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Experimental Investigation On Corn Cob Nano Composites In Terms Of Aspect Ratio And Thermal Degradation

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One agro-industrial trash that could be handled more effectively is great numbers in many nations, notably India. This study assesses the viability of using corncob (CB) as a cellulosic base to produce nanostructures through acidic hydrolysis, a substance that can be used as a reinforcing ingredient for creating nano-composites (NCS). 15 Ml of H_2SO_4 was used during every gram of cellulosic all over the hydrolysis, which took place at 60 °C for 40, 80, and 120 minutes. After only 45 minutes of hydrolysis, CB cellulose nano-crystals (cellulose-Nc's) showed excellent temperature resilience and reinforcing capability. NCS/ polyvinyl alcohol (PVOH) hybrids being supplemented with NCS120 nano-particles (NP), which have these nano-crystals (NC) have promised potential reinforcements because to their favorable proportions and temperature resistance. NCS 120 has shown the highest L/D ratio 55.21 and highest thermal degradation value as 264^{0}C when compared with NCS80 and NCS 40.

Key words: agro-industry, corncob, cellulose, nano-structure, nano-composites, nano-particle, tensile, nano-crystal.

1. INTRODUCTION

One of the many useful polymers included in plant bio-mass is cellulose, which means it is also cheap, reusable, sustainable, as well as environmentally benign. Cellulose nano-particles are made from residues such as grain from rice and kernels of corn and are resilient, stiff, and hydrophobic. These tiny cellulose molecules may find use in multifunctional ingredients, transmembrane technological advances, materials made of composites medical care, water purification, and industrial settings. Corn cobs, which are high in cellulose and lignin, can be utilized to make greener substances and petroleum that may have antioxidants plus absorbent UV qualities. Because of ecological worries, ecological materials consisting of garbage, reused components, and bio-sources are becoming more and more popular. The creation of more environmentally friendly polymer alternatives having equivalent structural and thermo qualities has come about from the growing worldwide reliance on polymers derived from hydrocarbons. Research has looked at modifying polypropylene (PP) using bio-carbon (BC) to enhance efficiency and lessen its effect on the natural world. Originating from 1, 4 diamino butane generated from hydrocarbons and spontaneously existing biodegradable sebacic acids, PA 4, 10 is a unique class of polyamide that is 72% biodegradable. This kind of polymer preserves polymer durability whilst using fewer hydrocarbons. Studies using BC, which is a byproduct of pyrolysis (PYS), were carried out to investigate the deterioration of conventional organic fibres. Burning biological materials produces bio-oil, syngas, and BC through the process of PYS.

The heat during pyrolysis, the raw material, and the duration of stay all affect the structural characteristics of BC. One possible feeding resource is corn cobs, which have a bio-based composition, and cheap manufacturing expenses, with a minimal ecological effect. The leftovers may be utilized as substances within vibrant composite objects. CB'S are pyrolyzed to form BC an agent that sequesters carbon and absorbs nutrients. Because of its reduced ashes amount and better BC rates at moderate pyrolysis conditions, it is appropriate for large-scale usage. Its substantial quantity of unstable stuff makes it a promising filling ingredient for green hybrids. Studies on the potential it is incorporated remain scarce. The utilization of CB residue for the sustainable synthesis of nano-cellulose and its application in bio composites has been extensively studied in recent years has attracted a lot of interest lately because of the possibility to lessen ecological pollution and promote sustainable development. Researchers have explored various methods for extracting nano-cellulose from corn cob residue, including enzymatic hydrolysis, mechanical grinding, and ultra-sonication. CB residue is an abundant agricultural waste that poses significant environmental concerns due to its disposal and burning, which releases harmful greenhouse gases. Nano-cellulose, a biodegradable and renewable nano-material, has shown great promise as a reinforcement material in bio composites because of his special qualities, like his tremendous endurance stiffness, and thermal stability. The extracted nano-cellulose has been characterized and found to have high crystalline, high aspect ratio, and good thermal stability. Several studies have investigated the use of nano-cellulose from corn cob residue as a reinforcement material in biodegradable polymers, such as Polylactic Acid (PLA) and poly-hydroxy-alkanoates (PHA). The findings suggest the inclusion of nano-cellulose enhances the mechanical and thermal attributes of the composites. Other research has looked into the application of nano-cellulose from corn cob residue in biodegradable packaging materials and bio-plastics.

