



The effect of packaging materials on the physicochemical stability of ground roasted coffee

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ABSTRACT

This research was intended to evaluate the effect of packaging materials on the stability of physicochemical properties of ground roasted coffee during storage at room temperature ($28\pm 2^\circ\text{C}$) and relative humidity (RH) 80% for 12 months. The packaging material used was aluminium-laminated polyethylene (ALP) and polyethylene terephthalate (PET). The physicochemical characteristics observed were non-volatile fractions consisted of moisture, crude fibre, ashes, the alkalinity of ashes, pH, microbiological load, total phenolic and caffeine contents. The results showed that the moisture content of ground coffee was below 5.0 % for both packaging materials. The overall adsorbed moisture of ground coffee was significantly different ($p < 0.05$) for both packaging materials, whereby the moisture content of ground roasted coffee packaged in PET was 4.15% while of those packaged in ALP was 3.38% by the end of 12 months storage. There was no significant decrease in the ashes, the alkalinity of ashes, crude fibre, pH, total phenolic and caffeine contents for both packaging materials. The total plate count (TPC) decreased from an initial of 1.2×10^3 to be 1.7×10^2 CFU/g. Both packed ground roasted coffees were microbiologically safe as it had TPC less than 10^5 CFU/g after 12 months storage. The results indicated that ALP and PET packaging can preserve the quality and stability of the ground coffee during storage.

Article history:

Received 28 Dec 2019

Revised 19 Feb 2020

Accepted 25 Feb 2020

Available online 29 Feb 2020

Keywords:

Ground roasted coffee
storage time
packaging materials
physicochemical stability
PET

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DOI: 10.5614/crbb.2019.1.2/ZTVC3720

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1. Introduction

Ground roasted coffee is the most popular beverage in the world. It is consumed for a refreshing drink, or as a daily routine regularly due to taste and stimulatory effect. Ground coffee containing hundreds of chemical compounds developed during roasting, such as carbohydrates, lipids, vitamins, minerals, and phenolic compounds with numerous health effects (Benkovic and Tusek, 2018).

The quality of ground coffee is strongly influenced by the roasting process. The roasting process also induces transformation of chemical contents during storage. Oxygen, light, temperature, moisture, packaging and extraneous odours have the most significant influence on the quality of coffee during storage even those can cause stalling. Massive odour and flavour losses are the consequence of the water solubility of essential oils of coffee and the formation of volatile flavouring substances with oxygen (Kreuml et al., 2013; Sage, 2012). Yeretian et al. (2017), stated that the change in aroma might due to oxidation, where aroma compounds oxidised to produce a new volatile compound so the coffee aroma fades. The oxidative processes can be prevented by protecting the coffee from oxygen by using packaging material that has barrier properties.

Commercially ground coffees are available in various type of packaging. The function of packaging is to protect the commercial value of coffee as long as possible by maintaining coffee unity with all of its characteristics (Ismail et al., 2013). The standard quality for the roasted and ground roasted coffee requires the type of packaging materials that may be used is airtight cans, glass

containers, or suitable metal foil or laminated containers with food-grade plastic lining. Among the type of metal foil packaging or lamination with plastic lining that is often used is flexible packaging such as aluminium laminated polyethylene (ALP) and polyethylene terephthalate (PET). These type of packaging materials most often used in single-serving packaging.

Research on the storage time of ground roasted coffee quality has been carried out widely. Most of the study evaluated the change of aroma during storage, such as Mayer and Grosch (2001) who assessed the changes in the volatile profile of freshly ground Arabica coffee and ground coffee. Cardelli and Labuza (2001) found the shelf-life of roasted and ground Arabica coffee based on consumer evaluations of product acceptability using a Weibull hazard analysis method was 20.6-24 weeks. Ross et al. (2006) studied the sensory change of ground roasted coffee which stored at room and freezing temperature. Kreuml et al. (2013) found a decrease in the sensory qualities after nine months of storage in vacuum packed. Gloss et al. (2014) monitored the loss of freshness index for commercial single-serve capsule systems packed in several packaging materials by using DMDS/MeSH freshness index. All previous research studied the shelf-life of bulk coffee which is packaged in cans or bags, no study on the shelf-life of single-serve coffee packaging.

Most of the research on the change of quality of ground and roasted coffee focused on the volatile component. The sensory quality of coffee is a combination of the volatile and non-volatile element which constitutes it. Moisture, ashes, crude fibre and microbiological loads are among structural components of the non-volatile component. It responds for the body and flavour of ground roasted coffee. Besides, some coffee exporting countries and

importing countries have set these parameters as a coffee quality requirement (standard coffee quality).

