temperature to 700°C (Salh & Raswl, 2018). The composite films were sliced into small pieces and tested using a microbalance and TGA equipment. The composite films were chopped into small pieces before being evaluated using micro-balance and TGA equipment. The temperature was determined via thermogravimetry analysis.

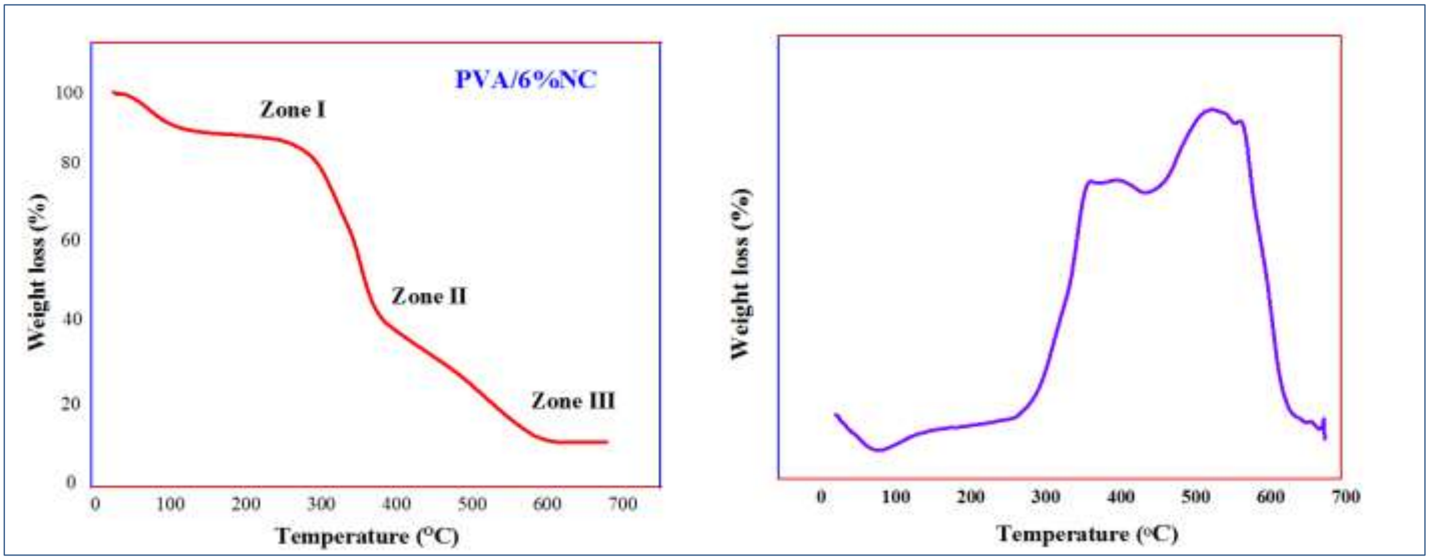


Figure 4.16: TGA and DTGA graph of PVA /6wt% NC

The thermo-gravimetric examination of 6wt% Nanocellulose reinforced PVA films reveals three main weight loss zones, as illustrated in Figure 4.16. The film loses weight in the first section due to the evaporation of physically weak and chemically strong bonding of water, which runs from 28.60°C to 290.78°C, and the weight loss is around 15.0 wt.%. The structural thermal degradation of PVA intermolecular hydrogen bonding, composite films caused the second transition zone to encompass a wider temperature range of roughly 290.78 °C to 380.20 °C, and the total weight loss in this range was about 80.56 wt.%.

The third stage has residue and weight loss above 380.20°C, which is likely owing to the cleavage backbone of PVA composite films or carbonaceous matter degradation. At 590.71°C, the overall weight loss in this range was only 0.85 wt. %. The reading in this region reveals essentially no weight loss as the temperature rises. Generally, it is discussed from several studies that reinforced nanocomposite has better thermal stability than neat PVA (Chin, 2020).

4.7.3. Mechanical tests

The mechanical properties of nanocomposite films were investigated by measuring tensile strength and elongation at break with a Testing Machine at Tana drilling and industries plc (Addis Ababa, Ethiopia). The mechanical properties of nanocomposites were investigated through the interaction of nanocellulose and PVA matrix.

The films were cut into rectangular pieces with dimensions of (10 mm by15 mm), and the analysis was carried out at a crosshead speed of 25 mm/min and a gauge length of 40 mm (Lani et al., 2014). The values of the sample's tensile strength and elongation at break are evaluated and reported as the average values of measurements for each of the compositions. The tests were used to compare the composite films' tensile strength and elongation at break to that of neat PVA.

4.7.3.1. Tensile strength

Concerning polyvinyl alcohol (PVA) and bamboo nanocellulose, four different weight percentage ratios were attempted. The tensile strength of the composites film increased linearly as the weight percentage of nanocellulose in the PVA matrix increased until an optimum point was reached then decreased. As shown in Figure 4.17, the tensile strength obtained for neat PVA, PVA/2wt % NC, PVA/4wt % NC, PVA/6wt % NC and PVA/8wt%NC was around 32.18MPa, 34.13MPa, 35.96Mpa, 37.60MPa, and 30.86 MPa respectively. PVA/6wt % NCs had the highest tensile strength of all composites, with a value of 37.6MPa and similar studies confirmed (Yu et al., 2013). The value indicates that adding 6wt% NC to the PVA matrix increases the tensile strength. This was done due to the existence of small particles with high aspect ratio and nanocellulose spreading in polyvinyl alcohols matrix suggested that there is intermolecular interaction between nanocellulose and neat PVA and the noble adherence at the filler/matrix which resulted in improved mechanical capabilities, is hydrogen bonding between the O-H groups of cellulose fibers and analogous groups of the PVA matrix.

The addition of 8% NC to the PVA solution resulted in a decrease in tensile strength. The excess amount of nanocellulose increases intermolecular interaction, which may compute between the interactions of PVA and NCs, and the frequency of faults in nanocomposites, such as bubbles trapped during sample preparation, increases with increasing reinforcement, resulting in a decrease in composite tensile strength.

