

## Modification of Egyptian Bagasse Kraft Pulping Using Yield - increasing Additives I-Effect on Chemical Properties

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**B**AGASSE is one of the potential raw materials for pulp in Egypt. So, our target is to improve the chemical properties of Kraft pulp using yield- increasing additives as anthraquinone (AQ), polysulfide (PS) and sodium borohydride ( $\text{NaBH}_4$ ). The effect of the bagasse to liquor ratio B/L ratio was also examined. The yield gains for AQ, PS,  $\text{NaBH}_4$ , AQ-PS and AQ- $\text{NaBH}_4$  were 4.73%, 6.46%, 2.99%, 12.15% and 4.05%, respectively for 1:4 B/L ratio. While the yield gains at 1:10 B/L ratio were 1.42%, 2.65%, 5.2%, 6.9% and 5.3%, respectively. Combination of AQ-PS gave synergistic effect. AQ,  $\text{NaBH}_4$  and AQ- $\text{NaBH}_4$  reduced the kappa number by 7%, 14%, 9%, 4.48%, 40.3% and 5.95% for 1:4 and 1:10 B/L ratios, respectively. More dissolution of lignin was promoted at higher B/L ratio. All additives preserved the carbohydrate yield more than the Kraft one, and showed higher delignification selectivity except for PS.

**Keywords:** Egyptian bagasse, Kraft pulp, Modified kraft pulp, AQ,  $\text{NaBH}_4$ , PS and Yield-increasing additives.

The use of agro-fiber wastes in paper production is beneficial in terms of environmental and socio-economic aspects. The production of non-wood plant pulps has increased rapidly<sup>(1)</sup> 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<sup>(2)</sup>. At present, some agricultural residues such as wheat and rice straws, sorghum stalks and some annual plants such as hemp and jute are used as raw materials for pulp and paper production<sup>(1,2)</sup>. Moreover, numerous studies have been performed to introduce new lignocellulosic fiber resources such as cotton stalks and bagasse for pulp and paper industries<sup>(3-5)</sup>.

Pulping yield depends on some factors such as the modification of the chemical reactions and the composition of the raw material. Thus the paper industry has recently been interested in taking advantage of an environmentally friendly process involving boron compounds in pulping processes<sup>(6)</sup>. To counteract the yield losses; due to polysaccharides degradation and lignin dissolution; several studies and trials were done to investigate the effect of

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addition of polysulfide, anthraquinone and borohydrate on kraft cooking. Further studies<sup>(6)</sup> which aimed at increasing the yield have largely focused on modifying the Kraft process with multiple liquor injection and impregnation techniques in order to minimize the alkali degradation of cellulose and hemicelluloses. With better understanding of the chemistry of the kraft process as well as the mechanisms for degradation and loss of polysaccharides in alkali solutions, attention is now mainly focused on developing ways of stabilizing the cellulose and hemicelluloses against alkaline attack thereby, 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<sup>(6)</sup>. It has been observed that addition of end group stabilizers (such as AQ and NaBH<sub>4</sub>) decreases significantly the peeling-to-stopping rate constants ratio<sup>(7)</sup>. Furthermore, addition of pulping additives (such as anthraquinone, polysulfide or sodium borohydride) to cooking liquor increases pulp yield through greater retention of hemicelluloses<sup>(8)</sup>. NaBH<sub>4</sub> particularly is an effective chemical for selective delignification<sup>(9)</sup>. It causes reduction of carbonyl group located on the end group of cellulose to hydroxyl group during the cooking and thus stops the probable peeling reaction. Which can occur in both cellulose and hemicelluloses<sup>(6)</sup>. The major effect of borohydride is to prevent the acceleration of glucomannan removal that otherwise occurs at 100°C<sup>(8)</sup>. Polysulfide pulping results in substantial yield gain which is brought about by oxidation of aldehyde end groups in carbohydrate chains to aldonic acids during cleavage, thereby stabilizing aldehyde end groups against further peeling<sup>(10,11)</sup>. The yield increase in PS pulping is proportional to the PS added to the cook<sup>(12)</sup>.

The aim of this study is to examine the effect of anthraquinone, polysulfide and sodium borohydride as well as the effect of bagasse to liquor ratio on the chemical properties of Egyptian bagasse Kraft pulping

### Materials and Methods

#### *Materials*

The depithed bagasse used in this study was received from Masr-Edfu pulp mill at Aswan-Egypt.

#### *Chemicals*

Sodium hydroxide (NaOH) and anthraquinone (AQ) were obtained from EL-Naser Pharmaceutical Chemical Company, Egypt. Sodium sulfide (Na<sub>2</sub>S) and Sodium borohydride (NaBH<sub>4</sub>) were obtained from Oxford Laboratory Reagent (India). Sodium chlorite and sulfur were obtained from AL-gomhorya Pharmaceutical Chemical Company, Egypt.

