

Development of reinforced paper and mitigation of the challenges of raw material availability by utilizing Areca nut leaf

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ABSTRACT: Paper industries are facing a raw material crisis and searching for alternate raw materials that may be able to help mitigate the issue. Many industries use agro-waste as a raw material, irrespective of it having low bleachability and poor mechanical strength.

Areca nut leaf (ANL) is a nonwood-based material that may be acceptable as an alternate source of raw material that contains 61.5% holocellulose and 13.6% lignin, which is comparable to other agro-wastes and hardwood pulps. Kraft anthraquinone pulping with 20% active alkali as sodium oxide (Na_2O), 25% sulfidity, and 0.05% anthraquinone produced 15 kappa pulps with about 38.5% pulping yield. The bleachability of ANL pulp was good, and 83.5% ISO brightness could be achieved using the $\text{D}_0(\text{EOP})\text{D}_1$ bleaching sequence. The ANL fiber has 33.8% better tensile, 54.5% better tear, and 15.2% better burst index than hardwood fiber. Similarly, 60.4% better tensile, 56.5% better tear, and 21.7% better burst index were observed in ANL than in wheat straw. Thus, the study revealed that Areca nut leaf can be used as an alternative raw material for papermaking, as well as to improve the physical property of paper products by blending it with inferior quality pulp.

Application: Areca nut leaf was used in this study as a potential raw material for papermaking, with good pulping and bleachability. The paper properties were also good as compared to other agricultural residues and hardwood pulp, so Areca nut fiber pulp can be used in place of hardwood to improve the physical properties of paper products, as agro-based paper industries are using hardwood pulp to improve its strength.

The Areca nut plant is commonly known as a palm plant and is cultivated for producing betel nut in tremendous quantities. The plant is grown in the coastal region of tropical countries in Asia and East Africa. India, Kerala, Assam and other places are major producers of areca nut and generate a huge amount of plant waste that is being burned or disposed of in the open field. This causes serious environmental problems of harmful gases (carbon monoxide, carbon dioxide, methane gas, etc.) because of burning and biodegradation in the open field. Thus, the present investigation was carried out for the utilization of Areca nut leaf (ANL). By utilizing ANL fiber, open field burning can be minimized, and the struggle for raw materials in the paper industry can be reduced.

The Areca nut plant has a very large evergreen leaf that whorls around the top of the stem. The ANL has a tubelike sheath at the base that is open at one side at maturity. The ANL fiber is a rich source of α -cellulose. Earlier works have shown that ANL fibers contain 75% or 66% α -cellulose [1,2]. Compared to the Areca nut husk fibers, which contain only 34%–35% cellulose, Areca seeds are found to have high cellulose content [3]. There is very little literature on the use of ANL fibers, especially in pulp and paper. Various rein-

forced polymer composite materials have been developed using the higher cellulose content of ANL fiber [1,4-5]. Researchers have worked on the use of Areca leaf sheets by blending them with wastepaper in a different proportion for developing paperboards with high resistance to water absorption and good tear and tensile strength [6]. Methods were developed for the utilization of Areca nutshells by softening their fiber through microbial treatment of the material. These methods can be commercially applied for developing value-added products like textiles, viscose, and polyesters [7]. Areca leaf sheaths were noticed as potential sources for extracting high crystalline nanocellulose fibers with about 72% cellulose content [8]. Areca nutshell was used as a raw material for making pulp, and bleaching was done with hydrogen peroxide to make handmade colored art paper [9]. Areca leaf sheath has traditionally been used to develop disposable plates and dishes as a sustainable packaging material in India for decades [10]. Based on the literature, no work has been reported on the extraction of pulp fibers by kraft pulping and elemental chlorine-free (ECF) bleaching, as well as its blending with other agro-residue pulp for developing improved mechanical strength properties of paper products.

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The current work evaluated ANL as a new raw material for paper manufacturing, keeping in mind that paper-based industries are facing a series of problems related to raw material availability and strength development in paper products. The whole Areca nut leaf was used to generate pulp via the kraft pulping process. Bleachability of the pulp was investigated using ECF bleaching. The strength properties of bleached pulp were determined, along with the comparative strength properties of other fibers, e.g., hardwood and wheat straw pulps. Successful utilization of ANL would provide an additional raw material for the paper manufacturing industries, as well as for improving the quality of the paper product in terms of strength properties.

