SYNTHESIS AND CHARACTERISATION OF NANOCELLULOSE CRYSTALS FROM PINEAPPLE LEAVES

PROJECT REPORT

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CERTIFICATE

This is to certify that the project report entitled **"SYNTHESIS AND CHARACTERISATION OF NANOCELLULOSE CRYSTALS FROM PINEAPPLE LEAVES"** is an authentic work done by SREELAKSHMI S, under my supervision at Department of Physics, St Teresa's College, Ernakulam, for the partial requirements for the award of Degree of Bachelor of Science in Physics during the academic year 2022-23. The work presented in this dissertation has not been submitted for any other degree in this or any other university.

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Place: ERNAKULAM
Date: 25-04-2023

St. Teresa's College (Autonomous) ★ Department of Physics ★ Ernakulam

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B.Sc.Physics

PROJECT REPORT

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Date: 25-04-2023

Examiners: 24/04/23

DECLARATION

We, SREYA SONAN and SREELAKSHMI S, final year BSc Physics students, Department of Physics, St.Teresa's College (Autonomous), Ernakulam, do hereby declare that the project work entitled **"SYNTHESIS AND CHARACTERISATION OF NANOCELLULOSE CRYSTALS FROM PINEAPPLE LEAVES "** has been originally carried out under the guidance and supervision of Dr. KALA M S, Professor, Department of Physics, St.Teresa's College(Autonomous), Ernakulam, in partial fulfilment for the award of the degree of Bachelor of Physics. We further declare that this project is not partially or wholly submitted for any other purpose and the data included in the project is collected from various sources and is true to the best of my knowledge.

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SYNTHESIS AND CHARACTERISATION OF NANOCELLULOSE CRYSTALS FROM PINEAPPLE LEAVES

ABSTRACT

Nanotechnology involves the manipulation of nanoparticles to design and develop nanostructures with new functions and properties. Cellulose is one of the most important biopolymers. Pineapple leaves which are considered to be a post harvest agricultural waste are rich in cellulose and can be used to synthesize nanocellulose. Nanocellulose is of increasing interest due to its wide range of applications in various fields of science and technology. Its excellent mechanical properties, good biocompatibility, interesting optical properties and renewable nature make it one of the best nanopolymers with a wide range of applications. This project reports the synthesis and characterization of nanocellulose crystals. Here nanocellulose was prepared from pineapple leaves using the method of alkali treatment and acid hydrolysis. The particle size of nanocellulose crystals was determined by XRD analysis. The average particle size of nanocellulose crystals were then found using the Debye Scherrer equation. Future scope and further applications of nanocellulose have also been detailed in this project.

CONTENTS

CHAPTER 1

INTRODUCTION

Nanoscience is an interdisciplinary science which deals with study of materials having dimensions in the range of nanoscale. The word Nano is derived from the Greek word "nanos", which means dwarf. Nano refers to one billionth and one nanometre means one billionth of a meter. The nanoscale materials have unique characteristics different from its bulk state and these properties can be tuned to design powerful potential devices. The technology used to control and manipulate matter at atomic level to create large structures with desired properties is called nanotechnology. This field is of great importance since properties of materials dramatically change as we go down to nanoscale. This is due to their large surface to volume ratio, large fraction of surface atoms, reduced imperfections and so on. The surface area of materials at nanoscale is very large compared to the same amount of their bulk counterparts and the fraction of atoms present on the surface of such small structures will be more. Atoms on the surface have a smaller number of neighbour atoms than those at the center. For this reason they have high surface energy. Reduction of size of the materials makes them more chemically reactive and can affect its electrical, mechanical, magnetic, structural and optical properties. At such a small dimension, these properties can no longer be described in terms of classical mechanics and Quantum mechanics comes into play. For example, gold nanoparticles show unique catalytic properties while gold in its bulk state is inert. Dependence of various properties of particles on their size allows us to manipulate them to engineer new properties.

Even though nanotechnology is a relatively new and emerging field, nanostructures were used much earlier. The Lycurgus cup from the British museum is one of the greatest examples of nanotechnology in the ancient world. When lit from outside it looks green and when lit from inside it looks reddish and purple. The explanation of this phenomenon was obtained only in 1990 after scientists analyzed this cup using AFM. It was found that dichroism observed in the cup was a result of the presence of nanoparticles. Besides this, glass windows of medieval cathedrals exhibited unusual optical properties. It is due to the fact that the glass was embedded with nanosized particles. The beginning of nanoscience was officially marked by a lecture by American physicist and Nobel laureate Richard Feynman entitled "There's plenty of room at the bottom". Feynman suggested that in future we would be able to manipulate and control things at an atomic scale.

