UNIVERSITI PUTRA MALAYSIA

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DEVELOPMENT OF CHOCOLATE POWDER TABLETS

BY

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ABSTRACT

A chocolate malted drink is a well-known product in Malaysia. Currently chocolate malt drink are being produced in a powder form to consumers. There are potential to develop the chocolate powder into tablets which is convenient to be diluted into drinks or be eaten or chewed. The objectives of this study are to investigate the psychochemical properties of chocolate powder and to develop a chocolate powder tablet. The chocolate tablets were analysed by measuring the dissolution time and accelerated shelf life. The physicochemical properties of the chocolate powder were analysed of its particle size, bulk density, tapped density, true density, porosity and moisture content volumes. The chocolate powder tablets were formed by using a 13-mm cylindrical die with forces applied from 2 to 9.8kN via the Universal testing machine. The dissolution time of the chocolate powder tablets was measured by using a dissolution tester with distilled water as dissolution medium. The shelf life were determined by measuring the moisture content during various storage times at extreme temperature (38 ± 2ºC). The study shows that the powder physicochemical properties such as particle sizes distribution can significantly affect the properties of the chocolate powder. For a dissolution test, it can be concluded that the dissolution rate decreased, as the porous material decreased and the compression force increased. To conclude, the shelf life of the tablet decreased as the moisture content increased during a nineteen days of storage with the highest volume moisture content of 22.2%.

ABSTRAK

Minuman malt coklat adalah suatu produk minuman yang amat dikenali di Malaysia. Kini, minuman malt coklat dipasarkan di dalam bentuk serbuk kepada pengguna. Terdapat beberapa potensi yang membolehkan serbuk minuman malt coklat diubah ke dalam betuk tablet dimana ianya mudah sedap dimakan ataupun dikunyah begitu sahaja. Antara objektif penyelidikan ini adalah untuk mengkaji sifat fizikokimia serbuk coklat dan menghasilkan serbuk coklat tersebut dalam bentuk tablet. Selain itu, coklat tablet juga melalui analisis penglarutan dan analisis jangka hayat. Sifat fizikokimia serbuk coklat dianalisis dengan saiz zarahnya, ketumpatan pukal, kepadatan yang ditekan, kepadatan sebenar, keliangan dan jumlah kelembapan serbuk malt coklat. Tablet serbuk coklat dibentuk menggunakan silinder berukuran 13mm dengan daya tekanan bermula dari 2kN hinggan 9.8kN melalui mesin ujian Universal. Penglarutan analisis bagi tablet serbuk coklat dijalankan melalui penguji larut degan menggunakan air suling sebagai media penglarutan. Jangka hayat serbuk coklat ditentukan berdasarkan jumlah kelembapan semasa proses penyimpanan di dalam suhu tinggi (38 ± 2ºC). Kajian ini menunjukkan bahawa sifat-sifat fizikokimia serbuk seperti saiz zarah dan pengedaran secara rawak oleh saiz zarah boleh mempengaruhi dan merubah sifat fizikokimia. Manakala untuk analisis penglarutan, ia boleh disimpulkan bahawa kadar larutan menurun kerana dipengahuri oleh peratusan keliangan yang berkurang dan peningkatan daya mampatan. Jangka hayat bagi tablet serbuk coklat berkurang kerana peningkatan jumlah lembapan yang terkandung di dalam tablet yang meningkat sepanjang proses penyimpanan selama sembilan hari dan mencatatkan jumlah kelembapan yang tertinggi sehingga 22.2%.

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CHAPTER 1

1. INTRODUCTION

1.1. RESEARCH BACKGROUND

Chocolate is a product based on cocoa. The cocoa beans have been undergoing a few processes before the formation of the chocolate and powder obtained. All over the countries have their own specific types of cocoa to be formulated. The largest cocoa plantation is in Ivory Coast following by Ghana and Indonesia. Malaysia once had been the largest cocoa production country in the 1980s but it has been declined due to the pod borer infestation and the focus is on the oil palm plantation because of the greater profitability (Beckett, 2008). In Malaysia, the cocoa is grown and planted by a small-holdings where the whole family work together (Wood & Lass, 1985).

While for the chocolate production, the largest country that produced high quality chocolates is in the US with over 20 billion dollars annually in retail sales (Maverick, 2015). Maverick (2015), also stated that the second largest country of a chocolate manufacturer is in Germany, following by Switzerland and Belgium.

Basically, in order to produce a cocoa powder as the final product, the cocoa beans will undergo two stages of processing which are cocoa primary processing and secondary processing. In the primary processing, the cocoa beans will be fermented for a few days and will be dried to produce dried beans before proceeding with the secondary processing. In the secondary processing, the dried cocoa beans will be cleaned, micronizing, breaking and winding (separation between nibs and shell).

The cocoa nibs are then roasted and proceeded with grinding process and form a cocoa liquor or cocoa mass. In order to produce the cocoa powder, the cocoa mass will be pressed and cocoa cake will be the product. The cake will be cooled, milled and sieved respectively and the final product will be the cocoa powder.

One of the applications of the cocoa powder is as an ingredient of a chocolate malt beverage. The chocolate malted beverage is actually a mixed of cocoa powder, sugar, malt extract, flavour and whey (Lannes & Medeiros, 2008). The consumption of beverages is a great source for diet and overall nutrition (Hamid et al., 2015).

There are 73.5% among Malaysian primary school children reported consuming chocolate malted drinks for at least once a week (Hamid et al., 2015). Hamid et al. (2015), also claimed that malted drink consumers had a greater micronutrient intakes and were more physically active than non-consumers. For a Malaysian adult, the intake of macronutrients from chocolate malted beverage met the recommendations for a healthy diet (Mirnalini et al., 2008).

The availability of chocolate malted drink is only in a powder form before it is reconstituted with water. Currently there are no chocolate malt powder in tablet form as a drink formulation in Malaysia. The only chocolate malt powder that has been commercialised in the tablet form is been used as a chewable candy which was produced by the Ovaltine brands'. While for the Milo brands which is recently been produced a Milo candy in a cube form. Both of this product only can be used as a chewable candy but not as a drink formulation.

The obvious characteristic of chocolate powder is the hygroscopic content. Hygroscopicity is the ability of powder to absorb the moisture from high relative humidity environment (Jaya & Das, 2005). The challenges in handling chocolate powder is that the physical and chemical properties of the powder may easily change because of the hygroscopicity. The hygroscopicity of the powder reduces by tableting (Saifullah et al., 2014). Thus, the aim of this study is to develop the chocolate malted drink in a tablet form.

1.2. PROBLEM STATEMENT

The chocolate malted beverage is in a powder form. Powder by means is a bulk assemblies consisting of solids, liquids and gases. The interactions between all three of these phases determine the bulk powder behaviour. In this study, the powder will be transformed into tablet form using compression method. The research is done with few reasons on the powder characterisations that leads to a study on change it into a tablet.

Firstly, powders are less convenient to use since it is in fine particle size and easily affected by the heat and surrounding factor because of the hygroscopicity of the chocolate powder. It also consumes more space than tablet in terms of packaging size and storage space. Furthermore, the powder also will give an inaccuracy of dose in case of bulk powder. The tablet will make an easiest way for the consumer to carry.

Tablets are the most shelf-stable choice and retain their potency over a longer period than powders. The hygroscopic characteristics that contain in the powder lead to the factors of the powder shelf life.

The powder particle characterization may offer many inconvenient factors such as the solution requires better control for handling and operating the powders. From the study, the powder behaviour is not completely dependent on the powder characteristics only, it is also can be affected by the environmental factors (Hausner, 1981). A tablet form transformation from a powder properties is actually can provide many benefits and consumer friendly.

1.3. OBJECTIVES

1. To determine the physicochemical properties of chocolate powder and to compare with commercialized chocolate powder.
2. To develop chocolate powder tablet.
3. To analyse the dissolution time and accelerated shelf life of developed chocolate powder tablets.

1.4. SCOPE OF THESIS

The formation of chocolate tablet that can be used as a chewable candy and more convenient in handling a chocolate malted drink in a tablet form. The direct compaction technique is used to form the tablet by using by the Instron Universal Testing Machine 5566, U.K.

CHAPTER 2

2. LITERATURE REVIEW

2.1. TABLET RAW MATERIAL

A chocolate malt drink is a famous drink consume in Malaysia as it provides high nutrition for consumers. The chocolate malt drink is a mixing of cocoa powder, sugar, creamer, malt extract and cream milk. The drinks can be served with hot or cold water. The benefits of consuming chocolate malt drink besides the nutrition it also supplies energy to the consumer. There are 73.5% among Malaysian primary school children reported consuming chocolate malted drinks for at least once a week (Hamid et al., 2015). Hamid et al. (2015) also concluded that the highest consumers are boys, indigenous children and those live in the East Coast region of Malaysia.

2.1.1. Nutrition facts of chocolate powders

The chocolate malted drink is rich of nutritional content. The chocolate malted powder is mainly made up of a cocoa, sugar, and creamer. It also contains of protein, fat and carbohydrate which supplies energy to the body. As shown in Table 2.1, there are various compositions of energy, protein, fat and carbohydrate content based on the types of chocolate malted drink. The surface composition can affect the flowability of the powder (Saifullah et al., 2016)

	Nutrition information per serving (30g)			
Components	Milo	Ovaltine	Cadbury	Oligo
Energy (kcal)	135	121	130	115
Protein (g)	9.5	1.2	2.0	1.1
Fat (g)	3.7	1.9	4.0	3.1
Carbohydrate (g)	24.4	24.8	22.0	20.6

Table 2. 1: The nutrition information for a commercialised chocolate powders.

By referring to the table above, Milo has a high amount of energy produced with 135kcal, following by Cadbury with 130kcal energy, Ovaltine with 121kcal energy and Oligo with 115kcal of energy content accordingly. Also, for protein content, Milo has a high amount of protein with 9.5g, following by Cadbury with 2.0g protein, Ovaltine with 1.2g protein and Oligo with 1.1g of protein content accordingly.

Next, for a fat content, Cadbury has a high amount of fat produced with 4.0g, following by Milo with 3.7g of fat, Oligo with 3.1g of fat and Ovaltine with 1.9g of fat content accordingly. While, for a carbohydrate content, Ovaltine has a high amount of carbohydrate produced with 2.48g, following by Milo with 24.4g of carbohydrate, Cadbury with 22.0g of carbohydrate and Oligo with 20.6g of carbohydrate content accordingly. The local chocolate malted drink powder is assumed to have the same properties as Oligo powder based on the physical powder properties as obtained in the result sections.

2.2. TABLETTING

2.2.1. Direct Compaction Technique

Direct compaction is a commonly used technique in order to produce tablet. There are advantages of direct compaction technique such as it shorten the development time, lowers manufacturing cost and recommended for heat sensitive product.

The manufacturing costs can be reduced through the elimination of the wet granulation process by the direct compression. The direct compression requires fewer unit operations in the tablet production where it involved two unit operations which are powder mixing and tableting (Ezani, 2015).

The direct compression also requires shorten development time since it requires less steps with less equipment and quicker scale up technique to produce tablets. These advantages lead to the most industry's preferred choice. Many researchers used the direct compression technique by using the same machine which is an Instron Universal Machine 5566 (Ezani, 2015). Previous study that have used the direct compression was tableting of pitaya powder (Yusof et al., 2012). Other than that, this compression technique has also been used in the development of fruit powder tablets to study the flow and physicochemical properties of tablet (Saifullah et al., 2016).

The heat sensitive product or powder is suitable using the direct compression technique in order to develop medicine tablets. This technique is the simplest way of combining the mixture without changing any composition of the tablet ingredients.

2.2.2. Uniaxial Die Compaction cycle

The uniaxial die compaction is where powder undergoes compaction process within a die cavity by the action of an upper punch at a constant velocity, while the lower punch will be in static position within the mechanical assembly as shown in Figure 1. In order to investigate the compressibility and compactability of powder this will be the main process needed to be done. The differences between the compressibility and compactibility is where the compressibility shown an ability to reduce volume, and compactability is the ability to form particle bonding (Yusof et al., 2009).