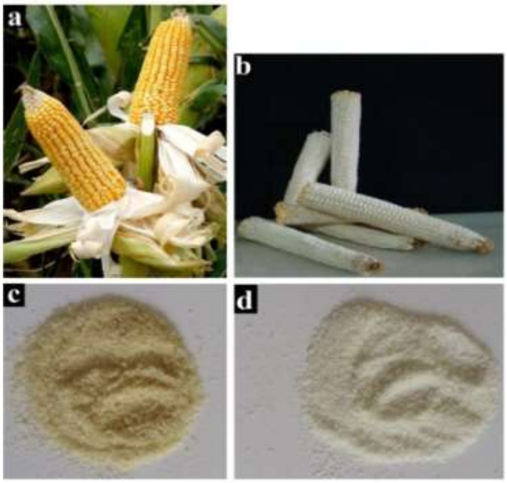

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Fig.1 (a) CB (b) CB prior to (c, d) further purification [6]

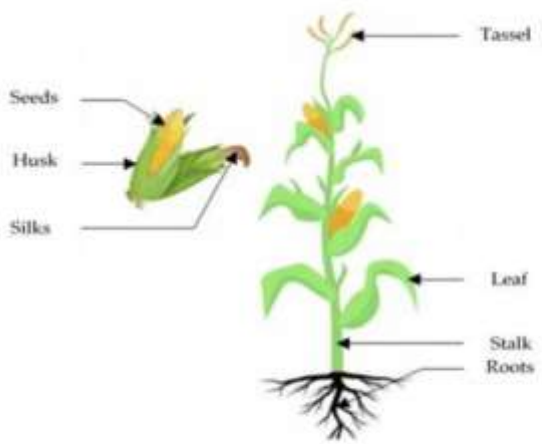

Fig. 2 Several CB components [10]

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Table.1 Attributes of CB Starch [10-15]

Component	Quantity
Density (g/cm^3)	1.4029–1.356
Melting point (C)	256 ^{0} –258^0
Boiling point (C)	205^0
Amylose (g/100g)	29.4–24.64
Amylopectin (g/100g)	75.36–72

Crude fats (g/100g)	7.13–0.32
Crude proteins (g/100g)	7.70–0.38
Ash (g/100g)	0.62–0.32
Phosphor (%)	0.09
Moisture (%)	10.82–10.45

Table.2 Attributes of CB Plant Components [16-28]

Component	Stalk	Hull	Husk	CB	Unit
Density	1.42	1.3231	1.49–1.18	0.8–1.2	g/cm^3
Porosity	58.51	-	88 ± 2	67.93	%
Cellulose	32–39	15.3	31.3–47	40–44	%
Hemi-cellulose	29.1–42	40.4	34–43.91	31–33	%
Lignin	5–38. 12	2.1–2.87	1.5–14.3	16–18	%
ASH	24.9	0.88–1.3	3.3–6.8	2.3–3.2	%
Moisture	3.32–6.4	4.2–8.59	7.6–8.7	6.38	%

CB'Scan now is used to produce nanocellulose substances according to a novel extraction technique created by Ismail et al., Sulfuric acid levels, ambient temperature processing, and mechanical treatments with ultrasound equipment are all part of the process. The concentrations with crystallinities of the resulting nanocellulose are excellent, whereas the resulting fiber NP range in size between 15.3 nm to 2.1 nm. The technique may effectively and economically recover nanocellulose from corncob waste in benign circumstances.

