Identifying the Conditions Required for the NaOH Method for Producing Pulp and Paper from Sorghum Grown In Turkey

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This study examines the possibilities of making pulp from sorghum grown in Bartın *via* the NaOH method. While setting the cooking conditions for producing pulp from annual plants, NaOH was used in quantities that amounted to 14, 16, 18, and 20% of the total weight of the dry sample. Optimum conditions were established relative to pulp yield, Kappa number, and a scoring table based on mechanical, optical, and physical values. Accordingly, optimum conditions for pulp production from sorghum stalks occurred when 20% NaOH was used at 120 °C, a 5:1 solution-to-stalk ratio, and a cooking time of 60 min.

Keywords: Sorghum bicolor sudanense; Pulp and paper; NaOH method

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INTRODUCTION

In parallel with an increasing population, increases in the demand for products with wood as the raw material will make it necessary to use non-wood replacement products. The ages of trees used for industrial manufacturing purposes vary depending on the species, but some can be as little as 5 to 10 years. Others could be a few times longer than the average human lifetime. Thus, there would be significant financial benefit associated with the use of annual plants as a source of raw material whenever possible. Therefore, growing annual plants as a source of raw material for energy and fiber has become a necessity.

Sorghum bicolor x *S. bicolor* var. *sudanense* is an annual summer plant (Almodares and Hadi 2009). Higher amounts of biomass can be obtained from this plant (Barbanti *et al.* 2006) with low precipitation than other plants that are sources of sugar (Neal *et al.* 2011). Keskin *et al.* (2005) conducted a study in Turkey and found the dry grass efficiency of sorghum to be between 1.40 to 1.54 tons per decare (1 daa = 1000 square meters). In Turkey, the biomass increase in forest stands is 217.50 kg/daa for red pines aged between 0 and 50 years (Durkaya *et al.* 2009), 90.13 kg/daa for Calabrian pine (Durkaya *et al.* 2010a), and 115.33 kg/daa for Crimean pine (Durkaya *et al.* 2010b). Sorghum is an important raw material due to its greater biomass efficiency compared to forest trees. Although the conditions in Turkey make it necessary to cultivate the land every year, which increases planting costs, this increase is outweighed by the optimal use of the soil and the high biomass productivity of the plants.

When sweet sorghum is examined, the pith and rind consist of different proportions of chemical components. While water-soluble sugar is high in the pith, the rind is richer in lignin and fiber. Therefore, the rind is a convenient raw material in the pulp and cellulose industry (Billa *et al.* 1997). In addition, when the pith is taken while green, the features of the resulting paper are improved. When cooking the rinds of sorghum, adding soda-anthraquinone increases delignification (Khristova and Gabir 1990).

In determining the optimum conditions in making pulp, pulps that have the highest yield and the lowest Kappa numbers are preferred. It has become a tradition to prefer and accept pulp that produces paper with the best mechanical and optical values. Acquiring all of the desired features in a single pulp is, for the most part, impossible. Thus, it is very difficult to identify optimum cooking conditions. In order to determine the optimum cooking condition, a scoring table (Table 7) was developed.

A literature review indicated that sorghum is examined by separating its pith and rind parts. These practices are not economical in industrial applications because they result in the loss of time and increased labor. Since the aim of our study was to look into the applicability of sorghum grown in Turkey as an industrial material, all stalk and leaves were used to make paper after the seeds were removed.

EXPERIMENTAL

Sweet sorghum, *i.e.*, *Sorghum bicolor* x *S. bicolor* var. *sudanense*, which is a variety of nutria honey, was planted in Bartın in May. Five kilograms of seeds were planted per decare. The altitude of the area where the sorghum was planted was 28 m, the slope was 5 to 6%, and the exposure of the area was towards the southwest. Bennett and Anex (2009) indicated that the harvesting season of sorghum is around 90 days after planting. Hence, after a three-month growing period, the plants were harvested and dried in the sun. Following an 8 day drying period, the plants were stored in a half-open location in which they were shielded from rain and direct sunlight. After the plants were dried, the average length of the samples was 212 cm and the average diameter was 1.20 cm. Of the total mass of a sweet sorghum plant, 70 to 75% is stalk, 10 to 15% is leaves, and 7% is grains.

