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The Investigations on Pulp and Paper Production with Modified Kraft Pulping Method from Canola (*Brassica napus* L.) Stalks

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Abstract

Aim of study: This study was carried out to evaluate canola (*Brassica napus* L.) stalks as an alternative raw material for pulp and paper production.

Area of study: This work was carried out in Kahramanmaraş Sütçü Imam University, Faculty of Forestry, Pulp and Paper Production Laboratory.

Material and Methods: Before pulping procedure, the chemical components of the canola stalks were investigated. The holocellulose, alpha cellulose, lignin, and ash contents and NaOH (1%) and cold-hot water solubilities were determined. Modified kraft cooking method was used for pulping from canola stalk by using sodium borohydride ($NaBH_4$). Sulfidity charge; 26, 28, 30%, and $NaBH_4$ charge; 0, 0.1, 0.3, 0.5% were changed while keeping constant temperature (150 °C), time (120 min) and alkali charge (20%). For determination of the optimum pulping condition, 12 cooking experiments were performed to canola stalks. In addition, the physical and optical properties of the papers produced from the obtained pulps were also determined.

Main results: The holocellulose (72.10%) and lignin (13.15%) contents of the canola stalks were found to be similar with hardwoods. The optimum cooking condition was determined as 20% active alkali, 28% sulfidity and 0.5% $NaBH_4$ charges (as NaOH) and the total pulp yield were obtained about 44.7% in this condition. Besides, the addition of $NaBH_4$ increased pulp yield by 24.1% and decreased kappa number by 9.7%.

Research highlights: It was concluded that obtained results demonstrate the suitability of canola stalk for pulp and paper production and $NaBH_4$ is an effective cooking additive for the pulp yield.

Keywords: *Brassica napus* L., canola stalk, $NaBH_4$, Kraft pulping

Kanola Saplarından (*Brassica napus* L.) Modifiye Kraft Yöntemi ile Kağıt Hamuru ve Kağıt Üretimi Üzerine Araştırmalar

Öz

Çalışmanın amacı: Bu çalışma, kanola (*Brassica napus* L.) saplarının kağıt hamuru ve kağıt üretiminde alternatif bir hammadde olarak değerlendirilmesini amaçlamıştır.

Çalışmanın alanı: Bu çalışma Kahramanmaraş Sütçü İmam Üniversitesi, Orman Fakültesi, Kağıt Hamuru ve Kağıt Üretim Laboratuvarı'nda yürütülmüştür.

Materyal ve Yöntem: Kağıt hamuru üretimine geçilmeden önce kanola saplarının kimyasal içeriği araştırılmıştır. Holoselüloz, alfa selüloz, lignin ve kül içerikleri ile NaOH (%1), soğuk ve sıcak su çözünürlükleri belirlenmiştir. Kanola saplarından kağıt hamuru üretimi için $NaBH_4$ ilaveli modifiye kraft yöntemi kullanılmıştır. Sülfidite %26, 28 ve 30, $NaBH_4$; %0, 0.1, 0.3, ve 0.5 oranlarında değiştirilirken aktif alkali oranı %20 olarak sabit tutulmuştur. Optimum koşulu belirlemek için 12 farklı koşulda pişirme işlemi yapılmıştır. Ayrıca elde edilen kağıt hamurlarından üretilen kağıtların fiziksel ve optik özellikleri de belirlenmiştir.

Sonuçlar: Kanola saplarının holoselüloz (%72.10) ve lignin (%13.15) içeriği yapraklı ağaç odunları ile benzer oranlarda bulunmuştur. Optimum pişirme, %20 aktif alkali, %28 sülfidite ve %0.5 $NaBH_4$ oranlarının kullanıldığı koşulda elde edilmiş ve bu koşulda kağıt hamuru toplam verimi %44.7 olarak bulunmuştur. Aynı zamanda $NaBH_4$ ilavesi ile toplam verim %24.1 oranında artarken kappa numarası %9.7 oranında azalmıştır.

Araştırma Vurguları: Elde edilen sonuçlar, kanola saplarının kağıt hamuru ve kağıt üretimi için uygunluğunu ve $NaBH_4$'ün hamur verimliliği için etkili bir pişirme katkı maddesi olduğunu ortaya koymuştur.

