

# Evaluation of pulping by propylene glycol

Zenat, A. N.\* and Enas, A. H\*\*.

Cellulose and Paper Department, National Research Center, El-Behoos street, Dokki, Cairo, Egypt 12622.

This study was carried out to acquire basic data necessary for the use of non wood pulp and save energy with different concentrations of propylene glycol (PG). Study was carried out by pulping bagasse with different concentrations of (PG) (80-100 %) at different temperatures (140 -170°C) at atmospheric pressure. We found that the best temperature was 150°C and best concentrations of (PG) was 90% for 2 hrs. By comparing this suitable conditions of pulping at atmospheric pressure by pulping under pressure we found that pulping at atmospheric pressure kappa no. reduced from 29.33 to 22.42 and brightness increased from 30.3 to 37.8 but pulping at pressure gave higher yield.

## INTRODUCTION

According to FAO report [1] the growth rate of the pulp and paper industry will be 3% per annum up to 1995 in developed countries, but it is estimated to be 5.1 % in the developing countries, and even 6% per annum in the Far East. This means that the use of non-woody plants in pulp and paper making will considerably increase in developing countries. Sugar cane bagasse is one of the most important non-wood fibrous raw materials in developing countries. The process to pulp bagasse for bleachable-grades with higher yields and better mechanical properties has attracted increasing attention. For chemical pulp production from bagasse the soda process is the most applied one. In some pulp mills the kraft process is also used. But both processes are not satisfying from the economical and environmental point of view.

Worldwide a large number of investigations have been performed to develop new processes or process modifications. The addition of anthraquinone in bagasse soda pulping accelerates pulp delignification and gains higher yields [2-4]. Applying this process it was possible to produce pulps with physical strength properties similar to kraft. Valladares et al. [5] added methanol or ethanol to an anthraquinone catalysed soda cook to improve the selectivity of lignin dissolution, resulting in high yield pulps with good strength properties. Due to the lower active alkali charge, the cooking time, ranging from 4 to 6 hours, was too long for an industrial production since kraft pulping requires a large capacity and recovery of chemicals is tedious. Thus novel systems must be developed that can be operated on a small scale with low consumption of energy and easy recovery of cooking chemicals. They must also be pollution-free. If cooking chemicals could be readily obtained from wood components, the possibility for construction of mills becomes more viable.

Organosolv pulping is suitable for the proposed mills, since such pulping is economical and pollution-free [6]. In addition, it yields major components of woods as valuable byproducts.

However, the separation of softwoods by such pulping not satisfactory since the pulping of softwoods requires harsh conditions, such as high cooking temperature and long cooking time, as compared to pulping of hardwoods [6-8].

In this study, we investigated pulping of softwoods with polyhydric alcohols as a novel form of organosolv pulping. Polyhydric alcohols might be produced from hemicellulose sugars, since those with two and three carbons, such as glycerol, can be produced by Newberg fermentation and/or other fermentations with specific yeasts[9]. Thus the development of polyhydric alcohol pulping could lead to establishment of a chemically self-supporting systems. Digestion of softwoods with triethylene glycol as cooking solvent has already been reported for preparation of high-energy food for ruminants[10]. The digestion was performed with glycol and Lewis acids as a catalyst. However, it was unclear from this work whether or not fibrous components that were obtained were utilized as pulp and as a source of cellulose.

The objective of our study is using polyhydric alcohol as organosolv pulping in presence of mineral acids instead of Lewis acid. In addition; operation at atmospheric pressure was investigated comparing with pulping under pressure. And also this organosolv pulping was studied with respect to delignification rates, yield, brightness, viscosity and chemical properties of the pulps.

## EXPERIMENTAL

### Raw Material

The fibrous raw material used in this work was depithed Egyptian bagasse obtained from El-Nasr Company for Sugar and Pulp Industry at Edfu. Bagasse was extracted with ethanol-benzene mixture (2: 1 v/v) for 8 hours, washed with distilled water, and left for air dry. The chemical composition of bagasse was 21.4% lignin, 25.9% hemicellulose, 78.3 % holocellulose, 55.3% alpha-cellulose, and 1.4% ash.