For all these reasons the present work was to study the stability of non-volatile components of ground roasted coffee during 12 months storage in a single serving packaging by using PET and ALP as a packaging material based on the standards quality for roasted and ground coffee. The single-serve packaging was chosen since there is great interest in using it in coffee industries. The importance of understanding the behaviour of the non-volatile component during storage thus to maintain their properties to maximize its shelf-life.

2. Materials and methods

2.1. General procedure

The ground roasted coffee and packaging materials used for the research were provided by Benua Coffee a local industrial coffee roaster in Palembang, Indonesia. The ground roasted coffee was blended coffee from Robusta coffee and Arabica coffee with the ratio 80:20, w/w. Flexible packaging materials consist of (I) aluminium laminated poly ethylene (ALP) made from 12 µm polyethylene terephthalate (PET) and 15 µm PE, laminated with 7 µm aluminium (Al) and 25 µm copolymer polypropylene (CPP); and (II) poly ethylene terephthalate (PET) made from 12 µm polyethylene terephthalate (PET), 9 µm silicon oxide SiO_x, 15 µm nylon (NY) with 65 µm random copolymer polypropylene (R-CPP). Freshly roasted Arabica and Robusta coffee were blended with the ratio 20:80 (w/w) and ground in a commercial grinder to drip brewing size (60 mesh). The blended ground roasted coffee then was packed in a unit sachet of ALP and PET measuring 5.0 cm x 2.0 cm for holding 7 g.

2.2. Assessment stability of ground roasted coffee

The moisture content was determined based on a gravimetric method (AOAC, 2005). The total ashes were determined by incinerating the samples at 550°C for 4 h (AOAC, 2005).

Total alkalinity of ashes was obtained by igniting the sample in muffle furnace until it was free from carbon. The ashes obtained was added with H₂O₂ 3 % and 20 ml of HCl 0.5 N and heated on the water bath for 10 min, filtered through ashless filter paper. Then, the filter paper was washed with hot water until free of acid. The filtrate and the washing were combined, and the alkalinity of ashes was determined by titrating against 0.1 N NaOH using phenolphthalein as an indicator. The alkalinity of ash was expressed as a number of ml of N NaOH per 100 g sample.

The crude fibre was determined gravimetrically after chemical digestion and solubilisation of other materials. The fibre residue weight was then corrected for ash content after ignition (AOAC, 2005).

The pH of the coffee samples was determined using pH analyser (Model No. ELICO LI 614 pH). About 1.0 g of ground roasted coffee was mixed with 10 ml hot water and cooled to room temperature.

The coffee brew was filtered by Whatman No. 1 filter paper. The filtrate was measured using a pH analyser.

Total plate count was determined based on AOAC (2005). The principle of total plate count analysis was the calculation of the number of bacterial colonies present in the dilution sample that were incubated for 24-48 h at 34-36°C; the petri dish was placed upside down in the incubator.

Total phenolic content (PC) was determined by using Folin-Ciocalteu's method as described by Contini et al. (2008) with slight modification. The tannic acid solution was used for calibration. Measurements were performed in triplicate and results were expressed as % tannic acid equivalent (TAE) (mg/g).

Caffeine content was determined by the HPLC method. Amount of 2.00 g ground roasted coffee samples were weighed and transferred into 250 ml beakers. A 100 ml of boiling distilled water was added and let to stand for 5 min with stirring. The solution was cooled and filtered into conical flasks. Five ml of the filtrate were pipetted into a clean 50 ml volumetric flask and adjusted to the mark with the mobile phase. The standards and the samples were run in the HPLC system.

The sensory evaluation conducted according to Cardeli and Labuza (2001) with slight modification. Sample coffee was removed at each sampling time and brewed with 150 ml hot water in ceramic cups (200 ml capacity) and covered with aluminium foil. Samples were then identified with random numbers and served to untrained tasters. Tasters consisted of 18 tasters (50% male, 50% female, 30-54 years of age) and allowed to participate in the study only if they regularly drank one or more cups of black coffee per day, without sugar or cream. Three tasters were used at the beginning of the test (0 months). The number of tasters was increased by C=1 at each sampling time until half of the tasters found the samples unacceptable. After that, the number of tasters for each period was increased by C+U, where U= number of unacceptable responses for the previous test time.

2.3. Data analysis

All experiments were conducted in triplicates and analysis of variance (ANOVA) and LSD tests were performed using SAS 9.1.3 software. The data were expressed as mean ± standard deviations. The confidence interval at 95% (p < 0.05) was used for statistical significance.

