UNIVERSITI PUTRA MALAYSIA

ANALYSIS OF PHYSICAL AND FLOW ABILITY PROPERTIES IN MEASURING CAKING FORMATION OF SELECTED COMMERCIAL SOFT BROWN SUGARS AVAILABLE IN THE MALAYSIAN MARKET

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ABSTRACT

Brown sugar is a sugar product with a distinctive brown colour due to the presence of molasses, known for its added flavour and faster caramelization properties are widely used as an ingredient for baking goods. The problem of caking in soft brown sugar has been causing quality deterioration as well as interfering manufacturing and handling processes in the industry. The aim of this study is to determine the possible factors leading to the caking of sugar in terms of physical properties, flowability properties and storage conditions. The investigated in this study are moisture content, colour absorbance as an indication of molasses content, mean particle size, and flowability properties of Carr's Compressibility Index and Hausner Ratio. A directly proportional graph of correlation between moisture and colour absorbance was established. The flowability properties of sugar is seemed to relate directly with the mean particle sizes. Compaction of brown sugar into sugar tablets and six days open air storage was carried out to imitate industrial warehouse storage conditions in order to investigate the factors of caking. The presence of excess moisture on the surface of sugar particles and fine mean particle sizes are found to be some of the factors which causes formation of solid bridges between particles and characterize its aptitude to cake. Flowability before and after six days open air storage is measured using the fixed funnel method in order to predict the flow property. In conclusion, the factors that causes caking are found to be high in moisture content and fine particle size as well as open-air and compacting storage condition.

ABSTRAK

Gula perang lembut adalah produk gula yang khas kerana kehadiran molase, terkenal dengan rasa tambahan dan sifat karamelisasi yang lebih cepat digunakan secara meluas sebagai ramuan untuk kegunaan bakeri. Masalah pembentukan kek gula dalam pembuatan gula perang yang lembut menyebabkan kemerosotan kualiti serta mengganggu pemprosesan dan pengendalian dalam industri. Tujuan kajian ini adalah untuk menentukan faktor-faktor yang mungkin membawa kepada pengambilan gula dari segi sifat fizikal, sifat aliran dan keadaan penyimpanan. Yang diteliti dalam kajian ini adalah kandungan kelembapan, penyerapan warna sebagai petunjuk kandungan molase, ukuran zarah rata-rata, dan sifat aliran yang dapat dilakukan oleh *Carr's Compressibility Index* dan *Hausner Ratio*. Graf korelasi berkadar langsung antara kelembapan dan penyerapan warna telah dibuat. Sifat aliran gula nampaknya berkaitan langsung dengan ukuran zarah min. Pemadatan gula merah ke dalam tablet gula dan penyimpanan udara terbuka selama enam hari dilakukan untuk meniru keadaan penyimpanan gudang industri untuk menyelidiki faktor-faktor kek. Kehadiran kelembapan berlebihan pada permukaan zarah gula dan ukuran zarah rata-rata halus didapati merupakan beberapa faktor yang menyebabkan pembentukan jambatan padat antara zarah dan menjadi ciri kebolehannya untuk membuat kek. Kemudahaliran sebelum dan selepas enam hari penyimpanan udara terbuka diukur menggunakan kaedah corong tetap untuk meramalkan sifat aliran. Kesimpulannya, faktor penyebab kek didapati tinggi kandungan kelembapan dan saiz zarah halus serta keadaan penyimpanan terbuka dan pemadatan.

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LIST OF ABBREVIATIONS

ICUMSA	International Commission for Uniform Methods of Sugar Analysis
IU	ICUMSA Unit
SB	Soft Brown Sugar
CI	Carr's Compressibility Index
HR	Hausner Ratio
p	Porosity
D	Diameter
R	Radius
F	Force
α	Angle of Repose
h	Height of pile
ρ	Density
ERH	Equilibrium Relative Humidity
CV	Coefficient of Variance
RT	Refractometric
W	Work done in Joule (J)
P	Breakage load in Newton (N)
σ_t	Tensile Strength in Pascals (Pa)

CHAPTER 1

INTRODUCTION

1.1 Background

Sugar, an essential ingredient in almost all food processing industries, does not only act as a sweetener, but plays a vital role in preservations, fermentations, colour and texture of food (Zaitoun et al., 2019). It is also an important source of energy supplied by a processed food to human body as the brain requires 130 grams of glucose a day to achieve and maintain optimum brain and nervous function (Gal et al., 2018).

Sugar denominates a broad phraseology which represents many different types of sweet carbohydrates made up of carbons, hydrogens and oxygens but is conventionally used to define sucrose, a disaccharide, which is a molecule composed of two monosaccharides or simple sugars structures joined by glycosidic bonds (Groves, 2018). Glucose is a major product of photosynthesis which occurs to allow plants to turn sunlight into energy and forms as a stored energy in the plant (*Sucrose - Definition, Structure, Uses | Biology Dictionary*, 2018). In order to obtain sucrose, fructose molecule derived from plants will form a linkage with glucose through an enzymatic extraction extracting a molecule of water by condensation reaction (Hassid & Putman, 1950).

Granulated sugar is one of the most widely used type of sugar and is the highest grade produced from the first refined syrup followed by other typical commercial sugars such as caster sugar, icing sugar, light brown sugar and dark brown sugar (Kaminski, 2020). These grades are differentiated by the particle size distribution and colour of sugar. Particle size distribution of sugar products is a fundamental criterion for the final product quality and used as a control indicator mechanism in sugar production. Coarse sugar has sturdy structure and is more resistant to heat, whereas finer particle sugar types like caster and confectionary or icing sugars make it easier to dissolve. The varying granulation of the sugar crystals greatly influences the handling of sugar, the dissolution time for batter preparation and the smoothness of food textures (Tiefenbacher, 2019). Granular materials are often subjected to caking phenomenon as it is a process of lumps bonding together by inter-particle forces (Specht, 2006). Sugar is the most common example of particles associated with caking due to its availability in granular form.

Brown sugar is a sugar product with a distinctive brown colour due to the presence of molasses, known for its added flavour and faster caramelization properties are widely used as an ingredient for baking goods. Brown sugar is one of the four mainly used sugar in Malaysia, according to *Malaysia Sugar Market Guide - (Tridge*, n.d.), with white sugar, fine sugar and palm sugar being the other three. The global market was valued at USD 72310 million for white sugar, and USD 33837 million for brown sugar during 2019 (More, 2021; Wood, 2020). From the local perspective, white sugar is valued at MYR 4960 million and no data was reported for local market revenue on 2019 (Hirschmann, 2021).

The sugar quality measures and analytical method of quality assurance tests for the final product in sugar industries are fixed by the international standard published by The International Commission for Uniform Methods of Sugar Analysis (ICUMSA) that was founded on 1987 and recognized by Codex Alimentarius Commission, the OIML, the EU and the US Food Chemicals Codex.

The term 'cake' by definition is a verb that occurs when a substance dries or harden into a solid mass (Oxford English Dictionary). In the crystallization field, it is also known as agglomeration which is a time-dependent phenomenon that can be defined as the process of formation of lumps or masses (Bradley & Deng, 2013). This phenomenon causes particles to lose its free-flowing properties. Hence, soft brown sugar has a tendency to cake since it appears in granular form, and causes particles to lose its free-flowing properties (Johanson, 2014).

The formation of darker range sugar colour is also another relevant parameter of sugar that breeds more range of sugar products. Colour is the crucial quality specifications of sugar where the first refine will produce whitest sugar with lowest colour reading in ICUMSA unit and increases as the boiling stages persists. Brown sugar is the outcome of multiple boiling stages in a vacuum pan before the exhaustion of final molasses, coated with molasses that made up the colour and flavour of the sugar. It does not contain any significant nutritional benefits other than giving aromatic and flavour to the end products (Temmers, 2017). Sugar has a hygroscopic property that holds onto moisture and do not stale as quickly, but molasses has even higher hygroscopic properties which makes brown sugar produces chewier products compared to white sugars.

Since sugar plays a major role in food industries, according to Gray (2020), sugar refineries faces high demand of sugar and it keeps increasing with the growth of food innovations, with the compound annual growth rate (CAGR) of 9.5% as reported by BCC Research (Dewan, 2015; Jenkins et al., 2015). A vital factor in continued sales growth is the quality of the product (Quain, 2019; Takeuchi & Quelch, 1983). Quality control is an essential part of sugar production process. It focuses not only on the look, texture and taste, but also on the solubility, propensity to aggregate, agglomerate and other properties that are essential for industrial processes with sugar as their raw material (*Quality Control of Sugar - Knowledge Base - Applications*, n.d.). ICUMSA has established the ICUMSA 420 standard for classifying the consistency of consuming sugar by colour. ICUMSA 420 has developed a standard for the values of edible sugar chromaticity from 45IU to 800IU (IU, ICUMSA Units). The lower the value, the lower the impurity (ACTTR, 2018).

1.2 Problem Statement

Since quality make an important contribution for product profitability, caking phenomenon is considered to be one of the deficiencies of sugar. Caking is an undesirable phenomenon for sugar crystals for it interferes the storage handling process, transportation process and production processing. It is detrimental to the industry considering the loss of functionality and a lower quality product. Moisture migration, storage conditions, particle size and attrition are the primary causes that can lead to formation of caking.

Brown sugar, in addition to pure sucrose, is the coating of molasses that imparts the distinctive flavour and colour (Lachmann & Haddonfield, 1959). The molasses having high properties of hydrophilic, hygroscopic and sticky form encourages the formation of lumps and thus resulting in the caking mechanism of sugar (Lee, 2008). When final product is stored for a few days, transported or exposed to the air at ambient temperature, the sugar has a tendency to lose some of its moisture and tend to cake and harden that forms lumps which are difficult to break and causes a number of handling and mixing problems to the industrial user and consumer (Lachmann & Haddonfield, 1959).

The product brown sugar which is also commercially known as soft brown sugar has a character of a dark brown colour with ICUMSA unit of 6000 and a fine particle size with mean particle size of 300μm (Edwards, 2000).