MATERIALS AND METHODS

Areca leaf was collected from agricultural lands in the urban areas of western Uttar Pradesh, India. Raw materials were chopped to about 2–3 cm in length, wet cleaned with hot water, and air dried to remove absorbed water. Chopped and cleaned materials were stored at room temperature for further investigation. An active alkali (AA) solution of 100 g/L was prepared by dissolving sodium hydroxide (NaOH) and sodium sulfide (Na₂S) in distilled water; sulfide was maintained at 25%.

Chemical proximate analysis

Determination of the chemical components of the material was done by following regular laboratory procedures as per the TAPPI Standard Test Methods specified in **Table I**. To facilitate the analysis, the samples needed to be ground in a Wiley mill to make dust, and the moisture content of the dust material was measured as per standard.

Pulping

Kraft pulping of ANL was done to produce about 15 kappa pulps. Cooking was performed in a rotary digester having four stainless steel bombs of 100-gm ANL chip capacity on oven-dry basis. The measured weight of the material was taken in this bomb, and to optimize the AA, different doses were charged with 0.05% of anthraquinone as a cooking aid that was added to the alkali. A 4:1 liquid-to-solid ratio

Parameter	Cooking Condition
AA, as Na ₂ O, on o.d. ANL	16%–22%
Sulfidity	25%
Anthraquinone, on o.d. ANL	0–0.05%
Liquid-to-solids ratio	4:1
Temperature	165°C
Time to temperature	60 min
Time at temperature	60 min
AA = active alkali; Na ₂ O = sodium oxide; ANL = Areca nut leaf.	

II. Cooking conditions during kraft pulping.

was maintained through the addition of water after the alkali was charged. The bombs were placed into a digester that was full of water as a heating medium. Cooking was done at a fixed 165°C temperature for 60 min, excluding the time to achieve this temperature from ambient, which was 60 min (time to temperature). The conditions are presented in **Table II**. Targeted kappa numbers were achieved by keeping all the conditions the same, except AA charges. After cooking, black liquor was collected to analyze residual AA, and the pulp was thoroughly washed with hot and cold tap water to remove incorporated black liquor from the pulps. The washed unbleached pulp was kept in a polyethylene bag and stored in a refrigerator for further analysis of pulping yield and kappa number.

Bleaching

The unbleached pulp was bleached using an ECF bleaching sequence of D₀(EOP)D₁ to get the maximum possible brightness of the pulp. Here, D₀ stands for the delignification stage, which includes chlorine dioxide (ClO₂) used to react with the remaining lignin; and (EOP) is the extraction stage, which includes sodium hydroxide, oxygen, and hydrogen peroxide together in the same stage to remove chlorinated and oxidizing lignin products from the pulp. It also

Test Parameter	Standard Test Method
Solvent extractive	TAPPIT 204 cm-97 “Solvent extractives of wood and pulp”
1% alkali solubility	TAPPIT 212 om-02 “One percent sodium hydroxide solubility of wood and pulp”
Holocellulose	Wise, Murphy, and D’Addieco [11]
Lignin	TAPPIT 222 om-06 “Acid-insoluble lignin in wood and pulp”
Ash	TAPPIT 211 om-12 “Ash in wood, pulp, paper and paperboard: combustion at 525°C”
Silica	TAPPIT 245 cm-07 “Silicates and silica in pulp (wet ash method)”

I. Standards followed during the chemical analysis of Areca nut leaf (ANL).

Parameter	Bleaching Stage		
	D ₀	(EOP)	D ₁
Temperature, °C	50–55	80	75
Time, min	40–45	90	180
Consistency, %	5	10	10

III. Bleaching conditions used in different stages.

helps to reactivate the remaining lignin for the next oxidation stage, D₁, which is the bleaching or oxidizing stage for the remaining lignin with ClO₂ to get higher pulp brightness. During the delignification stage, the dose of ClO₂ was optimized by applying different kappa factors to the pulp. The ClO₂ dose in the D₁ stage was also optimized to achieve high brightness of the pulp with minimum loss of chemicals during this stage. Chlorine dioxide bleaching (D₀ and D₁) was performed in polyethylene terephthalate jars, and the temperature throughout treatment was maintained in a water bath. The alkaline extraction (EOP) phase was performed in an electrically-heated reactor under pressurized conditions. After every bleaching stage, the pulp was thoroughly washed with distilled water to remove residual chemicals and reaction products. The process conditions followed at every stage of the bleaching are depicted in **Table III**.

