PULPING AND PAPERMAKING PROPERTIES OF ZARA PLANT

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The fast-growing Zara plant (a putative hybrid of *Pennisetum purpureum* Schumach.) is usually planted for cattle fodder. For the first time, Zara plant was evaluated for its pulping potential based on anatomical, chemical, and morphological characteristics. This plant was characterized by an acceptable amount of α-cellulose (40.32%) and lignin (18.4%), with medium fiber length (1.09 mm). Soda–anthraquinone (AQ) pulping of Zara plant was investigated. Anatomically, stems of Zara plant had porous structure, mainly composed of vascular bundles surrounded by parenchyma tissue. Consequently, they were easier to delignify and defibrate, and a pulp yield of 46.54% was obtained, with kappa number 15.45 under the conditions of 14% alkali charge for 120 min of cooking at 150 °C. Good papermaking properties were observed, the tensile index was 78.9 N.m/g at 43 °SR. The resulting pulps showed good bleachability in elemental chlorine-free (ECF) bleaching sequences ($D_0(EP)D_1$). Pulp brightness of 81.5%, with the viscosity of 16.0 mPaS, was obtained, with the consumption of 25 kg ClO_2/ton of pulp.

Keywords: pulp, non-woods, pulpability, bleachability, papermaking properties

INTRODUCTION

Paper and other cellulosic products (viscose, cellulose derivatives, *etc.*), which are produced from pulp, have become an indispensable part of our daily life. Nowadays, about 90-91% of the pulp is produced from wood.[1] However, the global consumption of paper and paperboard is steadily rising, 408 million tons were consumed in 2021, and it is projected to continue rising over the coming decade to reach 476 million tons by 2032.[2] Unfortunately, the available wood supply for papermaking will not fulfill the growing demand of pulp. Moreover, there remain environmental concerns for deforestation, and thus, sustainability challenges for paper industries over the world. As an overpopulated and forest-deficient country, Bangladesh is no exception, suffering from huge shortage of fibrous raw material, stressed to meet the demand of pulp, as all the paper mills of this country have to import pulp from abroad. All pulp mills in Bangladesh have shut down because of an acute shortage of fibrous raw materials. Therefore, to keep the sustainability of the paper sector, we have to find out alternative sources of fibrous raw materials, such as agricultural residues, grasses, and annual plants, for pulp production. As an agrarian country, these alternative non-wood fibers might be the future fibers for pulp mills in Bangladesh.

Non-wood or agro-based fibers are derived from selected tissues of various mono- or dicotyledonous plants, and are categorized botanically as grass, bast, leaf, or fruit fibers. Non-wood plants can be produced within a year compared to the long growth cycles of wood.[3] In order to reduce the pressure on forests, researchers are trying to find new non-woody raw materials having pulping potential. Jahan *et al.* characterized palm grass (*Cyperus flabelliformis* Rottb.) with an extremely high slender ratio, 32.2% α-cellulose, as well as evaluated its pulpability and quite acceptable papermaking properties.[4] Ferdous *et al.* studied bagasse (*Saccharum officinarum* L.), kash (*Saccharum spontaneum* L.) and corn stalks (*Zea mays* L.), and all three non-woods showed similar chemical and morphological properties, with good papermaking properties and bleachability.[5] Bhardwaj *et al.* compared soda and soda-anthraquinone (AQ) pulping of rice straw and reported that the addition of AQ in soda pulping increased pulp yield and reduced kappa number, as well as improved strength properties.[6] Different non-woods, such as jute fiber, rice straw, chia (*Salvia hispanica* L.), golpata (*Nypa fruticans* Wurmb), okra fiber (*Abelmoschus esculentus* Moench), cotton stalks, dhaincha (*Sesbania aculeata* Pers.) and pati (*Typha* sp.), were evaluated in different processes for pulping and papermaking at the Pulp and Paper Research Division of BCSIR Laboratories, Dhaka.[7-14]

Zara plant (a putative hybrid of *Pennisetum purpureum* Schumach.) belongs to the subfamily Panicoideae, family Poaceae. It is similar to Napier grass, which grows in different regions of Bangladesh and is mainly used as animal fodder and feed. It is a very fast-growing plant and can be cultivated in any season, with low cultivation costs. However, this plant has not been evaluated yet in the search for alternative non-wood fiber. In this investigation, Zara plant was characterized in terms of anatomical, chemical and morphological properties. Pulpability, bleachability and papermaking properties of Zara were also investigated.

