EXPLORING THE USE OF MICROWAVE IN COCOA BEANS MICRONIZING – ASSESSMENT OF SELECTED PHYSICAL AND MECHANICAL PROPERTIES, DRYING KINETICS AND BIOACTIVE COMPOUNDS

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ABSTRACT - *This study explains the use of microwave in conducting cocoa beans micronizing as opposed to the standard infrared dry beans micronizing that is widely used in the industry. The effect of microwave in reducing the moisture content, the drying kinetics profiles, and the mechanical properties of the beans after this treatment and the amounts of remaining bioactive compounds were studied. Four different moisture levels (43.54% - 12.00%) of cocoa beans were prepared and used to provide the micronizing profiles spectrum using 500 Watts of microwave power with 12 minutes micronizing time. The results showed that after micronized, the 12.00% moisture content of the beans became 7.52%, a reduction of more than 37% of moisture. The results also show the final temperature of the beans were between 89.2ºC to 136.5ºC. The drying rate shows a significant amount of moisture was removed in the 12.00% wet beans moisture content compared to the rest. The mechanical properties of the beans after the micronizing treatment represented by the modulus of rupture (MOR) was in the range of 169.16±45.3 to 69.87±13.1. Statistically, there was no significant differences in the remaining amount of total polyphenols and its sub-class compounds in the dried samples. This study provides some insight and determining factors in how microwave can be used in cocoa beans micronizing to intensify the cocoa processing steps.*

Keywords: bio-active compounds, cocoa beans, mechanical properties, micronizing, microwave

INTRODUCTION;

Cocoa processing involves the usage of thermal energy. There are two steps where heat is applied; that is in drying and micronizing. The drying process is conducted at farm level, while micronizing is conducted at factory level. The purpose of drying is to dry the beans until it's reached a level for safe storage. Micronizing process on the other hand is to ensure effective separation of the shells and the nibs. The purpose of micronizing is to give the cocoa beans a thermal shock in order to expand the beans and loosen the shell from the nibs (Beckett, 2009). The beans will normally appear puffy after the micronizing treatment. The infrared burner is widely used in the cocoa bean processing factory for this purpose. The standard procedure of micronizing is by heating the dry beans to 165ºC for two to three minutes, although the temperature and the duration might vary depending on the beans condition (Mulato, 2012). The heat will cause the beans to expand and this is known as 'puffing effect' of the beans, which mainly due to the quick evaporation of water between the shell and the bean. The 'puffed' beans are the physical condition of the beans, which will ensure effective separation of the shells and the nibs. If the micronizing is poorly executed, the beans will be poorly broken and the amounts of unbroken beans will be higher. Higher percentage of unbroken beans will affect the processing quality as well as delaying the processing time. On the other hand, the cocoa bean moisture content which stood at 7.5% at the beginning of the micronizing process will be reduced due to the heating and drying effect. Typically, the micronized bean moisture content is reduced to 3.9±0.5% (Anshary, 2015). Factors such as temperature, moisture and drying rate would affect the beans mechanical strength separation performance and the final moisture contents of the nibs. Similarly, the other valuable anti-oxidants compounds such as total polyphenols and other phenols which is heat sensitive will be affected (Andújar, *et al*., 2012).

However, the industry need is changing and more reliable, fast and clean techniques are required to process the beans. The main challenge of improving the quality of cacao beans is to find an easy and fast ways to process the wet cacao beans to become dried quality cacao beans for downstream product usage. Lately, the use of microwave for drying agricultural products are gaining momentum. This is due to the way the microwave drying works, whereby the heating are achieved through volumetric heating that drives the drying process. Other advantages of microwave drying includes a non-contact drying, pollution free and fast distribution of thermal energy. Furthermore, moisture and temperature distribution are two determining factors that affects the total polyphenols contents. Similarly, these two factors also affect the mechanical properties of the beans. In terms of drying effectiveness, the energy efficiency in drying is closely related to drying times and drying rate, which later leads to the drying efficiency of the product. The advantage of microwave drying have been reported in other agricultural produce (Figiel, 2009; Hathazi *et al*., 2019; Heydari, Kauldhar, & Meda, 2020; Prakash & Srivastava, 2015), with mostly reported a significant reduction in drying times.