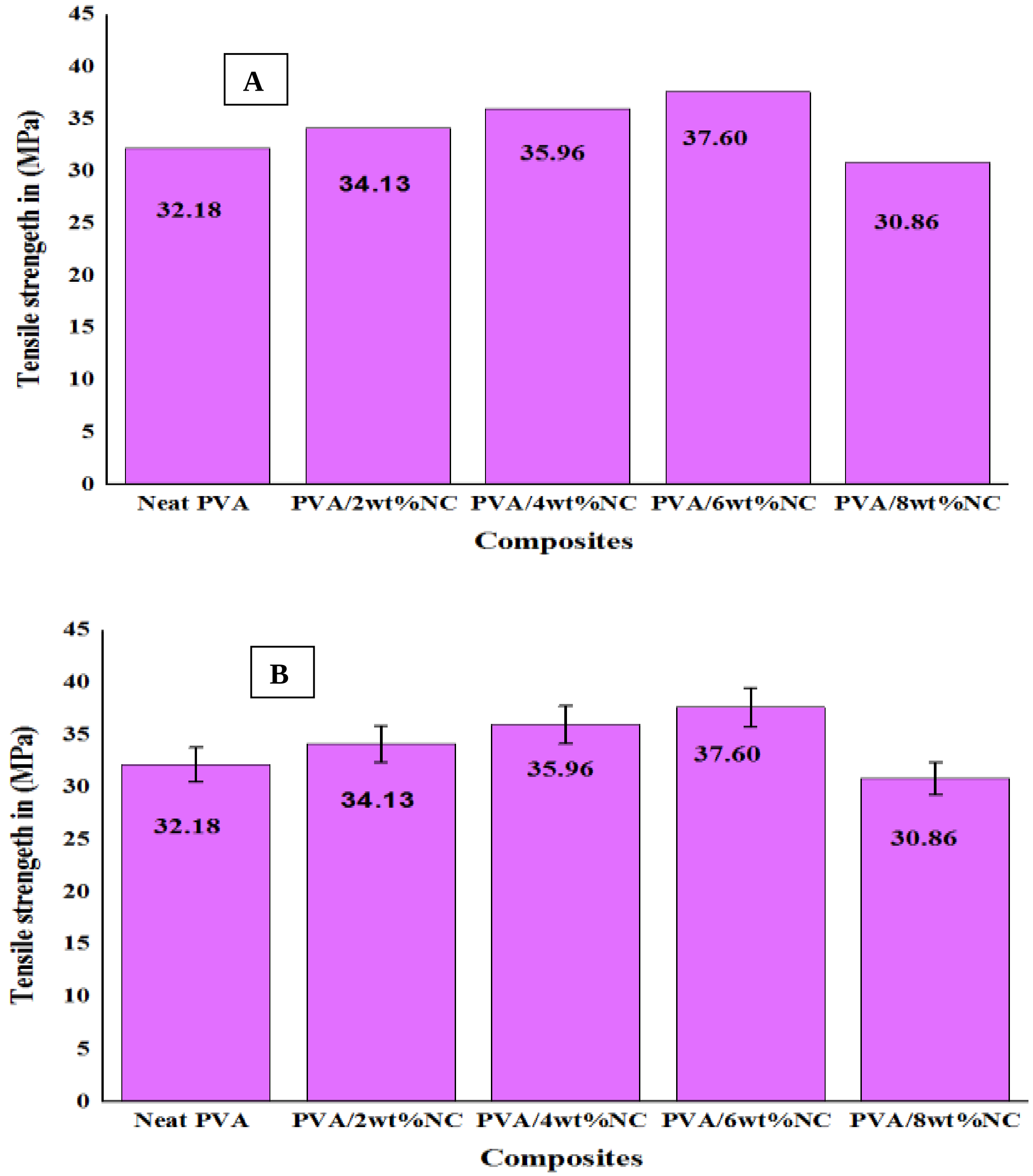


Figure 4.17: **A)** Tensile strength and **B)** Error bar for different nanocellulose loading

The strong interaction between the PVA matrix and the nanocellulose may be able to improve the nanocomposite properties by increasing the tensile strength of the nanocellulose linked through H-bonding.The percolation phenomenon increased in tensile strength of up to 6% NCs loading, which improved the mechanical properties of the reinforced composite films. Furthermore, uniform dispersion of nanocellulose within the PVA matrix may be capable of producing a continuous network as well as a strong interaction between the nanocellulose and the PVA matrix. As a result of this formation, composite films with improved mechanical properties were created.

4.7.3.2. Elongation at break

Percent elongation at break was calculated by dividing each specimen's extension at the moment of rupture by the initial grip separation and multiplying by 100. As shown in Figure 4.18, the average of each five test specimen’s elongations at the break was determined for all composite fabricated.

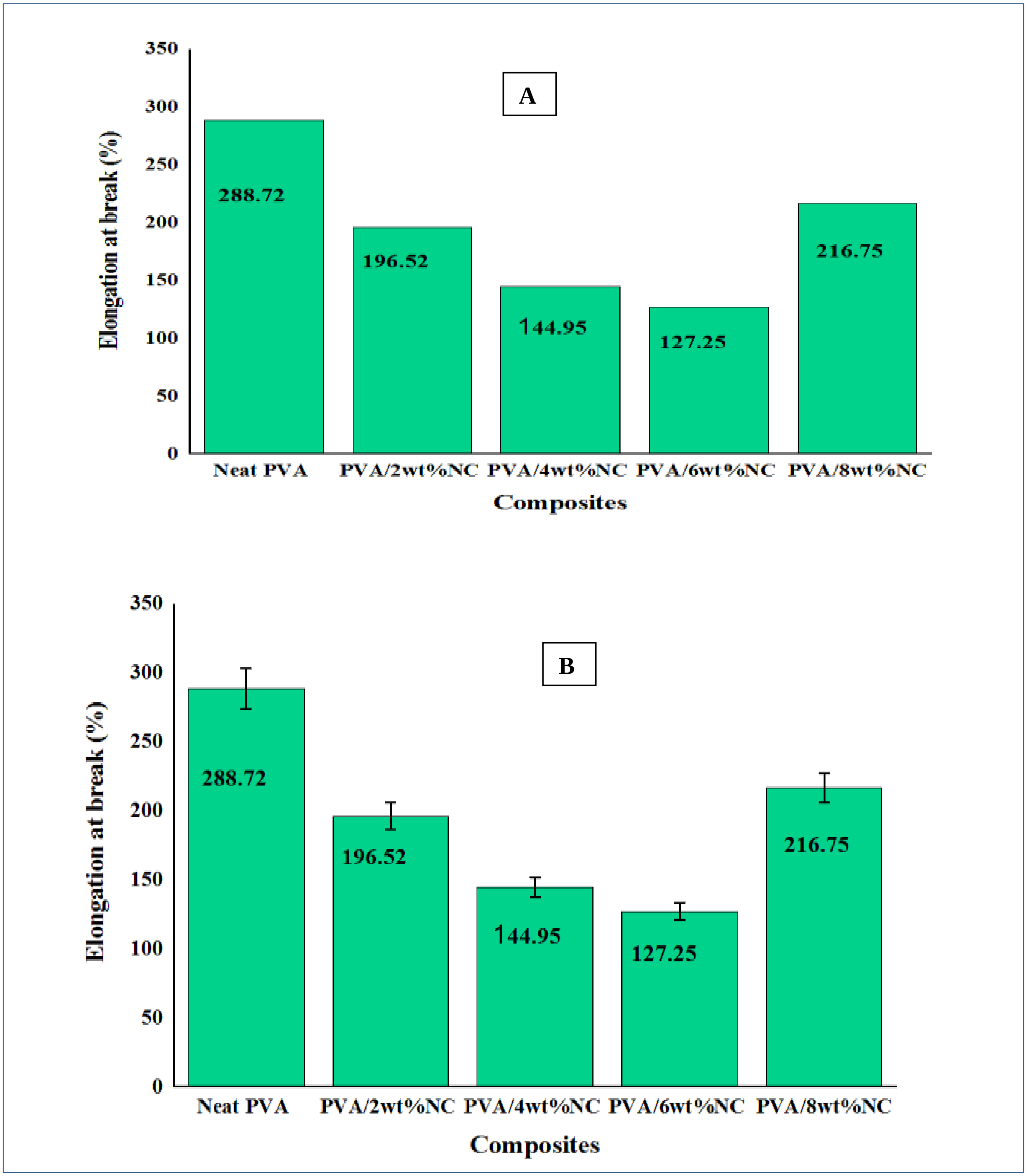


Figure 4.18: **A)** Elongation @break and **B)** Error bar for different nanocellulose loading

As seen in Figure 4.18, the number of nanocellulose filler particles in the PVA polymer matrix decreases percentages of elongation at the break until an optimum point and then increased. Elongations at the break for the composite films fall from 288.72 to 196.52, 144.95, and 127.25 when the amount of nanocellulose filler increases from 0 wt. % NC to 2 wt. % NC, 4 wt. %t NC, and 6 wt. % NC, respectively.The interaction of the link between the nanofiller and the PVA matrix is responsible for the composite film's decrease as loading increases. Similar studies were confirmed (Tang et al., 2015).

However, the reduction was noticed after the addition of 2% NC. This could be due to the rigidity of the fibers. Strong contacts between Nano cellulose and PVA chains may also impede chain mobility, obstructing flow and reducing ductility. Numerous researchers reported that the tensile strength and elongation at the break of nanocellulose reinforced with polyvinyl alcohols composites increased until optimum points, then decrement was perceived. Table 4.12 shows the summary of elongation at break and tensile strength of some studies literature.