3. Results and Discussion

The moisture content of ground roasted coffee significantly (p<0.05) increased during storage from an initial of 2.05±0.15% to 4.15±0.41% for PET and 3.38±0.31% for ALP respectively. Linear regression showed that the storage time and packaging materials had a strong correlation with the increase of moisture content (R²=0.96). This result is in good agreement with the study worked by Benckovic and Tusek (2018), Correa et al. (2016), and Votavova et al. (2009), who stated the moisture content of ground roasted

Table 1. Change on the moisture, ashes, alkalinity of ashes and crude fibre

Storage time, months	Moisture, %		Ashes content, %		Alkalinity of ashes	
	ALP	PET	ALP	PET	ALP	PET
0	2.05±0.15 ^{aA}	2.05±0.15 ^{aA}	1.48±0.21 ^{aA}	1.48±0.21 ^{aA}	61.67±0.23 ^{aA}	61.67±0.27 ^{aA}
4	2.25±0.04 ^{aA}	3.48±0.10 ^{bB}	1.19±0.10 ^{aA}	1.36±0.19 ^{aA}	60.12±0.42 ^{aA}	59.01±0.43 ^{bB}
8	2.80±0.72 ^{aA}	4.03±0.66 ^{bB}	1.42±0.22 ^{aA}	1.49±0.18 ^{aA}	58.48±0.64 ^{bA}	59.08±0.72 ^{bA}
12	3.38±0.31 ^{bA}	4.15±0.41 ^{bB}	1.39±0.21 ^{aA}	1.38±0.12 ^{aA}	58.65±0.32 ^{bA}	58.18±0.38 ^{cA}

^aMeans ± standard deviations with different superscripts within a column are significantly different at p < 0.05.

^AMeans ± standard deviations with different superscripts within a row are significantly different at p < 0.05. Each value represents triplicate analyses of the samples (n=3)

coffee exhibited a consecutive rise throughout storage. The increase in the moisture level due to transmission could result in favourable condition for microbial growth. ANOVA test showed that the storage time and the type of packaging materials had a significant effect on the moisture content. The increase of moisture content of ground roasted coffee packaged in ALP was smaller compared to PET (Table 1) showed that ALP is more effective in preventing the increase in moisture content due to the transmission of moisture from the surrounding. The result was consistent with other study carried out by Wong et al. (2016) who stated that aluminium foil had exhibited higher protective barriers against moisture content. Moreover, Pua et al. (2008) reported that aluminium foil was more superior to other polymeric materials even at 6 to 9 µm gauge. Most international standards for the quality of roasted and ground coffee set the moisture content shall not exceed 5.0 % at the time of packing (ICO, 2018). This means that the moisture content both of ground roasted coffee considered acceptable since it less than 5%.

Ashes are the inorganic matters or solid that are left after complete heating in the presence of oxidizing agents, which provides a measure of the total amount of minerals within a food, like Ca, Na, K and Cl. Determination of the ashes and mineral content of foods is important for several reasons: (i) the type and concentration of minerals contained affect the quality of foods,

including their taste, appearance, texture, and stability; (ii) food that contain high mineral, are more stable to microorganisms; (iii) some minerals *e.g.*, calcium, phosphorus, potassium and sodium are essential to a healthy diet whereas others can be toxic (*e.g.*, lead, mercury, cadmium, and aluminium). The most important minerals present in coffee are considered micronutrients essential for human health. At the initial, the ashes content of ground roasted coffee was 1.48±0.21%. There was a slight change in ashes content during storage. However, the change in ashes content both for those packed in PET and ALP were not significant. ANOVA results showed that both storage time and type of packaging materials had no significant effect on the ashes content. According to Ballesteros et al. (2014), the composition of ashes in roasted coffee consists of a variety of mineral elements including potassium, calcium, magnesium, sulphur, phosphorus, iron, manganese, boron, copper, and others. These elements are stable thus persistent. The compilation of national quality standards for roasted coffee and ground coffee by the International Coffee Council set a total ash content of 6 % by mass. Foods, which contained the ash content exceeds 6.00%, maybe indicated a significant quantity of impurities. This means that both types of packaging materials used are able to prevent contamination of impurities from the surrounding environment during storage.

