Evaluation of Tea Wastes in Usage Pulp and Paper Production

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The objective of this study was to characterize the properties of pulp and paper produced from tea (*Camellia sinensis*) wastes, an agricultural residue widely available in Turkey, using the kraft-anthraquinone (AQ) cooking method. The chemical components and fiber morphology of tea wastes were investigated. The results indicated that tea wastes had low holocellulose, cellulose, and α-cellulose contents and high lignin content. Also, the suitability of the fiber for pulp and paper production was examined, and the fiber length, fiber width, lumen diameter, and cell wall thickness were measured. According to these values, it was found that the strength properties of papers obtained from tea wastes were insufficient. Therefore, tea waste pulps were mixed with Turkish pine pulps at various ratios. Twelve different cooking experiments were performed on the tea wastes, and the cooking with the best pulp yield was used for mixing. The second cooking, with 0.1% AQ, gave the best yield (33.26%), an increase of about 3.51% compared to the first cooking with no AQ. The physical and optical properties of the papers were also examined. Results showed that paper properties were improved by increasing the Turkish pine pulp rate. Consequently, tea wastes can be used in pulp and paper production when combined with softwood pulps.

Keywords: Tea wastes; Pulp; Paper; Camellia sinensis; Turkish pine

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INTRODUCTION

The decreases in the availability of raw materials for pulp and paper production have led papermakers to search for new raw material resources. Several studies have been carried out to discover these resources (Sabharwal and Young 1996; Atchison 1996; Chandra 1998). As an alternative to wood-based raw materials, annual plants and agricultural wastes are the most important raw material resources for pulp and paper production. In paper industries based on wood, lignocellulosic annual plants were first used as raw materials in the early 19th century. Because of the decrease in forests, the demand for annual plants has rapidly increased, especially after the Second World War, and many small and large-scale mills have been established for pulp and paper production from annual plants (Eroglu 1983; Tutus 2000; Akgul 2007).

Tea factory wastes, which can be regarded as agricultural residues, could be an important raw material for the pulp and paper industries. Tea wastes are obtained from incorrectly harvested black tea leaves. As they are the main waste of tea factories, a high amount of tea wastes are produced. While this amount is between 3% and 5% by normal standards, misharvesting increases the actual amount to 17% to 18% (Kacar 1987; Tiftik 2006). Approximately 40,000 tons of tea wastes are produced in tea factories operating in the Black Sea region every year. Tea wastes are usually dumped in the environment or landfilled, causing environmental problems. These wastes must be considered more valuable such that there will be an economic motivation to prevent pollution (Ozturk 2011).

The kraft process is the most commonly used method for producing pulp suitable for papermaking. The main problem with this method is the high amount of residual lignin. While 90% of the lignin dissolves, the rest remains in the pulps (Gellerstedt *et al*. 1984; Uner 2003). Increasing the pulp yield in cooking is very important to the pulp and paper industries. Modifying kraft cooking, such as with AQ addition, is one way to increase pulp yield (Samp 2008).

In Turkey, tea wastes have been burned for energy production (Malkoc and Nuhoglu 2006). However, there is little to no information on the application of these resources for pulp and paper production. The objective of this study was to characterize the properties of pulp and paper produced from tea wastes, using a kraft-AQ cooking method.

EXPERIMENTAL

Materials

This study was performed at the Kahramanmaras Sutcu Imam University Faculty of Forestry, Pulp, and Paper Production Laboratory. Tea wastes were collected from tea factories operating in the Black Sea region of Turkey. Chemicals were supplied by Merck (Darmstadt, Germany). Raw material preparation, chemical analysis, fiber properties, and the physical and optical properties of handsheets were determined according to the relevant standard methods.

Chemical Compositions and Fiber Morphology

Subsamples of raw materials were analyzed for their α-cellulose, lignin, water, and NaOH (1%) solubilities and ash contents using standard test methods of the Technical Association of Pulp and Paper Industries (TAPPI). Extractives were investigated using the standard procedures of the American Society for Testing and Materials (ASTM 2013). The holocellulose and cellulose contents were determined according to Wise's (1962) chlorite and Kurschner-Hoffer's (1993) nitric acid methods, respectively. The fiber lengths and widths, lumen diameters, and cell wall thicknesses from the morphologic properties of the raw material were measured with a microscope. To measure the fiber morphologic properties of the specimens (0.5-mm thickness and 2-cm long, parallel to the fiber), the chloride method was performed (Wise and Karl 1962). In this method, specimens were immersed into chloride solution until they were defibered and measurements were applied with an Olympus BX51 microscope. The viscosities and kappa numbers of the pulps were determined according to TAPPI standards T230 om-08 (2008) and T236 om-13 (2013), respectively.

