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Full Length Research Paper

A comparison of soda and soda-AQ pulps from cotton stalks

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In this study, cotton stalks (*Gossypium hirsutum* L.) were cooked using soda and soda-anthraquinone (AQ) process. Nine soda cooks were conducted by changing cooking conditions including active alkali charge and pulping time. Soda-AQ cooks were obtained by adding 0.075, 0.10, 0.15, 0.2% AQ (based on o.d stalks) to optimum soda pulping. Adding AQ to soda pulps led to the increase in regarding to pulp yield and kappa numbers. On the other hand, soda-AQ pulps made from cotton stalks showed higher mechanical properties than soda pulps. The results indicated a major increase in pulp brightness when soda pulping was modified with %0.15 AQ. Also, the results showed that better pulp and paper can be produced from cotton stalks by soda-AQ process compared to the soda process.

Key words: Alkaline pulping, anthraquinone, cotton stalks, delignification, mechanical properties, yield.

INTRODUCTION

Fiber crops have begun to receive considerable attention in the last 10-15 years in forest industry to alleviate the shortage in wood raw material mostly because they are renewable resources. The use of agro-fiber wastes in paper production is beneficial in terms of environmental and socio-economic aspects.

Using non-wood raw materials for pulp and paper industry has a long history. The production of this type of pulp has increased more rapidly and today, several non-wood fiber resources are commercially utilized to manufacture chemical pulp and paper products, by a factor of two in Latin America and three in Africa and Middle East (Casey, 1990).

Several studies examined the viability of substituting wood based materials with crops residues from annual plants; such as rye straw (Usta, 1985), cereal straw (Wong, 1995), wheat straw (Moore, 1996), okra stalks (Atik, 2002) and licorice (Copur et al., 2007) to produce pulp and paper.

One of the agricultural residues, cotton stalks are available in large quantities in several parts of the world and produced by former Egypt which have no forests; cotton stalks are one of the agricultural residues available for pulping and papermaking (Ali et al., 2001). On the other hand, Turkey is the 7th cotton fiber producer in the world with an annual production of 655,000 MT which is 3.3% of total world production. Estimated cotton stalks remaining in the field is around 980,000 MT (Kirci et al., 1997).

But, there are several problems which have hindered commercial utilization of cotton stalks, for pulp and paper industry refers to the high cost of collection, transporting of the raw materials, storing of the residue materials and debarking which is made difficult due to the fact that cotton stalks are thinly branched, bushy plants. Some of these problems can be solved by small scale mills close to rural areas.

The objective of this study was to investigate the utilization of whole, undebarked cotton stalks for preparation of chemical pulps by soda and soda-anthraquinone (soda-AQ) methods and to evaluate some mechanical properties of the resulting pulps.

MATERIALS AND METHODS

The cotton stalks (*Gossypium hirsutum* L.) used in this work were obtained from the Adana and Kahramanmaras regions in Turkey. Stalks were cleaned from leaves, roots, branches, calyxes and soil. After that the cotton stalks were dried in the laboratory atmosphere,

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Table 1. Data on pulp characteristics.

Pulp No	A.A (%)	Max. Temp. (ºC)	Time to Max. Temp (min)	Time at Max. Temp. (min)	AQ (%)	Kappa mL/g	Viscosity (cm^3/g)	% ISO Brightness	% of o.d. wood yield	
1	15	160	30	30	-	97.52	832	17.6	33.06	13.39
2	15	160	30	60	-	87.31	821	16.8	36.25	7.05
3	15	160	30	90	-	78.96	815	17.0	34.83	5.10
4	18	160	30	30	-	77.02	828	17.0	29.39	12,6
5	18	160	30	60	-	74.33	806	17.1	35.80	5.14
6	18	160	30	90	-	56.49	794	18.0	30.38	2.76
7	20	160	30	30	-	68.98	805	18.8	31.84	9.10
8	20	160	30	60	-	57.44	765	21.3	28.47	4.30
9	20	160	30	90	-	44.39	737	21.3	28.57	1.86
10	18	160	30	60	0.075	67.28	818	21.5	36.86	1.27
11	18	160	30	60	0.1	66.90	832	22.1	37.87	1.07
12	18	160	30	60	0.15	59.63	872	22.5	37.98	1.01
13	18	160	30	60	0.2	55.00	879	21.9	37.94	0.94