Refining of pulp and handsheet preparation

The unbleached and bleached pulps obtained were refined according to TAPPI Standard Test Method T 248 sp-00

Parameter	Present Study	[8]	[12]	[13]
Extractives, %	12.7	1.51	10.86	0.72
Holocellulose, %	61.5	85.7	60.4	73.3
Lignin, %	13.6	12.8	16.6	19.6
Ash, %	10.4	--	10.9	—

IV. Chemical composition of Areca nut leaf (ANL) fibers.

Parameter	Areca Leaves (present study)	Rice straw [14]	Wheat Straw [15]	Bagasse [13]	Hardwood [16]
Extractives, %	12.7	3.4	3.2	—	1.8
Holocellulose, %	61.5	66.6	72.7	51–72	83.6
Lignin, %	13.6	13.2	19.9	27–32	25.7
Ash, %	10.4	12.7	5.2	1.5–5	1.4

V. Comparison of chemical content with other agro-waste material.

“Laboratory beating of pulp (PFI mill method)” in a PFI mill to reach 450 ± 25 mL freeness level. For the blending study, unbleached and bleached hardwood and wheat straw pulp were also refined to get the same freeness level. Preparation of handsheets of 60 ± 2 g/m² for all samples and blends of the samples in different proportions using a British handsheet former were needed to determine all physical properties of the paper and were performed as per TAPPI T 220 sp-06 “Physical testing of pulp handsheets.” Afterwards, the handsheets were kept at $27 \pm 2^\circ\text{C}$ temperature and $65 \pm 5\%$ relative humidity for about 24 h as per ISO 187:1990 “Paper, board and pulps—Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples” before further analysis of physical properties.

RESULTS AND DISCUSSION

Assessment of chemical components

The chemical composition of natural fibers varies with their place of origin. The main components found in the natural fibers are lignin, holocellulose (cellulose and hemicellulose), and a small amount of pectin and extractives. The chemical composition of ANL fibers were measured and are depicted in **Table IV**. It was noted that ALN contains more than 60% of holocellulose in its fibrous mass and is approximately comparable to the other materials, which are frequently used by the paper industry, as listed in **Table V**.

Active alkali dose optimization during pulping

Unbleached 15 ± 1 kappa number pulps were generated using different doses of AA with and without the cooking aid, 0.05% anthraquinone, during pulping with all other conditions (e.g., sulfidity, temperature, time, and liquid-to-solid ratio) held constant. As shown in **Table VI**, 20% AA as Na₂O was needed to produce the targeted 15 kappa pulp of 38.8% yield with 1.1% rejects. It was observed that, by adding a cooking aid, a 15%–20 % kappa reduction can be achieved for the same AA charge and that pulping yield increased by 7%.

Optimization of dosages of ClO₂ during the D₀(EOP)/D₁ stage

The first stage of a bleaching sequence is known as the delignification stage (D₀-stage), as most of the residual lig-

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Parameter	Ex-1	Ex-2	Ex-3	Ex-4	Ex-5	Ex-6
AA, %	16	18	20		22	
Pulping additives, %	–	–	–	0.05	–	0.05
Residual AA, g/L	2.6	4.1	5.1	5.6	6.5	9.6
pH, black liquor	11.6	11.8	12.1	12.0	12.4	12.3
Kappa number	29.9	25.8	18.0	15.2	14.4	11.5
Unscreened pulp yield, %	39.1	38.8	35.9	38.5	33.8	36.2
Reject, %	1.7	1.5	1.2	1.1	1.0	0.9

