Changes in physicochemical, antioxidative, and sensory properties in espresso coffee during refrigerated storage and their impacts on coffee milk

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Abstract. Coffee is a widely consumed commodity. Beside its stimulating effects, it also has beneficial health effect. The objective of this study was to observe the changes in physicochemical, antioxidant and sensory properties of espresso coffee during refrigerated storage to determine its shelf-life as ingredient for coffee milk. The analysis used in this study were pH, color, antioxidant activity, total phenolic content, and sensory test as triangle test and hedonic of flavor, aroma, body, and color. The results showed that espresso coffee has quality degradation in both chemical (pH and antioxidant) and sensory properties during refrigerated storage. The lightness of coffee milk made from espresso coffee after four-day storage significantly increased. Although there was changes in pH of espresso coffee, it didn’t change the pH of coffee milk produced. The reduction of antioxidant capacity could be observed from espresso coffee after 2 days storage, while the reduction of total phenolic content was observed after 4-day storage. Interestingly, the highest hedonic score was found in both of coffee products stored for 4 day in the refrigerator. The shelf life of espresso coffee for making coffee milk was six days based on triangle test.

1. Introduction

Coffee is the world most consumed beverages after water and the domestic consumption of coffee keep has increase to equivalent of 4.75 million 60-kilogram bags of coffee in 2018/2019 [1]. Brewed coffee contains bioactive compounds like chlorogenic acids and derivatives, trigonelline, nicotinic acid, 5-hydroxymethylfurfural, theobromine, theophylline, and caffeine [2]. Moreover, coffee consumption has been shown to reduce the risk of several cancers like colorectum, endometrium, esophagus, pancreas, bladder, breast, buccal cavity and pharynx, prostate, leukemia, and hepatocellular cancers [3].

Shelf-life is an important information in food for producers and consumers. Shelf-life is defined as a period of storage time until the product becomes unacceptable to consumers [4]. Shelf-life of the roasted and ground coffee at 1.0 kPa O₂ and 35°C determined by Weibull hazard methods is 22.5 weeks [5]. The shelf-life of brewed coffee at 20°C has been also determined by Manzocco and Lagazio [6] showing that brewed coffee kept for one day has significantly reduce in acceptability score due to sourness.
To ease the work of the barista, some companies like to prepare espresso (brewed coffee) in high amount and keep it refrigerated for further formulating products like coffee milk. However, during storage, there will be some changes in the espresso coffee which resulted in a reduction in the consumer acceptance. This reduction was also followed by physicochemical changes and antioxidant activity reduction. Therefore, this study will study the changes in physicochemical, antioxidant and sensory properties of espresso coffee during refrigerated storage to determine its shelf-life as an ingredient for coffee milk.

2. Materials and Methods

2.1. Materials
Coffee beans used were arabica coffee from Flores Bajawa. Coffee beans were ground into coffee powder using an automatic grinder (Eureka Zenith E65, Italy) and then extracted using an espresso machine (Nuova Simonelli Appia II, Italy), porta filter (Nuova Simonelli, Italy), tamper (Nuova Simonelli, Italy). Amount of 18 g of coffee powder was used to make 60 ml of espresso coffee using the espresso machine. The espresso coffee made was allowed to reach room temperature before refrigeration at 4°C for shelf life determination. Before analysis, espresso coffee was made into coffee milk by creamer and water addition and refrigerated for 2-3 h at 4-6°C.

2.2. Color and pH determination
Color analysis was determined using colorimeter (Portable Colorimeter 3nh NH310, China) with the results shown as CIE L*a*b*. Analysis of pH was conducted using digital pH meter (Eutech Instrument pH 700, United States) by following instrument’s guidelines.

2.3. Total phenolic analysis
Espresso coffee was first diluted for 100 times and then 0.5 ml of diluted espresso coffee was added with 2.5 ml of 7.5% Na₂CO₃ and 2 ml of 10% Folin Ciocalteu reagent [7]. After 30 minutes incubation, absorbance of the solution was measured using spectrophotometer UV-Vis at 765 nm. Gallic acid (0-200 ppm) was used to make standard curve.