A granulation technique was introduced as a solution to achieve free-flowing, noncaking sugar granules with larger particle size where during the boiling process that takes place in a vacuum pan, half of the capacity in the pan which has crystallized is transferred to another pan as seeds, and later moved back in to allow even boil of the sugar. This solves the caking formation but results in a large mean particle size of particle size formation which ranges from 600μm to 1900μm and is no longer soft as it claims.

This study relates to the discovery of factors affecting caking in soft brown sugar by evaluating the physical properties and flowability properties of commercially available soft brown sugar, because there is no statistical data readily available to assess the trend in brown sugar caking. Since caking of granulated materials are often associated with moisture contents and particle size, apart from just looking into physical and flowability parameters, it is also equally important to correlate the properties to validate the correspondence between the properties and to eliminate the possible factors leading to caking. Lastly, the effect of different storage conditions was investigated to the best suitable condition of storage to prevent caking.

1.3 Objectives

Based on the problem that has been stated, this research aimed to study the factors contributing to cake formation in soft brown sugars by:

1. To evaluate the physical properties and flowability properties of different types of commercial soft brown sugar available in the market,

2. To establish the correlation between the physical properties and the flowability properties of soft brown sugar, and

3. To determine the effect of different storage conditions on flowability properties of soft brown sugar.

1.4 Scope of Limitation

The scope of this study is limited to Soft Brown Sugar, a variety of refined sugar extracted from cane sugar. The samples were all manufactured locally from Malaysia and one from Philippines. The storage conditions are also limited to industrial method which are open-air environment and stacked by 0.5 ton of bulk package. Other conditions of storage will not be included in this study.

The results established by this study contributes to the industrial research and development to focus on few factors that plays a major role in formation of caking.

CHAPTER 2

LITERATURE REVIEW

2.1 Sugar Refinery Production Process

2.1.1 Conventional Method of Sugar Refinery Production

A sugar refinery is a refinery that undergoes a method of processing raw sugar from its plant source of either beet sugar or cane sugar into white refined sugar and a few other grades of refined sugar including brown sugar. The refining process, which is also known as purification process is necessary to remove the film of molasses containing impurities and colouring matter covering the sugar crystals of raw sugar produced by sugar mills (*Purification of Sugar - The Canadian Sugar Institute*, n.d.). The following process flow is according to Baikow (2013).

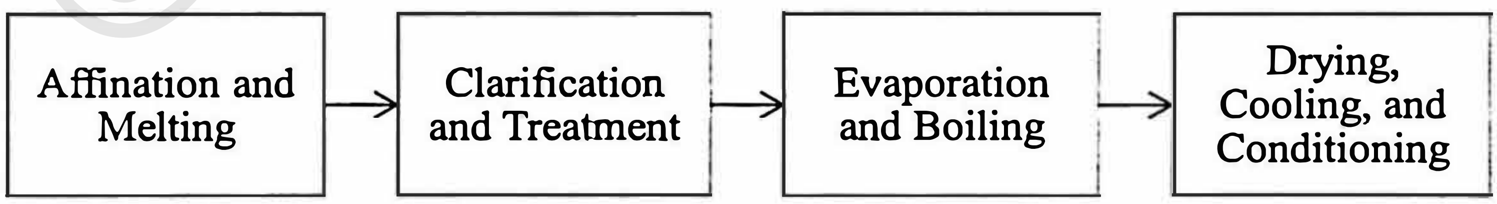


Figure 2-1: Process Flow of Sugar Refining (Baikow, 2013).

The refining process is started off with affination process, a purely mechanical process that consist of softening the adhering molasses film, rubbing it off with other crystals, dissolving it in affination greens which are also known as affinated molasses. This process is a mingling of raw sugar with heated affinated molasses which is later obtained after centrifuged with a ratio affinated molasses to sugar of 75:25, to take off the adhering molasses coating from the surface of the raw sugar crystal. It is separated in a centrifugal machine which spins at 1200 rpm at most, thereby separating particles of different densities by centrifugal force where the denser particle which is the molasses will move outward in radial direction and the other way goes to the less dense particles, sugar crystals where it moves towards the centre, and then is further washed by a water spray to free it off from the remaining molasses. The washed sugar obtained from separating with affinated molasses is now in a semi-liquid state is mixed with sweet water and crop liquor before it goes down to the melter tank where brix of the raw liquor is controlled at 61-65 by adjusting the ratio of addition of sweet water. Raw liquor is sieved with the addition of alpha amylase enzyme to help break down starch chains into low molecular weight products which are glucose, maltose and maltotriose and being transferred to the raw liquor buffer tank. The main purpose of this process is to remove the high-coloured molasses film.

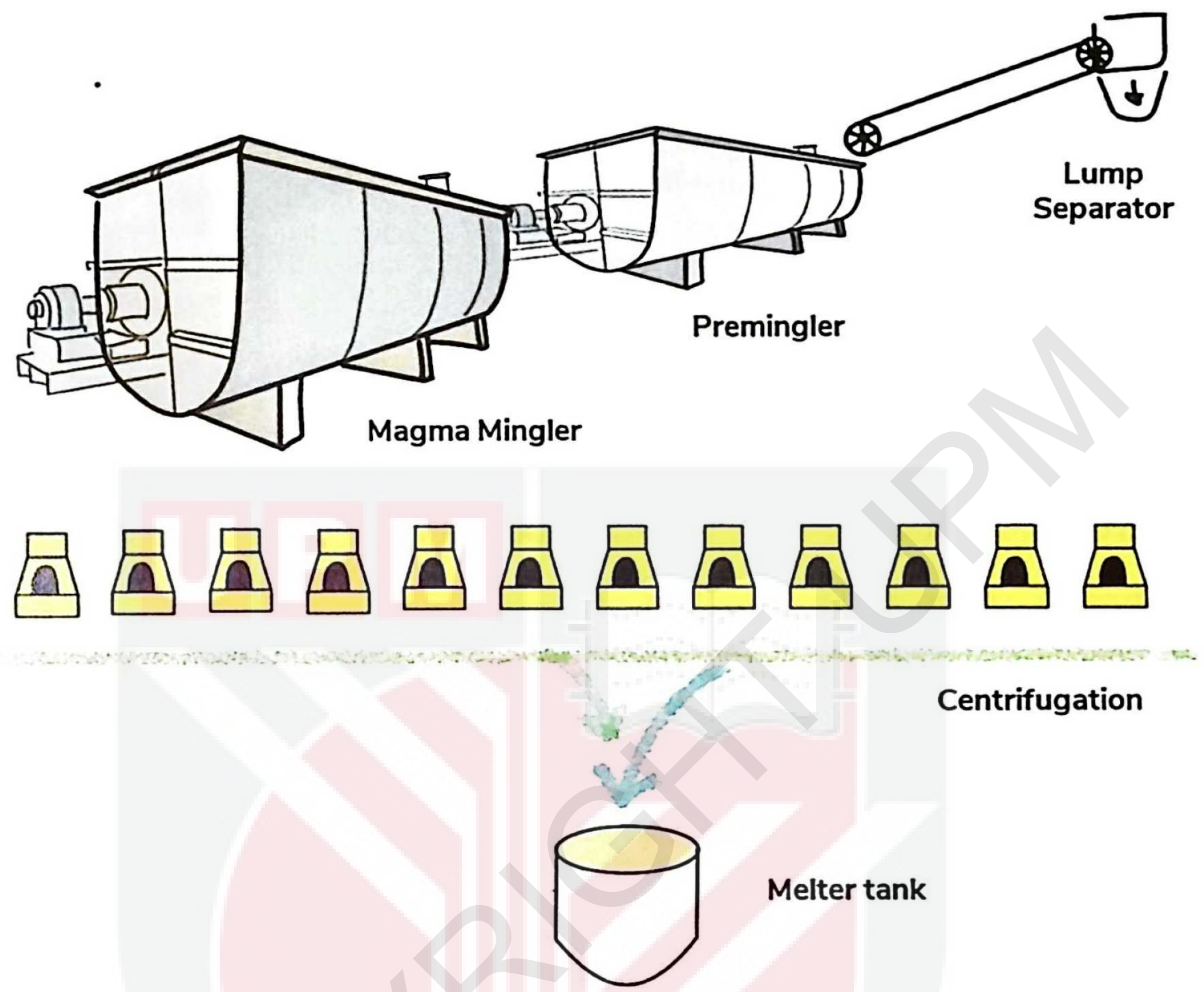


Figure 2-2: Affination process of sugar refining

The clarification step is required to remove all possible non-sugar components mainly the colour compound of the sugar, which is why this process is also known as decolourization. Two methods for clarification were suggested which is phosphatation and carbonation. The above Figure 2-3 shows the carbonation of liquor for clarification. Carbonation takes place by the reaction of carbon dioxide gas and hydrated lime that forms calcium carbonate to precipitate impurities. Carbonated liquors from the carbonated liquor tank will go through filtration in a rotary leaf filter to filter off the calcium carbonate sludge, producing mud cake and sweet water of 9-

10 brix reading by a filter press. Clear liquor is further decolourized in an ion exchange resin (IER) column where resin is inserted by manual, regenerated with NaCl salt after 10-15 hours of use, and regenerated again with hydrochloric acid (HCl) after 40 cycle uses. The process undergoes a secondary filtration using a smaller size of rotary leaf filter producing sludge cake and fine liquor with brix controlled at 65.