specimens were chopped to the length of 3-4 cm, and prepared according to Tappi T 257 om-85 for chemical analyses. Holocellulose and α-cellulose contents were determined according to the chloride dioxide (Wise and Karl, 1962) and Tappi T 203 om-71 methods, respectively. The following tests were performed to determine the lignin (Tappi T 222 om-98) and ash (Tappi T 211 om-93) contents. The extractives were also determined by alcohol-benzene (Tappi T 204 cm-97), cold and hot-water (Tappi T 207 om-93) and 1% NaOH (Tappi T 212 om-98) methods.

Pulping trials were made in an electrically heated rotary laboratory type 15 liters digester. The experimental procedures were divided into two main categories. Firstly, to determine optimum soda pulping conditions of cotton stalks 9 cooking trials were made. In these trials, alkali charge (AA) and pulping time at maximum temperature were varied as indicated in Table 1. Soda-AQ cooks were made by adding 0.075%, 0.1, 0.15, 0.2 AQ (based on o.d stalks) to optimum soda pulping (Table 1). For each experiment, 600 g oven-dry cotton stalks were cooked in digester. The liquor-to-straw ratio (L/kg) was 5:1, maximum temperature was 160ºC and time to maximum temperature was 30 min for all cooking.

The produced pulps were disintegrated and washed with hot tap water and then screened using a laboratory flat screen with a slot width of 0.15 mm. The yield contents of the pulps and rejects were determined according to Tappi T 210 by gravimetric measurements in the laboratory environment. The screened yield was determined through duplicate analyses. Adding the yield of rejects to the screened yield gave total pulp yield. The kappa number (Tappi T 236) and viscosity (SCAN cm 15:88) of the pulp samples were the averages of duplicate analyses.

Refining was made on a PFI mill, in accordance with Tappi T 248. Pulp samples were collected at three predetermined intervals of 0, 0.5 and 1 min in PFI mill. Freeness was measured using a Schopper Riegler device according to ISO Standard 5267-1 and laboratory hand sheets for physical tests were produced in a Rapid Köthen machine in accordance with relevant ISO standard 5269-2 method. Tensile properties, tearing resistance (Elmendorf method) and burst strength of hand sheets were measured according to ISO standards of 1924, 1974 and 2758, respectively. The brightness and opacity of the pulps were also determined according to appropriate ISO 2469 and 2471 standard methods, respectively.

RESULTS AND DISCUSSION

Chemical composition of raw material

The chemical composition of cotton stalks are given in Table 2. In this table, results of chemical compositions of other nonwood plants determined in previous studies were also illustrated (Ozturk, 1995; Akgul, 1997; Eroglu et al., 1992; Tank et al., 1985; Bostanci, 1980; Dogan, 1994). Although the holocellulose content value of cotton stalks is close to other non wood fiber resources, cotton stalks contain high lignin and α-cellulose content. Additionally, the cotton stalks contain significantly less ash content than other non wood fiber resources. In terms of extractive contents, the cotton stalks comprise nearly low amount of extractives than others.

Screen yield, rejects yield, kappa number, and viscosity

Screened yield, rejects, kappa number and SCAN viscosity values of 13 experiments were given in Table 1. When the time fixed at 30 min, the active alkali ratio is increased from 15 to 18%; the screen yield decreases in 3.67%. If alkali ratio is increased from 18 to 20% the screen yield increases in 2.45%. In 60 min, if active alkali ratio is increased from 15 to 18% the screen yield decreases in 0.45%. If alkali ratio is increased from 18 to 20% the screen yield decreases in 7.33%.