Table 4.12: Comparison of mechanical properties of PVA composite films with literature

Nano cellulose loading	Tensile strength (MPa)	Elongation @break (%)	Source
0%NC	27	48	(Frone et al., 2011)
2%NC	32	29	
4%NC	34	25	
0%NC	100	25	(Zhou et al., 2012)
2%NC	108	40	
6%NC	114	50	
8%NC	102	45	
0%NC	16.6	44.6	(Spagnol et al., 2018)
4%NC	25.5	52.3	
6%NC	70	41.6	
8%NC	33.1	50.6	

5. CONCLUSION AND RECOMMENDATION

5.1. Conclusion

In this study, nanocellulose was successfully synthesized from highland bamboo (*Arundinaria Alpina*) using sulfuric acid hydrolysis. The synthesis was carried out in four basic stages namely pretreatment, alkali treatment, bleaching, and acid hydrolysis respectively. The optimization process condition was carried out for acid hydrolysis temperature, reaction time, and acid concentration through design expert software and the result was 40℃, 60 min, and 61.404 wt.% respectively. The synthesized nanocellulose's characteristics, including crystallinity, functional group, and particle size was studied. The crystallinity of the generated products improved after each successive treatment with the treated bamboo, according to an XRD examination. The crystallinity indexes of bamboo and Nano cellulose were 44.60 and 74.07% respectively and the average crystallite size was found to be 35.20nm. These XRD results demonstrate that lignin, hemicellulose, and other amorphous portions of bamboo were reduced.

The average particle size was determined to be 106.5nm with monomial graphs, and the poly-dispersity index was 0.0632. The structure of the nanocellulose could be confirmed by the structural analyses, Fourier transforms infrared spectroscopy. Water absorption capacity, mechanical testing, and thermal stability were used to characterize the reinforced Nano cellulose in PVA films using different sets of bamboo powder Nano cellulose suspension created by solvent casting. The water absorption capacity of the samples reduces linearly as the amount of nanocellulose added increases. Neat PVA with 58.921% water absorption capacity when compared to PVA/8%NC 24.729%, films performed better in terms of reducing water absorption capacity. The maximum tensile strength was 37.60 MPa with the addition of 6% NC, while the minimum was 32.18 MPa without the addition of reinforcement and finally decreases after optimum points were reached. According to the findings, bamboo is the best lignocellulosic materials for generating Nano cellulose, and reinforcing nanocellulose with polyvinyl alcohol at a lower nanocellulose ratio increases the water absorption and mechanical properties of composite materials and polymer films.

5.2. Recommendation

Nanocomposite materials have significant advantages for the current world by reducing the problem caused due to petroleum-based resources which can affect the environment. So by replacing these resources with biological-based products such as nano-biocomposites is acceptable in terms of economic feasibility, environmental sustainability, and social life. Therefore, it is important to address the future works that are not included in this research and other incremental research from other biological-based Lignocellulosic materials.

- In this research, the acid hydrolysis method was used for Nanocellulose extraction. But it is better if simultaneously use enzymatic, and mechanical methods need to be done to investigate the variation that could be a rise in the quality and quantity of the NCs yield.
- Due to the unavailability of some characterization equipment such as Atomic Force Microscopy, Scanning Electron Microscopy, Transmission Electron Microscopy it will be recommended for future researchers.
- Different factors can affect the yield of nanocellulose during acid hydrolysis. In this study, only the effects of reaction temperature, hydrolysis time, and acid concentration at constant solid cellulose to the acid ratio on yield were tested. Therefore, other parameters such as solid cellulose to the acid ratio that can affect the yield and properties of nano cellulose should also be tested.
- In this research bamboo chips from bamboo, tree-based nanocellulose was investigated, but also it is possible to produce nanoparticle from bamboo leaves for nanocomposite application and it is recommended for future researches to investigate this issue.
- Optimization process parameters that affect the yield of the nanocomposite.

REFERENCES

Abbate, F., Iulianelli, G. C. V, Inês, M., & Tavares, B. (2016). *The Use of Cellulose Nanofillers in Obtaining Polymer Nanocomposites : Properties , Processing , and Applications*. *May*, 257–294.

Abdel-Halim, E. S. (2014). Chemical modification of cellulose extracted from sugarcane bagasse: Preparation of hydroxyethyl cellulose. *Arabian Journal of Chemistry*, *7*(3), 362–371. https://doi.org/10.1016/j.arabjc.2013.05.006

Abitbol, T., Rivkin, A., Cao, Y., Nevo, Y., Abraham, E., Ben-shalom, T., Lapidot, S., & Shoseyov, O. (2016). ScienceDirect Nanocellulose , a tiny fiber with huge applications. *Current Opinion in Biotechnology*, *39*(June), 76–88. https://doi.org/10.1016/j.copbio.2016.01.002

Ahmad, I., Thomas, S., Wiley, A. D. J., Ioelovich, M., & Kinds, V. (2017). *Handbook of Nanocellulose and Cellulose Nanocomposites . Editors : H . 1*.

Alves, H., Pires, W., Neto, F., Oliveira, N., & Pasquini, D. (2013). Extraction and characterization of cellulose nanocrystals from corncob for application as reinforcing agent in nanocomposites. *Industrial Crops & Products*, *44*, 427–436. https://doi.org/10.1016/j.indcrop.2012.10.014

Anuar, S., Warren, B., & Andreas, G. (2017). Thermal stability of processed PVC / bamboo blends : effect of compounding procedures. *European Journal of Wood and Wood Products*, *75*(2), 147–159. https://doi.org/10.1007/s00107-016-1148-5

Ariadurai, S. (2014). *Bio-Composites : Current Status and Future Trends Bio-Composites : Current Status and Future Trends*. *May*.

Arora, A., Nandal, P., Singh, J., & Verma, M. L. (2020). Nanobiotechnological advancements in lignocellulosic biomass pretreatment. *Materials Science for Energy Technologies, 3,* 308–318. https://doi.org/10.1016/j.mset.2019.12.003

Aslam, M., Kalyar, M. A., & Raza, Z. A. (2018). *Polyvinyl Alcohol : A Review of Research Status and Use of Polyvinyl Alcohol Based Nanocomposites*. https://doi.org/10.1002/pen.24855

Ayeni, A. O., Adeeyo, O. A., Oresegun, O. M., & Oladimeji, T. E. (2015). Compositional analysis of lignocellulosic materials: Evaluation of an economically viable method suitable for woody and non-woody biomass. *American Journal of Engineering Research*, *44*, 2320–2847. www.ajer.org

Beyan, S. M., Amibo, T. A., Prabhu, S. V., & Ayalew, A. G. (2021). Production of Nanocellulose Crystal Derived from Enset Fiber Using Acid Hydrolysis Coupled with Ultrasonication, Isolation, Statistical Modeling, Optimization, and Characterizations. *Journal of Nanomaterials*, *2021*. https://doi.org/10.1155/2021/7492532

Bhat, A. H., Dasan, Y. K., Khan, I., Soleimani, H., & Usmani, A. (2017). Application of nanocrystalline cellulose: Processing and biomedical applications. In *Cellulose-Reinforced Nanofibre Composites: Production, Properties and Applications*. Elsevier Ltd. https://doi.org/10.1016/B978-0-08-100957-4.00009-7

Carley, K. M. (2014). *Response Surface Methodology*. *October 2004*.

Chin, S. C. (2020). Thermal and mechanical properties of bamboo fi ber reinforced composites. *Materials today Communications*, *23*(September 2019), 100876. https://doi.org/10.1016/j.mtcomm.2019.100876

Ching, Y. C., Rahman, A., Ching, K. Y., Sukiman, N. L., & Chuah, C. H. (2015). *Preparation and Characterization of Polyvinyl Alcohol- Based Composite Reinforced with Nanocellulose and*. *10*(2), 3364–3377.

Chu, P. K., & Liu, X. (2008). Biomaterials fabrication and processing: Handbook. *Biomaterials Fabrication and Processing Handbook*, 1–701. https://doi.org/10.1201/9780849379741

Cobos, M., De-La-Pinta, I., Quindós, G., Fernández, M. J., & Fernández, M. D. (2020). alcohol) / Graphene Oxide – Silver Nanoparticles. *Polymers*, *12*(723), 1–18.