2.4. Antioxidant capacity
Antioxidant determined on espresso coffee was FRAP (Ferric Reducing Antioxidant Power), the ability of the sample to reduce oxidized compounds [8]. The oxidized compound used in this analysis was FeCl₃. First of all, espresso coffee was diluted into 100x then 1 ml of diluted espresso coffee was added with 2.5 ml of 0.2 M phosphate buffer pH 6.8 and 2.5 ml 1% K₃Fe(CN)₆ solution. After 20 min incubation at 50°C, 2.5 ml of 10% trichloro acetate (TCA) were added, homogenized, and centrifuged 3000 rpm for 10 min. About 5 ml of supernatant was collected and added with 5 ml of distilled water and 1 ml of 0.1% FeCl₃. Absorbance was measured at 700 nm using spectrophotometer. Quercetin (0-200 ppm) was used to make standard curve.

2.5. Sensory analysis (triangle and hedonic tests)
Triangle and hedonic test were carried out using methods described by Meilgard [4]. Shelf life determinations of espresso coffee and coffee milk were done by triangle test. Hedonic tests were conducted to determine the acceptance of the product. About 8 trained panelists (barista) were asked to give the answer on triangle and hedonic test for espresso coffee and 30 untrained panelists were asked for coffee milk product. Hedonic test was conducted with the range answer of 1-5, which 1 represents really don’t like and 5 represents really like.

2.6. Statistical analysis
Results were shown as mean ± standard deviation from 3 replications. Analysis of variance (ANOVA) following Duncan’s post hoc test was conducted to observe the significant difference among sample groups at p<0.05.
3. Results and Discussion

3.1. The changes in color, pH and total phenolic contents and antioxidant capacity of espresso coffee and coffee milk during storage

Espresso coffee is an ingredient used to make milk coffee. The changes on physicochemical properties of espresso coffee is very important in shelf-life determination. The changes in physicochemical properties of espresso coffee observed from color $L^*a^*b^*$ value, pH value, total phenolic contents and antioxidant capacity. Moreover, these changes impact on the coffee milk produced was also observed from color $L^*a^*b^*$ and pH values.

The changes of espresso coffee’s and coffee milk’s color during storage can be seen in Table 1. Although there were no significant changes in espresso coffee during storage up to 8 days, the milk coffee showed significant changes during storage. It was suspected that espresso coffee is too dark to be measured by colorimeter, thus the color did not show significant changes.

Table 1. The changes in color values of espresso coffee and coffee milk during storage

| Storage time (day) | $L^*$     | $a^*$     | $b^*$     | $\Delta E^*$ |
|--------------------|----------|----------|----------|--------------|
| Espresso Coffee    |          |          |          |              |
| 0                  | 13.57 ± 0.52<sup>a</sup> | -9.49 ± 0.79<sup>a</sup> | -1.68 ± 0.07<sup>a</sup> | -          |
| 2                  | 13.64 ± 0.65<sup>a</sup> | -9.34 ± 1.51<sup>a</sup> | -1.62 ± 0.17<sup>a</sup> | 1.92        |
| 4                  | 13.40 ± 0.23<sup>a</sup> | -9.85 ± 0.59<sup>a</sup> | -1.69 ± 0.10<sup>a</sup> | 1.03        |
| 6                  | 13.25 ± 0.21<sup>a</sup> | -9.97 ± 0.45<sup>a</sup> | -1.67 ± 0.13<sup>a</sup> | 0.77        |
| 8                  | 13.07 ± 0.08<sup>a</sup> | -10.61 ± 0.46<sup>a</sup> | -1.75 ± 0.09<sup>a</sup> | 1.23        |
| Coffee Milk        |          |          |          |              |
| 0                  | 27.63 ± 0.66<sup>b</sup> | 7.57 ± 1.15<sup>a</sup> | 8.38 ± 1.61<sup>c</sup> | -          |
| 2                  | 32.05 ± 1.53<sup>b</sup> | 6.07 ± 0.10<sup>a</sup> | 10.74 ± 0.65<sup>b</sup> | 5.47        |
| 4                  | 46.04 ± 4.06<sup>a</sup> | 7.41 ± 0.80<sup>a</sup> | 14.68 ± 0.94<sup>a</sup> | 19.47       |
| 6                  | 50.37 ± 3.10<sup>a</sup> | 7.43 ± 0.60<sup>a</sup> | 14.62 ± 0.37<sup>a</sup> | 23.62       |
| 8                  | 47.37 ± 4.75<sup>a</sup> | 7.29 ± 0.90<sup>a</sup> | 14.57 ± 0.90<sup>a</sup> | 20.74       |

