

Bagasse Pulping by Using Caro's Acid

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ABSTRACT

Caro's acid (persulphuric acid) has been proposed as a promising reagent used in pulping of lignocellulosic materials. Bagasse was subjected to caro's acid pulping under different pulping conditions of per acid concentration (2~8 percent), pulping temperatures (40~60°C) and pulping time (120~240 min). The influence of different parameters of caro's acid pulping process on the chemical and strength properties of the produced pulps had been studied. Peracid concentration and pulping temperature are the most important variables of the pulping process. High degree of delignification and good defiberation were achieved at moderate pulping conditions in the studied range.

Pulping experiments of bagasse by using caro's acid had been carried out for determination of the optimum conditions of the process. In such case to effectively tackle the problem, a specific design of an experiment is selected depending on the goals to be achieved in the experimentation. The study could enable us to expect the bagasse pulp properties (yield, Kappa number, DP) that be stated as function of the independent variables of caro's acid pulping process (per acid concentration, pulping temperature and pulping time).

1. Introduction

Current industrial processes for pulping wood and annual plants have evolved slowly over many decades. Although these processes are complex and energy intensive, they are fairly efficient. Their major disadvantage is their negative impact on the environment. Even the best current technology is unable to suppress the odors emitted by kraft mills.

The discovery of new methods for more easily or more efficiently pulping of lignocellulosic materials could lead to the development of new more efficient, less environmentally troubles and energy saving pulp-

ing processes.

Recent investigations had studied the possibility of using organic per acid^{1,2)} for lignocellulosic pulping. Peracetic acid and performic acid readily delignify wood and other lignocellulosic materials.³⁻⁶⁾

Recently, it was found that solutions of caro's acid (persulphuric acid) at atmospheric pressure is very effective in delignification of wood.⁷⁻⁹⁾ The discovery of the pulping ability of caro's acid led to a consideration of the potential end uses as a promising reagent to produce chemicaltype pulps. Based on the studies conducting to date,¹⁰⁾ caro's acid pulping of hard woods and annual plants appear to be very economical-

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ly viable and less environmentally troublesome compared with the current processes. The objective of this research was the utilize Caro's acid (persulphuric acid) in pulping of depithed bagasse which is considered the most important raw material for pulp manufacture in Egypt. The research had studied the influence of pulping parameters, e.g., per acid concentration, temperature and pulping time on the chemical and strength properties of the produced bagasse pulps. Optimizing the pulping conditions and anticipation of the produced pulp properties could be considered an ultimate goal in this study.

2. Experimental

2.1 Material

Depithed bagasse was used in this study. It has the following analysis : Lignin 20.90 percent, α -cellulose 40.10 percent, hemicellulose 20.40 percent and ash 1.05 percent.

2.2 Methods

2.2.1 Preparation of Caro's acid (Persulphuric acid)

Caro's acid was prepared by placing a known weight of hydrogen peroxide in flask. The flask is kept in a ice bath. The sulphuric acid (95 percent) is added dropwise using a funnel while the mixture is agitated. The peracid and peroxide concentrations were measured by a method proposed by Greenspan and Mackellar.⁽¹⁾ Caro's acid used in this study had been analysed as follows :

Persulphuric acid percent: 9.45
Hydrogen peroxide percent: 2.85
Total active oxygen percent: 5.95

2.2.2 Pulping

Bagasse pulping was carried out by using different caro's acid concentrations from 2 to 8 percent (based on bagasse weight) at pulping temperatures 40, 50 and 60°C for pulping time from 120 to 240 min with liquor ratio 10 : 1. The chemical analysis of the produced pulps were determined by TAPPI Standard methods.

2.2.3 Paper sheet formation

Pulps were beaten to 40 °SR in a Jekro Beater. Hand paper sheets of basis weight 68 g/m² were formed according to SCAN Standard. Strength properties (breaking length, burst factor and tear factor) were tested according to TAPPI standard method.