Pulp and Paper Production

Air-dried samples were cut into pieces about 6 cm long. Twelve cooking experiments were performed on tea wastes, using kraft-AQ processes, to determine the optimum pulping conditions (Table 1). The Turkish pine pulps used in this study were obtained with the kraft cooking conditions given in Table 1.

Pulping experiments were conducted in an electrically heated laboratory rotary digester of 15-L capacity and 25-kg/cm^2 maximum pressure. All pulps were washed and screened on a 0.15-mm slotted screen. The screened pulp yields and screen reject rates are also presented.

Table 1. Cooking Conditions of Tea Wastes and Turkish Pine Woods

Pulping Condition	Unit	Tea Wastes	Turkish Pine
Active Alkali	%	18, 20, 22	24
Sulfidity	%	25	30
Total Alkali	%	22	28
AQ Charge	%	0, 0.1, 0.3, 0.5	0
Cooking Temperature	°C	150	175
Time to Maximum Temperature	min	40	40
Time at Maximum Temperature	min	60	120
Liquor-to-raw Material Ratio	L/kg	6/1	5/1

Screened pulps were beaten in a Hollander beater to 50±3 °SR freeness, according to TAPPI standard T200 sp-96 (1996). Ten handsheets per test sequence, with grammage of 70 g/m^2, were prepared using a Rapid-Kothen sheet former according to ISO standard 5269-2 (2004). The breaking length (TAPPI T494 om-01 (2006)), burst index (TAPPI T403 om-97 (1997)), tear index (TAPPI T414 om-12 (2012)), brightness (ISO 2469 (2014)), and opacity (TAPPI T425 om-96 (1996)) of the handsheets were investigated.

According to the chemical components and fiber morphology of tea wastes, this raw material has high potential for use as short fiber in pulp and paper production. Therefore, tea wastes pulps (TWP) were mixed with Turkish pine pulps (TPP) to improve the resulting paper properties. The mixing rates (%) of TWP and TPP were 100+0, 90+10, 80+20, 70+30, 60+40, 50+50, 40+60, 30+70, 20+80, 10+90, and 0+100. The pulps that gave the best total yield were used in these mixtures with TPP.

RESULTS AND DISCUSSION

Chemical Compositions and Fiber Morphology of Tea Wastes

The chemical compositions of tea wastes and some wood types and annual plant are presented in Table 2.

Table 2. Chemical Compositions of Tea Wastes, Wood, and Annual Plants (Kirci 2006; Tutus *et al.* 2014)

Chemical Components (%)	Tea Wastes	Hardwoods	Softwoods	Annual Plants
Holocellulose	60.81±1.14	72 to 82	63 to 74	65 to 79
Cellulose	29.42±0.57	38 to 55	55 to 61	40 to 52
Alpha Cellulose	26.53±0.58	-	-	35 to 47
Lignin	36.94±0.34	18 to 26	25 to 32	15 to 19
Ash	4.53±0.10	0.2 to -0.7	0.2 to 0.7	2 to 7
Extractives	15.22±1.16	1 to 6	1 to 6	5 to 9
1% NaOH Solubility	54.14±0.25	12 to 25	8 to 10	28 to 47
Hot Water Solubility	31.38±1.84	1 to 8	1 to 5	3 to 19
Cold Water Solubility	10.86±0.95	0.2 to 4	0.5 to 4	3 to 16

The holocellulose, cellulose, and α-cellulose contents of tea wastes were lower than those of hardwoods and softwoods. This is because tea wastes have lower hemicellulose content and higher lignin content as compared to wood. The high NaOH solubility is due to the presence of low-molecular weight carbohydrates and other alkali-soluble matter (Tutus and Eroglu 2003). Also, NaOH solubility indicates the extent of fiber degradation during a pulping process (Zawawi *et al.* 2014). Water solubility provides a measure of the tannins, sugars, gums, and coloring matter. The water solubility of tea wastes was higher than that of other species because of high coloring matter content (ASTM D1110-84 (2013)).

The ash content in tea wastes was similar to values found in the literature (Table 2). Also, the lignin content was higher than that of hardwoods and softwoods. Lignin functions as an adhesive to bind cellulose together in the fibers. Lower lignin content makes the fiber stronger and harder to break (Tran 2006; Zawawi *et al.* 2014).

