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Full Length Research Paper

Chemical, morphological and anatomical properties and evaluation of cotton stalks (*Gossypium hirsutum* l.) in pulp industry

Ahmet Tutus[1]*, Ahmet Cenk Ezici[1] and Saim Ates[2]

[1]Department of Forest Industrial Engineering, Faculty of Forestry, KSU, Kahramanmaras-Turkey.
[2]Department of Forest Industrial Engineering, Faculty of Forestry, Kastamonu University, 37100, Kastamonu-Turkey.

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In this study, chemical composition and morphological properties of cotton stalks growing in Turkey were investigated. Typical chemical properties determined were the amount of holocellulose, α-cellulose, cellulose, lignin, ash, silica, solubility of cold and hot water and 1% soda and alcohol–benzene. The holocellulose, α-cellulose, cellulose, lignin contents and solubility of cold and hot water and 1% soda and alcohol–benzene in cotton stalks were determined similar to those of some common non-wood and hardwood raw materials. Also, sodium borohydride ($NaBH_4$) modified kraft pulping was performed by using cotton stalks and the effects of $NaBH_4$ addition on the total pulp yield and chemical properties of pulps were investigated. In order to determine the optimum cooking parameters, 30 different laboratory experiments were performed. It was noted that the increasing level of $NaBH_4$ improved the screened pulp yield, pulp viscosity and degree of polymerization, reduced the kappa number. Consequently, total pulp yield was increased (15.66%) by $NaBH_4$ addition. Pulp viscosity and degree of polymerization were found to be higher than those of kraft one by about 1.60 and 1.73%, respectively. However, kappa number of $NaBH_4$ added to kraft pulp is lower (15.86%) than those of kraft one.

Key words: Cotton stalks, chemical composition, fiber properties, $NaBH_4$, pulp.

INTRODUCTION

The worldwide consumption of paper and board products increases continuously due to several reasons, e.g., population growth, better literacy, development of communication, and industrialization in developing countries. Although, wood is the major source of fiber supply for papermaking, the lack of forest resource in many areas of the world imposes the use non-woods fibers as important alternative fibrous sources for papermaking industries. In 2005, global production of virgin pulp for paper and paperboard was 187.6 million metric tons, from which 17.4 million metric tons made from non-wood fibers, accounted for 9.27% (Bowyer et al., 2007).

The use of agro-fiber wastes in paper production is beneficial in terms of environmental and socio-economic aspects. The production of non-wood plants pulps has increased more rapidly and today, several non-wood fiber resources are commercially utilized to manufacture chemical pulp and paper products in China, India, Latin America, Africa, Middle East and Turkey. At present, some agricultural residues such as wheat and rice straws, sorghum stalks and some annuals plants such as hemp and jute are used as raw materials for pulp and paper production (Rousu et al., 2002; Ashori, 2006). Moreover, numerous studies have been performed to introduce new lignocellulosic fiber resources for pulp and paper industries (Sarwar et al., 2006; Shatalov and Pereira, 2006; Tran, 2006; Wanrosli, 2007).

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 997.000 tons which is

*Corresponding author: E-mail: atutus@ksu.edu.tr. Tel: +90-5374517621.

about 4% of total world production. Estimated cotton stalks remaining in the field is around 4 million tons (TUIK, 2008).

The paper industry has recently been interested in taking advantage of an environmentally friendly process of utilizing boron compounds in pulping processes. Pulping yield depends on some factors, such as the modification of the chemical reactions and the composition of the raw material during pulping. To counteract the yield losses, several studies and trials were done with the addition of polysulfide, anthraquinone and borohydrate to modified cooks. Studies related to pulp yield increasing focused on modifications to the kraft process with multiple liquor injection and impregnation techniques in order to minimize the alkali degradation of cellulose and hemicelluloses. When the chemistry of the kraft process was better understood and the mechanisms for degradation and loss of polysaccharides in alkali solutions were realized, the investigation focused on ways to stabilize the cellulose and hemicelluloses against alkaline attack, leading to higher pulp yields. Stabilization of the polysaccharides against alkali degradation occurs with conversion of carbonyl groups with a reducing or an oxidizing agent, and these reactions prevent a further peeling reaction (Courchene, 1998; Tutuş and Eroğlu, 2003). Addition of pulping additives (anthraquinone, polysulfide or sodium borohydride) to cooking liquor increases pulp yield through greater retention of hemicelluloses (Tutuş and Eroğlu, 2004).

