

Extraction and Characterization of Nanocellulose from Eragrostis Teff Straw

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Abstract

This work presents the extraction and characterization of nanocellulose from agricultural waste (Eragrostis Teff) straw. The proximate analysis of the straw was done with three triplicates and the average value was recorded. The moisture, ash, volatile matter, and fixed carbon content were 6.4%, 5.1%, 74.2%, and 15.6%, respectively. The extractives, lignin, hemicellulose, and cellulose content were found to be 8.5%, 17.5%, 29.5%, 36.5%, respectively. The cellulose was extracted from teff straw by hot water treatment, acid-chlorite delignification, and alkaline hydrolysis process respectively. The values of process parameters such as temperature, time, and sodium hydroxide concentration for alkaline hydrolysis were 57.6°C, 1hour, and 4(w/v) % respectively to obtain a cellulose yield of 78.7%. The nano cellulose particles were extracted from the cellulose by acid hydrolysis process (48%v/v sulfuric acid) at 35°C for 30 min and the yield was 42.8%. The characterization of nano cellulose was done by using Fourier-transform infrared spectroscopy, dynamic light scattering, x-ray diffraction, and thermogravimetry analysis/ derivative thermogravimetry analysis to determine the functional group, particle size distribution, crystallinity, and thermal stability respectively. The Fourier-transform infrared spectroscopy result shows that the reduction and total removal of lignin, hemicellulose, and other amorphous parts found in teff straw. The average particle size was found to be 101.6 nm with a polydispersity index of 0.047. The crystallinity index of teff straw and nano cellulose was 47.7 and 77.1% respectively. The thermogravimetry analysis shows the thermal stability of the nanocellulose sample was lower than that of cellulose and teff straw samples.

Keywords: Teffstraw, Nanocellulose, Alkaline hydrolysis, Acid hydrolysis

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1 Introduction

Nanoscience and technology is a broad and interdisciplinary area of research that has been growing explosively worldwide in the past few years and nanomaterials are cornerstones of this area. Nano-scale materials are defined as a set of substances where at least one dimension is approximately in a nanometer range[1]. Nano-materials are used for different applications because of their unique optical, magnetic, electrical, and other properties. In recent years, different nanomaterials such as carbon-based nanoparticles, ceramic nanoparticles, and metal nanoparticles were developed and used for different applications. However, the need for non-renewable sources such as fossil fuel to produce these materials remains a challenge because of this; research progression in environmental science continues to push for materials that are renewable, biocompatible, and less toxic[2]. The extraction of nanoparticles from renewable resources such as lignocellulosic material, bacteria, and tunicate is attracting the attention of researchers.

Nanocellulose is one of the promising nanomaterials that can be extracted from lignocellulosic materials. The properties of nanocellulose make it an interesting material for many applications such as paper and paperboard production, food products, antimicrobial agent, coating, cosmetics, a medical field for drug delivery, the implant of soft tissue, blood vessel replacements, and others. Different methods can be used to obtain nanocellulose from different lignocellulosic materials. These are acid hydrolysis, mechanical treatment, and enzymatic hydrolysis [3] [1]. The widely used method by different researchers is the acid hydrolysis method. It is an easy and fast method to produce nanocellulose with better properties and small particle size. Different works were done on this area to extract nanocellulose from different materials like bagasse, rice husk, wheat straw, flax, hemp, sisal, jute, corn straw, cotton, and pineapple[4][5][6].

Teff straw, the abundant and indigenous lignocellulosic material in Ethiopia, is produced on large scale. Ethiopia is a large producer of teff in the world and currently producing a large amount of teff annually in different areas and in 2017, teff accounted for about 24% of the nationwide grain-cultivated area. It is one of the main cereal crops mostly cultivated in Ethiopia and Eritrea for food[7].

In 2015-2016, 2.87 million hectares (22.95%) of land was covered with Teff which was cultivated by 6.56 million farmer households approximately 3.73 million tons of Teff grain was

assumed to be produced in the country [8]. In most cereals, approximately 2lb of straw is expected to be produced per 1lb of grain. Taking this into consideration, Ethiopia's annual production of Teff straw is estimated to reach about 7.46 million tons. Teff straw is used for different applications such as animal feed, mulching, and construction. It contains around 38% cellulose, 27% hemicellulose, 18% lignin, and 10% extractives [9]. It is the cheapest material and around 15% of this material is leftover in the field and disposed of annually but, its recognition in further scientific studies does not get attention when compared to other lignocellulosic materials.

A very few Ethiopian scholars try to investigate the use of teff straw for biosilica synthesis[10], for the removal of heavy metals [11], and bio-methane production [9]. Nowadays, as the industry attempts to reduce its dependence on petroleum-based products there is an increasing need to investigate more environmentally friendly with improved properties and sustainable materials to replace the existing ones. So, this study attempt was done to evaluate the effectiveness of teff straw as a cellulose and nanocellulose source.