Costa, L. A. S., Assis, D. D. J., Gomes, G. V. P., & Jania, B. A. (2015). Extraction and characterization of nanocellulose from corn stover. *Materials Today: Proceedings*, *2*(1), 287–294. https://doi.org/10.1016/j.matpr.2015.04.045

Darder, M., Aranda, P., & Ruiz-Hitzky, E. (2007). Bionanocomposites: A new concept of ecological, bioinspired, and functional hybrid materials. *Advanced Materials*, *19*(10), 1309–1319. https://doi.org/10.1002/adma.200602328

Das, M., & Chakraborty, D. (2006). *Influence of Alkali Treatment on the Fine Structure and Morphology of Bamboo Fibers*. *102*(June), 5050–5056. https://doi.org/10.1002/app.25105

Davoudpour, Y., Aprilia, N. A. S., Mustapha, A., & Dungani, R. (2013). *Nanocellulose Based Polymer Nanocomposite: Isolation, Characterization and Applications*. 1–31.

Design, I., Centre, D., Khas, H., & Gate, K. (1992). *Mechanical behaviour of bamboo and bamboo composite*. *27*, 4598–4604.

Ditzel, F. I., Prestes, E., Carvalho, B. M., Demiate, I. M., & Pinheiro, L. A. (2017). Nanocrystalline cellulose extracted from pine wood and corncob. *Carbohydrate Polymers*, *157*, 1577–1585. https://doi.org/10.1016/j.carbpol.2016.11.036

Dong, P., Prasanth, R., Xu, F., Wang, X., Li, B., & Shankar, R. (2015). Eco-friendly polymer nanocomposite—properties and processing. In *Advanced Structured Materials* (Vol. 75). https://doi.org/10.1007/978-81-322-2470-9_1

Dp, C. (2016). *Journal of Textile Science & Engineering Synthesis , Characterization and Application of nano cellulose for enhanced performance of textiles*. *6*(2). https://doi.org/10.4172/2165-8064.1000248

Embaye, K. (2000). The indigenous bamboo forests of Ethiopia: An overview. *Ambio*, *29*(8), 518–521. https://doi.org/10.1579/0044-7447-29.8.518

Fahma, F., Hori, N., Iwata, T., & Takemura, A. (2017). *PVA nanocomposites reinforced with cellulose nanofibers from oil palm empty fruit bunches (OPEFBs)*. *29*(5), 323–329. https://doi.org/10.9755/ejfa.2016-02-215

Fall, A. B. (2013). *Colloidal interactions and orientation of nanocellulose particles* (Issue december).

Faria, M., Thomas, S., & Pothan, L. A. (2015). *Utilization of various lignocellulosic biomass for the production of nanocellulose : a comparative study*. https://doi.org/10.1007/s10570-015-0554-x

Filler, N., & Othman, S. H. (2016). Bio-nanocomposite Materials for Food Packaging Applications : Types of Bio-nanocomposite Materials for Food Packaging Applications : Types of Biopolymer and Nano-sized Filler. *Italian Oral Surgery*, *2*(December 2014), 296–303. https://doi.org/10.1016/j.aaspro.2014.11.042

Fowler, P. A., Hughes, J. M., & Elias, R. M. (2006). *Biocomposites : technology , environmental credentials and market forces*. *1789*(July), 1781–1789. https://doi.org/10.1002/jsfa

Frone, A. N., Panaitescu, D. M., Donescu, D. A. N., Radovici, C., & Ghiurea, M. (2011). *PVA Based Composite Films with Cellulose Fibers Prepared by Acid Hydrolysis*. *2*.

Furukawa, Y. (2013). Polymer chemistry. *Science in the Twentieth Century*, 547–563. https://doi.org/10.4324/9781315079097-35

Gallardo-Sánchez, M. A., Diaz-Vidal, T., Navarro-Hermosillo, A. B., Figueroa-Ochoa, E. B., Casillas, R. R., Hernández, J. A., Rosales-Rivera, L. C., Martínez, J. F. A. S., Enríquez, S. G., & Macías-Balleza, E. R. (2021). Optimization of the obtaining of cellulose nanocrystals from agave tequilana weber var. Azul Bagasse by acid hydrolysis. *Nanomaterials*, *11*(2), 1–21. https://doi.org/10.3390/nano11020520

Gan, P. G., Sam, S. T., Faiq, M., & Omar, M. F. (2019). *Thermal properties of nanocellulose-reinforced composites : A review*. *48544*. https://doi.org/10.1002/app.48544

George, J. (2015). *Cellulose nanocrystals : synthesis , functional properties , and applications*. 45–54.

Hajeeassa, K. S., Hussein, M. A., Anwar, Y., Tashkandi, N. Y., & Al-Amshany, Z. M. (2018). Nanocomposites containing polyvinyl alcohol and reinforced carbon-based nanofiller: A super effective biologically active material. *Nanobiomedicine*, *5*, 1–12. https://doi.org/10.1177/1849543518794818

Halib, N., Perrone, F., Id, M. C., Dapas, B., Farra, R., Abrami, M., Chiarappa, G., Forte, G., Zanconati, F., Pozzato, G., Murena, L., Fiotti, N., Lapasin, R., Cansolino, L., Grassi, G., & Id, M. G. (2017). *Potential Applications of Nanocellulose-Containing Materials in the Biomedical Field*. 1–31. https://doi.org/10.3390/ma10080977

Hallensleben, M. L., Fuss, R., & Mummy, F. (2015). Polyvinyl Compounds, Others. *Ullmann's Encyclopedia of Industrial Chemistry*, 1–23. https://doi.org/10.1002/14356007.a21_743.pub2

Han, S., Yao, Q., Jin, C., Fan, B., Zheng, H., & Sun, Q. (2016). *Cellulose Nanofibers From Bamboo and Their Nanocomposites With Polyvinyl Alcohol : Preparation and Characterization*. 1–9. https://doi.org/10.1002/pc

Harmsen, P., & Huijgen, W. (2010). *Literature Review of Physical and Chemical Pretreatment Processes for Literature Review of Physical and Chemical Pretreatment Processes for*. *January*.

Heriberto, J., Nascimento, O., Luz, R. F., Galvão, F. M. F., Daniel, J., Melo, D., Oliveira, F. R., Ladchumananandasivam, R., & Zille, A. (2015). Extraction and Characterization of Cellulosic Nanowhisker obtained from Discarded Cotton Fibers. *Materials Today: Proceedings*, *2*(1), 1–7. https://doi.org/10.1016/j.matpr.2015.04.001

Hossain, I., Zaman, H., & Rahman, T. (2018). *Derivation of Nanocellulose from Native Rice Husk*. *20*, 20–23.

Ioelovich, M. (2012). *Optimal Conditions for Isolation of Nanocrystalline Cellulose Particles*. *2*(2), 9–13. https://doi.org/10.5923/j.nn.20120202.03

Jabbar, A., Widiarto, S., Yuwono, S. D., Adnan, S., & Azhar, A. H. (2016). *Nanocellulose prepared by acid hydrolysis of isolated cellulose from sugarcane bagasse Nanocellulose prepared by acid hydrolysis of isolated cellulose from sugarcane bagasse*. https://doi.org/10.1088/1757-899X/107/1/012045

Jara, A. A., Razal, R. A., Migo, V. P., Acda, M. N., Calderon, M. M., Florece, L. M., & Peralta, E. K. (2020). *Production of nanocellulose and biocomposites from kawayan kiling (Bambusa* *vulgaris Schrader ex Wendland) shoots*. *10*, 10–17.

Jasmani, L., Rusli, R., Khadiran, T., Jalil, R., & Adnan, S. (2020). Application of Nanotechnology in Wood-Based Products Industry: A Review. *Nanoscale Research Letters*, *15*(1). https://doi.org/10.1186/s11671-020-03438-2

Julie Chandra, C. S., George, N., & Narayanankutty, S. K. (2016). Isolation and characterization of cellulose nanofibrils from arecanut husk fibre. *Carbohydrate Polymers*, *142*, 158–166. https://doi.org/10.1016/j.carbpol.2016.01.015

Kalia, S., Dufresne, A., Cherian, B. M., Kaith, B. S., Av, L., Njuguna, J., & Nassiopoulos, E. (2011). *Cellulose-Based Bio- and Nanocomposites : A Review*. *2011*. https://doi.org/10.1155/2011/837875

Kandhola, G., Djioleu, A., Rajan, K., Labbé, N., Sakon, J., Carrier, D. J., & Kim, J. W. (2020). Maximizing production of cellulose nanocrystals and nanofibers from pre-extracted loblolly pine kraft pulp: a response surface approach. *Bioresources and Bioprocessing*, *7*(1). https://doi.org/10.1186/s40643-020-00302-0

Kaur, N., Saxena, S., Gaur, H., & Goyal, P. (2017). *A Review on Bamboo Fiber Composites and its Applications*.

