Effects of fat content and grinding level of cocoa nibs on physicochemical characteristics of espresso cocoa

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Abstract
Grinding is an important factor in espresso coffee preparation and the optimal grinding level is needed to improve its characteristics. However, a problem arises where ground cocoa nibs change from solid to fluid mass at finer level due to high fat content (52%) in the cocoa bean. Thus, the aim of this study was to determine the effect of fat in Ivory Coast cocoa nibs and grinding level on particle size and the physicochemical characteristics including viscosity (mPa), pH, foam index (%), density (g/mL), total solid (mg/mL) and extraction (%) of espresso cocoa (ECOC). Solvent extraction was used to produce defatted cocoa nibs (40%, 34% and 20%) and was ground at four different grinding levels (i.e., 10, 30, 50 and 70) to extract a cup of ECOC using espresso machine. The grinding level 70 (1665.0±28.30 µm) contributed to significantly (p<0.05) coarsest particle size compared to the smallest grinding level 10 (340.0±21.20 µm). However, only grinding level 50 and 70 were selected as they contributed a good extraction of ECOC with targeted volume of 40 mL. Overall, ECOC with fat content of 20% at grinding level 50 produced significantly (p<0.05) the most viscous (5.63 mPa), the highest foam index (10.83%) the highest density (1.00 g/mL), the highest total solid content (24.36 mg/mL), and the highest extraction value (10.37%) of ECOC compared with another type of ECOC and control. This study is important to develop high quality ECOC as another alternative beverage for cocoa.

Keywords
Espresso, Espresso cocoa, Cocoa beans, Grinding level, Fat content, Particle size, Physicochemical characteristic

Introduction
An espresso is a concentrated brew, often thick beverage with a layer of dense foam, created with a machine by percolation of hot water (88-92°C) through a basket of tightly packed roasted ground coffee, for a limited amount of time at very high pressure approximately 9 bars (Navarini et al., 2004; Illy and Viani, 2005; Albanese et al., 2009; Bartel et al., 2015). The preparation technique has a significant influence on the taste, aroma and composition of coffee brews. A shot of espresso refers to the beans which is finely ground and usually tampered and placed in a portafilter followed by extraction of espresso using the espresso machine. The perfect shot shows noticeable spectrum of colours whereas light frothy top layer crema cascades down from light tan shades to darker brown. Other than Italy, the popularity of espresso coffee (EC) extraction method has been increased in many other countries around the world with more than 50 million cups of espresso are consumed daily (Illy and Navarini, 2011). This may influence the interest in using roasted cocoa nibs as espresso similar to EC. In addition, Vanderhoven (2007) claimed that it is possible to produce richly flavor beverage brewed using ground cocoa substrate with the additional portion of husk material.

Cocoa powder as one of cocoa-based product has been used in cocoa beverage production. During the production, grinding process has been done as one of the treatment. However, grinding converts nibs into a thick chocolate colored fluid paste, known as liquor which contain about half of cocoa butter (50–55% w/w) (Asep et al., 2008). Unlike coffee bean (Arabica and Robusta) with fat content of about 7–17% (Speer and Kollin-Speer, 2006), cocoa nibs are rich in fat content. Cocoa butter is usually removed using mechanical expression and solvent extraction. Usually, during grinding process of cocoa beans, heat will be produced thus change the nibs into a thick paste called cocoa liquor containing cocoa butter (Meursing et al., 1994). The process describe above
may interfere the grinding process of the roasted bean during the preparation of newly developed ECOC.

In espresso drink preparation, grinding plays an important role to produce optimal grinding grade for the obtainment of a good-quality EC. Numerous studies have been performed to evaluate the effect of grinding levels on the physicochemical characteristics of EC. Andueza et al. (2003), reported that smaller grinding size of coffee bean produces persistent foam, which is one of the characteristics of good quality EC. Roth (2010) claimed that the perfect shot will have a two millimeter-thick layer of crema or dense golden-brown foam on top. In addition, the viscosity of EC has been reported to be increased inversely with increased grinding size. The EC is more concentrated with smaller particle size and therefore becoming more viscous (Andueza et al., 2003). However, to the best of our knowledge, similar studies on the effect of fat in cocoa nibs and grinding level on ECOC have not been reported.

High fat content in cocoa nibs causes problem during grinding and tends to form paste thus partial removal of fat is believed able to facilitate the espresso preparation which also involves grinding. Therefore, the aim of this study was to determine the effect of fat in cocoa nibs and grinding on the physicochemical characteristics of newly developed concentrated espresso drink with improve espresso characteristics.