Nano materials have a wide range of applications in various fields of our life. The discovery of STM, AFM, XRD etc has revolutionized the nano field. Silver nanoparticles are used as antimicrobial agents for treatment of wounds. Nanoparticles are used in cancer treatment for delivering drugs directly into the tumor cells without affecting the healthy cells. Broken bones may take at least six weeks for healing and the process may be long and tedious. Scientists in Japan have found that carbon nanotubes can help to fasten this healing process. Nanotechnology is also used in food packaging and processing industries. Nanoparticles can be used to deliver vitamins and nutrients to food without changing its taste and appearance. Researchers are trying to develop nanocapsules containing nutrients that shall be released when nanosensors detect deficiency of nutrients in our body. Nanotechnology can be used to fabricate powerful electronic devices with less power consumption and improved performance. The field of nano science is influencing every single field of science, in unpredictable ways and yes! Feynman is right, There's plenty of room at the bottom.

Nowadays, green, renewable and sustainable materials and their applications have become increasingly important. These materials can be used as potential solutions for most of the problems faced by our environment. Biopolymers are one among such materials. They are the polymers produced from natural sources, which are gaining worldwide interest, since they are renewable, environment friendly and easily available. Among them cellulose is the most abundant and renewable Biopolymer. They are the chief part of the plant cell wall and help the plants to remain stiff and strong. Cellulose is made of thousands of D-glucose units, which are linked together by β-1,4 glycosidic bonds. This unique structure is the reason for the unique properties of cellulose. This polymer is an inexhaustible raw material for industrial uses since they have the potential to be modified.

Cellulose at the nanoscale is called Nanocellulose. It is a promising bio nanoparticle that has wide range of applications in various fields. Increased demand of this material is due to its unique properties which include biocompatibility, Renewability, biodegradability, excellent mechanical and optical properties. All these properties make nanocellulose high performance renewable materials for advanced applications. Its promising performance and abundance makes nanocellulose a material of great interest. Due to its nanoscale dimensions, Nanocellulose exhibits various advantageous features over bulk materials. Compared to bulk cellulose which contains both crystalline and amorphous regions, nanocellulose crystals have a larger specific surface area, higher surface to volume ratio, greater specific strength, high surface chemical reactivity, high mechanical strength and unique liquid crystalline characteristics. There are three types of nanocellulose- nanocellulose fibers, nanocellulose crystals and bacterial nanocellulose. Nanocellulose can be derived from plants as well as bacteria. Cellulose from plants is typically a mixture with lignin, pectin and hemicellulose, whereas the bacterial cellulose is pure. Cellulose nanocrystals can be obtained from natural sources like rice husk, corn, cotton, pineapple leaves, sisal, wood pulp etc. They may have needle like, rod like, ribbon like, or spherical shape having diameters ranging from 5 to 20 nm and length from 100 nm to $1\mu m$.

Here the cellulose nanocrystals are isolated from pineapple leaves by alkali treatment followed by acid hydrolysis. Pineapple is one of the most loved fruits all around the world. But the leaves of this fruit are considered as agricultural waste, which are available in abundance. The disposal of pineapple leaves after harvest is one of the major postharvest problems faced by the farmers. However, Pineapple leaves which are rich in cellulose can be exploited to extract nanocellulose.

Though acid hydrolysis using sulphuric acid is one of the oldest processes, it remains the most common method of preparation of nanocellulose crystals. It starts with alkali and bleaching pretreatment followed by acid hydrolysis. Pretreatments are done to obtain pure cellulose while the second stage is done for converting cellulose to nanocellulose. The nanocellulose thus synthesized are then characterized by X ray diffraction, a process x rays are exploited to study the crystal structure.

Nanocellulose crystals can be used for various applications. It can interact with various molecules due to high surface to volume ratio and abundance of functional groups. This makes cellulose nanocrystals attractive for sensing applications. Nanocellulose reinforced composites are good options to replace the use of petroleum based products. They have a broad spectrum of applications in various biomedical, electronic and chemical fields, including drug delivery, bioimaging, tissue engineering, reinforcement of functional materials and fabrication of structural color materials. Nanocellulose can be further developed to design high performance organic materials that are environment friendly and cost effective.