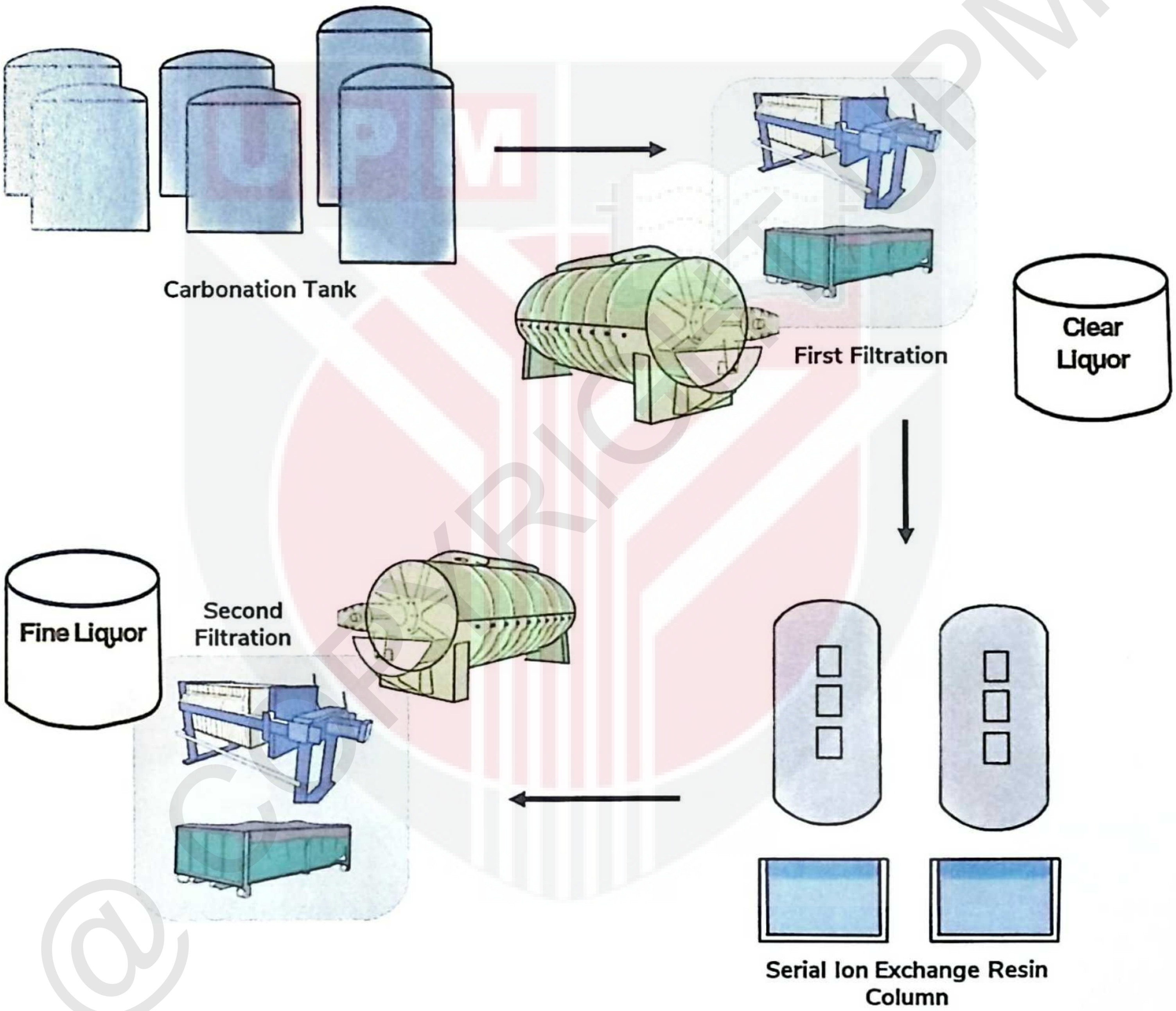


Figure 2-3: Clarification process of sugar refinery by carbonation

Fine clarified liquor with 65 brix is passed through a plate evaporator to elevate the brix reading to 68-69 and then boiled in a vacuum pan. The boiling takes place in a vacuum condition to keep the temperature at low level to minimize colour formation and the inversion or degradation of sucrose.

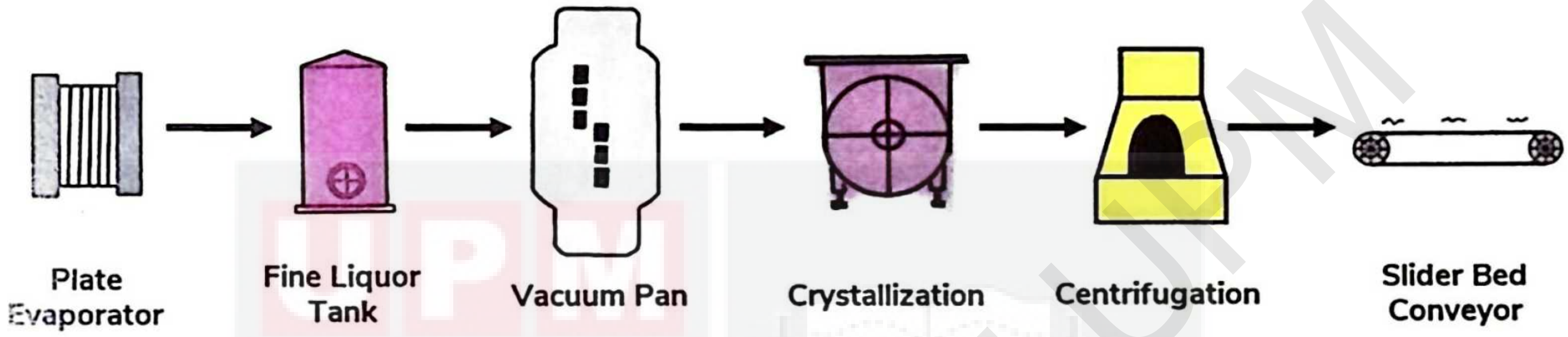


Figure 2-4: Evaporation and boiling process in sugar refinery process

The boiling time rises as the boiling stage extends, and longer boiling time is needed if the brix of sugar is lower. Boiled and crystallized sugar will go through another centrifugal step to separate sugar from molasses that is to be further boiled in the next stage. The first stage produces Industrial Grade sugars (IG), second to fourth stage produces commercial white sugar of C1 and A1 and the next few stages produces few brown sugar grades before finalized as final molasses. Sugars are transferred to finishing process through a grasshopper or a slider bed conveyor where it vibrates as it moves to avoid clumping of sugar.

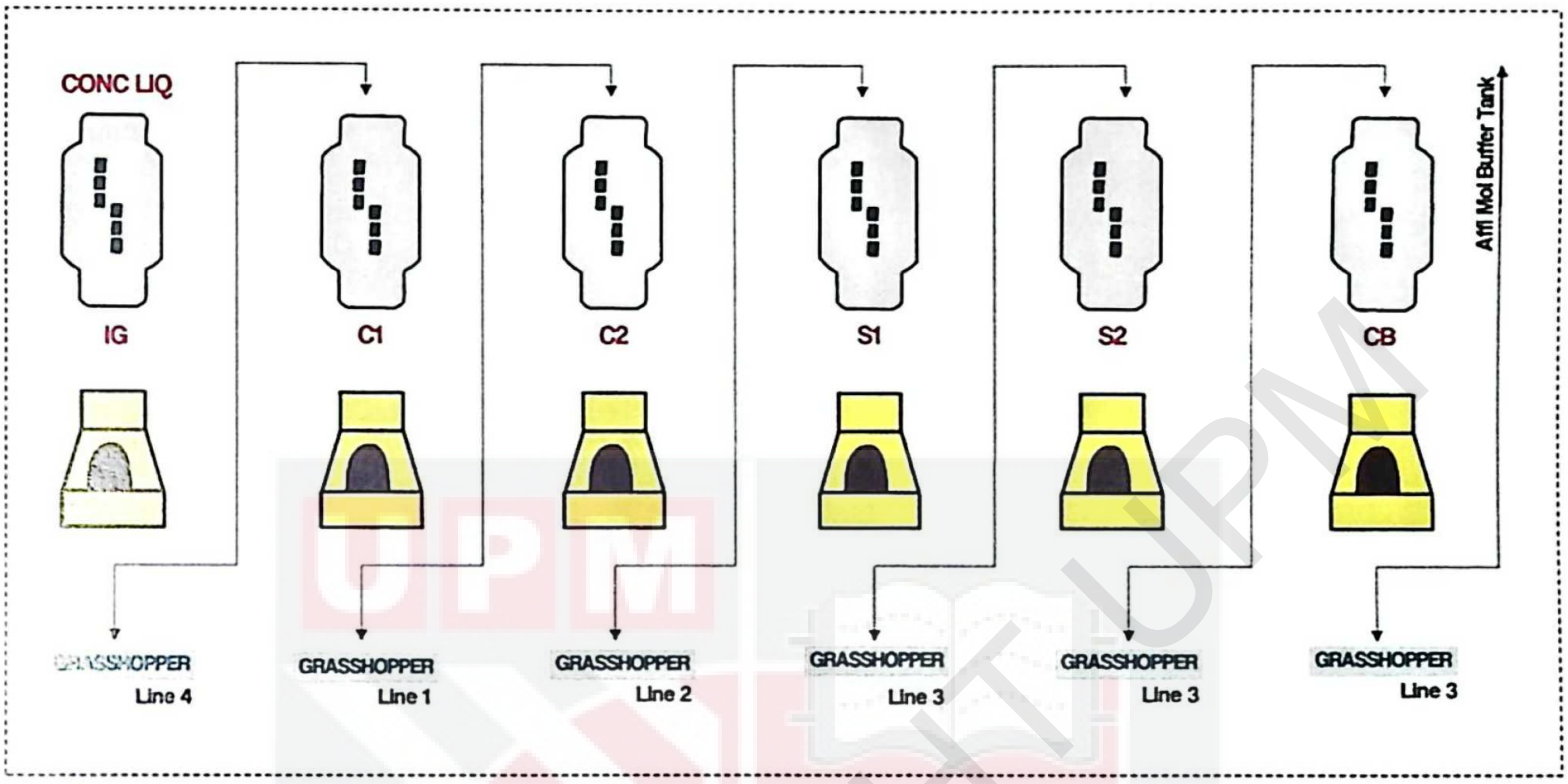


Figure 2-5: Boiling stages of sugar refining process

2.1.2 Recovery House for Soft Brown Sugar Production

Sugar from the fourth or fifth stage of boiling will undergo a further boiling cycle with the addition of a ratio of molasses, until the final molasses is exhausted to produce brown sugar. This process is to recover the maximum amount of sucrose possible. A few grades of brown sugar products are produced from the recovery house which includes light brown sugar, dark brown sugar and aromatic brown sugar. The products differ by colour, such that the more E-magma added the higher the colour.