When the time fixed at 90 min, the active ratio is

Table 2. Chemical compositions of cotton stalks with different agricultural residues.

Raw Materials	Chemical components						Solubility				Literature
	Holocellulose (%)	Cellulose (%)	α-cellulose (%)	Lignin (%)	Pentosans (%)	Ash (%)	Alcohol-benzene (%)	%1 NaOH solubility	Hot water solubility (%)	Cold water solubility (%)	
Cotton stalks	72.2	-	41.6	19.3	-	2.40	6.10	42.9	17.8	16.7	Determined
Cotton stalks	76.6	46.5	38.9	19.5	-	2.30	6.50	21.6	11.1	10.3	14
Wheat straw	74.6	48.5	41.1	15.9	-	5.10	5.80	43.6	12.0	7.89	15
Rye straw	74.1	51.5	44.4	15.4	-	3.20	9.20	39.2	13.0	10.2	16
Corn stalk	64.8	45.6	35.6	17.4	-	7.50	9.50	47.1	14.8	-	16
Tobacco stalk	67.6	46.5	37.5	19.5	-	7.30	6.50	42.9	19.1	15.8	17
Sunflower stalk	74.9	47.6	37.5	18.2	-	8.20	7.00	29.8	16.5	15.5	18
Kenaf	81.2	54.4	37.4	14.5	-	4.10	5.00	34.9	12.8	11.7	19

increased from 15 to 18%; the screen yield decreases in 4.45% and decreases in kappa numbers in 20%. Kappa number decreases depending on Alkali ratio delignification. The lowest kappa number was obtained at 20% AA after 44.39% screen yield and 90 min cooking. If alkali ratio is increased from 18 to 20% the screen yield decreases in 1.81% (Figure 1). Regardless of the cooking time, when the active alkali ratio increases, continuous screen reject values decrease. For cotton stalks cooking conditions which affect yield and kappa number are examined for optimum conditions.

The optimum cooking conditions are determined for 18% active alkali at 60 min. On the other hand, regardless of the cooking time if the activate alkali ratio increases, screen residue decreases continuously. An increasing at active alkali and cooking times cause in cellulose's DP decrease in pulping.

The effect of adding AQ into optimum soda pulping showed that the addition of 0.075%, 0.1, 0.15, 0.2 AQ led to a yield increase of 2.96, 5.78, 6.09 and 5.98% and kappa number decrease of 10.5, 11.1, 24.7 and 35.1%, respectively (Figure 2). The differences in pulp yield can be attributed to the retention of hemicelluloses and lignin, once the absolute proportion of cellulose remains constant with pulping (Atik, 2002; Copur et al., 2007). A higher pulp yield with soda-AQ pulps, compared to the soda method, was due to the higher hemicellulose retention (Atik, 2002; Copur et al., 2007; Lowendahl and Samuelson, 1978; Nelson and Irvine, 1992). This shows that addition of AQ in soda pulp can be used to reach a certain kappa number in a shorter cooking time resulting in advantages of shorter process and reduced energy consumption. The yield observed for cotton stalk was lower and kappa number was higher compared to the other non wood resources, such as reed (Kuang, 1986), hemp (Dharm et al., 2005) and okra stalks (Atik, 2002). In terms of pulping rejects, adding AQ in pulping lowered the amount of screening rejects compared to the soda pulp (Table 1). It was observed that a significant reduction in pulp rejects was obtained when 0.2% AQ was used in soda pulping. On the other hand addition of AQ in soda pulping resulted in higher viscosity compared to the soda pulp. Adding AQ into optimum soda pulping, the results showed that the addition of 0.075%, 0.1, 0.15, 0.2 AQ led to a viscosity increase of 1.49, 3.23, 8.19 and 9.06%, respectively.