Materials and Methods

Samples and equipments

The Ivory Coast cocoa bean (Theobroma cacao L.) was supplied by Barry Callebaut Malaysia Sdn. Bhd, an international cocoa bean importer which located at Pelabuhan Klang, Selangor. Boncafe espresso gourmet ground coffee is made of roasted and blended Arabica coffee blended from Central America, South America and Africa. The coffee consists of individual packed containing 200g of coffee bean and hermetically sealed.

The equipments used to prepare ECOC were Rancilio Rocky Grinder (USA) and Rancilio Silvia Semi-Automatic Espresso Machine (USA). A grinder (Rancilio Rocky Grinder, USA) was used to grind the non-defatted and defatted roasted cocoa nibs. The grinding scale of the grinder ranged from 10 to 80 with 10 being the finest setting and 80 being the coarsest. During preliminary analysis, four different levels (10, 30, 50 and 70) were selected to study the grinding ability of all samples to be used for ECOC preparation. However, two grinding levels (50 and 70) were found to be suitable to grind non-defatted and defatted roasted local cocoa nibs and further proceed as a beverage. Particle size analysis for non-defatted and defatted cocoa nibs at each grinding level was compared using digital micrometer (Mitutoyo, Japan). The analysis was done in duplicate.

Preparation of ECOC for analysis

A total of 9.4 g of ground cocoa nibs was weighed
using a weighing balance (Sartorius, German) and was used to extract a cup of ECOC before filled into the portafilter. After the ground cocoa nibs had been compressed with a tamper, the ECOC was extracted using an espresso machine. A thermometer and a digital timer were used to maintain the extraction temperature and percolation period. Fixed tamping pressure (≤10 lbs) and percolation time (within 30 s) were used for ECOC preparation after the preliminary analysis. The water output temperature was fixed in the range of 80-90ºC. The ECOC preparation process was repeated three times for analysis.

**Volume, foam index, and density determination**

The volume and foam index of the ECOC beverage was determined immediately after the extraction process, and it was measured using a 100-mL graduated cylinder according to Anduenza et al. (2003). The volume was measured to determine the volume of ECOC and compared with targeted volume (40 mL). The foam index was defined as the ratio (%) of ECOC foam and the total volume of ECOC in millimetres. This value was compared with the foam index of EC. The density of ECOC was also determined by measuring the mass of 40 mL of ECOC. Density was calculated as follows by dividing the mass of ECOC with the 40 mL total volume.

\[
\text{Density} = \frac{\text{Mass (g)}}{\text{Volume (40 mL)}}
\]

**pH and viscosity measurements**

The pH of the ECOC was determined using an electronic pH meter (Jenway, UK), which was calibrated with buffer solutions at pH 4 and 7. The viscosity analysis of the ECOC beverage was also analyzed to compare with EC viscosity using a viscometer (Rheolab QC, Anton Paar, USA) at temperature 22ºC, and the results were expressed as dynamic viscosity \( \eta = \frac{\nu}{\delta} \) (mPa s).

**Total solids and extraction**

The total solids and extraction analysis was performed according to Bartel et al. (2015). This study is important in determining the building blocks of aroma, taste, and body of espresso drink. Total solids of ECOC were determined by oven-drying (Memmert, Germany) at temperature 102±3ºC for 18 hours. ECOC with 40 mL volume was dried in an oven until a constant weight was obtained. Extraction was determined from the percentage of total solids with respects to the 9.4 g of ground roasted cocoa dose to determine the percentage of ground cocoa nibs, by weight, which were dissolved in the water.

**Statistical analysis**

A one-way analysis of variance (ANOVA) was performed on all the tests, and Tukey’s test at a significance level of \( p<0.05 \) was utilized. A two-way analysis of variance (ANOVA) also was performed to test the interaction between two factors. ANOVA was performed using Minitab 16 software (Minitab Inc., USA). Correlation analysis was also applied to determine the significant effect between fat content and grinding level on physicochemical characteristics using SPSS 18 (PASW Statistics 18, Inc., USA).