$NaBH_4$ causes reduction of carbonyl group located on the end group of cellulose to hydroxyl group during the cooking and stops the probable peeling reaction because it is a powerful reducing agent. Thus, a decrease in yield during cooking can be prevented. This reaction can occur in both cellulose and hemicelluloses (Courchene, 1998; Hafızoğlu and Deniz, 2007). Peeling reaction initiated in carbonyl groups in the end units is prevented by the conversion of carbonyls to hydroxyls by borohydride. The major effect of borohydride is to prevent the acceleration of glucomannan removal that otherwise occurs at 100˚C (Tutuş and Eroğlu, 2004).

The aim of this study was to determine the chemical and morphological properties and kraft-$NaBH_4$ pulping properties of cotton stalks were investigated to evaluate the potential utilization of cotton stalks fibers in pulp production. We also focused on the characteristics; total pulp yield, kappa number, viscosity and degree of polymerization of the pulp samples obtained from kraft-$NaBH_4$ method.

MATERIALS AND METHODS

Materials

The cotton stalks (*Gossypium hirsutum* L.) used in this work was obtained from the Kahramanmaras regions in Turkey. Stalks were cleaned from leaves, roots, calyxes and soil to determine chemical, morphological properties and evaluation in pulp production.

Methods

Chemical properties of raw materials

The cotton stalks were chipped to the length of 3 - 4 cm, and prepared according to Tappi T 257 om-85 for chemical analyses. Cotton stalks were analyzed for holocellulose, α-cellulose, lignin, pentozan, ash, silica and solubility rations, (cold and hot water, 1% soda and alcohol-benzene) in accordance with the applicable TAPPI standards: T 203 os-71, T 222 om-88, T 222 om-98, T 19 m-50, T 211 om-85, T 244 om-88 and T 207 om-88, respectively (Anonymous, 1992). The cellulose content of cotton stalks was determined according to Kurscher-Hoffer nitric acid method (Browning, 1967). All measurements were repeated three times.

Morphological and anatomical properties of raw materials

To measure fiber morphologic properties of the specimens (0.5-mm thickness and 2 cm long is in parallel to fiber), chloride delignification method was applied. In this method, specimens were immersed into chloride solution until they were defibred and later, morphologic properties were measured. A drop of macerated sample was taken on a slide and fiber length, fiber width, lumen width and cell wall thickness were measured by using vizopan microscope. For measuring fiber length, fiber width, lumen width and cell wall thickness and diameter, 200 fibers were measured from the slides and average reading was taken.

For anatomical properties, cotton stalk stem of about 1 cm as autoclaved followed by immediate storage in a mixture of equal volume of glycerin, ethyl alcohol and water till sectioning with sliding microtome. Then, transversal section slide was prepared and investigated on a microscope.

Pulping and chemical properties of pulp samples

3 controls and 27 $NaBH_4$ added, total of 30 cooking experiments were performed for kraft-$NaBH_4$ process to determine optimum pulping conditions. Active alkali and $NaBH_4$ rations and cooking time were varied at three levels as 14, 16, 18 and 0.1, 0.3, 0.5% and 90, 120, 150 min, respectively. The cooking trials were carried out in 15 L, electrically heated laboratory type, cylindrical type rotary digester and governed with digital temperature control system. At the end of pulping, pressure was relieved to atmospheric pressure then pulps were washed, disintegrated in a laboratory type, pulp mixer with 2 L capacity and screened on a Noram type pulp screen with 0.15 mm slotted late. Pulp yield was determined as dry matter obtained on the basis of oven dried (O.D) raw material. The reactor was loaded with 600 g (O.D) cotton stalks chips for each trial and cooked with appropriate chemicals needed as shown in Table 3.