#### *pulping*

Kraft pulping is the dominant chemical pulping process in the world, it uses NaOH and Na<sub>2</sub>S. For each experiment, 10 g oven dry bagasse was cooked in 250 ml stainless steel autoclave at 170 °C for 150 min after reaching the

temperature in 60 min. The cooks were made at 20% alkali charges and 20% sulfidity. The bagasse-to-liquor ratio for all cooks (g/ml) were 1:4 and 1:10. The autoclaves after being purged with nitrogen, were sealed and heated in an oil bath in which the temperature was raised from the ambient to the desired temperature. Kraft cooks were prepared using NaOH + Na<sub>2</sub>S (K) or by adding 0.1% AQ (based on o.d. bagasse), 1%, 2% and 3% NaBH<sub>4</sub> (based on o.d. bagasse), 0.1% AQ + 3% NaBH<sub>4</sub>, 3% PS (based on o.d. bagasse), and 0.1% AQ + 3% PS. All of these additives were added to the autoclaves along with the bagasse. At the end of the pulping, pressure was reduced to atmospheric pressure and then the produced pulp was disintegrated, washed with tap water till free of black liquor and then air dried. Every experiment was repeated and mixed well to get enough pulp for chemical analysis. The  $\alpha$ -cellulose, yield, ash, and lignin contents of pulp samples were determined according to Tappi T203, T210, T211 and T222, respectively. While, holocellulose content was determined according to wise *et al.* <sup>(13)</sup> and kappa number was calculated from the equation:

$$\text{Klason lignin} = \text{kappa number} \times 0.15 \quad \text{Tappi-T236}$$

The values of chemical analysis were duplicated to ensure good results.

## Results and Discussion

**TABLE 1. Chemical constitution of bagasse.**

Holocellulose %	$\alpha$ -cellulose %	Lignin %	Ash content %
78	59	20	1.75

Bagasse consists of holocellulose ( $\alpha$ -cellulose + hemicellulose), lignin and ash .

### *Pulping yield*

One of the disadvantages of Kraft pulping is the low pulp yield because Kraft chemicals are not selective for lignin and large amount of hemicelluloses are dissolved in the strong alkaline environment especially in the initial phase of cooking. Because pulp yield is highly desirable so, our target, as well as many researchers <sup>(14-17)</sup>, is focusing on increasing yield by protecting the carbohydrates (mostly hemicelluloses) from degradation and improving fiber strength by modification of the pulping process using yield – increasing additives such as anthraquinone (AQ), sodium borohydride (NaBH<sub>4</sub>) and polysulfide (PS). These additives stabilize the reducing ends of carbohydrates against peeling either by oxidation to carboxylic acid end group as in case of AQ and PS <sup>(18)</sup> or by reduction of carbonyl groups to hydroxyl groups using NaBH<sub>4</sub> <sup>(15,19)</sup>. Chemical analysis data are compiled in Table 2 while the effect of additives on percentage yield is graphically represented in Fig. 1&2 . Addition of AQ to the cooking liquor for B/L ratios of 1:4 and 1:10 resulted in increased yield by 4.73% and 1.42%, respectively due to increasing the cellulose yield

and, to a lower extent, glucomannan<sup>(15)</sup> because AQ stabilizes the carbohydrates from further degradation. Different authors<sup>(15,16,20-26)</sup> proved that AQ increases the yield and accelerates the delignification rate due to its dual effect. Similarly, using 3% polysulfide (PS) in Kraft cooking increased the pulp yield by 6.46% and 2.65% for B/L ratios 1:4 and 1:10, respectively. This trend is in agreement with the findings of Cöpur and Tozluoglu<sup>(15)</sup> and Jiang<sup>(10)</sup>. This effect of PS is mainly as a result of increasing the retention of hemicellulose, especially glucomannan which dissolves in early stage and almost completely during kraft cooking followed by dissolution of xylan<sup>(27)</sup>. Landmark<sup>(18)</sup> claimed that the PS stabilizes the polysaccharides against peeling reactions by oxidation of end group and the stabilization of hemicellulose begins early at 90 °C which is fast at the beginning then decreases quickly. Simultaneous addition of 0.1% AQ and 3%PS resulted in a relative yield increase of about 12.15% and 6.19% for 1:4 and 1:10 B/L ratios, respectively. This is considerably higher than the yields observed from independent addition of either of the additive. This synergistic effect of AQ and PS has been observed previously<sup>(10,17,18,28)</sup>.