Khalil, H. P. S. A., Bhat, I. U. H., Jawaid, M., Zaidon, A., Hermawan, D., & Hadi, Y. S. (2012). Bamboo fibre reinforced biocomposites : A review. *Materials and Design*, *42*, 353–368. https://doi.org/10.1016/j.matdes.2012.06.015

Khan, A., Rangappa, S. M., Siengchin, S., & Asiri, A. M. (2020). Biofibers and biopolymers for biocomposites: Synthesis, characterization and properties. In *Biofibers and Biopolymers for Biocomposites: Synthesis, Characterization and Properties*. https://doi.org/10.1007/978-3-030-40301-0

Kindu, M., & Universit, T. (2016). *Status of bamboo resource development , utilization , and research in Ethiopia : A review*. *July*.

Kong, I., Tshai, K. Y., & Hoque, M. E. (2015). *Manufacturing of Natural Fibre-Reinforced Polymer Composites by Solvent Casting Method*. 331–349. https://doi.org/10.1007/978-3-319-07944-8

Kumar, A., Negi, Y. S., Choudhary, V., & Bhardwaj, N. K. (2014). *Characterization of Cellulose Nanocrystals Produced by Acid-Hydrolysis from Sugarcane Bagasse as Agro-Waste*. *2*(1), 1–8. https://doi.org/10.12691/jmpc-2-1-1

Lam, E., Male, K. B., Chong, J. H., Leung, A. C. W., & Luong, J. H. T. (2012). Applications of functionalized and nanoparticle-modified nanocrystalline cellulose. *Trends in Biotechnology*, *30*(5), 283–290. https://doi.org/10.1016/j.tibtech.2012.02.001

Lani, N. S., Ngadi, N., Johari, A., & Jusoh, M. (2014). *Isolation , Characterization , and Application of Nanocellulose from Oil Palm Empty Fruit Bunch Fiber as Nanocomposites*. *2014*.

Lin, N., & Dufresne, A. (2014). Nanocellulose in biomedicine : Current status and future prospect. *European polymer journal*, *59,* 302–325. https://doi.org/10.1016/j.eurpolymj.2014.07.025

Lugt, P. Van Der, & Dobbelsteen, A. A. J. F. Van Den. (2005). *An environmental , economic and practical assessment of bamboo as a building material for supporting structures*. 1–9. https://doi.org/10.1016/j.conbuildmat.2005.02.023

Mandal, A., & Chakrabarty, D. (2011). Isolation of nanocellulose from waste sugarcane bagasse and its characterization. *Carbohydrate Polymers*, *86*(3), 1291–1299. https://doi.org/10.1016/j.carbpol.2011.06.030

Mefteh, F. Ben, Frikha, F., Daoud, A., Bouket, A. C., Luptakova, L., Alenezi, F. N., Al-anzi, B. S., Oszako, T., Gharsallah, N., & Belbahri, L. (2019). *Response Surface Methodology Optimization of an Acidic Protease Produced by Penicillium bilaiae Isolate TDPEF30 , a Newly Recovered Endophytic Fungus from Healthy Roots of Date Palm Trees (Phoenix dactylifera L .)*. 20894. https://doi.org/10.1021/ol200770k.

Mohammad, B., Islam, T., Alam, M. M., Torino, P., Patrucco, A., & Mon-, A. (2014). *Preparation of Nanocellulose : A Review*. *February 2015*. https://doi.org/10.14504/ajr.1.5.3

Moon, R. J., Martini, A., Nairn, J., Simonsen, J., & Youngblood, J. (2011). Cellulose nanomaterials review: Structure, properties and nanocomposites. In *Chemical Society Reviews* (Vol. 40, Issue 7). https://doi.org/10.1039/c0cs00108b

Mora, J. I. (2008). *Extraction of cellulose and preparation of nanocellulose from sisal fibers*. 149–159. https://doi.org/10.1007/s10570-007-9145-9

Morais, J. P. S., Rosa, M. D. F., De Souza Filho, M. D. S. M., Nascimento, L. D., Do Nascimento, D. M., & Cassales, A. R. (2013). Extraction and characterization of nanocellulose structures from raw cotton linter. *Carbohydrate Polymers*, *91*(1), 229–235. https://doi.org/10.1016/j.carbpol.2012.08.010

Moubarik, A., Grimi, N., & Boussetta, N. (2013). Composites : Part B Structural and thermal characterization of Moroccan sugar cane bagasse cellulose fibers and their applications as a reinforcing agent in low density polyethylene. *COMPOSITES PART B*, *52*, 233–238. https://doi.org/10.1016/j.compositesb.2013.04.040

Mugo, S. M. (2020). Isolation of Cellulose Nanofibers from Oryza sativa Residues via TEMPO Mediated Oxidation. *Journal of Natural Fibers*, *00*(00), 1–13. https://doi.org/10.1080/15440478.2020.1764454

Nascimento, P., Marim, R., Carvalho, G., Celso, R., Cid, G., Celso, R., & Cid, G. (2016). *Nanocellulose Produced from Rice Hulls and its Effect on the Properties of Biodegradable Starch Films*. *19*(1), 167–174.

Nilsson, C. (2017). *Preparation and characterization of nanocellulose from wheat bran*.

Nishiyama, Y., Sugiyama, J., Chanzy, H., & Langan, P. (2003). Crystal Structure and Hydrogen Bonding System in Cellulose Iα from Synchrotron X-ray and Neutron Fiber Diffraction. *Journal of the American Chemical Society*, *125*(47), 14300–14306. https://doi.org/10.1021/ja037055w. 1–127.

Oliveira, F. B. De, Bras, J., Teresa, M., Pimenta, B., Aprigio, A., & Belgacem, M. N. (2016). Production of cellulose nanocrystals from sugarcane bagasse fibers and pith. *Industrial Crops & Products*. https://doi.org/10.1016/j.indcrop.2016.04.064

Osorio, L., Trujillo, E., Lens, F., Ivens, J., & Verpoest, I. (2018). *In-depth study of the microstructure of bamboo fibres and their relation to the mechanical properties*. https://doi.org/10.1177/0731684418783055

Pandya, S., & Universiy, P. (2015). *nanocomposites and it ' s application-review nanocomposites and it's application -review - Shivani Pandya introduction : December*. https://doi.org/10.13140/RG.2.1.2798.9840

Phanthong, P., Reubroycharoen, P., Hao, X., Xu, G., & Abudula, A. (2018). Nanocellulose : Extraction and application. *Carbon Resources Conversion*. https://doi.org/10.1016/j.crcon.2018.05.004

Plengnok, U., & Jarukumjorn, K. (2020). Preparation and characterization of nanocellulose from sugarcane bagasse. *Biointerface Research in Applied Chemistry*, *10*(3), 5675–5678. https://doi.org/10.33263/BRIAC103.675678

Posom, J., & Sirisomboon, P. (2017). *Evaluation of the higher heating value , volatile matter , fixed carbon and ash content of ground bamboo using near infrared spectroscopy*. https://doi.org/10.1177/0967033517728733

Rahimi Kord Sofla, M., Brown, R. J., Tsuzuki, T., & Rainey, T. J. (2016). A comparison of cellulose nanocrystals and cellulose nanofibres extracted from bagasse using acid and ball milling methods. *Advances in Natural Sciences: Nanoscience and Nanotechnology*, *7*(3). https://doi.org/10.1088/2043-6262/7/3/035004

Ramos, M., Valdés, A., & Garrigós, M. C. (2016). Multifunctional Applications of Nanocellulose-Based Nanocomposites. In *Multifunctional Polymeric Nanocomposites Based on Cellulosic Reinforcements*. Elsevier Inc. https://doi.org/10.1016/B978-0-323-44248-0.00006-7

Rivai, H., Hamdani, A. S., Ramdani, R., Lalfari, R. S., Andayani, R., Armin, F., & Djamaan, A. (2021). Research on Production and Characterization of Alpha Cellulose Derived from Rice

Straw (Oryza sativa L.). *Technological Innovation in Pharmaceutical Research Vol. 3*, *52*(08), 68–75. https://doi.org/10.9734/bpi/tipr/v3/1698c

Saba, N., Jawaid, M., Sultan, M. T. H., & Alothman, O. Y. (2017). *Green Biocomposites for Structural Applications Green composites*. https://doi.org/10.1007/978-3-319-49382-4

Salh, S. H., & Raswl, D. A. (2018). *Thermal stability of polymer composite films based on polyvinyl alcohol doped with different fillers*. *2*(2), 5–10.

