Bilge International Journal of Science and Technology Research

ISSN: 2651-401X
e-ISSN: 2651-4028
Research Article

2022, Volume: 6, Issue: 1, 29-37
Received: 22.12.2021; Accepted: 01.03.2022
DOI: 10.30516/bilgesci.1038727

Characterization of Chickpea (*Cicer arietinum*) Stalk Pulp and Evaluation in Paper Production

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Abstract: In this study, the evaluability of chickpea stalks generated after harvest in chickpea production was investigated in pulp and paper production. Also, paper production was carried out by blending the chickpea stalk fibers with primary and secondary fibers in certain proportions and the effects of chickpea stalk fibers on the paper properties were determined. The modified kraft method was used in the pulping of chickpea stalks and anthraquinone (AQ) was added to the cooking liquor as a catalyst. Some chemical, mechanical and optical properties of the pulps produced with the addition of different AQ charges were compared and the 0.7% AQ added cooking experiment gave the best results. The yield and viscosity values of the chickpea stalk pulps increased by 12.6% and 34.2%, respectively and the kappa number decreased by 46.7% with the addition of 0.7% AQ to cooking liquor. Paper production was carried out by blending the fibers obtained from an optimum cooking condition with primary and secondary fibers at certain rates and the effects of chickpea stalk fibers on the paper properties were examined. Depending on the amount of chickpea stalk fibers, the mechanical properties of the papers produced with secondary fibers and the optical properties of the papers produced with primary fibers improved. The strength losses that occur during the recycling of waste paper could be reduced by blending the chickpea stalk fibers with secondary fibers and it is possible to produce various paper types such as writing-printing paper by blending the short fiber chickpea stalk fibers and long fibers.

Keywords: Chickpea stalk, pulp, paper, anthraquinone, primary fiber, secondary fiber.

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Citation: Çiçekler, M., Özdemir, A., Tutuş, A. (2022). Characterization of Chickpea (*Cicer arietinum*) Stalk Pulp and Evaluation in Paper Production. Bilge International Journal of Science and Technology Research, 6(1): 29-37.

1. INTRODUCTION

Cellulose is one of the most available polymers on the planet. Wood, cotton, and other lignocellulosic materials like agricultural waste and annual plants contain cellulose (Akgül et al., 2018; Gündüz et al., 2014; Moon et al., 2011). Natural cellulose fibers retain their strength even when wet. It is the combination of these properties, as well as strength and flexibility, which distinguishes cellulose as a valuable material for paper manufacturing. By far, the most common source of papermaking fiber is wood (Yaşar et al., 2017). Wood is primarily made up of fibers with only a few nonfibrous elements such as pith and parenchyma cells. However, due to forest resource protection laws, difficulties in obtaining wood raw materials have arisen. Many organizations advocate the agricultural wastes and residuals used as cellulose sources in the paper industry, owing to rising environmental concerns (Ateş et al., 2014; Camarero et al., 2004; Güler and Beram, 2018).

Agricultural waste is unwanted or non-sellable material that is entirely produced from agricultural activities which are directly related to the cultivation or breeding of animals with the main purpose of generating profits or livelihoods (Dai et al., 2018; Viets, 1975). Agricultural wastes, whose annual production amounts are quite high, are one of the most abundant renewable resources in the world (Bian et al., 2019; Jordan et al., 2021; Mancera et al., 2012). The reuse of agricultural wastes has gained great importance in terms of both environmental and commercial aspects. In terms of the environment, leaving agricultural wastes to rot or burned on the land causes soil, water, and air pollution. It is economically important to transform agricultural wastes into products with high added value such as pulp, paper, fiberboard, particleboard and composites. It is important to note that in most cases, non-wood raw materials have a low density, a porous structure, and a low lignin content, which requires less energy and chemicals for fiber separation during the pulping process. Due to the restrictions on non-reusable resources, the demand for renewable materials, especially cellulose-based, has been increasing (Hapani et al., 2020; Mohanty et al., 2005).

