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Production of Dissolving Grade Pulp from Poplar Wood by Ethanol-Water Process

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Abstract: The possibilities of dissolving grade pulp production from poplar by an ethanol-water process were investigated. The effects of ethanol ratio, cooking temperature and acid catalyst ratio on unbleached, bleached and alkali-purified pulps were studied. It was seen that catalyst ratios exceeding 0.01% caused serious yield and viscosity losses. The results showed that with the catalyst ratio to pulping liquor exceeding 0.01%, pulp yield and viscosity were reduced to an unacceptable level. The best pulping result was obtained at 40% ethanol consistency, 180 ºC pulping temperature and 150 min reaction time without the addition of acid catalyst to the pulping liquor. After bleaching and alkali purification, the pulp obtained had a 30.7% yield, 95.8% α-cellulose content, 677 cm^3/g viscosity, 83% ISO brightness, 1.80% residual pentosans, 0.33% copper number and 0.14% ash content. However, brightness was increased to 88.5% ISO level with the application of a multistage bleaching (CEHDED) sequence instead of chlorite bleaching without serious losses in yield or α-cellulose content at normal viscosity levels. It is concluded that these pulps meet dissolving grade pulp quality requirements.

Key Words: Poplar, organosolv pulping, ethanol-water pulping, dissolving pulp, alpha-cellulose

Etanol-Su Pişirme Yöntemiyle Kavak Odunundan Çözünür Hamur Üretimi

Özet: Bu çalışmada kavak odunundan etanol-su yöntemiyle selüloz türevleri endüstrisinde kullanılacak çözünür hamur üretimi imkanları araştırıldı. Esmer, ağartılmış ve alkali ile saflaştırılmış hamur özellikleri üzerine etanol oranı, pişirme sıcaklığı ve asit katalizör oranının etkisi çalışıldı. %0,01'in üzerindeki asit konsantrasyonunun hamur verimi ve viskozitesi üzerinde ciddi kayıplara neden olduğu görüldü. En iyi sonuçlar %40 etanol konsantrasyonunda, 180 ºC pişirme sıcaklığı ve 150 dakika reaksiyon süresinde pişirme çözeltisine asit katalizör ilave edilmeden elde edildi. Ağartma ve alkali saflaştırma işleminden sonra elde edilen hamurun verimi %30,7 ; alfa selüloz oranı %95,8 ; viskozitesi 667 cm^3/g ; kalıntı pentozan oranı %1,80 ; parlaklık değeri %83 ISO; bakır sayısı %0,33 ve kül oranı %1,14 olarak belirlendi. Klorit ağartması yerine geleneksel CEHDED ağartma dizini uygulandığında verim ve hamur viskozitesinde kayıp olmaksızın daha yüksek parlaklık derecesinde hamur üretilebilmiştir. Bu koşullarda elde edilen hamurun selüloz türevleri endüstrisinin kalite gereklerini karşıladığı sonucuna varıldı.

Anahtar Sözcükler: Kavak, organosolv pişirme, Etanol-su pişirmesi, Çözünür hamur, alfa-selüloz

Introduction

Pulping is done under acidic conditions at elevated temperatures in some solvent based pulping systems. Because the decomposition of hemicelluloses is predominant in these conditions, it is easy to produce dissolving grade pulp with extremely high α-cellulose content. Alcohol-water systems can also be used to produce dissolving grade pulp. Both lignin and hemicellulose degradation reactions may occur in pulping medium catalyzed by mineral acids or acidic salts.

Spruce chips can be delignified with aqueous methanol using calcium chloride as the catalyst. When the calcium chloride content of cooking liquor is 0.05%, dissolving grade pulp can be produced. Even after bleaching, 98% of the original α-cellulose can be preserved (Paszner and Chang, 1983).

According to Sabatier and co-workers (1989), the pH of the reaction medium is the most important parameter affecting the selectivity of ethanol-water pulping. Acid catalysts provoke carbohydrate hydrolysis. This leads to low pulp yields. Dissolving pulps can be produced in this method through adequate control of the pulping parameters.