The chemical components of the sorghum, *i.e.*, α-cellulose (Han and Rowell 1997), holocellulose (Browning 1967), and TAPPI T 222 (2002) standards were used for determining lignin. TAPPI T 207 (1999) was used for cold-hot water solubility, TAPPI T 204 (1997) was used for alcohol solubility, and TAPPI T 212 (2002) was used for 1% NaOH solubility. Belayachi and Delmas (1995) found the highest efficiency using depithed sorghum when the solution-to-stalk ratio was 3:1 with the addition of 14% NaOH, and, when they increased the ratio to 5:1 and 7:1, the yield decreased. Since the whole stem was used in this study, the pith and leaves absorbed a significant amount of the solution that was added. Therefore, the 3:1 solution-to-stalk ratio was inadequate, so the ratio was changed to 5:1.

Huang *et al.* (2008) found the highest yield in depithed sorghum stalks at 150 °C when cooking was done using NH_4OH-KOH-AQ. However, they also found that carbohydrate degradation occurred and yield decreased with the increase of temperature. Because NH_4OH and KOH are weaker alkalis than NaOH, this temperature may be necessary for the reaction. However, since NaOH is a strong base, in this study the cooking process was initiated at a lower temperature (120 °C). As in the conditions shown in Table 1, the eight pulps that were produced from the samples were cut into 5 cm pieces and cooked for a total of 130 min (including 70 min of heating and 60 min of cooking at the maximum temperature).

Table 1. Conditions Used to Make Pulp from Sorghum

Cooking No.	NaOH (%)	Maximum Cooking Temperature (°C)
1	14	120
2	14	130
3	16	120
4	16	130
5	18	120
6	18	130
7	20	120
8	20	130

Pulp was produced by placing 600 g of dry raw material in a rotating electric boiler. Ten sample papers weighing 75 ±2 g/m^2 were made from the pulp beaten up to 50 °SR according to the ISO 5269-2 (2013) standard. The thicknesses of the sample papers were measured after they were conditioned at 23 ±2 °C and 50 ±2% relative humidity for 24 h according to the TAPPI T 402 (2003) standard. Breaking length, burst index, and tearing index were determined according to TAPPI T 494 (2001), TAPPI T 403 (2002), and TAPPI T 414 (1998) standards, respectively. The opacities of the papers were determined according to the TAPPI T 519 (2002) standard, and brightness was determined according to TAPPI T 525 (2002). Surface smoothness was determined according to ISO 8791-2 (2013), and air permeability was determined according to the ISO 5636-3 (2013) standard. Statistical evaluation was conducted using SPSS statistical software.

In this study, the highest values of breaking length, brightness, burst index, tearing index, opacity, whiteness, and surface smoothness at 50 SR° and the values at which the difference between those values was not statistically significant at 5% were taken into account, and a scoring table (Table 7) was developed in order to determine the optimum condition. Moreover, the highest values (*) and the value(s) at which the difference between those values were not statistically significant at 5% were each graded as 1, while all of the remaining values were graded as 0.

In the bleaching process, the level of bleaching of the pulp increases as the Kappa number decreases. Therefore, the lowest value is essential. While writing the Kappa numbers in the table, the lowest values and the value(s) in which the difference between those values is not statistically significant at 5% were each graded as 1, while all of the remaining values were graded as 0.

RESULTS AND DISCUSSION

To be consistent with industrial practice, the seeds were removed from the sorghum plant in this study. The chemical analyses were conducted for the samples, including all of the body and leaves of the plants (but excluding the seeds), and the results are compared with the results of other similar studies in Table 2. As can be seen in Table 2, the holocellulose, lignin, and α-cellulose contents of sorghum stalks were 71.0%, 13.0%, and 40.3%, respectively. Belayachi and Delmas (1995) found the holocellulose content in depithed and extracted samples to be 69.0 and 61.6%, respectively. In the same study, the lignin contents in the depithed and extracted samples were 16.1 and 14.9%, respectively.