Chickpea is the world's second most significant legume and is cultivated in at least 33 countries in South Asia, East Africa, West Asia, North Africa, Australia and Southern Europe (Singh, 1997). In 2019, approximately 14.2 million tons of chickpeas were produced in the world (FAO, 2022). *Cicer arietinum* L. is one of the earliest legumes to be produced around the globe, and it is thought to have originated in an area near Syria in present-day Turkey. Chickpea is in a tribe of its own, *Cicereae Alef* and 43 species have been reported: 33 perennial, 9 annuals (including the cultivated one), and one unspecified (Van der Maesen, 1987). As a result of the interviews with the farmers producing chickpea, it was determined that 1 kg of chickpea stalk is obtained from one kilogram of chickpea production. In line with these data, approximately 14.2 million tons of chickpea stalks are produced annually in the world. These stalks are either used in animal feed production or left in the soil and rotted. It has become extremely important to obtain products with high added value from lignocellulosic material, which is found in such a large amount.

Wood, annual plants and waste paper are used as raw materials in the production of paper and pulp in the world. Considering some laws and regulations, it has become difficult to obtain wood raw materials as mentioned before, and papermakers have focused on using waste paper and annual plants as raw materials. The most widely used pulp production method in the world is chemical pulping. Chemical pulping gives higher quality fibers than mechanical and semi-chemical pulping methods. The sulfate (kraft) method, one of the chemical pulping methods, gives the strongest fibers and is one of the most widely used chemical pulping methods in the world. Pulping methods are modified to improve some optical, chemical and mechanical pulp and paper properties. These modifications, it is aimed to slow down or stop the peeling reactions that occur in alkaline cooking processes. Therefore, some chemical additives such as anthraquinone (AQ) and boron compounds are added to the cooking liquor. These additives protect the shortening of cellulose chains by reducing end-groups of hemicellulose and cellulose so it modifies keto and the aldehyde groups to the hydroxyl group by reducing easily (Çiçekler & Tutuş, 2021; Saraçbaşı et al., 2016; Tutuş et al., 2015). Anthraquinone (AQ), a redox catalyst, oxidizes the reducing ends of cellulose and hemicelluloses in pulp and protects against alkaline degradation such as peeling reaction (Samp, 2008; Tutuş et al., 2016).

In this study, pulps were produced from chickpea stalks by the kraft-AQ cooking method, and some chemical, mechanical and optical properties of these pulps were characterized, then their usability with primary and secondary fibers was investigated.

2. MATERIAL AND METHODS

2.1. Material

The chickpea stalks used in the study were obtained from the experimental fields of Kahramanmaraş Sütçü İmam University, Faculty of Agriculture (Turkey). The chemical components and fiber dimensions and parameters of the chickpea stalks determined by (Özdemir et al., 2020) were given in Table 1.

Table 1. The chickpea stalks chemical components and fiber dimensions

Chemical Components		Fiber Dimensions and Parameters	
Holocellulose content (%)	78.8	Fiber length (mm)	0.89
Cellulose content (%)	52.6	Fiber width (μ)	18.3
Alpha cellulose content (%)	47.8	Lumen diameter (μ)	11.7
Lignin content (%)	17.1	Cell wall thickness (μ)	3.27
Ash content (%)	4.70	Felting rate	50.2
Extractive (%)	4.40	Elasticity coefficient	62.6
1% NaOH solubility (%)	39.7	Runkel ratio	0.68
Cold water solubility (%)	16.0	Rigidity coefficient	18.7
Hot water solubility (%)	12.8	F factor	272.5

Chemicals used in cooking processes were purchased from Merck Inc. (Germany). In the study, fibers obtained from red pine chips by the kraft method were used as primary fibers and fibers recycled from old corrugated cardboard (OCC) were used as secondary fibers.