CHAPTER-2

SYNTHESIS AND XRD CHARACTERISATION

SYNTHESIS METHODS

There are two major approaches for synthesizing nanoparticles - bottom up method and top down method. In the top down method, the bulk material is finely crushed by mechanical or chemical means to form nanoparticles. This method is a cheap method but is time consuming. In the bottom up method atoms and molecules are assembled into larger nanostructures. This type of fabrication method is expensive but is faster than the top down method.

The fabrication of nanocellulose involves two steps. First one is the pretreatment, which is followed by the mechanical methods. The pretreatment methods can be done either using enzymes or by using alkali. In the method of enzyme hydrolysis, enzymes with the ability of selective hydrolysis are used to degrade lignin and hemicellulose content without disturbing the cellulose content, whereas in alkali treatment, an alkali is used. Most commonly used alkali for this purpose is NaOH. The mechanical methods are carried out to convert cellulose to nanoscale. Different mechanical methods are high pressure homogenization, grinding, cryocrushing, ball milling and chemical hydrolysis.

Here nanocellulose is synthesized by alkali treatment followed by acid hydrolysis. Acid hydrolysis using sulphuric acid is one of the most common methods for the preparation of nanocellulose from pineapple leaves. This method starts with the alkali and bleaching pretreatment of pineapple leaves, which is then followed by acid hydrolysis. Pretreatments are carried out to purify the cellulose whereas the acid hydrolysis is done to bring it down to nanoscale.

CHARACTERIZATION TECHNIQUE

The discovery of x-rays by Wilhelm Conrad Roentgen was a breakthrough for scientific disciplines, making new technological developments and possibilities for research. In 1912 Max Von Lau, a German physicist discovered that x rays can be scattered by atoms in the crystal. Thus crystals can be used as a three dimensional diffraction grating for x rays. This phenomenon was called x ray diffraction. The x ray diffraction technique is one of the precise tool for determining and studying the crystal structure. It requires no sample preparation and is non destructive. X rays diffracted by crystal lattice give a unique pattern of peaks of reflection at different angles and of different intensities. The principle behind x ray diffraction is Bragg's law and it is given by

$$\mathbf{2d \sin\theta = n\lambda} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where n =order of diffraction

θ=angle of diffraction

λ=wavelength of x rays

d=interplanar spacing

Since, sin θ<1,   λ<2d

This implies that diffraction in crystals could be observed only if the wavelength of x rays is comparable to the size of atoms. SInce the wavelength of x rays are nearly 1Å, which are of order of size of atoms, it satisfies the condition for diffraction.

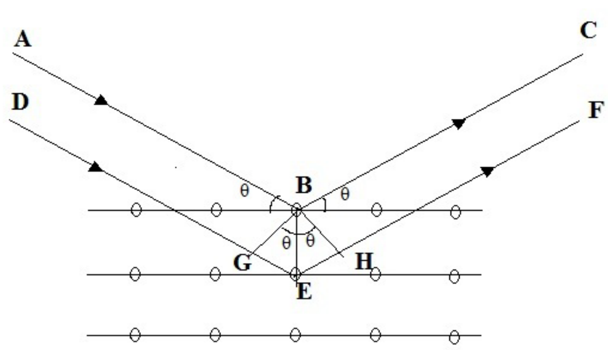

Fig-1 Bragg's law

Let AB be the incident x-ray that strikes the crystal and get reflected along BC from an atom at B and let θ be the glancing angle. Similarly let another x-ray DE which is parallel to AB is incident on the crystal at E and gets reflected along EF.

To find the path difference between the reflected rays

Join BE and draw normals BG and BH as in the figure.

Consider △BGE

Sin θ=GE/BE

GE=BE Sin θ

GE= d sin θ ………………………….(2)

Consider △BHE

sin θ= EH/BE

EH= BE sin θ

EH=d sin θ……………………………..(3)

The path difference between BC and DE = GE+BH = d sinθ+ d sinθ =2d sinθ…….(4)

According to the condition for constructive interference, the reflected rays will undergo constructive interference if the path difference between any two consecutive rays is an integral multiple of x-ray wavelength λ.