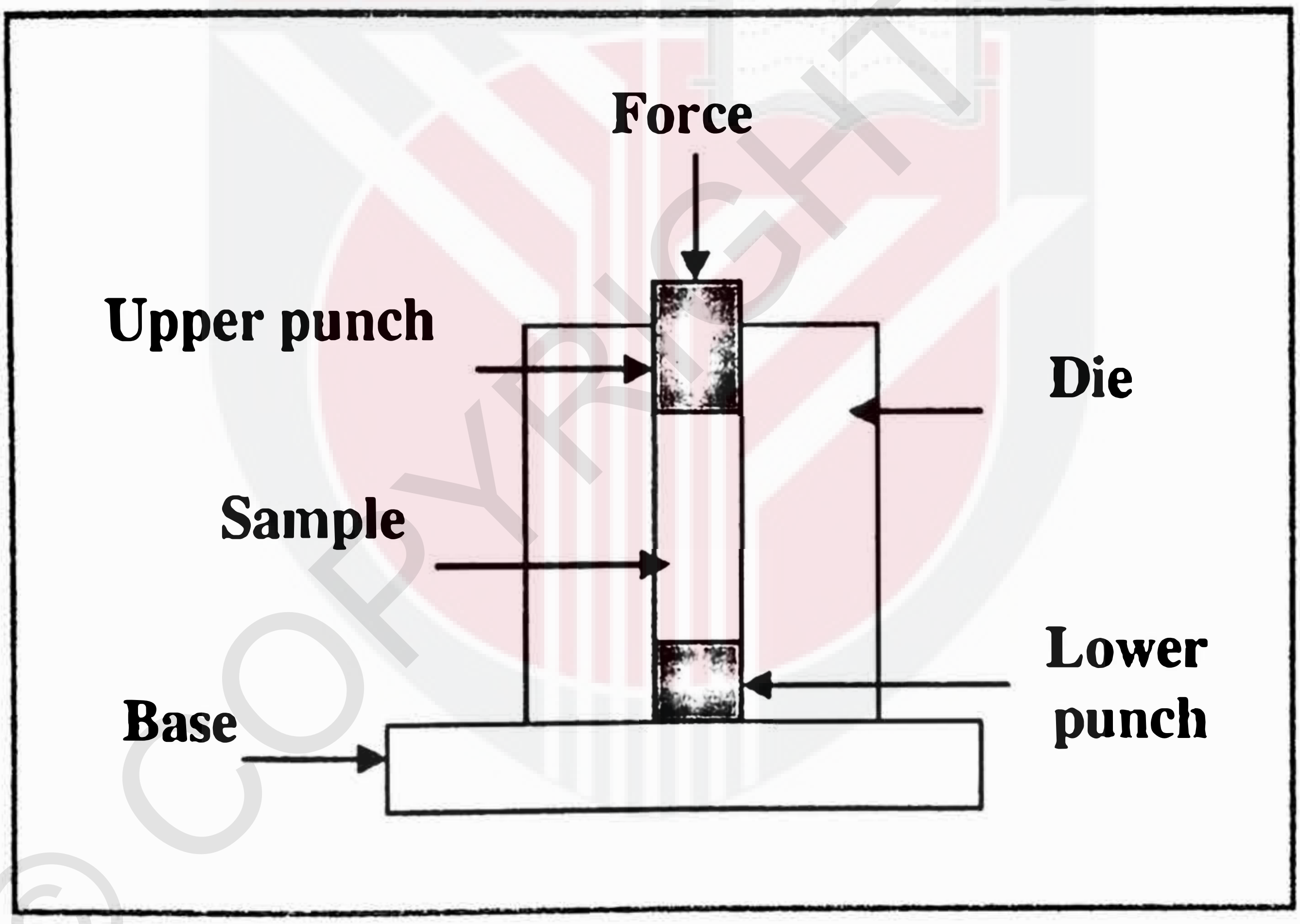

Figure 2. 1: A schematic diagram of uniaxial die compaction (Yusof et al., 2009).

A universal testing machine, Instron 55666 (Canton MA, USA) was used to compress chocolate powder into tablets by pouring the powder into the cylindrical uniaxial die of 20mm diameter. There are four stages of compression using uniaxial die compaction as shown in Figure 2.2.

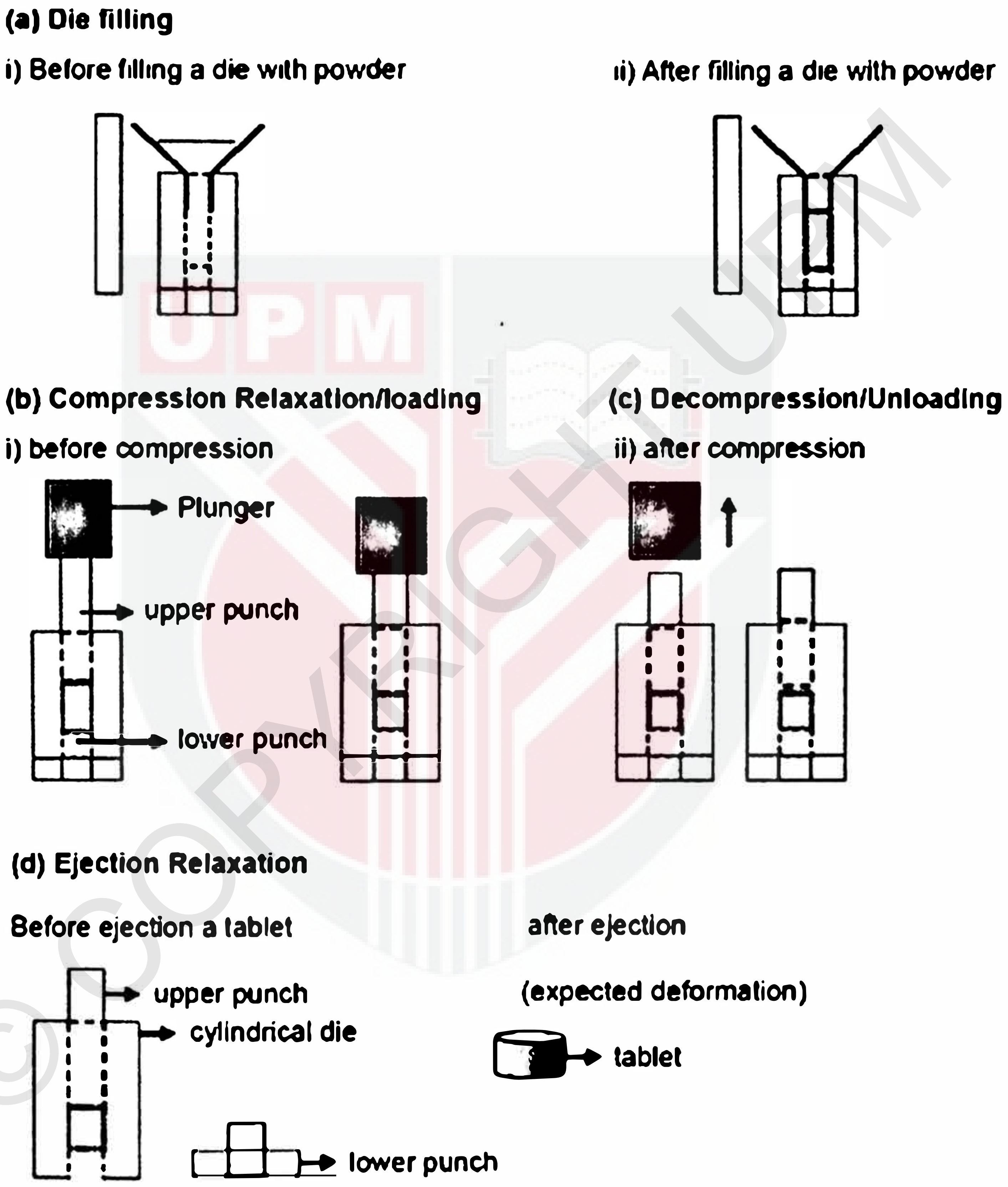

Figure 2. 2: The major processing stages for compression using uniaxial die compaction (Yusof et al., 2010).

The first stage of the compression is the die filling by referring to the Figure 2.2 (a). The schematic diagrams show the diagram of the die compaction before filling with powder and a die compaction after the filling as drawn in Figure 2.2 (a, i) and Figure 2.2 (a, ii), respectively. Die filling is whereby the powder mixture is poured under gravity into the feed shoe running over the die opening (Yusof et al., 2010).The second stage shown in the Figure 2.2 (b) is the compression relaxation or loading phase. During the second stages, a plunger is been used to pressing the chocolate powder against the upper punch.

The third stage shown in the Figure 2.2 (c) is the decompression or unloading phase. During the third stages, the plunger used is removed from the upper punch to allow the tablet relaxation and an elasticity phenomenon to take place.

The last stages is the phase of relaxation ejection of the tablet as shown in in Figure 2.2 (d). This is where the tablet was removed from the die. The tablet development objectives was determined. Other than that, the tablet will be used to find the compressibility and compactibility characteristics of the chocolate powder. Besides, the tablet will be used to analyse the dissolution time.

2.3. DISSOLUTION TEST

Dissolution is a process of dissolving a solid particles into a solvent and turns to a liquid medium (May et al., 2014). Dissolution testing has been used in many sectors of industry and it also have widely used in the development of pharmaceutical product which has been used for optimization of formulation and quality control (Kumar & Bhatnagar, 2014). Kumar & Bhatnagar (2014) also stated that the test can been used to investigate the drug degradations profiles, stability and shelf life studies, and physical and mechanical testing of dosage forms.

2.4. ACCELERATED SHELF LIFE

Shelf life is actually a period of time of a product that is acceptable and meets the consumers' expectations regarding food quality and safety (Martins et al., 2008). The main purpose of food and the food packaging technologies in industry is to extending the shelf life of the food products (Gibbs, 2015).

The method used for the shelf life determination by the studies of frozen foods are using the sensory analysis while evaluates the taste, odour, body, texture, colour and appearance (Fu & Labuza, 1997). Other than that, a shelf life prediction also can be estimated from the relationship between the moisture content, the sticky point temperature and the colour change during storage (Jaya & Das, 2005).

CHAPTER 3

3. MATERIAL AND METHODOLOGY

3.1. TABLET RAW MATERIALS

3.1.1. Selection of raw materials

In this research, the main chocolate powder of the chocolate malted drink that has been chosen is from commercialised local company which known as A powder. While for the comparison of other commercialized chocolate malted drink brands were chosen are Milo 3 in 1, Cadbury Hot Chocolate Drink 3 in 1, Oligo 3 in 1 and Ovaltine 3 in 1. All the chocolate malted drink chosen contain all the main ingredients that have been stated; cocoa, sugar, and creamer.

3.2. PHYSICAL PROPERTIES

The physical powder properties consist of determination of particle size, bulk density, tapped density, true density and porosity. The tests were carried out by using a raw material in the form of powders before transforming it into a tablet form at the laboratory by doing it either manually or by the specific equipment or machines.

3.2.1. Particle size

The particle size of the chocolate powders was carried out by using a dry dispersion method and measured a particle size analyzer (Malvern Mastersizer 2000 Instrument Ltd., U.K.). To analyze the size of the particles, a laser light diffraction technique by Mastersizer 2000 utilizes was used (Saifullah et al., 2016). For this property, the data were recorded automatically by the placed the powder samples in the particle analyzer.

3.2.2. Bulk density

The bulk density of the powder samples was measured manually by pouring 2g of the chocolate powder into a 10mL graduated measuring glass cylinder. The bulk density was determined by the calculation of the ratio of the mass of the powder to the volume occupied by the powder (Goula et al., 2004).

3.2.3. Tapped density

The tapped density of the powder samples was determined by using an envelope density analyser with T.A.P. density option (GeoPyc 1360 Pycnometer Micromeritics, U.S.A.). The chamber internal diameter used is 25.4 cm with typical force factor of 51N. The cycle of the determination used is 5 cycles for each types of chocolate powder. The sample weight recorded was entered into the computer program, and the data such as volume and density will be calculated automatically by the systems (Saifullah et al., 2016).