In autocatalyzed alcohol-water systems, enough acidity can be ensured with releasing of acetic acid from wood by hydrolytic cleavage of acetyl groups in the hardwood's glucuronoxylan at elevated temperatures. The ethanol-water pulping procedure has been studied with regard to its suitability for the production of dissolving pulp from beech wood. Pulp of rather good quality was produced in a pilot plant. The viscose fibers processed from the pulps were comparable with those made from sulfite pulps (Peter and Hoglinger, 1986).

Koll and Lenhardt (1987) treated birch wood chips in a flow apparatus with ethanol-, 1-butanol- and ethyleneglycol-water (50/50, v/v) mixtures. Higher temperatures (225-250 ºC) can be used in batch delignification. Hemicelluloses and lignin can be separated from pulp without severe decomposition of pentosans. The pulps were delignified by more than 90% and were bleached easily. Due to the high pulping temperature, the cellulose had low DPw values and it may be used as dissolving pulp.

Organocell^{TM} (APR) red oak pulp has been produced with a kappa number of 14 and a pulp yield of 43% in autocatalyzed pulping conditions. Aspen and birch woods seemed to be easier to delignify. APR pulps can successfully replace the hardwood kraft components in printing and tissue grades. The potential of this process in producing dissolving pulp has been shown in pilot viscose production runs (Lora and Aziz, 1985).

The aim of this work was to optimize the cooking conditions of auto- and acid-catalyzed ethanol-water batch pulping system in order to produce dissolving grade pulp. Poplar wood was chosen as the raw material. The effects of ethanol concentration, catalyst ratio to wood, pulping time and temperature on some properties of pulp were determined.

Materials and Methods

Poplar (*Populus euroamericana*) wood chips were selected as the raw material in this study. Wood samples were taken from a poplar plantation in Tokat province in Turkey. Selected trees were 12 years old and had a 28-30 cm root collar diameter.

Kleinert's (1974) original pulping procedure was adapted for batch type pulping in order to obtain dissolving grade pulp at laboratory scale. Pulping was done in a 15-l capacity laboratory type rotary digester with a digital temperature controller. In order to find the optimum cooking conditions, the ethanol ratio to pulping liquor, pulping temperature, time and acid catalyst ratio to wood were changed systematically. Accordingly, 31 organosolv and 3 kraft and prehydrolyzed-kraft (PHK) control pulps were obtained. The pulping conditions are given in Table 1. After every organosolv pulping trial, each pulp was pressed to up to 25% dry-matter content followed by treatment with 8% caustic soda to oven-dry pulp mass at 10% pulp consistency and 30 min extraction time. Then, the pulp was washed with tap water to remove residual alkali and alkali-soluble lignin as well as some hemicellulose fractions.

The organosolv pulps were both delignified and bleached with an improved chlorite method. For this, 2 g of oven-dried pulp, 2 g of sodium chlorite, 0.67 g of sodium acetate, 1.33 g of acetic acid, 1 drop of formic acid and 45 ml of distilled water were put into a 100 ml flask, mixed thoroughly and left for 16 h at constant temperature of 25 ºC. Then pulps were washed with sufficient distilled water (Nelson and Irvrine, 1992).

The delignified and bleached pulps were purified with cold alkaline at room temperature using a 10% NaOH charge on the oven-dried pulp. Alphα-cellulose, pentosans, ash, copper number and brightness values of pulps were determined according to the relevant Tappi standards. Scan-cm 15:88 standard method was applied for pulp viscosity determinations.

A single kraft and a couple of PHK cooking trials were performed using poplar wood chips for comparison. Bleaching and purification were done using the procedures mentioned above. In addition, organosolv pulp obtained at optimum conditions (pulp code 9) was also bleached with a multistage (CEHDED) bleaching sequence.

Results and Discussion

Some properties of the unbleached ethanol-water, kraft and PHK pulps obtained from poplar wood are given in Table 1. It is well known fact that the peeling of acetyl groups in the o-acetyl-4-o-methyl glucuronoxylan at higher temperatures aids in decreasing the pH level of pulping liquor during the autocatalyzed ethanol-water pulping from hardwoods (Paszner and Cho, 1989). As shown in Table 1, especially at a pH below 3.5, free acetic acid acted as a catalyst and good fiber liberation occurred. Longer pulping at the maximum temperature lowered the pH of pulping liquor and the kappa number. Although the addition of acid catalyst to pulping liquor is expected to accelerate the delignification reactions, it was observed that excess acid catalyst (H_2SO_4) addition to pulping liquor caused serious viscosity and yield losses probably due to

Table 1. Pulping conditions and unbleached pulp properties of organosolv, kraft and prehydrolyzed kraft (PHK) pulps.