Apart from its nutrients, pleasant flavor, aroma, and color, cocoa are also known for offering many health benefits (Andújar et al., 2012; Pucciarelli, 2013), because it is an excellent source of antioxidants (Carrillo *et al*., 2013; Othman, *et al*., 2007). Many different bioactive compounds are present in cocoa, such as polyphenols, flavonoids and methylxanthines (caffeine and theobromine), among others. These phytochemicals can be present at different concentrations depending on diverse factors like cocoa variety and cocoa processing, which can lead to the presence of new bioactive compounds (Quelal-Vásconez *et al*, 2020).

The purpose of this study is to explore the use of microwave in heating and partially applying heat treatment to the wet cocoa beans to see the potential of these techniques as an alternative in micronizing process. Therefore, this study was designed to explore the possibility of using the microwave in cocoa processing steps, especially to intensify the process flow since microwave was known for its rapid and quick moisture removal. The effects of microwave on the bioactive compounds were also determined as one of the responds factor in order to add value to the finish products.

MATERIALS AND METHOD

Sample preparation

Fresh fermented beans were obtained from Stesen Penyelidikan dan Pembangunan Koko Jengka Pahang. The samples were stored in a chiller (-30ºC) before used. The wet beans moisture content of the sample was determined using AOAC (2000) method. A total of 200 gm of fermented beans was used as a sample in every batch of study cycle. The initial wet beans moisture contents were derived from weight reduction method. The amounts of moisture to be removed in order to get the intended moisture was pre calculated and once the weight reached the amounts the drying stop and the sample moisture were calculated using the AOAC method. The experiments were conducted in three replications and the average value of the initial moisture contents were reported.

The drying experiment

An experimental set-up unit known as the microwave laboratory dryer (MLD) has been specially fabricated for this study as shown in Figure 1. The unit was designed to allow better propagation of microwave electromagnetic fields. In addition, the MLD was equipped with power regulator and a circulator unit to absorb any reflected waves. The MLD has a load cell which allows uninterrupted weight reduction data recording in order to increase the drying data accuracy. Teflon circular pan, measuring 90 mm diameter and 28 mm thickness was placed on top of the load cell. Another important feature from this unit comes in the form of an infrared thermocouple.

Figure 1: The microwave laboratory dryer experiment set up

The infrared thermocouple allows for a better temperature profiles data acquisition, in which the temperature data collection can be free from electromagnetic field interference. The magnetron is rated at 1.2 kW and the system was equipped with a circulator system. The circulator system diffuses any reflected waves and protects the magnetron generator unit. The cavity of the MLD has a 310 mm depth by 315 widths and 180 mm height dimensions. A stirrer, made from stainless steel in the shape of fan blade was fixed at the top of the cavity and rotated at 5 rpm. The function of the stirrer is to distribute the electromagnetic waves in order to achieve the uniform distributing pattern. An axial box fan, which can generate 150 to 200 m^3/hr of air flow, is added to the system to drive out surrounding moisture in the cavity.

Drying experiments

The drying process was conducted in the MLD experimental setup. Weight loss of the sample is determine at every 3 minutes interval. Since the MLD is complete with analytical balance, the sample was not draw out at the time interval. The experiments was conducted at three replications and means value was used in the data. The amount of moisture removal was plotted against the time and represented as drying rate curve.

Dried beans strength

Modulus of rupture (MOR) also known as flexural strength, or bend strength, or transverse rupture strength is a material property, defined as the stress in a material just before it yields in a flexure test. This mechanical property is widely used to determine the strength of the materials and widely used in the woods study (Ouertani *et al*., 2018; Zhu & Kaliske, 2011). The effect of MLD on dried beans mechanical properties in term of its mechanical strength and its fractural strength were determined by using a texture analyzer (Stable Microsystems TA.XT plus, UK) with a compression test mode at test speed of 2.0 mm s^{-1} and penetration distance of 10 mm. The result was given in force-deformation curve (force versus time) and the value for fracturability was calculated. The first peak from the curve indicated the fracturability point and hardness was on the maximum peak. The texture measurement was conducted on 30 pieces of cocoa beans samples and the average value was reported.

Total polyphenols determination

The analysis was carried out using defatted sample. The amounts of total polyphenols were determined using a method described by Markham and Bloor (1998). The total polyphenol content was determined by the spectrophotometric method of Folin-Ciocalteu. The sample was ground in a high speed laboratory mill until its particle size was reduced to approx 90µm. The mill cocoa samples (1 g) will be extracted with 100 mL of acetone:water (70:30; v:v) under reflux at 60ºC for 2 hours. Acetone was removed under vacuum at 30ºC. The analysis was performed using UV-Visible Spectrometry Shimadzu. The blank was used as a reference cell, the slid width 0.2mm with 765nm of wavelength. The cuvette was used as a sample container which was transparent to the electromagnetic radiation. The standard was prepared by diluting gallic acid.. The result was expressed as gallic acid equivalent on a dry weight basis.

