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Nanocellulose Extraction from Jute Wastes through Chemical Pre-Treatment

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Abstract—*In the present studies an attempt have been made to develop nano crystalline, a high-end value product ($ 550-1000 per kilo, from the low cost jute stick ($ 0.075 or around INR ~5 per kilo) through chemical treatment. Dilute sulphuric acid have been found be effective at a concentration of 50-60% for breakdown of cellulose long chain. The alphacellulose yield is more than 50% and the yield of nano cellulose is around 30%. As of now the nanocellulose has been produced from wood which is a major concern for deforestation. The findings may mitigate not only the problems associated with the availability of wood (the conventional raw material for preparing nanocellulose) due to serious concern about deforestation, but also utilization of high volume agricultural waste/ by-product like jute stick and jute caddies along with generation of extra economic benefits for jute cultivation.*

Keywords*: Jute waste, Caddies, Sticks, Nanocellulose, NCC*

1. INTRODUCTION:

Cellulose is the predominant organic molecule on earth. The major component in the plant cell wall is cellulose. Cellulose is a linear polysaccharide polymer with many glucose monosaccharide units. The acetal linkage is beta which makes it different from starch (Nishiyama *et al.*, 2003 and Subramanian *et al.*, 2008). Cellulose contains both amorphous and crystalline structures. Nanocrystalline cellulose (NCC), also called cellulose nanocrystals, is typically a rigid rod-shaped monocrystalline cellulose domain (whisker) with 1-100 nm in diameter and tens to hundreds of nanometers in length (Ruiz, *et al.*, 2000). NCC has a high degree of crystal structure (more than 70%), a very big length-diameter ratio (around 70), and a large surface area (150 m^2/g). NCC can be used for many applications such as regenerative medicine (Fleming *et al.*, 2001), optical application (Revol, *et al.*, 1998), automotive application (Dahlke *et al.*, 1998), composite materials (Avella *et al.*, 1993; Jiang & Hinrichsen, 1999) and so on.

In India, a huge quantity (around 4 Mt per annum) of jute (Corchorus spp.) stick is being produced as a primary by-product of jute fibre (economic part) cultivation (Nayak et al., 2013). Moreover, Indian Jute Industry generates nearly 40000 tonnes of processing wastes as by products commonly known as Jute Caddies (Nayak et al., 2012). In common practice these biomass are either burnt in boiler or used as such as firewood for domestic energy purposes. It is found from the experiment that if this stick/biomass of jute could be explored further as a new source of high-end value product, especially, Nanocellulose, a huge return can be achieved.

2. MATERIALS AND METHODS:

Jute Caddies:

Jute caddies were obtained from three jute mills and analysed separately for their components. The composition of jute caddies was determined by removal of oil and grease by extraction with trichloroethylene under reflux in a Soxhlet apparatus for 6 h, followed by opening and cleaning in a trash analyzer. The oil and trash (bark, jute stick remnants, etc.) free material was treated with 0.5 per cent hydrochloric acid to remove the remaining inorganic impurities.

Scouring of Caddies

Caddies can be scoured by an aqueous or a non-aqueous method. While the aqueous method has the advantage of employing simple equipment like, steel vat, it generates effluents. A non-aqueous scouring method employing solvent like n-hexane or toluene, though efficient and clean, requires much higher capital investment and working capital. Jute caddies were scoured with different chemicals in aqueous system as well as with organic solvents in a non- aqueous system at different temperatures and time intervals.

Opening and Cleaning of Scoured Caddies

The scoured caddies contained remnants of plant bark, broken pieces of jute stick, thick fibrous roots and some dust. Opening and cleaning operation to remove these materials was carried out, using an opener machine which used both mechanical and pneumatic actions to clean the fibres. If the caddies are not cleaned before pulping the paper obtained is dotted with tiny dark spots of bark and dust particles.

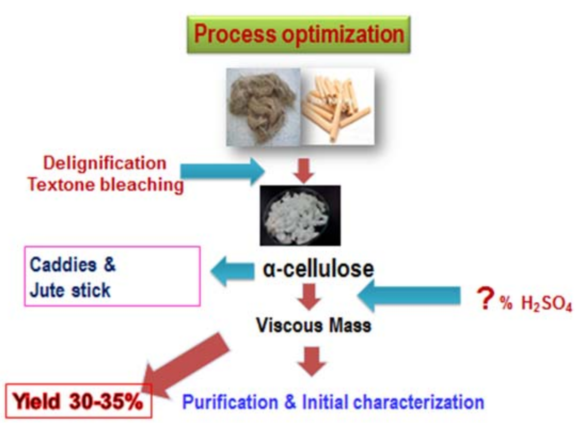
Pre-treatment of jute caddies:

Pre-treatment is normally done to swell the fibres before subjecting them for extraction of cellulose. The defatting was done by applying the Leavitt-Danzer method. In this process, two types of chemicals were used, namely, toluene ($C_6H_5CH_3$) and ethanol (C_2H_6O), with ratios of 2 : 1. The extraction process was done using the extraction column (Soxhlet extractor, Round Bottom Flask, Liebig Condenser, Heater, Membrane, and Thermometer). Then, the chopped jute fibers were immersed in the extraction column. This process was continued for 3 hours at 150^0C. The collected fibers were later placed in the forced air convention oven for 24 hours at 75∘C. The delignification was implied using acetic acid (CH_3COOH) and hydrogen peroxide (H_2O_2) in present titanium oxide (TiO_2) in a round bottom vector vessel. Then, the dewaxed jute fibers were placed in the round bottom vessel. This process was continued for 3 hours at 130∘C. After this, the collected fibers were carefully washed and placed in the forced air convention oven for 24 hours at 70^0C.