The fiber length, fiber width, lumen diameter, and cell wall thickness of tea wastes were measured as 3.7 mm, 870.9 µm, 291.8 µm, and 289.5 µm, respectively. The fiber parameters were determined according to the following equations,

Felting power = Fiber length/fiber width (1)
Elasticity coefficient = Lumen diameter*100/fiber width (2)
Rigidity coefficient = Cell wall thickness/fiber diameter*100 (3)
Runkel's proportion classification = Cell wall thickness*2/lumen diameter (4)
Mulsteph's proportion = Cell wall area/fiber breadth cross section area*100 (5)
F Factor = Fiber length/cell wall thickness*100 (6)

The fiber parameters calculated with these equations are given in Table 3. In the pulp and paper industries, long and short fiber concepts are used. Measurement of the fibers constituting pulp, and accordingly, the relationships between pulp properties, are also important. For example, increases in fiber length affect strength properties but can cause paper malformation (Kirci 2006; Akgul and Tozluoglu 2009).

Table 3. Fiber Parameters of Tea Wastes and Wood Species (Alkan *et al*. 2003; Istek *et al*. 2009; Tutus *et al.* 2014)

Fiber Parameters	Tea Wastes	Softwoods	Hardwoods
Felting Power	42.49	60 to 80	40 to 55
Elasticity Coefficient	33.52	55 to 75	35 to 55
Rigidity Coefficient	33.24	13 to 20	15 to 35
Runkel's Proportion Classification	1.98	0.3 to 0.8	0.5 to 1.8
Mulsteph's Proportion	88.77	50 to 60	55 to 90
F Factor	127.81	400 to 580	150 to 300

±: Standard deviation

Felting power is widely used to determine the suitability of raw material for paper production. This power is important to the physical properties of paper such as breaking length, burst index, and tear index. It is desired to be between 70 and 90 for softwood and 40 and 60 for hardwoods. The felting power of tea wastes was 42.49. This value was similar to that of hardwoods. Therefore, the physical properties of papers obtained from tea wastes pulps were lower than those of softwood. The Runkel value of the raw material was higher than that of other species. This means that this raw material has thick cell walls. Tea waste fibers had a high rigidity coefficient of 33.24. This value negatively affects the tensile, tear, burst, and double fold resistance of paper (Hus *et al.* 1975; Akgul and Tozluoglu 2009a). Elasticity coefficient is an important factor that has a positive effect on the mechanical strength due to a larger number of bonds between fibers (Saikia *et al.* 1997; Dutt *et al.* 2008). The elasticity coefficient of tea waste fibers was lower than that of hardwoods and softwoods. Mulsteph's proportion, indicated by dividing a cell wall by the cross-sectional area, determines the effects of the cell wall on the physical properties of paper. Thin wall fibers are easily crushed during paper production, positively affecting either paper density or resistance properties (Akgul and Tozluoglu 2009a). Mulsteph's proportion of the tea wastes was 88.77, which was higher than that of other species (Table 3). Higher F factor (flexibility), as calculated by dividing the fiber length into the wall thickness, determined that the flexibility of papers from these types of fibers would be better. The F factor of tea wastes was calculated as 127.81. This value is lower than those of hardwoods and softwoods.

Pulp and Paper Properties

The cooking conditions, pulp yields, and some chemical properties of tea wastes are given in Table 4. According to Table 4, the best total yield and kappa number were achieved in the second and first cooking experiments, respectively. The viscosity and degree of polymerization (DP) values of the third cooking experiment were higher than others. When compared with the first cooking (control), the total yield of the second cooking increased by about 3.5%. One reason for this increase is that AQ oxidizes the reducing end of polysaccharides in pulp, protecting it from alkaline degradation.

Table 4. Cooking Conditions, Pulp Yields, and Some Chemical Properties of Tea Wastes

Cooking No	Active Alkali (%)	AQ Charge (%)	Black Liquor pH	Total Yield (%)	Kappa Number	Viscosity (cm^3/g)	DP
1	18	0	13.5	32.14±0.52	137±1.65	806±5.66	1184
2	18	0.1	12.9	33.26±0.49	162±1.86	910±6.05	1354
3	18	0.3	12.1	30.18±0.25	172±1.93	986±5.87	1480
4	18	0.5	12.4	30.36±0.36	169±1.78	928±4.56	1383
5	20	0	12.8	29.68±0.48	176±2.01	925±3.45	1378
6	20	0.1	12.2	27.96±0.45	178±2.36	846±3.58	1250
7	20	0.3	11.8	28.79±0.56	164±1.96	873±4.56	1294
8	20	0.5	11,3	27.47±0.33	168±2.00	926±5.87	1380
9	22	0	12.0	26.75±0.37	171±1.85	863±4.85	1277
10	22	0.1	11.7	26.77±0.49	169±2.67	981±5.01	1184
11	22	0.3	11.1	27.03±0.35	164±1.55	781±6.08	1354
12	22	0.5	11.0	27.69±0.42	171±1.86	856±6.12	1480