Preparation of extracts for epicatechin and catechin determination

Quantitative HPLC preparative method was used for the catechin and epicatechin determination. Defatted cocoa bean samples were extracted with 80% aqueous acetone (80 ml) for 30 min at 50°C using a sonicator. The mixture was then filtered through a filter paper (Whatman No. 4) and the residue of glassware was washed with 80 % aqueous acetone and total volume of filtrate was made up to 100 ml in a volumetric flask. Ten mL of extract was dried on a rotary evaporator at 45°C. The extraction obtained was suspended with 5ml deionised water. For following filtration, SPE cartridge C18 was preconditioned using 5 ml methanol and continuously preconditioned by 5 ml deionised water. Then, the pooled extract was injected through the SPE cartridge and the resulting residue was discarded. The epicatechin and catechin, which was retained by the SPE cartridge was eluted with about 10 ml of 40 % aqueous MeOH. The final volume elute was made up to 10 ml in a volumetric flask. 1.5 ml of final extraction was filtered through a nylon filter into a vial 10 microliter of this final solution was injected during the HPLC analysis.

RESULTS AND DISCUSSION

Temperatures profiles

The profiles of temperature were steep for the first three minutes, but eventually show a flatten curve towards the end of 12 minutes. This could be due to the presence of abundant moisture in the samples and heating was initiated by the electromagnetic fields. Samples exposed to microwave power rapidly develops a high internal temperature due to the amounts of water in the beans that reacts to the water ionized polarity (Ibrahim & Osman, 2012) Higher wet beans moisture provides gradual and moderate temperature profiles. In the first three minutes, the temperatures rise in the beans was constant amongst the different beans moisture, except for the 12.00% bean samples where the temperature progression was steeper as shown in Figure 1.

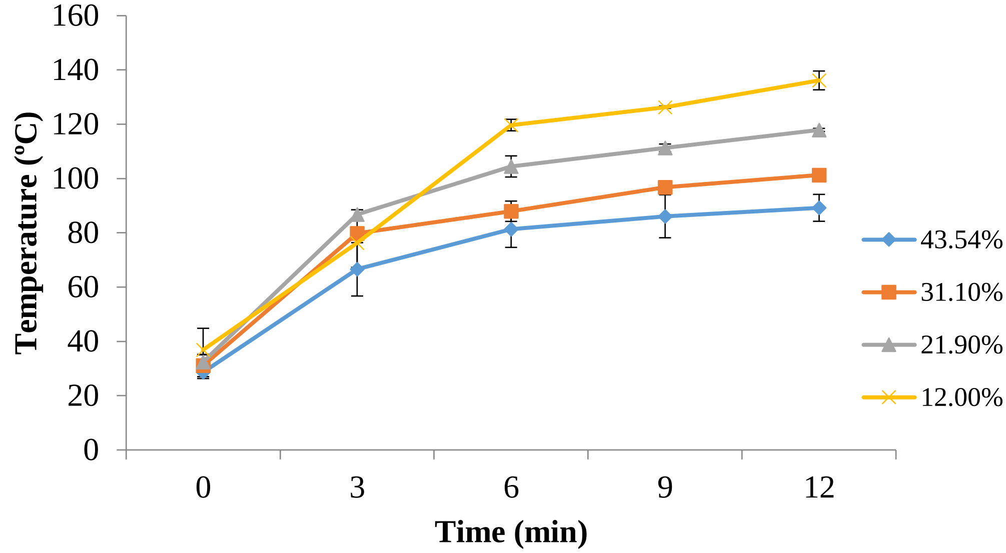
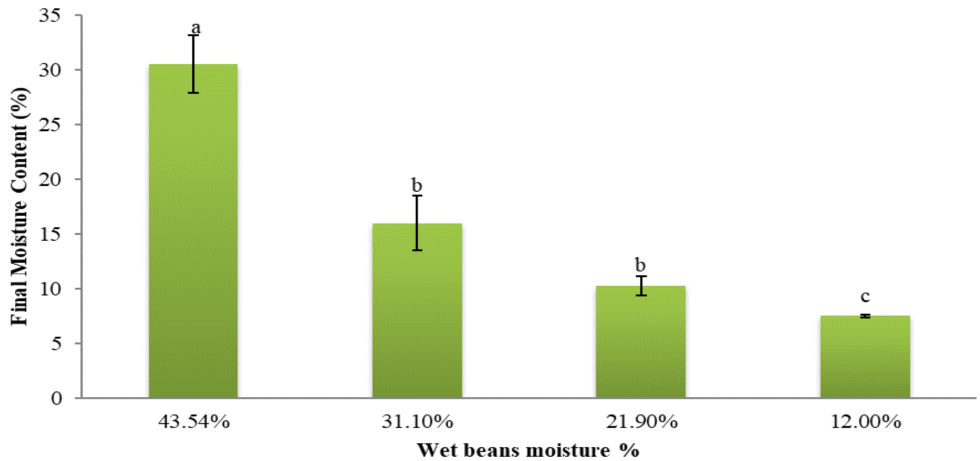