PULP CODE	PULPING CONDITIONS					PULP PROPERTIES (UNBLEACHED)			
	Ethanol	Pulping time	Pulping temp.	H_2SO_4 additionto to liquor	Black liquor pH	Screened yield	Reject	Total yield	Kappa No.
	(%)	(min)	(°C)	(%)		(%)	(%)	(%)	
1	40	90	180	0.00	3.40	39.17	12.9	52.07	61
2	40	90	180	0.01	2.97	33.86	5.42	39.38	27
3	40	90	180	0.02	2.06	20.72	2.89	23.61	17
4	40	90	180	0.025	1.90	18.58	2.62	21.20	15
5	40	120	180	0.00	3.02	42.06	8.62	50.68	57
6	40	120	180	0.01	2.90	36.22	2.19	38.41	21
7	40	120	180	0.02	1.97	16.36	2.01	18.37	15
8	40	120	180	0.025	1.95	12.37	2.08	14.45	16
9	40	150	180	0.00	3.06	42.47	5.30	47.77	55
10	40	150	180	0.01	2.86	35.87	1.46	37.33	19
11	40	150	180	0.02	1.90	14.27	2.28	16.55	14
12	40	150	180	0.025	1.87	12.80	1.48	14.28	14
13	50	90	180	0.00	4.10	44.49	11.02	55.51	67
14	50	90	180	0.01	3.15	40.44	3.17	43.61	29
15	50	90	180	0.02	2.80	22.96	2.80	25.76	16
16	50	90	180	0.025	2.50	15.39	2.08	17.47	13
17	50	120	180	0.00	3.17	43.01	8.20	51.21	58
18	50	120	180	0.01	2.80	32.28	2.25	34.53	26
19	50	120	180	0.02	2.40	15.03	1.42	16.45	16
20	50	120	180	0.025	1.90	9.55	2.01	11.56	15
21	50	150	180	0.00	3.14	42.82	6.69	49.51	54
22	50	150	180	0.01	2.76	28.36	1.20	29.56	22
23	50	150	180	0.02	1.92	12.80	1.90	14.70	13
24	50	150	180	0.025	1.78	6.55	2.10	8.65	13
25	50	90	170	0.01	3.80	29.29	28.42	57.71	63
26	50	90	170	0.02	2.60	22.93	3.09	26.02	20
27	50	120	170	0.01	3.73	32.77	3.74	35.51	58
28	50	120	170	0.02	2.60	21.61	2.27	23.88	17
29	50	150	170	0.01	3.67	28.94	2.77	29.71	52
30	50	150	170	0.02	2.53	15.11	1.13	16.24	13
31	40	150	180	0.00	3.06	42.47	5.30	47.77	55
K	-	60	170	-	13.06	49.66	0.01	49.67	22
PHK1	-	60	170	-	12.76	34.50	0.00	34.50	7
PHK2	-	60	170	-	12.45	33.22	0.00	33.20	8

K: Kraft pulp (active alkali:16%, sulphidity: 20%), PHK1 and PHK2: kraft pulps prehydrolyzed with hot water (170 °C) and mineral acid (0.25% HCl at 140 °C), respectively.

the acceleration of polysaccharide degradation reactions at low pH levels. The hydrolytic degradation reactions of carbohydrates were quite fast when more than 0.01% mineral acid was added to the pulping liquor. It can be noted that the peeling off of acetyl groups at higher temperatures and the prolonged cooking time might lower the pH level.

There is a good relationship between the end pH of black liquor and viscosity, yield and kappa number of unbleached poplar ethanol-water pulp (Figures 1-3).