3.2.4. True density

The true density of the powder samples was determined by using a gas pycnometer (AccuPyc II 1340 Micromeritics, U.S.A.). The volume of the samples was measured by using the helium gas displacement method with 5 cycles used. The sample weight recorded was entered into the computer program, and the data will be calculated automatically the systems (Saifullah et al., 2016).

3.2.5. Porosity

The porosity of tablets was measured manually by using an equation from the value of tablet density and the true density of the tablet ingredients. The equation used was as stated below (Sun, 2005):

$$\varepsilon\,(\%) = \left(1 - \frac{\rho b}{\rho true}\right) \times 100 \tag{3.1}$$

Where, ε is the porosity, ρ_b is the bulk density of the tablet, ρ_{true} is the true density of the tablet ingredients.

3.3. CHEMICAL POWDER PROPERTIES

The chemical properties is the determination of moisture content of the powder.

3.3.1. Moisture content

The moisture content of the chocolate powder was measured by using a moisture analyser. The testing was repeated for three times to obtain an average reading. The moisture content of the chocolate powder was determined automatically by the instrument (Saifullah et al., 2016).

3.4. POWDER FLOW PROPERTIES

Powder flow properties consist of the determination of Hausner ratio and Carr index. The powder flow properties can be proceed right after the determination of tapped density and bulk density inside the physical or powder properties.

3.4.1. Hausner ratio and Carr index

In order to investigate the flow properties of the raw materials, the Hausner Ratio (Hausner, 1981) and Carr Index (Carr, 1965) were calculated by using a values of tapped density and bulk density as follows:

$$HR = \frac{Td}{Bd} \tag{3.2}$$

$$CI = \frac{Td - Bd}{Td} \times 100 \tag{3.3}$$

Where, *HR* is the Hausner Ratio, T_d is the tapped density, B_d is the bulk density and *CI* is the Carr Index.

From the calculations, the different ranges between the Carr Index and Hausner Ratio have been defined by Lebrun et al., (2012) as presented in Table 3.1.

Flowability	Carr Index (CI), %	Hausner Ratio (HR)
Excellent	0-10	1.00-1.11
Good	11-15	1.12-1.18
Fair	16-20	1.19-1.25
Passable	21-25	1.26-1.34
Poor	26-31	1.35-1.45
Very poor	32-37	1.46-1.59
Very, very poor	>38	>1.60

Table 3. 1: The Flowability Classification (Lebrun et al., 2012)

3.5. TABLETTING

A tableting chocolate powders were carried out by using a direct compaction method (Saifullah et al., 2016). A cylindrical uniaxial die of 20mm was used by pouring the raw material inside it with the weight of 2g. A universal testing machine (Instron Universal Testing Machine-5566, Canton MA, UK) was used to compress the powder samples into tablet form. A force was set up at 9.8 kN with a compaction speed of 5 mm/min by using the Bluehill Software (Canton MA, USA) which is the operating software for the Inston Machine. The thickness of the tablets was measured to calculate the final volume and density of the powder in tablet form. There are also various range of force was applied to determine the pressure-volume relationship which are 4kN, 6kN and 8kN at the same weight of powder.

The Kawakita & Lüdde (1971) model was used to determine the compressibility of the samples. The mathematical formula of this equation is as follows:

$$\frac{P}{C} = \frac{P}{a} + \frac{1}{ab} \tag{3.4}$$

Where a and b are constants, C is the degree of volume reduction under applied pressure P. C is calculated from the initial volume of the powder and final volume in tablet form. The mathematical formula of this equation is as follows:

$$C = \frac{Vo-V}{V} \tag{3.5}$$

Where v_o is the initial volume and v is the final volume.

3.6. DISSOLUTION TEST

Dissolution test was used to measure the dissolution time between the local chocolate malted drink powder and the foreign commercialised chocolate powder brands. The dissolution test will be carried out in a dissolution tester (Pharma Test D-63512, Germany) at 100 rpm paddle speed. The dissolution medium was used during the test is the distilled water.

To analyse the amount of solute present in the samples collected from the dissolution chamber, an Ultraviolet spectrophotometer (HACH DR2800, United States) was used (Saifullah et al., 2016). The light absorbance by the samples was measured at a wavelength of 560nm. Using the following equation to calculate the percentage of dissolution/solute release:

$$\text{Percentage dissolution at any time} = \frac{At - Ao}{Af} \times 100 \tag{3.6}$$

Where, A_o is the Absorbance of a control sample (fresh medium), A_t is the Absorbance of the sample at any time, A_f is the Absorbance of the sample when complete dissolution occurs.

The dissolution been tested by using a dissolution tester (Pharma Test D-63512, Germany) at 100rpm paddle speed with 500ml dissolution medium used (Saifullah et al., 2014). The suitable temperature to carry out the dissolution test is at 37 ± 0.5°C (Kumar & Bhatnagar, 2014).

3.7. ACCELERATED SHELF LIFE

The accelerated shelf life was determined by using the same method used in Mango powder researched by Jaya & Das (2005) which was determined by the moisture content during the storage process. For this studies of chocolate tablet accelerated shelf life, every 2g of tablet samples will be placed inside the incubator with a temperature range of 38 ± 2ºC and environment maintained at 90% Relative Humidity (RH) by the salt solution.

The shelf life will be determined by the moisture content changes of the tablet with the storage time where the moisture is measured by using the moisture analyser.

CHAPTER 4

4. RESULTS AND DISCUSSION

4.1. PHYSICOCHEMICAL AND FLOW PROPERTIES OF CHOCOLATE POWDERS

The raw material that has been used in this experiment is labelled as A powder which is the local commercialised powder, Milo powder which labelled with B, Cadbury powder labelled with C, Ovaltine powder labelled with D, and Oligo powder labelled with E. The physical and chemical properties of the powders were determined in this section which include the moisture content of the powders, true density, tapped density, bulk density and porosity. For a flow properties, it will be determined by the Hausner's ratio (HR) and Carr Index (CI) equations.

4.1.1. Particle size distribution

A dry dispersion method was used to measure the particle size and size range of the powder samples and were measured by using a particle analyser (Malvern Mastersizer 2000 Instrument Ltd., U.K.).

Material properties	Powder samples A	B	C	D	E
Mean particle size (µm)	176.41	129.90	260.57	210.42	262.40
Span	3.58	4.11	2.03	5.52	4.05

Table 4. 1: Experimental values of particle size distribution of different powders

By referring to the Table 4.1, it shows the mean particle size distribution for powder A, powder B, powder C, powder D and powder E are 176.41 µm, 129.90 µm, 260.57 µm, 210.42 µm and 262.40 µm respectively. According to the Table 4.1, powder E has the largest mean particle size and followed by powder C, powder D, powder A and powder B.

While for the span value which is the width of the distribution based on particle sizes for powder A, powder B, powder C, powder D and powder E are 3.58, 4.11, 2.03, 5.52, and 4.05 respectively. Powder D has the highest span values followed by powder B, powder E, powder A and powder C. The higher the span values, the wider the distribution of powder particles (Ezani, 2015). Moreover, the particle size and particle size distribution can provide better flow properties and compaction behaviour which will be influenced in compactibility and rearrangement of particles (Saifullah et al., 2016).

4.1.2. Densities of powders

Table 4.2 shows the experimental values for true density, tapped density, bulk density, Hausner's ratio (HR), Carr Index (CI) and porosity of A powder, B powder, C powder, D powder and E powder.

Powder samples	A	B	C	D	E
True density (kg/m^3)	1373.8 ± 1.5	1414.7 ± 0.8	1039.3 ± 2.2	1517.5 ± 1.9	1367.3 ± 0.4
Tapped density (kg/m^3)	697.8 ± 3.1	699.8 ± 5.1	607.6 ± 1.8	792.8 ± 6.2	695.3 ± 4.4
Bulk density (kg/m^3)	504.3 ± 7.4	560.8 ± 9.2	444.1 ± 5.6	600.1 ± 10.0	594.2 ± 10.0
Hausner's ratio, HR	1.38 (Poor)	1.25 (Fair)	1.38 (Poor)	1.32 (Passable)	1.17 (Good)
Carr Index, CI (%)	28 (Poor)	20 (Fair)	27 (Poor)	25 (Passable)	15 (Good)
Porosity (%)	63.29	60.36	57.55	61.21	56.54

Table 4. 2: Experimental values of densities for each powder

By referring to the Table 4.1, the highest amounts of true, tapped and bulk densities contains is having by the powder D with 1517.3 kg/m^3, 792.8 kg/m^3 and 600.1 kg/m^3 respectively. The next higher amounts of true and tapped contains is having by the powder B with 1414.7 kg/m^3 and 699.8 kg/m^3 respectively.

The following up higher amounts of true and tapped densities is having by the powder A with 1373.8 kg/m^3 and 697.8 kg/m^3 respectively. The lower amounts of true and tapped densities is having by the powder E with 1367.3 kg/m^3 and 695.3 kg/m^3 respectively. While the lowest amounts of true and tapped densities is having by the powder C with 1039.3 kg/m^3 and 607.6 kg/m^3 respectively.

There are different arrangement of powders that contain higher amount of bulk density after the powder D which is followed by powder E, powder B, powder A and powder C with 594.2 kg/m^3, 560.8 kg/m^3, 504.3 kg/m^3 and 444.1 kg/m^3 respectively. The different arrangement of bulk densities from others arrangement of densities may be caused by the different particle sizes of the samples. The different sizes of particles in a powder tend to allow the smaller particles to fill up the pores made by the larger particles of the powder (Saifullah et al., 2016). Saifullah et al. (2016), also stated that the wide range of particle size results in high initial packing can increase the bulk density volume.

The Hausner's Ratio (HR) and Carr Index (CI) values from the table above, shows that the powder E have a good flowability characteristic with 1.17 and 15% respectively. Next, powder B has fair flowing characteristic based on HR and CI values with 1.25 and 20% respectively. The passable flowing characteristic is consist by powder D with 1.32 HR value and 25% of CI. While for powder A and C, both have poor flowing characteristics with the same HR values which is 1.38 but different CI percentages which are 28% and 27% respectively.

The flowability characteristics may be varies by depending on the powder particle morphology such as particle sizes and shape of the powder particles with the interaction between the powder particles (Saifullah et al., 2016). It is also claimed that, the Knowlton (1994) stated that the flowability has a great influence on transportation, formulation, mixing, compression and packaging.

Based on the Table 4.1, powder A have the highest percentage of porosity with 63.29% and following by powder D, powder B, powder C and powder E with 61.21%, 60.36%, 57.55% and 56.54% respectively. Other than that, porosity also can be affected by the bulk density because of an inter relationship of both properties inside the calculations. While the bulk density can be affected by the uniform particle shape and wide range of particle sizes of the powder because of the void spaces filled by the small particles (Saifullah et al., 2016).