<sup>a,c</sup>Mean ± standard deviation in each column with the same letter are not significantly different ($p>0.05$) determined by Analysis of Variance with Duncan’s post hoc test

Milk coffee showed increase in lightness ($L^*$) value after 4 days storage and the intensity of yellow color increased after 2 days of storage. It may be due to degradation of melanoidin pigment produced during roasting coffee because of Maillard and caramelization reactions [9]. Table 2 shows the changes in pH value of espresso coffee and coffee milk during storage. The significant changes in pH value was found only in espresso coffee but not in milk coffee. For espresso coffee, there were increase in pH value during storage up to 4 days and interestingly it decreased afterwards. The lowest pH value was observed in espresso coffee stored for 8 days, while the highest pH value was observed in espresso coffee stored for 4 days. Rossa et al. [10] reported that coffee kept at 4°C for more than 10 days showed significant decrease in pH value; however, this study found significant reduction in pH value in espresso coffee storage for 8 days.

Table 2. The changes in pH value of espresso coffee and coffee milk during storage

| Storage time (day) | pH value of espresso coffee | pH value of coffee milk |
|--------------------|-----------------------------|------------------------|
| 0                  | 5.13 ± 0.01<sup>c</sup>    | 7.19 ± 0.01<sup>a</sup> |
| 2                  | 5.19 ± 0.02<sup>b</sup>    | 7.16 ± 0.02<sup>a</sup> |
| 4                  | 5.23 ± 0.01<sup>a</sup>    | 7.18 ± 0.02<sup>a</sup> |
| 6                  | 5.20 ± 0.01<sup>b</sup>    | 7.17 ± 0.01<sup>a</sup> |
| 8                  | 5.06 ± 0.02<sup>d</sup>    | 7.16 ± 0.03<sup>a</sup> |
The changes of total phenolic content and antioxidant capacity of espresso coffee is shown in Table 3. The total phenolic content showed significant reduction after 4 days of storage, while total antioxidant capacity showed significant reduction after 2 days of storage. The reduction of total phenolic content is commonly caused due to oxidation of phenolic content by polyphenol oxidase enzymes.

| Storage time (day) | Total phenolic content (mg GAE/ml) | Antioxidant capacity (mg QE/ml) |
|-------------------|-----------------------------------|---------------------------------|
| 0                 | 9.43 ± 0.20<sup>a</sup>           | 11.39 ± 0.04<sup>a</sup>       |
| 2                 | 8.98 ± 0.48<sup>a</sup>           | 11.04 ± 0.06<sup>b</sup>       |
| 4                 | 7.52 ± 0.20<sup>b</sup>           | 10.86 ± 0.07<sup>c</sup>       |
| 6                 | 7.77 ± 0.29<sup>b</sup>           | 9.64 ± 0.08<sup>d</sup>        |
| 8                 | 7.28 ± 0.24<sup>b</sup>           | 9.15 ± 0.03<sup>e</sup>        |

The increase in hedonic acceptance of coffee may due to the increase in pH value which may be caused by reduction of chlorogenic acid during coffee storage [11]. For hedonic rating of odor attribute, there was decrease in the hedonic rating in espresso coffee and coffee milk kept for 6 days. This is caused by evaporation of volatile compounds in espresso coffee during storage.