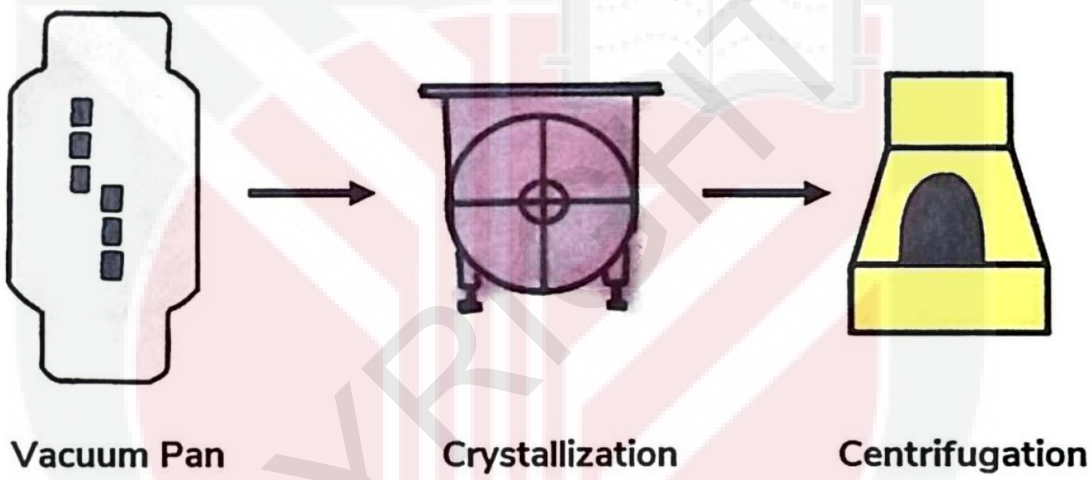


Figure 2-6: Boiling stage in recovery house of sugar refining process for brown sugar production

2.2 Physical Properties of Soft Brown Sugar

The main sources of sugar are sugar beets and sugar cane and are usually sold without its plant source clearly identified. Both extracts equal amount of sucrose and is chemically equivalent. From the same extraction, sugar is able to be distinguished into few grades that are classified according to the sugar colour ranging from 50 ICUMSA for white sugar types up to 6000 ICUMSA for brown sugar types which results from number of boiling stages and the sugar particle sizes of mean particle size ranging from 75μm to 750μm, with moisture percentage below 1% (based on quality specifications from Sugar Industrial Training).

Those sugar grades established varieties of sugar types for retails and goes by many different names, the common ones differentiated by colour are known as white sugar and brown sugar, and the ones classified by sizes are coarse sugar, fine sugar, castor sugar and icing sugar or confectionary sugar.

From the brown sugar type, it can be extensively categorised into different shades of colour from light brown sugar to dark brown sugar and also the particle sizes associated. Soft brown sugar is dark in colour quantified at minimum of 3000 ICUMSA unit with a soft granular particle size with a mean particle size of 300μm (Edwards, 2000).

Regardless of the sugar type, the chemical properties for all sugar remains the same with the chemical formula of sucrose which is $C_{12}H_{22}O_{11}$ thus having the same molar mass or molecular weight, solubility and density value but different physical appearance (*Sucrose (C12H22O11) - Structure, Properties, Uses, and FAQs*, n.d.).

Based on the processing method of crystallization that sugar undergo, the structure of sugar is classified as crystalline. It is made of a repeating unit-cell which is sucrose molecules. (Palzer & Sommer, 2011).

2.3 Caking Quantifications

2.3.1 Interparticle Interactions

Based on a recent research on caking of white crystalline sugar by Rogé & Mathlouthi, (2003), caking of crystalline structured particles are caused by a wetting of particle surface that causes plasticization and dissolution to happen.

High humidity state causes the liquid layer to form on the crystal surfaces that will initiate the development of liquid bridges. The high content of water contributes to plastic deformation, making crystals stick together. This briefly causes the dissolution and recrystallization of sugar crystals that makes them grow together as a lumped particle (Chen et al., 2015).

Based on Figure 2-7, 4 stages of formation of caking described by Gutman (1995) are,

1. Pendular stage

This is the initial stage of caking where the particle behaviour is still free flowing,

2. Funicular stage

This stage established permanent contacts between particles,

3. Capillary stage

This stage is reached when the moisture content elevates such that it is sufficient to develop liquid bridges between particles,

4. Drop stage

The final stage of when the dissolution of particles is obtained and is dominating.

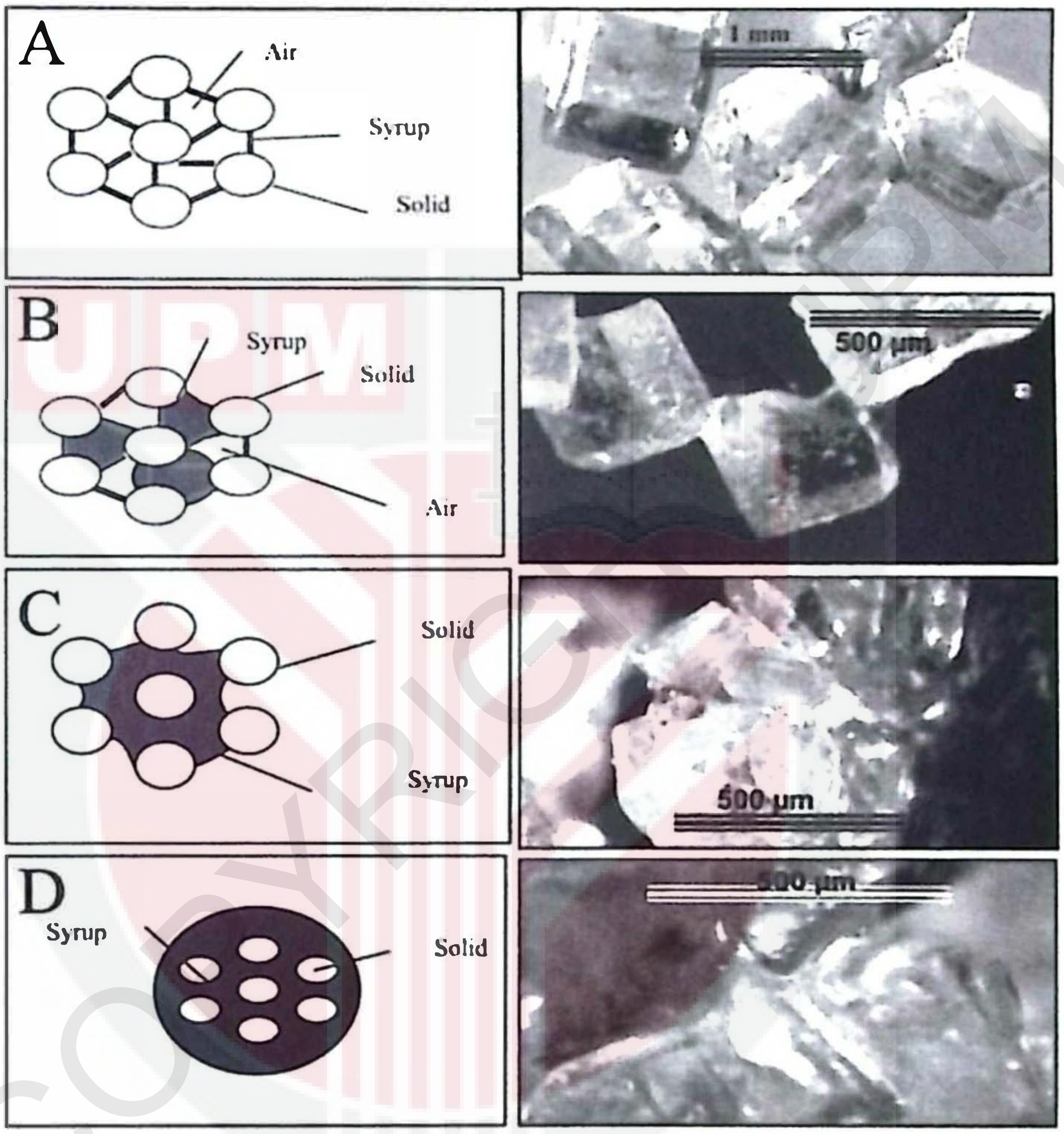


Figure 2-7: Schematic and microphotography illustration of the stages of wetting of sugar crystals by Gutman (1995)

A quantitative measure for this condition is defined by Aguilera et al., (1995) as the state of the system at any time relative to the initial state. Two morphological indicators of the state of the system are:

1. The ratio of the instant system porosity to initial system porosity

$$\frac{p(t)}{p_o} \quad (2.1)$$

2. The ratio of interparticle bridge diameter to particle diameter

$$\frac{D_{bridge}}{D_{particle}} \quad (2.2)$$

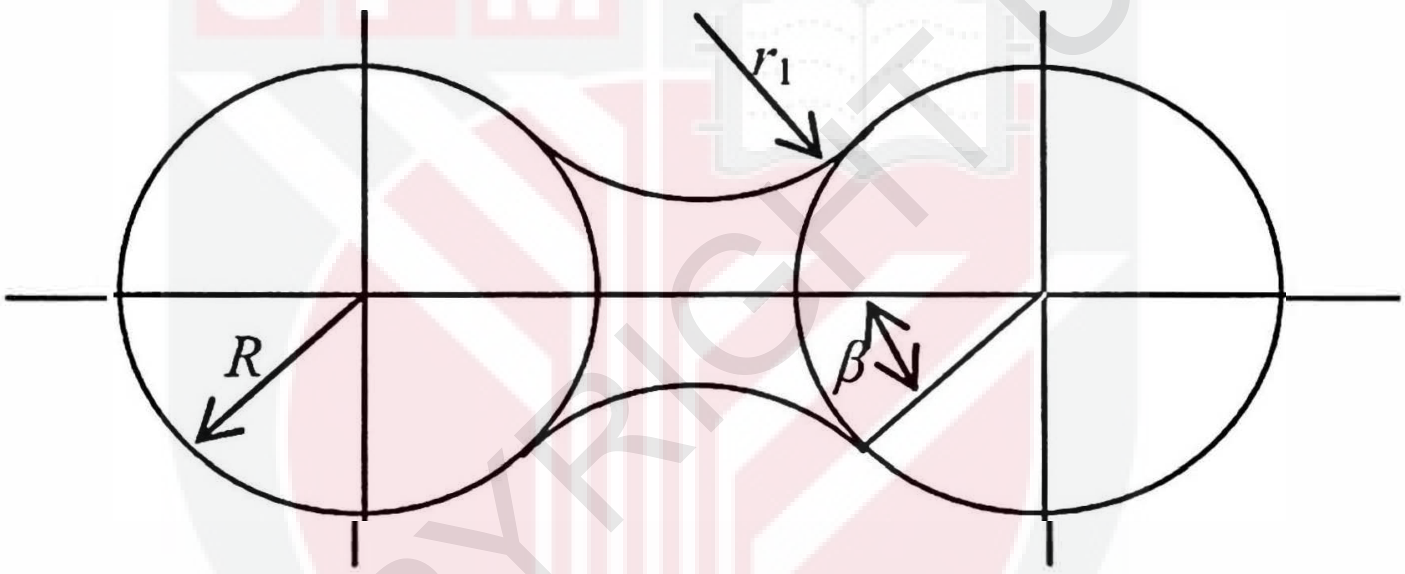


Figure 2-8: Illustration of a liquid bridge

From the stages explained, liquid is recognised to play a crucial role in the caking phenomenon and can be present between particles as a result of vapour condensation. Seville et al. (1997) have done a thorough analysis of capillary forces where it is an initial process that can contribute to the creation of subsequent cake. There are several experiments exploring the interaction between strength of agglomeration and interparticle forces.