The yield contents of the pulps and rejects were determined according to Tappi T 210 by gravimetric measurements in the laboratory condition. The screened yield was determined through duplicate analyses. Adding the yield of rejects to the screened yield gave total pulp yield. The kappa number of the pulps was determined according to Tappi T 236 85 -cm. The pulp viscosity, which is indicative of the damage of the cellulosic chain, was determined according to SCAN 15:88 cm. The degree of polymerization (DP) of fibers was calculated using the following equation, where viscosity is the SCAN 15:88 cm. All measurements were repeated two times (Anonymous, 1988). $DP^{0.905} = 0.75 \times$ viscosity.

Table 1. Chemical analyses of whole cotton stalks comparison with same annual plants and woods.

Raw materials	Holocellulose (%)	Cellulose (%)	α-cellulose (%)	Lignin (%)	Ash (%)	Silica (%)	Alcohol benzene (%)	1 % NaOH (%)	Cold water (%)	Hot water (%)	References
Cotton stalk	75.6	45.5	39.8	18.2	2.52	0.48	6.1	30.9	11.7	15.3	Determined
Wheat straw	77.0	52.1	39.8	18.1	7.04	5.43	5.52	40.8	7.7	12.4	(Tutuş and Eroğlu, 2003)
Rice straw	70.9	48.2	35.6	17.2	16.6	14.9	3.5	49.2	10.7	16.2	(Tutuş et al., 2004)
Rye straw	74.1	51.5	44.4	15.4	3.2	1.5	9.2	39.2	10.2	13.0	(Usta and Eroglu, 1988)
Kenaf	77.9	54.4	37.4	14.5	4.1	-	5.0	34.9	12.8	11.7	(Dogan, 1994)
Bamboo	70.5	53.4	43.3	24.5	1.4	-	3.9	25.1	-	6.5	(Deniz and Ates, 2002)
Coniferous	70-81	42-51	40-45	24-32	<1	-	1-8	8-14	1-4	1-6	
Deciduous	63-90	55-38	36-49	21-25	<1	-	1-7	15-22	1-5	1-8	(As et al., 2002)

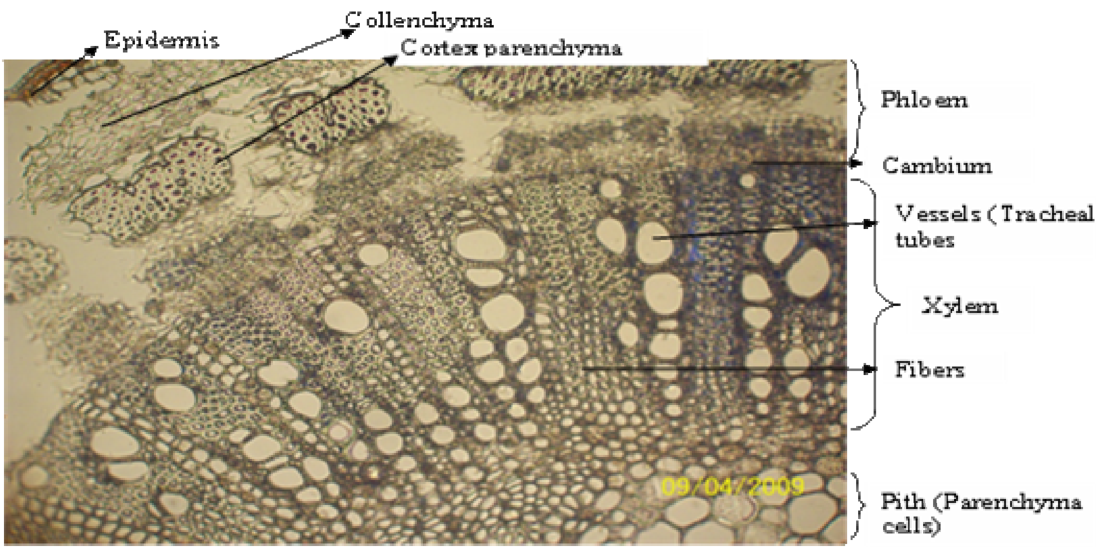

Figure 1. Transversal sections of cotton stalks stem.