Anahtar kelimeler: *Brassica napus* L., kanola sapları, $NaBH_4$, Kraft pişirmesi

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Introduction

Cellulose is one of the most abundant biopolymers in the world. It is found in wood, cotton and other lignocellulosic materials such as agricultural wastes and annual plants (Moon, Martini, Nairn, Simonsenf & Youngblood, 2011; Gunduz, Asik, Aydemir & Kilic, 2015). Wood raw material is generally used as a cellulose source in pulp and paper production. However, difficulties have arisen in the procurement of wood raw materials due to the laws on the protection of forest resources. Difficulties of supplying sources for pulp and paper production have given rise papermakers to seek new sources. Some researchers have been done to discover new raw materials. As a result of these investigations, it has been reported that annual plants and agricultural wastes can be evaluated in pulp and paper productions as substitute to wood. The use of annual plants for pulp and paper production has begun in the 19th century. Paper consumption has increased with population growth and the advancement of technology and therefore the use of annual plants in pulp and paper production also increased rapidly after the Second World War (Eroglu and Deniz, 1987; Rowell, Young & Rowell, 1997; Tutus 2000; Gencer, Eroglu & Deniz, 2005; Akgul, 2007; Tutus and Cicekler, 2016).

The evaluation of agricultural wastes in pulp and paper industry provides both economic and environmental benefits. The manufacturing of pulps from non-wood materials has increased and nowadays some non-wood fiber sources are commercially handled to produce pulp and paper in some countries such as Turkey, Africa, India and China. In 2016, there are approximately 82 million tons of agricultural wastes in Turkey (Faostat, 2018). Wheat and rice stalks are main agricultural wastes used in pulp and paper production. In addition to these, sorghum, jute and hemp stalks are also used as raw materials in Turkey and world (Rousu, Rousis & Anttila, 2002; Ashori, 2006; SKSV, 2018). Plentiful studies have also been carried out on new raw material as a fiber source in pulp and paper industry (Sarwar, Nasima & Khalidul, 2006; Shatalov and Pereira, 2006; Tran, 2006; Wanrosli, Zainuddin, Lawb & Asro, 2007; Tutus, Cicekler, Ozdemir & Altas, 2014; Tutus, Kazaskeroglu & Cicekler, 2015;).

Canola (*Brassica napus* L.) is one of the most important industrial plants in many countries of the world. With the begin of the Second World War in 1940s, canola production increased and nowadays it is the oil seed plant with the fastest growth rate (Guler, Kara & Dok, 2005). Annual canola production in the world is around 170 million tons. It was approximately 12.000 tons of canola production in Turkey. After the canola seeds are collected, approximately 20% of the stalks remain in the field (Banuelos, Bryla & Cook, 2002; Enayati, Hamzeh, Mirshokraie & Molaii, 2009; Tiras, 2009). According to this calculation, approximately 34 million tonnes of canola stalks per year remain in fields. It was concluded that this amount demonstrates the suitability of canola stalk in numerous products.

The kraft method is the most used method for obtaining pulp suitable for papermaking. Increasing the pulp yield in pulping is very important to the pulp and paper industries. Modifying kraft cooking, such as with sodium borohydride ($NaBH_4$) addition, is one way to increase pulp yield (Courchene, 1998; Tutus and Eroglu, 2003; Tutus and Eroglu, 2004; Tutus and Usta 2004; Hafizoglu and Deniz, 2007; Istek and Gunteki, 2009).

In many countries, especially in Turkey, canola stalks are burned for heating or energy production. There is little information on the utilization of these raw materials in paper industry. The aim of this study was to evaluate canola stalks for pulp and paper production, using Kraft and Kraft-$NaBH_4$ cooking methods.

Material and Method

Material

Canola stalks were taken from Karacabey Agricultural Management Directorate (Bursa). Chemicals used in this study were supplied by Merck and Sigma. Chemical, optical and physical properties of the canola pulps and raw material preparation were investigated according to relevant standards.

Chemical Analysis

The canola stalks were cut into the length of 2-4 cm, and grained to 60 mesh fractions in a willey mill. Prepared materials were used to investigate its chemical components according to standards given in below;

Holocellulose content: Wise chloride method (Wise and Murphy, 1962).

Lignin content: TAPPI T222 om-11

Alpha cellulose content: TAPPI T203 cm-09

Ash content: TAPPI T211 om-02

Alcohol-Benzene solubility: TAPPI T204 cm-07

Cold-Hot Water solubility: TAPPI T207 om-88

1% NaOH solubility: TAPPI T212 om-12

Three replicates were performed for each treatment and mean values were used.