Reagent grade (BDH chemicals Ltd Poole, England) propylene glycol (PG) was used for organosolv

pulping.

### Pulping

25 grams of bagasse were firstly pretreated with 95% PG at liquor ratio of 1:6 (w/v) in one-liter flask and heated at 60°C for 30 minutes. The liquor was filtered, then fresh liquor of PG having different concentrations (80 - 100%) and 0.29% sulfuric acid (wt/wt of liquor). The pulping time and temperature were varied from 1-3 hours and 140-170°C, respectively. The flasks were put in an oil bath and heated under reflux for specified reaction time. After pulping the crude pulp was filtered and washed successively with 80 % aqueous acetone and water.

Pulping was also carried out under pressure in an autoclave using the same conditions of PG concentrations, pulping time, liquor ratio and catalyst at 150°C. After washing, the pulp was screened to remove the unreacted bagasse. The yield of screened pulp and rejects were determined.

### Isolation of organosolv lignin

Bagasse was delignified at atmospheric pressure or on autoclave by PG at different concentrations and reaction times. The waste liquor was collected and lignin was precipitated by adding to it five volumes of water. Precipitated organosolv lignin was filtered out, washed with water, and vacuum dried at 65°C.

### Pulp properties

The pulp sheets were prepared with a Buchner funnel (12.5 mm diameter) and dried by a rotating drum at 100°C for 2 hours. Brightness was determined using an Hunter-lab calorimeter. Kappa number was determined according to TAPPI standard T-236m. Degree of polymerization was determined according to SCAN-cm 15:88.

Holocellulose, hemicellulose, and a-cellulose were determined according to the standard methods [11].

## RESULTS AND DISCUSSION

In order to study the influence of the cooking

temperature on pulp yield, rejects, degree of polymerization, kappa no, optical properties and laboratory trials were performed simulating a best condition for the pulp production. Three different cooking temperatures were studied 150,160,170°C due to at 140°C no pulping occur and also three concentrations of (PG) were 100, 95 and 90% due to 85% no pulping occur.

### Effect of different concentration of (PG) pulping at 150°C

It is clear from Table 1 that the results indicate that the selectivity of the lignin dissolution was practically independent of operating conditions. When concentration of PG reached a value 100% the selectivity decreased considerably due to lignin condensation [12]. It was clear from kappa number increased 34.88 for 3 hours while one hour was 30.56. This was also clear for 95% PG the kappa number 36.15 for 3 hours but 29.05 for one hour. But 90% PG concentration gave more lignin dissolution especially for 2 hours gave lower kappa number 22.45, also higher brightness and higher holocellulose reached 37.8%. But DP and a cellulose were slightly reduced.

The best favorable conditions for pulping with PG at atmospheric pressure was 90% for 2 hours at 150°C which gave higher pulp yield, lower rejects and lower kappa number.

### PG Pulping at 160°C

Also, it was clear from Table 2 that the best conditions for pulping was 90% PG but at 1 hour gave lower kappa number 27.32 and also higher yield 42.14%. It was observed that the best concentration for delignification was 90%PG only for one hour gave higher yield, higher brightness small significantly effect on DP and higher holocellulose percent reached 92.16.

### PG Pulping at 170°C

It was clear that at higher temperature 170°C the viscosity decreased with the increase with the

Table 1 : Effect of pulping time and PG concentration on chemical composition of bagasse at 150°C and atmospheric pressure.