Santos, R. M. dos, Flauzino Neto, W. P., Silvério, H. A., Martins, D. F., Dantas, N. O., & Pasquini, D. (2013). Cellulose nanocrystals from pineapple leaf, a new approach for the reuse of this agro-waste. *Industrial Crops and Products*, *50*, 707–714. https://doi.org/10.1016/j.indcrop.2013.08.049

Sharma, A., Thakur, M., Bhattacharya, M., & Mandal, T. (2019). Commercial application of cellulose nano-composites – A review. *Biotechnology Reports*, *2018*, e00316. https://doi.org/10.1016/j.btre.2019.e00316

Shchipunov, Y. (2012). *Bionanocomposites : Green sustainable materials for the near future **. *84*(12), 2579–2607.

Slavutsky, A. M., & Bertuzzi, M. A. (2014). Water barrier properties of starch films reinforced with cellulose nanocrystals obtained from sugarcane bagasse. *Carbohydrate Polymers*, *110*, 53–61. https://doi.org/10.1016/j.carbpol.2014.03.049

Song, K., Zhu, X., Zhu, W., & Li, X. (2019). Preparation and characterization of cellulose nanocrystal extracted from Calotropis procera biomass. *Bioresources and Bioprocessing*, *6*(1). https://doi.org/10.1186/s40643-019-0279-z

Spagnol, C., Fragal, E. H., Witt, M. A., Follmann, H. D. M., Silva, R., & Rubira, A. F. (2018). Mechanically improved polyvinyl alcohol-composite films using modified cellulose nanowhiskers as nano-reinforcement. *Carbohydrate Polymers*, *191*(February), 25–34. https://doi.org/10.1016/j.carbpol.2018.03.001

Tang, C., Tian, Y., & Hsu, S. (2015). *Poly(vinyl alcohol) Nanocomposites Reinforced with Bamboo Charcoal Nanoparticles: Mineralization Behavior and Characterization*. 4895–4911. https://doi.org/10.3390/ma8084895

Thakur, M., Sharma, A., Ahlawat, V., Bhattacharya, M., & Goswami, S. (2020). Process optimization for the production of cellulose nanocrystals from rice straw derived a -cellulose. *Materials Science for Energy Technologies*, *3*, 328–334. https://doi.org/10.1016/j.mset.2019.12.005

Thamer, N. A., Muftin, N. Q., & Al-Rubae, S. H. N. (2018). Optimization properties and characterization of green synthesis of copper oxide nanoparticles using aqueous extract of cordia myxa L. Leaves. *Asian Journal of Chemistry*, *30*(7), 1559–1563. https://doi.org/10.14233/ajchem.2018.21242

Wang, Jie, Liu, X., Jin, T., He, H., Liu, L., Wang, J., Liu, X., Jin, T., He, H., & Liu, L. (2019). Preparation of nanocellulose and its potential in reinforced composites : A review Preparation of nanocellulose and its potential in. *Journal of Biomaterials Science, Polymer Edition*, *30*(11), 919–946. https://doi.org/10.1080/09205063.2019.1612726

Wang, Jinlong, Li, X., Song, J., Wu, K., Xue, Y., & Wu, Y. (2020). *Direct Preparation of Cellulose Nanofibers from Bamboo by Nitric Acid and Hydrogen Peroxide Enables Fibrillation via a Cooperative Mechanism*.

Wegner, T. H., & Jones, E. P. (2005). *Biomass*.

Wijaya, C. J., Ismadji, S., Aparamarta, H. W., & Gunawan, S. (2019). Heliyon Optimization of cellulose nanocrystals from bamboo shoots using Response Surface Methodology. *Heliyon*, *5*(July), e02807. https://doi.org/10.1016/j.heliyon.2019.e02807

Xie, H., Du, H., Yang, X., & Si, C. (2018). *Recent Strategies in Preparation of Cellulose Nanocrystals and Cellulose Nanofibrils Derived from Raw Cellulose Materials*.

Xie, J. (2018). *Preparation of Cellulose Nanofibers from Bamboo Using Microwave Liquefaction*.

Yoon, S., Park, M., & Byun, H. (2012). Mechanical and water barrier properties of starch / PVA composite films by adding nano-sized poly (methyl methacrylate- co -acrylamide) particles. *Carbohydrate Polymers*, *87*(1), 676–686. https://doi.org/10.1016/j.carbpol.2011.08.046

Yu, H., Qin, Z., Liu, L., Yang, X., Zhou, Y., & Yao, J. (2013). Comparison of the reinforcing effects for cellulose nanocrystals obtained by sulfuric and hydrochloric acid hydrolysis on the mechanical and th- ermal properties of bacterial polyester. *Composites Science and Technology*, *87*, 22–28. https://doi.org/10.1016/j.compscitech.2013.07.024

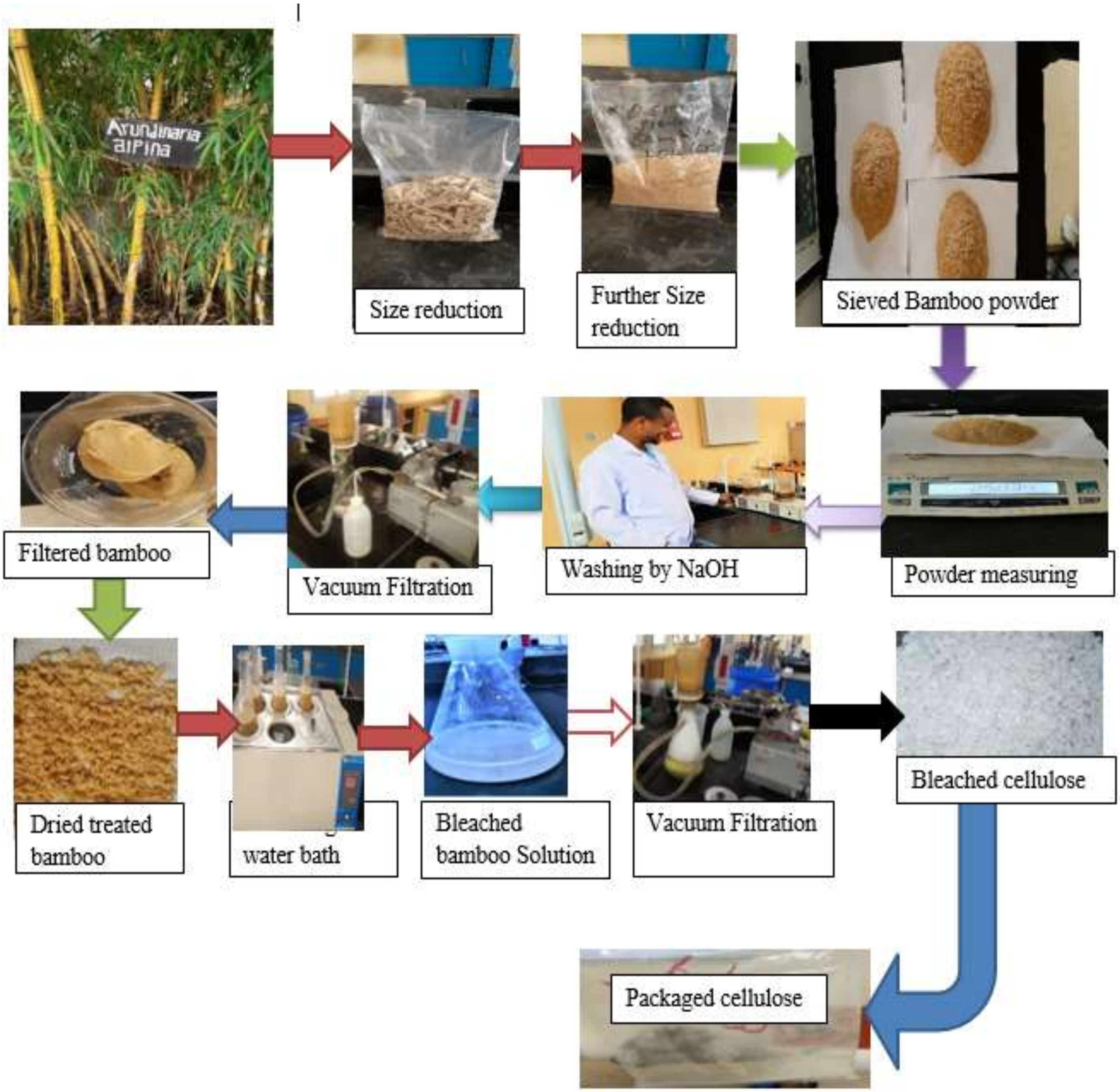
Zhao, J., Zhang, W., Zhang, X., Zhang, X., Lu, C., & Deng, Y. (2013). Extraction of cellulose nanofibrils from dry softwood pulp using high shear homogenization. *Carbohydrate Polymers*, *97*(2), 695–702. https://doi.org/10.1016/j.carbpol.2013.05.050