EXPERIMENTAL

Raw material

Zara plant was collected from the northern region of Bangladesh, sun-dried and cut into 2-3 cm pieces in length. The moisture factor of the plant was determined for the subsequent experiments.

Morphological properties

A small portion of Zara plant was macerated in a 1:1 solution of HNO_3 and $KClO_3$. A drop of the macerated sample was taken on a slide and fiber length was examined under a profile projector (Nikon V-12, Japan).

Chemical analysis

The extractive (T204 om88), 1% alkali solubility (T 212 om98), water solubility (T207 cm99), Klason lignin (T222 om83), acid-soluble lignin (TAPPI UM 250 1991) and ash content (T211 os76) were determined in accordance with Tappi Test Methods (2003–4).[15] Holocellulose was determined by treating extractive-free wood meal with $NaClO_2$ solution.[16] The pH of the solution was maintained at 4.0 by adding CH_3COOH–CH_3COONa buffer and α-cellulose was determined by treating holocellulose with 17.5% NaOH.

Anatomical and morphological properties

For anatomical analysis, plant samples were collected from 25 days old plants 30 cm above the ground level and were fixed in FAA solution. The slides for anatomical observation by light microscopy were prepared following the procedure described by Sarwar and Prodhan (2000).[16] Samples were sectioned using a sliding microtome (Leica SM2010R), having a thickness of 5.0 µm, and slides of transverse section were prepared, which were further investigated under a light microscope (Oxion Euromex Microscope, equipped with an Euromex microscope camera CMEX-18 PRO USB 3.0, 18 MP, with CMOS Sensor).

For the measurements of fiber morphological properties, these samples were macerated in a solution containing 1:1 HNO_3 and $KClO_3$. A drop of macerated sample was taken on a slide, and fiber length and diameter were measured on a Euromex-Oxion image analyzer using Image Focus Alpha software. For measuring fiber length and fiber width, 200 fibers were measured from the slides and average values were taken.

Pulping

To determine pulpability of Zara plant, pulping was carried out through soda-anthraquinone (AQ) process in a closed electrically heated digester, with a constantly rotating device, varying the alkali charge, cooking temperature and time, under the following conditions: active alkali: 12%, 14%, 16% and 18% as NaOH; anthraquinone (AQ): 0.10%; liquor to feedstock ratio: 1:8; temperature: 140 °C, 150 °C and 160 °C; time: 60, 90, 120 and 150 minutes.

After digestion, the pulp was washed untill free from residual chemicals, and screened on a flat vibratory screen (Yasuda, Japan). The screened pulp yield, total pulp yield, and screened reject were determined gravimetrically as percentage of o.d. raw material. The kappa number (T 236 om-99) of the resulting pulp was determined in accordance with Tappi Test Methods.

$D_0(EP)D_1$ bleaching

Pulps were bleached by the $D_0(EP)D_1$ bleaching sequences (where D represents chlorine dioxide and EP represents peroxide-reinforced alkaline extraction). In the first stage (D_0) of the $D_0(EP)D_1$ bleaching sequences, the pulp was bleached with 2% ClO_2 for 45 min at 70 °C, maintaining 10% pulp consistency. The pH was adjusted to 2.5 by adding dilute H_2SO_4. In the alkaline extraction stage, the pulp was extracted with 2% NaOH and 0.5% H_2O_2 (on o.d. pulp) for 120 min at 70 °C. Pulp consistency was 10%. The end pH in the D_1 stage was adjusted to 4.0 by adding dilute NaOH. In the third stage (D_1), the pulp was again bleached with 1% ClO_2 for 45 min at 70 °C. After bleaching, the brightness was determined in accordance with Tappi Test Method T525 om 92.

Evaluation of pulps

After the disintegration of Zara pulp, handsheets of about 60 g/m^2 were made in a Rapid Kothen Sheet Making Machine according to German Standard Method Nr 106. The sheets were tested for tensile (T494 om-96), burst (T403 om-97), and tear strength

(T414 om-96) according to TAPPI standard test methods.