	Propylene glycol concentration								
	100 %			95 %			90%		
Pulping time	1hr	2hr	3hr	1hr	2hr	3hr	1hr	2hr	3hr
Screened yield, %	35.88	35.51	36.28	38.11	36.35	38.52	43.27	40.07	41.89
Rejects, %	6.61	4.44	1.05	4.34	2.56	0.97	1.28	0.0	0.0
Total yield,	42.49	39.95	37.33	42.45	38.91	39.49	44.55	40.07	41.89
Holocellulose, %	88.69	90.41	83.02	89.94	92.41	82.22	92.54	93.77	85.22
a-Cellulose, %	87.59	85.46	80.54	86.99	83.66	80.25	85.22	81.96	75.13
Hemicellulose, %	12.38	11.29	10.08	11.92	9.64	9.05	10.33	9.01	9.00
Kappa no.	30.56	29.16	34.88	29.05	26.06	36.15	26.11	22.45	32.44
Brightness	30.7	32.3	28.5	32.4	35.5	27.8	35.7	37.8	29.5
D.P.	1356	1270	997	1321	1146	969	1253	1100	889

simultaneous increase in cooking temperature and cooking time. All different concentrations of PG pulping kappa number increased due to lignin condensation. But only 90% PG for 1 hour gave slightly reduced kappa number 30.99, higher brightness 30.5 and lower viscosity 938.

Generally from all our experiments by pulping of PG at different temperatures at atmospheric pressure the best temperature was 150°C this improve that the delignification can be proceed at lower temperature this has been reported [13&14] due to homogeneity of the cook increased which enhances the penetration and diffusion of the cooking chemicals into wood chips and leads to

lower amount of rejects [15].

#### PG Pulping at 150°C on autoclave

By comparing effect of pulping by PG at atmospheric pressure with pulping on autoclave we noticed that from Table 4 the best concentration of PG which gave higher delignification rate, low brightness 30.3, DP slightly reduced 938 and kappa number 29.33 was 90% concentration and pulping for hour. But pulping at atmospheric pressure at that temperature was better than autoclave.

It was clear from Fig. 1 effect of pulping time at different temperature on kappa number. Best conditions obtained at a temperature 150°C for 2

**Table 2 : Effect of pulping time and PG concentration on chemical composition of bagasse at 160°C and atmospheric pressure.**

	Propylene glycol concentration								
	100 %			95 %			90%		
Pulping time	1hr	2hr	3hr	1hr	2hr	3hr	1hr	2hr	3hr
Screened yield, %	34.38	34.78	35.61	36.06	37.42	39.11	42.14	41.38	42.83
Rejects, %	4.31	2.68	0.0	2.52	0.69	0.0	1.22	0.0	0.0
Total yied,	38.69	37.46	35.61	38.58	38.11	39.11	43.36	41.38	42.83
Holocellulose, %	88.58	86.76	80.00	88.87	88.99	83.55	92.16	89.56	84.15
a-Cellulose, %	84.41	84.25	79.15	83.04	81.87	78.24	80.76	77.22	73.27
Hemicellulose, %	10.59	10.36	9.00	10.00	9.77	9.17	10.22	9.02	9.00
Kappa no.	30.32	30.82	37.98	30.13	30.45	34.24	27.32	28.01	34.52
Brightness,	30.6	30.4	26.3	30.6	30.6	28.8	34.9	33.1	28.7
D.P.	1146	1146	958	1129	1100	938	1015	918	724

**Table 3 : Effect of pulping time and PG concentration on chemical composition of bagasse at 170°C and atmospheric pressure..**

	Propylene glycol concentration								
	100 %			95 %			90%		
Pulping time	1hr	2hr	3hr	1hr	2hr	3hr	1hr	2hr	3hr
Screened yield, %	41.01	36.49	38.32	42.41	41.47	42.41	45.62	46.14	46.28
Rejects, %	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total yield,	41.01	36.49	38.32	42.41	41.47	42.41	45.62	46.14	46.28
Holocellulose, %	83.36	82.63	80.04	84.62	82.91	83.51	88.36	83.95	82.52
a-Cellulose, %	82.11	80.55	77.62	81.44	79.02	75.61	78.53	75.32	73.01
Hemicellulose, %	9.52	9.33	8.23	9.11	9.00	8.01	8.55	8.15	8.00
Kappa no.	33.88	35.77	38.34	32.13	35.36	36.24	30.99	33.99	35.02
Brightness,	29.1	27.5	26.3	29.7	28.0	27.0	30.5	28.8	27.6
D.P.	1191	997	918	977	958	899	938	899	724

hours pulping gave lower kappa number 22.45 by 90% PG concentration pulping. Also clear from this figure all pulping increased kappa number with prolonged increase with time due to lignin condensations.