±: Standard deviation

Copur and Tozluoglu (2008) reported that adding AQ into kraft pulp resulted in pulps with higher viscosity, indicating that the addition of these additives prevented cellulose degradation in pulping.

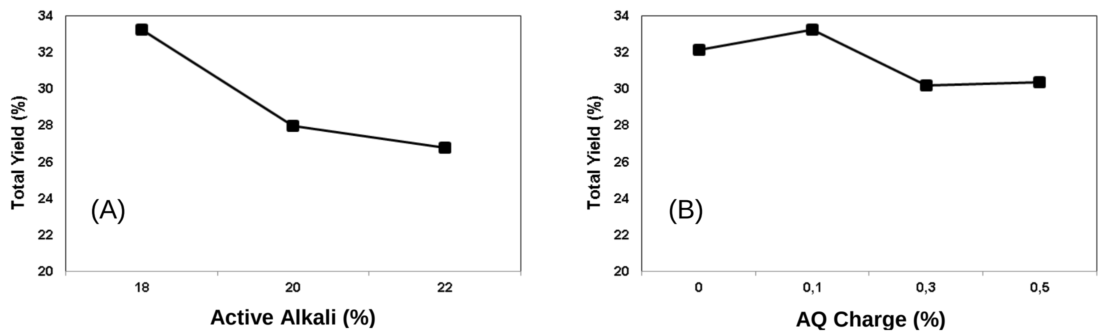

Fig. 1. Effects of active alkali (A) and AQ (B) on total pulp yield

Figure 1(A) shows that the highest total yield was achieved when the lowest tested value (18%) of active alkali was used in pulping. For this reason, these pulps were compared with each other to determine effect of AQ on the total yield. As shown in Fig. 1(B), the best total yield was obtained when 0.1% AQ was used in kraft pulping (cooking 2). In one study, the pulp yields from cotton stalks were increased using AQ. The total yield was 37.98%. Under the same conditions without AQ, the pulp yield was 35.80%. As a result, the pulp yield was increased by about 6.1% (Akgul and Tozluoglu 2009b).

Pulps obtained from the second cooking experiment were mixed with TPP and handsheets were produced to determine their physical and optical properties. The measured physical and optical properties of the mixed pulps are given in Table 5.

Table 5. Physical and Optical Properties of Mixed Pulps

Mix. Rate (%)	TWP + TPP	100 + 0	90 + 10	80 + 20	70 + 30	60 + 40	50 + 50	40 + 60	30 + 70	20 + 80	10 + 90	0 + 100
Breaking Length (km)		2.82 ±0.19	2.75 ±0.16	3.60 ±0.21	3.89 ±0.21	4.20 ±0.26	4.52 ±0.22	5.13 ±0.30	4.63 ±0.22	5.09 ±0.24	5.54 ±0.32	5.51 ±0.31
Tear Index (mN·m²/g)		2.24 ±0.15	2.23 ±0.11	2.26 ±0.19	3.51 ±0.30	3.65 ±0.33	4.34 ±0.25	4.65 ±0.24	6.26 ±0.24	6.21 ±0.22	7.10 ±0.32	6.15 ±0.32
Burst Index (kPa·m²/g)		1.66 ±0.12	1.71 ±0.09	2.02 ±0.20	2.29 ±0.19	2.44 ±0.24	2.92 ±0.11	3.25 ±0.33	3.55 ±0.38	3.79 ±0.26	4.16 ±0.22	4.49 ±0.29
Bulk (cm³/g)		1.77 ±0.15	1.64 ±0.10	1.72 ±0.11	1.70 ±0.22	1.86 ±0.15	1.61 ±0.09	1.56 ±0.15	1.56 ±0.14	1.55 ±0.22	1.49 ±0.17	1.36 ±0.12
Density (g/cm³)		0.56 ±0.01	0.61 ±0.05	0.58 ±0.03	0.59 ±0.05	0.54 ±0.03	0.62 ±0.04	0.64 ±0.04	0.64 ±0.04	0.64 ±0.05	0.67 ±0.03	0.74 ±0.06
Brightness (ISO)		12.49 ±0.76	12.53 ±0.77	13.38 ±0.67	13.44 ±0.49	13.33 ±0.62	14.00 ±0.92	14.59 ±0.85	13.85 ±0.53	13.78 ±0.65	15.42 ±0.68	15.8 ±0.72
Opacity (ISO)		99.09 ±1.28	99.59 ±1.28	99.42 ±1.43	99.91 ±1.62	99.85 1.32±	98.94 ±1.28	99.62 ±1.47	99.60 1.65±	99.47 1.47±	99.01 1.98±	98.7 0.94±