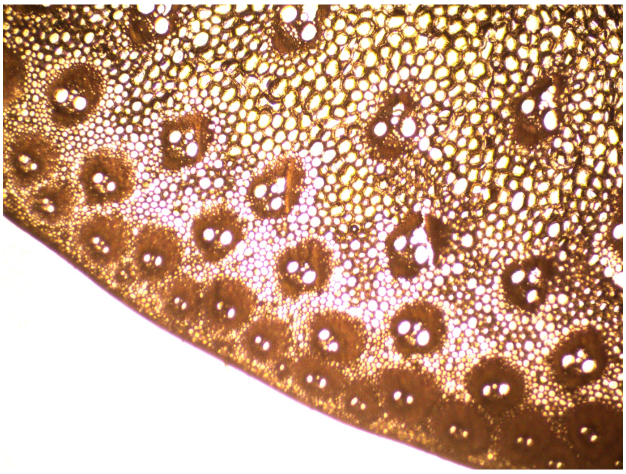
Fiber quality of screened pulp samples was determined using a Fiber Quality Analyzer–360, OpTest, Canada. The viscosity of the pulp was then measured according to Tappi Test Method T 230 om-94.

RESULTS AND DISCUSSION

Anatomical properties

The outline of Zara stems is more or less circular; the anatomical structure of the stems is broadly divided into three regions, *viz*. epidermis, hypodermis, and ground tissue (Fig. 1). The epidermis consists of single-layer elongated or squarish parenchyma cells, compact and without any intercellular space. A thin layer of cuticle, consisting of cutin, is present on the outer surface of the epidermis. Underneath the epidermis, a hypodermis layer, composed of 1-5 layers of thick-walled sclerenchyma cells, occurs as discontinuous patches intermingled with chlorenchyma, chloroplast-containing parenchyma cells (Fig. 1). The vascular bundles are conjoint, collateral, closed, and usually surrounded by a sclerenchyma bundle sheath. The bundle sheath of outer smaller vascular bundles are sometimes associated with hypodermis. The vascular bundles, consisting of xylem and phloem without cambium in between them, are scattered throughout the ground tissue, extends from below the hypodermis to the centre of the stem. Among the components of phloem tissue, the sieve tube and companion cell could be identified easily. The xylem tissue consists of xylem vessels, xylem fibre, and xylem parenchyma; vessels are commonly arranged in V-shape, rarely in Y-shape. The ground tissue consists of a mass of thin-walled, round, or polygonal-shaped parenchymatous cells having well-defined intercellular spaces among them.

There are no significant differences between the stem anatomy of Zara grass and its parent species *P. purpureum* (Napier grass).[17] The internal structure (anatomy) of the Zara (plant) stem is also similar to that of other most common non-wood pulp and paper-making materials like rye (*Secale cereale* L.), wheat (*Triticum aestivum* L.), maize (*Zea mays* L.), and others.[18-20] These plant bodies are rich in uniform thin-walled parenchyma tissue and a relatively small amount of thick-walled sclerenchyma tissue. The deposition of lignin is closely linked with the process of cell wall thickening, making cell walls rigid and waterproof. The higher amount of holocellulose and low lignin content in the cell wall of parenchyma tissue might be used as an indicator of the better quality of the paper produced.[18]



Figue 1: Transverse section of Zara stem

Chemical composition

Table 1 shows the chemical characteristics of Zara fiber and compared with whole bagasse. The Klason lignin content in Zara was 20.19%, which is very close to the Klason lignin content in kash and corn stalk and slightly lower than in whole bagasse.[5] As shown in Table 1, the acid soluble lignin (ASL) in Zara was about 1% higher than in bagasse. ASL is composed of low-molecular-weight degradation products and hydrophilic derivatives of lignin with the syringyl nucleus.[21] The holocellulose content in Zara fiber was 64.6%, which was better than those of corn stalk and bagasse,[5] and lower than that of bamboo.[22] The holocellulose content in Zara fiber is very close to the holocellulose content in wheat straw and better than in rice straw, those two non-woods being commonly used for pulping.[23] Holocellulose is the sum of α-cellulose and hemicelluloses. The α-cellulose content in Zara (40.3%) was very close to the α-cellulose content in bagasse (39.3%). The α-cellulose content is directly related to pulp yield and papermaking properties,[24] therefore, this parameter is very important in selecting a pulping raw material.