**TABLE 2. Chemical analysis of pulp samples .**

Method	Yield %		Kappa no.		Viscosity C.Pa		Holocellulose %		α-cellulose %		1%NaOH solubility	
	1:4	1:10	1:4	1:10	1:4	1:10	1:4	1:10	1:4	1:10	1:4	1:10
<b>B:L ratio</b>	1:4	1:10	1:4	1:10	1:4	1:10	1:4	1:10	1:4	1:10	1:4	1:10
<b>Kraft (K)</b>	57.77	56.50	33.33	22.33	11.84	12.80	91.80	94.25	70.60	70.90	11.40	11.05
<b>K-0.1% AQ</b>	60.50	57.30	31.00	21.33	12.20	12.98	94.25	96.87	71.40	71.00	10.50	10.60
<b>K- 1%BH</b>	58.41	58.12	30.00	20.00	11.86	13.66	94.50	96.50	72.15	72.50	10.60	9.10
<b>K- 2%BH</b>	59.36	59.00	28.67	16.00	11.96	13.74	94.90	97.00	72.50	73.15	10.30	9.00
<b>K- 3%BH</b>	59.50	59.44	29.47	13.33	12.26	15.02	95.73	97.60	73.15	74.00	10.00	7.75
<b>K-3% BH +0.1% AQ</b>	60.11	59.50	30.33	21.00	12.02	12.16	94.60	94.90	72.55	72.20	9.35	8.40
<b>K-3% PS</b>	61.50	58.00	52.60	32.00	12.35	14.28	93.00	94.53	70.80	71.00	11.30	10.15
<b>K-3% PS +0.1% AQ</b>	64.79	60.00	43.33	30.60	12.50	14.40	93.40	95.50	70.95	71.20	10.90	11.15

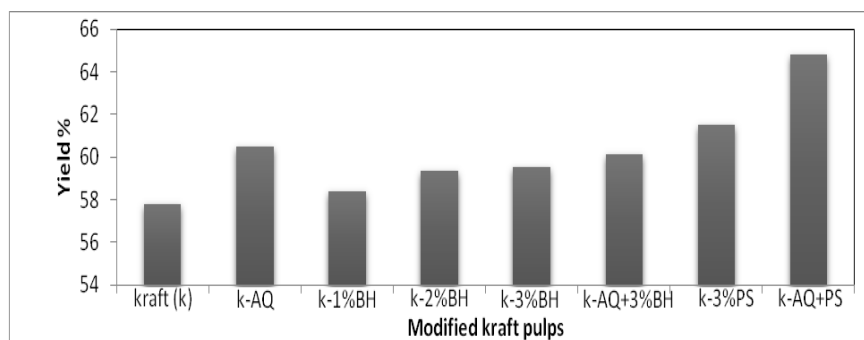


Fig. 1. Effect of additives on the yield % at 1:4 B/L ratio.

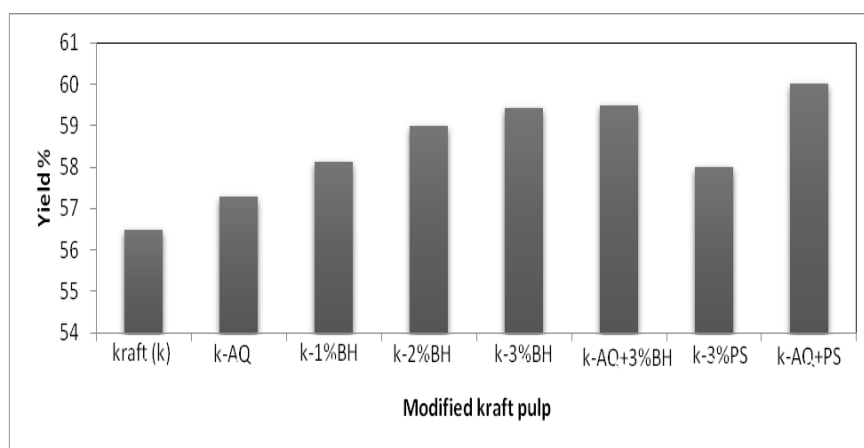
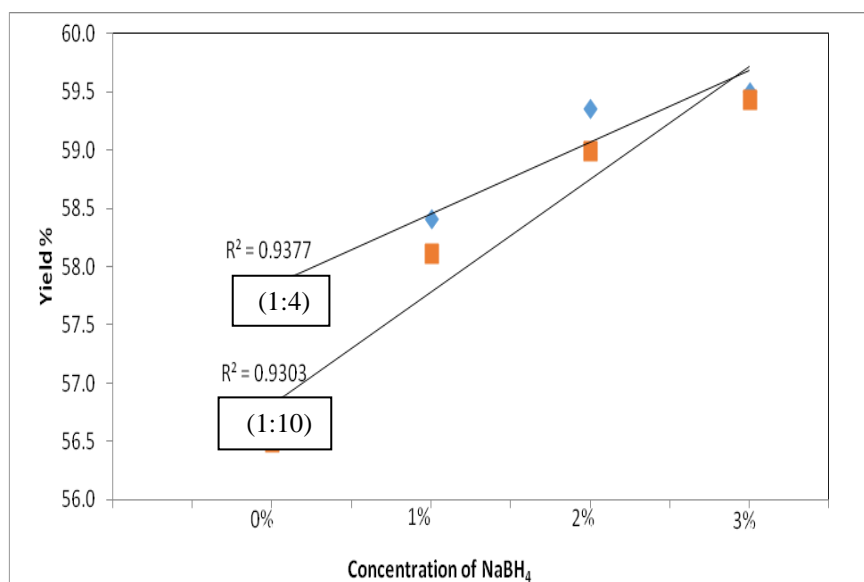