The capillary power, illustrated in Figure 2-8 shows the liquid wets the solid particles. For particles usually smaller than 1 mm in diameter, the two components of the capillary force are the reduced hydrostatic pressure and the axial surface tension, and the resulting capillary force, Fc, is

$$F_C = \frac{2\pi R\gamma}{1 + \tan\left(\frac{\beta}{2}\right)} \tag{2.3}$$

where,

F_C = capillary force,

R = particle radius,

γ = interfacial surface tension between the liquid and air, and

β = bridge half-angle.

2.3.2 Flowability

In order to express the flowability of granular particles, different parameters such as angle of repose, Carr's index and Hausner ratio are used according to the analysis by Hadjittofis et al. (2017).

Flowability itself is demonstrated as the measure of flow rate, which is a ratio of mass to time as expressed below. (V. B. Khot*, 2017)

$$Flowability = \frac{Mass}{Time} \tag{2.4}$$

1. Angle of repose

The angle of repose is the simplest method to express flowability. The sides of the pile are at a definite quantifiable angle with the horizontally levelled surface as granular solids are stacked on a flat surface (Figure 2-9).

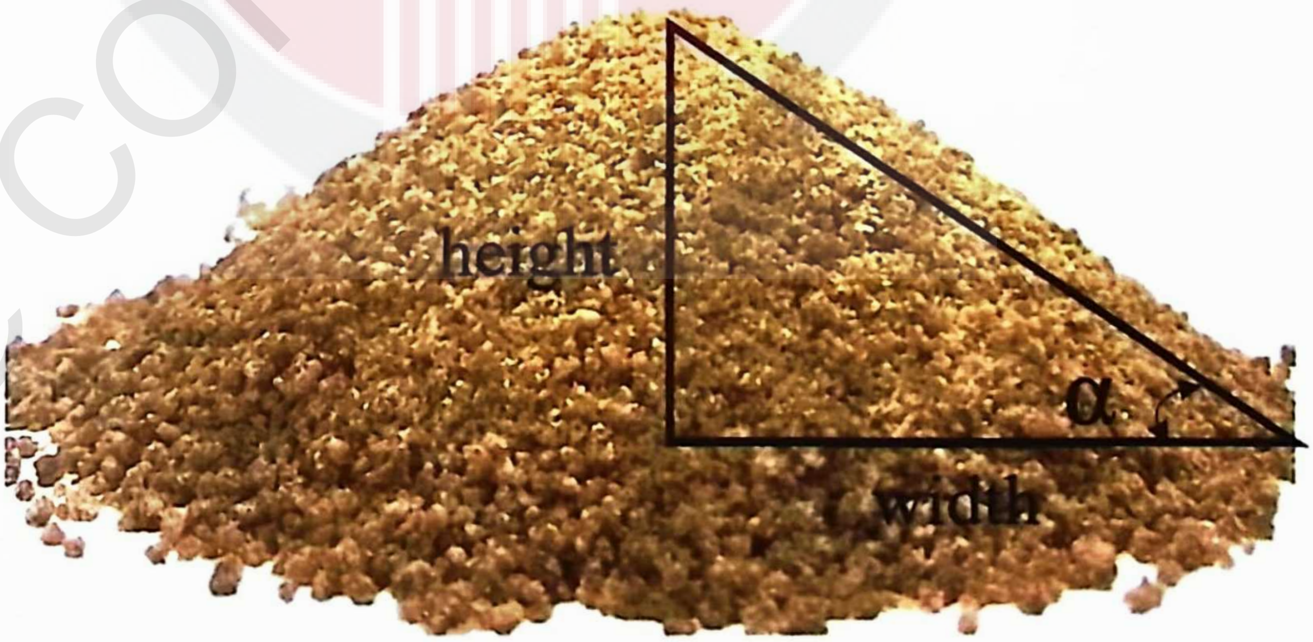


Figure 2-9: Illustration of angle of repose for a powder material

This angle is known as the angle of repose of the material. The angle is low when the grains are smooth and rounded, and high for very fine and sticky materials. Materials with low angle of repose are easily flowable and can be transported using gravitational force and requires a small amount of energy. The angle of repose is related to the free flowability properties of particulate materials in bulk forms (Teunou et al., 1999). Material that are considered freely flowable typically have an angle of repose below 30 degrees, whilst materials with an angle of repose more than 55 degrees are strongly cohesive, sticky, caking and non-flowable (Beakawi Al-Hashemi & Baghabra Al-Amoudi, 2018).

Interparticle interactions that involve adhesion, cohesion, frictional forces, mechanical forces due to interlocking influence the flow of powder particles. A number of other variables can also affect powder flowability. The lower the powers of adhesion and cohesion, the higher the flowability would be (Hadjittofis et al., 2017).

The angle of repose based on V. B. Khot*, 2017 can also be determined by an equation below.

$$\tan \alpha = \frac{2h}{d} \tag{2.5}$$

Where,

α = the angle of repose,

h = height of the cone, and

d= the diameter at the base

From the angle of repose degrees calculated as in Equation (2.5), the flow of sugar can be classified into its properties according to Table 2-1.

Table 2-1: Flow Property according to Angle of Repose degrees

Flow Property	Angle of Repose Degrees (°)
Excellent	25-30
Good	31-35
Fair	36-40
Passable	41-45
Poor	46-50
Very Poor	51-55
Extremely Poor	>66

2. Carr's Compressibility Index (CI)

Another representation of flowability established by Hadjittofis et al. (2017) is Carr's compressibility index which represents a numerical value expressed as percentage that depends on both apparent and tap densities of materials. The densities can be determined accurately by ASTM standards.

CI is a measure of powder bridge strength and stability based on compressibility index where lower value indicates better flow properties. A flow category based on its CI value is established by Shah et al., (2008) as shown in the table below.

Table 2-2: Flow category based on Carr's Index value by Shah et al. (2008)

Carr's Index scale (%)	Flow category
<10	Excellent
11-15	Good
16-20	Fair
21-25	Passable
26-31	Poor
32-37	Very poor
>38	Extremely poor

The value of Carr's index is determined by the following formula.

$$Carr\ Index\ (\%) = \frac{\rho_T - \rho_A}{\rho_T} \times 100 \quad (2.6)$$

Where

ρ_T = tap density, and

ρ_A= apparent density.

3. Hausner ratio (HR)

Hausner ratio is the other numerical value representation of measuring flowability. Hausner ratio is a measure of the interparticle friction (Shah et al., 2008). The value also depends on the two densities values which is tap density and apparent density which is measured according to the ASTM standard, but is calculated disparately. The formula established by Kaleem et al., (2020) is as shown.

$$Hausner\ ratio = \frac{\rho_A}{\rho_T} \quad (2.7)$$

Where,

ρ_T = tap density, and

ρ_A= apparent density.

The HR value scales to demonstrate the flow category is as shown in Table 2-3 by The flow Shah et al., (2008).

Table 2-3: Flow category based on Hausner ratio value

Hausner Ratio scale	Flow category
<1.11	Excellent
1.12-1.18	Good
1.19-1.25	Fair
1.26-1.34	Passable
1.35-1.45	Poor
1.46-1.59	Very poor
>1.60	Extremely poor

2.3.3 Moisture Migration

Rodgers & Lewis (1963), and many authors hence, have differentiated between three forms of moisture in refined sugar.

1. Free moisture

A dilute solution that forms a thin layer on the surface of the crystals when discharged from the centrifugal. This moisture is extracted reasonably easily and rapidly in dry conditions.

2. Bound moisture

Often known as migratable moisture, concentrated solution on the surface of the crystals, associated and trapped by amorphous sugar. This low permeability amorphous shell is produced during the initial drying of the sugar when the release of the moisture is too fast to enable the sucrose to crystallise on the surface of the crystal. This moisture is the major factor of caking, which must be removed by conditioning.

3. Inherent moisture

Moisture that is trapped within the crystals and removed only by dissolution or grinding. There seems to be no evidence of the migration of this moisture, and it is assumed that it does not play a role in caking.

Caking happens as sugar retains moisture when the relative humidity increases above the Equilibrium Relative Humidity (ERH) of sugar. This absorption reduces the partial pressure of the water at the boundary, and the moisture migrates from the warm core to these areas.

Moisture migration is one of the two dominant causes responsible for caking other than compressive caking (Christakis et al., 2006). It happens through a cycle of moisture adsorption or desorption between air and solid particles. The steps of moisture migration according to Christakis et al. (2006) is summarised as below.

1. Wetting and moisture absorption,
2. Liquid bridging,
3. Drying and moisture desorption,
4. Hardening and solid bridging, and
5. Compacting and caking.