VI. Active alkali (AA) dose optimization for targeted kappa pulp.

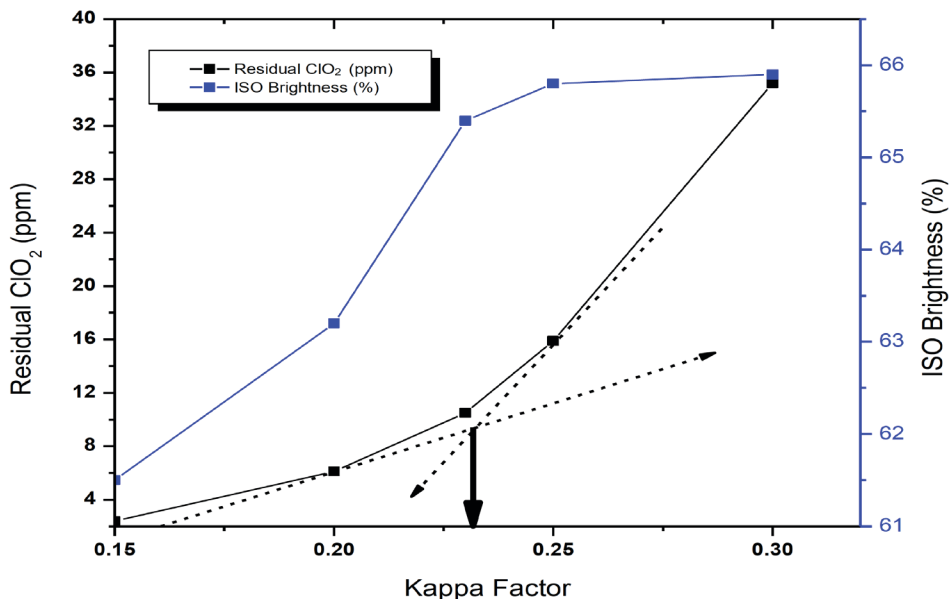
Particular	Ex-1	Ex-2	Ex-3	Ex-4	Ex-5
Kappa no.	15.0				
D₀-Stage (retention time: 45 min; temperature: 55°C; consistency: 5%)					
Kappa factor	0.15	0.20	0.23	0.25	0.30
ClO ₂ added, %	0.86	1.14	1.31	1.42	1.71
End pH	2.5	2.4	2.6	2.4	2.6
Residual ClO ₂ , ppm	2.4	6.1	10.5	15.9	35.2
ClO ₂ consumed, %	99.5	99.0	98.7	97.9	96.1
(EOP)-Stage (H₂O₂: 0.5%; temperature: 75°C; time: 90 min; end pH: 10.5)					
Brightness, % ISO	61.5	63.2	65.4	65.8	65.9
(EOP) kappa number	2.2	1.9	1.6	1.5	1.5

VII. Optimization of chlorine dioxide (ClO₂) charge at D₀ stage.

nin is removed at this stage. Different kappa factors were tested to optimize the ClO₂ charge, and a kappa factor of 0.23 was determined to be the optimum ClO₂ charge, as shown in **Table VII** and **Fig. 1**. The dotted line in Fig. 1 represents the tangent drawn on the curves of X-Y plane, and the point of intersection of the tangent is shown as the optimum kappa factor. Chlorine dioxide optimization at the D₁ stage was carried out by applying various doses of the chemicals from 0.5%–2.0% on the ovendry pulp for four different sets, as shown in **Table VIII**. In the final bleaching with ClO₂, a maximum brightness of about 83% ISO was achieved, and no further improvement in the brightness of pulp was observed beyond 1.0% dose of the chemical. As a result, a 1.0% dose of ClO₂ was determined to be the optimum value in the final stage of the D₀(EOP)D₁ bleaching sequence. Our finding to achieve this brightness is of great importance, as an earlier study showed a maximum of 69.2% brightness with 17.07% hydrogen peroxide bleaching [9].