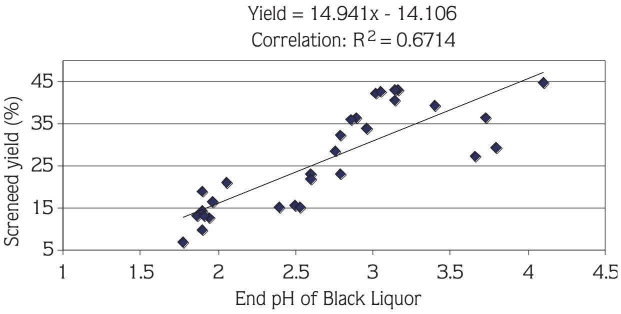

Figure 1. Correlation between end pH of black liquor and yield.

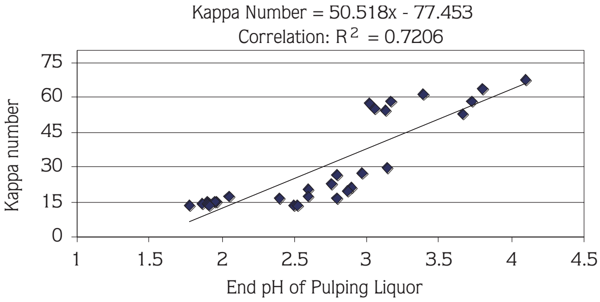

Figure 2. Correlation between end pH of black liquor and kappa number.

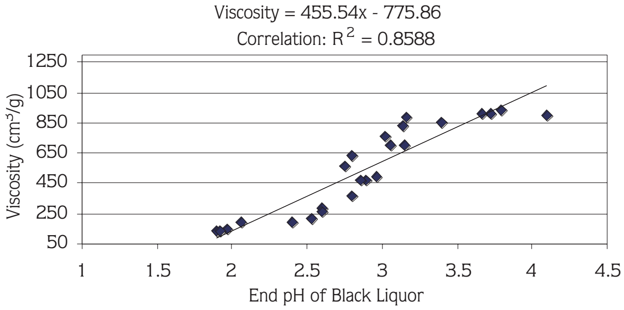

Figure 3. Correlation between end pH of black liquor and pulp viscosity.

Figure 1 indicates that the correlation between the yield and end pH of black liquor is linear and statistically significant (R^2 = 0.6714). It was understood that hydrolytic degradation and cleavage reactions were dominant and excessive yield losses took place at lower pH levels.

The effects of end pH values on the kappa number are shown in Figure 2. The relationship between the end pH and the kappa number of pulp is linear and statistically significant (R^2 = 0.7296). It is clear that the decreasing pH of pulping liquor with increasing acid catalyst ratio and the prolonging of pulping time accelerated both delignification and carbohydrate dissolution.

There was also a statistically significant relation between end pH and pulp viscosity. As a result, R^2 was calculated as 0.8588. It is possible that acidic hydrolysis in acidic medium breaks off the cellulose molecule chains.

Some properties of the pulps bleached by chlorite and purified by cold alkali extraction are given in Table 2. After chlorite delignification, the yield and α-cellulose content of autocatalyzed poplar organosolv pulp varied from 34 to 37% and from 87 to 90%, respectively. Alpha-cellulose contents were 96.7% and 97.0% in eucalyptus PHK pulp (Rabinovich, 1986) and beech PHK pulp (Kerr and Harwood, 1976), respectively. Although the α-cellulose contents of the pulps were consistent with the specifications given in the literature for viscose based film and artificial fiber applications and filler for plastics (Hinck et al., 1985), they must undergo cold alkali purification at the preparation of other cellulose derivatives such as cellulose acetate, cellulose nitrate and cellulose ethers, but an α-cellulose ratio of pulp above 95% can be achieved with cold alkali extraction to oven-dried pulp using 10% alkali. Indeed, α-cellulose content must be over 96% for production of such cellulose derivatives (Masura, 1987; Kosaya and Kosheleva, 1976).

After cold alkali extraction, residual pentosans in the pulp were reduced to below 3.5%. Copper numbers and ash ratios were 0.28-0.33% and 0.13-0.18% respectively. Residual pentosans and ash in a dissolving pulp are considered contaminants for the preparation of cellulose derivatives, but these values were below the recommended limits for highly pure sulfite- and hardwood PHK-based dissolving grade pulps (Hinck et al., 1985; Sixta et al., 1999).

Table 2. Bleached and purified pulp properties of organosolv, kraft and prehydrolyzed kraft (PHK) pulps.