2.2 Pulp Production

Chickpea stalks were chipped in 6-8 cm dimensions after being purified from their impurities and non-lignocellulosic structures. To be used in each cooking, 500 g oven-dried samples, whose water content was determined, were kept in polyethylene bags and made ready for cooking processes. Four different cooking trials were applied to chickpea stalks, using the kraft-AQ pulping method and the cooking conditions were presented in Table 2.

Table 2. Cooking conditions applied to the chickpea stalks

Conditions	Value
Active alkali (%)	20
Sulfidity (%)	25
AQ charge (%)	0, 0.3, 0.5, 0.7
Cooking temperature (°C)	160
Time to max. temp. (min)	40
Time at max. temp. (min)	100
Liquor to stalk ratio (l/kg)	5/1

Cooking processes were carried out in an electrically-heated rotary digester resistant to high temperature (350 °C) and pressure (25 bar), and the digester rotates 4 revolutions per minute. The pulps obtained after the cooking processes were washed with plenty of tap water on a 200-mesh screen to remove the black liquor. Then, the pulps were transposed to a slotted (0.15 mm) screen in order to separate the uncooked parts (screen rejects). After the screening processes were completed, pulp yields (screened and screen rejects) were determined according to the amount of raw material used at the beginning of the cooking process. The viscosity values and kappa numbers as chemical properties of pulps were determined according to ISO 5351 and ISO 302 standards, respectively.

2.3 Paper Production

The pulp that gave optimum properties under the conditions given in Table 2 was used in the paper production. Test papers were produced by blending chickpea stalk fibers, primary (virgin) and secondary (recycled) fibers. The fibers were gradually beaten to 35±2 SR° freeness level in a laboratory-type Hollander device before paper production. According to ISO 5269-2 standard, ten test papers with 80±3 ($g.m^{-2}$) grammages were manufactured from each blend by using Rapid Kothen (RK-21) semi-automatic paper machine. Then, the papers were conditioned in a conditioning room for 24 hours at 23±1 °C and 50±2% relative humidity in accordance with the ISO 187 standard.

2.4. Analysis of Mechanical Properties

Printability and runnability depend on the mechanical properties of papers. Breaking length, burst and tear indices, which are main indicators of the mechanical properties of papers, were determined according to ISO 1924-2 (breaking length-50 mm/min), ISO 2759 (burst index) and ISO 1974 (tear index) standards. Fig. 1 indicates where the breaking length, burst and tear test samples were taken from the papers.

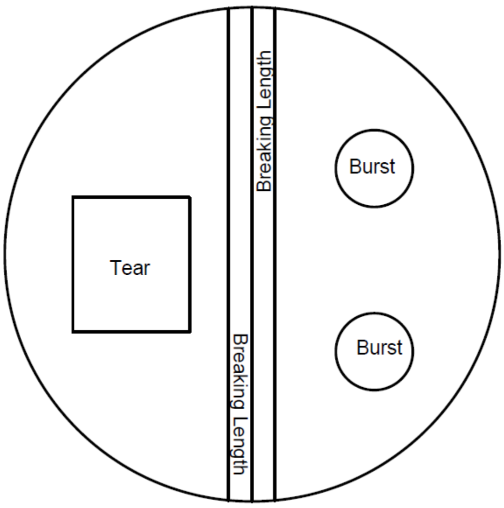

Figure 1. Parts used in determining the mechanical properties of papers.

The mentioned mechanical tests were replicated 10 times on the papers obtained from each fiber blend.

2.5. Analysis of Optical Properties

The optical properties of the papers are a kind of indicator of the printing quality of the paper. Whiteness, brightness and yellowness values of papers are the prominent parameters in optical properties. These values were measured by Datacolor Elrepho 450x spectrometer according to ISO 2470 (whiteness), ISO 11475 (brightness) and ISO 17223 (yellowness).

2.6. Statistical Analysis

One-way analysis of variance (SPSS for Windows, Version 16.0) was used to test whether there is a statistically significant difference between the means of independent groups. Then, the Duncan test which indicates the effects of chickpea stalk fiber content on the mechanical and optical properties of papers was applied.