The particles surfaces are hydrated or moistened by moisture as it absorbs moisture, and followed by formation of inter-particle liquid bridges by cohesion and adhesion. Along with recrystallization, solid bridges initiated when excess water evaporates during a drying period from the surface of the particles. The recrystallized solid bridges formed exhibit the requisite strength to hold the two particles in contact. These irreversible consolidation of bridges results in the agglomeration of particles. This would result in a moisture migration loop if the wetting and drying cycles are repeated. Interparticle space is limited and there is a thickening of interparticle bridges. Thus, caking strengths would depend on the rate of moisture migration and the solubility of the hygroscopic granular materials.

In the case of practically pure sugar, the accelerated method of drying introduces excess moisture trapped in the middle of the particle. In the case of crystalline materials, some of this moisture is part of the crystal structure which is known as inherent moisture. In time, the residual moisture migrates to the outer surface of the particle, forming a thin, syrupy outer coat of saturated sugar solution. This moisture is known as attached moisture and is responsible for the migration of moisture. (Leaper et al., 2002)

Usually, bound moisture accounts for less than 0.02% of the mass of a typical sugar molecule, while the water concentration in this layer is closer to 40%, measured by drying method loss. Until the crystals are mechanically destroyed, the inherent moisture content which is usually 0.025–0.04% by particle mass, does not take part in the moisture migration process and can only be obtained when the overall moisture content is calculated, usually by Karl Fischer examination. (Leaper et al., 2002)

Water adsorption isotherms indicates the relationship between moisture content and equilibrium relative humidity or water activity, and scanning electron microscopy (Chitprasert et al., 2006). It is established using the microclimate method. Equilibrium relative humidity (ERH) of gas-tight jars were fixed with saturated salt solutions (Greenspan, 1977). Sucrose was maintained at 20°C of temperature varies ERH until equilibrium was reached. At a given equilibrium relative humidity (ERH), water content was calculated and the sorption curve established as below (M. Mathlouthi & Rogé, 2003).

$$Water\ content = f(ERH) \tag{2.8}$$

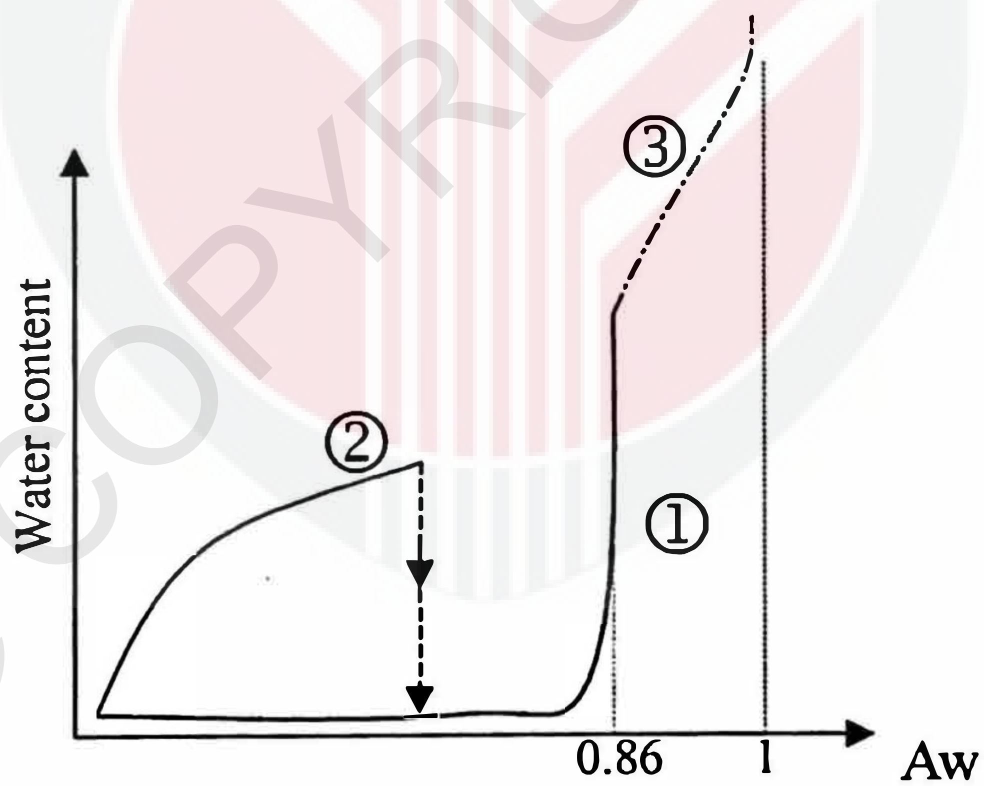


Figure 2-10: General shape of adsorption isotherms of (1) crystalline sucrose, (2) amorphous sucrose and (3) saturated solution. (M. Mathlouthi & Rogé, 2003)

2.4 Industrial Methods on Overcoming Caking Problems

A number of methods were suggested by authors as an approach to overcome caking problems in sugar industries. The behaviour of sugar during caking was investigated in order to propose a method of solution.

1. Decaking

Decaking, according to (M. Mathlouthi & Rogé, 2003), is an act of recovering a flowing sugar after caking happens. This method is done by flowing humid air of 60% ERH followed by a gradual drop to achieve 40% relative humidity in a silo with a design consisting of a very fine stainless mesh, a conical base which allows homogenization of air diffusing through the sugar, and an insulated wall to prevent heat loss.

In this case, it enables the re-crystallization of sucrose found in the bridges formed during caking. Following this regulated treatment of relative humidity, decaking is achieved and sucrose flowability is back to normal (Mathlouthi & Roge, 2001).

2. Pulverizing

Pulverised brown sugar will even out the particle size distribution and lowers the coefficient of variance (CV) value. It will also has a free-flowing properties as the moisture content decreased to 1% and the sticky molasses coating will get dry and solidify. (Lachmann & Haddonfield, 1994).

3. Molasses Refining

Food containing molasses has been faced with the challenge of drying out and allowing a free-flowing property. This method of prevention by Hollviken, Pehrson, & Löddeköpinge, (1998) has suggested that the molasses is refined using a membrane filtration to have an enhanced drying properties with a refractometric (RT) value of 8-45%. This separation method is to separate the low-molecular part from the high-molecular fraction since it contains higher sucrose purity. This will also result in lower hygroscopic property which induce the caking phenomenon.

4. Additives

The addition of additives act as an anti-caking agent and will causes sugar to behave with an improved storage quality such that with free flowing ability and non-caking property (Graham et al., 1994). Anti-caking agents are anhydrous substances that act by removing additional moisture or by wrapping particles to make them more water-repellent, avoiding the development of liquid bridges that initiate the formation of lumps (Frank, 2015).

5. Liquid Screening

A study was conducted by Chen et al., (2015) which focuses on the adhesion process during formation of crystal bridges that leads to caking. Rinsing liquids was used as a surface treatment to prove that high adhesive free energy prevents bonding of particles that leads to caking, contrary to water that in.

Mohamed Mathlouthi, (2014) performed an experiment measuring the degree of caking in corresponding to the deviation of ERH, temperature and particle size of sugar by image analysis. It is shown that the sample with smallest particle size of below

250µm has the worst flowability and it should be removed as in the case of caking, it acts as a base of the cohesion. A method of isopropanol washing was used as a rinsing liquid to remove the fine particles mentioned.

6. Environmental Control

A number of researches including Aguilera et al., (1995), Chen et al., (2017), Chitprasert et al., (2006), Rastikian & Capart, (1998), Zafar et al., (2017) has concluded that environmental conditions has greatly influenced the development of caking.

Sugar products within a supply chain faces many environmental shifts that contribute to thermal and moisture gradients within the bulk. As should be evident at this point, the tight control of moisture content and storage at low temperatures, when possible, are key factors in minimizing the risk of caking of sugar. (Aguilera et al., 1995)

7. Conditioning

Also known as curing, conditioning and curing is a method that the sugar must undergo for a certain period before it proceeds to packing or transporting in bulk (Excell, 1984). Conditioning is a process to remove excess moisture by subjecting the sugar to low humidity air conditioning silos for a set period of time. This method of prevention suggested by Excell, (1984) and Meadows, (1993) results in a drop in moisture content of sugar thus prevent wetting, forming of liquid bridges and caking phenomenon.

8. Cooling

A period of cooling sugar to ambient temperature was inserted between the hot dried process and packaging. This is according to Lachmann & Haddonfield, (1959) and Rastikian & Capart, (1998), to ensure the molasses coating of the sugar dried entirely without leaving any stickiness property.

9. Moisture-proof packaging

Lachmann & Haddonfield, (1959) suggested that packaging of sugar should be provided with moisture proof linings as to prevent moisture migration between the sugar particles and the atmosphere. Exposed sugar will lose its moisture under certain climatic conditions therefore resulting to hardening and caking.

10. Sieving

Chitprasert et al., (2006) suggested the removal of small sized sugar particles of below 425μm while Mohamed Mathlouthi, (2014) suggested removal of particles below 250μm. Both suggestions proposed the idea of removal fine sized particles as it plays the role as a base and binder in caking formations. Zafar et al., (2017) mentioned that these small particles have an adhesive force that promotes higher tendency to deform plastically.

11. Dehydration

Downton et al., (1989) recommends products should be in a dehydrated condition to prevent caking. This condition is achieved by further drying such that the current drying temperature and time were increased.

Table 2-4: Summary of caking preventions suggestions by previous research

Authors	Suggestion
M. Mathlouthi & Rogé, 2003; Mohamed Mathlouthi, (2014)	Decaking
Graham et al., (1994)	Pulverizing
Hollviken, Pehrson, & Löddeköpinge, (1998)	Molasses Refining
Graham et al., (1994)	Additives
Chen et al., 2015; Mohamed Mathlouthi, (2014)	Liquid Screening
Aguilera et al., 1995; Chen et al., (2017); Chitprasert et al., (2006); Rastikian & Capart, 1998; Zafar et al., (2017)	Environmental Control
Excell, 1984; Meadows, (1993)	Conditioning
Lachmann & Haddonfield, 1959; Rastikian & Capart, (1998)	Cooling
Lachmann & Haddonfield, (1959)	Moisture proof Packaging
Chitprasert et al., 2006; Zafar et al., (2017)	Sieving
Downton et al., (1989)	Dehydration

2.5 Factors Affecting Caking

Based on the preventions applied by previous research to prevent and overcome caking problems, similar repeating factors were discovered that plays a big role in contributing to caking.