4.1.3. Moisture content of powders

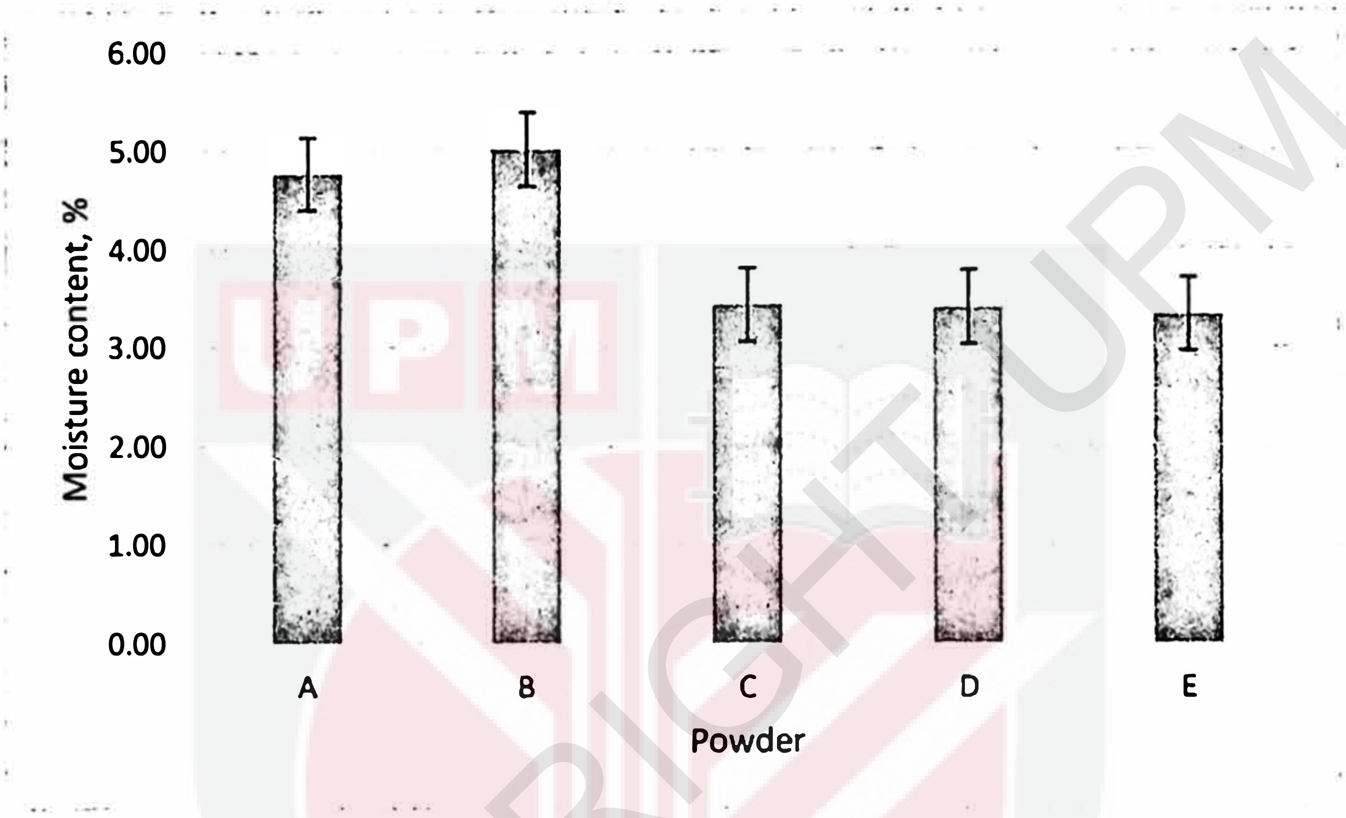

Figure 4. 1: Moisture content for various types of chocolate powders.

As shown in Figure 4.1, there are various average moisture content of the powder which are 4.77 ± 0.13%, 5.02 ± 0.49%, 3.43 ± 0.26%, 3.40 ± 0.40% and 3.33 ± 0.27% for powder A, powder B, powder C, powder D and powder E respectively. For powder A and powder B, the moisture content is quiet close compared to powders C, D and E. There are also have a slightly different of the moisture content percentage in between powders C, D and E.

The moisture content in every powder particle is an important factor for food industry, since the moisture content may affect the shelf life of the powders. This is because the probability of shelf life for the powders may be come shorter by the presence of microbe and be a spoilage to the powder.

4.2. TABLET PROPERTIES

4.2.1. Applied pressure-Volume relationship

For the applied pressure-volume relationship, the compression speed used is 5.0 mm/min with a pressure of 2kN, 4kN, 6kN, 8kN and 9.8kN. Every sample was compacted at the mass of 2g for the pressure volume relationship tested with a 20mm diameter of uniaxial die.

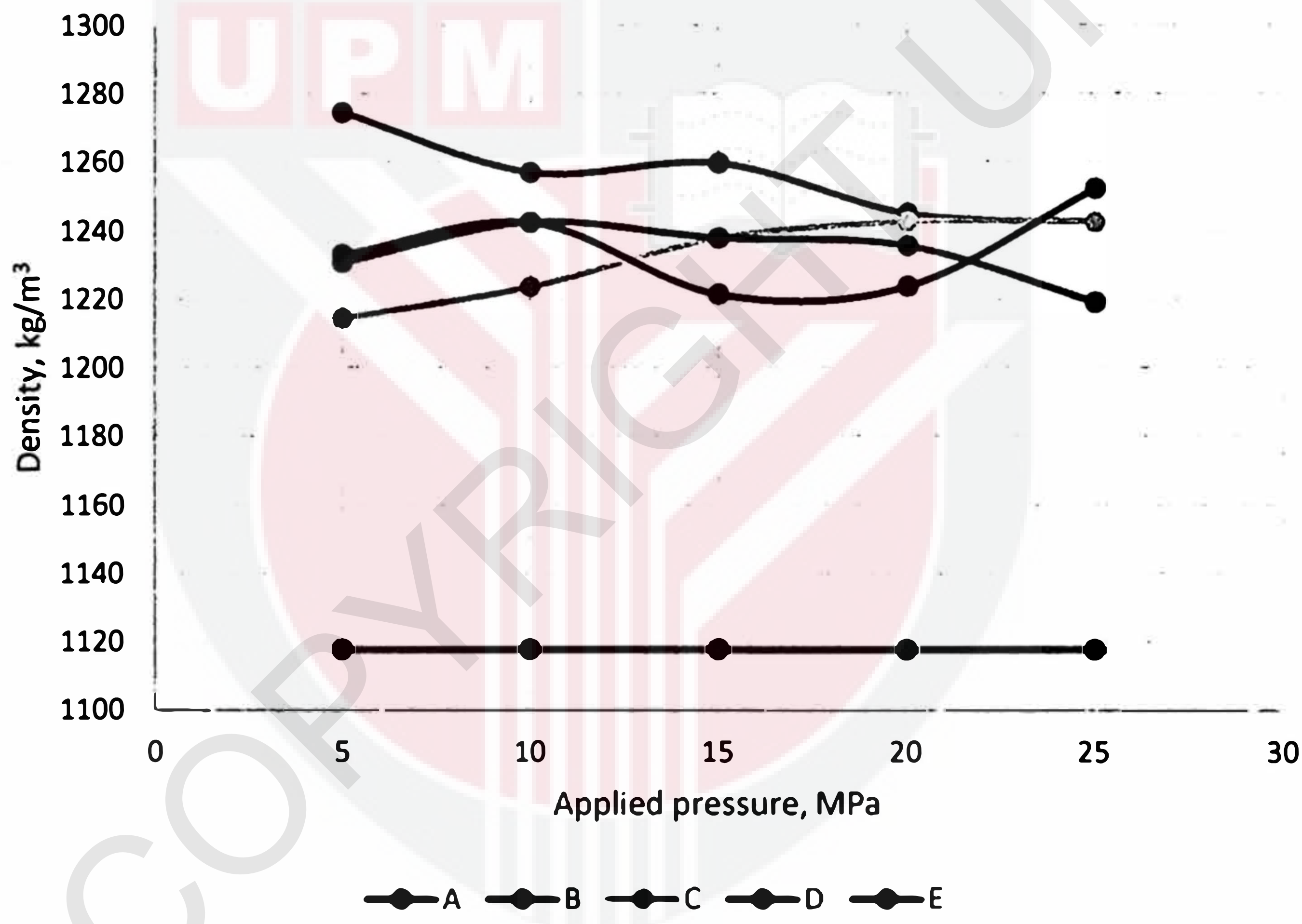

Figure 4. 2: The applied pressure-volume relationship of various types of powders.

Based on the Figure 4.2, the density of tablet B has remained constant for all various amount of pressure applied. This is because, tablet B contains of constant particle sizes of powder and have fair flowability characteristics as stated at the previous discussion.

While for tablet A, D and E, have an inconstant graph lines as the density is increased and decreased for an applied pressure. The variation of density may cause by the distribution of random particle sizes during the compaction.

Besides, the distribution of particle sizes of the chocolate powder have caused a smaller particle filled in the void spaces, hence reduce the compacts volume (Yusof et al., 2009).

For tablet C, the graph line is increasing in density with the increasing of the applied pressure. This show that the powder have a same distribution of particle sizes inside the sample. Yusof et al. (2010) stated that as the pressure increases, the density of the powder increases.

4.2.1.1. Kawakita and Lüdde Equation

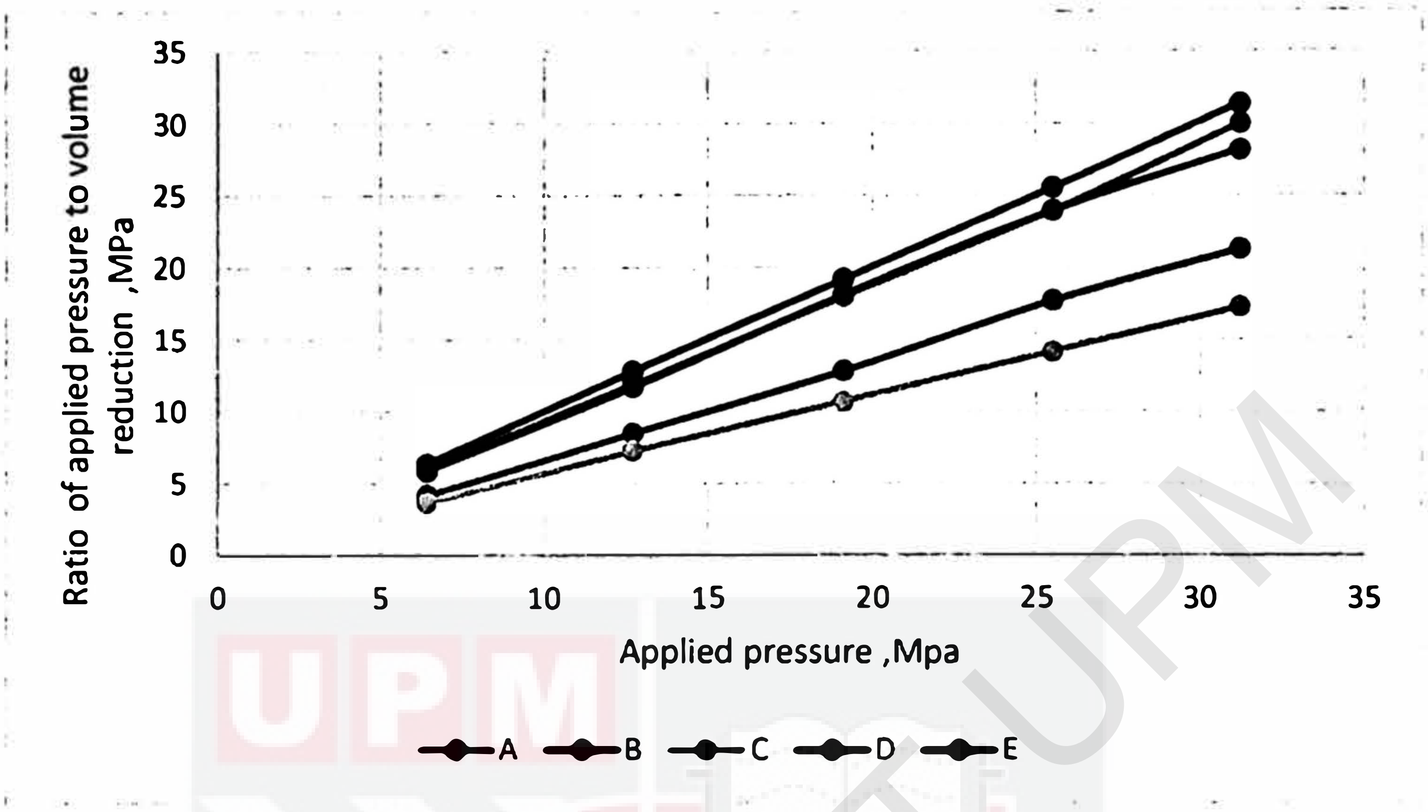

Figure 4. 3: Experimental data fitting into Kawakita and Lüdde equation for chocolate powders at ambient conditions.