Measurement of physical properties of pulp handsheets

The physical properties of handsheets of the unbleached and bleached ALN, wheat straw, and hardwood pulps of the same freeness level and basis weight were measured to compare their behavior, as presented in **Table IX** and **Table X**. All the physical properties of ANL pulp handsheets were found to be higher than wheat straw and hardwood pulp handsheets. It was calculated that 39.3% and 2.1% higher tensile index was observed for ANL sheets than for wheat straw and hardwood, respectively, in the case of unbleached pulp handsheets. However, in the case of bleached pulp handsheets, tensile index for ANL sheets was observed at about 60.4% and 33.8% higher than for wheat straw and hardwood, respectively, as shown in Table X. Similarly, the tear index of ANL pulp handsheets was observed 49.2% and 39.7% higher than wheat straw and hardwood pulp handsheets, respectively, in the case of unbleached pulp sheets; in the case of bleached pulp



1. Residual chlorine dioxide (ClO_2) and ISO brightness (%) of pulp vs. kappa factor curve.

Particular	Results			
Kappa no.	15			
D ₀ -Stage (temperature: 55°C; time: 45 min; cy: 5%)				
Kappa factor			0.23	
ClO_2 added, %			1.31	
End pH			2.6	
Residual ClO_2 , ppm			10.5	
ClO_2 consumed, %			98.7	
(EOP)-Stage (temperature: 75°C; time: 90 min; cy: 10%)				
NaOH added, %	2.07			
H_2O_2 added	0.5			
Final pH	10.7			
H_2O_2 consumed, %	94.4			
Brightness, % ISO	65.2			
(EOP) kappa number	1.5			
D ₁ -Stage (temperature: 75°C; time: 180 min; cy: 10 %)				
ClO_2 added, %	0.5	1.0	1.5	2.0
Final pH	3.4	3.6	3.2	3.6
Residual ClO_2 , ppm	90.0	60.0	59.4	44.5
ClO_2 consumed, %	99.1	97.5	95.1	92.5
Brightness, %	80.2	83.1	83.4	83.1
CIE whiteness	79.2	82.4	82.8	82.4
ClO_2 = chlorine dioxide; NaOH = sodium hydroxide; H_2O_2 = hydrogen peroxide.				

VIII. Optimization of chlorine dioxide (D_1) dose for $\text{D}_0(\text{EOP})\text{D}_1$ bleaching of pulp.

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Parameter	Areca Leaves	Wheat Straw	Hardwood
CSF, mL (before refining)	715±10	760±10	800±10
CSF, mL (after refining)	425±10	425±10	425±10
Basis weight, g/m ²	60±2	60±2	60±2
Bulk, cc/g	2.09±0.15	2.12±0.05	2.07±0.12
Tensile index, N.m/g	33.3±2	20.2±3	32.6±2
Tear index, mN.m ² /g	6.3±0.4	3.2±0.3	3.8±0.6
Double fold, no.	45±2	19±1	25±3

IX. Mechanical properties of unbleached pulp handsheets (mean of four readings is reported, along with the standard deviation).

Parameter	Areca Leaves	Wheat Straw	Hardwood
CSF, mL (before refining)	560±10	600±10	660±10
CSF, mL (after refining)	425±10	425±10	425±10
Basis weight, g/m ²	60±2	60±2	60±2
Bulk, cc/g	2.15±0.12	2.1±0.14	2.10±0.10
Tensile index, N.m/g	64.4±3	25.49±3	42.6±2
Tear index, mN.m ² /g	10.8±0.5	4.7±0.5	4.9±0.5
Burst index, kpa-m ² /gm	4.6±0.02	3.6±0.02	3.9±0.01
Double fold, no.	151±2	20±1	30±1

X. Mechanical properties of bleached pulp handsheets (mean of four readings is reported, along with the standard deviation).

sheets, it was about 56.5% and 54.6% higher than for wheat straw and hardwood, respectively.