Figure 1: Temperature profiles during wet beans micronizing

Clearly, the lower wet beans moisture gave impact on the final temperature of the beans. The temperature of the beans was rapidly increasing in the 12.00% wet bean moisture due to low moisture content in the beans. Therefore, as the energy is applied, all the moisture was easily loss to the surrounding due to the weak bond between the molecules in the cocoa beans (Uzel, 2018). In electromagnetic heating, the temperature profile closely follows the power absorption distribution where more electromagnetic power exposure results in more heat dissipation amongst the samples and therefore leads to temperature rises (Scaman & Durance, 2005). On the other hand, in microwave heating, the volumetric heat absorption depends on the electromagnetic frequency, thickness of the object and the dielectric properties (Hossan & Dutta, 2012). Upon completion of the 12 minutes exposure of microwave electromagnetic waves, the product temperature was 136.15±3.46ºC for 12.00% wet beans moisture. For 21.90% wet beans moisture, the temperature was 117.9±0.56 ºC. In the other two wet beans moisture, 31.10 and 43.54% shows a final beans internal temperature of 101.3±0.00ºC and 89.2±4.94ºC, respectively. The temperature distribution along the propagation direction is greatly influenced by the wave frequency. For lower frequency, the temperature distribution was more uniform, but the variation in temperature was observed for the microwave heating with alternate hot and cold spots especially for the domestic microwave oven (f=2450 MHz) heating (Hossan & Dutta, 2012).

Final moisture content

Moisture content is one of the important determining factors in heating, drying, roasting as well as micronizing in cocoa beans processing. In normal micronizing practice, the moisture content after the micronizing treatment was kept at below 3.9+0.5%. The mechanical and thermal properties of the beans are affected due to the moisture content. The amount of final moisture contents of the beans after the micronizing treatment was as shown in Figure 2. The moisture reduction was found to be reduced correspondingly to its wet beans moisture content samples.



Values are mean ± standard deviation of three replications. Mean value with same superscript letters do not showed significant different ($p < 0.05$)

Figure 2: Final moisture content of the beans

After the exposure of the electromagnetic at 500 Watt power for 12 minutes, the final moisture contents were 30.49+2.46 and 16+2.51 for 43.54 and 31.10% wet beans moisture respectively. Whereas, for the 21.90 and 12.00% wet cocoa beans the final moisture content were 10.29+0.86 and 7.52+0.11, respectively. The amount of final moisture content relied heavily on the product temperatures. High temperature indicates more conversion of water to vapor pressure and thus provide an effective moisture removal. The highest moisture removal in this study was in the 31.10 and 21.90% wet beans samples, where close to 50% amount of water reduction were removed. Microwave energy affects the entire volume of the material, and due to its peculiar volumetric heating, the water was removed from the layers of the seed core to outside. Bouraoui *et al.* (1993), reported that a high MW power level (ca. 800 W) could cause overestimation of moisture content in some seed samples. This may probably be due to the development of high vapor pressure and restriction of vapor flux flow from inside to outside of the seed. The other reason could be due to the sudden change of physio-chemical characteristics of grains such as rice (Jafari, *et al.* 2018). The moisture determination was also related to the development of final product temperature and the amount of vapor diffusion from inside to outside. Therefore, the moisture content of the beans could be inaccurate.