Path difference = n λ.……………………(5)

From equation 3 and 4 ,we can write

2d sinθ = n λ.……………………………(6)

This equation is called Bragg's law. This law describes the general relationship between the wavelength of incident x rays, diffraction angle and the spacing between crystal lattice points. The x rays can penetrate the solid without disturbing their structure. The diffracted x rays from different crystal planes give information about internal atomic arrangement and the lattice parameters of the crystal. The position of the diffraction peak is determined by the unit cell of the crystal and each peak represents each lattice planes. Amorphous materials, which do not have a long range order, cannot produce any significant peak in the diffraction pattern. The crystallite size of particle is calculated using the Debye Scherrer formula, which is given by

$$D=\frac{K\,\lambda}{\beta cos\theta}\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(7)$$

Where D = crystalline size of particle

K = *Scherrers constant (0.98)*

λ= x ray wavelength

β = full width at half maximum

CHAPTER 3

MATERIALS AND METHODS

MATERIALS

Pineapple leaves, NaOH solution, Hydrogen peroxide (H_2O_2), Sulphuric Acid (H_2SO_4), Distilled water.

Preparation of nano cellulose from pineapple leaves

Alkali Treatment:

Nanocellulose isolated from pineapple leaves is a mixture of pectin, lignin, hemicellulose and other compounds in smaller quantities. So to remove all those mixtures, shade dried pineapple leaves are powdered. 5g of powdered pineapple leaves are then weighed in a weighing balance. 5g of NaOH pellets is taken in 100 ml of distilled water and is placed on the magnetic stirrer so that the NaOH pellets are dissolved in the distilled water. A magnetic bead is used to stir the solution. This magnetic bead is then cleaned with acetone. After that in a beaker 5g of powdered pineapple leaves is dissolved in the NaOH solution (5%) and this beaker is placed in a vessel with water on the magnetic stirrer. A magnetic bead cleaned with acetone is used to stir this solution at 70° C of temperature for 2 hrs. After that the sample is washed several times with distilled water by centrifugation and filtered till the pH level is neutral. Then it is dried in the oven at 60°C for 2hrs.

Bleaching:

In a beaker, 3g of Alkali treated sample is mixed with 30 ml of NaOH solution which was made by dissolving 4g of NaOH pellets in 100 ml of distilled water and this is mixed with 30ml of Hydrogen peroxide(H_2O_2) and is placed on the magnetic stirrer for 2hrs at 50°C of temperature. The treatment is carried out until the sample becomes white in color. Then the sample is washed several times with distilled water by centrifugation and filtered till the pH level is neutral. Then it is dried at 100°C of temperature in the oven for one and half hours. Bleaching is done to increase the cellulose content and to remove the left out lignin and hemicellulose.

Acid Hydrolysis:

Now we have the microcellulose. 1.0024 gm of microcellulose is weighed and this is mixed with 9.2 ml of sulphuric acid (H_2SO_4) and 10.8 ml of distilled water and it is then stirred by placing it on a magnetic stirrer at 50℃ of temperature for 2 hrs. Then 40 ml of cold water is added to the reaction mixture to stop this reaction and centrifuged several times with distilled water to remove the excess acid . Then it is dialyzed for 5 days to neutralize and eliminate the sulfate ions. And it is dried in an oven for one and half hours at 100°C . Finally, the Nanocellulose crystals are synthesized.

Fig 2- Steps in the synthesis of Nanocellulose

Alkali treated pineapple leaves

Bleached pineapple leaves

Acid Hydrolyzed pineapple leaves

CHAPTER 4

RESULTS AND DISCUSSIONS

The alkali treatment was performed to remove lignin, hemicellulose, pectins, waxes and other substances .Then bleaching was done to increase the content of cellulose in the sample. The change in color of the sample indicates the removal of lignin, hemicellulose, pectin etc. The white color shows the high cellulose content in the sample. X-ray diffraction was done to investigate the crystalline behavior of the sample. The pattern contains crystalline peaks. The presence of high cellulose content was verified by the presence of these peaks. These characteristic peaks correspond to crystalline planes of (101), (002), and (004) and these peaks indicate the crystal structure of nanocellulose crystals.The peaks obtained at 2θ of around 18°, 22° and 34°, were compared with the JCPDS data(JCPDS no. 00-050-2241) and was found to be in agreement.