Table 2. Comparison of the Results of the Chemical Analysis of Sorghum Stalks with the Results of Other Studies

Components (%) and solubility	Experimental	DE*	E*	DE**	E**	DE†
Holocellulose	71.0	68.0	61.6	69.0	61.6	-
Alpha-Cellulose	40.3	-	-	-	-	-
Cellulose	-	48.8	45.0	48.8	45.0	46.2
Lignin	13.0	16.0	14.9	16.1	14.9	22.9
Alcohol	15.3	-	-	-	-	-
Alcohol-benzene	-	11.4	24.0	11.4	24.0	2.7
1% NaOH solubility	47.1	52.1	63.1	52.1	63.1	36.1
Hot water solubility	19.7	25.3	43.8	23.3	43.8	4.2
Cold water solubility	15.1	-	-	-	-	2.7

DE= depithed extracted; E= extracted

*Belayachi and Delmas 1997, ** Belayachi and Delmas 1995, †Huang *et al.* 2008

The 1% NaOH solubility of sorghum stalks was 47.1% (Table 2). The solubilities of the depithed and extracted samples in 1% NaOH were 52.1 and 63.1%, respectively. Belayachi and Delmas (1997) found that the holocellulose contents in depithed samples and the extracted samples were 68.0 and 61.6%, respectively. In the same study, the lignin contents in the depithed samples and the extracted samples were 16.0 and 14.9%, respectively. Qi *et al.* (2013) found the lignin contents in the pith and the rind of crushed sweet sorghum were 17.4 and 19.7%, respectively. The proportional value of the rind, in which cellulose and lignin are mostly found, increases with the removal of the pith or squeezing the stalks; it is inevitable that the results of this study would be different. In addition, since the extraction content for the extracted samples were at different degrees, it is possible that they would be incompatible with our study.

The morphological features of the sorghum stalk are given in Table 3. The length of the fibers is an important criterion in determining the compatibility of a raw material for pulp production.

Table 3. Some Morphological Features of Sorghum Fibers

	Sorghum rind			Sorghum leaves		
Dimensions	Mean	Standard deviation	Variance of coefficient	Mean	Standard deviation	Variance of coefficient
Fiber length (mm)	2.31	0.59	0.25	1.38	0.36	0.26
Fiber width (µm)	16.0	3.45	0.22	10.26	4.50	0.44
Lumen diameter (µm)	5.58	2.80	0.50	4.44	3.51	0.79
Wall thickness (µm)	5.21	1.42	0.27	2.91	0.99	0.34

In this study, the fiber length in the leaves of sweet sorghum was measured as 1.38 mm. This value was 0.74 mm in wheat straw (Deniz *et al.* 2004), which is a significant product in agriculture in Turkey and a significant source of fiber; it also was found that the length of the fibers in cotton stalks was 1.01 mm (Gençer *et al.* 2001), and that it was 1.15 mm in rye straw (Usta and Eroğlu 1987).

Considering these facts, it is possible to say that sorghum is convenient for pulp production. Dutt *et al.* (2008) found fiber lengths in *Ipomea carnea*, *Cannabis sativa*, *Picea abies*, and *Pinus kesiya* to be 0.6, 1.8, 2.3, and 2.3 mm, respectively. It can be said that the length of the rind fibers of sorghum stalks (2.31 mm) make them a viable substitute in pulp production, replacing the above-mentioned species.

The elasticity coefficient, modulus of rigidity, Runkel ratio, and felting ratio of sorghum rind and leaf are given in Table 4. Elasticity coefficient depends on the individual elasticity of fibers. Rind and leaf elasticity coefficients of sorghum are found as 34.90 and 43.28, respectively. Such fibers are in the flexible fibers group and are preferred for paper manufacturing. The modulus of rigidity of rind and leaf are determined as 34.90 and 43.28, respectively. The high modulus of rigidity is the result of the wall thickness of the fiber. Runkel ratios of rind and leaf are found as 1.87 and 1.31, respectively. This group of fibers has a thin wall of thickness. They are more easily collapsed during paper manufacturing compared to fibers with thick walls and produce more inter-fiber connections. The felting ratio of many hardwood fibers is found below 70. That ratio being below 70 shows that the resistance properties of paper decrease (Albert *et al.* 2011). The felting ratios of sweet sorghum rind and leaf are determined as 144.65 and 134.65, respectively. With these values, sweet sorghum rind and leaf can be used for pulping.