**Results and Discussion**

The particle size of food powders is an important property as it affects the desired end use of product properties (Davies, 1984; Shittu and Lawal, 2007). Roasted cocoa nibs with different fat contents were ground at four different grinding levels (i.e., 10, 30, 50 and 70) to produce ECOC with good physicochemical characteristics. Table 1 shows particle size distribution for each grinding level where particle size significantly increases \( p<0.05 \) when grinding level increases. For example, grinding the cocoa nibs with a fat content of 40% at level 70 resulted in a larger particle size (1277.5 µm) than grinding the cocoa nibs at levels 10 (558.8 µm). Table 1 shows the fat content of cocoa nibs did affect the particle size distribution. The particle size of ground cocoa nibs with fat content of 40%, 34% and 20% at grinding level 10 shows significantly small \( p<0.05 \) in particle size which were 558.5 µm, 432.5 µm and 340 µm respectively. Furthermore, the fat content did affect the grinding ability of cocoa nibs where cocoa nibs with high fat content (52%, non-defatted) were unable to be ground at grinding level 10 and 30. In this study, grinding at 10 using ground cocoa nibs with fat content 20% shows the nearest particle size with the average particle size for EC which was 200-240 µm (Mahlkonig, 2011).

The performance of ECOC at each grinding level was also determined. Based on Anduenza et al. (2003), the targeted volume of ECOC that should be used in this study was 40 mL. Grinding level 50 and 70 (Table 2), which were the large particle size formation shows good extraction ability during the espresso preparation compared to other different fat content. Petracco et al. (2001), stated that the pH for EC is between 5.2 and 5.8. In this study, the pH of ECOC was found to be within the acceptable range for espresso drinks. The pH value of ECOC using non-defatted cocoa nibs (52%) at grinding level 70 shows significantly \( p<0.05 \) the highest pH value, 5.80, compared to defatted cocoa nibs (40%, 34%
The pH also gradually increased with the increased of grinding level. Foam index of ECOC gradually increased with the decreased of fat content in roasted cocoa nibs. However, only the foam index of ECOC using defatted cocoa nibs (20%) at grinding level 50 showed significant increment (p<0.05) which has the highest foam index (10.83%) compared to non-defatted cocoa nibs (52%) which was 6.68% (Table 3). The foam index decreased with the increased of grinding level. For example, significantly high foam index (p<0.05) was formed for ECOC with 20% fat of cocoa nibs between grinding level 50 (10.83%) and 70 (7.50%).

The foam formation happened as hot water is forced through the beans under pressure and extracts the natural oil or fat content in the bean, suspending it in tiny microbubbles of air. Therefore, there is mixture of tiny oil droplets in an aqueous solution which contained few components such as protein-like material, sugars, caffeine and acids. (Illy and Navarini, 2011). In addition, the foam is created as a direct result of carbon dioxide (CO₂) within the cell structure of coffee beans, which is created during the roasting process. These bubbles is then supported by melanoidin to form the foam (Illy and Navarini, 2011).

ECOC with non-defatted (52%) cocoa nibs do not performed good formation of foam index compared to the defatted nibs due to the high fat content. According to practical experience, whole milk has poor foaming behaviour compared to skim milk which has low fat content (Anderson et al., 1988). The foam formation indicates good extraction ability of espresso as high foam development contributes to the high quality of espresso. The foam formation indicates good extraction ability of espresso as high foam development contributes to the high quality of espresso. It is important to produce persistent foam as it is responsible for the visual acceptability of espresso drinks (Illy and Viani, 2005).

The density and viscosity of ECOC produced from roasted cocoa nibs with different fat content

Table 1. Particle size of non-defatted and defatted ground cocoa nibs at different grinding level

<table>
<thead>
<tr>
<th>Cocoa nibs fat content (%)</th>
<th>Particle size (µm)</th>
<th>Grinding Level</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10</td>
<td>30</td>
</tr>
<tr>
<td>52 (control)</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>40</td>
<td>656.8±1.77</td>
<td>663.9±6.30</td>
</tr>
<tr>
<td>34</td>
<td>432.5±17.70</td>
<td>441.3±15.93</td>
</tr>
<tr>
<td>20</td>
<td>340.0±21.29</td>
<td>437.9±7.68</td>
</tr>
</tbody>
</table>

*All values are shown as mean±standard deviations (n = 3)
Small letters a, b, c indicate significant different (p<0.05) among different cocoa nibs fat content at the same grinding level.
Capital letters A, B, C indicate significant different (p<0.05) among different grinding level at the same cocoa nibs fat content
N/A = Not applicable
and from different grinding levels were determined. Density was a physical property of matter (substance present in ECOC), as it measures the heaviness of objects at a constant volume (Maclin et al., 1997). Table 3 also shows that the density of ECOC made from roasted cocoa nibs with different fat contents and grinding levels ranged from 0.96 g/mL to 1.00 g/mL. Anduenza et al. (2003), stated that the density for EC was between 1.010 g/mL to 1.014 g/mL. In this study, the density of ECOC was found to have the similar acceptable range for espresso drinks. However, significant different of density (p<0.05) was only observed between control (52%, fat) at value 0.98 g/mL and defatted nibs at 20% fat content (1.00 g/mL). For grinding level, significant different (p<0.05) was found at cocoa nibs with fat content 34% and 40% between grinding level 50 and 70.