Fig. 2. Effect of additives on the yield % at 1:10 B/L ratio.

Our results are in a good agreement with the synergistic effect reported in literature<sup>(29-32)</sup>. One possible mechanism by which this effect occur is that PS may participate in the AHQ-lignin and AQ-carbohydrate redox system<sup>(33)</sup>. Increasing the yield gain for 1:4 than 1:10 B/L ratios in case of Kraft-PS or Kraft-AQ-PS may be due to increasing polysulfide concentration consequently, increasing the stabilization of polysaccharides<sup>(17)</sup>.

Figure 3 shows that the relative yield increases with increasing concentration of  $\text{NaBH}_4$  at both B/L ratios of 1:4 and 1:10. At low concentrations however, the additive seems to be more effective at B/L ratio of 1:10. This trend was confirmed by several studies<sup>(14,15,19,20,34-37)</sup>. Our data are in agreement with findings of Akgül *et al.*<sup>(34)</sup> who claimed that, increasing in yield is due to carbohydrates' preserving mechanism of  $\text{NaBH}_4$  where, Kraft- $\text{NaBH}_4$  better preserved the carbohydrate chains and improved the pulp viscosity through the lower kappa number. Borohydride prevents the acceleration of glucomannan removal.



**Fig. 3.** The relation between percentage yield and concentration of NaBH<sub>4</sub> at 1:4 and 1:10 B/L ratios, respectively.

#### Delignification

The total lignin in pulp is the sum of insoluble lignin (klason lignin) and acid soluble lignin. Addition of 0.1% AQ to the cooking liquor decreased the kappa number by 7% and 4.48% at 1:4 and 1:10 B/L ratio, respectively, which refers to increasing the rate of delignification by addition of AQ because it facilitates  $\beta$ -ether cleavage producing lower molecular weight lignin fragments through its redox cycle. Several authors<sup>(10,38-40)</sup> showed a reduction in kappa number (7.07%-15%) when 0.1% AQ was used in Kraft pulping. It can be concluded that pulps with lower kappa numbers could be achieved when AQ is added to Kraft pulping specially in case of 1:10 B/L ratio. Generally, polysulfide pulping increased the kappa number. Our data are in accordance with Cöpur and Tozluoglu<sup>(15)</sup> who mentioned that if low kappa numbers are desired with polysulfide pulping the active alkali should be increased because polysulfide sulfur reacts with some hydroxide and oxidizes organic materials resulting in a reduction in hydroxide ions in white liquor and consequently a reduction in lignin delignification. Another studies<sup>(41-43)</sup> noted that the kappa number of polysulfide pulps was higher compared to the Kraft method. On the other hand, several authors<sup>(10,17,31,44)</sup> indicated that polysulfide pulps gave lower kappa numbers than that of corresponding Kraft pulp. Anyway, the effect of polysulfide pulping on kappa number is a contentious issue.

The presence of sodium borohydride in Kraft pulping affect the rate of delignification in both 1:4 and 1:10 B/L ratios where 1%, 2% and 3% NaBH<sub>4</sub> and 0.1% AQ-3%NaBH<sub>4</sub> reduced the kappa number by 10%, 11.52%,14% and 9% for 1:4 B:L ratio and by 10.4%, 28.35%, 40.30% and 5.95% in case of 1:10

B/L ratio compared to the main Kraft pulp, respectively. These results indicated a linear relation (Fig. 4) between pulp yield and kappa number with strong regression specially for 1:10 B/L ratio as also observed earlier<sup>(31,45)</sup> for kraft-NaBH<sub>4</sub> pulps. More dissolution of lignin was obtained for higher B/L ratio because in the high liquor to wood L/W ratio cooks wood components could easily diffuse and remain in the liquor, while in the low L/W ratio cooks a certain fraction of dissolved components reabsorbed on the fibers<sup>(46)</sup>. Joachimiak *et al.*<sup>(47)</sup> claimed that in the higher L/W ratio the higher value of extracted substances could be gained. Increasing the concentration of NaBH<sub>4</sub> decreased the kappa number which was assured by Istek and Gonteki<sup>(35)</sup> who found that increasing NaBH<sub>4</sub> from 1% to 3% for Kraft cook reduced the kappa number due to acceleration of delignification. Tutus *et al.*<sup>(48)</sup> reported also a reduction in kappa number for NaBH<sub>4</sub>-kraft cooking of cotton stalks.