Sandeep Gairola et al. carried a novel processing method for turning corn, as well as maize stalks into nano-cellulose materials, was introduced. Sulfuric acid amounts, ambient temp processing, and physical therapy using ultrasound equipment are all part of the procedure. Excellent rates, as well as crystallinities of nano-cellulose, are obtained, while the ensuing fabric nanostructures span in dimension from 15.3 nm to 2.1 nm. The process is a potentially reusable material since its production is economical, environmentally benign, and retains its original quality .Ethan Watt et al., worked on a biodegradable BC infill made of maize cob have been utilized to create Polyamide 4, 10, a reusable polyamide. Supplement durability under elevated temps is enhanced by PYS and 76% of the content in bio-composites is present. Despite increases in tensile strength and heating bending heat, bio-composites retain comparable structural and thermal characteristics. This might lessen the need for polyamide materials derived from hydrocarbons. To comprehend PA 4, 10's capabilities, the current study has concentrated on applying bio-composites to a variety of sectors, notably the manufacturing of automobile parts. Chun, K et al., investigation found that the oil from coconuts has been utilized to create a cocoa butter connector (CCBC), a novel biological bridging reagent. The investigation looked at how the related to machinery, heat, and architectural characteristics of CB full PLA environmentally friendly combinations were affected by the filling amount plus CCBC. The addition of CB reduced mechanical toughness along with extension upon breakage however enhanced rigidity modulation, according to the findings. These features were boosted, the glassy threshold was rose, and the crystallization procedure was done using CCBC. Yiling Wan et al., worked with wood ash (WA), polypropylene (PP), and CB grain were used to make this combination. Malefic grafting PP was utilized as a compatibilizer, while WA was utilized as the strengthened filler material. The inclusion of WA enhanced the elastic and tension moduli despite increasing humidity digestion, according to the findings BC is solid filler with low cost that may find use in bio-composites. Up to 76% more bio content and compound characteristics can be obtained using it. This work examined PA 4, 10/BC bio-composites at various load values and examined their shape, framework, toughness, as well as thermal order to describe the impact of decomposition frequency on cornfield corn of corn BC. Hudson Alves Silverio et al., researched on the utilization of corn cobs as an origin of cellulose to make nano-particles for nanotechnology is investigated in this work. Employing H_2SO_4, the hydrolysis procedure was carried throughout 30 to 60 minute at 45°C. The resultant cellulose nano-crystals are thermally stable, morphologically uniform, and crystalline. The nano-particles (NCS120) exhibited a greater capacity for reinforcement after a 60-minute retrieval period. Junjie Guan et al., investigated on the structural and chemical characteristics of blown foamy compounds comprised of cellulose/corn cob and starch acetate are assessed in this investigation. The practical features of builds produced by blending starches acetic plus cellulose, CB'S, and varying levels of ethanol were contrasted. The findings suggest that whereas cellulose does not affect the axial growth of cornstarch acetate bubbles, CB mixes can. Increased compressive capacity is correlated with increased corncob percentage.

Pradeep Kumar Gandam et al., research exposed that because corncob lignocelluloses has fewer ashes plus mineral extraction but is richer in xylan, it is a promising resource for bio-refinery chemicals. Up to 99% delignification, 100% hemicellulose solubilization, and 95% cellulose extraction can be obtained with preprocessing techniques. Xylooligosaccharides, ethanol, and foundation glucose are examples of bio-chemical frameworks generated from CBS. Both established and novel preprocessing techniques are applied, and information from the SCOPUS library about corncob preparation is examined. Chan Ming Yeng et al., effects of chemical alteration and infill concentration on the related to machinery, heat, and morphological characteristics of CB bio-composite materials were investigated in this work. Mechanical and heat characteristics were enhanced by the addition of acrylic acid, which also enhanced interpersonal contact. With the use of FTIR and penetrating optical microscopes, the connection was verified. Barbash et al., investigated the cellulose and nitro-cellulose (NC) separation process using the globe's biggest grain agricultural product, corn crop residues (CCR). Researchers discovered that per acetic acid was used in an eco-friendly technique to produce NC. According to the research, the NC nano-particles were multiple microns long and possessed a longitudinal dimension of 5-65 nm. NC may minimize the use of chemical supplementary compounds and enhance carton indications. In their melted mix investigation, Moran et al., discovered that PA 4, 10 and PA 6, 10 were soluble and outperformed homo-polymer materials. Additional research recommends increasing diffusion and retention modulation, eliminating volatile elements, and adding infill's. The large resolution and increased strengthening impact of cellulose nano-tunes NTs, as demonstrated by Otaegi et al., investigations, boosted nano-filler distribution along with electronic and biomechanical characteristics when a soluble PA 6/CNT master-batch, or was incorporated with a PA 4,10 matrices. Husseinsyah S et al., The mechanical toughness and suppleness of bio-composites composed of polypropylene and CB were the main subjects of the investigation. These characteristics were reduced when CB was added, whereas the durability, as well as elasticity of compatibilizers, was increased. Overall, the literature suggests that CB residue is a promising source of nanocellulose for sustainable synthesis and application in bio composites.

Methodology:

Collection and Processing of CB Residue: Corn cob residue was collected from local farms, cleaned, and processed to remove impurities. The residue was dried and ground into a fine powder using a high-speed grinder.