2 Materials and Methods

2.1 Materials

The raw material, teff straw, was collected from the agricultural field near Jimma Agaro town. Sodium hydroxide (FINEM, 97%), sulfuric acid (UNICHEM, India, 98%), sodium chlorite (HIMEDIA, 80%) were purchased from Ranchem industry and trading plc, Addis Abeba. Acetic acid glacial (Neolab, 96%), filter paper (90mm) in diameter were purchased from Alkane Plc, Addis Abeba. All chemicals are analytical grade and used as received.

2.2 Methods

2.2.1 Raw material Preparation

Teff straw was collected, sorted, and washed with distilled water. Then, the sample was sun-dried for 5 days and oven-dried for 6 hours at 60°C. After that, the straw was grinded and sieved to obtain 0.5 mm.

2.2.2 Proximate Analysis of raw material

The moisture content of the teff straw was determined by the oven drying method. This was carried out at a temperature of 103 ± 2 °C for 2 hours following the ASTM D 1037 (1991). Then it was calculated by the equation;

$$\text{Moisture content} = \frac{W_i - W_f}{W_i} \times 100\% \quad (1)$$

Where, W_i is initial mass before drying and W_f is the final mass after drying

The ash content was determined using ASTM D 2017 (1998). 3g of straw placed in a pre-weighed crucible was incinerated in a muffle furnace at 600°C until complete ash was achieved (2 hours). The crucible was then transferred into desiccators for cooling and calculated by;

$$\text{Ash Content} = \frac{W_a - W_c}{W_b - W_c} \times 100\% \quad (2)$$

Where, W_a , is the weight after complete ashing + crucible

W_b is the weight before ashing + crucible and W_c is the weight of the empty crucible

The volatile matter was determined according to ASTM, E-872. 2g of the samples of the straw was incinerated in a crucible at a temperature of 650 °C for 6 minutes and allowed to cool down in a desiccator. Based on a dry basis the volatile matter content was calculated by using the equation;

$$\text{Volatile content} = \frac{W_i - W_f}{W_i} \times 100 \quad (3)$$

Where W_i is the initial weight of the sample and W_f is the final weight of the sample

The fixed carbon of the straw was calculated according to standard (ASTM D3172 – 13) by subtracting the sum of ash content (%) and volatile matter (%) from 100. The fixed carbon is defined as the residue left after removing the volatile matter and the ash from the substance.

$$\text{Fixed Carbon} = 100 - (\text{Ash content} + \text{Volatile matter}) \times 100\% \quad (4)$$

2.2.3 Compositional analysis of Teff straw

The water and ethanol extractives were determined according to the method used by [12]. 10 g of sample was taken and mixed in 200 ml of distilled water. Then, the solution was kept in a boiling water bath at 80°C for 3 hours then cooled and vacuum filtered. After that, it was dried at 105°C in the oven until a constant weight was obtained. To determine the ethanol extractives, 5% ethanol was prepared and the water extractive free sample (5g) was added and mixed. Then, the solution was kept in a water bath of 80°C for 3 hours. Then, cooled and filtered. After that, it was dried at 105°C until a constant weight was obtained. The amounts of total extractives were calculated as,

$$\text{Extractives}(\%) = \frac{M_i - M_f}{M_i} \times 100 \quad (5)$$

Where M_i is the initial mass and M_f is the final mass of the sample.

The hemicellulose content was determined according to the method ASTM D5896-96 (2012). 1g of the sample from the dried extractive free sample was taken and 10 ml of 0.5 mol of NaOH solution was added to it. Then the solution was kept in a boiling water bath for 3 hours at 80°C. After that, the residue was washed with distilled water and filtered until neutral pH was achieved. Then, the sample was dried at 105°C and the amount was calculated by the equation;

$$\text{Hemicellulose content} = W_i - W_f \quad (6)$$

Where W_i is the initial weight and W_f is the final weight

Lignin content was estimated according to the method of the institute of paper chemistry, Appleton, Wisconsin as described in [13]. About 2 g of extractive sample was treated with 20 ml 72% sulfuric acid and the acid was added to the sample drop-wise with constant stirring. After that, the sample was allowed to stand and left overnight at room temperature. The sample was then transferred to a round flask, diluted to 3% sulfuric acid, and boiled for 4 h. The lignin was filtered on a pre-weighed filter paper and washed with hot distilled water until neutrality. The lignin was then dried at 105°C for 6 h and estimated according to the following equation.