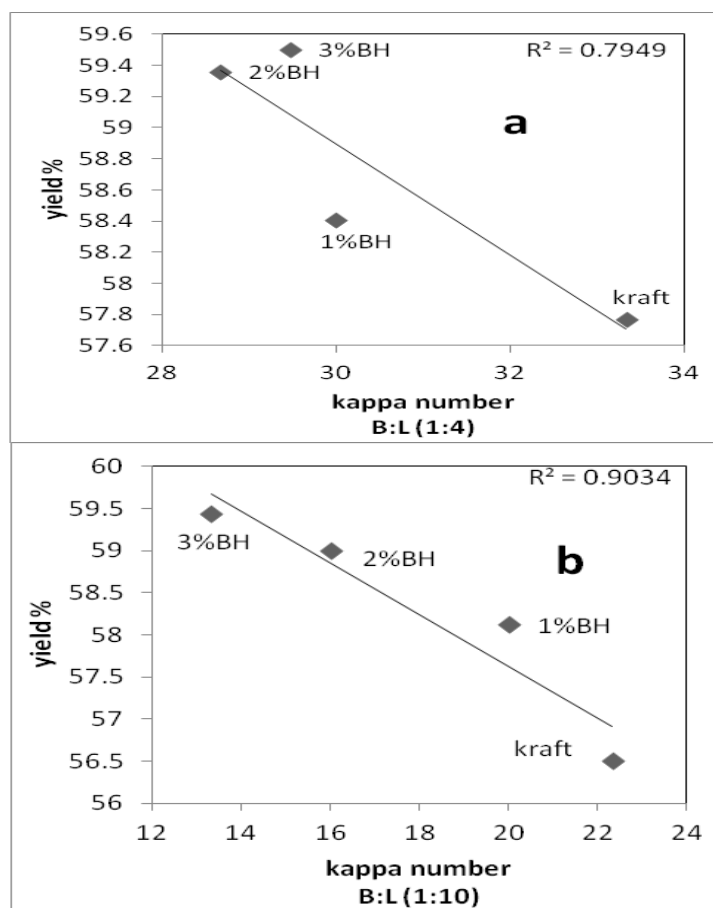


Fig. 4. Effect of sodium borohydride concentration on the relation between yield% and kappa number of Kraft pulps for 1:4 and 1:10 B/L ratios.

In a trial to explore the effect of combination 0.1% AQ-3% NaBH<sub>4</sub> on kraft cooking, improving the yield by 4.05% and 5.31% and reducing kappa number of resulting pulp by 9% and 5.96% for 1:4 and 1:10 B/L ratios, respectively was observed.

#### Viscosity of pulps

Viscosity measurements are usually determined to evaluate the degradation of cellulose in cooking and bleaching. Introducing AQ and PS in kraft pulping resulted in pulps with higher viscosities indicating that addition of these additives decreased cellulose degradation in pulping.

PS or combination of PS-AQ gave the most favorable effect on pulp viscosity (except 3% NaBH<sub>4</sub> for higher B/L ratio) compared to the other pulps in which they increase pulp viscosity by 4.31%, 5.57% and 11.56%, 12.5% for 1:4 and 1:10 B/L ratios, respectively. Improving the viscosity by these additives preserved the degree of polymerization of the cellulose chains which resulted in higher viscosity by increasing the percentage of carbohydrate yield. Our data is in accordance with the literature<sup>(14,19,34,37,44,49,50)</sup>. The results pointed out that NaBH<sub>4</sub> preserved the carbohydrate chains and improved the viscosity of the pulps through to the lower kappa number<sup>(14,34)</sup>. Increasing the rate of NaBH<sub>4</sub> from 1% to 3% improved the beneficial effect on pulp viscosity as shown in Fig. 5. The significant effect of the higher 1:10 B/L ratio showing acceleration in delignification and carbohydrate preservation resulting in higher viscosity.