RESULTS AND DISCUSSION

Chemical composition and some solubility values of the cotton stalks were determined and shown in Table 1. Table 1 show that the chemical properties of cotton stalk and their comparison with some annual plants, coniferous and deciduous wood which are the main fibrous raw materials. As shown in Table 1, the average holocellulose content of cotton stalks was found as 75.6%; most annual plant and coniferous were between 70 - 81%, α-cellulose in cotton stalks (45.5%) is higher than some annual plants, softwood and hardwoods (35 - 45%). Lignin content of cotton stalks was found as 18.2%, which is comparable with all softwoods (25 - 32%), especially with scots pine were 26.0% (Tutuş et al., 2004); it is however, substantially lower than all hardwood (21 - 25%) and 24.5% in bamboo (Deniz and Ates, 2002), 17 - 26% in same annual plants and hardwoods (As et al., 2002). Cotton stalks also showed similar solubility with some annual plants. But solubility of hardwood and softwoods lower than cotton stalks and some annual plants. As shown in Table 1 that compared with coniferous woods, all main chemical composition and some solubility values are in normal range (As et al., 2002). High NaOH solubility rate in cotton stalks compare with coniferous can be due to the presence of low molecular weight carbohydrates and other alkali soluble matters.

The anatomical structure of cotton stalks was studied on transverse sections (Figure 1). The light microscopy observation revealed the prevalence of four distinct tissue systems: vessels, fibers, parenchyma cells and collenchymas. The general structure of cross section of

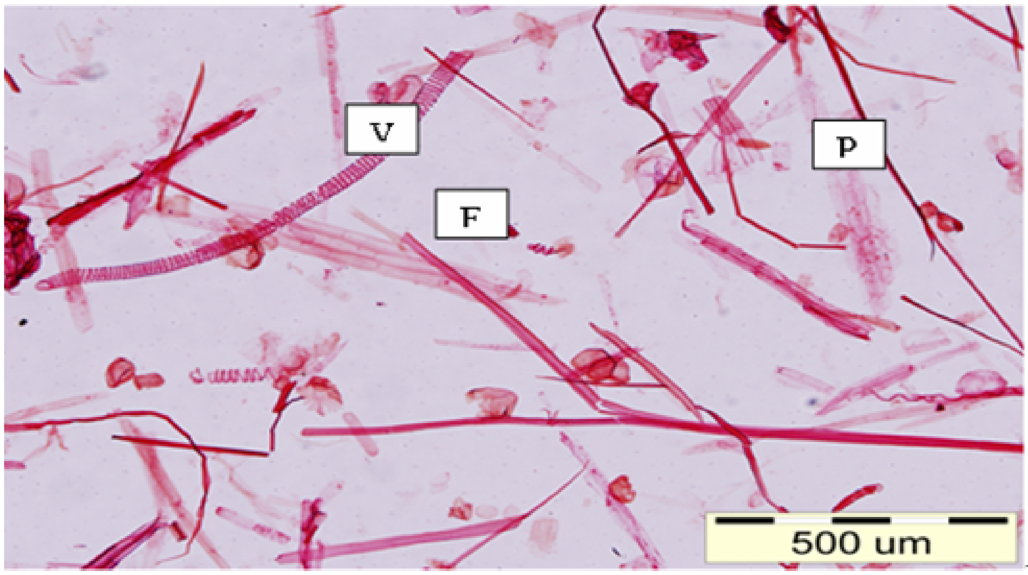

Figure 2. Macerated sample of cotton stalks stem (F: fibers, P: parenchyma cells, and V: vessels).

Table 2. The results of fiber properties of cotton stalks and comparison with common papermaking fibers.

Samples	Fiber lengths (mm)	Fiber width (µm)	Lumen width (µm)	Cell wall thickness (µm)	References
Cotton stalks	0.81	24.98	16.75	4.12	Determined
Wheat straw	0.74	13.20	4.02	4.59	(Deniz et al., 2004)
Rice straw	0.89	14.80	6.40	4.20	(Tutuş et al., 2004)
Canola stalks	1.17	23.02	12.50	5.26	(Enayati et al., 2009)
Coniferous	3-7	32-43	15-30	13 - 17	(As et al., 2002)

cotton stalks from bark to core is displayed in Figure 1.