$$\text{Lignin content (\%)} = \frac{W_d}{W_i} \times 100 \quad (7)$$

Where W_d is the weight of the dried sample and W_i is the initial weight

The cellulose content was determined by considering the other constituents of lignocellulose and by subtracting the sum of extractives, hemicellulose, and lignin from a hundred.

$$\text{Cellulose \%} = 100 - (\text{hemicellulose} + \text{lignin} + \text{extractives}) \quad (8)$$

2.3 Extraction of Nanocellulose from Teff Straw

2.3.1 Delignification process

Delignification is the process in which lignin, pectin, and other extractives were removed and this process was conducted according to the procedure described by [14] and [15] with some modification. The fiber was treated with hot distilled water before the delignification process to remove water extractive materials. The fiber was treated at 75 °C when a ratio of fiber to water is 1:20 g/ml for an hour. Then the fiber was treated with acidified sodium chlorite solution (0.7%) w/v at 75°C in a water bath for 2 hours under continuous mechanical stirring (400 rpm). The pH of sodium chlorite was adjusted to 4 by glacial acetic acid (5% v/v). The ratio of fiber to acidified sodium chlorite solution was 1:25 g/ml. This process was repeated four times until the color of the fiber was turned to white. The residue was vacuum filtered and washed with distilled water and this process was repeated until the pH of the filtrate becomes neutral. Then the residue was oven-dried overnight at 50°C and kept for further process.

2.3.2 Isolation of Cellulose

The cellulose was extracted from holo-cellulose by an alkaline hydrolysis process using sodium hydroxide solution. The dried holo-cellulose was treated with hot 4% w/v NaOH solution at 60 °C in a water bath for 1 hour. The fiber to solution ratio was 1:20 g/ml under constant mechanical string with 400 rpm. Then, the residue was vacuum filtered and washed several times until the yellow color disappeared and the pH of the filtrate becomes neutral.

2.3.3 Extraction of Nanocellulose

The nanocellulose extraction was conducted under a modified procedure adopted by [16] and [14]. The 10 grams of cellulose was hydrolyzed with a sulfuric acid solution (48% v/v) under continuous mechanical stirring (500 rpm) at 35 °C in a water bath for 30 minutes. The ratio of cellulose to a sulfuric acid solution was kept at 1:25 g/ml. To stop the hydrolysis process 10-fold of distilled water (2500 ml) was added to the reaction mixture.

Then the suspensions were centrifuged at 3000 rpm for 5 minutes to remove the acids. Then, the suspensions collected from the centrifugation process undergo the dialysis process to remove the remaining acids and the dialysis process was done with a dialysis tube with a specification of 12-14 kDa MWCO. The suspensions were kept under dialysis in distilled water for 10 days by changing the water daily and collected after the pH of the product near neutral. After that, it was sonicated using probe ultrasonicator at 25 kHz for 5 minutes at (Leather Industry and Development Institute (LIDI), Ethiopia) to obtain uniformly dispersed nanocellulose suspension to avoid agglomeration of the nano cellulose particles. Finally, the yield of nanocellulose was calculated by dividing the final weight of nanocellulose after freeze-drying for the initial weight of the cellulose submitted to hydrolysis.

$$Yield = \frac{W_f}{W_i} \times 100\% \quad (9) \quad \text{Where,}$$

W_f is the final weight and W_i is the initial weight

2.4 Characterization of Nanocellulose

2.4.1 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

The chemical composition assessment of teff straw, cellulose, and the nanocellulose suspensions were conducted using Fourier transform infrared spectroscopy (FTIR). FTIR spectra were obtained using the Perkin Elmer FTIR spectrometer (Perkin Elmer, 65, USA) at Addis Ababa University, Department of Chemistry (Addis Ababa, Ethiopia). This analysis was done for the three materials to see the changes that happened to the functional group of the straw after different steps of pretreatments. The analysis was done with the range of 4000 – 400 cm^{-1} and

KBr is used for solid samples (teff straw and cellulose) and the NaCl window was used for nanocellulose suspension.

2.4.2 Particle Size Distribution

Dynamic Light Scattering Analysis is one of the well-known methods to determine the particle size distribution and average particle size of the nanoparticle. Due to the different treatments on the teff straw, there is a change in dimensions since the fiber chains breakdowns into smaller particles. The determination of particle size distribution and average particle size of nanocellulose was done at Addis Ababa Science and Technology University (Addis Ababa) by using the Zetasizer Nano instrument (ZE3600).

2.4.3 Crystallinity Analysis

The X-ray Diffractometer (XRD) instrument was used to see the changes in the crystallinity of the teff straw and nanocellulose. The XRD analysis was done at Jimma Institute of Technology, School of Materials Science and Engineering, Jimma. DW-XRD-Y7000 instrument was used with Cu K α radiation. The scanning range was $2\theta = 10$ to 70 degrees at a scanning speed of $0.03^\circ/\text{s}$.