Pulp and Paper Production

Total of 12 cooking experiments (3 control and 9 $NaBH_4$ added) were performed to determine optimum pulping conditions and effects of $NaBH_4$ on pulp properties. Sulfidity charge; 26, 28, 30%, and $NaBH_4$ charge; 0, 0.1, 0.3, 0.5% were changed while keeping constant temperature (150 °C), time (120 min) and alkali charge (20%). The pulping conditions performed canola stalks were given in Table 1.

Table 1. Pulping conditions for canola stalks

Pulping conditions	Unit	Value
Active Alkali charge	% (as NaOH)	20
Sulfidity charge	% (as NaOH)	26, 28, 30
$NaBH_4$ charge	% (as NaOH)	0, 0.1, 0.3, 0.5
Cooking temperature	°C	150
Cooking time	Min	120
Cooking liquor/stalk	L/kg	5/1

The cooking trials were carried out in rotary digester and governed with digital temperature control system and electrically heated. Obtained pulps were washed with tap water to remove black liquor and disintegrated. Then, the pulps were screened on a 0.15 mm with a Somerville Screen. Viscosity and kappa values of all pulps were also determined according to TAPPI T230 om-08 and TAPPI T236 om-13 standards, respectively.

The pulps were beaten to 50±5 °SR (Schopper-Riegler) freeness level in a hollander beater and ten test papers with grammages of 70 $g.m^{-2}$ were produced with using a Rapid-Kothen sheet former according to ISO 5269-2. The physical and optical properties of the papers were also determined according to relevant standards given in below;

Breaking length (km): TAPPI T494 om-06

Burst index ($kPa.m^2.g^{-1}$): TAPPI T403 om-15

Tear index ($mN.m^2.g^{-1}$): TAPPI T414 om-88

Brightness (% ISO): ISO 2470-1.

Statistical Analysis

Statistical analysis of the obtained data was performed with the SPSS statistical package. Data of chemical, physical and optical properties of the pulp were analyzed using a computerized statistical program to determine variance, and by applying the Duncan test at a $P \leq 0.05$ confidence level.

Results and Discussion

Chemical Components of Canola Stalks

The chemical components of canola stalks were presented in Table 2 with those of some other annual plants.

Table 2. Chemical components of canola stalks and some annual plants

Chemical Components	Current Study	*A. membranaceus* (Tutus et al., 2014)	*Papaver somniferum* (Tutus et al., 2011)	Cotton stalks (Tutus et al., 2010)	Wheat stalk (Tutus 2000)	Corn stalk (Eroglu et al., 1992)	*Phyllotachy bambusoides* (Deniz Et al., 2017)
Holocellulose (%)	72.10	77	80	76	77	65	70.5
Alpha cellulose (%)	42.55	50	52	40	40	36	43.3
Lignin (%)	13.15	24	19	18	18	17	24.5
Ash (%)	8.20	5.50	4.70	2.50	7.10	7.50	1.35
Extractives (%)	8.50	5.60	-	6.10	5.50	9.50	3.94
%1 NaOH (%)	40.4	29.4	30.3	30.9	40.9	47.1	25.1
Hot water (%)	16.4	8.40	10.4	14.2	12.2	14.8	6.47
Cold water (%)	13.6	7.20	5.10	11.7	7.65	-	-

According to Table 2, canola stalks contain 72.10% holocellulose, 42.55% alpha cellulose, 13.15% lignin and 8.2% ash. The main components such as holocellulose and lignin of the canola stalks were comparable with previous studies (Eroglu, usta & Kırcı 1992; Eroglu and Deniz, 1998; Tutus, 2000; Tutus, Ezici & Ates, 2010; Tutus et al., 2011; Tutus et al., 2014; Kiaei et al., 2014). The holocellulose content of canola stalks were found to be lower than other annual plants due to lower hemicellulose content (Tutus and Cicekler, 2016). While the holocellulose content of canola stalks was similar with that of softwood and hardwood (Kirci, 2006), the lignin content was found to be lower. Another notable property of canola stalks is 1% NaOH solubility. As a result of alkali-soluble matters and low molecular weight carbohydrates, the NaOH (1%) solubility is higher (Tutus and Eroglu 2003). This solubility indicates fiber degradation during cooking trials (Zawawi, Mohd, Angzzas, Halizah & Ashuvila, 2014). In addition, substance soluble in tannins, resins, water, polyphenols, and fragments of lignin and hemicelluloses can leach into alkaline solutions (Potucek, Gurung & Hajkova, 2014). In a study in which the chemical components of canola were determined, ash, lignin, alpha cellulose, and holocellulose contents were found as 8.2%, 17.3%, 42%, and 73.6%, respectively (Enayati et al., 2009). According to these results, canola stalks were consistent with literature (Ekhtera, Azadfallah, Bahramin & Rovshandeh, 2009; Enayati et al., 2009; Mohammad, Enayati, Hamzeh & Roustaei, 2010; Hosseinpour and Latibati, 2012).