Phloem (bark), cambium, xylem, and pith sections are shown together. Phloem occupies a narrow space and its outer part is covered by epidermis cells. Cortex parenchyma is present under the epidermis. The cambium layer is distinguishable between phloem and xylem. Xylem cells are distinguishable by their corrugated shape. The vessels (tracheal tubes) are diffused through the xylem. The pith part is large and consists of thin-walled parenchyma cells.

Macerated sample from cotton stalks was studied on vizopan microscope (Figure 2). The morphological properties of cotton stalks and their comparison with common papermaking fiber resources are summarized in Table 2. The results show that the cotton stalks contained short fibers with a mean length of 0.81 mm. The cotton stalks fibers are as long as the short fibers of non-wood plants such as rice straw. However, they are longer than wheat straw as same in hardwood fibers. The fiber diameter and lumen width of cotton stalks are similar to those of canola stalks fibers and same softwood fibers.

On the other hand, cell wall thickness of cotton stalks fibers is thicker than those of same non-wood, hard and softwood fibers. Overall morphological properties of cotton stalks fibers are satisfactory for papermaking, although, they would be classified as short fibers. Pulping conditions and experimental design for obtaining kraft-$NaBH_4$ pulps from cotton stalks was shown in Table 3. Rejects, screened yield, total pulp yield and some chemical properties of cotton stalks pulp samples obtained with kraft-$NaBH_4$ methods were shown in same Table (Table 3).

The pulp yield increasing and delignification maximizing studies are based on raw material modifications, pulping additives (anthraquinone, polysulfide, boron compounds etc.), and cooking modifications. A pulping additive, sodium borohydride, acts as a catalyst. It protects the reducing end groups from a peeling reaction and increases the screened yield of pulp (Istek and Gonteki, 2009). Cooking conditions and influence of different cooking parameters on cotton stalks pulps are shown in Table 3. In control 3 (C3) kraft pulping, the highest total yield (39.32%) and the lowest kappa number (39.11) was obtained. The benefits of $NaBH_4$ addition into kraft pulping were a significant increase in pulp yield, pulp viscosity, degree of polymerization and reduction in

Table 3. Cooking conditions and yield and some chemical properties of cotton stalks kraft-$NaBH_4$ pulps.

Cooking No.	Active alkali (%)	Cooking time (min.)	$NaBH_4$ ratio (%)	Screened yield (%)	Reject (%)	Total yield (%)	Kappa no	Viscosity (cm^3/g)	DP
C1	14	120	-	33.40	3.84	37.24	40.35	998	1499
C2	16	120	-	35.19	3.13	38.32	39.72	975	1461
C3	18	120	-	37.05	2.27	39.32	39.11	907	1349
1	14	90	0.1	38.85	2.20	41.05	39.28	917	1365
2	14	120	0.1	39.73	1.72	41.45	37.32	1003	1508
3	14	150	0.1	38.47	1.31	39.78	36.36	991	1488
4	16	90	0.1	39.70	1.77	41.47	37.25	1007	1514
5	16	120	0.1	40.22	1.55	41.77	36.23	985	1478
6	16	150	0.1	39.81	1.09	40.90	35.92	974	1460
7	18	90	0.1	39.96	1.64	41.60	36.32	950	1420
8	18	120	0.1	40.67	1.47	42.14	35.27	910	1354
9	18	150	0.1	39.79	0.60	40.39	35.07	854	1262
10	14	90	0.3	39.95	1.84	41.79	37.41	1037	1564
11	14	120	0.3	41.20	1.59	42.79	35.38	1018	1533
12	14	150	0.3	39.64	1.50	41.14	34.13	1001	1504
13	16	90	0.3	40.02	1.67	41.69	35.96	1021	1538
14	16	120	0.3	41.44	1.52	42.96	34.76	1007	1514
15	16	150	0.3	40.37	1.01	41.38	34.42	992	1489
16	18	90	0.3	40.65	1.59	42.24	35.45	1011	1521
17	18	120	0.3	42.67	1.45	44.12	34.41	965	1447
18	18	150	0.3	40.96	0.61	41.57	33.87	952	1423
19	14	90	0.5	39.97	1.75	41.72	35.92	1051	1588
20	14	120	0.5	41.80	1.57	43.37	34.78	1031	1554
21	14	150	0.5	40.39	1.44	41.83	33.32	1018	1533
22	16	90	0.5	41.57	1.61	43.18	34.65	1004	1509
23	16	120	0.5	42.88	1.42	44.30	33.42	1045	1578
24	16	150	0.5	41.95	0.92	42.87	31.31	1021	1538
25	18	90	0.5	40.50	1.50	42.00	35.87	994	1493
26	18	120	0.5	42.11	1.36	43.47	33.35	1010	1519
27	18	150	0.5	41.50	0.60	42.10	33.19	992	1489