3. RESULTS AND DISCUSSION

3.1 Chickpea Stalk Pulp Properties and Effects of Anthraquinone

The yield and some chemical properties of the pulps obtained from chickpea stalks by the modified kraft method were given in Table 3.

Table 3. Yield and chemical properties of chickpea stalk pulps produced by the kraft-AQ cooking method

AQ Charge (%)	Screened Yield (%)	Screen Reject (%)	Total Yield (%)	Kappa No	Viscosity ($ml.g^{-1}$)	DP
0	37.9^{d}	1.07^{d}	39.0^{d}	53.5^{d}	421^{d}	577^{d}
0.3	40.4^{c}	0.17^{c}	40.1^{c}	37.5^{c}	446^{c}	615^{c}
0.5	40.7^{b}	0.12^{b}	40.9^{b}	34.5^{b}	511^{b}	715^{b}
0.7	43.9^{a}	0.07^{a}	43.9^{a}	28.5^{a}	565^{a}	800^{a}
Sig	.000	.000	.000	.000	.000	.000

* According to Duncan's mean separation test, mean values with the same lower-case letters are not significantly different at the 95 % level of confidence.

AQ addition in cooking liquor enhanced the pulp yield, viscosity and degree of polymerization (DP) values of the chickpea stalk pulps. AQ oxidizes the reducing end of the polysaccharide in the pulp, preserving it from alkaline deterioration (Liu et al., 2017; Masrol et al., 2018; Shao et al., 2017). Peeling reactions that cause yield loss are reduced and pulp yields increase (Fig. 2).

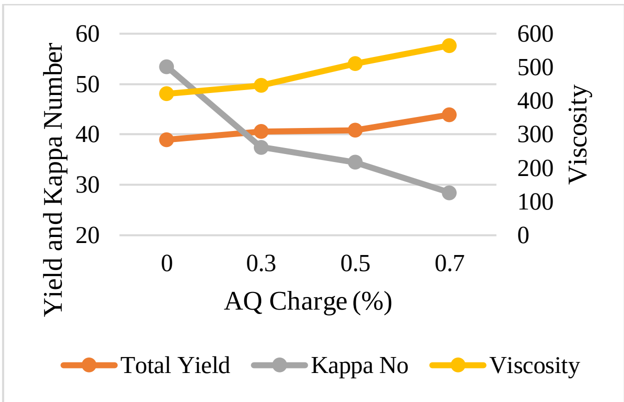

Figure 2. Effects of AQ on yield, kappa number and viscosity of the chickpea stalk pulps

Besides, the viscosity and DP values of the pulps were also increased by the addition of AQ into the cooking liquor. According to statistical analyzes, 0.7% AQ added cooking experiment gave the best results in pulp yield and chemical properties. With the addition of 0.7% AQ, the total pulp yield, viscosity and DP values of the chickpea stalk pulps were increased by about 12.6%, 34.2% and 38.6%, respectively. Kappa numbers, which are indicators of the remaining lignin content in the pulp, decreased by about 47% with the addition of 0.7% AQ (Fig. 2). AQ is an important catalyst that has been used widely in alkaline wood pulping for its effectiveness in an acceleration of delignification (Fišerová et al., 2006; Masrol et al., 2018). Table 4 shows some mechanical and optical properties of chickpea stalk pulps.

Table 4. Mechanical and optical properties of chickpea stalk pulps produced by kraft-AQ cooking method

AQ Charge (%)	Breaking Length (km)	Burst Index ($kPa.m^2.g^{-1}$)	Tear Index ($mN.m^2.g^{-1}$)	Whiteness (ISO%)	Brightness (ISO%)	Yellowness
0	4.10^c	1.84^b	2.91^a	34.4^c	26.3^c	34.8^b
0.3	4.26^d	1.89^c	2.94^a	36.1^b	27.7^b	33.8^a
0.5	4.44^b	1.97^a	2.93^a	36.6^b	28.0^b	34.4^{ab}
0.7	4.62^a	1.94^a	2.95^a	39.8^a	29.5^a	36.9^c
Sig	.000	.000	.154	.000	.000	.000

According to Table 4, AQ addition to the cooking liquor had a positive effect on both mechanical and optical properties of the pulps. The breaking length and burst index of the pulp obtained from the 0.7% AQ-added cooking trial increased by approximately 12.7% and 5.4% compared to the AQ-free cooking trial (Fig. 3).