Table 4. Morphological Features of Sorghum Fibers

Features	Sweet Sorghum rind	Sweet Sorghum leaf	*Sorghum halepense* (Albert *et al.* 2011)
Elasticity coefficient	34.90	43.28	33.79
Modulus of Rigidity	32.55	28.36	33.05
Runkel Ratio	1.87	1.31	1.90
Felting Ratio	144.65	134.65	90.37

Screened yield, reject, and total yield for the pulp from sorghum stalk and the Kappa number are given in Table 5. The highest total pulp yield was obtained at a NaOH ratio of 14%, and it decreased because of the increase in the degradation of lignin due to the increased NaOH ratio. The decreases in the Kappa number validated this conclusion.

Using the soda-AQ method, Belayachi and Delmas (1997) cooked sorghum samples that had been depithed and extracted separately at 160 °C for 20 min for chemical pulp production. The screened yield in this study was found to be 43% in the depithed samples and 31.7% in extracted samples. When the change of the Kappa numbers given in Table 5 based on NaOH ratio was examined, it was evident that the Kappa number decreased as the NaOH ratio increased. According to the results of the Tukey test, except for the decrease in Kappa number when the NaOH ratio increased from 16% to 18%, the decreases in the Kappa numbers observed as the NaOH ratio increased were statistically significant at the 95% level. The lowest Kappa number in this study was 7.45 in cooking number 8. Belayachi and Delmas (1997) found it to be 10.3 in depithed pulp and 9.4 in extracted whole sorghum. Belayachi and Delmas (1995) found the pulp yield to be 45.8%, and the Kappa number was 11.6 when they took the solution/stalk ratio as 5/1 in 14%

NaOH. The screened yield at 14% NaOH and the 5/1 solution/stalk ratio in our study was 43.38% at 120 °C and 43.56% at 130 °C, and the Kappa numbers were 13.40 and 12.65, respectively.

Table 5. Some Pulp Properties of Sorghum Stalk

Cooking No.	Screened yield (%)	Reject ratio (%)	Total yield (%)	Kappa Number
1	43.38	4.94	48.32	13.40
2	43.56	2.87	46.43	12.65
3	42.96	2.78	44.44	12.10
4	43.87	1.59	44.36	11.05
5	40.12	2.85	42.97	10.65
6	41.84	0.99	40.83	9.85
7	38.39	1.01	38.90	9.45
8	38.00	0.56	38.56	7.45

Physical and optical properties for paper samples from sorghum stalks are given in Table 6. Surface smoothness increased as the NaOH ratio increased, becoming almost stable at 18 to 20%. This is because the cellulose increased proportionally. The porosity of the paper increased as the NaOH ratio increased. As a result, resistance to air permeability decreased. Fluctuation is one of the results of this. When the NaOH ratio was increased from 14% to 16%, breaking length decreased by 8% and as the NaOH ratio increased from 18% to 20%, breaking length increased by 7.6%.

Table 6. Physical and Optical Features of the Paper Samples of Sorghum Stalks

CN	T (μm)	SS (mL/min)	AP (mL/min)	O (%)	B (%)	BL (km)	BI (kPa.m^2/g)	TI (mN.m^2/g)
1	113.83	442.97	79.63	96.10	30.53	2.17	3.80	30.90
2	112.33	444.47	65.51	96.04	30.82	1.99	3.59	30.77
3	113.17	482.93	62.15	96.06	31.79	1.96	3.81	32.88
4	112.17	501.07	33.23	96.00	32.11	1.86	3.74	32.52
5	112.00	544.23	37.27	95.99	35.08	2.06	3.73	34.92
6	112.17	541.57	57.10	96.10	35.27	2.09	3.69	34.00
7	112.00	540.80	66.20	96.09	36.87	2.25	3.56	37.53
8	112.00	532.50	69.35	96.17	37.73	2.28	3.52	37.32