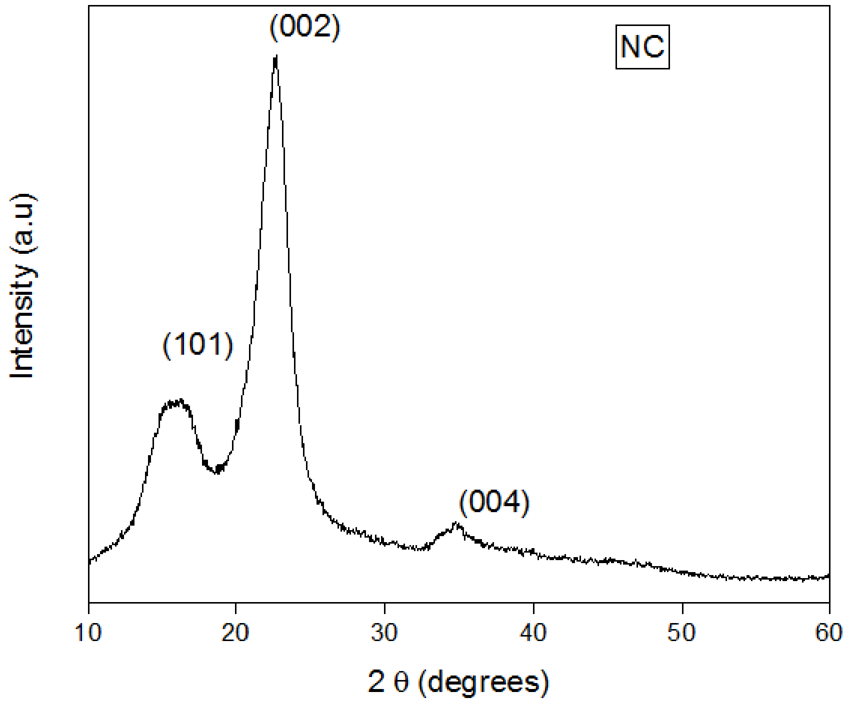

Fig 6

XRD Pattern of Cellulose Nanocrystals.

The crystallite size of the particle is calculated using the debye Scherrer equation.

$$D=\frac{K\lambda}{\beta cos\theta}$$

where D=crystallite size of the particle
K=Scherrers constant, which is approximately equal to 0.92
λ=Wavelength of x ray
β= Full width half maximum

Table 1

2θ (Degree)	θ (Degree)	θ (Radians)	β (Degree)	β (Radians)	Particle size $D=\frac{K\lambda}{\beta cos\theta}$ (nm)
16.05599°	8.027995°	0.14004	5.187	0.09048	1.565nm
22.40449°	11.202245°	0.1954169	3.27909	0.057202	2.52nm
					Average particle size=2.0425nm

<u>Substitutions and Calculations.</u>

The wavelength of x ray λ = 1.54178 Å
K= 0.92
2θ= 22.40449°
θ= 11.202245°= 0.1954169 rad
β= 3.27909° = 0.057202rad
D= K λ /β cosθ = 2.52 nm

The average particle size D = $\frac{1.565+2.52}{2}$ =2.0425 nm
Thus the particle size was determined from the XRD analysis.

CHAPTER 5

APPLICATIONS AND FUTURE SCOPE

The demand for green renewable environmentally friendly materials is increasing day by day to produce high performing materials which have low or no negative impacts on our environment. In such a context cellulose is a material of growing interest. Nanocellulose are bio nanomaterials with unique mechanical and physical properties which makes them the most attractive renewable material for advanced applications. They exhibits excellent properties such as high surface to volume ratio, high young's modulus, biocompatibility, renewability and so on. These amazing properties of nanocellulose crystals can be tailored to develop powerful and cost effective devices. Through surface modification and by manipulating its unique properties, nanocellulose crystals can have a wide variety of applications in numerous fields of our lives, which may include bioimaging, sensing, drug delivery, tissue engineering, manufacturing of structural color materials, food packaging, reinforcing paper and so on. It can be used as a potential replacement of plastics in the near future.