Pulp strength properties

After cotton stalks with soda, soda-AQ methods are used to make pulps. The reason is that in soda method remaining cooking time and/or active alkali amount makes pulp beating easy. Furthermore remaining cooking time and/or active alkali ratios effect of the paper resistance were given in Table 3.

Adding AQ had some effect on the ease of pulp refining (Table 3). A faster rate of refining associated with AQ could be attributed to differences in the relative amounts of holocellulose and lignin contents and higher hemicellulose content enhancing swelling and flexibility.

Refining had a positive effect on tensile and burst strength for all pulps, and when pulps were refined to 0.5 and 1 (min) in PFI mill, the tensile index of the pulps increased as shown in Table 3. Higher tensile and burst indices were observed when pulps were produced using higher active alkali for soda pulping. Higher tensile and burst indices brought about by refining could be explained with the refining effects of external and internal fibrillations. Also, higher active alkali could be the reason of the pulps with lowest lignin and highest hemicellulose contents. When the unrefined (0 min) optimum soda and soda-AQ pulps were compared, the obtained data indicated 66.3, 72.9, 60.2 and 33.1% increase in tensile

Table 3. Pulp PFI data.

Pulp	PFI (min)	SR (mL)	Tensile index (Nm/g)	Tear index (mNm^2/g)	Burst index ($kPam^2/g$)
Soda-%15AA (30 min)	0	39	16.3	2.54	0.75
	0.5	54	19.3	4.51	1.07
	1	59	23.1	5.94	1.47
Soda-%15AA (60 min)	0	35	16.7	6.32	0.99
	0.5	46	17.2	5.54	1.01
	1	58	18.9	6.17	1.22
Soda-%15AA (90 min)	0	31	14.9	5.79	0.80
	0.5	47	18.8	5.52	1.07
	1	59	20.3	5.43	1.30
Soda-%18AA (30 min)	0	30	18.9	5.67	0.89
	0.5	48	22.1	6.36	1.22
	1	58	22.6	6.41	1.25
Soda-%18AA (60 min)	0	30	16.6	3.84	0.77
	0.5	54	24.4	4.88	1.35
	1	59	24.6	5.76	1.38
Soda-%18AA (90 min)	0	34	12.3	3.52	0.54
	0.5	60	20.8	5.33	1.32
	1	78	28.0	4.08	1.41
Soda-%20AA (30 min)	0	40	22.7	5.86	1.20
	0.5	49	25.5	6.37	1.38
	1	54	27.3	6.78	1.52
Soda-%20AA (60 min)	0	45	20.3	4.45	1.03
	0.5	65	28.9	4.70	1.51
	1	73	29.1	6.34	1.67
Soda-%20AA (90 min)	0	38	19.4	3.91	1.06
	0.5	59	25.6	4.59	1.39
	1	66	27.7	5.48	2.09
Soda-%0.075 AQ	0	45	27.6	5.91	1.31
	0.5	64	31.1	5.62	1.67
	1	69	35.6	6.61	1.94
Soda-%0.1 AQ	0	49	28.7	6.31	1.49
	0.5	67	33.7	5.65	1.77
	1	74	35.9	6.12	1.95
Soda-%0.15 AQ	0	48	26.6	4.89	1.37
	0.5	61	28.5	5.54	1.53
	1	70	28.6	6.34	1.76
Soda-%0.2 AQ	0	40	22.1	4.23	0.95
	0.5	62	27.5	4.54	1.46
	1	67	28.5	6.63	1.68

indices (Figure 3) and 70.1, 93.4, 77.9 and 23.4% increase in burst indices (Figure 4) in soda-AQ pulps, respectively. The soda-AQ pulp produced using with 0.1% AQ had the highest tensile and burst indices.