To conduct the XRD analysis the samples need to be dried in advance, which will affect the results as the structure changes upon drying. The nanocellulose suspension used for XRD analysis was freeze-dried at (-30°C) at Addis Ababa Science and Technology University (Addis Ababa, Ethiopia) to obtain dry powder nanocellulose particles. The following equation was used to calculate the crystallinity from the different peaks obtained.

$$\text{Crystallinity} = \frac{A_{\text{crys}}}{A_{\text{total}}} \times 100\% \quad (10)$$

Where A_{crys} Area of crystalline peaks and A_{total} is a total area of all peaks

2.4.4 Thermogravimetry Analysis

Thermogravimetry analysis (TGA) was conducted to investigate the thermal stability of the samples. This was conducted with instrument TGA - 4000 Perkin Elmer at Jimma institute of technology, Jimma, Ethiopia. This was conducted for all samples with nitrogen gas. Samples were heated from 25°C to 500°C under a flow of nitrogen gas. The flow rate of nitrogen gas was

20 ml/min with a heating rate of 10°C/min. The Derivative Thermogravimetry (DTGA) data also recorded.

3 Result and Discussion

The nanocellulose was extracted from teff straw. The yield of cellulose was 78.47% and the yield of nanocellulose was 42.87%.

3.1 Proximate analysis of straw

It is important to understand the component of lignocellulosic materials before application. Different proximate analysis was conducted to investigate the moisture, ash, volatile, and fixed carbon content of the straw. The result obtained in this work is in agreement with the literature.

Table 1: Result of proximate analysis

Parameters	Average result (%)	Literature value
Moisture content	6.4 ± 3	7.3
Ash content	5.1 ± 2	4.0
Volatile matter	74.2 ± 1	74.7
Fixed carbon content	15.6 ± 5	14.0

Source for literature value: [10]

3.2 Compositional Analysis

The compositional analysis of lignocellulosic material is very important to understand the suitability of the material for the targeted application. This means it is necessary to know the component of the teff straw before extraction of the cellulose. The cellulose, hemicellulose, and lignin content of the straw played a great role in the yield of the final product. The result is presented in table 2. The cellulose content of teff straw determined was 36.5 %.

3.3 Functional group Analysis

The study of the functional group of the raw materials, cellulose, and nanocellulose was done by FTIR and the results were interpreted based on the peak observed at each functional group.

As the spectra observed from the result shows there are different spectral bands. FTIR spectra of nanocellulose having sharp bands but almost similar to that observed in cellulose. A similar result was reported by [17]. The peak at 3344-3490 cm^{-1} shows that the stretching of intramolecular hydrogen bonds of O-H groups of cellulose became narrower after the different treatments due to the removal of part of the amorphous components which is in agreement with [18]; [6]. The peak around 2852 cm^{-1} – 2942 cm^{-1} corresponds to the C–H stretching of methyl and methylene groups in cellulose which decreased after alkaline and bleaching treatments [19]. The other peak located at 1730 cm^{-1} shows the presence of acetyl and uronic ester groups of the hemicellulose or the ester linkage of the carboxylic group of ferulic and *p*-coumaric acids of lignin and hemicelluloses [20]. This was reduced and practically disappeared after the treatment process. The peak at 1632 cm^{-1} is due to the water absorbed by the teff straw [21].

Table 2: Result of Compositional analysis of teff straw

Parameters	Average value
Extractives	8.5
Lignin	17.5
Hemicellulose	29.5
Cellulose	36.5

The other located at 1512 cm^{-1} in the teff straw sample is due to the C-H deformation of methyl, methoxyl groups of lignin, and aromatic C=C ring stretching. This peak was disappeared in cellulose and nanocellulose which shows the absence or reduction of lignin and hemicelluloses after bleaching and alkali treatment. The spectral bands at 1427 cm^{-1} and 1384 cm^{-1} are due to CH₂ scissoring and C-H bending in cellulose respectively [17]. The peaks observed at 1253 cm^{-1} and 1045 cm^{-1} in teff straw and disappeared in cellulose and NC were due to the C-O out of plane stretching due to the aryl group of lignin and C-O-C pyranose ring stretching respectively [22]. This shows the effectiveness of the chemical treatments to remove lignin and hemicellulose. The spectral bands at 1155 cm^{-1} and 1118 cm^{-1} are due to the C-C stretching in cellulose and C-OH skeletal vibration in lignin respectively [22].