Sample	A	B	C	D	E
a	1.44	0.99	1.83	1.04	1.10
b	-2.26	-48.38	2.04	-4.54	3.56
R^2	0.9993	1	0.9999	0.9995	0.9977

Table 4. 3: The constant characteristics and regression for various sample of chocolate powders

Figure 4.3 shows an example of fitting the ratio of the applied pressure to the volume of reduction as the function of applied pressure for various types of powders.

The constant *a* in the Kawakita and Lüdde relationship represents the initial porosity, and the constant *b* is related to the cohesiveness of the powder (Yusof et al., 2012).

Based on the Table 4.3, the values of the constant characteristics, a and b derived from the Kawakita equation, for tablet A with regression R^2 are 1.44, -2.26 and 0.9993 respectively. While for tablet B, the values of the constant characteristics, a and b derived from the Kawakita equation with regression R^2 are 0.99, -48.38 and 1 respectively.

The tablet D has a constant characteristics, a and b derived from the Kawakita equation with regression R^2 are 1.04, -4.54 and 0.9995 respectively. And the last properties is for tablet E which have a constant characteristics, a and b derived from the Kawakita equation with regression R^2 are 1.10, 3.56 and 0.9977 respectively.

Tablet C has a constant characteristics, a and b derived from the Kawakita equation with regression R^2 are 1.83, 2.04 and 0.9999 respectively. As the results from the Figure 4.2, the tablet C is following the theory of density increased as pressure increased and to be compared with the results in Figure 4.3, it shows that the data of applied pressure-volume relationship is valid since it have the lowest ratio of applied pressure to volume reduction.

4.2.2. Dissolution of chocolate powder tablets

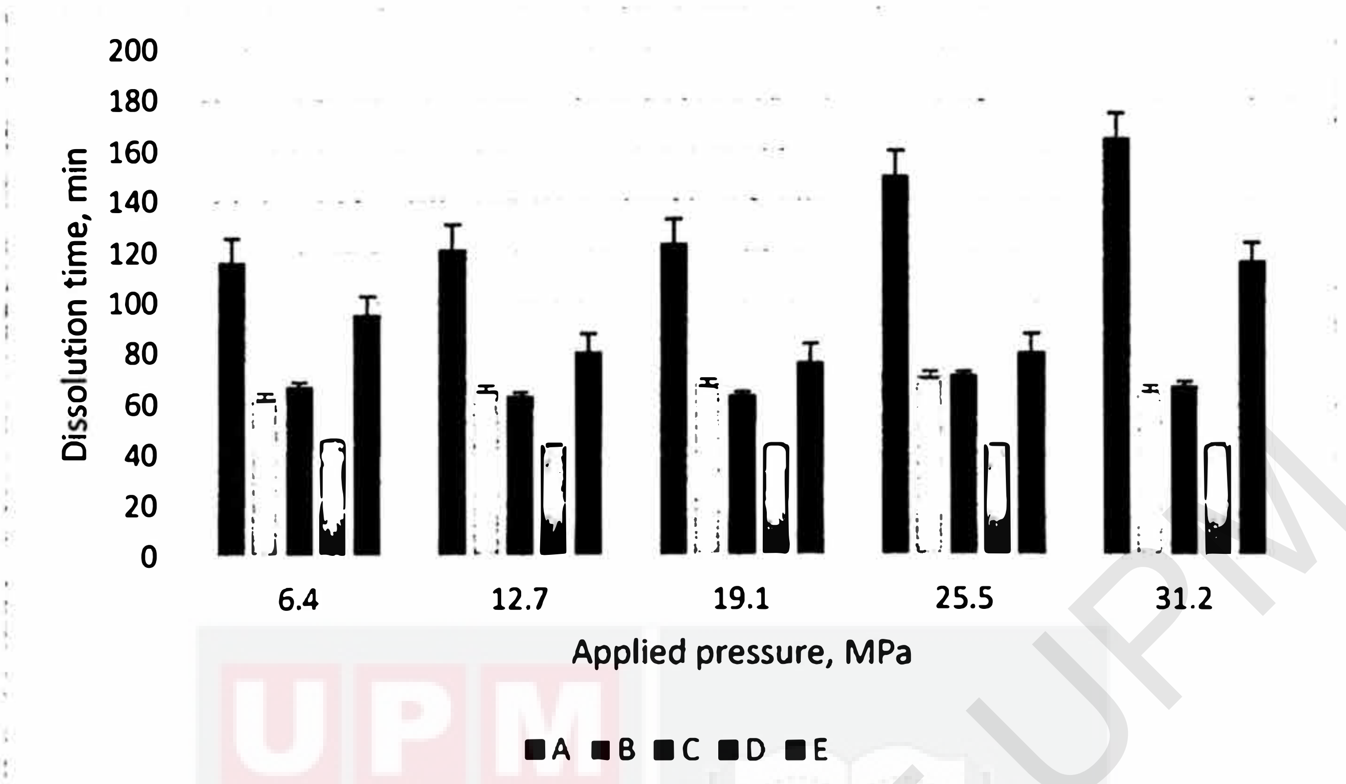

Figure 4. 4: Dissolution time by applied pressure of chocolate powders.

The dissolution test has been made by dissolving various types of tablet with different pressure applied to the tablet. Based on the Figure 4.4, there is slightly different dissolution time taken among tablet B and tablet C. For tablet D, it has the lowest dissolution time taken as compared to the other powder tablet types. It also shows that, the dissolution time taken taken for tablet B, tablet C and tablet D does not influence by the amount of applied pressure.

In contrast, for tablets A and E there are quite far difference of the dissolution time taken and also it has a various range of dissolution time taken along the amount of pressure applied.

The dissolution time based on Table 4.1 and Table 4.3, the shortest time of dissolution consume is 44 min for tablet D with the porosity of 61.21%. Next, following by tablet B with 67 min of dissolution time at the percentage of porosity 60.36%. It however has the same dissolution time for tablet C with the percentage of porosity 57.55%.

For tablets E, the dissolution time is 87 min with the percentage of porosity 56.54%. The porosity has a direct relationship with dissolution of any powdery material because the less porous material, the lower the dissolution rate (Saifullah et al., 2016).

But for tablet A, the dissolution time is 127 min with the percentage of porosity 63.29% which is supposed to have a less percentage of porosity because of the dissolution time. The error might be caused by the hygroscopic characteristic and the powder has been exposed to the environment for a long period which affected the physical characteristics.

4.2.3. Accelerated shelf life of the powder tablets

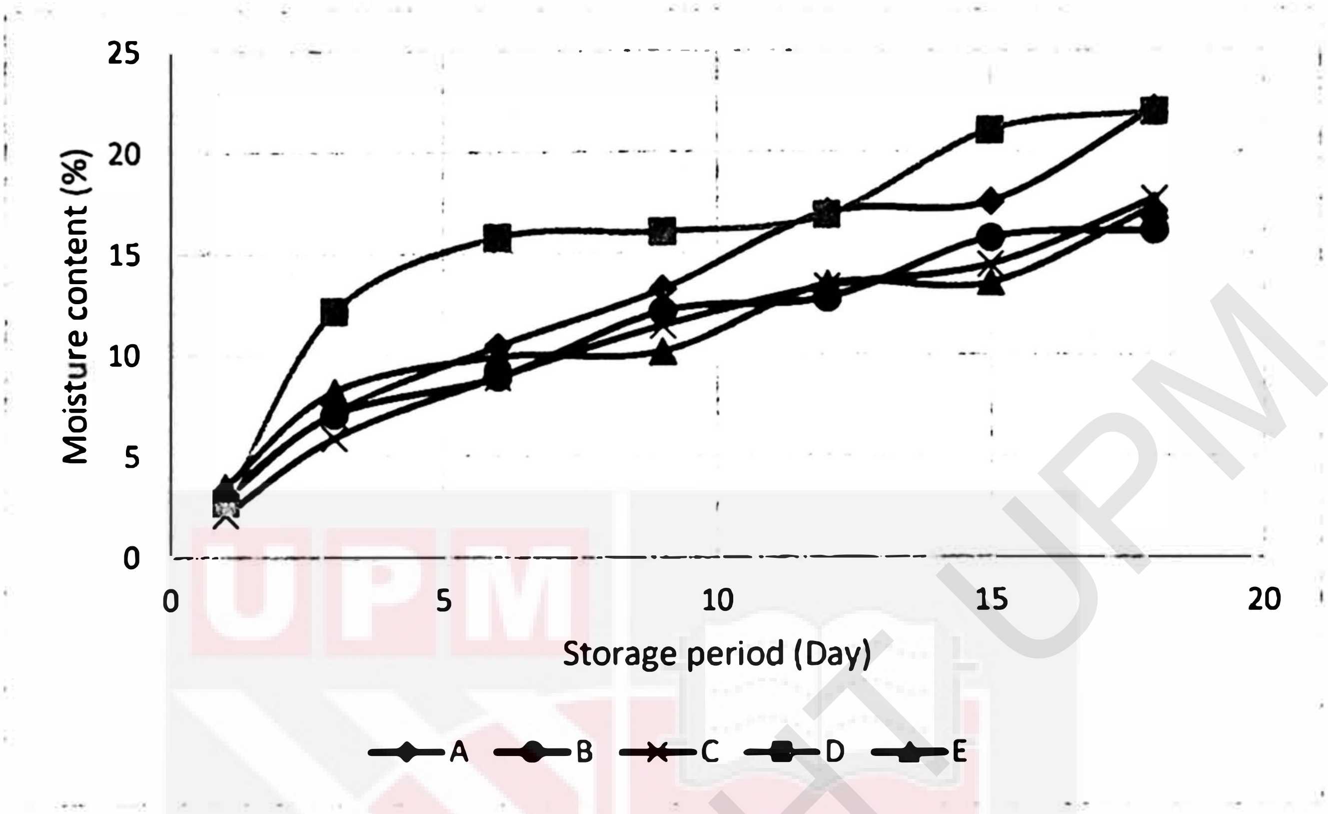

Figure 4. 5: Moisture content of chocolate powders for an accelerated shelf life.

The accelerated shelf life was determined by the moisture content changing over a period of time. Based on Figure 4.5, moisture content in the first five days for tablets D increased drastically, but for tablets A, B, C and E the moisture content were increasing steadily. This is may be because of the large porosity percentage that make the tablets D easily absorbed the air and increase the moisture content in the tablets. Even though tablets A had the largest porosity, the results may be affected by the hygroscopicity characteristics that change the tablet properties as it is due to exposure to the surrounding while conducting the experiment.

As shown in the Figure 4.5, the moisture content of the tablet A initially is 2.8% and have increased drastically to 15.8% by day six. At the end of day eighteen, the highest moisture content is the tablet A with a percentage of 22.2%, following by tablet D with 22.0% and tablet C with 17.7% of moisture content. It is then followed by the tablet E with 17.3% moisture content and the lowest moisture content is the tablet B with 16.2%.

By day six of storage, the tablets for all samples were getting soft and sticky as compared to the first day of the experiment. The initial conditions of the tablets are hard structures and not sticky also easy to handle. As day goes by, the tablets started to have a separation of a liquid below the tablets by day nine. The orange liquid solutions that had been released might be the sugar or malt contents. By day nineteen, tablet A is the first tablet that started to have mould above the tablet surface and followed by tablet D, tablet C, tablet E and tablet B.

As the moisture content is high, the shortest the shelf life of the tablet as it increased the chance of microbes to grow rapidly and spoil the tablets (Ezani, 2015).

CHAPTER 5

5. CONCLUSION AND RECOMMENDATIONS

5.1. CONCLUSION

The development of chocolate malted drink powder in a tablet form has undergone a few tests. The physical and chemical powder properties need to be studied first before proceeding with compressibility test in order to form a chocolate tablet. The important physical properties of powder consist of bulk density, tapped density, true density, flowability which were determined the Hausner Ratio (HR), Carr Index (CI), and porosity. While for chemical properties, it is determined by the moisture content of the chocolate malt powder.