On the other hand, adding AQ in soda pulping increased the tear index of the pulps (0 min). This indi-cates that adding AQ into soda pulping resulted in strong fibers compared to the soda pulp. Adding 0.075, 0.1, 0.15, 0.2% AQ (based on o.d stalks) into soda pulp resulted in an increase of 53.9, 64.3, 27.3 and 10.3% in tear indices

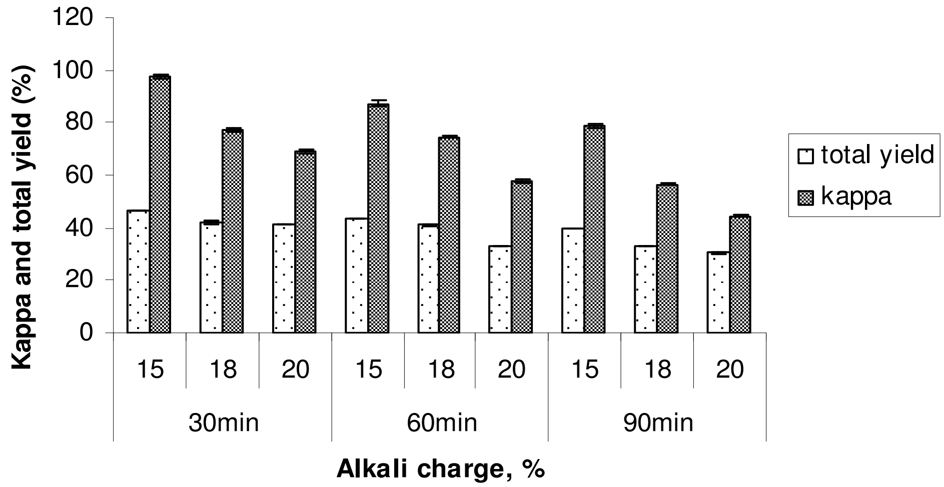

Figure 1. Total yield and kappa with soda pulping methods.

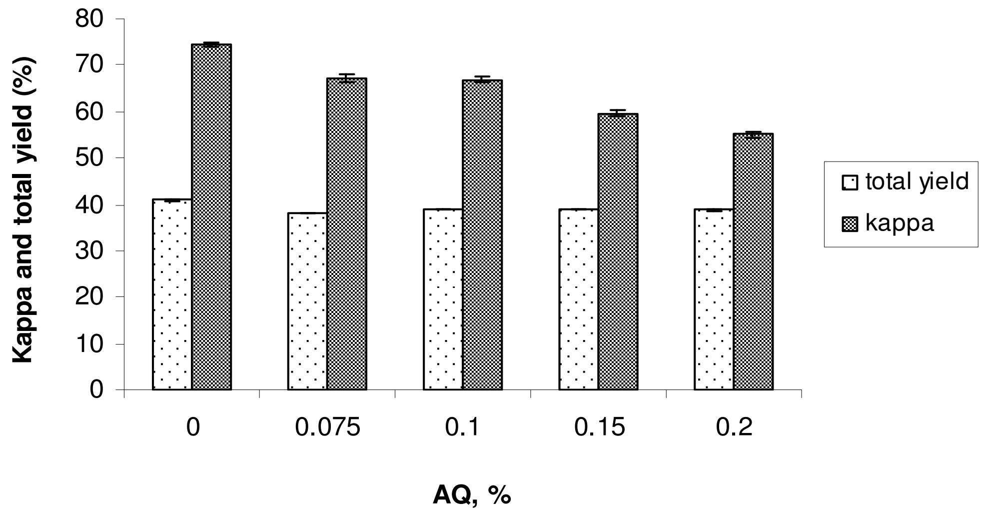

Figure 2. Total yield and kappa with soda-AQ pulping methods.