The pentosan content in Zara plant was slightly higher than in bagasse (16.9% vs 16.8%). Hemicelluloses act as a strength promoter of pulp. Mobarak *et al*. (1973) extracted hemicelluloses from hardwood and rice straw pulps, and added to the pulps to restore the original pentosan content.[25] The obtained paper sheets showed higher strength. The addition of 1% (w/w) of hemicelluloses to cellulosic pulp increased papermaking properties by 30%.[26]

Table 1
Chemical and morphological properties of Zara plant

Parameters	Zara	Bagasse (Ferdous *et al*. 2020)[5]
Extractives (%)		
Cold water	13.24	8.4
Hot water	36.93	12.13
1% NaOH	39.19	39.6
Ethanol:benzene (1:2)	2.19	2.2
Holocellulose (%)	64.61	62.2
α-cellulose (%)	40.32	39.3
Pentosan (%)	17.95	16.9
Klason lignin (%)	18.42	20.4
Acid-soluble lignin (%)	3.10	2.28
Ash (%)	5.17	0.67
Fiber length, L (mm)	1.09	2.28
Fiber diameter, D (μm)	21.14	20.4
Slender ratio (L/D)	51.56	79.2

Table 2
Effect of different parameters on yield and kappa number of pulps obtained from Zara plant

NaOH (%)	AQ (%)	Cooking temperature (°C)	Cooking time (min)	Pulp yield (%)			Kappa Number
				Screened	Reject	Total	
12	0.10	140	120	46.24	4.24	50.48	21.40
14	0.10	140	120	44.99	4.02	49.01	18.38
16	0.10	140	120	43.93	3.47	47.40	15.80
18	0.10	140	120	42.72	2.87	45.59	14.03
12	0.10	150	120	45.15	2.16	47.31	18.68
14	0.10	150	120	45.56	1.28	46.54	15.46
16	0.10	150	120	43.34	0.67	44.01	14.17
18	0.10	150	120	42.51	0.17	42.68	12.03
12	0.10	160	120	43.03	0.98	44.03	16.23
14	0.10	160	120	42.66	0.35	43.01	14.01
16	0.10	160	120	40.77	0.13	40.90	12.15
18	0.10	160	120	39.12	0.08	39.20	11.40
14	0.10	150	60	39.54	8.97	48.51	20.87
14	0.10	150	90	42.07	5.25	47.32	19.46
14	0.10	150	150	43.01	1.08	44.09	14.01

Cold water solubility in Zara plant was 13.24%, while hot water solubility was 36.93%. This is attributed to the presence of higher inorganics, tannins, gums, sugars and lower molecular carbohydrates. The one percent alkali solubility in Zara plant was 39.19%, which was very close to the alkali solubility of bagasse (Table 1). This value was lower than the one percent alkali solubility in whole corn stalk in different locations of USA, as reported by Byrd *et al*.[27] Ethanol/toluene solubles in Zara plant reached 2.19%, while the ethanol-benzene extract in whole bagasse was 8.5% according to Andrade *et al*.[28] Extractives in Zara plant were found slightly higher as compared to other grasses.[29] The extractives are dissolved or saponified in alkaline pulping, and have the tendency to form pitch, and inhibit the delignification through the formation of condensates with lignin.[30]

The ash content in Zara plant was higher than in hardwood,[31,32] but comparable to other straws,[5,33] which caused rather serious difficulties during chemical recovery and poor drainage of pulp during papermaking.

The average fiber length and diameter of Zara plant was 1.09 mm and 21.14 µm, respectively, which are within the non-wood category.[33] As shown in Table 1, the fiber length of Zara plant was shorter than in bagasse. A higher fiber length contributes to higher tearing strength of paper.[5,34] The slenderness ratio of Zara was 51.56. Generally, the acceptable value for slenderness ratio for papermaking is more than 33.[35] The higher slenderness ratio means longer fiber with thin width, which is expected to produce stronger paper.

