The effect of brewing time on pH values, polyphenols content, and antioxidant activities of coffee husk tea (cascara tea)

S Maharani*, I Mustikawati, L Nailufhar and S Istiqomah
Study Program of Agroindustrial Technology Education, Universitas Pendidikan Indonesia, Bandung, Indonesia

*shinta.maharani@upi.edu

Abstract. Coffee husk tea, well-known as cascara tea, is a by-product of coffee processing. It contains phenol compound that has antioxidant activity. Total phenol compound is extracted by brewing process. It is commonly called polyphenol compounds. Polyphenols extraction process is deeply connected to brewing method, especially the time. The purpose of this study was to determine the effect of brewing time on pH, polyphenols content, and antioxidants activity of cascara tea. This study was carried out in Completely Randomized Design with variations of brewing time in 5 levels from 5 minutes, 7 minutes, 10 minutes, 12 minutes, and 15 minutes. The results showed that the variation of brewing time significantly affected the levels of pH values, polyphenols content, and antioxidant activity of cascara tea. The brewing time of cascara tea for 10 minutes has the highest level of polyphenols content and antioxidant activity. Meanwhile pH values was decreased during brewing process.

1. Introduction
Coffee, one of the most traded commodities in the world, is commonly processed into coffee beverage. Coffee beverage is prepared from roasted coffee beans obtained from coffee cherries. The two coffee bean varieties dominating the global coffee market are Arabica (Coffea arabica) and Robusta (Coffea canephora). However, the coffee cherries need to go through an intricate process to produce coffee beans. Therefore, coffee production generates a lot of coffee wastes and by-products [1].

The main solid by-products from cultivation and preparation of coffee are spent coffee grounds, by-products of coffee fruit (coffee cherry), and bean processing (coffee husk, peel, and pulp) [2]. The coffee husk, collected after the coffee bean has been removed from the coffee cherry, is usually discarded as useless waste or used as compost. Further research showed that coffee husks can be processed into energy drinks, food bars, and food supplement due to high concentration of caffeine and polyphenols [3].

Nowadays, coffee husk is utilized as the main ingredient to produce unique tea beverage known as cascara tea. Cascara, meaning "husk" in Spanish, is a dried coffee husk from the coffee fruit. Cascara tea is not exactly the same as the common tea beverage made from Camellia sinensis. Cascara is dried in the sun before they are packaged and sold [1]. The main visual difference between cascara and tea leaves is cascara looks similar to dried raisins or peanut husks.

Cascara tea products have been circulating in the global market but are still hardly found in Indonesia due to lack of public knowledge and interest in the utilization of coffee by-products [4]. Cascara tea contains phenol compounds [1]. Phenol compounds in cascara tea have antioxidant activities [5].
Antioxidants are compounds that can inhibit oxidation. Antioxidants function as a cellular layer of protection between oxidants and delicate cell structures in all human tissues. Antioxidants can intercept or extinguish the damaging oxidative properties of oxidants [6].

The brewing process of cascara tea is one of the way to extract the polyphenols. Polyphenols extraction process is deeply connected to brewing method, especially the brewing time. The antioxidant activities and total phenolic contents varied depending on the brewing temperature and length of hot water infusion [7]. The brewing process allows extraction of some acid compounds that led to different pH values of tea. The increasing duration of brewing causes the chance of longer contact between brewing water and tea. The optimization of brewing time on tea can better extract a large amount of bioactive compounds and antioxidant capacity that led to the optimal chemical properties and sensorial quality because polyphenols are water-soluble compounds [8]. This study was conducted on the effect of brewing time on pH values, polyphenols content, and antioxidant activities of cascara tea. The results of this study were expected to provide information on the best brewing time of cascara tea.

2. Methods

2.1. Materials
The raw material used in this study was coffee husk tea (cascara) derived from Garut Arabica Coffee from Java Coffe Crown. Materials used in chemical analysis were methanol, distilled water, DPPH solution, gallic acid, Folin Ciocalteu 10%, sodium carbonate 7.5%.