Viscosity is an important characteristic for food application as it contributes to the texture or feeling in the mouth when a product was eaten. A similar pattern with the percent of foam index was observed in viscosity of ECOC where grinding level 50 shows high viscosity compared to grinding level 70. Table 3 shows that the most viscous ECOC (5.63 mPa) was obtained at grinding level 50 using defatted cocoa nibs (20%). It shows significantly (p<0.05) high viscosity of ECOC than that at the grinding level of 70 (4.47 mPa). This study was consistent with Anduenza et al. (2003), who reported that fine blends of Arabica:Robusta:Robustatorrefacto produce the most viscous EC (1.30 mN/m²s) and that the coarse blends produce the least viscous EC (1.19 mN/m²s). This is due to smaller particle size of cocoa nibs will produce large surface extraction and increase the extraction rate of cocoa bean soluble solids (Daniel Ephraim, 2010). Therefore, a higher number of soluble solids brewed results in more particle-particle interactions and increased resistance to flow that increase the viscosity. Table 3 also shows the initial viscosity of ECOC at grinding level 50 using cocoa nibs with fat content 52% (non-defatted) was significantly (p<0.05) increased from 4.87 mPa to 5.63 mPa (20%).

According to Anduenza et al. (2003), the extraction value decreased when the particle size increased for EC beverage. For example, Table 3 shows that ECOC extraction value at grinding level 70 with 52% fat was significantly (p<0.05) decreased (7.16%) from grinding level 50 (10.37%). A significant difference (p<0.05) also was found in ECOC produced from different fat content at grinding level 50 where extraction value produced from cocoa nibs with 20% (10.37%) was higher compared to 52% (control) fat of ECOC (6.71%).

In this study, it was shown that the extraction percentage value of ECOC made from roasted cocoa at different fat content and grinding levels ranged from 3.74% to 10.37% (Table 3). The most acceptable percentage of extraction for EC was from 18 to 22%, whereas below 16% was considered to be underdeveloped and above 24% was over extracted (Lingle, 1996; Anduenza et al., 2007). However, the extraction value of ECOC was found not to be within the acceptable range for espresso drinks and was considered as underdeveloped. This might be due to a larger particle size of ground cocoa nibs used in the present study to brew ECOC (Table 1) compared to EC. Small particle size increases surface area therefore allows a greater additional contact between coffee grounds and hot water (Bladyka, 2015). Smaller particles also help to reduce the distance from the center of each particle thereby increasing

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**Table 3. Physicochemical characteristics of ECOC produced from roasted cocoa nibs at different fat content and grinding level**

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Grinding level</th>
<th>Cocoa nibs fat content</th>
<th>Reference value*</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50</td>
<td>70</td>
<td></td>
</tr>
<tr>
<td><strong>pH</strong></td>
<td>6.72±0.04**</td>
<td>6.62±0.08**</td>
<td></td>
</tr>
<tr>
<td>Foam index (%)</td>
<td>6.68±0.29**</td>
<td>6.52±0.76**</td>
<td></td>
</tr>
<tr>
<td>Density (g/mL)</td>
<td>0.96±0.01**</td>
<td>0.99±0.01**</td>
<td></td>
</tr>
<tr>
<td>Viscosity (mPa)</td>
<td>6.43±0.29**</td>
<td>6.32±0.68**</td>
<td></td>
</tr>
<tr>
<td>Total solid</td>
<td>18.71±0.11**</td>
<td>16.36±0.30**</td>
<td></td>
</tr>
<tr>
<td>Extraction (%)</td>
<td>6.71±0.22**</td>
<td>6.75±0.13**</td>
<td></td>
</tr>
</tbody>
</table>

*All values are shown as mean±standard deviations (n = 3)

Small letters a, b, c indicate significant different (p<0.05) among different grinding level at the same cocoa nibs fat content. Capital letters A, B, C indicate significant different (p<0.05) among different cocoa nibs fat content at the same grinding level.

* Citation from literature.
extraction of oils, solubles, and other flavoring materials (Lingle, 1996).

The physicochemical characteristics of ECOC were analyzed using two-way ANOVA analysis to determine if there is an interaction between fat content in cocoa nibs and grinding level (Table 4). In this study, each fat content and grinding level significantly affected (p<0.05) the particle sizes and other physicochemical characteristics of ECOC such as pH, foam, density, viscosity, total solid, extraction and concentration. However, only particle size of cocoa nibs showed significant interaction (p<0.05) towards both the fat content and grinding level.