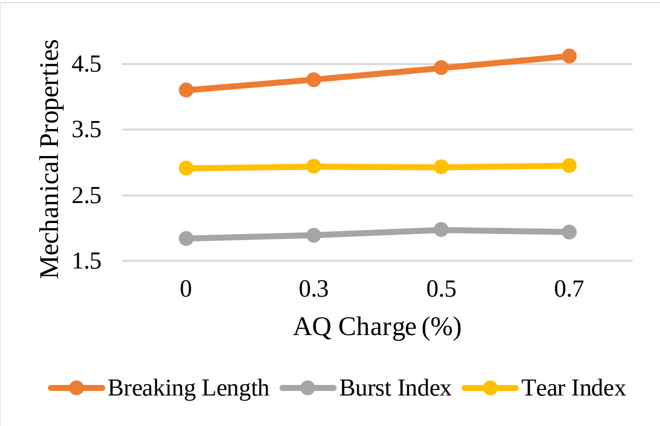

Figure 3. Effects of AQ on the mechanical properties of chickpea stalk pulps

It was observed that AQ had no impact on the tear strength of the pulps. Since AQ slows down or stops the peeling reactions occurring in the cellulose chains, pulps with a high degree of polymerization (DP) are obtained. Thus, the strength properties of pulps with high DP are better (Hassan et al., 2013; Khristova et al., 2006; Sarwar Jahan et al., 2012; Tutuş et al., 2016). Whiteness and brightness values of the AQ-added pulps were found to be higher than AQ-free pulps. On the contrary, the yellowness values of the pulps were negatively affected by using AQ (Fig. 4).

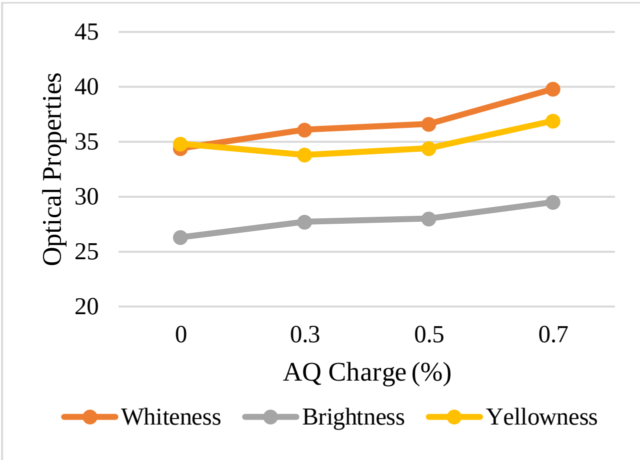

Figure 4. Effects of AQ on the optical properties of chickpea stalk pulps

As mentioned before, due to the effectiveness of AQ in accelerating delignification, the amount of lignin that adversely affects the optical properties of the paper is less. In addition, when the kappa numbers of the pulps in Table 4 are examined, it is clearly understood that AQ reduces the amount of lignin remaining in the pulps (Fišerová et al., 2006; Masrol et al., 2018).

As a result of the analysis of the data in Tables 3 and 4, optimum conditions for pulp production from chickpea stalks were obtained with the addition of 0.7% AQ. Fibers obtained from 0.7% AQ-added cooking trial were used in paper production with primary and secondary fibers.

3.2 Properties of Papers Produced with Chickpea Stalk and Primary Fibers Blends

Table 5 indicates some mechanical and optical characteristics of papers made with chickpea stalk (CF) and primary (PF) fiber blends.