2.2. Research methods
The brewing method was done according to SNI 1902-2016 about Black Tea [9]. 2.8 grams of cascara tea was put in a filter bag made of non-woven fabric and added boiled water until the volume reached 140 ml. This experiment was laid out in one-factor factorial arrangement with the variations of the brewing time from 5 minutes, 7 minutes, 10 minutes, 12 minutes, and 15 minutes. The chemical analysis of cascara tea was carried out after brewing to measure pH values, polyphenols content, and antioxidant activities. The equipment used were pH meter (OHAUS), UV-VIS spectrophotometer, cuvette, micropipette, volumetric pipette (10 ml, 2 ml, and 1 ml), vortex mixer, analytical balance, test tube, hot plate, dark-colored bottles, aluminium foil, measuring flasks (250 ml, 100 ml, 50 ml, and 25 ml), and glass cups (100 ml and 300 ml). Table 1 shows the analysis method performed in this study.

| Analysis               | Method            | Source                          |
|------------------------|-------------------|---------------------------------|
| pH Value               | pH meter          | SNI 6989.11:2019 [10]           |
| Polyphenol Content     | Folin Ciocalteu   | SNI 3143:2011 [11]              |
| Antioxidant Activity   | DPPH              | Sudaryat et al. [12]            |

The pH value is a measure of the acidity or alkalinity of a solution. In this study, pH measurements were carried out using pH meter. The indicator cathode tip of pH meter was washed using distilled water before pH meter was used and then the indicator cathode tip was immersed in cascara tea samples. The measurement results were shown on the pH meter.

The levels of polyphenol of cascara tea were calculated according to the Follin-Ciocalteu method with Gallic acid as standard. The Follin-Ciocalteu method are based on the oxidation reduction (redox) reaction of the phenolic hydroxyl group. The selectivity of this test is the oxidation of phenolic compounds.

The antioxidant activities of cascara tea were determined using free radical scavenging assay or DPPH (2,2-diphenyl-1-picrylhydrazyl) assay according to Sudaryat et al. [12] with minor modification. Gallic acid was used as standard. In this method, DPPH acts as free radicals which are suppressed by antioxidants from the samples, where DPPH will be captured by antioxidants through the donation of hydrogen atoms from antioxidants to form reduced DPPH-H.
2.2.1. Statistical analysis. The collected data was analyzed descriptively. The differences of data were analyzed using One-Way ANOVA and Duncan’s Multiple Range Test (DMRT) with 95% confidence interval (ρ = 0.05). The final data was analyzed using IBM SPSS statistic App. 25 ver.

3. Results and discussion

3.1. The pH value of cascara tea
The results of pH value of cascara with brewing-time variation are shown in Figure 1. The a, b, c, d notations mean that each value with the same notation is not significantly different and vice versa in DMRT (ρ = 0.05).

![Figure 1. The pH value of infusion water of cascara with brewing-time variation.](image)

Figure 1 shows that the acidity of cascara tea was significantly increased when the brewing time was longer. The higher value of pH indicates the lower acidity of a solution [13]. Cascara tea brewed for longer time exhibited more acidic compounds. The acid content extracted in cascara tea is not only phenolic acid but all acidic compounds. Coffee husk contains several acidic compounds including chlorogenic acid, ferulic acid, caffeic acid, siringat acid, kumaric acid, vanic acid, gallic acid, and hydroxybenzoic acid [4].

The important factors that affected the pH value are the type and time of brewing. The titratable acidity in hot coffee extracts was larger than that measured in cold brew coffee extracts, which could indicate that hot brewing was able to extract more acids and additional acidic compounds [14]. The maximum acidity extracted in this study was in brewing time for 15 minutes that was affected by a prolonged extraction time under hot extraction because a longer brewing time allows increased extraction of some compounds [15].

Further analysis by the Duncan's Multiple Range Test revealed that the brewing time has a significant effect on the pH value of cascara tea. The brewing time of cascara tea for 5 minutes and 10 minutes had significant difference between other treatments. Meanwhile, the brewing time of cascara tea for 7 minutes, 12 minutes, and 15 minutes did not have significant differences between other treatments.