2.5.1 Environmental Conditions

The method of decaking, conditioning and environment control was the most suggested preventions in overcoming caking problems. As needs to be obvious at this stage, environmental condition is one of the major factors affecting caking, this includes shifting from day to night and transporting from one place to another under different weather conditions. However, controlling the environment is out of reach as the product somehow needs to be packed in smaller packings and exposed to different conditions and weather.

2.5.2 Particle Size Distribution

A study by Rogé & Mathlouthi (2003) concluded that the particle size affects flowability. It is proven by comparing water vapor sorption isotherms of groups of standard sucrose crystals to water vapor adsorption of 100% of fine crystals, and to the behaviour of amorphous sugar (Roth, 1976). These findings support Werner's (1963) claim that fine sugar crystals with a particle size of less than 250µm are the most hygroscopic since it absorbs more water when the relative humidity rises.

Pulverizing of sugar, liquid screenings and sieving are methods proposed that points out the presence of fine sized particles and unevenly variant of particles sizes are the factors of caking. Based on an experiment conducted by Mohamed Mathlouthi, (2014), he observed the degree of caking in corresponding to the deviation of ERH, temperature and particle size of sugar by image analysis. The results were presented on a three-dimensional histogram, and it is observed that samples with smallest particle size of below 250µm has the worst flowability. This proves that crystals of fine sizes have a relatively larger and disordered structure which makes it more hygroscopic than the larger particles, and the aptitude to caking is greatly dependent on the heterogeneity of composition (CV) of sugar. These particles therefore play a big role as a cement of cohesion in the case of caking.

2.5.3 Molasses Hygroscopic Property

Hygroscopicity is a property of a substance that have the ability to absorb and retain moisture. Molasses, which is the coating of brown sugar that gives off the colour and taste, is considered a very hygroscopic matter with a sticky property in addition. That is therefore the reason of method of refining molasses and cooling product between drying and packing was suggested.

2.5.4 Bound Moisture

Bound moisture as explained in 2.3.3, is the layer of moisture that subjects to caking. As stated in 2.2, the allowable moisture content for soft brown sugar is known to be below 1%. A number of preventions was established aiming to remove this layer of moisture, in other words, excess moisture which is the addition of additives, conditioning, moisture proof lining for packaging and dehydration.

2.5.5 Storage Compaction

Investigation of M. Mathlouthi & Rogé (2003) on the caking of food powder has shown that compaction affects the flowability and the cohesiveness of sugar.

Compaction happens when bulk is tacked or placed under pressure which is commonly the case in industrial storage as a method to minimize space usage. Similar sizes of 50kg to 500kg bags were stacked, grouped on a pallet and wrapped to ease the transportation process.

The hygroscopic behaving particles will initiate caking by compaction where solid bridges were formed as a result of wetting of sugar, followed by drying (M. Mathlouthi & Rogé, 2003). Compaction can go to the extent of not being able to be reversed when the removal rate of water relative to the recrystallization rate is too quick, and decaking is possible but is damaging to the quality of sugar.

CHAPTER 3

METHODOLOGY

3.1 Experimental Design Overview

Figure 3-1 shows the experimental design flowchart overview. A total of six experiments will be conducted in order to study the factors contributing to cake formation in soft brown sugars. This is achieved by considering the properties of each sample and correlate it with the flowability and caking degree.

To begin, each sugar samples were measured the physical properties of moisture content, colour, and density, and moisture was measured right out from the packaging to prevent influence from external factors such as humidity conditions. Samples are compacted into tablets to measure the compaction strength using the Brazilian Test. Samples are then carried on with angle of repose experiments, left in open air storage to imitate a warehouse condition for six days, and then measured the angle of repose again. Flowability parameters of Hausner ratio and Carr's index can be computed from the density's values.

The investigated parameters were used to prove the factors affecting caking involving bound moisture content, molasses content for hygroscopic property and mean particle size and type of storage conditions that affects caking.

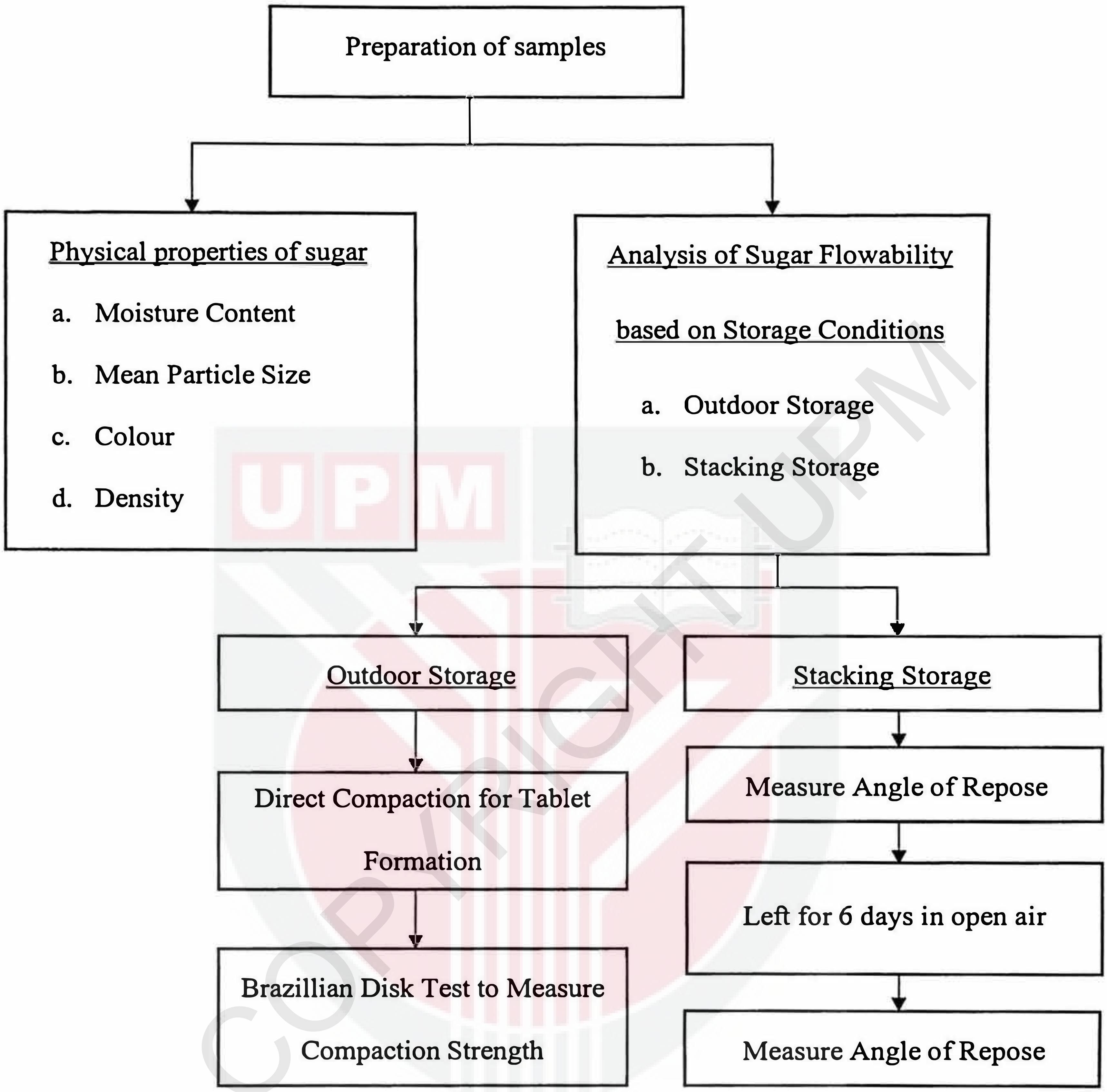


Figure 3-1: Experimental design flowchart

The equipment used are moisture analyser, tapped density analyser, analytical balance, universal testing machine, temperature and humidity sensor and image processing software ImageJ. While the materials needed are 250ml conical flask, 50ml measuring cylinder, filter funnel, clear plastic cups, cereal box, digital camera, cardboard base, and blue screen background.

3.2 Sample Preparations

This experiment uses seven different samples of soft brown sugar available commercially from five different manufacturers. The samples are listed as below:

Table 3-1: Table of samples

Sample Name	Name	Company	Country Of Origin	Packaging
CSR 1	Soft Brown Sugar Non-caking	Central Sugar Refinery Sdn. Bhd. (CSR)	Malaysia	
CSR 2	Caking soft brown sugar	Central Sugar Refinery Sdn. Bhd. (CSR)	Malaysia	
CSR 3	Soft Brown Sugar Non-caking with large particle size	Central Sugar Refinery Sdn. Bhd. (CSR)	Malaysia	
MSM	Soft Brown Sugar	MSM Prai Berhad	Malaysia	
DD	Soft Brown Sugar	Dapur Desa	Malaysia	
RWF	Muscovado Sugar	Radiant Whole Food	Philippines	
CFO	Molasses Sugar	Country Farm Organics	Malaysia	

Approximately 300-500g of sugar was prepared for every sample, labelled with the sample number and kept in a zip lock bag to prevent moisture migration.

The molasses content for all three CSR samples uses a mixture of different tank of molasses, named D2 and S3 or S1. The composition for D2 molasses remained at 40% while S1 or S3 will be added accordingly to make the soft brown sugar colour darker. No data of molasses content was obtainable for other samples.

3.3 Physical Properties

3.3.1 Moisture Content

In reference to the South African Sugar Technologists' Association (SASTA) Lab Manual, *Method 8 . 2 – Refined Sugar : Moisture by Oven Drying*, (2005), this method is used to estimate the free moisture content in a sugar particle by loss on drying. Dish was preheated at (105 ± 1°C) for 30 minutes and allowed to cool for 1 hour before adding 10 ± 0.0001g of each sugar sample on separate dishes. All dishes are covered and heated in the oven operating at atmospheric pressure and (105 ± 1°C) for 3 hours ± 5 minutes. Dish were cooled down to room temperature before weighing.