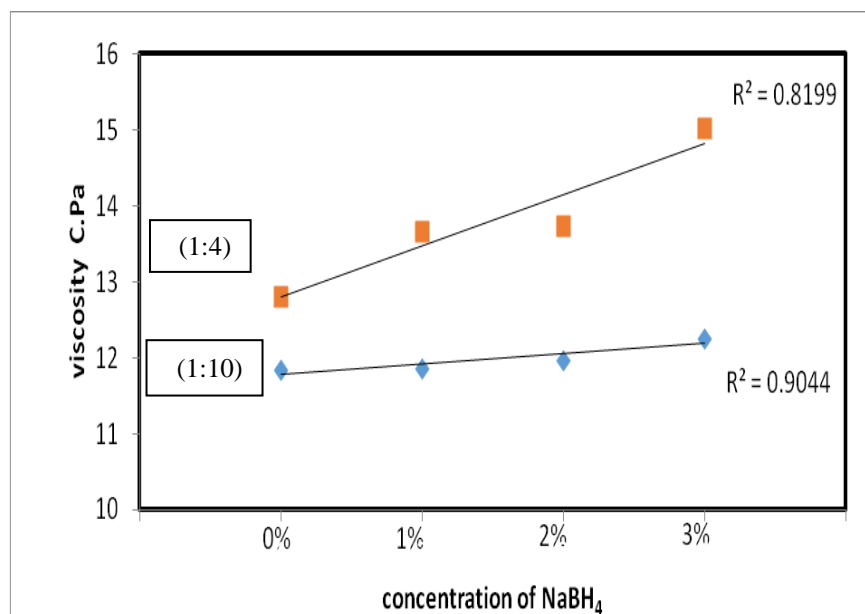


Fig. 5. The relation between viscosity and concentration of NaBH<sub>4</sub> for 1:4 and 1:10 B:L ratios.



*Holocellulose and  $\alpha$ -cellulose of pulps*

The reducing ends in carbohydrates (mostly hemicellulose) can be stabilized, and thus less peeling occurs, by using AQ, PS and  $\text{NaBH}_4$  during pulping. This can be remarked by the determination of holocellulose and  $\alpha$ -cellulose as indication of polysaccharides and cellulose, respectively. AQ, PS and  $\text{NaBH}_4$  improved the holocellulose and  $\alpha$ -cellulose in kraft pulp samples by protecting carbohydrates from degradation.  $\text{NaBH}_4$  has the highest protecting effect which appears through the higher values of holocellulose and  $\alpha$ -cellulose. Increasing the rate of  $\text{NaBH}_4$  in both B/L ratios increased the holocellulose and  $\alpha$ -cellulose content of kraft pulp samples due to increasing the protecting effect of  $\text{NaBH}_4$  which assured from the good regression of the straight line relationship (Fig.6). Our data are compatible with Tutus *et al.* <sup>(19)</sup> who used these additives in Kraft pulping of cotton stalks. Samples of 1:10 B/L ratio showed higher values of holocellulose and  $\alpha$ -cellulose contents which may also refer to increasing the protecting effect of these additives.

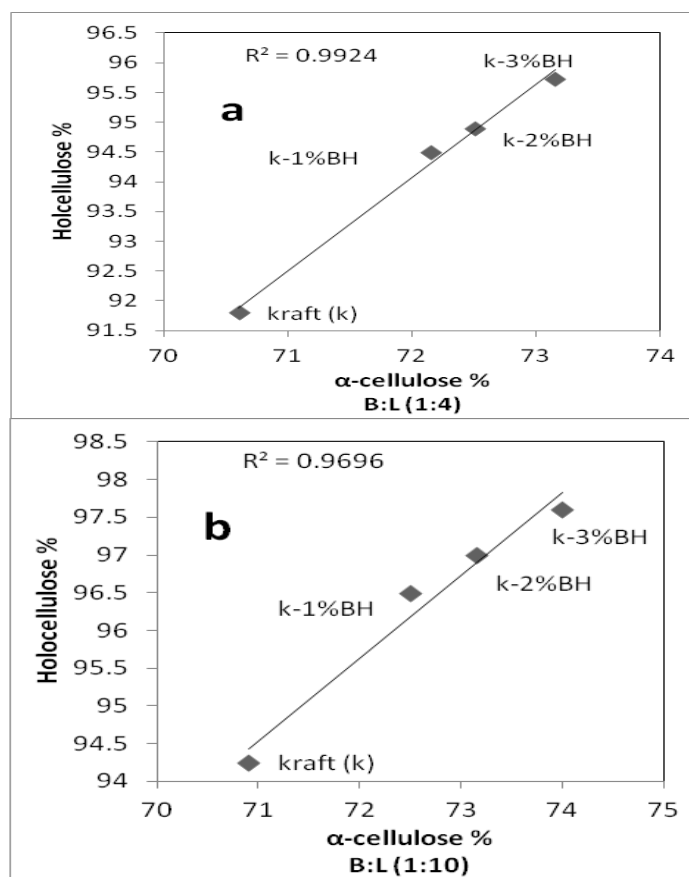


Fig. 6. Effect of sodium borohydride concentration on the relation between holocellulose % and  $\alpha$ -cellulose % for 1:4 and 1:10 B/L ratios.