Note: Sulfidity ratio: 25%, cooking temperature: 160°C and liquor/raw material ratio: 5/1 were constant in all cooking experiments, C= control.

kappa number and reject ratio.

The results indicated that the highest yield increase was observed when C2 kraft pulp was modified with 0.5% $NaBH_4$ (cooking no 23). The yield increase observed was 15.61% (relative percentage). This increase in pulping yield is expected and is explained by the stabilization of glucomannan and xylan. In addition, $NaBH_4$ addition causes a reduction of carbonyl groups located on the end group of cellulose to hydroxyl group during the cooking and stops the probable peeling reactions because $NaBH_4$ is a powerful reducing agent; therefore, prevents yield decrease in cooking (Courchene, 1998; Hafızoglu and Deniz, 2007; Istek and Ozkan, 2008; Copur and Tozluoglu, 2007). Generally, it has been observed that in Figure 3, total yield was increased and kappa no was decreased with the increasing addition level of $NaBH_4$ for kraft processes. Increase of $NaBH_4$ regularly reduced the kappa number of the pulps. Increasing $NaBH_4$ from 0 - 0.5% for C2 cooking reduced the kappa number from 39.72 - 33.42 (cooking no 23). This result can be ascribed to the acceleration of delignification by $NaBH_4$ (Akgül et al., 2007) obtained similar results indicating that adding $NaBH_4$ during brutia pine kraft pulping decreased the kappa number from 29.6 - 27.2.

This results show that the addition of $NaBH_4$ increased pulp viscosity and degree of polymerization (Figure 3). The highest viscosity and DP was observed 1051 cm^3/g and 1588 in cooking no 19, respectively. The viscosity and degree of polymerization increase could be because the addition of $NaBH_4$ preserved of cellulose chains and resulted in higher viscosity and DP (Akgül et al., 2007).