3. Results and Discussion

3.1 Effect of persulphuric acid concentration and pulping temperature

3.3.1 Yield

Fig. 1a shows the effect of persulphuric acid concentration on the yield of produced bagasse pulps. At pulping temperature 40°C and 50°C, the screened pulp yield increases with increasing of persulphuric acid concentration till certain concentration after which pulp yield decreases with increasing of persulphuric acid concentration. At higher pulping temperature 60°C, the screened pulp yield decreases with increasing of persulphuric acid concentration, i.e., pulp yield not pass through maxima. The appearance of the maxima at lower pulping temperature is due to the presence of considerable amount of shives which is reduced with increasing of persulphuric acid concentration. At higher pulping tem-

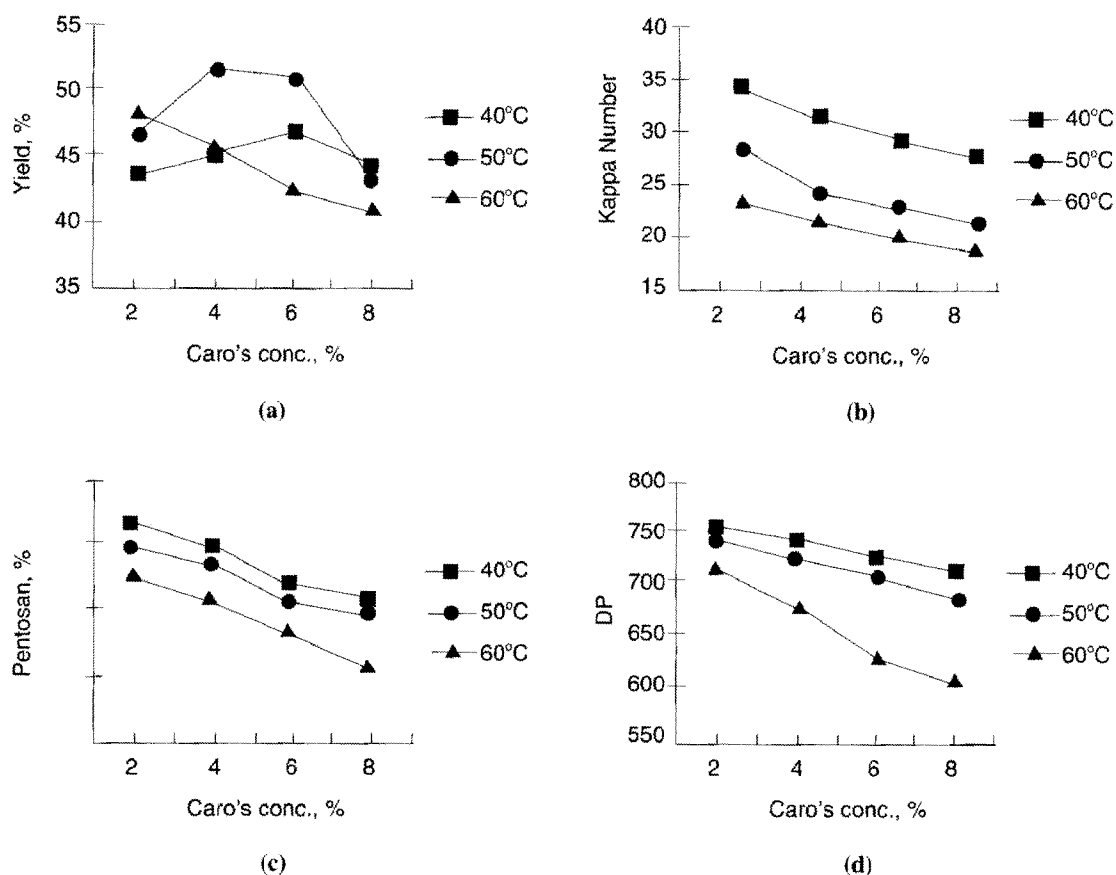


Fig. 1. Effect of Caro's acid concentration (based on raw material) on the properties of the produced pulps at different temperatures for 3 hrs at 10:1 liquor ratio.

perature a minute amount of shives is produced so, the feature of pulping yield vs. persulphuric acid concentration not pass through maxima.

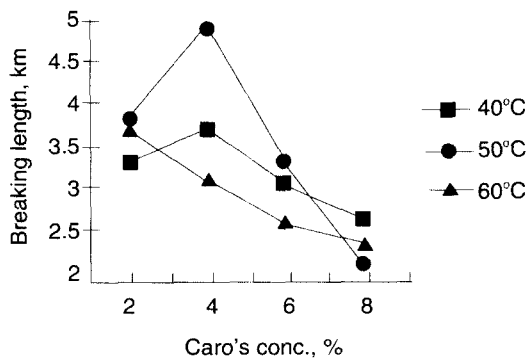
3.1.2 Kappa number

Kappa number is greatly affected by persulphuric acid concentration and pulping temperatures as shown in Fig. 1b. Principle effect of pulping temperature has manifested at low and moderate concentration of persulphuric acid since the rate of decrease in Kappa number values of the produced pulps was considerable. at high persul-