±: Standard deviation

The breaking length and tear index of papers of mixing rate 10+90 were 5.54 km and 7.10 mN·m^2/g, respectively; these values were higher than those of other mixtures. Figure 2 shows that the physical properties of the papers were improved with increasing TPP rate. This was because the fiber properties such as fiber length, width, and other parameters of the Turkish pine were better than those of tea wastes. The optical and physical properties of papers containing 30% TWP and 70% TPP were suitable for paper production, and it was found to be an appropriate mixing rate when considering the economics.

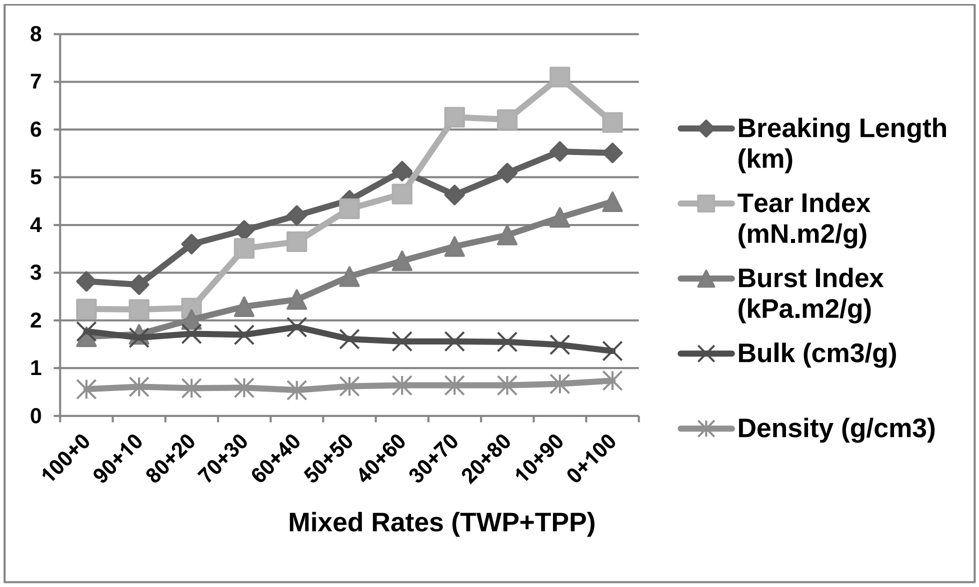

Fig. 2. Effect of mixing rate on physical properties

Several researchers found that fiber length directly affects the physical properties of paper. This finding led to the conclusion that long fibers are stronger than short fibers (Diaz *et al.* 2007; Pickering and Beg 2008; Thumn and Dickson 2013).

CONCLUSIONS

1. With increases in active alkali ranging from 18 to 22%, the total pulp yield was decreased from 32.14% to 26.75%. The best total yield, 33.26%, was found in the cooking experiment with 0.1% AQ. According to these results, the most appropriate cooking parameters for the tea wastes were 18% active alkali and 0.1% AQ charge.
2. Felting power, elasticity and rigidity coefficients, and Mulsteph's proportion of tea wastes fibers were similar to those of hardwood fibers. Therefore, tea wastes can be used by the pulp and paper industry as short fiber. These fibers can be used successfully in all of the areas in which the short fibers are applied. The optical properties of tea wastes pulps can be easily to the desired color values by bleaching.
3. The optical and physical properties of papers containing 30% TWP and 70% TPP were examined, and considering economics, it was concluded that this was an appropriate mixing rate. Based on this, TWP can be used in any kind of paper or cardboard production when mixed with softwood pulps at certain rates.
4. When pulp and paper production raw material shortage is considered, using tea wastes in pulp and paper production would contribute significantly to raw material procurement and economy.

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