One of the most promising applications of nanocellulose crystals is the fabrication of sensors. Owing to the presence of abundant highly reactive functional groups on their surface, they are highly sensitive and readily react with molecules around them. Hence they can be used to design sensors. Nanocellulose crystals can form composites with other materials for sensing gas, chemicals, proteins, enzymes etc. Nanocellulose based sensing devices can be used for sensing pollutants in the atmosphere, poisonous substances in food, etc. Polyaniline-cellulose nanocrystal based sensors are fabricated for detection of ammonia gas. The electrical resistance of these composites were found to increase in the presence of ammonia, which was a result of deprotonation of aniline. When exposed to ammonia, Polydiacetylene based sensors were reported to change its color from blue to red. But this did not work at low temperatures to be applied on food industry. However when cellulose nanocrystals were incorporated into polydiacetylene based sensors, the sensitivity of sensors was found to be improved.It formed more sensitive ammonium sensors even at very low temperatures. NO_2 is a harmful gas. Cellulose nanocrystals -iron oxide composite based sensors are ideal for the detection of NO_2.Biosensors can also be developed using nanocellulose. An example of a biosensor based on cellulose nanocrystals is the sensor which is used for the detection of HNE (Human Neutrophil Elastase detection). Cellulose nanocrystals conjugated with a peptide can be used for detection of HNE. HNE hydrolyses the tripeptide which is covalently attached to cellulose nanocrystals and then releases a colored dye which can be detected via spectroscopy to determine the amount of HNE present in the system. These tripeptide-cellulose nanocrystal sensors are more sensitive towards HNE than tripeptide-paper sensors. This is due to the fact that the surface area of cellulose nanocrystals was much larger than that of cellulosic paper which is used in tripeptide paper sensors.. Among the nanocellulose types cellulose nanocrystals have been receiving more interest due to their superior characteristics such as possession of abundant hydroxyl groups,high surface area and high mechanical strength.

Another interesting application of nanocellulose is in the food packaging industry, which is currently dominated by the use of plastics. Cheap packaging has been possible since the birth of plastics and now it is the need of the hour to replace the plastic by green biodegradable materials. Biopolymers like cellulose can be considered as a good candidate for this purpose.Nanocellulose is one of the most interesting plant biodegradable polymers that is available in abundance and has the potential to replace plastics. Owing to the distinctive properties of nanocellulose like tunable surface chemistry, barrier properties, mechanical strength ,crystallinity and non toxicity, it is an emerging material for food packaging applications. Nanocellulose crystals can be corporated into various polymers for making green composites and can improve the properties of those materials. Plenty of hydroxyl groups on the surface of nanocellulose allows its chemical crosslinking with other polymers, which increases the strength of such composites further. Nanocellulose also has the ability to form hydrogen bonds that form a dense network that impedes various gas molecules to pass through it thereby maintaining a low oxygen atmosphere. Hence it can act as an oxygen barrier when used in food packaging. Oxygen barrier is necessary for food packaging as it can spoil the food.

Nanocellulose crystals can be used as a potential candidate for green electronics. Nanocellulose offers the fabrication of environmentally friendly and cost effective electronic devices including sensors,actuators, memory devices etc. It can be used as a reinforcing filler in electronic devices where strength is a factor. It exhibits excellent strength, stiffness and high mechanical properties. It is widely used in transparent electronics due to its excellent optical properties. The optical properties of nanocellulose can be manipulated to fabricate liquid crystals devices and electronic displays. Low thermal expansion of nanocellulose is another important property which makes it desirable for heat generating devices. They are also used in the field of energy storage devices like supercapacitors, batteries and fuel cells etc, owing to their high conductivity and large surface area. The higher conductivity of cellulose nanocrystals is due to the large number of charge carriers and acidity of sulphuric acid introduced during its extraction process.

These are only some of the applications of nanocellulose. The studies are going to explore more and more applications of nanocellulose. We expect that nanocellulose-based materials will certainly improve the people’s quality of life in the future .Its unique properties makes it an excellent material for a wide range of applications..

CHAPTER 6

CONCLUSION

In this study, eco-friendly cellulose nanocrystals were successfully prepared from Pineapple leaves which is an agro-industrial waste. The method adopted for the isolation of nanocellulose was found successful. This work studied nanocellulose extraction processes by alkali treatment, bleaching and acid hydrolysis. To remove pectin, lignin, hemicellulose and other smaller compounds Alkali treatment is done. By Bleaching method the cellulose content is increased and the left out lignin, pectin and hemicellulose in the alkali treatment were also removed and microcellulose is obtained. By Acid hydrolysis microcellulose becomes nanocellulose, that is the size of nanocellulose was reduced into nanoscale. The XRD result showed an increase in crystallinity which is a clear indication of removal of lignin and hemicellulose that were present. The particle size of nanocellulose crystals were also calculated from the x-ray diffraction pattern

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