Table 2. Change on the crude fibre and pH

Storage time, months	Crude fibre, %		pH		Total plate count, CFU/g	
	ALP	PET	ALP	PET	ALP	PET
0	18.85±0.23 ^{aA}	18.85±0.23 ^{aA}	5.15±0.16 ^{aA}	5.15±0.16 ^{aA}	1200±100 ^{aA}	1200±100 ^{aA}
4	17.21±0.42 ^{aA}	18.27±0.63 ^{aB}	5.13±0.21 ^{aA}	5.13±0.13 ^{aA}	740 ± 60 ^{bA}	2200±300 ^{bB}
8	18.39±0.22 ^{bA}	17.65±0.58 ^{bB}	5.23±0.19 ^{aA}	5.14±0.11 ^{aA}	260 ± 82 ^{cA}	380± 90 ^{cA}
12	17.40±0.21 ^{bA}	18.60±0.29 ^{aB}	5.12±0.08 ^{aA}	5.13±0.09 ^{aA}	170 ± 60 ^{cA}	190± 20 ^{cA}

^aMeans±standard deviations with different superscripts within a column are significantly different at $p < 0.05$.

^AMeans±standard deviations with different superscripts within a row are significantly different at $p < 0.05$.

Each value represents triplicate analyses of the samples (n=3)

The alkalinity of ashes is a measure of the presence of combined cations with organic acids. The alkalinity of the ashes is defined as the sum of cations, other than the ammonium ion, combined with the organic acids. The alkalinity of ashes will be expressed in milliequivalents per 100 grams of dry matters in roasted coffee. It not only indicates the presence of carbonates but also shows potential mineral adulteration. Roasted coffee with alkalinity under 57 considered as adulterated (Cramer, 1992). The test result showed that the alkalinity of ashes slightly changes during 12 months storage (from 61.67 to 58.18). Statistical analysis showed that storage time had a significant effect on the alkalinity of ashes while the type of packaging and its interaction had no significant effect on the alkalinity of ashes. This result in good agreement with Cramer (1992) who stated that the alkalinity of ashes in roasted coffee varied between 60.7 to 68.6.

The crude fibre of ground roasted coffee at the initial was 18.85±0.23%. There was a slight change in the crude fibre content during storage for both packaging materials. However, the change on the crude fibre content was not significant. ANOVA showed that neither storage time nor the type of packaging and its interaction had a significant effect on the crude fibre of ground roasted coffee. This means that the crude fibre of ground roasted coffee packed in ALP and PET is relatively stable during storage. According to Chandaka et al. (2017), the crude fibre consists of 60-80% cellulose and (4-6%) lignin, plus some mineral matters. These components have a little food value but provide the bulk necessary for proper peristaltic action in the intestinal tract and beneficial in treating or preventing constipation, haemorrhoids, diverticulosis, coronary heart diseases, and some cancer. Crude fibre is part of insoluble fibre found in the edible portion of the plant cell wall (Dai and

Chau, 2017). Yang et al. (2017) stated that crude fibre makes up a small part of dietary fibre. It only a rough indicator does not truly reflect the total fibre in food. Almost 60% of dietary fibre of coffee is composed by polysaccharides, Maillard reaction products, and other non-identified substances (Moreira et al., 2015).

The pH value of ground roasted coffee was varied between 5.12 to 5.23 (Table 2), and can thus be considered to be slightly acidic. Basavaraj et al. (2014) stated that the perceived acidity of coffee brews has always been recognised as an essential criterion for a good cup of coffee. He also noted that acidity level assessed by the organoleptic scoring matched with the acidity level measured by pH meter, thus the pH of a coffee has been found to correlate with the perceived acidity in coffee. The sample which has higher pH value gets lesser the score for the sensory perceive acidity. The lower pH of roasted coffee is due to the acid content in coffee which can be classified into three groups namely aliphatic, chlorogenic, and alicyclic carboxylic and phenolic acids. ANOVA result showed that neither storage time nor the type of packaging and its interaction had a significant effect on the pH of ground roasted coffee. This means that the pH of ground roasted coffee packed in ALP and PET is stable during storage.

The total plate count is the enumeration of aerobic, mesophilic organisms that grow in aerobic conditions under moderate temperatures of 20-45°C, including all aerobic bacteria, yeast, moulds, and fungi that grow in the specific agar. This count includes all pathogens and non-pathogens and is used to determine the hygienic status of food produced. Schages et al (2018) stated that coffee could be contaminated with different microorganisms including both bacteria and fungi (including yeasts). The mould contamination can occur on coffee beans as a result of improper processing during harvesting, drying, and inadequate storage