Kraft-$NaBH_4$ pulping process better, preserved the

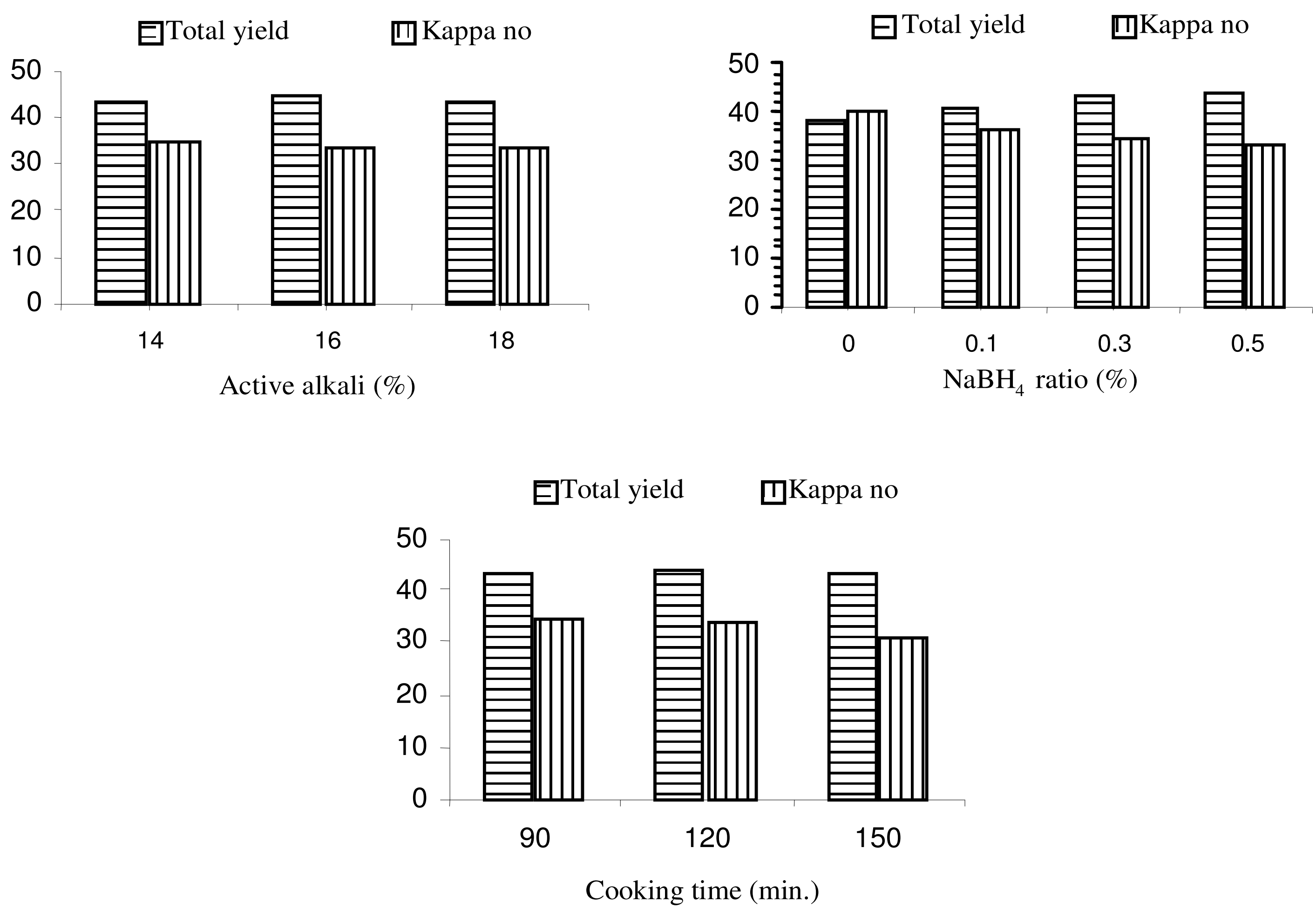

Figure 3. The effect of cooking conditions on total yield (%) and kappa no.

carbohydrate chains and improved the viscosity and DP of the pulps through the lower kappa number. As shown in Table 3 and Figure 4, when all other conditions were kept constant, total yield, pulp viscosity and DP increased until 16% active alkali ratio and then decreased. However, total yield, pulp viscosity and DP increased until 120 min cooking time and then decreased. Consequently, total pulp yield, pulp viscosity and DP slightly increased with the increasing active alkali and cooking time.

Conclusions

The following conclusions may be drawn from this investigation: The lignin, holocellulose and α-cellulose in cotton stalks are comparable to same non-wood, hardwood and softwood, cotton stalks have substantially higher hot and cold water, alkali and alcohol-benzene solubility than that of softwoods and hardwoods, which caused lower pulp yield. All main chemical composition (holocellulose, cellulose and lignin contents) are in normal range compare with comparable to same non-wood, hardwood and softwood cotton stalks have lower amount of fiber and higher content of short parenchyma cells and tracheal tubes. The pulp yields, viscosities and DP were increased and kappa number was decreased with increasing $NaBH_4$ ratio. Total pulp yield, pulp viscosity and DP slightly increased with the increasing active alkali and cooking time. Cotton stalk kraft-$NaBH_4$ pulps can be used in the production of high quality printing and writing papers as well as in newspaper and packing papers by the mixture long fibrous materials.