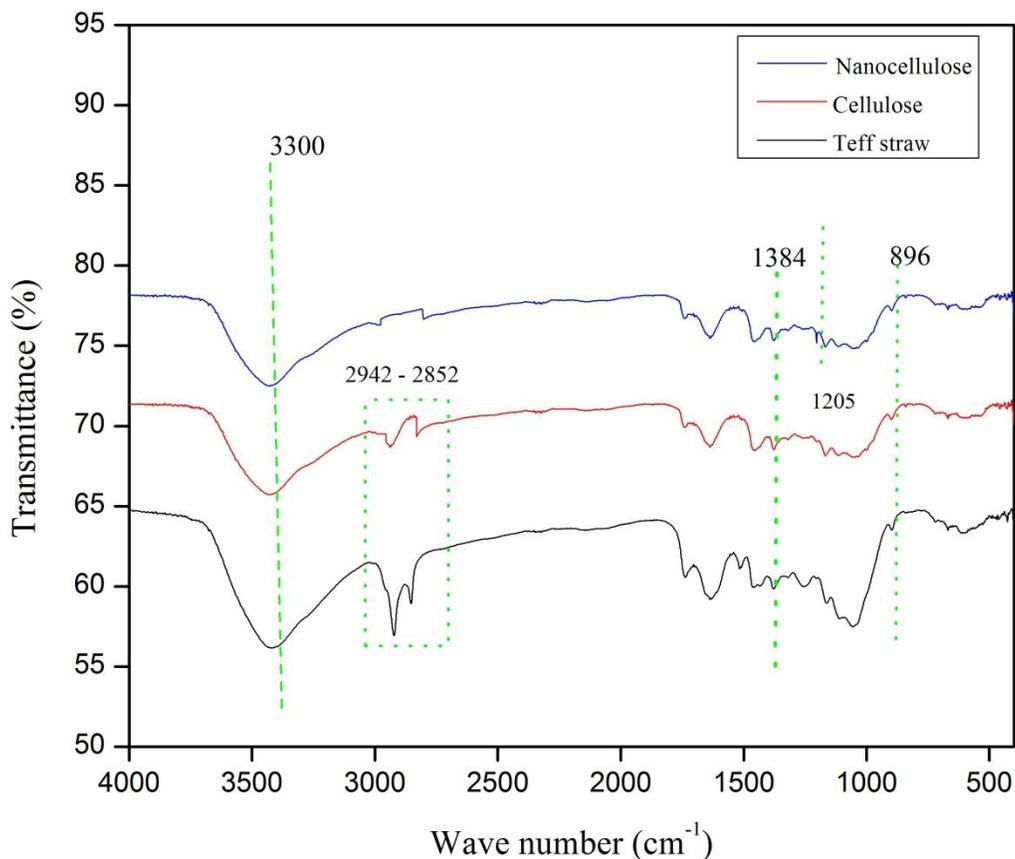


Figure 1: FTIR Spectra of Teff straw, cellulose, and nanocellulose sample.

The peak observed at 892 cm^{-1} shows the presence of β -glycosidic linkages in cellulose [23]. The appearance of the new small spectral band at 1205 cm^{-1} in nanocellulose is due to the presence of S=O vibration after the acid hydrolysis process [24]. The spectral bands at 1427 cm^{-1} and 895 cm^{-1} represent Cellulose I content. The peaks at 1322 cm^{-1} , 1431 cm^{-1} , and 1372 cm^{-1} are typical of pure cellulose[18]. Generally, the same results were reported by [25]; [22], and [21]

3.4 Particle Size

Dynamic Light Scattering was the method used in this work to determine particle size distribution and average particle size. This instrument gives particle size distribution, the average particle size of nanocellulose suspension, and the Polydispersity index of the sample. The results derived by DLS were given in figure 2 and table 3. As the below figure shows the peak obtained was Uni-modal and the particle size distribution was in a range of 50 nm to 200 nm. The Z-average particle size given by the instrument was 101.6 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 intensity. The polydispersity index (PDI) describes the uniformity of the sample in suspension. The higher the PDI, the less uniform the sample is. In general, samples with PDI less than 0.1 (<0.1) corresponds to monodisperse and gives the Unimodal peak. Samples with PDI greater than 0.1 (>0.1) are polydisperse and can give the bimodal or tri-modal peaks. The PDI for this product was 0.047 which is less than 0.1 and confirms that the nanocellulose suspension had good uniformity.