3.2. Polyphenols content of cascara tea
Polyphenols in cascara tea are important for the benefit of human health because of their scavenging ability on free radicals due to their hydroxyl groups [16]. The levels of polyphenols content of cascara tea with brewing-time variation are shown in Figure 2. The a, b, c, d notations mean that each value with the same notation is not significantly different and vice versa in DMRT (ρ = 0.05).
Figure 2. The levels of polyphenols content of cascara tea with brewing-time variation.

Figure 2 shows that cascara tea brewed in 5 minutes to 10 minutes exhibited increased levels of polyphenols. Polyphenols content of cascara tea brewed in 5 minutes, 7 minutes, and 10 minutes were increased due to longer brewing time. The brewing time affected the polyphenols extracted from cascara tea [5]. The longer duration of brewing caused the chance of longer contact between brewing water and tea. The extraction process become better and the total polyphenols were increased because polyphenols are water-soluble compounds. However, cascara tea brewed in 12 minutes and 15 minutes exhibited lower polyphenols content.

The lack of temperature control during the course of the study was one of the factors causing the decreased polyphenols content. The optimum polyphenols content of cascara tea can be obtained by maintaining the temperature of water during brewing process. Temperatures that are too high or too low can affect the chemical content that can be extracted during the brewing process. One of the compounds that is possible to be degraded during the brewing process is epicatechin [5]. Brewing at a temperature of 98°C led to catechins degradation by 20% [17]. Brewing white tea at 98°C for 7 minutes exhibited the highest polyphenol content [8]. Acid is one of the polyphenol compounds that can be extracted during process. Several studies has been reported that chlorogenic acid and protocatechuic acid are the dominant polyphenols in all analysed samples [5].

Further analysis by the Duncan's Multiple Range Test revealed that the brewing time has a significant effect on the polyphenols content of cascara tea. The brewing time of cascara tea for 5 minutes and 10 minutes had significant differences between other treatments. Meanwhile, the brewing time of 7 minutes, 12 minutes, and 15 minutes did not have significant differences.

3.3. Antioxidant activity of cascara tea

Polyphenols are chemical compounds contained in plants and are strong antioxidants [16]. Antioxidants are molecules that can neutralize free radicals by receiving or donating an electron to eliminate the condition of "unpaired electrons" [6]. Antioxidant activities of cascara tea were determined based on the inhibition percentage of DPPH free radicals. The antioxidant activities of cascara tea with brewing-time variation are shown in Figure 3. The a, b, c, d notations mean that each value with the same notation is not significantly different and vice versa in DMRT (ρ = 0.05).

Figure 3. Antioxidant activity of cascara tea with brewing-time variation.
Figure 3 shows that the brewing time of cascara tea for 5 minutes to 10 minutes had an increase in antioxidant activity. Meanwhile, the brewing time of cascara tea for 12 minutes and 15 minutes had a decrease in antioxidant activity. This was directly in accordance with the levels of polyphenols. The sample that perform the highest antioxidant activity (Figure 3) have the highest polyphenol content (Figure 2). This result is in good accordance with Geremu that the increase in polyphenol content was in line with the increase in DPPH free radical scavenging capacity and thus antioxidant activities increased [16]. Cascara tea brewed in 5 minutes and 7 minutes had antioxidant activities that did not differ significantly as shown in Figure 3.

In addition to catechin compounds, there are also other antioxidants that play a role in inhibiting DPPH radicals. Various studies have shown a strong correlation between the capture of DPPH free radicals and the total amount of polyphenols extracted. Study of white tea showed a strong correlation between the capture of DPPH free radicals and total polyphenols in the steeping of the tea [18]. Further analysis by the Duncan's multiple range test revealed that the brewing time had significant effect on the antioxidant activities of cascara tea. The brewing time of cascara tea for 5 minutes and 7 minutes did not have significant difference. Meanwhile, the brewing time of cascara tea for 10 minutes, 12 minutes, and 15 minutes had significant differences between other treatments.

4. Conclusions
The brewing time of cascara tea significantly affect pH values, polyphenols content, and antioxidant activity of cascara tea. Cascara tea brewed for 10 minutes has the highest level of polyphenols and antioxidant activity. The best brewing time of cascara tea is 10 minutes to reach the optimum levels of polyphenol and antioxidant. Meanwhile, the longer brewing time increased the acidity of cascara tea.

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