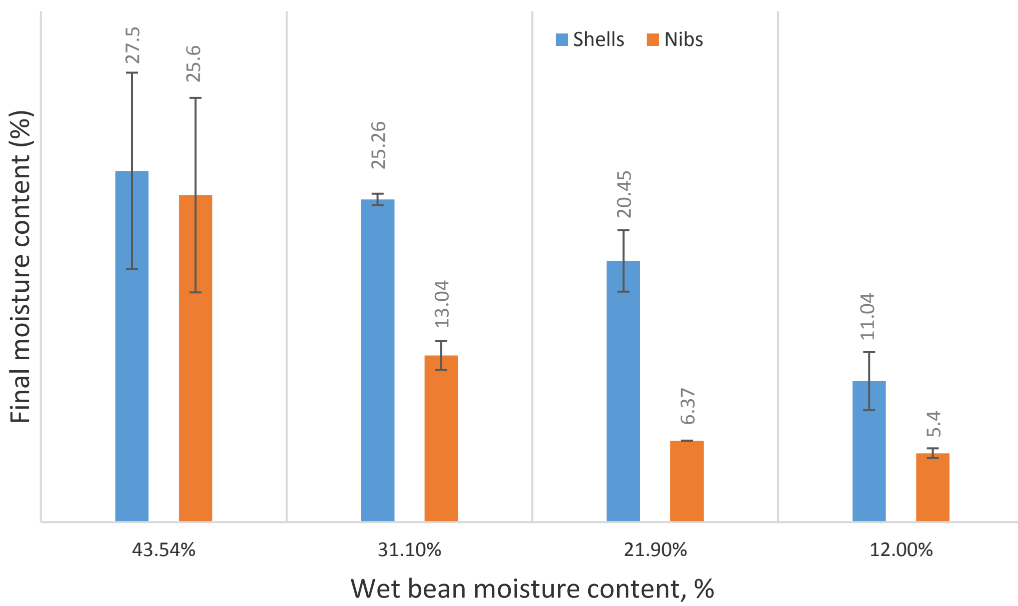


Figure 3: Final moisture content of shells and nibs

Figure 3 shows the moisture contents of the shell and nibs. A distinct differentiation can be seen between shell and nibs moisture contents. This suggests an evidence that the volumetric heating occurred in the nibs, thus the nibs moisture content was much lower than the shells. It also suggests that the accumulation of moisture to the outer layer of the beans were due to the intense vapor pressure that occurred inside the beans. The high variation of the final moisture contents in the 43.54% wet beans sample was perhaps due to the formation of hot and cold spot in the sample. The abundant amount of moisture and the inability of microwave to produce uniformity drying are one of the disadvantages of microwave drying. Despite of the installation of waveguides and mixers in the preparation in the experiment set up, the drying process was non-uniform due to the presence of hot spots. The amount of nibs moisture content was 5.4±0.38 for the 12.00% wet beans moisture. Whereas for 21.90% wet beans moisture, the nibs final moisture was 6.37±0.01. These two samples of wet moisture contents exhibit potential in conducting beans micronizing using electromagnetic due to two factors. The first factor is the final nibs moisture which was close to the requirement for effective micronizing. The other factor is the difference between the nibs and shell moisture, which implies the presence of voids between the shell and nibs, which are crucial for the separation of shells and nibs in latter processing step. Even though in the 31.1% wet beans sample the gap between shells and nibs final moisture content was high, the final moisture contents of the nibs were not close to the requirement of effective micronizing (3.9±0.5%).

Drying rate profiles

Drying rate profiles of the experiment are as shown in Figure 4. The drying rate exhibits a falling rate period just like most of other agricultural products. At the onset of the drying, the microwave energy was used to heat the moisture in the beans. This result in slowest progression of drying rate as shown in 43.54, 31.10 and 21.90 % wet beans sample. This phase requires the microwave energy to increase the temperature of the products until its reach evaporation state. From here, the vapor started to move to the outer layer of the beans and thus initiates the moisture removal process. The rates of moisture removing dictates the characteristics of the drying kinetics of the product. The highest drying rate of samples was in the range of 0.83 to 1.32 kg of water/kg of dry matter for 43.54 to 21.1% beans moisture, which happen at the end of the 12 minutes duration. However, the 12.00% beans moisture samples show a different drying rate path and the highest drying rate was at 9 minutes with 3.86 kg of water/kg of dry matter. This could be due to the limited amount of water available in the samples, thus the conversion of water to vapor were more rapid. In microwave drying, the amount of water presents largely affects the microwave absorption properties and therefore, the presents of high moisture serve as an advantage for higher moisture removal. Similar studies on other agricultural products also reported a decreased trend of drying rate towards the end of drying process (Firihu & Sudiana, 2016; Gursoy, *et al*. 2013). Drying rate normally exhibit a higher rate at the first one hour in convection mode of drying and the rate decreases with time. Similarly, the microwave drying also show a similar pattern but at a much rapid and fast time frame.