CN=Cooking No, T=Thickness, SS=Surface Smoothness, AP=Air permeability, O=Opacity, B=Brightness, BL=Breaking length, BI=Burst index, TI=Tear index

The air permeability of paper increases as the delignification effect and the hemicellulose ratio of NaOH decrease; as a result, the burst index decreases. In the present study, the burst index under optimum conditions was found to be 3.56 kPa.m^2/g, whereas this value was 3.85 kPa.m^2/g (İstek and Özkan 2008) in papers produced *via* the Kraft method from *Populus tremula*. Under these conditions, when considered in terms of burst index, sorghum could be considered to have similar features to *Populus tremula*, which is a hardwood. For both temperatures, as the NaOH ratio increased, the proportional cellulose in the pulp increased with lignin delignification. Therefore, the pulp's resistance features increased, which increased the tear index. Lignin gives the brown color to the pulp, and, as the lignin content decreased with the increasing NaOH ratio, whiteness and brightness (a function of whiteness) also decreased.

The gaps between the fibers in the paper increased when the NaOH ratio was increased at 18%. Thus, the light permeability of the paper increased. The opacity value decreased because the light scattering index of cellulose is higher than that of air. With the increasing of temperature from 120 to 130 °C and increasing of NaOH from 18 to 20%, the ease of passage of light through the paper decreased. Therefore, the opacity value increased and the fluctuation is a result of all these.

Table 7 provides the averages of the physical, mechanical, and optical features of the papers that were produced. Scoring was done based on these values, and the scores in the columns are summed from top to bottom; the column where there are the samples which were cooked at 120 °C, and in which 20% NaOH was used based on complete dry sample length, had the highest value with 6 points. As a result, it was found that optimum values for pulp production from sorghum with the NaOH method were 120 °C, a 20% NaOH ratio, a 5:1 solution-to-stalk ratio, and 60 min of cooking.

Table 7. Scoring of Pulp and Papers Results

Cooking parameters								
NaOH Ratio (%)	**14**		**16**		**18**		**20**	
Temperature (°C)	**120**	**130**	**120**	**130**	**120**	**130**	**120**	**130**
Tear index	0	0	0	0	0	0	1(37.53)*	1(37.32)
Breaking length	0	0	0	0	0	0	1(2.25)	1(2.28)*
Burst index	1(3.80)	0	1 (3.81)*	0	0	0	0	0
Brightness	0	0	0	0	0	0	1(36.87)*	1(3673)
Opacity	1 (96.10)	0	0	0	0	1(96.10)	1(96.10)	1(96.17)*
Screened yield	1 (43.38)	0	0	1(43.87)*	0	0	0	0
Kappa number	0	0	0	0	0	0	1(8.45)	1(7.45)*
Surface smoothness	0	0	0	0	1 (544.23)*	1(541.57)	1(540.80)	0
Total	4	0	1	1	1	2	6	5

CONCLUSIONS

1. Pulp production from sorghum stalks requires fewer chemicals, less process time, and lower temperature compared to wood material, based on previous studies' data. In the present work it was found that sorghum can be used as an alternative raw material for pulp production.
2. The lengths of the rind and leaf fibers of sorghum stalks (2.31 and 1.38 mm) make them a viable substitute in pulp production. On the other hand, mechanical, physical and optical performance of sorghum handsheets presented satisfactory results.

3. Optimum values for pulp production from sorghum with the NaOH method were determined as 120 °C, a 20% NaOH ratio, a 5:1 solution-to-stalk ratio, and 60 min of cooking.

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Article submitted: December 17, 2014; Peer review completed: February 20, 2015; Revised version received: March 13, 2015; Accepted: March 16, 2015; Published: March 25, 2015.
DOI: 10.15376/biores.10.2.2850-2858