Table 3: Z-Average particle size from DLS instrument

		Size (d.nm)	% Intensity
Z- average (d.nm) : 101.6	Peak 1:	91.9	100.0
PDI : 0.047	Peak 2:	0.000	0.0
Result quality: Good	Peak 3:	0.000	0.0

Different authors reported the particle sizes between 3 nm to 600nm ranges. Zhou et al., [26] and Fattahi and Dadashian [27] reported the nanocellulose size of 115 nm and 70 nm respectively with bimodal peak. Sofla et al., [19] and Chattopadhyay [28] reported the nanocellulose size of approximately 100 nm and 348 nm respectively with a Unimodal peak. Heriberto et al. [29] extracted nanocellulose from discarded cotton fibers with an average particle size of 235 nm with a Unimodal peak by using a dynamic light scattering instrument.

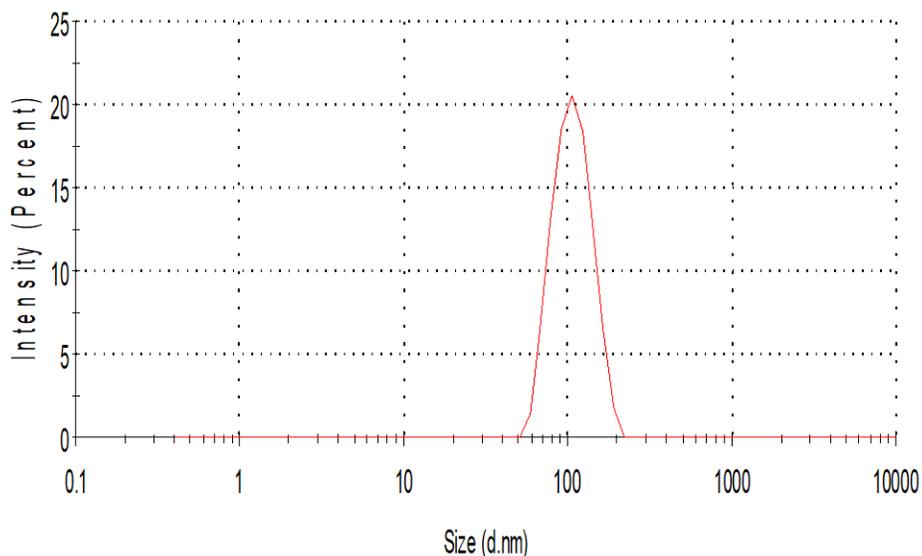


Figure 2: Particle size distribution obtained by Dynamic Light Scattering

3.5 Crystallinity Index

The diffractometer peaks obtained from XRD analysis scanned over 10 to 70 degrees with a step increment of $0.03s^{-1}$ were given in figure 3. It was done for three samples, teff straw, cellulose, and nanocellulose to see the difference in their crystallinity after chemical treatments. The high-intensity peak located at 22.79° for nanocellulose describes the crystalline nature of the material and the intensity value shows the amount of crystalline structure. There are two major peaks for all samples around 2θ of 15 degrees and 22 degrees. The crystallinity index was calculated by equation 10. First, the crystallinity of nanocellulose was calculated and the crystalline peak area and total area for all samples were calculated. The crystallinity index was found to be 77.185%. The crystallinity index of teff straw was also calculated in the same way as calculated for nanocellulose and found to be 47.736%.

Table 4: Crystallinity index of teff straw and nanocellulose

Samples	Crystallinity Index (%)
Teff straw	47.7
Nanocellulose	77.1

The crystallinity indexes of untreated teff straw and nanocellulose were given in table 4. The CI of teff straw was 47.736% without any chemical treatment and it had the lowest CI compared to the chemical treated samples.