Table 5. Some mechanical and optical characteristics of the papers made with CF and PF blends

Blending ratios (%)	Breaking Length (km)	Burst Index ($kPa.m^2.g^{-1}$)	Tear Index ($mN.m^2.g^{-1}$)	Whiteness (ISO%)	Brightness (ISO%)	Yellowness
100^{PF}	6.35^a	3.26^a	9.69^a	28.53^g	20.46^g	43.41^f
$50^{CF}+50^{PF}$	5.43^b	2.64^b	5.78^b	32.92^f	23.99^f	39.85^e
$60^{CF}+40^{PF}$	5.38^c	2.66^b	5.43^c	34.18^e	24.95^e	39.16^d
$70^{CF}+30^{PF}$	5.13^d	2.73^{cd}	4.85^d	35.61^d	26.30^d	37.65^c
$80^{CF}+20^{PF}$	4.95^e	2.76^d	4.25^e	36.80^c	27.25^c	37.43^{bc}
$90^{CF}+10^{PF}$	4.89^f	2.81^e	3.93^f	38.69^b	29.04^b	37.11^{ab}
100^{CF}	4.61^g	2.94^f	2.94^g	39.78^a	29.51^a	36.96^a
Sig.	.000	.000	.000	.000	.000	.000

As it is known, fibers produced from softwood by the kraft method are quite strong compared to annual plant and hardwood fibers. One of the main reasons for this is that softwood fibers are longer than others (Madakadze et al., 1999; Przybysz Buzała et al., 2018; Schönberg et al., 2001; Wan Rosli et al., 2009). Many paper qualities are influenced by fiber dimensions, especially strength, and fiber dimensions contribute to the optical and surface properties of paper (Hiltunen & Paulapuro, 2011; Larsson et al., 2018).

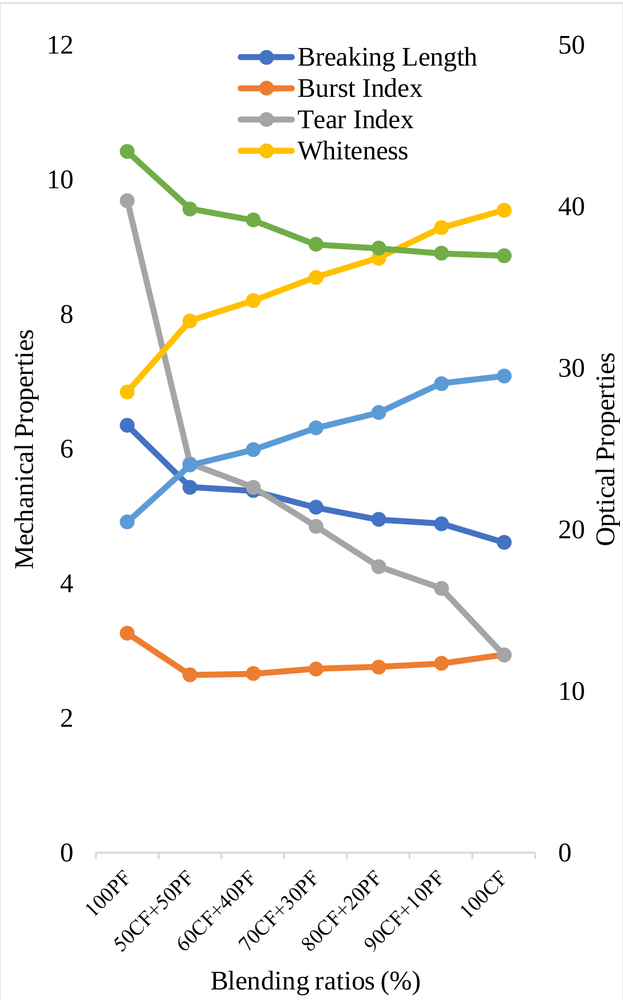

Figure 5. The mechanical and optical properties of the papers produced with CF and PF blends