From this study, it can be observed that the density properties are correlated where the bulk density can be influenced by the particle shape and random distribution particle sizes of the powder. Whereas for the porosity, it was affected by the amount of bulk density. The variation of flowability of the powder depends on the physical properties of the powder, the powder morphology as mentioned previously and the surface composition such as fat, protein, carbohydrate and moisture content.

The compressibility and compactibility of the chocolate powder into a tablet form can be concluded to be influenced by the particle size and particle size distribution. For a random distribution particle sizes, as the pressure applied increases, the density of the powder increases. The supported analysis were made by the Kawakita and Lüdde equation which showed the validation of data tabulated.

The analysis of dissolution test for the tablets was made to analysed the time taken required for a tablet to dissolve. It can also be concluded that, as the rate of dissolution lowers, less porous material will be obtained. Furthermore, higher compression force also leads to the lower dissolution rate.

For the shelf life of the tablet, it shows that the hygroscopicity of the powder have caused a faster absorption of moisture. As the moisture content increased, the shelf life of the tablet becomes shorter. It was also observed that the tablet had separation of liquid and become very sticky with formation of mould on the tablet surface.

For development of chocolate powder tablet, this study indicates that chocolate powder has the potential to be a formulated drink tablet and also it can be made as chewable candy that provides energy and good nutrition to the consumers. Furthermore, the formation of chocolate powder into a tablet formed provide a convenient feels in terms of storage space.

5.2. RECOMMENDATIONS FOR FUTURE STUDIES

In this study, it is found that the formation of powder into a tablet form was successfully developed.

In further studies, it is suggested to do the formulation of the powder towards the tablet development. As the original weight of one sachet of a formulated drink powder is 30g, to maintain the taste for one glass drink as it is formed in a 2g of chocolate tablet, this study are recommended.

Furthermore, the next suggestion for further studies is to do the shelf life test at the room temperature. As a random experiment has been tabulated along the accelerated shelf life test, the results show that the conditions of the tablet were still in the hard structure until day nineteen. Therefore, it is suggested to do this test to analyse the changes of the tablet conditions at the room temperature.

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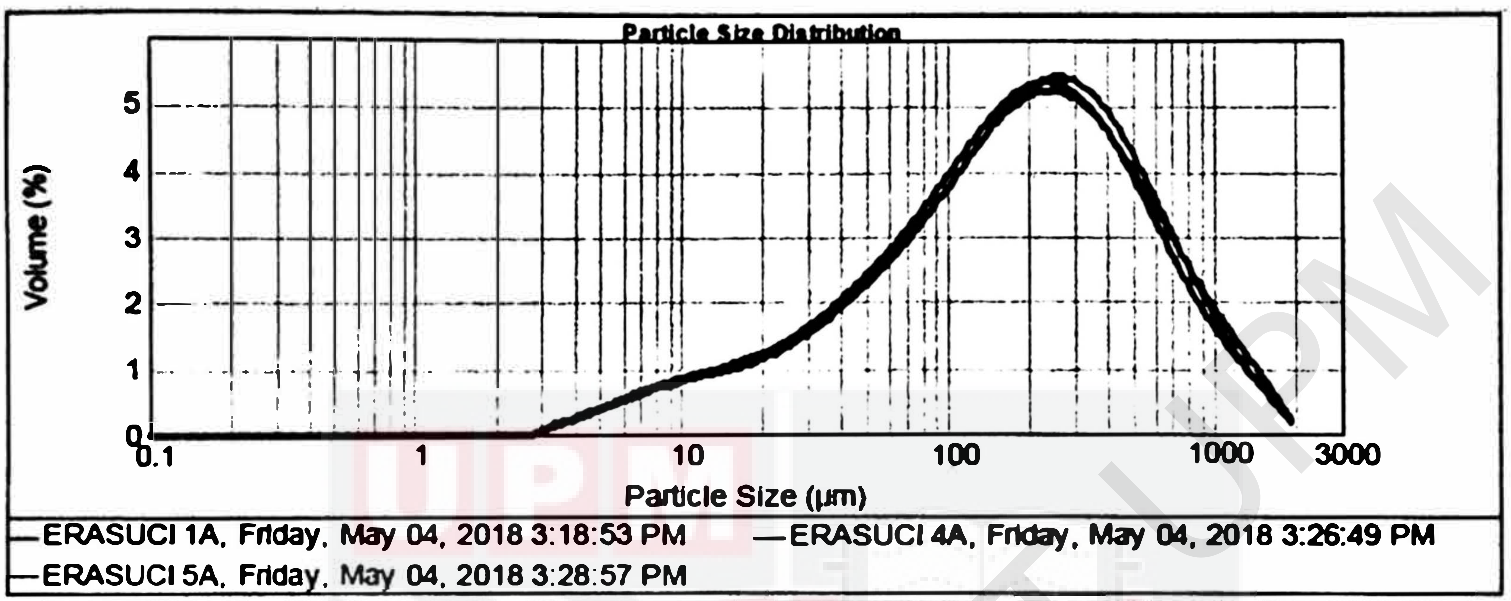
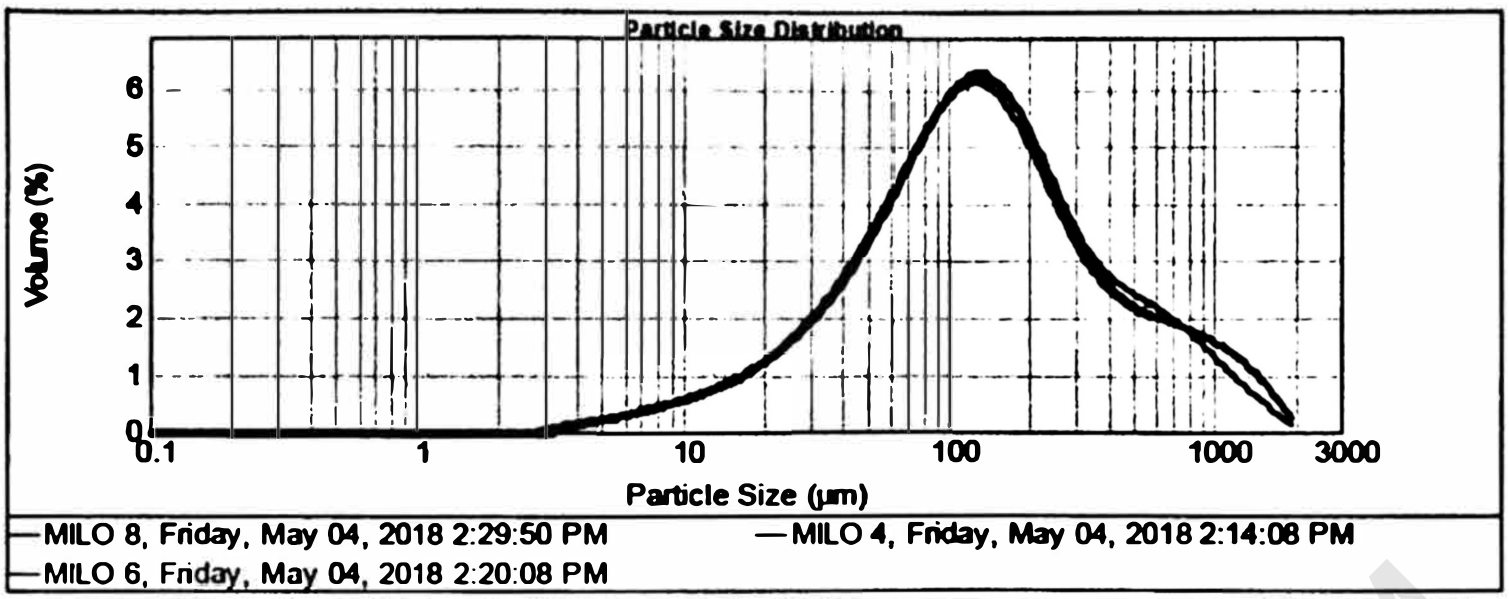
APPENDICES

APPENDIX A: THE PARTICLE SIZE DISTRIBUTION



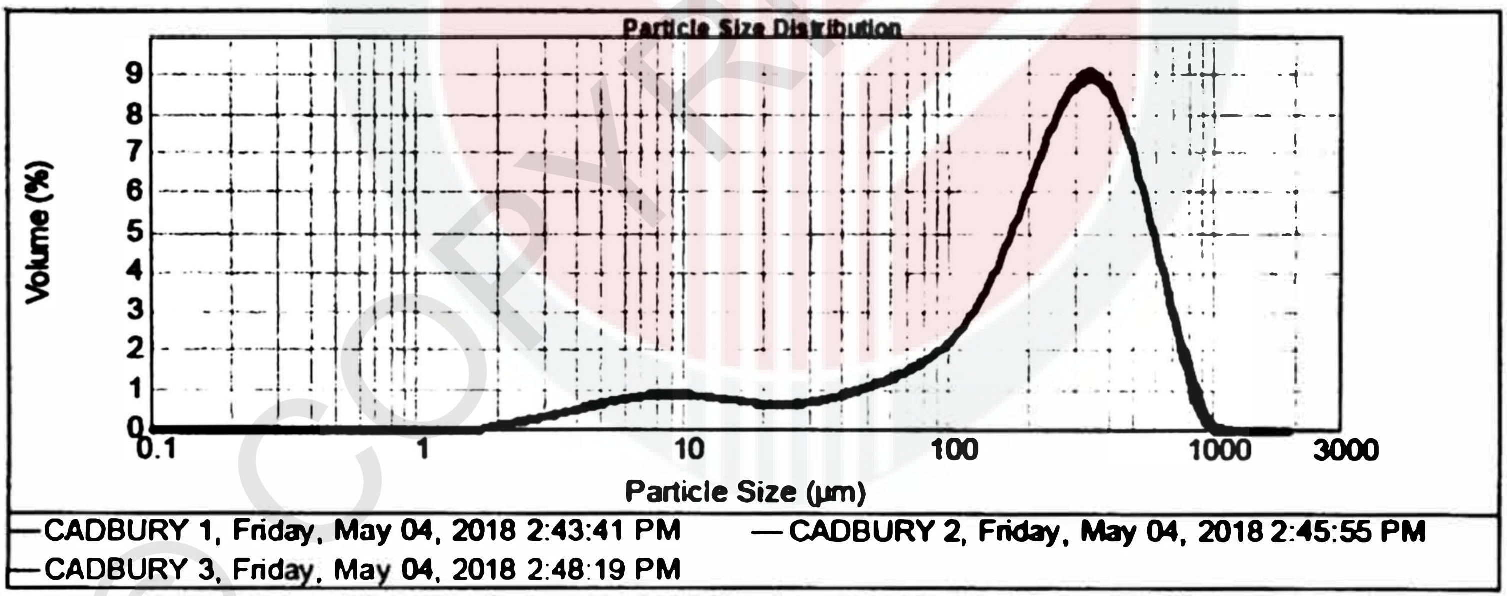
Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %	Size (µm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.00	11.482	0.85	120.226	4.11	1258.925	0.84
0.011	0.00	0.120	0.00	1.259	0.00	13.183	0.91	138.038	4.38	1445.440	0.59
0.013	0.00	0.138	0.00	1.445	0.00	15.136	0.97	158.489	4.61	1659.587	0.33
0.015	0.00	0.158	0.00	1.660	0.00	17.378	1.05	181.970	4.77	1905.461	0.08
0.017	0.00	0.182	0.00	1.905	0.00	19.953	1.15	208.930	4.84	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.00	22.909	1.27	239.883	4.83	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.00	26.303	1.41	275.423	4.72	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.07	30.200	1.57	316.228	4.51	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.15	34.674	1.75	363.078	4.22	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.23	39.811	1.96	416.869	3.85	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.32	45.709	2.17	478.630	3.44	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	0.41	52.481	2.40	549.541	2.99	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	0.49	60.256	2.66	630.957	2.55	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	0.58	69.183	2.91	724.436	2.13	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	0.66	79.433	3.20	831.764	1.75	8709.636	0.00
0.079	0.00	0.832	0.00	8.710	0.73	91.201	3.50	954.993	1.41	10000.000	
0.091	0.00	0.955	0.00	10.000	0.79	104.713	3.81	1096.478	1.11		
0.105		1.096		11.482		120.226		1258.925			