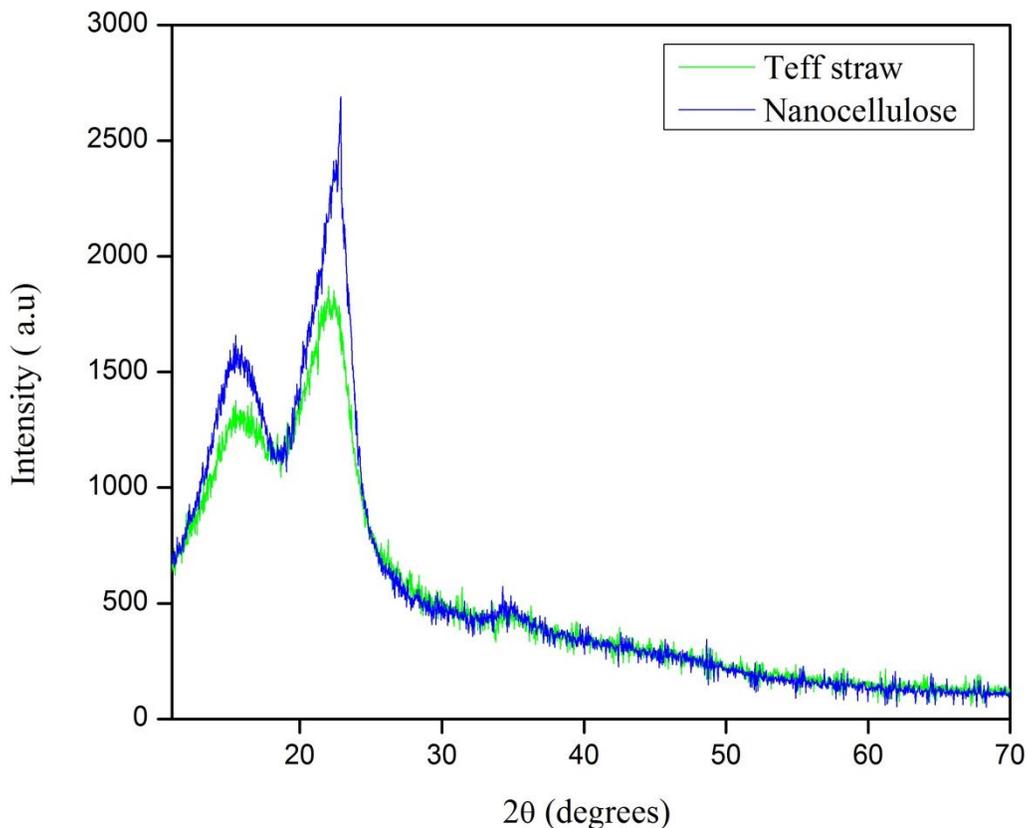


Figure 3: X-ray Diffraction patterns for nanocellulose

After acid hydrolysis of cellulose, the crystallinity index was raised to 77.185 %. This means the acid hydrolysis process had played an important role in the degradation of the amorphous parts. Kumar *et al.*, 2014 [17] reported that the nanocellulose extracted from sugarcane bagasse with a crystallinity index of 72.5% that characterized by XRD. Ioelovich [30] also extracted nanocellulose with a 76% crystallinity index from commercial MCC.

3.6 Thermal analysis

Thermogravimetry analysis (TGA) is one of the well-known methods to determine the thermal stability of materials. Cellulose, hemicellulose, lignin, and other extractives in plant fiber decompose at different temperatures due to their different chemical structures.

Figure 4 and 5 shows the TGA and DTGA curves of untreated teff straw and extracted cellulose. TGA curves of both samples reveal three regions of mass loss. The first loss starts at lower temperatures around 25 °C - 230°C, which is due to the release of moisture and volatile compounds, the second is between 230°C -375°C and the third is above 376 °C which corresponds to char formation.

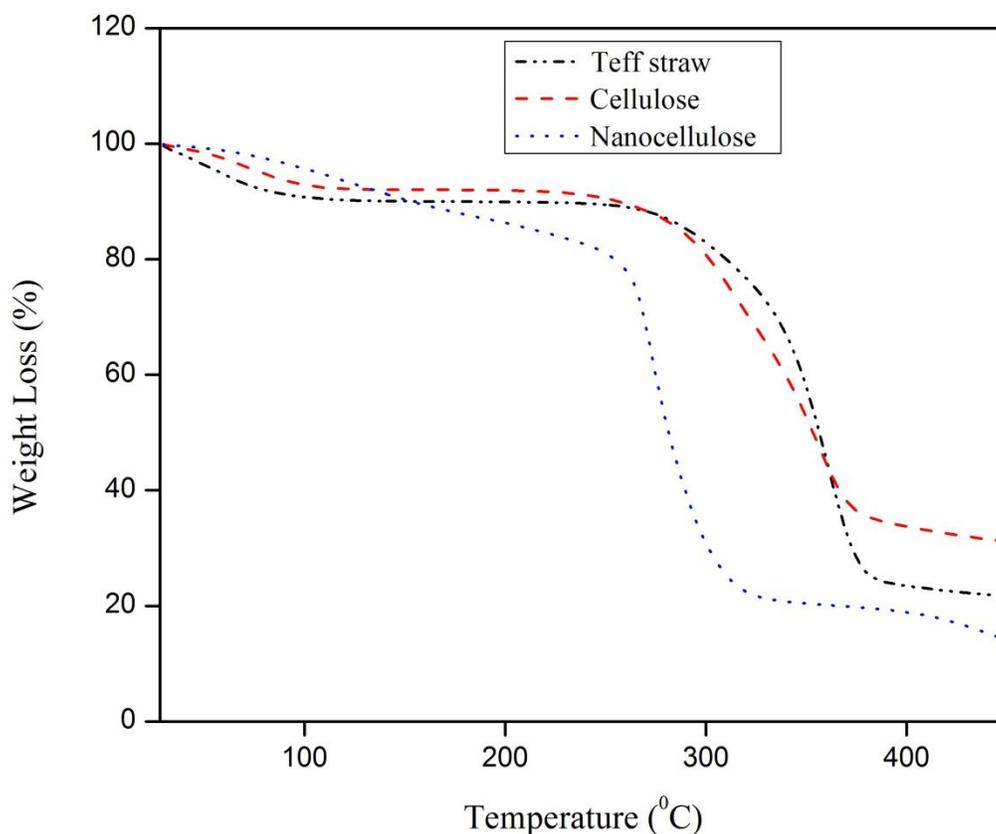


Figure 4: TGA curves of teff straw, cellulose, and nanocellulose.