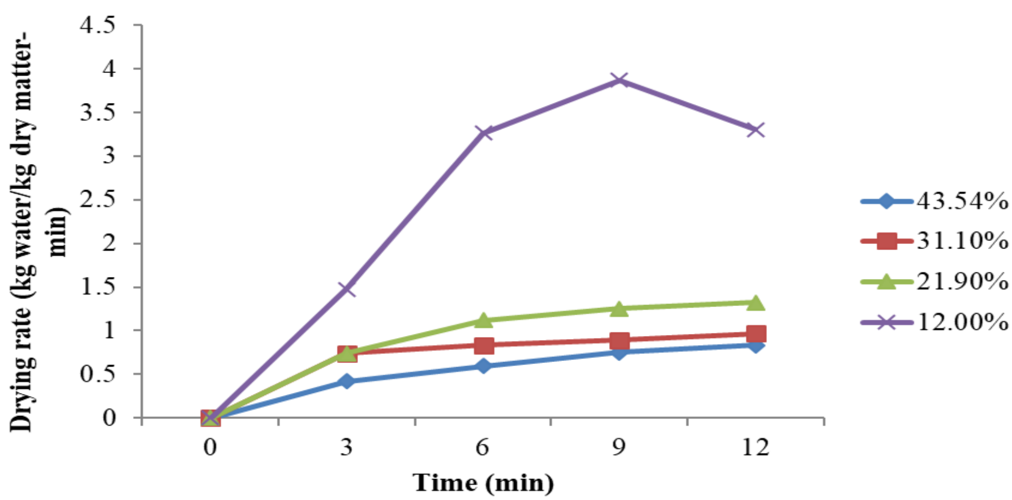
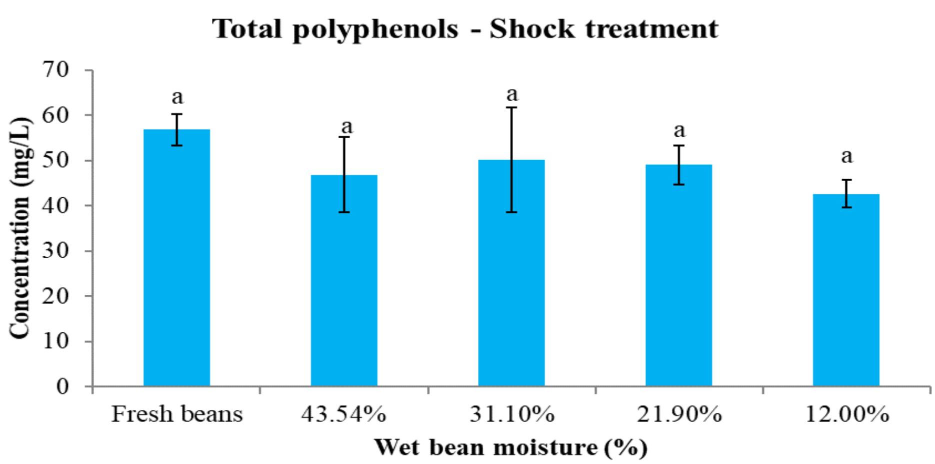


Figure 4: Drying rate of micronized wet beans

Changes in total polyphenols

Polyphenols are the most relevant bioactive cocoa compounds found to date. Polyphenol loss is generally ascribed to multiple factors, such as diffusion of soluble polyphenols into fermentation sweating, enzymatic oxidation, and non-enzymatic oxidation, the latter happened especially during sun drying. Most of polyphenols (80-90%) were lost during the first 48 hours but the remaining amount can be considered high (Albertini et al., 2015). Figure 5 shows a remaining amount of total polyphenols in this study. For comparison, the amount of total polyphenols in the fresh beans were determined and serve as a reference. The remaining amount of total polyphenols in the fresh beans (after fermentation) was 56.79±3.48 mg/L. The least amount of total polyphenols was 42. 57±3.02 mg/L, found in the 12.00% wet beans sample. The other remaining amount of total polyphenols were 46.85±8.25, 50.16±11.58 and 49.02±4.32 for 43.54, 31.10 and 21.90% wet beans moisture, respectively. Despite of the differences in the amount of the remaining total polyphenols, the amounts were found to be not significantly different. What causes the insignificant amount of total polyphenols reduction is unclear, but rapid drying and instant conversion from water to vapor could cause the total polyphenols compounds to remain in the beans. The other reason could be due to the non-uniformity in drying, where the total polyphenols compounds could remain in the cold spot areas. For the purpose of preserving the total polyphenols compounds, this could serve as an advantage. This study also suggests if microwave were to be used in the wet beans micronizing, the amount of the total polyphenols compounds presents in the nibs could be an additional value especially for the usage in the nutrition and health supplementary finish products. The high products value derived from the wet beans micronizing could compensate for the high production cost.