Extraction of Cellulose Nano-fibers: A novel eco-friendly method was employed to extract cellulose nano-fibers from CB residue. The methods involved- Mechanical grinding using a high-speed grinder, Enzymatic treatment using cellulose enzymes (10% w/w) at 50°C for 2 hours. , Ultrasonication using a high-frequency ultrasonic processor (20 kHz, 100 W) for 30 minutes.

Description of Nano-cellulose: The taken out nano-cellulose was described by means of Transmission electron microscopy (TEM), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and Thermogravimetric analysis (TGA) and Dynamic light scattering (DLS)

Preparation of Bio-Composites: The extracted nano-cellulose was incorporated into bio-composites using a biodegradable polymer matrix at various loadings (5%, 10%, 15% w/w).

Evaluation of Bio-Composites: The qualities that are chemical, heat, plus physical of the bio-composites were evaluated using Tensile testing (ASTM D638), Dynamic mechanical analysis (DMA), Thermogravimetric analysis (TGA), scanning electron microscopy (SEM) and Water absorption testing (ASTM D570).

Extraction Methods

Several methods have been explored for extracting nanocellulose from CB residue, including:-Enzymatic hydrolysis- This method involves the use of enzymes to break down the cellulose in CB residue. Mechanical grinding: - this method involves the use of mechanical forces to extract nanocellulose. Ultrasonication: -this method involves the use of high-frequency sound waves to extract nanocellulose.

The properties

The extracted nanocellulose has been characterized and found to have high crystallinity, high aspect ratio and good thermal stability. These properties make it an ideal reinforcement material for bio- composites.

High crystallinity: - Nano-cellulose from CB residue has been found to have high crystallinity, which makes it suitable for reinforcement in bio-composites.

High aspect ratio: nano-cellulose from CB residue has been found to have a high aspect ratio, which makes it suitable for reinforcement in bio-composites.

Good thermal stability:- nanocellulose from CB residue has been found to have good thermal stability, which makes it suitable for reinforcement in bio-composites.

Applications

Several studies have investigated the use of nanocellulose from CB residue as a bolstering material in bio-degradable polymers: - such as polylactic acid (PLA) and polyhydroxyalkanoates (PHA). The findings point to the inclusion of nano-cellulose enhances the mechanical and thermal attributes of the composites. Bio-Plastics:-nanocellulose from CB residue has been used as a reinforcement material in bio-plastics. Biodegradable polymers, biomedical applications, water treatment, textile industry and biodegradable packaging materials.

Advantages of Nano-Cellulose from CB Residue

Nanocellulose from CB residue has several advantages, including: renewable and bio-degradable, abundant and low-cost feedstock, high strength and stiffness, good thermal stability and Can be used as a reinforcement material in biodegradable polymers.

2 Experimental

2.1 Materials and methods

Corncobs from the local market were utilized in the investigation, alongside agents such as H2SO4 (90- 95%wt), PVOH (MW 80,000–90,000) NaOH, KOH, NaClO2, CH_3COOH (of technical grade 85) cellulose membrane (CM), powder was employed in the aggregates' creation.

2.2. The process of creating cellulose NC’s

2.2.1. Sanitation

At first, a mixer was used to grind unprocessed CB so that it could go across a 30 correspond sieve. Subsequently, the CB had been exposed to a water-based concentration of 5% (w/w) NAOH for 3 hours at 80°C. After being mechanically stirred and repeatedly cleaned using till all of the alkalizes had disappeared followed by drying in a furnace with airflow for 36 hours at 25°C. Following this procedure, the yarns are cleaned using a mix containing equivalent volumes (v: v) of aqua chlorine (1.5%wt $NaClO_2$ in waters) and acetate buffer (20 g NaOH and 80 ml CH_3COOH, concentrated to one liter of pure waters). For 5 hours, the whitening procedure took place at 60°C. After being stained, the fibers' are periodically cleaned in a purified solution before their pH reaches neutrality.