As the DTGA curve shows the maximum peak reached 354°C for teff straw and 361°C for cellulose particles. This means the untreated teff straw has lower thermal stability which is due to the presence of pectin, hemicellulose, and lignin. The extracted cellulose shows better thermal stability. The nanocellulose reached a maximum peak at 271°C. The nanocellulose extracted by sulfuric acid shows lower thermal stability than that of extracted cellulose and teff straw. This may be raised from the presence of sulfate groups produced in nanocellulose during the acid hydrolysis process and the thermal degradation of sulfate groups starts at lower temperatures. The other reason is an increase in the surface area of nanoparticles.

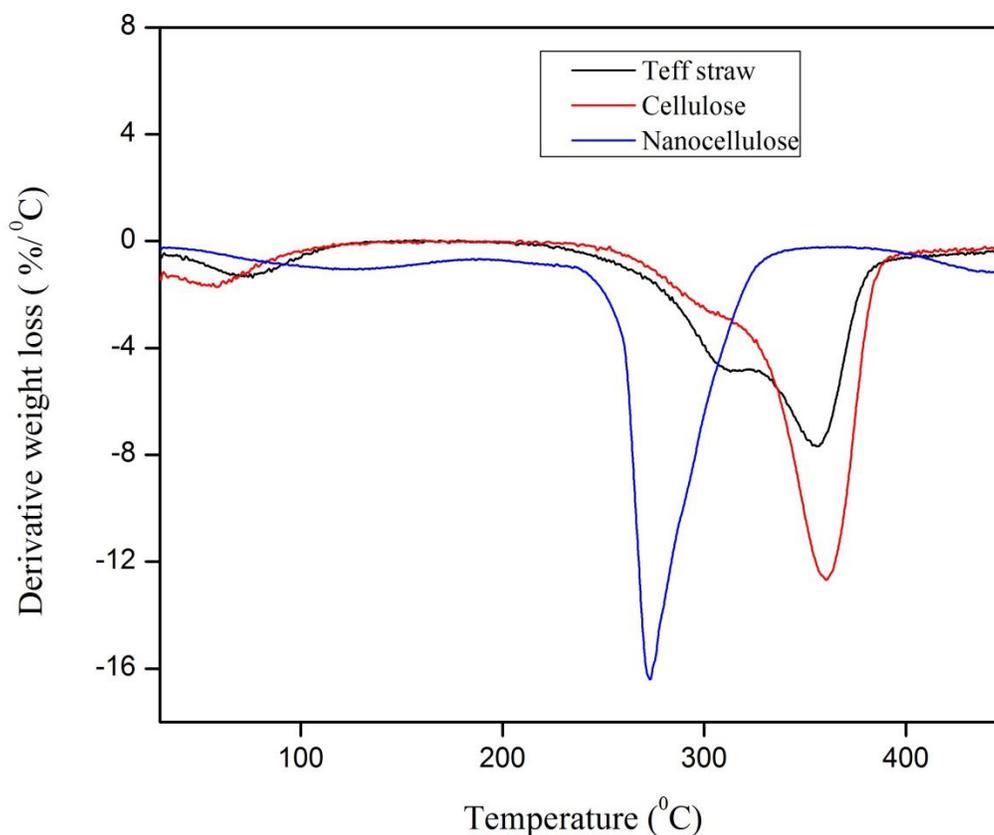


Figure 5: DTGA curves of teff straw, cellulose, and nanocellulose.

4 Conclusion

Nanocellulose has been successfully extracted from teff straw (*Eragrostis Teff*) by delignification and alkali treatment and acid hydrolysis with a yield of 42.8 %. The proximate analysis and compositional analysis of raw material were also determined. Cellulose was extracted by a hot alkali treatment method and nanocellulose was extracted by an acid hydrolysis method. The functional group, average particle size and particle size distribution, crystallinity index, and thermal stability of the extracted nanocellulose were determined by fourier-transform infrared spectroscopy, dynamic light scattering, x-ray diffraction, and thermogravimetry analysis/ derivative thermogravimetry. The properties are ideal and recommended for the application of nanocellulose in different areas.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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