Appendix A. 1: The particle size distribution of powder A



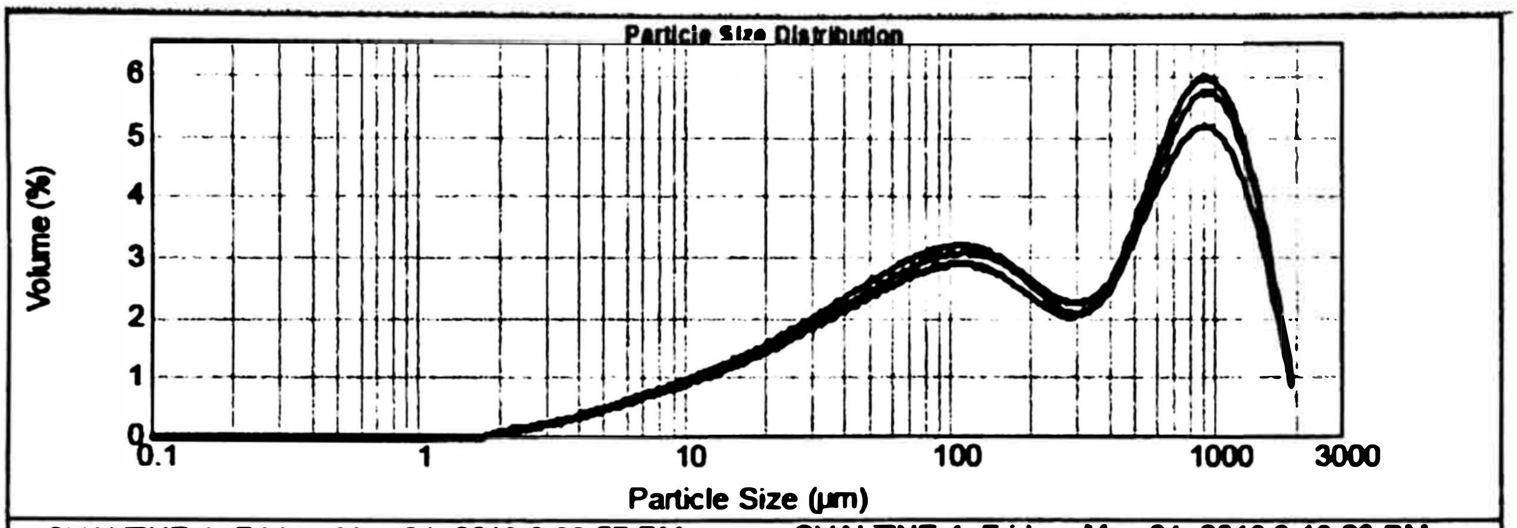
Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.00	11.482	0.60	120.226	6.67	1258.925	0.67
0.011	0.00	0.120	0.00	1.259	0.00	13.183	0.74	138.038	6.59	1445.440	0.46
0.013	0.00	0.138	0.00	1.445	0.00	15.136	0.88	158.489	6.32	1659.587	0.25
0.015	0.00	0.158	0.00	1.660	0.00	17.378	1.00	181.970	4.89	1905.461	0.06
0.017	0.00	0.182	0.00	1.905	0.00	19.953	1.17	208.930	4.37	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.00	22.909	1.37	239.883	3.80	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.00	26.303	1.60	275.423	3.29	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.00	30.200	1.88	316.228	2.86	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.08	34.674	2.17	363.078	2.59	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.12	39.811	2.53	416.869	2.31	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.17	45.709	2.94	478.630	2.16	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	0.22	52.481	3.40	549.541	2.01	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	0.27	60.256	3.80	630.957	1.86	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	0.33	69.183	4.40	724.436	1.65	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	0.40	79.433	4.88	831.764	1.41	8709.636	0.00
0.079	0.00	0.832	0.00	8.710	0.47	91.201	5.28	954.993	1.10	10000.000	
0.091	0.00	0.955	0.00	10.000	0.54	104.713	6.05	1096.478	0.80		
0.105		1.096		11.482		120.226		1258.925			

Appendix A. 2: The particle size distribution of powder B



Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.00	11.482	0.75	120.226	2.81	1258.925	0.00
0.011	0.00	0.120	0.00	1.259	0.00	13.183	0.70	138.038	3.51	1445.440	0.00
0.013	0.00	0.138	0.00	1.445	0.00	15.136	0.66	158.489	4.36	1659.587	0.00
0.015	0.00	0.158	0.00	1.660	0.00	17.378	0.60	181.970	5.32	1905.461	0.00
0.017	0.00	0.182	0.00	1.905	0.10	19.953	0.57	208.930	6.30	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.15	22.909	0.56	239.883	7.20	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.21	26.303	0.58	275.423	7.83	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.29	30.200	0.63	316.228	8.09	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.37	34.674	0.71	363.078	7.88	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.46	39.811	0.81	416.869	7.13	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.56	45.709	0.90	478.630	5.88	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	0.63	52.481	1.08	549.541	4.58	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	0.70	60.256	1.21	630.957	3.10	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	0.76	69.183	1.38	724.436	1.81	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	0.79	79.433	1.60	831.764	0.64	8709.636	0.00
0.079	0.00	0.832	0.00	8.710	0.80	91.201	1.88	954.993	0.00	10000.000	
0.091	0.00	0.955	0.00	10.000	0.78	104.713	2.28	1096.478	0.00		
0.105		1.096		11.482		120.226		1258.925			

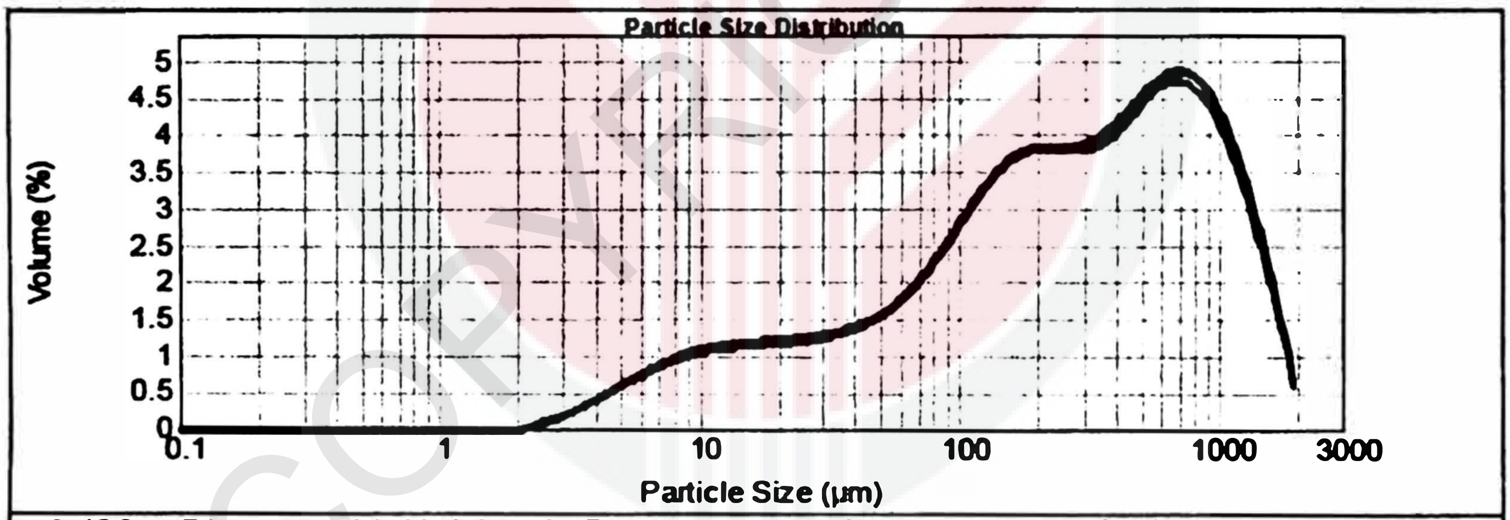
Appendix A. 3: The particle size distribution of powder C



—OVALTINE 1, Friday, May 04, 2018 3:02:57 PM —OVALTINE 4, Friday, May 04, 2018 3:10:26 PM
—OVALTINE 3, Friday, May 04, 2018 3:07:29 PM

Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.00	11.482	0.98	120.226	2.84	1258.925	3.56
0.011	0.00	0.120	0.00	1.259	0.00	13.183	1.08	138.038	2.74	1445.440	2.89
0.013	0.00	0.138	0.00	1.445	0.00	15.136	1.19	158.489	2.59	1659.587	1.55
0.015	0.00	0.158	0.00	1.660	0.00	17.378	1.30	181.970	2.41	1905.461	0.27
0.017	0.00	0.182	0.00	1.905	0.07	19.953	1.43	208.930	2.22	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.11	22.909	1.57	239.883	2.07	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.15	26.303	1.71	275.423	2.00	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.20	30.200	1.86	316.228	2.08	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.26	34.674	2.00	363.078	2.28	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.32	39.811	2.15	416.869	2.62	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.39	45.709	2.29	478.630	3.09	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	0.47	52.481	2.44	549.541	3.61	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	0.56	60.256	2.57	630.957	4.11	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	0.63	69.183	2.70	724.436	4.49	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	0.71	79.433	2.79	831.764	4.69	8709.636	0.00
0.079	0.00	0.832	0.00	8.710	0.80	91.201	2.86	954.993	4.57	10000.000	
0.091	0.00	0.955	0.00	10.000	0.86	104.713	2.89	1096.478	4.20		
0.105		1.096		11.482		120.226		1258.925			

Appendix A. 4: The particle size distribution of powder D



—OLIGO 1, Friday, May 04, 2018 2:54:01 PM —OLIGO 2, Friday, May 04, 2018 2:55:54 PM
—OLIGO 3, Friday, May 04, 2018 2:58:02 PM

Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %	Size (μm)	Volume In %
0.010	0.00	0.105	0.00	1.096	0.00	11.482	1.03	120.226	2.99	1258.925	2.78
0.011	0.00	0.120	0.00	1.259	0.00	13.183	1.05	138.038	3.21	1445.440	2.04
0.013	0.00	0.138	0.00	1.445	0.00	15.136	1.07	158.489	3.36	1659.587	1.18
0.015	0.00	0.158	0.00	1.660	0.00	17.378	1.09	181.970	3.43	1905.461	0.28
0.017	0.00	0.182	0.00	1.905	0.00	19.953	1.10	208.930	3.44	2187.762	0.00
0.020	0.00	0.209	0.00	2.188	0.06	22.909	1.12	239.883	3.42	2511.886	0.00
0.023	0.00	0.240	0.00	2.512	0.11	26.303	1.14	275.423	3.42	2884.032	0.00
0.026	0.00	0.275	0.00	2.884	0.18	30.200	1.17	316.228	3.48	3311.311	0.00
0.030	0.00	0.316	0.00	3.311	0.27	34.674	1.22	363.078	3.59	3801.894	0.00
0.035	0.00	0.363	0.00	3.802	0.37	39.811	1.28	416.869	3.78	4365.158	0.00
0.040	0.00	0.417	0.00	4.365	0.48	45.709	1.37	478.630	4.00	5011.872	0.00
0.046	0.00	0.479	0.00	5.012	0.59	52.481	1.50	549.541	4.21	5754.399	0.00
0.052	0.00	0.550	0.00	5.754	0.69	60.256	1.67	630.957	4.35	6606.934	0.00
0.060	0.00	0.631	0.00	6.607	0.79	69.183	1.89	724.436	4.35	7585.776	0.00
0.069	0.00	0.724	0.00	7.586	0.88	79.433	2.15	831.764	4.19	8709.636	0.00
0.079	0.00	0.832	0.00	8.710	0.94	91.201	2.43	954.993	3.88	10000.000	
0.091	0.00	0.955	0.00	10.000	1.00	104.713	2.73	1096.478	3.57		
0.105		1.096		11.482		120.226		1258.925			