*1%NaOH solubility of pulps*

Hot 1%NaOH extracts low molecular weight carbohydrates consisting mainly of hemicelluloses and degraded cellulose in wood and pulp. All additives showed lower values (except PS-AQ for 1:10 B/L ratio) than kraft pulp due to the carbohydrate preserving effect of these additives while, the higher 1:10 B:L ratio showed much lower values for AQ and NaBH<sub>4</sub>. Generally, PS and PS-AQ had higher values than AQ or NaBH<sub>4</sub> which may be due to the higher lignin content. As expected, increasing the level of NaBH<sub>4</sub> improved the carbohydrate protection consequently lowered the alkali solubility values of the produced pulps as shown in Fig. 7 which is confirmed by the strong regression of straight lines.

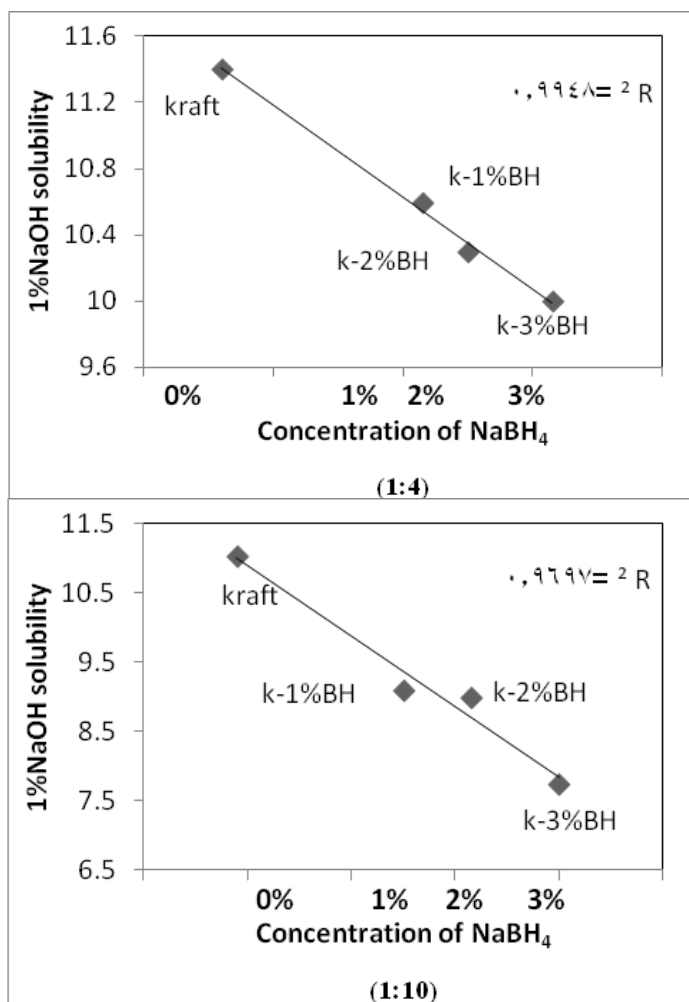


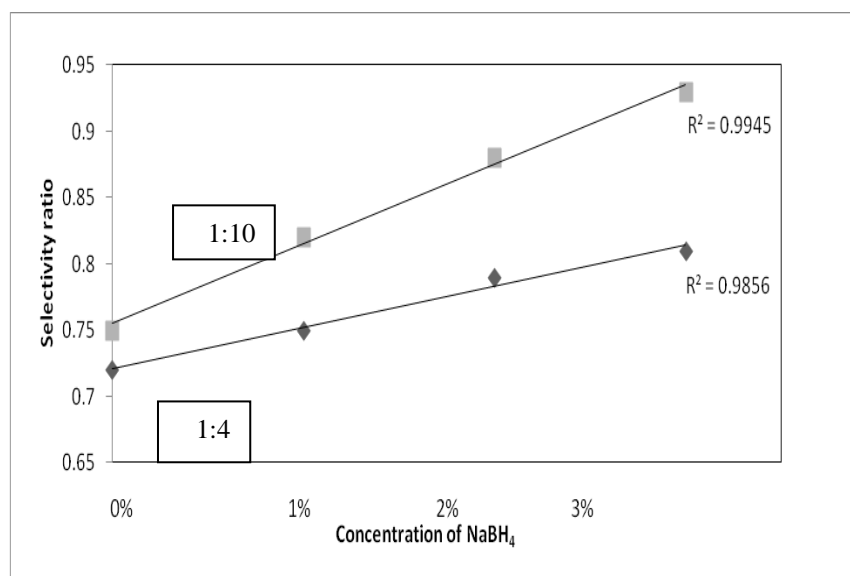
Fig. 7. Effect of sodium borohydride concentration on the alkali solubility of modified kraft pulps for 1:4 and 1:10 B/L ratios.