Blending of ANL and hardwood fiber with wheat straw

The strength properties of different blends of hardwood and ANL with wheat straw fiber are given in **Table XI** and **Table XII**. The blending of wheat straw fiber with ANL fibers showed better physical properties than blending with hardwood fiber. As can be seen from Table XI, by adding 25%, 50%, and 75% ANL fiber to the wheat straw fiber, the tensile index showed improvement by approximately 28.6%, 77.6%, and 109.4%; tear index by 21.3%, 59.6%, and 95.7%; and burst index by 5.6%, 13.9%, and 19.4%, for the respective blends. Similarly, in Table XII, when wheat straw pulp was blended with hardwood pulp at 25%, 50%, and 75%, the strength properties of wheat straw pulp also increased; tensile index improved by 12.2%, 20.0%, and 26.3%; tear index by 8.2%, 30.6%, and 51.0%; and burst index by 2.8%, 5.6%, and 5.6% for the respective blends. Thus, more improved physical properties of wheat straw pulp handsheets were observed when

blended with ANL fiber as compared to hardwood fiber. The blending of wastepaper with ANL sheath pulp has been done elsewhere to make high-quality paperboard, and improved tear, tensile and burst strength was observed [6].

CONCLUSIONS

The chemical content of Areca nut leaf fiber is comparable to wheat straw and hardwood, which are materials currently being used by the paper industry. The lignin content of the material is lower as compared to hardwood. The bleachability of the material is also good, and more than 80% ISO brightness could be achieved by using a D₀(EOP)D₁ bleaching sequence. Better physical properties in terms of tensile, tear, and burst index were observed as compared to wheat straw and hardwood pulp handsheets. The ANL fibers contain cellulose comparable to other materials and required low AA during pulping to achieve the targeted kappa pulp. Good bleachability of the pulp was also noted. The most important finding of our work was the high physical properties of paper from ANL fibers, which were higher than those for

Parameter	Wheat Straw:Areca Nut Leaves				
	100:0	75:25	50:50	75:25	100:0
Grammage, g/m ²	60±2	60±2	60±2	60±2	60±2
Bulk, cc/g	2.1±0.22	2.3±0.21	2.9±0.23	2.9±0.24	2.8±0.24
Tensile index, N.m/g	25.5±3	32.8±2	45.3±3	53.4±4	64.6±5
Tear index, mN.m ² /g	4.7±0.5	5.7±0.5	7.5±0.7	9.2±1	10.8±1
Burst index, kpa-m ² /gm	3.61±0.02	3.8±0.02	4.1±0.02	4.33±0.01	4.58±0.03
Double fold, no.	20±1	35. ±3	60±4	90±3	151±4

XI. Blending with wheat straw pulp (mean of four readings is reported, along with the standard deviation).

Parameter	Wheat Straw:Hardwood				
	100:0	75:25	50:50	75:25	100:0
Grammage, g/m ²	60±2	60±2	60±2	60±2	60±2
Bulk, cc/g	2.1±0.02	2.62±0.02	3.0±0.02	3.16±0.03	2.12±0.03
Tensile index, N.m/g	25.5±2	28.6±4	30.62±3	32.19±3	35.8±5
Tear index, mN.m ² /g	4.9±0.5	5.3±1	6.4±0.7	7.4±0.8	8.3±1
Burst index, kpa-m ² /gm	3.6±	3.7±1.5	3.8±2	3.8±1.5	3.9±2
Double fold, no.	188±5	106±7	32±3	20±2	10±1

XII. Blending of hardwood pulp with a wheat straw pulp (mean of four readings is reported, along with the standard deviation).

hardwood and wheat straw pulp handsheets. Thus, ANL fiber can be used as a substitute for hardwood pulp and where agro-based industries are challenged by the low mechanical properties of paper products. Our next step with Areca nut leaf will be to develop flexible packaging materials that require high strength properties using other agro-residues. **TJ**

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ABOUT THE AUTHORS

We chose to research this topic in order to promote use of Areca nut leaf (ANL), which is a huge agro-waste, as a raw material for making paper, as well as to mitigate raw material scarcity facing the paper industry. We found that ANL fiber can be used as a substitute for hardwood pulp and that it also imparts improved mechanical strength compared to hardwood and wheat straw pulp.

Industries are searching for alternates to scarce raw materials, and ANL provides one option. In addition, agro-based industries like paper have faced low mechanical strength in products. By using ANL, issues with mechanical properties may be minimized.

Our next step is to develop flexible packaging materials that require high strength properties using ANL in conjunction with other agro-residues.



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