Table 5 shows the correlation between the physicochemical characteristics of ECOC, different fat content and grinding levels. The selected fat content of cocoa nibs had significant effects (p<0.05) on most of the ECOC characteristics (total solid, extraction percentage and concentration) except for viscosity. The fat content was negatively correlated (r=-0.528) with foam index of ECOC where the decrease of fat content of cocoa nibs led to high foam index formation of ECOC. Similarly, the density of ECOC were negatively correlated (r=-0.639) which referred to lower grinding level used in this study produced a higher density of ECOC. In the case of ECOC viscosity, the finer or lower grinding levels resulted in a higher viscosity of ECOC (r=-0.892).

The fat content was negatively correlated (r=-0.528) with foam index of ECOC where the decrease of fat content of cocoa nibs led to high foam index formation of ECOC. Similarly, the density of ECOC were negatively correlated (r=-0.639) which referred to lower grinding level used in this study produced a higher density of ECOC. In the case of ECOC viscosity, the finer or lower grinding levels resulted in a higher viscosity of ECOC (r=-0.892).

**Conclusion**

The different fat content (52%, 40%, 3% and 20%) and grinding levels (50 and 70) of roasted cocoa nibs, significantly (p<0.05) affected the particle size of cocoa nibs and ECOC physicochemical characteristics, including viscosity, density, total solids and extraction of ECOC. However, statistically (two-way analysis of variance) shows that only particle size had significant interaction (p<0.05) on both factors. In addition, the physicochemical characteristics, including pH, foam index, density, total solids and extraction were significantly correlated (p<0.05) with the grinding levels of 50 and 70 used in this study. ECOC brewed using cocoa nibs with 20% fat content at the grinding level of 50 with the smallest particle size (836 µm) of cocoa nibs showed better characteristics and produced significantly (p<0.05) the highest foam index (10.83%), total solids content (24.36 mg/mL), extraction value (10.37%), high in density (1.00 g/mL), and the most viscous (5.63 mPa) ECOC compared to control. It is also recommended to use cocoa nibs with low-fat content and grind it at a lower level to increase the foam index, density, viscosity and extraction of ECOC.

**Table 4. Effect of Grinding level and cocoa nibs fat content on particle size and physicochemical parameters of ECOC (F and p values)**

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Grinding level: A</th>
<th>Cocoa nibs fat content: B</th>
<th>Interaction: A x B</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>F</strong></td>
<td><strong>p</strong></td>
<td><strong>F</strong></td>
<td><strong>p</strong></td>
</tr>
<tr>
<td>Particle size</td>
<td>3885.92</td>
<td>0.000***</td>
<td>0.000***</td>
</tr>
<tr>
<td>pH</td>
<td>26.44</td>
<td>0.000***</td>
<td>0.70</td>
</tr>
<tr>
<td>Foam</td>
<td>17.19</td>
<td>0.001***</td>
<td>1.77</td>
</tr>
<tr>
<td>Density</td>
<td>30.39</td>
<td>0.000***</td>
<td>0.57</td>
</tr>
<tr>
<td>Viscosity</td>
<td>312.20</td>
<td>0.000***</td>
<td>0.18</td>
</tr>
<tr>
<td>Total solid</td>
<td>150.76</td>
<td>0.000***</td>
<td>0.27</td>
</tr>
<tr>
<td>Extraction</td>
<td>150.80</td>
<td>0.000***</td>
<td>0.14</td>
</tr>
</tbody>
</table>

**Table 5. Correlation between cocoa nibs fat content, grinding level and physicochemical characteristics of ECOC**

<table>
<thead>
<tr>
<th>Characteristics</th>
<th>Fat content</th>
<th>Grinding level</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>0.579**</td>
<td>0.630**</td>
</tr>
<tr>
<td>Foam Index (%)</td>
<td>-0.618**</td>
<td>-0.528**</td>
</tr>
<tr>
<td>Density (g/mL)</td>
<td>-0.588**</td>
<td>-0.639**</td>
</tr>
<tr>
<td>Viscosity</td>
<td>0.365</td>
<td>-0.892**</td>
</tr>
<tr>
<td>Total Solid (mg/mL)</td>
<td>-0.561**</td>
<td>-0.702**</td>
</tr>
<tr>
<td>Extraction (%)</td>
<td>-0.561**</td>
<td>-0.702**</td>
</tr>
</tbody>
</table>

**Correlation significant level at p<0.01**
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