Pulp and Paper Properties

Yields, viscosities and kappa numbers, optical and physical properties of the pulps obtained from canola stalks were given in Table 3.

According to Table 3, the best applicable charges were determined as 28%, 20%, and 0.5% for sulfidity, active alkali, and $NaBH_4$, respectively (11th trial). The total yield and kappa number of the pulps obtained with 11th trial were found as 44.69% and 33.82, respectively. It is clearly seen that $NaBH_4$ has significant effect on pulp yield when compared with 2nd trial (Fig. 1).

Table 3. Yields, kappa numbers, viscosities, physical and optical properties of canola pulps

C. No	Active alkali (%)	Sulfidity (%)	$NaBH_4$ (%)	Total yield (%)	Kappa No
1	20	26	0	34.17	42.10
2	20	28	0	36.01	37.47
3	20	30	0	40.88	39.72
4	20	26	0.1	41.62	40.06
5	20	28	0.1	34.46	33.79
6	20	30	0.1	38.48	34.91
7	20	26	0.3	36.84	47.86
8	20	28	0.3	36.59	33.95
9	20	30	0.3	40.15	35.73
10	20	26	0.5	43.14	34.19
11	20	28	0.5	44.69	33.82
12	20	30	0.5	42.05	39.33
C. No	Viscosity ($ml.g^{-1}$)	Breaking Length (km)	Tear index ($mN.m^2.g^{-1}$)	Burst index ($kPa.m^2.g^{-1}$)	Brightness (%ISO)
1	1060	3.77	2.98	1.90	18.98
2	1008	3.73	2.95	1.83	19.09
3	1075	3.89	2.37	1.73	18.85
4	1005	3.66	1.28	1.48	20.93
5	1168	3.13	1.93	1.38	21.89
6	1098	3.90	1.61	1.50	20.52
7	1110	4.04	1.76	1.69	21.47
8	1106	3.39	1.56	1.39	20.91
9	1077	3.23	1.35	1.35	20.27
10	1141	3.13	1.62	1.42	22.74
11	1079	2.80	1.52	1.09	21.69
12	1042	3.68	1.09	1.57	20.52

The pulp yield, kappa number and viscosity value of canola stalks were presented in Table 4 with those of some other annual plants.

Table 4. Pulp properties of canola stalks and some annual plants

Literature	Cooking Method	Total Yield (%)	Kappa Number	Viscosity ($ml.g^{-1}$)
Canola stalks (current study)	Kraft-$NaBH_4$	44.69	33.82	1079
Cotton stalks (Tutus et al., 2010)	Kraft-$NaBH_4$	44.30	33.42	1045
Wheat stalk (Tutus, 2000)	Soda	50.46	18.10	686
Hordeum vulgare (Pahkala, 2001)	Soda-AQ	48.30	19.90	-
Avena sativa (Pahkala, 2001)	Soda-AQ	42.30	14.40	1180
Secale cereale (Pahkala, 2001)	Soda-AQ	48.20	12.50	1100

Table 4 indicates that the pulp properties obtained from the canola stalks were in accordance with the literature.

The end groups of carbohydrates are protected from peeling reactions by using $NaBH_4$ as a catalyst in cooking processes (Istek and Ozkan 2008; Tutus & Cicekler, 2016). Since $NaBH_4$ reduces the peeling reactions during cooking, the yield has increased. With using $NaBH_4$ in cooking liquor, the total yield has increased about 8.7% and kappa numbers and viscosity values have decreased. Besides, screen reject has also increased from 1.15% to 2.03%.

Kappa number indicates the pulp bleachability and it is generally used to determination lignin content of pulp. The kappa number decreased by approximately 9.7% with the addition of $NaBH_4$ to the cooking liquor when compared with 2^{nd} and 11^{th} trials. These decreases in kappa number indicate that the lignin content of the pulps is low and it can be easily bleached. Hart and

Connell (2006) reported that bleachability tends to decrease as the kappa number increases.