Soda-anthraquinone (AQ) pulping

The pulping potential of Zara plant was optimized by varying alkali charge, cooking temperature, cooking time with respect to pulp yield and degree of delignification (Kappa number) through the soda-anthraquinone (AQ) process. As shown in Table 2, total pulp yield and kappa number decreased with increasing alkali charge, cooking time and increasing temperature. Zara plant fibers were anatomically loosely bonded like those of bagasse, straw, kash, corn stalk, *etc*., therefore, it was expected that the defibration of Zara plant could be achieved at lower temperature.[5,36,37] In 120 min of cooking at 140 °C, total pulp yield and kappa number decreased from 50.48% to 45.59% and from 21.40 to 14.03, respectively, with increasing active alkali charge from 12% to 18%. Similarly, pulp yield and kappa number decreased from 49.01% to 43.01% and from 18.38 to 14.01, respectively, with increasing cooking temperature from 140 °C to 160 °C at 120 min of cooking with 14% alkali charge. Screened pulp yield increased from 39.54% to 45.56% with increasing cooking time from 60 min to 120 min with 14% alkali charge at 150 °C, further increase of cooking time decreased screened pulp yield. Screened pulp yield of Zara plant (45.56%) at 14% alkali charge and 150 °C was higher than in bagasse (36.11%), corn stalks (32.82%), kash (43.77%) under similar cooking conditions,[5] and was also higher than in chia and wheat straw.[9,23] This can be explained by the higher amount of α-cellulose content (Table 1). The delignification of Zara was much easier than that of some other non-woods, as the kappa number of Zara was 15.46 at 14% alkali charge and 150 °C for 120 min of cooking, which was much lower than that of bagasse (33.43, same conditions),[5] kash (41.61, same conditions),[5] chia (83.3, 14% alkali at 170 °C),[5] jute stick (32.5, 16% alkali at 170 °C),[9] dhaincha (19.4, 16% alkali at 170 °C)[38] but lower than in corn stalks (9.95%),[39] as corn stalks contain less lignin than Zara.

Figure 2 shows the relationship between kappa number and alkali concentration at different cooking temperatures, where kappa number decreased linearly with increasing alkali concentrations, as the concentration of neucleophile (OH^-) increases, resulting in higher delignification rate. The delignification rate at 150 °C was higher than that at 140 °C and 160 °C. This is why pulping at higher temperature increases the delignification ability of the pulping system, but in the final phase delignification has slowed down considerably due to depletion of reactive lignin units. Figure 3 shows the relationship between total pulp yield and kappa number of Zara plant pulp at different cooking temperatures with respect to alkali charge, where both pulp yield and kappa number decrease with increasing cooking temperature, as a certain number of α-O-4 (at initial phage) and β-O-4 (at bulk phage) ether linkages within the lignin macromolecules are cleaved mainly due to nucleophilic addition reactions through the formation of quinonemethide-intermediate.[40] As shown in Figure 2, the total pulp yield of Zara plant at kappa number of 15.46 was 46.54% at 150 °C, which was 0.9% lower than the pulp yield at 140 °C at the same kappa number. At these temperatures, the screened reject was 1.28% and 4.02%, respectively. Therefore, the optimum cooking temperature was 150 °C for Zara plant pulping.

The screened reject of Zara plant in soda-AQ pulping also decreased with increasing alkali charge, cooking time and temperature. The screened reject was very high at 140 °C, it was

2.87% even with 18% alkali charge. The maximum screened reject (8.97%) was observed in 60 min of cooking at 140 °C in 14% alkali charge, which decreased to 1.08% with increasing the alkali charge to 18%. In practice, the rejects were returned to the digester and cooked again with the incoming raw material. Tran cooked knots from hardwood fiber line with 15% and obtained screened yield approximately 65% and rejects 0.2%, and estimated the total pulp yield as the sum of the screened pulp yield and 65% of the rejects.[41] Therefore, the estimated total pulp yield will be higher.

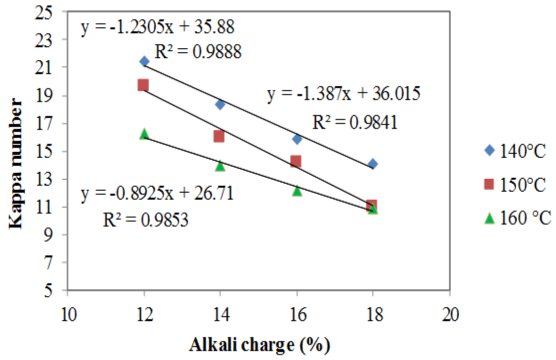


Figure 2: Delignification with respect to alkali charge in soda-AQ pulping of Zara at different temperatures

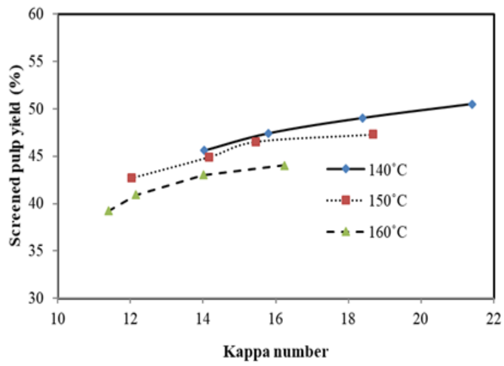


Figure 3: Variation of pulp yield with kappa number in soda-AQ pulping of Zara at different temperatures