They were then processed using an air-circulating kiln beginning at 60°C over 36 hours (de Rodriguez et al., 2006; and Siqueira et al., 2010a). During all of these chemical-based interventions, the fibre concentration ranged from 2 to 8% (w/w). The processed CB (PCB) represented the substance that remained post-cleansing**.**

2.2.2. Purification of nano-crystals of cellulose (NCOC)

Following the chemical therapy, the PCB is ground in a mixer, run over a 30-netting screen, and subsequently utilized in the alkaline hydrolyzed method of extracting nano-materials (NM). The process of hydrolysis is out for 120 minutes at around 60°C with aggressive continuous agitation. We utilized 10 ml of H_2SO_4 per gram of PCB. This hydrolysis process was halted right away by diluting the solution multiple times using a chilled liquid, and then the surplus acidity was removed by centrifuging the mixture for 20 minutes at 5000 rpm. Following that, non-reactive sulphate categories, minerals, and accessible starches were treated out of the precipitation using normal water till a pH level of zero was attained, which took around 2 days. Afterwards, during the kidney transplant procedure, the solution was ultrasonically treated over 30 minutes and kept at 2 °C in the fridge. A few $CHCl_3$ droplets have been applied on top of the PCB frame as a protective layer. The NCS- NC has been identified as NCS40, NCSC80, and ncs120 based on when the removal is done.

2.3. Making NC ready

To standardize the placement of NCS'S in the mixture, PVOH 3% (w/v) aquatic concentrations were incorporated using NCS40, NCS80 and ncs120 watery cancellations, and then ultrasonic therapy was applied lasting 10 minutes. The NCS40 to PVOH gravity proportions remained at 3:97, 6:94, and 9:91, correspondingly.NCS/PVOH hybrid flicks have been produced via molding at about 25 °C over 36 hours in an air-circulating oven, incorporating a 2-8% NCS charge plus a clean PVOH sheet. Every film had an ultimate mass of 0.8 g. Before examination as well as evaluation, the PVOH sheet with NCS/PVOH hybrids is kept in vacuumed sacks.

2.4 Characterization

Morphology: The morphology of nanocellulose has been studied using techniques such as TEM and SEM.

Crystallinity: The crystallinity of nanocellulose has been studied using techniques such as XRD and FTIR.

Thermal Properties: The thermal properties of nanocellulose have been studied using techniques such as TGA and DSC.

2.4.1 Gravimetric analysis

A portion of the NCS suspension with a specified quantity was dried at 100 °C for 24 hours in an air-circulating kiln to determine the digestion output.

2.4.2 Chemical composition

The TAPPI T13M-54 conventional technique had been used to quantify the lignin concentration. Browning (1967) reported that the acid chlorite method was used to measure the level of holo-cellulose (alpha-cellulose + hemi-cellulose); the -cellulose concentration had been determined by processing the holo-cellulose using KOH concentrations as 10 and 30% (w/w); the hemi-cellulose concentration being discovered simply deducting the holocellulose portion using the -cellulose portion. level; the % has been utilized to calculate the amount of ash disparity among the spun fibre's starting mass and the 6 hours of boiling at 600°C Trindade et al., 2005). For every, a mean from 3 repetitions was established.

2.4.3 Characterizing Heat

Heat stability of NCS40, NCS80 and NCS120 was assessed via the application Shimadzu TGA. DTG-60H apparatus. The parameters for the experiment include an N_2 atmosphere using the 20 ml per minute stream and a warming velocity of 5°C per minute. Specimen weight among five grams and 800°C, through thermal variation among 35 through Al skillet with 10 mg.

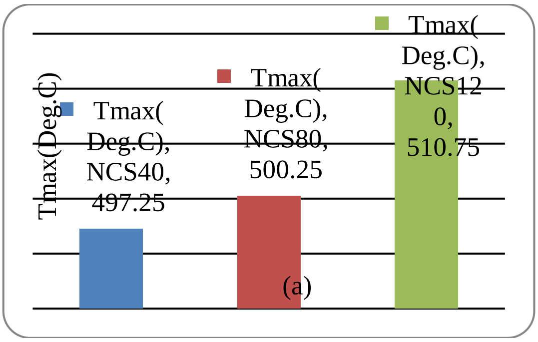
2.4.4 SEM Analysis

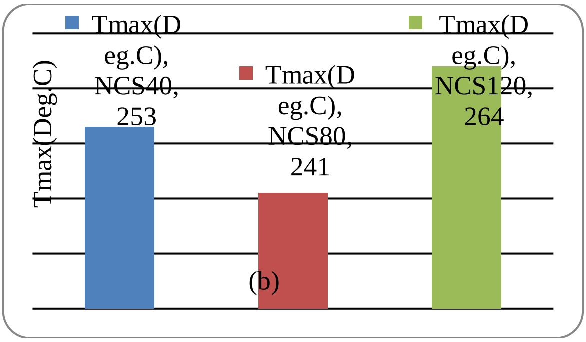
NCS's appearance was assessed using SEM under JSM-7500F microscopy. After applying a covering of the carbon (C) between 0.5 and 5 nm in thickness the specimen was subjected to an accelerated voltage between 0 and 12 kV. Optical imagery analysis was used to identify the size of NCS.