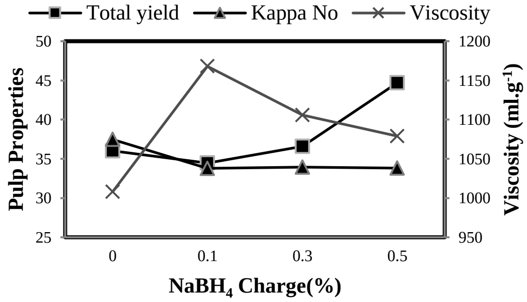

Figure 1. Effects of $NaBH_4$ on canola pulp properties (sulfidity: 28%, active alkali: 20%)

Viscosity is generally used to investigate degree of chemical damage to fibers or degree of polymerization of cellulose chain in fibers. In this study, viscosity value of the pulp obtained 11th trial was higher than that of 2nd trial about 6.6%.

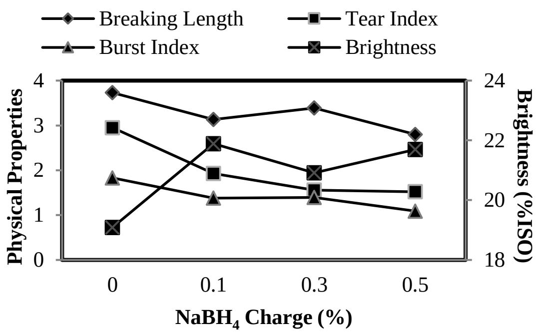

Figure 2. Effects of $NaBH_4$ on physical and optical properties (sulfidity: 30%, active alkali: 20%)

In addition, $NaBH_4$ is also used in pulp bleaching as a bleaching agent (Tutus & Usta, 2004; Okan et al., 2013). The brightness value of the pulp obtained by adding $NaBH_4$ to the cooking liquor has increased about 13.6% (2nd and 11th trails). In a study, Akgul and Temiz (2006) have used $NaBH_4$ in pulp production from Uludag Fir. They found that the brightness values of pulps have increased by using $NaBH_4$. However, the physical properties of the papers have decreased (Fig. 2). Many studies have reported that $NaBH_4$ is used to improve the bleaching performance and obtain high-brightness pulps (Sjöström and Eriksson, 1968; Ni, Sommerville & van Heiningen, 2001; Wang, Li, Ni & Zhang, 2004; Dang, Elder & Regauskas, 2007; Saracbasi, Sahin & Karademir, 2016).

To clearly understand the effect of $NaBH_4$ on the pulp and paper properties, the sulfidity charge (28%) and the active alkali charge (20%) were kept constant and the $NaBH_4$ charge was changed to 0, 0.1, 0.3 and 0.5. Then, variance analysis and Duncan test were performed on obtained data and presented in Table 5.

Table 5. Effects of $NaBH_4$ on pulp and paper properties

C. No	$NaBH_4$ (%)	Total yield (%)	Kappa No	Viscosity ($ml.g^{-1}$)
2	0	36.01b	37.47b	1008c
5	0.1	34.46c	33.79a	1168a
8	0.3	36.59b	33.95a	1106b
11	0.5	44.69a	33.82a	1079a
Sig.	-	.023	.047	.004
C. No	Breaking Length (km)	Tear index ($mN.m^2.g^{-1}$)	Burst index ($kPa.m^2.g^{-1}$)	Brightness (%ISO)
2	3.73a	2.95a	1.83a	19.09b
5	3.13b	1.93b	1.38b	21.89a
8	3.39a	1.56c	1.39b	20.91b
11	2.80b	1.52c	1.09c	21.69a
Sig.	.044	.024	.016	.031

According to the results of the variance analysis, it is seen in Table 5 that the $NaBH_4$. charge has a significant effect on all pulp and paper properties. Mean values with the same lower-case letters are not significantly different at 95% confidence level according to Duncan's mean separation test. it can easily be understood that the pulp and paper characteristics, especially total yield and brightness values were changed by the addition of $NaBH_4$ to the cooking liquor.

Conclusions

The paper industry is in a serious strait due to the lack of wood raw materials. Therefore, the evaluation of agricultural wastes as an important cellulose sources will play an important role for solving this problem. As a result of this study, it has been determined that canola stalks can be used as an alternative non-wood raw material for pulp and paper industry. The holocellulose and alpha cellulose content of the canola stalks is within acceptable limit for pulp and paper production.

Many methods are being developed to increase the yield in pulp production. In this study, $NaBH_4$ was found to be effective on the yield by adding it to the cooking solution. It increased the pulp yield about 24.1% (2nd and 11th trials). Besides, it also improved the pulp brightness value about 13.6%. Parallel to this, it also reduced the kappa number and facilitated the bleaching of pulp.

Evaluation of generally burned or disposed canola stalks in pulp and paper production would be an important contribution to the country economy such as Turkey, where wood resources are limited.

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