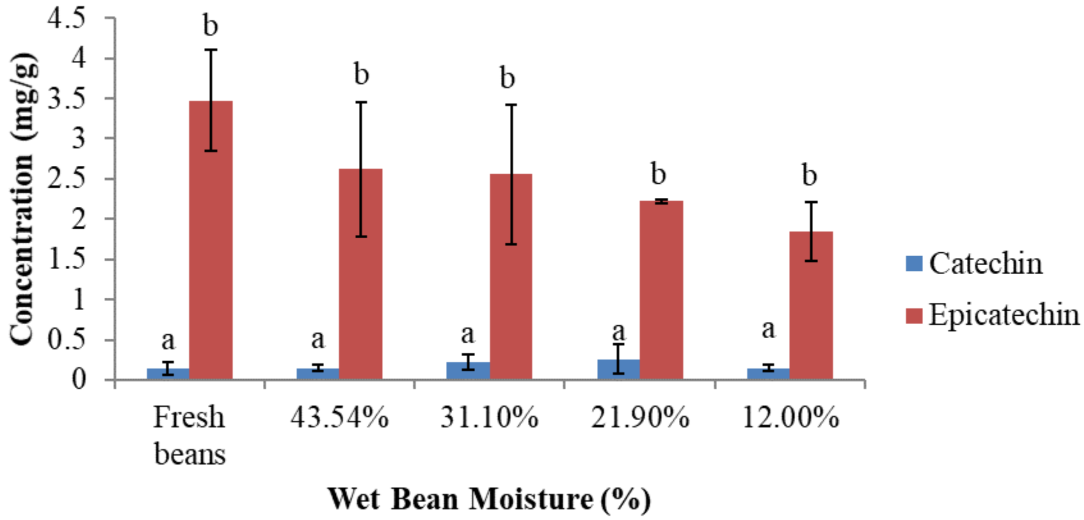
Values are mean ± standard deviation of three replications. Mean value with same superscript letters do not showed significant different ($p < 0.05$)

Figure 5: Changes in total polyphenols content

Changes in catechin and epicatechin

Polyphenols and especially epicatechin, the most abundant compound of the catechin family, was surely present in larger amount in fresh seeds. In this study, the amount of epicatechin was found higher compared with catechin as shown in Figure 6. It can be seen that the loss of epicatechin was proportionate to the wet beans moisture samples. The highest amount of epicatechin was 2.62$\pm$0.83 mg/g of the 43.54% wet beans sample. There were slight reduction of epicatechin content of the 31.10% wet beans sample, where the amount of epicatechin was 2.55$\pm$0.86 mg/g. In these two samples, the reduction of epicatechin was more than 20% compared with the fresh beans. Clearly, the moisture contents and temperature have an effect on the epicatechin amount in the beans. Further reduction of epicatechin was observed in the 21.90 and 12.00% wet bean samples. In the 21.90% wet beans sample, the remaining amount of epicatechin was 2.22$\pm$0.02 mg/g, whereas in the 12.00% wet beans sample, the amount of epicatechin was 1.85$\pm$0.36 mg/g. Despite of the different amount of epicatchin at respective beans moisture, the amount was not statistically different between the wet beans samples and the fresh beans.