*Delignification selectivity*

The delignification selectivity is defined as the ratio of dissolved lignin to dissolved carbohydrates. It was calculated in Table 3 as mentioned by Biswas *et al.*<sup>(50)</sup>. The amount of lignin removed by AQ or NaBH<sub>4</sub> is higher than that of Kraft pulp in both B/L ratios due to the delignification effect of these additives while PS retarded it. On the other hand, the carbohydrate loss was decreased with AQ, PS and NaBH<sub>4</sub> addition. This resulted in higher selectivity ratio of Kraft-AQ, Kraft-AQ-PS, Kraft- NaBH<sub>4</sub> and Kraft -AQ-NaBH<sub>4</sub> for both B/L ratios compared to Kraft one. The selectivity ratio increased by increasing the level of NaBH<sub>4</sub> as observed from the strong regression for the straight lines of selectivity ratio and borohydride concentration (Fig.8). All additives showed higher carbohydrate yield than kraft pulp which may be due to a retardation of alkaline peeling reactions.

**TABLE 3. Delignification selectivity of modified kraft pulps for both 1:4 and 1:10 B:L ratios.**

Additives	B:L ratio	Kraft (k)	K-0.1% AQ	K-1% BH	K-2% BH	K-3% BH	K-3% BH + 0.1% AQ	K-3% PS	K-3% PS + 0.1% AQ
selectivity ratio	1:4	0.72	0.82	0.75	0.79	0.81	0.80	0.64	0.89
	1:10	0.75	0.79	0.82	0.88	0.93	0.86	0.70	0.84



**Fig. 8. The relation between selectivity ratio and concentration of modified kraft-NaBH<sub>4</sub> pulps for 1:4 and 1:10 B:L ratios.**

### Conclusion

Egyptian bagasse kraft pulp can be modified using yield-increasing additives. Addition of anthraquinone, polysulfide and sodium borohydride significantly increased the pulp yield especially when AQ and PS are used together due to their synergistic effect. AQ and NaBH<sub>4</sub> were observed to decrease kappa number while PS or PS-AQ increased it. With the exception of AQ-NaBH<sub>4</sub> at 1:10 B/L ratio, all the additives showed higher viscosities than Kraft pulps. The additives appear to improve holocellulose and  $\alpha$ -cellulose of pulp samples by protecting carbohydrates from degradation. On the other hand, increasing the concentration of NaBH<sub>4</sub> increased the yield, viscosity, holocellulose and  $\alpha$ -cellulose, and decreased the kappa number and 1% alkali solubility. In general, Increasing the B/L ratio resulted in increased yield, viscosity, holocellulose and  $\alpha$ -cellulose but decreased kappa number of pulps.

Improving the chemical properties of bagasse kraft pulp could be achieved by using anthraquinone, polysulfide and sodium borohydride.

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## تعديل لب مصاص القصب المصرى المحضر بطريقة الكرافت باستخدام إضافات محسنة للنتائج

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يعتبر مصاص قصب السكر من الخامات الأساسية لصناعة لب الورق في مصر. لذلك فهدفنا هو تحسين الخواص الكيميائية لللب الكرافت باستخدام مواد محسنة للنتائج مثل الانثراكينون وعديد الكبريتيد وبوروهيدريد الصوديوم . كما تم دراسة تأثير نسبة المصاص إلى محلول الطبخ ولقد زادت حصيللة الناتج عند إضافة الانثراكينون وعديد الكبريتيد وبوروهيدريد الصوديوم والانثراكينون مع عديد الكبريتيد والانثراكينون مع بوروهيدريد الصوديوم بمقدار 4.73% و6.46% و2.99% و12.15% و4.05% على الترتيب وذلك عندما كانت نسبة المصاص إلى محلول الطبخ 4:1 . بينما زادت حصيللة الناتج بمقدار 1.42% و2.65% و5.2% و6.9% و5.3% على الترتيب عندما كانت نسبة المصاص إلى محلول الطبخ 10:1 . عند استخدام الانثراكينون مع عديد الكبريتيد أعطى تأثيرا متناغما. لقد قلل استخدام الانثراكينون بمفرده أو مختلطا مع عديد الكبريتيد من رقم الكابا بمقدار 7% و14% و9% و4.48% و40.3% و5.95% بالنسبة لنسبتي المصاص إلى محلول الطبخ 4:1 و10:1 على الترتيب . كما أن التركيز العالى من نسبة المصاص إلى محلول الطبخ أذاب كمية أكبر من اللجنين . الطبخ باستخدام الإضافات احتفظ بناتج الكربوهيدرات أكثر من الطبخ بالكرافت فقط ، كما بينت الإضافات وجود درجة عالية من انتقائية اللجنين (ما عدا استخدام عديد الكبريتيد).