| Storage time (day) | Taste | Odor | Body | Color |
|-------------------|-------|------|------|-------|
| Espresso coffee   |       |      |      |       |
| 0                 | 3.40 ± 0.86<sup>ab</sup> | 3.90 ± 0.80<sup>a</sup> | 3.47 ± 0.90<sup>a</sup> | 4.03 ± 0.56<sup>a</sup> |
| 2                 | 3.27 ± 0.94<sup>ab</sup> | 3.77 ± 0.73<sup>a</sup> | 3.23 ± 0.82<sup>a</sup> | 3.93 ± 0.91<sup>ab</sup> |
| 4                 | 3.70 ± 0.84<sup>a</sup> | 3.63 ± 0.72<sup>a</sup> | 3.63 ± 0.89<sup>a</sup> | 4.13 ± 0.82<sup>a</sup> |
| 6                 | 3.17 ± 0.79<sup>b</sup> | 3.17 ± 0.79<sup>b</sup> | 3.53 ± 0.86<sup>a</sup> | 3.93 ± 0.74<sup>ab</sup> |
| 8                 | 2.93 ± 0.83<sup>b</sup> | 2.87 ± 0.57<sup>b</sup> | 3.63 ± 0.89<sup>a</sup> | 3.57 ± 0.63<sup>b</sup> |

Coffee milk

| Storage time (day) | Taste | Odor | Body | Color |
|-------------------|-------|------|------|-------|
| 0                 | 3.13 ± 0.64<sup>b</sup> | 3.63 ± 0.52<sup>a</sup> | 2.75 ± 0.71<sup>a</sup> | 3.88 ± 0.64<sup>a</sup> |
| 2                 | 2.38 ± 1.19<sup>bc</sup> | 3.13 ± 0.64<sup>ab</sup> | 3.13 ± 0.83<sup>a</sup> | 3.50 ± 0.53<sup>ab</sup> |
| 4                 | 4.00 ± 0.76<sup>a</sup> | 3.00 ± 0.93<sup>ab</sup> | 3.38 ± 0.92<sup>a</sup> | 3.13 ± 0.64<sup>bc</sup> |
| 6                 | 2.50 ± 0.76<sup>b</sup> | 2.63 ± 0.74<sup>b</sup> | 2.88 ± 1.13<sup>a</sup> | 2.88 ± 0.64<sup>bc</sup> |
| 8                 | 1.63 ± 0.52<sup>c</sup> | 2.38 ± 0.52<sup>b</sup> | 3.00 ± 1.07<sup>a</sup> | 2.63 ± 0.74<sup>c</sup> |

*<sup>a</sup>-<sup>c</sup>Mean ± standard deviation in each column with the same letter are not significantly different (<i>p</i> > 0.05) determined by Analysis of Variance with Duncan’s post hoc test
From triangle test conducted on trained panelists, correct answer from panelists only reached minimum required correct answer in the espresso coffee kept for 8 days (Table 5). The same results finding also can be seen in coffee milk result using 30 untrained panelists. The correct answer by untrained panelist reached minimum number of panelists to answer correctly after 8 day of refrigerated storage. Therefore, the shelf-life of espresso coffee in refrigerated condition was 6 days.

### Table 5. Triangle test results of espresso coffee and coffee milk

| Sample         | Number of panelists | Min. number of panelists to answer correctly | Number of panelists answered correctly |
|----------------|---------------------|---------------------------------------------|---------------------------------------|
| Espresso coffee |                     |                                             |                                       |
| Day -0 and Day-4 | 8                   | 7                                           | 6                                     |
| Day -0 and Day-6 | 8                   | 7                                           | 6                                     |
| Day -0 and Day-8 | 8                   | 7                                           | 8                                     |
| Coffee milk     |                     |                                             |                                       |
| Day -0 and Day-4 | 30                  | 15                                          | 15                                    |
| Day -0 and Day-6 | 30                  | 15                                          | 14                                    |
| Day -0 and Day-8 | 30                  | 15                                          | 17                                    |

*p<0.01 for espresso coffee (trained panelist) and p<0.05 for coffee milk (un-trained panelist)

### 4. Conclusion

The lightness of coffee milk made from espresso coffee after four-day storage significantly increased, which may due to degradation of melanoidin compounds produced during roasting. Although there was changes in pH of espresso coffee during storage, it did not significantly change the pH of coffee milk produced. Moreover, the reduction of antioxidant capacity can be observed from espresso coffee after 2 days storage, while the reduction of total phenolic content was observed after 4-day storage. Interestingly, the highest hedonic score was found in the espresso coffee and coffee milk stored for 4 day in refrigerator which may due to lower acidity observed by pH value. The shelf life of espresso coffee for making coffee milk was six days based on triangle test result.

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