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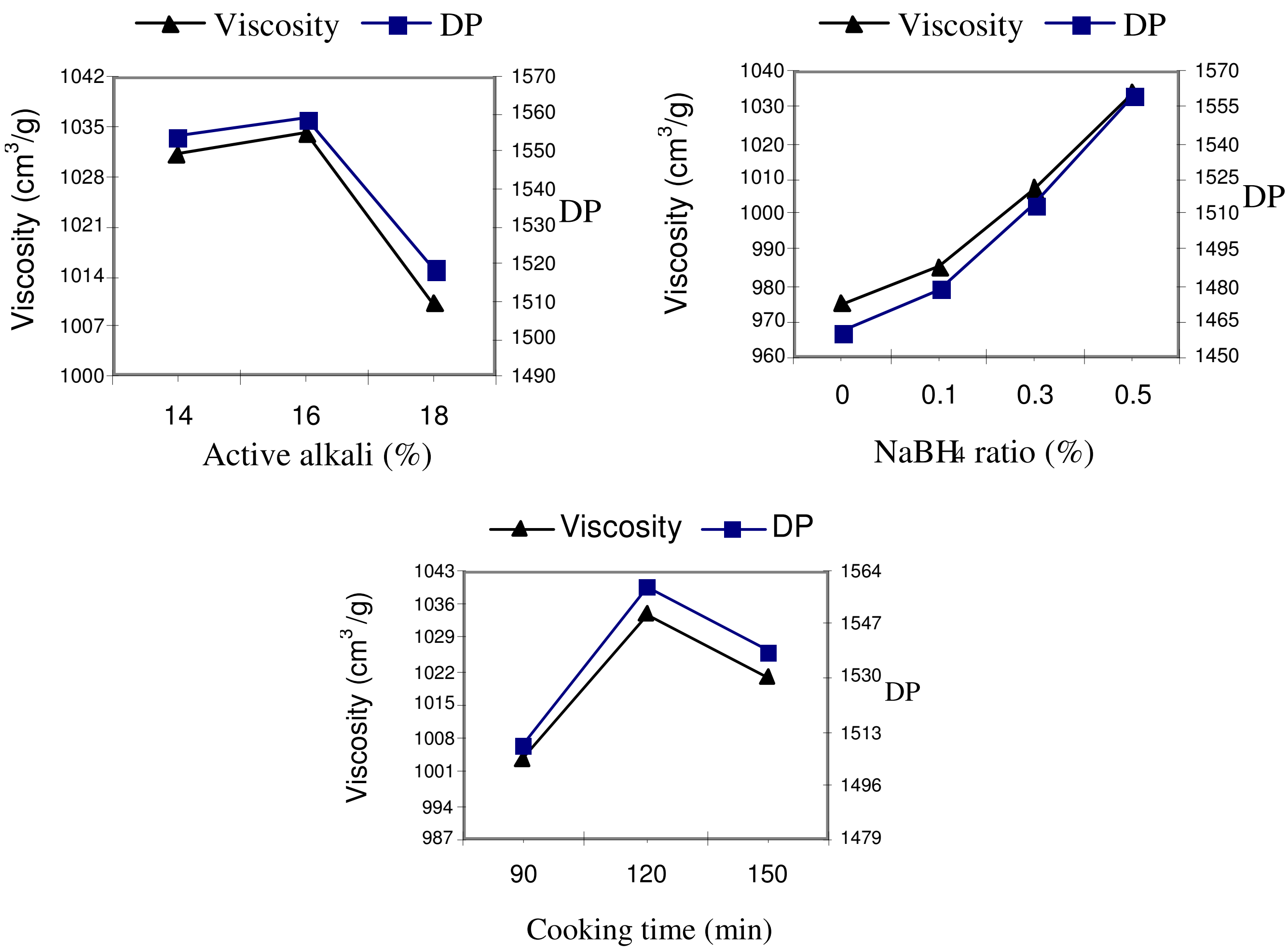

Figure 4. The effect of cooking conditions on viscosity (cm^3/g) and DP.

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