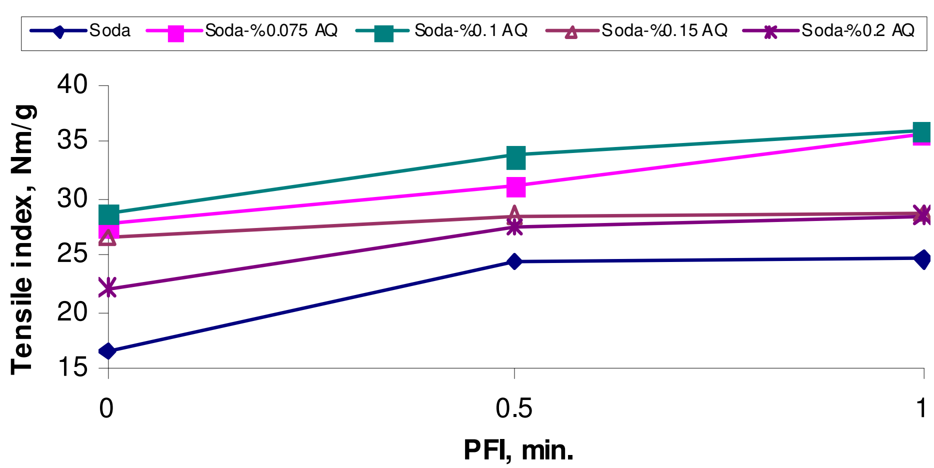

Figure 3. Effect of AQ on tensile strength.

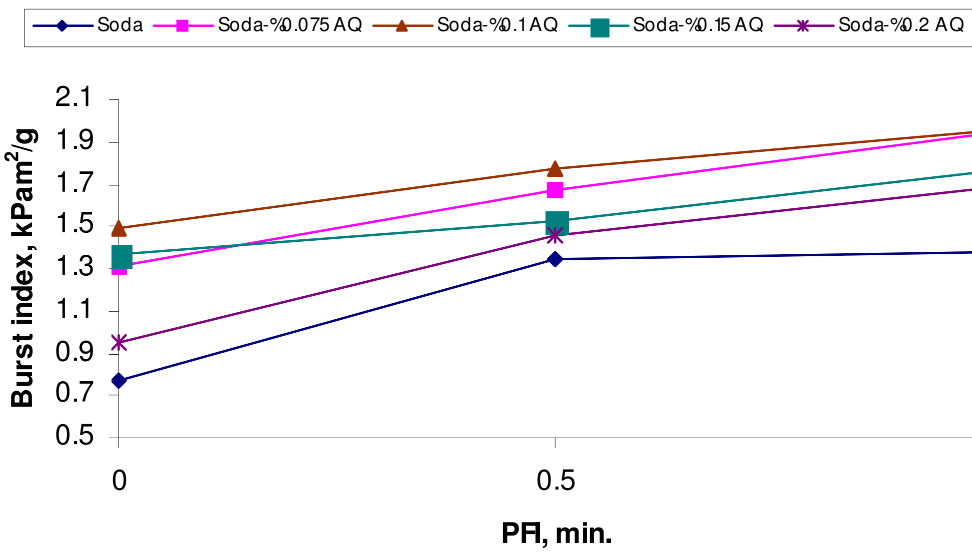

Figure 4. Effect of AQ on burst strength.