On the other hand, catechin was more difficult to determine due to its limited amount that was present in the dried nibs. In this study, the amounts of catechins were 0.22$\pm$0.09 and 0.26$\pm$0.18 mg/g of the 31.10 and 21.90% wet beans sample respectively. These amounts were 31.8 and 42.3 percent increase from the catechin amount in the fresh beans. On the other hand, the 43.54 and 12.00% wet beans sample contains a similar amount of catechin, at the rate of 0.15 mg/g. The reason for the increased in the catechin amounts was due to the epimerization process where the conversion of epicatechin to catechin took place at high temperatures (Hurst *et al*., 2011; Kofink, *et al.*, 2007; Kothe, *et al.*, 2013). Generally, the amount of catechin doesn't follow the reduction trend in the total polyphenols and the epicatechin. This could be due to the complexity and variations in each replication analysis. The large variation of the standard deviation value explained the difficulties in getting the precision results. Various studies on the retention of polyphenols found that these compounds reduced considerably during processing, particularly during fermentation, drying and roasting (Albertini *et al*., 2015; Hurst *et al*., 2011; Payne *et al.*, 2010). In terms of flavanol retention capability, a temperature of the cocoa beans must be kept below 140ºC in order to get the highest retention (Kothe *et al*., 2013).



Values are mean ± standard deviation of two replications. Mean value with same superscript letters do not showed significant different ($p < 0.05$)

Figure 6: Changes in epicatechin and catechin

Mechanical strength of the beans

The changes in the mechanical strength of the beans after wet beans micronizing is as shown in Table 1. Generally, the mechanical strength of the cocoa beans is represented by two units, one is the breaking force (Newton) and the other is the modulus of rupture. These two factors represent the hardness of the beans which gave indication on the effective breaking and separation process. The result shows that the breaking force of the beans was in the range of 6.5 to 5.9 kN for 43.54 to 21.90% wet bean moisture. In the 12.00% wet beans sample, the breaking force was 3.6$\pm$1.3 kN. The reduction in the breaking force was an indication that the force needed to break the beans was less and the beans became more brittle after the micronizing process. This indicates that less power was needed to break the beans. The changes of the strength of the beans are related to the amount of final moisture content of the beans and its final temperature. Less moisture in the beans is an indication that significant amounts of moisture have been dissipated due to the rapid conversion of water to vapor inside the beans. The rapid mass transfer in the microwave drying often creates a patterns that leads to the formation of porosity during drying of agricultural products. Porous beans are normally brittle and hence the force required for breaking is lesser. It can be seen from Table 1 that the force required to break the beans was 3.6 kN of the 12.00% wet beans sample, whereas the force required to break the 43.54% wet beans sample was 6.58 kN, an increase of almost twofold. Modulus of rupture (MOR) is a measure of the maximum load-carrying capacity or strength and is defined as the stress at which the material breaks or ruptures (based on the assumption that the material is elastic until rupture occurs). In this study, the MOR value corresponds with the breaking force for each wet beans sample. The highest MOR was found in the 43.54% wet beans sample, which was 169.15 while the 12.00% wet beans sample MOR was only 69.87. This translated as the 12.00% wet beans sample was the most brittle and has low resistant when subject to breaking force. In contrast, the 43.54% wet beans sample has more resistances compared to the rest of the wet beans samples. On the other hand, the mechanical properties of the beans give an indication of how thermal treatment affects the strength of the materials and whether the micronized beans could facilitate the breaking and consequently ease the separation process. Micronization of other products has been attempted by other researchers and in the case of using microwave as a source of heating, certain advantageous has been reported (Arntfield *et al*., 1997; Heydari *et al*., 2020; Scanlon, Cenkowski, Segall, & Arntfield, 2005).

Table 1: Breaking force and modulus of rupture of cocoa beans in beans micronizing

beans moisture, %	Breaking force (Newton)	Modulus of rupture
43.54	6.58±0.22	169.15±45.33
31.10	6.43±0.30	160.16±55.76
21.90	5.95±0.14	113.22±11.30
12.00	3.62±1.31	69.87±13.11

CONCLUSION

The use of microwave for wet beans micronizing show some potential when the temperature and the final moisture content achieved its required value. These will help and conditioned the nibs in the roasting step and the shell to be separated in the winnowing section. The drying kinetics of the beans in the microwave as depicts by the drying rate curve show similar moisture removal trends as other agricultural products and other method of drying, with the present of the falling rate in the curve. The bioactive compounds retention was noticeable in the dry nibs although there are differences among the wet beans moisture samples. Similarly, the amount epicatechin were found more abundant compared with catechin. The mechanical strength show that the drier the nibs the lesser the strength. The less mechanical strength of the beans could facilitate the breaking and winnowing process which happen at the latter stage. Nevertheless, some results derived from this study are inconsistent, because research on this matters are scarce and furthermore microwave drying tends to produce inconsistency and products scorching during high microwave power.

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