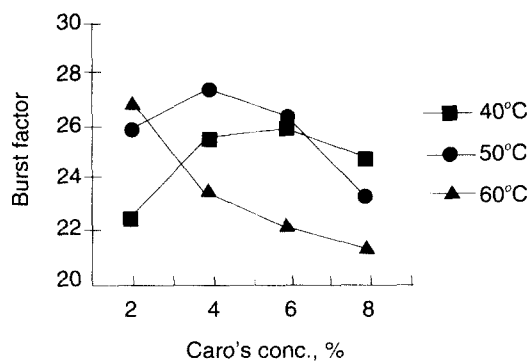
phuric acid concentration, small decrease in Kappa number has been achieved by increasing in pulping temperature. Therefore, at certain pulping temperature, moderate persulphuric acid concentration could be suitable for achieving of good degree of delignification.

3.1.3 Pentosan content :

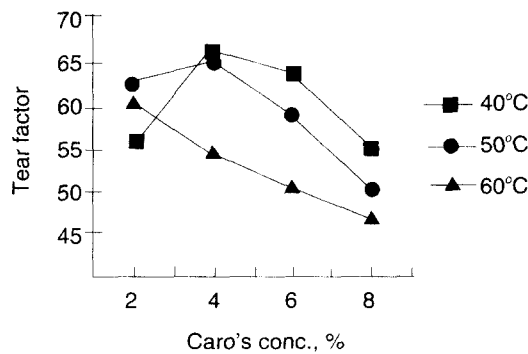
It's important to follow the effect of pulping temperatures and persulphuric acid concentration on the pentosan content in the produced pulps as shown in Fig. 1c. The importance of this following is referred to the



(a)



(b)



(c)

Fig. 2. Effect of Caro's acid concentration (based on raw material) on the strength properties of the produced pulps at different temperatures for 3 hrs at 10: 1 liquor ratio.

sensitivity of this pulp component (pentosan content which is a measure of hemicellulose) toward the acidic pulping conditions. The hydrolysis action of persulphuric acid upon hemicellulose fraction, especially at high pulping temperatures, results in notable decrease in the pulp yield.

3.1.4 Degree of polymerization (DP)

As shown in Fig. 1d, both increasing of pulping temperatures and persulphuric acid concentration results in decreasing in the degree of polymerization of the mean cellulosic chains. The sever degradative effect has been appeared at high pulping temperatures and persulphuric acid concentrations.

3.1.5 strength properties:

Figs. 2a, 2b, 2c show the effect of pulping temperature and persulphuric acid concentration upon the strength properties (breaking length, burst factor and tear factor) of paper sheets made from the produced pulps, it's clear that, at low and moderate pulping temperatures the strength properties could be enhanced by increasing of persulphuric acid till certain concentration could result in decrease in the strength properties. The increasing in the strength properties of the pulps till certain, per acid concentration may be attributed to the increasing of the degree of delignification which enhances the fibrous homogeneity and intera and inter-fiber bonding. The decreasing of the strength properties at higher per acid concentration my be refered to great decrease in pentosan content and the sever degradative action on the mean cellulosic chains, i.e., reducing in pulp DP.

At higher pulping temperature the strength properties decrease with increasing of persulphuric acid concentration within the range of peracid concentration studied. In conclusion,

Table 1. Effect of pulping time on the properties of the produced pulp by using 4 percent caro's acid (based on raw material) at 50°C and 10:1 liquor ratio.

Pulping time, min.	Screened yield, %	Kappa number	DP	Strength properties		
				Breaking length/km	Burst factor	Tear factor
120	46.20	29.85	755	3.77	28.37	66.35
180	51.55	24.34	730	4.85	27.24	65.45
240	48.70	22.65	645	3.65	26.21	45.80

the values of the breaking length and burst factors of pulps produced by moderate per acid concentrations and temperatures are acceptable. The tear properties of the produced pulps are still low even at low per acid concentration and pulping temperatures. In general, low tear values are considered a big problem in acidic pulping conditions.

3.2 Effect of pulping time

Table 1 shows the effect of pulping time upon the chemical and the strength properties of bagasse pulps produced by persulphuric acid pulping. It's clear that, the degree of delignification is enhanced by increasing the period of pulping time. From Kappa number values, Table 1, one can conclude that the bulk of delignification could be achieved at moderate pulping period 180 min after which a slight decrease in Kappa

numbers has been shown.