By studying the effect of different pulping methods by PG on kappa number it was clear from Fig.2 that pulping with PG at atmospheric pressure with different concentrations gave lower kappa number than pulping by PG in autoclave. The best kappa number was obtained by pulping for 2 hours with concentration 90 % which reached 22.45 much lower than under pressure by 7 points.

It was clear from Fig. 3 that viscosity of the pulp produced by pulping with different concentration of PG had higher viscosity than pulping under pressure. This means that pulp had a higher stabilization and protection of the carbohydrate, particularly the long-chain cellulose acromolecules against degradation during the PG pulping at atmospheric pressure.

It was clear from Fig. 4 that generally brightness in all pulping conditions by PG/atmospheric pressure was much better than autoclave especially with one hour by concentration 90 % which reached 37.8%.

#### CONCLUSIONS

Pulping by PG at atmospheric pressure was much better than pulping in autoclave. Brightness was much higher by pulping at atmospheric pressure. The low lignin content, high a cellulose and unbleached brightness make these pulps easily bleachable. The best concentration of PG and the best time were 90% and 150° respectively viscosity was obtained by pulping by PG at atmospheric pressure. These properties suggest that the pulps could be used not only for papermaking but also as dissolving pulp and as sources of highly crystalline cellulose. In addition, lignin was recovered in high yield by precipitation into water. There for, PO pulping seems to be a promising

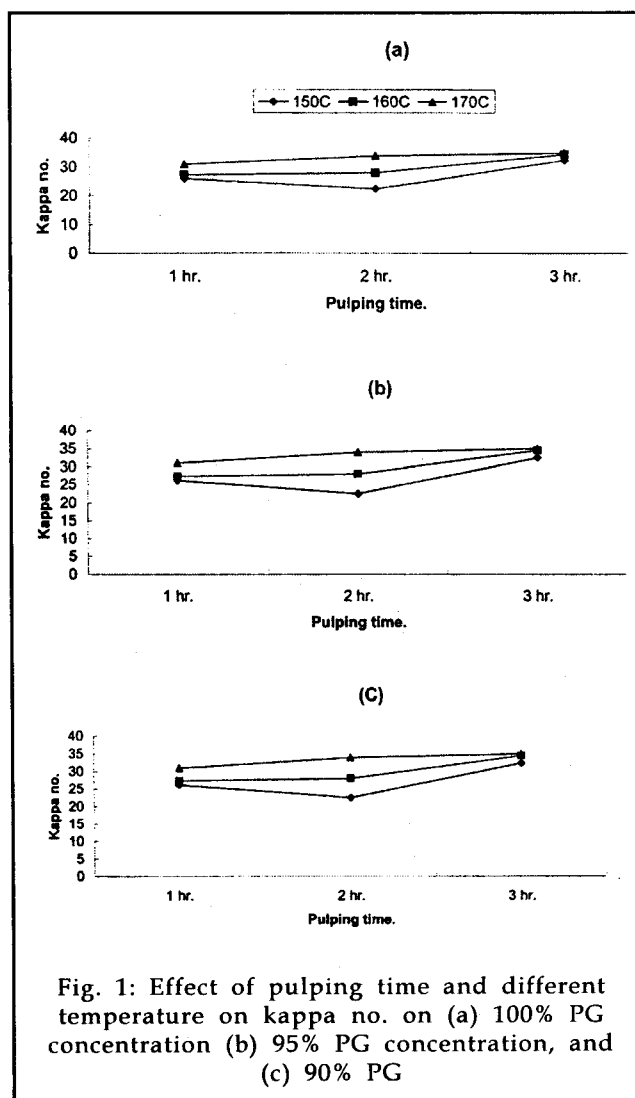


Fig. 1: Effect of pulping time and different temperature on kappa no. on (a) 100% PG concentration (b) 95% PG concentration, and (c) 90% PG

Table 4 : Effect of pulping time and PG concentration on chemical composition of bagasse at 150°C and on autoclave.