The effect of fiber properties on paper strength led to the common consensus that paper with the desirable mechanical properties could only be produced from long fiber (Hiltunen & Paulapuro, 2011; Li & Kim, 2018; Watson & Dadswell, 2017). While the long fibers generally improve the mechanical of the paper, the short fibers mostly increase the surface and printing properties (Özdemir et al., 2020; Wan Rosli et al., 2009). As can be seen from Fig. 5, decreases in mechanical properties occurred in parallel with the addition rate of CF to PF. As indicated in Table 1, the fibers obtained from chickpea stalks were classified as short fiber (0.89 mm). Accordingly, the strength properties of the papers produced by blending with CF decrease.

However, when the optical properties were examined, whiteness, brightness and yellowness values were positively affected by the addition of CF. As can be seen from Figure 4, the whiteness and brightness values increased with the addition of AQ to the cooking liquor. Besides, since chickpea stalk fibers have thin-walled (3.27 micron) fibers, they reflect light better than thick-walled fibers (Hubbe et al., 2008; Scallan & Borch, 1974). For these reasons, the optical properties improve as the CF ratio increases in papers produced from CF and PF blends.

3.3 Properties of Papers Produced with Chickpea Stalk and Secondary Fibers Blends

In Table 6, some mechanical and optical characteristics of papers manufactured with CF and secondary fibers (SF) blends were presented.

Table 6. Some mechanical and optical characteristics of papers manufactured with CF and SF blends

Blending ratios (%)	Breaking Length (km)	Burst Index (kPa.m^2.g^{-1})	Tear Index (mN.m^2.g^{-1})	Whiteness (ISO%)	Brightness (ISO%)	Yellowness
100^{SF}	3.80^e	2.39^e	5.46^a	35.25^f	26.47^f	37.15^c
50^{CF}+50^{SF}	4.11^d	2.41^d	4.73^c	36.82^e	27.84^e	36.51^{ab}
60^{CF}+40^{SF}	4.26^{cd}	2.45^d	4.97^b	37.10^d	28.05^d	36.53^{ab}
70^{CF}+30^{SF}	4.33^c	2.76^{bc}	3.97^d	37.80^c	28.64^c	36.25^a
80^{CF}+20^{SF}	4.39b^c	2.81^b	3.97^d	38.13^b	28.84^b	36.26^a
90^{CF}+10^{SF}	4.45^b	2.89^{ab}	3.39^e	38.19^b	28.95^b	36.82^{bc}
100^{CF}	4.61^a	2.94^a	2.94^f	39.78^a	29.51^a	36.96^c
Sig.	.000	.000	.000	.000	.000	.000

Corrugated cardboard papers production in the world is generally carried out by using the pulp obtained from the recycling of waste papers (Barbash & Yashchenko, 2020; Pereira et al., 2020). Fibers recovered from waste paper are called secondary fibers in the literature, and typically the hornified fibers shorten in length and lose their flexibility and take on a rigid structure (Yin et al., 2016; Zhang et al., 2017). At the same time, the surface areas of the fibers become narrower and they lose their fiber-fiber bonding potential (Biermann, 1993; Clark, 1978; Mckee, 1971; Minor, 1994).