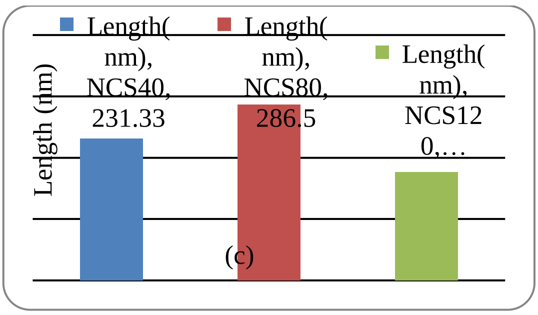
2.4.5 AFM Analysis

SPM-9600 instrument was used to conduct AFM investigations to assess the geometry. On a recently cleft mica terrain, a small amount of a concentrated nano-scale aquatic solution was applied, and it was allowed to airflow dry. AFM pictures have been taken in the active phase at ambient temperature, employing Si points that had a curved perimeter of less than 12 nm and a springing variable force of 32 N m^{-1}. The scans of the scan had been set to 0.5 Hz.

Results & Discussions


Tmax(Deg.C)
Tmax(Deg.C), NCS40, 497.25
Tmax(Deg.C), NCS80, 500.25
Tmax(Deg.C), NCS120, 510.75
(a)


Tmax(Deg.C)
Tmax(Deg.C), NCS40, 253
Tmax(Deg.C), NCS80, 241
Tmax(Deg.C), NCS120, 264
(b)


Length (nm)
Length(nm), NCS40, 231.33
Length(nm), NCS80, 286.5
Length(nm), NCS120,…
(c)

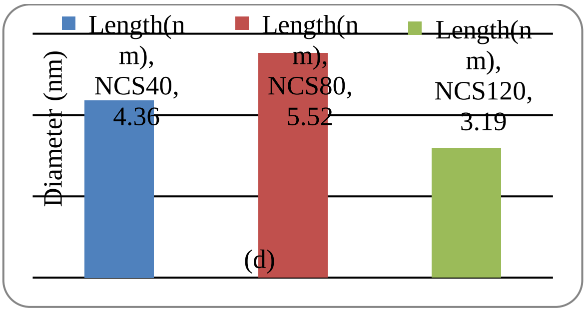


Fig.3a Temperature Distribution among the NCS water evaporation basis

(b) Thermal degradation (c) Length of NCS (d) Diameter of NCS

With reference to the Fig 3a NCS120 has shown the highest temperature distribution as 517.5^{0}C, the lowest temperature distribution as 497.25^{0}C in case of NCS80 was noticed. From Fig 3b on the basis of thermal degradation NCS120 has shown the highest temperature distribution as 264. ^{0} C, the lowest temperature distribution as 241^{0}C in case of NCS80 was noticed. From Fig 3c the highest length of yield was observed for NCS80 as 286.5 nm, where as the lowest length of yield was observed for the NCS120 as 176.133 nm. From Fig 3d NCS 80 has shown higher diameter in size as 5.52 nm, where as NCS120 as shown the lowest diameter as 3.19 nm. The L/D ratio is highest for NCS120 as 55.21 where as the L/D ratio is lowest for NCS 80 as 51.90.

Conclusion

This study demonstrates a sustainable approach to synthesize nano-cellulose from corn cob residue and its application in bio composites. The developed bio composites offer a promising alternative to traditional materials, reducing environmental impact and promoting a circular economy. The study suggests that corn cob residue is a promising source of nano-cellulose for sustainable synthesis and application in bio composites. Further research is needed to optimize the extraction methods and explore new applications for nano-cellulose from corn cob residue. The bio composites showed improved thermal stability.

Challenges and Limitations

Despite the advantages of nano-cellulose from corn cob residue, there are several challenges and limitations, including: extraction methods can be time consuming and costly, nano-cellulose can be prone to aggregation, can be difficult to disperse in polymers and limited understanding of the properties and behavior of nano-cellulose from CB residue.

Future Research Directions

Future research directions include: optimizing extraction methods to reduce time and cost, developing methods to enhance dispersal as well as inhibit agglomeration, investigating the properties and behavior of nano-cellulose from CB residue and exploring new applications for nano-cellulose from CB residue.

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