PULP CODE	BLEACHED PULP PROPERTIES				ALKALI-PURIFIED PULP PROPERTIES						
	Yield	Viscosity	Alpha-cellulose	Bright-ness	Yield	Viscosity	Alpha-cellulose	Pentosans	Brightness	Copper no.	Ash
	(%)	(cm^3/g)	(%)	(%ISO)	(%)	(cm^3/g)	(%)	(%)	(%ISO)	(%)	(%)
1	33.74	844	87.73	70.71	30.20	714	93.42	3.65	74.63	0.29	0.16
2	31.41	490	88.39	77.61	28.15	460	94.23	2.90	79.67	0.39	0.18
3	19.29	189	79.78	80.80	16.59	177	84.50	1.80	83.41	0.62	0.17
4	--	--	--	--	--	-	--	--	--	-	-
5	36.67	755	88.08	71.85	31.20	677	94.84	2.50	77.54	0.30	0.15
6	33.35	428	88.46	79.42	30.97	410	93.47	2.40	81.56	0.40	0.15
7	15.45	146	74.56	81.49	12.16	136	80.15	1.78	86.07	0.73	0.13
8	--	--	--	--	--	-	--	-	--	-	-
9	37.16	701	90.11	71.92	30.74	677	95.67	1.80	82.87	0.33	0.14
10	32.39	471	88.37	79.81	27.63	365	92.88	1.78	84.06	0.43	0.16
11	13.55	126	72.25	81.69	9.67	117	76.32	1.62	86.56	1.03	0.14
12	--	--	--	--	--	-	--	-	--	-	-
13	37.26	892	86.97	65.83	30.42	675	94.47	2.70	73.46	0.28	0.13
14	36.32	696	87.11	74.89	33.03	514	94.32	2.28	79.28	0.31	0.18
15	21.39	364	83.43	75.68	18.11	304	89.44	1.80	83.12	0.59	0.14
16	--	--	--	--		-	--	-	--	-	-
17	37.12	889	87.46	68.87	31.19	612	94.58	2.23	74.15	0.29	0.16
18	29.75	555	87.53	78.61	25.50	491	93.43	2.11	81.32	0.41	0.15
19	13.64	192	81.23	80.33	11.45	185	82.62	1.68	85.96	0.84	0.14
20	--	--	--	--	--	-	--	-	--	-	-
21	37.31	825	87.92	68.98	32.91	519	94.90	1.83	76.71	0.30	0.14
22	26.29	561	84.70	79.13	22.20	306	93.31	1.79	81.47	0.58	0.18
23	12.06	131	74.02	80.67	9.05	135	81.17	1.56	86.36	0.85	0.11
24	--	--	--	--	--	-	--	-	--	-	-
25	25.98	935	84.90	66.36	22.36	575	94.58	3.88	71.36	0.14	0.13
26	21.34	279	85.00	70.96	18.82	272	90.00	2.35	82.28	0.49	0.17
27	30.19	912	85.60	66.75	27.57	472	94.06	3.59	72.22	0.28	0.15
28	19.63	263	84.43	71.75	18.01	231	86.24	1.96	85.35	0.54	0.15
29	26.53	907	84.00	67.00	23.59	344	91.67	3.12	73.94	0.70	0.17
30	13.65	207	80.14	75.95	12.75	181	84.84	1.74	85.46	0.76	0.18
31*	34.68	683	90.66	87.15	31.52	698	95.01	2.67	88.45	0.36	0.11
K	46.34	996	86.92	72.58	44.80	930	88.98	9.83	75.88	0.34	0.10
PHK1	31.81	738	94.96	79.15	30.78	616	96.42	3.37	82.65	0.40	0.07
PHK2	31.27	837	95.14	78.22	29.30	741	96.12	3.74	80.49	0.41	0.07

* Bleached with CEHDED sequence

Acid catalyst addition of up to 0.01% to pulping liquor made the best viscosity control. Yield losses were only 2-4% after the 0.01% acid addition, but serious yield losses, lower viscosity levels and decreases in the α-cellulose content were observed with a sulfuric acid ratio over 0.01%.