conditions. Even though the roasting temperature of coffee beans can eliminate the mould contaminant, however, some spores are not entirely eliminated and would be carried over in coffee products (Kusumaningrum and Rasyidah, 2019). Test results showed that the total plate count decreased from the initial 1.2×10^3 to be 1.7×10^2 CFU/g at the end of 12 months storage for the ground roasted coffee packaged in ALP. ANOVA showed that storage time and its interaction with the type of packaging materials had a significant effect on the total plate count, while the type of packaging materials does not. The decrease in the total plate count is caused by several factors. One, lack of oxygen available in the package. Total plate count also referred to as an aerobic plate count, which requires free oxygen for survival and growth. Lack of oxygen causing the microbes cannot survive even die. Two, low levels of moisture content (<5%) is a barrier to microbial growth. Three, coffee contains several compounds such as caffeic acid, chlorogenic acid, trigonelline and phenolics which has an inhibitory effect against the growth of the microorganism (Fardiaz, 1995). This result is in accordance with the study conducted by Ismail et al. (2013) who found the decreased in the plate count and the count of yeast and moulds during coffee bean storage.

Table 3. Total phenolic content and caffeine content of ground roasted coffee

Storage time, months	Total phenolic content, megTAE/g		Caffeine, %	
	ALP	PET	ALP	PET
0	110.48±2.3 ^{aA}	110.48±2.3 ^{aA}	2.143±0.06 ^{aA}	2.143±0.06 ^{aA}
4	109.88±1.6 ^{aA}	109.73±2.1 ^{aA}	2.117±0.06 ^{bA}	2.116±0.05 ^{bA}
8	109.36±2.8 ^{aA}	108.47±1.9 ^{aA}	2.116±0.03 ^{bA}	2.110±0.03 ^{bA}
12	108.58±3.1 ^{aA}	108.26±1.7 ^{aA}	2.113±0.02 ^{cA}	2.113±0.02 ^{bA}

^aMeans±standard deviations with different superscripts within a column are significantly different at $p < 0.05$.

^AMeans ± standard deviations with different superscripts within a row are significantly different at $p < 0.05$.

Each value represents triplicate analyses of the samples (n=3)

There were no statistically significant differences in the number of total phenolic content during storage for both packaging materials (Table 3). In contrast, Raseetha and Abdullah (2016) found that the total phenolic content decreasing during storage time. Polyphenols are secondary metabolites that are produced by plants to protect themselves against plant diseases and insects, they also play essential roles in human health in protecting against a variety of many diseases associated with oxidative stress and free radical-induced damage. Chlorogenic acids are the most prevalent phenolic compound found in coffee (Farah, 2012).

Table 4. Weibull sensory data

Storage time, months	Acceptability	
	ALP	PET
0	+++	+++
4	++++	++++
8	+++++	+++++
12	+++++	+++++

+: acceptable sample as assessed by an untrained taster
-: unacceptable sample as assessed by an untrained taster

Caffeine content is the most important properties of coffee. It determines the strength, body, bitterness, and flavour of brewed coffee and also used as an indication of coffee quality. Caffeine affects as a stimulant to the nervous system, relaxation bronchial muscle and secretion (Kinuthia et al., 2017; Perez-Sarinana and Saldana-Trinidad, 2017; Van Cuong et al., 2014; Nuhu, 2014; Farah, 2012). Test results showed that the caffeine content of ground roasted coffee was ranged 2.11 - 2.14%. There was a slight

change in the caffeine content during storage for both packaging materials, however, the change on the caffeine content was not significant. ANOVA showed that neither storage time nor the type of packaging materials had a significant effect on the caffeine content of ground roasted coffee (Table 3). Most international standards require caffeine content 1.0% minimum on a dry basis.

The quality most of foods and beverage decreases over time, even under ideal handling condition. However, test results showed that till the end of 12 months of storage, the moisture, ashes content, alkalinity of ashes and total plate count of ground roasted coffee still met the quality standard requirements. The result of the acceptance test for the ground roasted coffee that has been stored for 12 months showed that all panelist stated that the sample was acceptable (Table 4). These results confirmed that the packaging of ground roasted coffee by using ALP or PET able to minimize quality degradation during storage.

4. Conclusion

Selecting a suitable packaging material is very important for coffee manufacturing industries. Packaging materials significantly ($p < 0.05$) influenced the moisture content and total plate count of ground roasted coffee. The study suggested that ALP materials were better suited for keeping ground roasted coffee than of PET.

Acknowledgement

The authors would like to thank you to Palembang Institute for Industrial Research and Standardization, Ministry of Industry, and PD. Sahang Mas which had facilitated this research.

Conflict of Interest

The authors declare no conflict of interests in this research.

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