The severe degradative effect on the mean cellulosic chain (reduction in degree of polymerization) has been noted at long period of pulping time (240 min).

The strength properties (breaking length, burst factor and tear factor) could be enhanced from short pulping time, (120 min) to moderate pulping time (180 min). This may be attributed to the improving of degree of delignification. The negative effect of pulping time has appeared at long pulping period since decreasing of the strength properties has been shown. This may be attributed to the reducing of degree of polymerization of the produced pulps.

3.3 Optimizing of bagasse pulping by using of Caro's acid

The work stated out the relation among

Table 2. Experimental matrix for 2³ full factorial experimental design

Exp. No.	Experimental Matrix														
	Factors on natural scale			Coded Factors									Kappa number	DP	Yield %
	Z ₁	Z ₂	Z ₃	X ₀	X ₁	X ₂	X ₃	X ₁ X ₂	X ₁ X ₃	X ₂ X ₃	X ₁ X ₂ X ₃	Y ₁	Y ₂	Y ₃	
1	2	40	120	+1	-1	-1	-1	+1	+1	+1	-1	41.50	775	42.60	
2	8	40	120	+1	+1	-1	-1	-1	-1	-1	+1	30.85	720	45.33	
3	2	60	120	+1	-1	+1	-1	+1	+1	-1	+1	27.63	733	47.55	
4	8	60	120	+1	+1	+1	-1	-1	-1	+1	-1	23.55	627	42.35	
5	2	40	240	+1	-1	-1	+1	-1	-1	+1	+1	36.88	746	41.25	
6	8	40	240	+1	+1	-1	+1	+1	+1	-1	-1	28.22	715	43.45	
7	2	60	240	+1	-1	+1	+1	-1	-1	-1	-1	25.35	690	46.28	
8	8	60	240	+1	+1	+1	+1	+1	+1	+1	+1	20.11	605	40.20	

the process independent variables and dependent variables of the process. The independent variables are per acid concentration Z_1 , pulping temperature Z_2 and pulping time Z_3 . The dependent variables are Kappa number Y_1 , degree of polymerization Y_2 and pulp yield Y_3 . A full factorial experimental design is one in which all possible combination of the factors at all levels involved in the experiment are used. The number of possible combinations of be performed during the experiment (N) could be determined as follows:

$$N = n^k$$

where n: number of levels

k: number of factors

The terms level of factor will refer to the boundary of the regain to be searched for a given process variable. It is usual to pass from Z_1, Z_2, \dots, Z_k coordinates to a new dimension less system coordinates X_1, X_2, \dots, X_k as shown in Table 2. In this study, $K=3$ at two levels. Thus the number of all possible combination (N) of three factors each fixed at two levels equals to 8. In the case of fuller regression equation containing two and three factor interaction coefficients.

$$Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{123}X_1X_2X_3$$

Where b_i : coefficients of the regression equation which can be determined in a way of matrix calculus.

Therefore:-

$$\begin{aligned} \text{Kappa number } (Y_1) &= 29.57 - 3X_1 - 2.25 X_2 - 2.75 X_3 + 4 X_1 X_2 + 1.25 X_2 X_3 \\ \text{DP } (Y_2) &= 768 - 41.6 X_1 - 52.2 X_2 - 40.3 X_3 - 4.6 X_1 X_2 - 0.4 X_1 X_3 - 0.9 X_2 X_3 + 1.7 X_1 X_2 X_3 \\ \text{Yield } (Y_3) &= 47.3 - 0.53 X_1 - 1.63 X_2 - 0.3 X_3 - 3.3 X_1 X_2 + 1.3 X_1 X_3 - 1.78 X_2 X_3 - 0.12 X_1 X_2 X_3 \end{aligned}$$

Thus, one can predict a certain property of bagasse pulps produced from caro's acid pulping by equation in which the interactions between the independent variables of the process are expressed.

4. Conclusions

Based on the studies conducting to date, Caro's acid pulping of annual plants appears to be very close to economic viability. In this study, The process produced bagasse pulps with moderate values of chemical and strength properties.

The deterioration of the pulp properties was manifested at higher Caro's acid used and higher pulping temperature.

The study has attempted to state out a mathematical formula to anticipate the effect of the interactions of pulping factors on the produced pulp properties.

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