Appendix A. 5: The particle size distribution of powder E

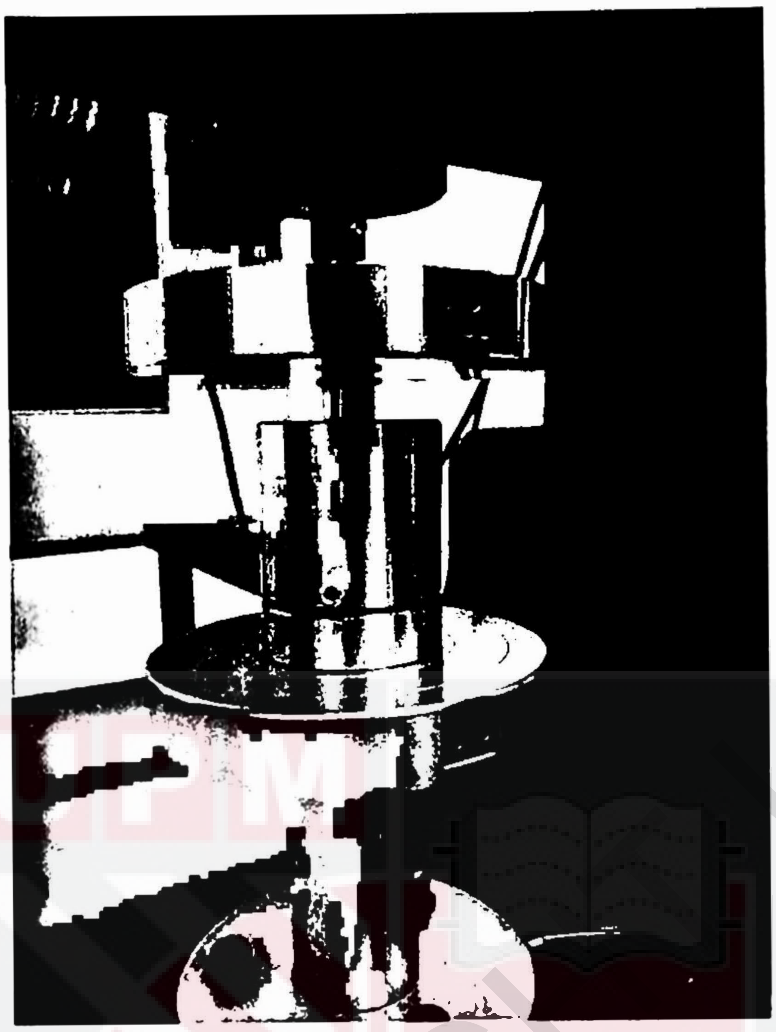
APPENDIX B: THE RAW MATERIALS USED

Appendix B. 1: The main raw material of chocolate powder from the commercialised local chocolate malted drink powder.

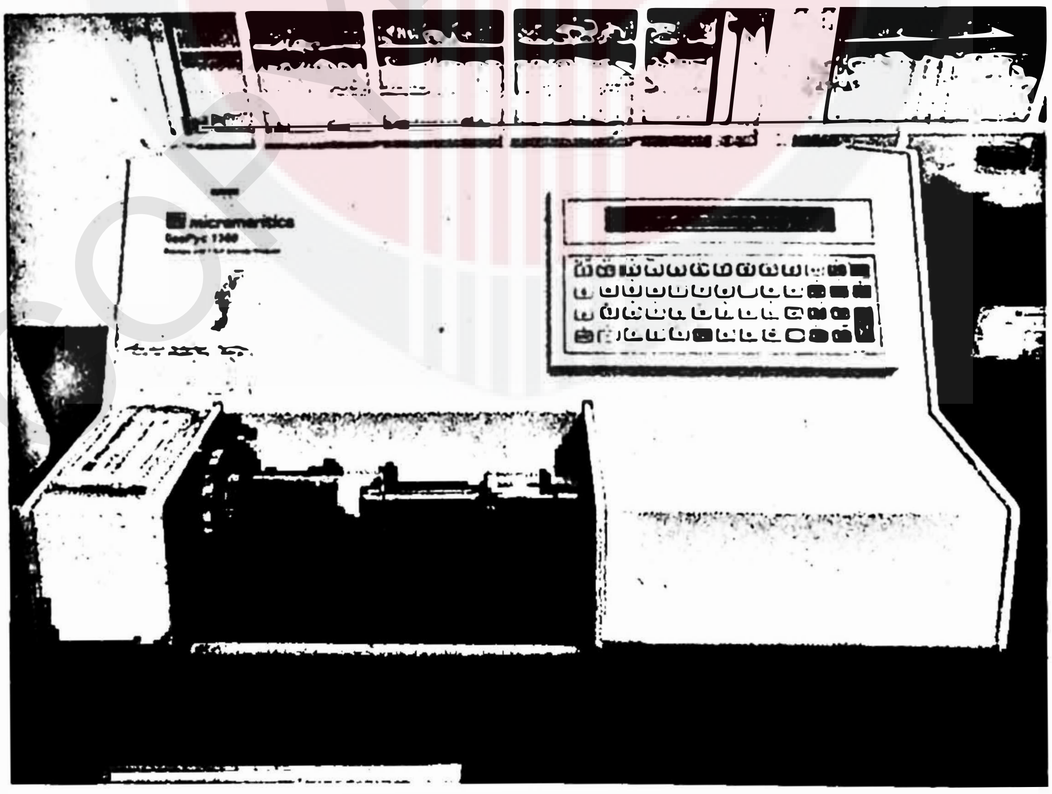
Oligo 3 in 1 **Ovaltine 3 in 1** **Milo 3 in 1** **Cadbury 3 in 1**

Appendix B. 2: The commercialised chocolate malted drink powders used.

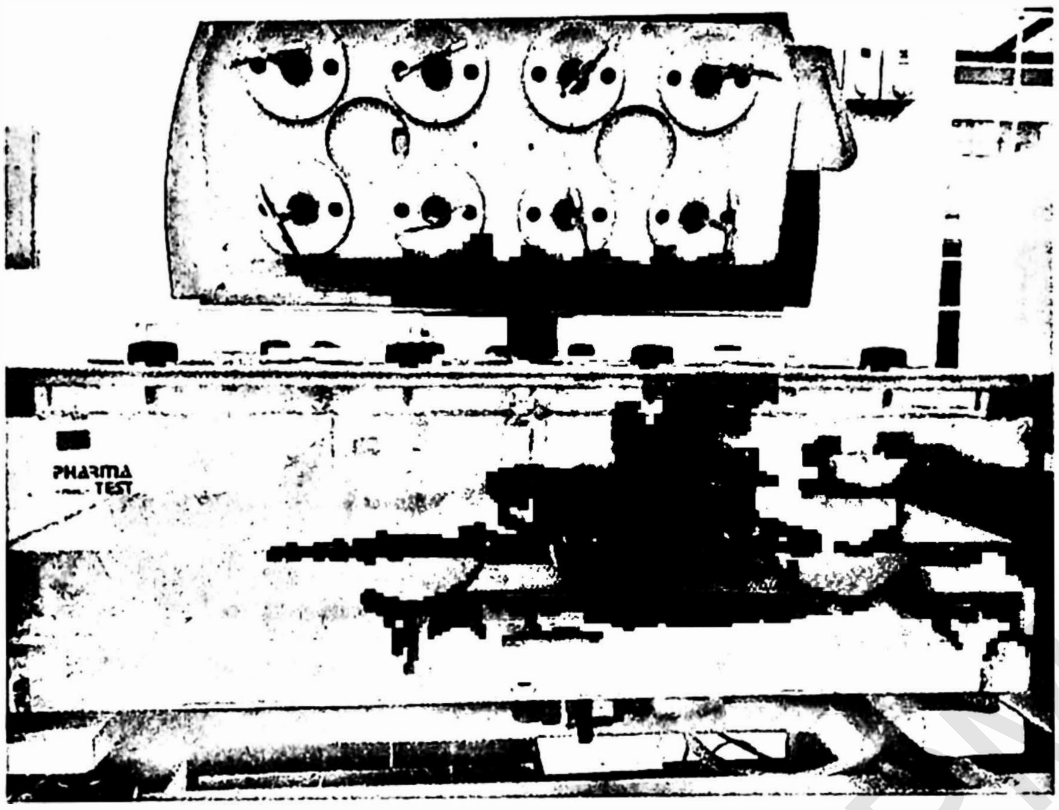
APPENDIX C: THE EQUIPMENTS USED



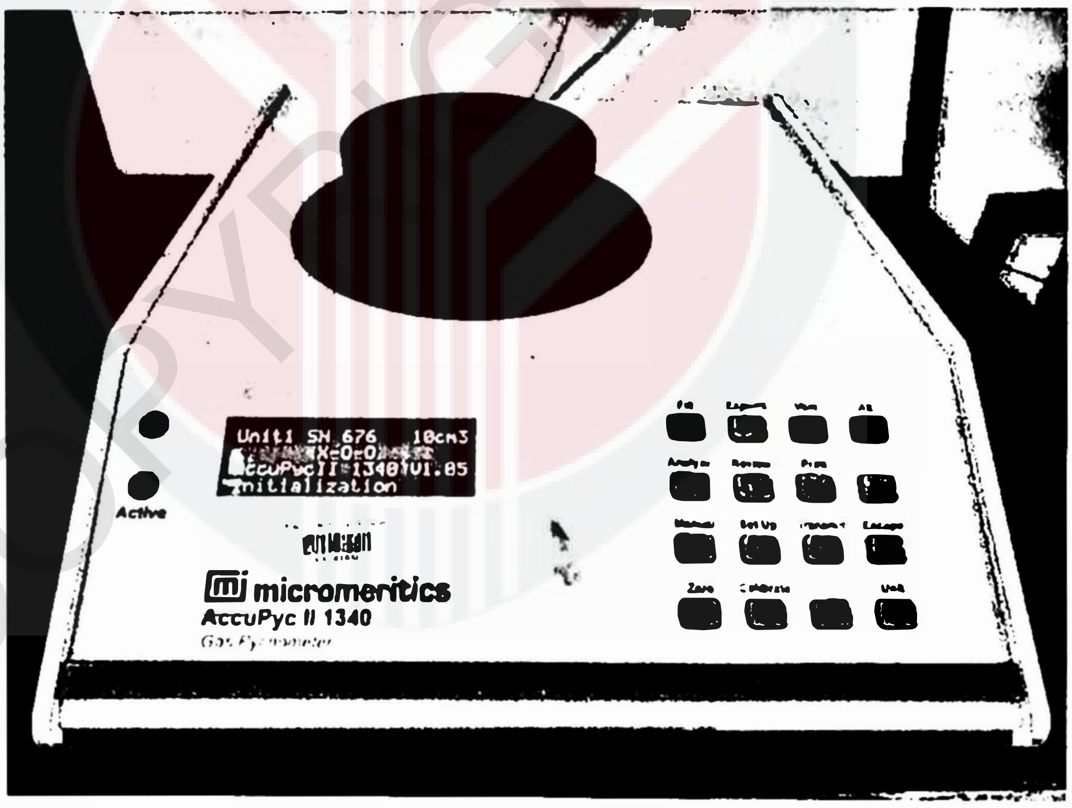
Appendix C. 1: The compaction machine (Instron Universal Testing Machine-5566, Canton MA, U.K.)



Appendix C. 2: The envelope density analyser with T.A.P. density option (GeoPyc 1360 Pycnometer Micromeritics, U.S.A.).



Appendix C. 3: The dissolution tester (Pharma Test D-63512, Germany)



Appendix C. 4: The true density analyzer (AccuPyc II 1340 Micromeritics, U.S.A.).

APPENDIX D: THE ACCELERATED SHELF LIFE

Appendix D. 1: The chocolate tablet inside the desiccator for an accelerated shelf life testing.