Zhou, Y. M., Fu, S. Y., Zheng, L. M., & Zhan, H. Y. (2012). *Effect of nanocellulose isolation techniques on the formation of reinforced poly (vinyl alcohol) nanocomposite films*. *6*(10), 794–804. https://doi.org/10.3144/expresspolymlett.2012.85

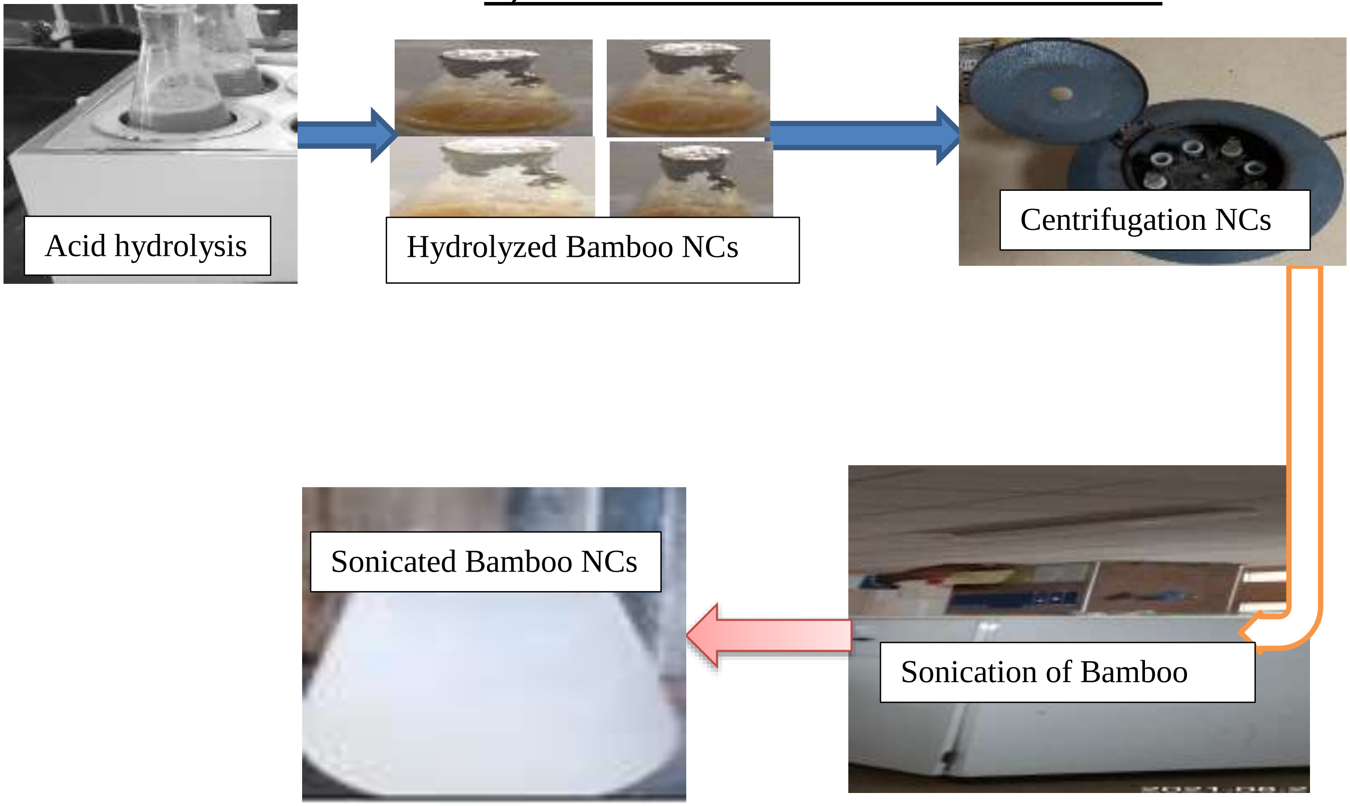
APPENDIX

Appendix A: Some laboratory experiment procedure

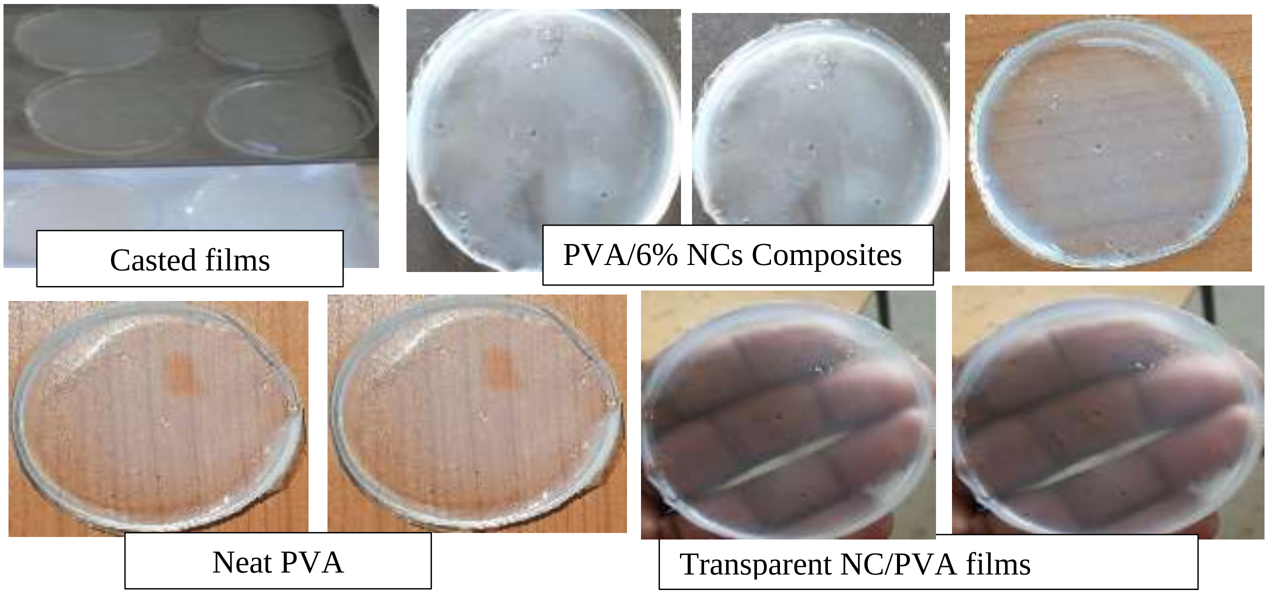
I) Bamboo Cellulose Extraction



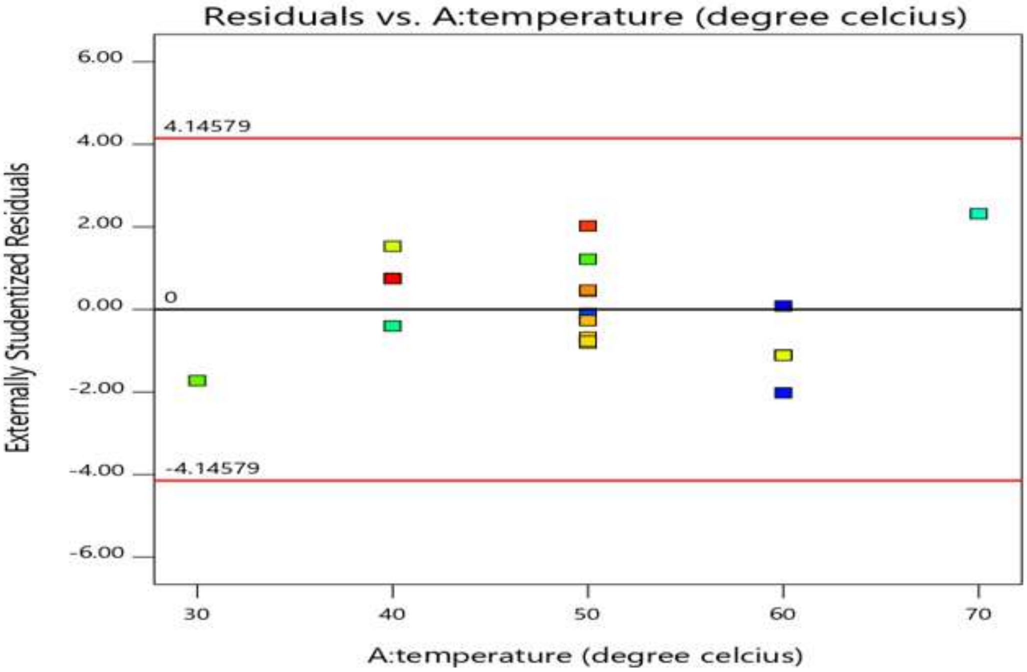
II) Bamboo Nano-Cellulose Extraction Procedure



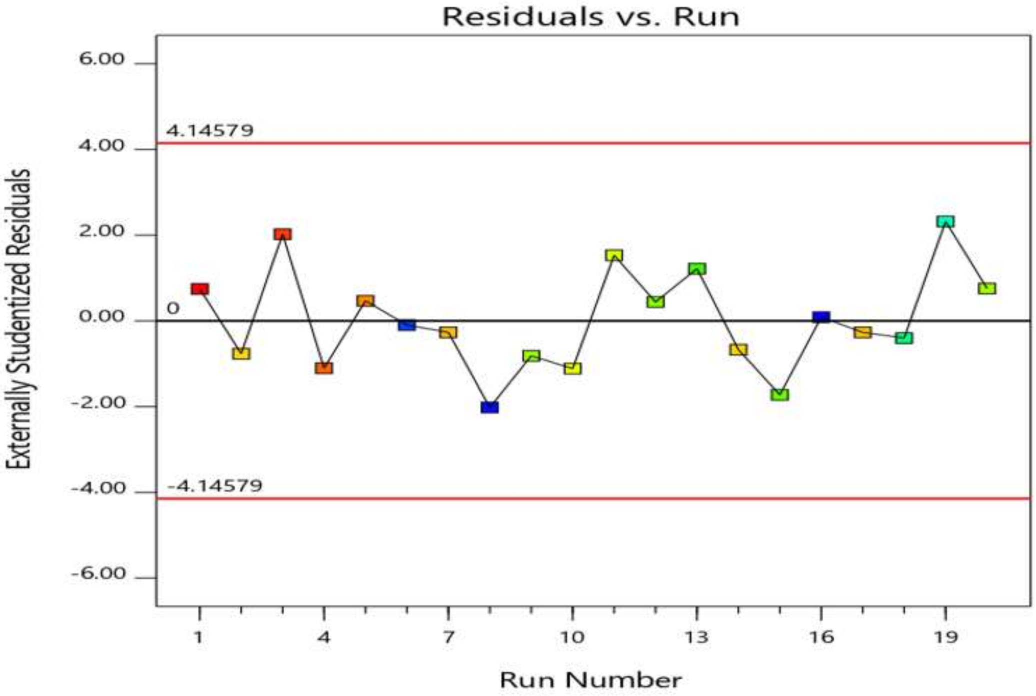
II) Nano-Cellulose/PVA synthesized composite



Appendix B: Some results from design expert software



The plot of residuals versus temperature



A plot of residuals versus the run number

Lack of Fit Tests

Table B$_1$: Lack of fit test from ANOVA

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Linear	315.82	11	28.71	32.73	0.0006	
2FI	139.49	8	17.44	19.88	0.0022	
Quadratic	**6.95**	**5**	**1.39**	**1.58**	**0.3131**	**Suggested**
Cubic	0.1702	1	0.1702	0.1940	0.6780	Aliased
Pure Error	4.39	5	0.8772			

Response Desirability

Table B$_2$: Optimal possible solutions

Number	Temperature	Reaction Time	Concentration	Yield	Desirability	
1	**40.000**	**60.000**	**61.404**	**43.155**	**0.985**	**Selected**
2	40.000	60.000	61.488	43.155	0.985	
3	40.001	60.000	61.313	43.155	0.985	
4	40.000	60.000	61.691	43.153	0.984	
5	40.000	60.000	61.844	43.151	0.984	
6	40.000	59.822	61.504	43.146	0.984	
7	40.000	59.740	61.113	43.140	0.984	
8	40.088	60.000	61.358	43.128	0.983	
9	40.000	60.000	59.891	43.110	0.982	
10	40.000	59.999	63.045	43.102	0.981	

Appendix C: Crystallite sizes

Table C$_1$: Peak data list and average crystallite size for Nanocellulose