Brightness was generally low (74-82% ISO) after chlorite delignification (Table 2). However, brightness could increase to 88.5% ISO level (pulp code 31) with the application of multistage bleaching (CEHDED) without serious losses of yield or α-cellulose content at normal viscosity levels. The brightness of poplar EW pulps

Table 3. Dissolving grade pulp properties obtained poplar and other raw materials.

Raw-materials/Pulping Method	Yield	Viscosity (%)	α-cellulose (cm^3/g)	Pentosans (%)	Copper no. (%)	Ash (%)	Brightness (%)	References (%ISO)
Linters-Kraft	-		98±0.5	-	0.4	0.05	85±1	Olsen, 1938
Eucalyptus-PHK	-	604	96.7	1.4	0.4	-	92.5	Rabinovich, 1986
Beech-PHK	34.0	475	97.0	-	-	-	88.2	Kerr and Harwood, 1976
Poplar-PHK1	29.3	741	96.1	3.0	0.41	0.10	80.5	Determined
Poplar-PHK2	30.8	616	96.4	3.4	0.40	0.07	82.7	Determined
Poplar ethanol-water (bleached with chlorite)	30.7	677	95.7	1.8	0.33	0.11	82.9	Determined
Poplar ethanol-water (bleached with CEHDED)	31.5	698	95.01	2.67	0.36	0.11	88.5	Determined
Poplar Kraft	44.8	930	88.90	9.83	0.34	0.10	75.9	Determined

bleached by a CEHDED sequence is comparable to beech (Kerr and Goring, 1976) and eucalyptus PHK pulps (Rabinovich, 1986).

Accordingly, the optimum cooking conditions of poplar organosolv pulp with respect to dissolving grade pulp properties were as follows: ethanol concentration in pulping liquor: 40%; maximum pulping temperature: 180 ºC; pulping time: 150 min; and liquor to wood ratio: 8 to 1. After bleaching and alkali purification, pulp was obtained in 30.7% yield with a 95.8% α-cellulose content at 677 cm^3/g viscosity level, brightness of 83% and copper number of 0.33% and 0.14% ash. The properties of pulp bleached with a CEHDED bleaching sequence and purified with cold alkali extraction were similar, with the exception of the brightness value of 88.5% ISO.

Table 3 compares the properties of ethanol-water, PHK and conventional kraft pulps obtained from poplar wood with dissolving grade industrial pulps obtained in earlier reports by Olsen (1938), Rabinovich (1986) and Kerr and Harwood (1976). Viscosity, α-cellulose content, Copper number and ash content of the organosolv poplar pulp were comparable with those of the hardwood based commercial dissolving grade pulps and poplar PHK pulps. Similarly, the residual pentosans of these pulps were lower than those of poplar PHK pulps. Pulp yield, on the other hand, was similar to that of poplar PHK pulp, but lower than that of eucalyptus PHK pulp. It is not possible to produce a dissolving grade pulp from paper grade kraft pulp without applying a prehydrolysis stage before pulping (see pulping code K in Table 2), probably because of the conversion of some hemicellulose fractions to an alkali-insoluble form.

Conclusions

The characteristics of dissolving grade poplar organosolv pulp were comparable to those of poplar and other hardwood PHK pulps. Therefore, bleached ethanol-water poplar pulp is suitable for producing viscose grade dissolving pulp, but cold alkali purification of the pulp seemed to be necessary for preparing other cellulose derivatives.

It is possible to control pulp viscosity and accelerate the delignification reactions by adding inorganic acid to pulping liquor. However, exceeding charges over 0.01% are not adequate for this purpose due to serious yield, α-cellulose and viscosity losses.

The potential drawbacks of kraft pulping related to bad-odor problems, sulfur usage and bleaching problems could be eliminated considerably by applying ethanol-water pulping processes.

The recovery of solvent (alcohol) from black liquor is easy and effective when compared with the volatilization capabilities of water from the black liquor in the multiply effect evaporator in a kraft recovery system. A simple distillation unit is sufficient for alcohol recovery. It is not necessary to concentrate and burn the black liquor as in the kraft process. Lignin is precipitated and separated from black liquor after distillation. The remaining liquor consists of sugar originated hemicelluloses and cellulose degradation, and it is easy to convert them to commercially valuable by-products such as alcohol, furfural and glycerol. Thus renewable resources can be utilized more efficiently.

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