	Propylene glycol concentration					
	100 %		95 %		90%	
Pulping time	1hr	2hr	1hr	2hr	1hr	2hr
Screened yield, %	43.48	42.52	44.06	43.43	43.51	44.94
Rejects, %	5.91	2.03	2.53	1.92	0.0	0.0
Total yied,	49.39	44.55	46.59	45.35	43.51	44.94
Holocellulose, %	81.91	81.06	82.91	80.28	89.85	81.78
a-Cellulose, %	80.13	78.35	79.85	77.95	79.02	76.51
Hemicellulose, %	11.08	10.00	10.49	9.79	9.28	9.01
Kappa no.	38.65	37.52	35.84	38.11	29.33	34.85
Brightness	27.0	26.4	28.0	27.4	30.3	28.9
D.P.	959	919	948	939	938	918

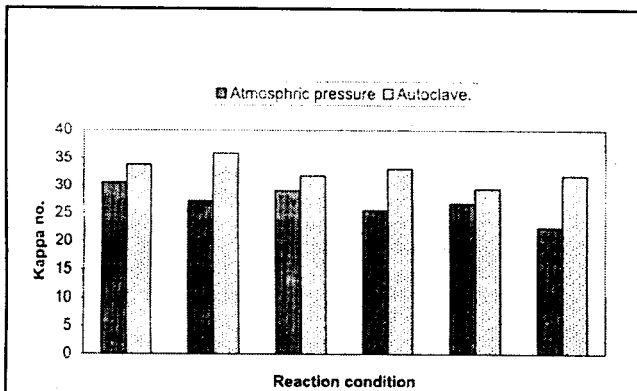


Fig. 2 : Effect of pulping method on kappa no. at different PG concentrations and times.

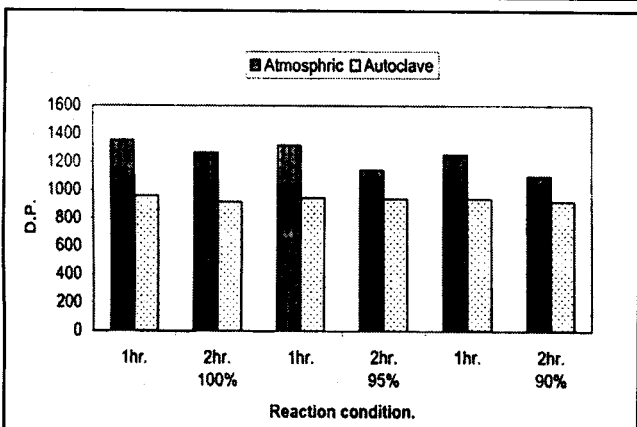


Fig. 3: Effect of pulping method on D.P. at different PG concentrations and times.

method for pulping soft wood as compared to other type of autocatalysed alcohol organosolv pulping.

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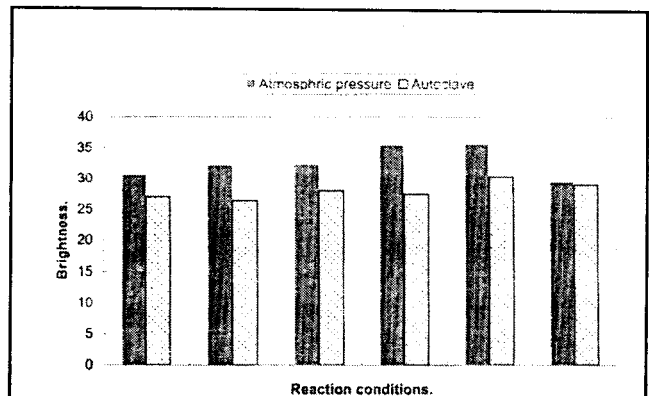


Fig. 4: Effect of pulping method on brightness at different PG concentrations and times.

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