Extraction of Alpha cellulose:

Alpha cellulose from jute caddies were extracted using following scheme. The delignified jute was washed and dried properly to obtain holocellulose. Hollocellulose contain a certain percentage of hemicellulose. These hollocellulose is further treated with higher alkali concentration (18% NaOH). The macerated hollocellulose was filtered and washed properly to obtain alpha cellulose.

Fractionation of A



Fractionation of alpha cellulose to Nanocellulose:

The dried alpha cellulose sample was subjected to acid treatment for further fractionation. Sulphuric acid 50% was found to be suitable for fractionation of alpha cellulose. The sample was treated in a magnetic stirrer for one hour. When there is a white precipitation in the reaction vessel the reaction is considered to be completed. It is then cooled and kept overnight. The precipitated sample is then subjected to rigorous centrifugation for 5-6 times at 15000 rpm till the pH of the reaction mixture becomes normal. At normal pH the reactant is taken to probe sonicator where it is treated for 2 hrs in interrupted sequences. The mixture becomes colloid and turned to be viscous mass which is subjected for DLS analysis.

3. RESULTS AND DISCUSSION:

The composition of jute caddies and jute sticks obtained from the commercial mill and from ICAR-NIRJAFT were analysed and average values are presented in Table 1.

Constituents	Jute Caddies (%)	Jute stick (%)
Fat & wax	9.28	1.9
α-cellulose	61.05	40.80
Pentosan	19.61	22.10
Lignin	15.07	23.50
Ash	2.39	1.00

It is found that jute caddies contain high amount of waxy materials. This may be due to the treatment of jute with batching oils. The jute caddies contained 61.05% alpha cellulose whereas, jute stick contained nearly 41% alphacellulose. Jute stick contained grater amount of lignin (23.5%) than the jute caddies (15.07%). The pentosane content in both caddies and stick were more or less similar and ranged between 19.61 to 22.10%.

Yield of cellulosic fractions :

The composition of jute caddies collected from different sources is presented in Table 2. It was observed that the caddies from NIRJAFT mill yielded 57.49% alpha cellulose. Fractionation of this cellulose yielded 32.35% of cellulose in nano range. The commercial mill caddies were yielded 59.40% alphacellulose from which 30.24% nanocellulose obtained. In comparison the plain jute yielded 61.48% alpha cellulose which was converted to nanocellulose with a yield of 35.56 %.

Caddies source	Initial weigh t (g)	Holocellulos e yield (%)	α-Cellulos e yield (%)	Nano cellul ose yield
NIRJAFT Mill	30	83.93	57.49	32.35
Commerci al Mill	30	88.60	59.40	30.24
Plain Jute	30	88.25	61.48	35.56

Optimization of acid concentration efficient yield of Nanocellulose:

The sulphuric acid concentration was optimized with correlation of yield of nanocellulose in specific nano range. Perusal of data presented in table showed that 50% concentration of sulphuric acid is optimum to yield the cellulose in the nano range. The higher concetration of acid did not show any significant results in terms of conversion of cellulose into nanocellulose. In both the concentration of 64 and 70% the samples were charred and no yield result could be obtained. The nano sized particle size were ascertained through dynamic light scattering.

Acid concentration (% H2SO4)	Initial wt of α-cellulose (g)	Particle size (nm)					Yield (%)
		d10 (dia less than 10%)	d25 (dia less than 25%)	d50 (dia less than 50%)	d75 (dia less than 75%)	d90 (dia less than 90%)	
40	1	12.03	24.52	45.56	72.79	100	0.36
45	1	20.39	40.75	76.57	119.9	161.1	5.95
50	1	20.56	45.35	80.23	146.23	150.2	8.19
64	1	-	-	-	-	-	
70	1	-	-	-	-	-	

4. CONCLUSION

Cellulose is abundant in earth and wasted abundantly without knowing its potentiality. Knowing the importance of nanocellulose there is huge demand of cellulose which is often obtained through deforestation. Deforestation, however, considered to be sin in the modern times in the context of global warming and climate change. Jute is a heavy cellulose yielding crop with duration of 2-3-4 months. Utilization of jute cellulose for achievement of nanocellulose can protect valuable wood and forests. However, jute is considered as one of the important commercial crops in many parts of our country. Although there is a source of jute waste biomass which can be explored for extraction of high value product like nanocellulose. In the context, initial characterization of jute stick and caddies were done and converted to alphacellulose. The acid hydrolysis process was further optimized and a good amount of yield was obtained. It is found that the yield of α-Cellulose from jute Caddies and jute stick were 57.49 and 32.52%, respectively. The viscous mass of colloidal cellulose was obtained through hydrolysis of α-Cellulose which was found to be in nano range. The yield of viscous mass was found to be 32-35% which may be exploited further for production of value added products.

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