Table 3
Papermaking properties of Zara plant

Beating time (min)	°SR	Tensile index (N.m/g)	Tear index (mN.m^2/g)	Burst index (kPa.m^2/g))	Breaking length (Km)
0	18.5	46.19	5.56	3.01	4.71
10	30.0	76.24	5.43	4.07	8.05
20	43.0	78.90	4.62	4.11	7.78
30	56.0	82.34	4.38	4.81	8.40
40	65.0	88.17	3.79	4.85	9.00

Table 4
Fiber quality analysis of Zara pulp

Measured parameters of Zara pulp	Results
Fines (%)	32.40
Mean length (mm)	0.83
Mean width (µm)	21.14
Mean curl index	0.07
Mean kink index (mm^{-1})	0.86
Degree of external fibrillation (%)	0.90
Coarseness (mg/m)	0.15

Papermaking properties

The papermaking properties of Zara plant pulps at different beating degrees are shown in Table 3. In the unbeaten state, the drainage resistance (°SR) was 18.5, which was close to that of other non-wood soda-AQ pulps.[5] The °SR value in the unbeaten state is very important for pulp processing, a higher value hampers pulp washing and screening. The tensile index of the pulp in the unbeaten state was 46.19 N.m/g. A good tensile index of Zara plant pulp in the unbeaten state can be explained by higher fines (32.4%) and external degree of fibrillation (0.9%), which facilitates fiber bonding (Table 4). As expected, tensile and burst indexes increased and tear index decreased with beating degree. The tensile and burst indexes increased from 46.19 N.m/g to 78.9 N.m/g and from 3.01 kPa.m^2/g to 4.11 kPa.m^2/g, while tear index decreased from 5.56 mN.m^2/g to 4.62 mN.m^2/g with increasing

°SR value from 18.5 to 43. These values were better or similar to those of other non-wood pulps[42,5,23] and higher than those of tropical hardwoods.[31,43-44]

Table 4 shows the average weighted length, width, fine percentage, curl, kink indexes, degree of fibrillation and coarseness of Zara plant pulp. These parameters of pulp fiber impact the properties of the pulps and the quality of the produced papers. The fines content was 32.4%, which are generated from non-fibrous parenchymatic cells. The high fines content may correspond to more flexible fibers. Paper formation is greatly affected by fines and fiber length. Reducing fibre length has little direct effect on the sheet structural and optical properties, but does reduce papermaking properties.[45] The coarseness of pulp fiber is related to the fiber wall thickness and influences the fiber collapse degree and development of bulk.[46] This parameter of pulp is important for tissue production.[47] The coarseness of Zara plant pulp was 15.0 mg/100 m, which was higher than in bagasse, corn stalk and kash pulps.[5]

Bleaching

The pulp was bleached by the $D_0(EP)D_1$ bleaching sequences. The final pulp brightness was 81.5% using 25 kg ClO_2/ton of pulp, which was comparable to other non-wood pulps.[5] The bleached pulp viscosity was 16 mPa.s. The viscosity of Zara plant pulp was better than that of bagasse, corn stalk and kash pulps.[5]

Table 5
Pulp brightness and viscosity of bleached Zara pulp

Parameters	Results
Bleaching yield (%)	84.2
Brightness (%)	81.5
Viscosity of bleached pulp (mPa.S)	16.01

CONCLUSION

Zara plant is characterized by a good amount of α-cellulose and lower content of lignin, with medium fiber length, and the xylem tissue consists of vessels, fiber and parenchyma. Anatomically, Zara plant stems have a porous structure, mainly composed of vascular bundles surrounded by parenchyma tissue; consequently, the delignification was easier, and the kappa number of the pulp reached 15.46 using only 14% alkali charge at 150 °C for 2 h, where pulp yield was 46.54%. The tensile index of unrefined pulp was very high (46.19 N.m/g) due the high percentage of fines (32%), this value increased to 88.17 N.m/g at 65 °SR. The pulp also showed good bleachibility and proved suitable for printing and packaging grade paper.

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