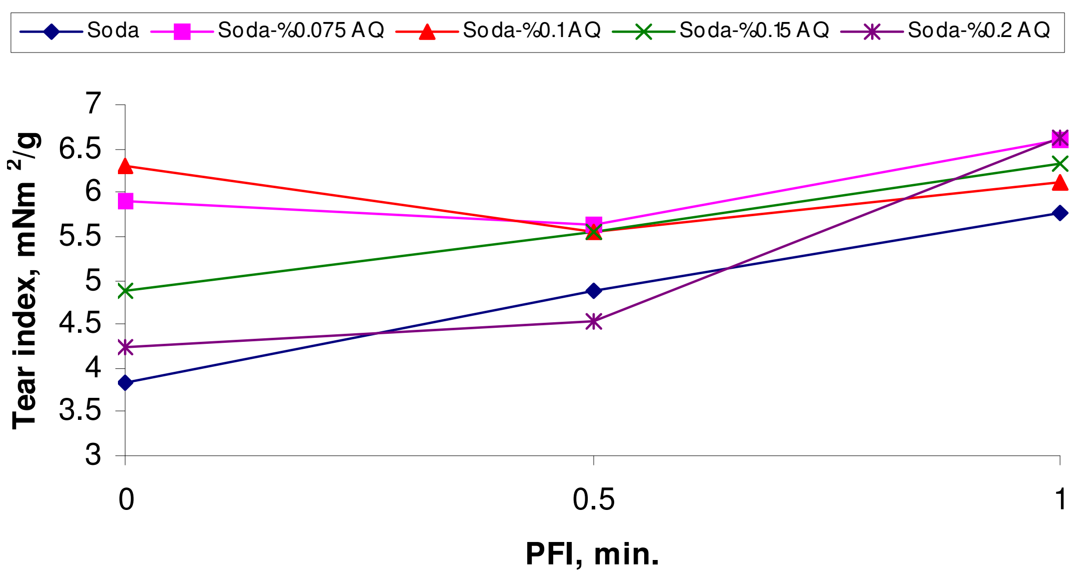

Figure 5. Effect of AQ on tear strength.

(0 min), respectively (Figure 5).

Optical properties

The Elrepho brightness of unbleached hand sheets of optimum soda and soda-AQ pulps was measured and given in Figure 6. Hand sheets from soda-AQ pulps gave 25.9, 29.7, 32.0 and 28.4% higher brightness compared to the soda pulp, respectively. The increase in pulp brightness with AQ pulp could be explained by lower lignin content in AQ pulps. The soda-AQ pulp produced using 0.15% AQ gave highest brightness value.

Conclusions

The effects of AQ on soda method with using cotton stalks as a raw material were studied. Pulps produced from cotton stalks showed that cotton stalk fibers could be an alternative raw material for pulp and paper industry. A significant reduction in pulp rejects was observed with addition of AQ in soda method. Adding AQ into the soda pulp led to an increase in pulp yield and reduction in kappa number. A major increase on pulp brightness was observed when 0.15 % AQ was added into the soda pulp. Finally, addition of AQ resulted in easier refine of the pulps and the refined soda-AQ pulps displayed the

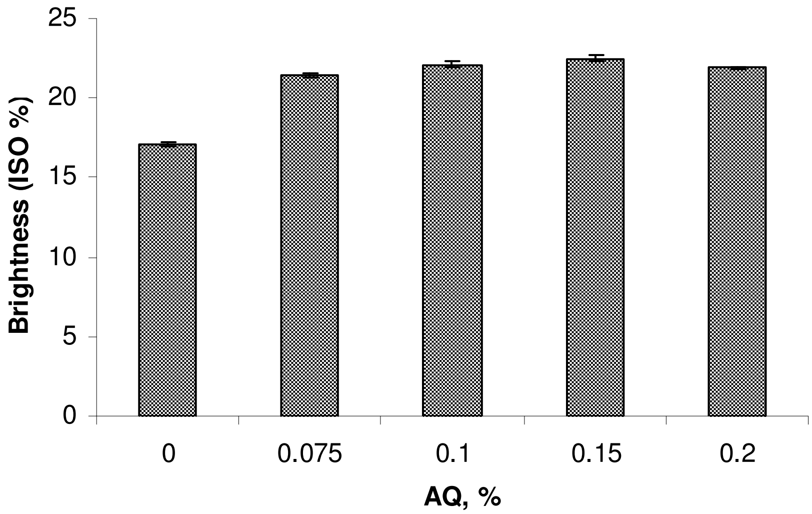

Figure 6. Effect of AQ on brightness.

highest tensile index.

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