Peak No.	2θ (degree)	d(A)	FWHM (Counts)	Intensity (Counts)	Crystallite Size (nm)	Integrated Area(nm^2)	Total area (nm^2)
1.	15.759	5.57580	0.0787	1506	26.91	4647	1829613
2.	22.864	3.88967	0.3149	2320	17.92	4245	
3.	32.174	2.78241	0.1968	753	43.90	13302	
4.	34.290	2.61517	0.2755	630	31.50	1803174	
5.	38.117	2.36092	0.1574	484	55.78	14245	

Appendix D: IR Spectrum table by frequency range

Table D1: IR spectrum table by frequency range

Absorption (cm^{-1})	Functional Group	Compound Class
3700-3200	O-H stretching	Alcohol
3400-3300	N-H stretching	aliphatic primary amine
3350-3310	N-H stretching	secondary amine
3300-2500	O-H stretching	carboxylic acid
3200-2700	O-H stretching	Alcohol
3333-3267	C-H stretching	Alkyne
3100-3000	C-H stretching	Alkene
3000-2840	C-H stretching	Alkane
2830-2695	C-H stretching	Aldehyde
2260-2222	C≡N stretching	Nitrile
2260-2190	C≡C stretching	Alkyne
2175-2140	S-C≡N stretching	Thiocyanate
2160-2120	N=N=N stretching	Azide
2145-2120	N=C=N stretching	Carbodiimide
2140-2100	C≡C stretching	Alkyne
2000-1900	C=C=C stretching	Alleyne
2000-1650	C-H bending	aromatic compound
1750-1735	C=O stretching	Esters
1740-1720	C=O stretching	Aldehyde
1720-1706	C=O stretching	carboxylic acid
1678-1648	C=C stretching	Alkene
1650-1580	N-H bending	Amine
1648-1638	C=C stretching	Alkene

1440-1395	O-H bending	carboxylic acid
1420-1330	O-H bending	Alcohol
1415-1380	S=O stretching	Sulfate
1390-1310	O-H bending	Phenol
1372-1335	S=O stretching	Sulfonate
1342-1266	C-N stretching	aromatic amine
1210-1163	C-O stretching	Ester
1150-1085	C-O stretching	aliphatic ether
1124-1087	C-O stretching	secondary alcohol
1085-1050	C-O stretching	primary alcohol
1070-1030	S=O stretching	Sulfoxide
730-665	C=C bending	Alkene