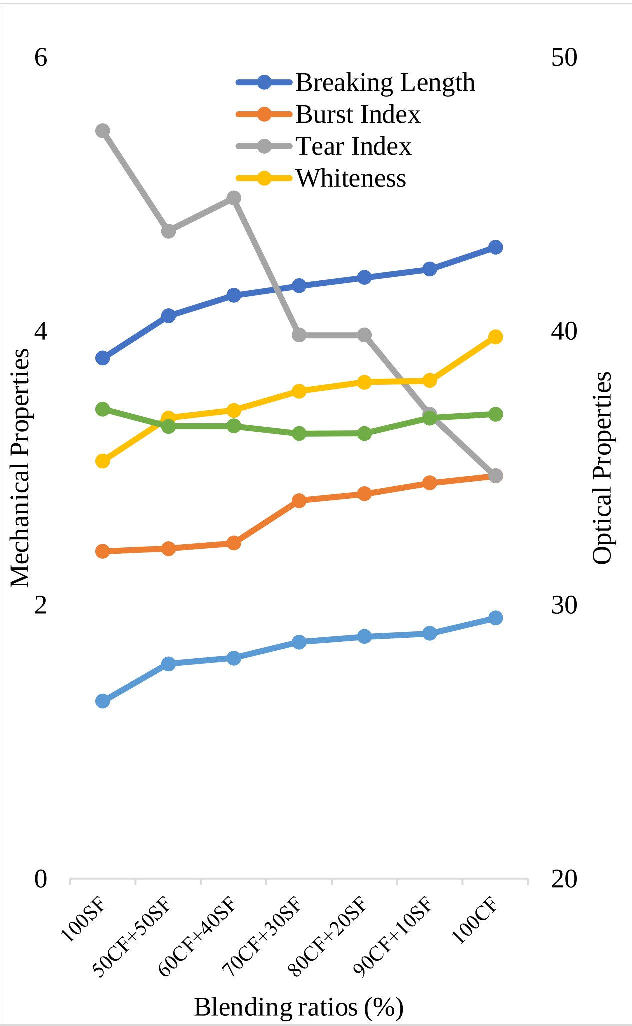

Figure 6. The mechanical and optical properties of the papers produced with CF and SF blends

The mechanical properties of old corrugated cardboard (OCC) pulps consisting of secondary fibers were lower than that of CF (Fig. 6). The fiber lengths and surface areas of SF pulps are lower than virgin pulps as mentioned above. Since the optical properties of CF are higher than that of SF, there has been an increase in optical properties in parallel with the CF addition ratio. This variation is caused mostly by differences in the fibers themselves, as well as the existence of different pollutants that have been properly eliminated from the pulp during processing (Obradovic & Mishra, 2020). The presence of pollution in paper made from secondary fibers can influence the optical properties as well as mechanical properties, though the effect of the changing characteristics of the fibers themselves is considerably more substantial in this case (Yin et al., 2016).

4. CONCLUSION

Optimum results in pulp production from chickpea stalks with a modified kraft method were obtained by adding 0.7% AQ to the cooking liquor. The breaking length and burst index of the pulp obtained by AQ added to the cooking liquor increased from 4.10 km to 4.62 km, and 1.84 kPa.m^2.g^{-1} to 1.94 kPa.m^2.g^{-1}, respectively. The total pulp yield was enhanced by 15.8% with the addition of 0.7% AQ. The utilization of chickpea stalks in pulp production would increase fiber availability in countries with limited wood resources. The mechanical and optical properties of the papers obtained by mixing the chickpea stalk with primary and secondary fibers in certain proportions were not significantly affected. The strength properties of the papers produced with the mixture of the fibers recovered from the old corrugated cardboards and the chickpea stalk fibers increased in parallel with the chickpea stalk fiber content. It was observed that the optical properties of the produced papers increased when the primary and chickpea stalk fibers were blended. Short fiber chickpea stalk pulps can be used in the production of many kinds of paper by blending it with long fibers in certain proportions. Besides, the losses in the strength properties of the paper produced in waste paper recycling can be eliminated by adding chickpea stalk fibers. Correspondingly, the possibility of using chickpea stalk fibers in the production of different papers is quite high.

Acknowledgments

This research was conducted as part of the "YÖK 100/2000 Ph.D. Scholarship" and the "TÜBİTAK 2244-Industrial Ph.D. Program". The authors would like to thank YÖK and TÜBİTAK for their assistance.

Ethics Committee Approval
N/A

Peer-review
Externally peer-reviewed.

Author Contributions
All authors have read and agreed to the published version of the manuscript.

Conflict of Interest
The authors have no conflicts of interest to declare.

Funding
The authors declared that this study has received no financial support.

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