The moisture of sugar samples was calculated based on the before and after weight such that substituting into this equation.

$$Moisture\ of\ sugar\ (\%) = \frac{(M_2 - M_3)}{(M_2 - M_1)} \times 100 \qquad (3.1)$$

In this research, a digital moisture analyzer AND MX-50 with an accuracy of 0.01% and 0.001g which uses Loss on Drying (LOD) method to determine moisture content was used using the same parameters from the SASTA Lab Manual heating 20g of sample under a uniform halogen lamp at 105°C. This method express moisture in wet basis (wb).

3.3.2 Mean Particle Size

Upon measuring the mean particle size samples, according to Kumara et al., (2012) the method of image analysis was used using a public domain image processing software, ImageJ (Image Processing and Analysis in Java, National Institute of Health and the Laboratory for Optical and Computational Instrumentation (LOCI, University of Wisconsin, United States).

Images of the sugar particles were taken by a digital camera at constant distance and optical zoom by sticking the surface of the samples on a tape, so that the particles do not stack in the photo. A blue-coloured background was used, in referring to the colour complementary wheel to maximize the contrast between the brown sugar particles. A photo of scaled ruler at same distance and optical zoom was also taken to be used as calibration, and calibration was denoted as 69.6046 pixels/mm.

Images were adjusted by applying threshold to differentiate particles to be in red and background is in white. The red area was measured limited to threshold and recorded, and number of particles for the measured area was counted using the 'multi-point' cursor.

The mean of particles was calculated by using the formula:

$$\text{Mean particle size} = \frac{\text{Area}}{\text{number of particles}} \qquad (3.2)$$

3.3.3 Colour Absorbance

The colour of sugar directly relates to the degree of refining and can be used to estimate the molasses content.

The International Commission for Uniform Methods of Sugar Analysis (ICUMSA) established Method GS1/3-7 to be used to measure raw sugars, brown sugars and coloured syrups colour using spectrophotometric colour determination at 420nm.

Using Beer's Law, an absorption spectroscopy is utilized to transmit coloured building paper light through a sample and detected on a smartphone (iPhone 11, Apple Inc., California, United States) using an RGB application. Two different RGB application was installed to get the average value, Colorimeter RGB (Dmitry Svishchov, Smyk Sergii, and Color_Meter (Dogan Agcay, Palo Alto, California).

The Beer-Lambert Law express absorbance in terms of intensities as:

$$A = log_{10}\left(\frac{I_o}{I}\right) = \varepsilon l C \tag{3.31}$$

Where,

A = absorbance,

I_o= intensity of blank,

I = intensity of sample,

ε = molar absorptivity or molar extinction coefficient,

l = light path, and

C = concentration.

Soft brown sugars, having a colour value of that lies in between 2000-7000 ICUMSA UNIT (IU), were expected to follow the specifications below:

Table 3-2: ICUMSA sample dilution ratio according to colour range

ICUMSA Colour Range (IU 7.0)	Cell length (mm)	Sample (g)	Water (g)
2000-7000	10	10 ± 1	90 ± 1

Based on the above table, the dilution ratio of sample to water is 10g to 90g, were mixed in a 250ml clear plastic cup and dissolved by swirling at room temperature, and a plain filtered water was prepared in the same cup as blank. Using the same blue background to enhance the contrast between the sugar solution, the application's reading on blue colour was recorded.

3.3.4 Density

Bulk Density

Bulk density is a ratio of the sample mass and its volume, including the interparticular void volume. It is measured by using a graduated cylinder method, where a suitable amount of sample is poured carefully in a 50ml tarred graduated measuring cylinder (Fini et al., 2008, Yaakub et al., 2018). The volume was then read straight from the cylinder and and substituted in the equation to measure the bulk density:

$$Bulk\ Density = \frac{Mass\ of\ sample\ (g)}{Occupied\ volume\ (ml)} \qquad (3.42)$$

Tapped Density

The tapped density is a measure of bulk density after tapping the container containing samples. It is measured using the tapped density analyser (GeoPyc 1360 Micromeritics, U.S.A) where an amount of 2 ± 0.001g was inserted into the glass tube and covered using a piston tip

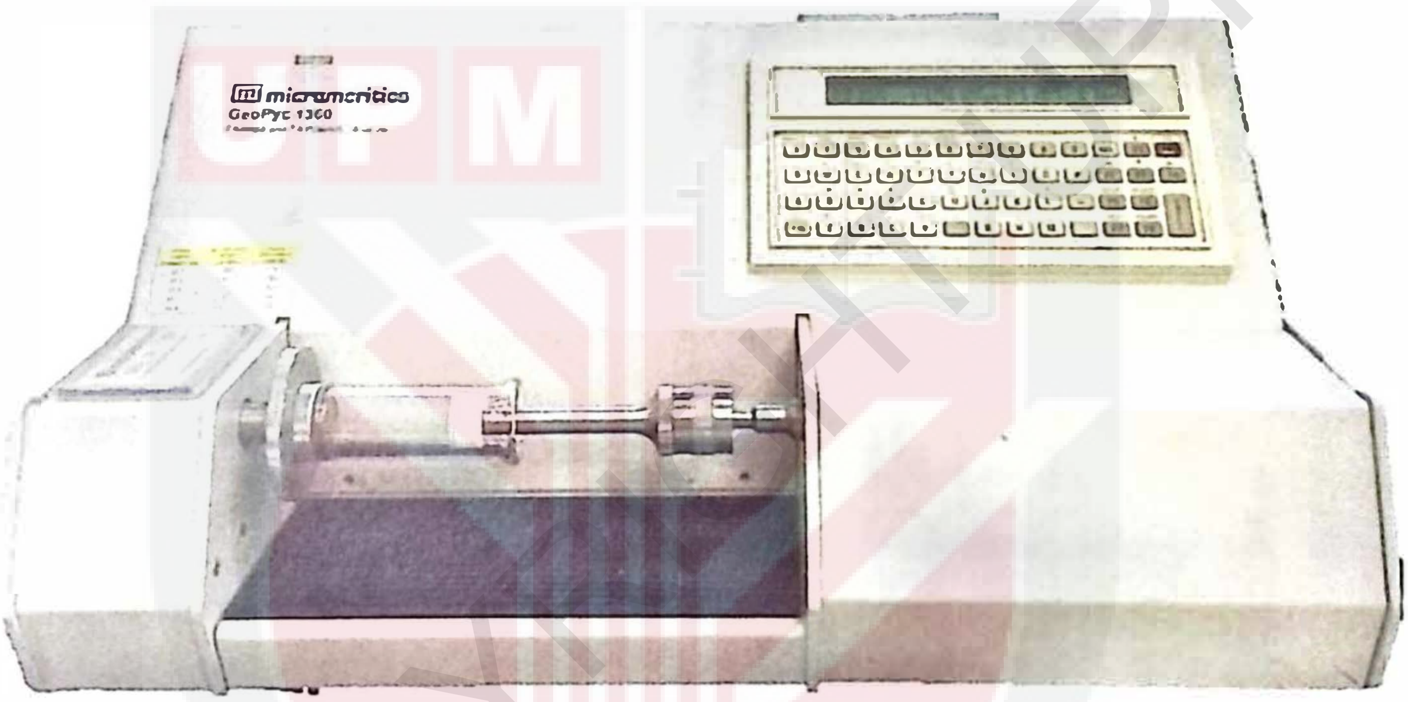


Figure 3-2: Tapped density analyser GeoPyc 1360 Micromeritics, U.S.A

3.4 Flowability Properties

3.4.1 Powder Flowability Indicator

Hausner Ratio

From the results of bulk and tapped densities, the Hausner ratio is the ratio of tapped density to bulk density and it was calculated such that it obeys the equation:

$$Hausner\ Ratio = \frac{Tapped\ Density}{Bulk\ Density} \qquad (3.53)$$

Carr's Index

Similarly, the Carr's index was calculated based on the densities measured earlier by substituting into the equation:

$$Carr's\ Index = \frac{Tapped\ Density - Bulk\ Density}{Tapped\ Density} \times 100 \qquad (3.6)$$

3.4.2 Angle of Repose

A method of measuring the angle of repose of sugar using the fixed funnel method according to Beakawi Al-Hashemi & Baghabra Al-Amoudi, (2018).

The sugar samples were poured in a funnel on a flat horizontal base, slowly raising from the base to a fixed height of 8cm. A heap was formed, and the width and height were recorded and the angle of repose was determined by the equation:

$$Angle\ of\ Repose, \alpha = \tan^{-1}\left(\frac{2 \times height}{width}\right) \qquad (3.7)$$

3.5 Pilot Storage

Two storage condition was considered which is open-air outdoor storage and bags stacking storage. Both conditions act as a factor affecting flowability of sugar.

3.5.1 Flowability Analysis for Outdoor Storage

To imitate the industrial warehouse condition for storage of packed sugar assuming the packaging is not well lined with moisture-proof lining, a bag of sample unzipped was left in the open air with a controlled humidity and temperature by a humidity and temperature sensor (Mi Temperature and Humidity Monitor 2, Xiaomi, China) for six days. The chosen timeframe was concluded to be the best representation of industrial storage period. The angle of repose of samples were measured before and after the six days period.

3.5.2 Compaction Strength in Stacking Storage

Using the universal testing machine (model 3382, Instron MA, U.S.A.), the compaction strength was measured by compacting 2 ± 0.01g of samples into a tablet, poured into the 13mm diameter punch die set with a compaction force of 0.5kN to imitate the packed products in 50kg bags. The load speed of 0.1mm/s while the load is being loaded is 0.167mm/s (Anuar, 2009).

Tablet dimensions, thickness or height and diameter, were measured immediately after ejection. The results were taken on average in three places. The tablet was then placed in a tiny container and kept over silica gel at room temperature in a desiccator. After the compression, the next tablet testing, the Brazilian disk was carried out.

The work involved in the compression process is represented by the area under a force-displacement curve. The force-displacement method has been utilized in foundational research on the energy changes that occur during powder compaction. The following equation demonstrates that the area under the force-displacement curve, which is the integral of force and displacement equals to work:

$$W = \int F \cdot dx \tag{3.8}$$

Where,

W= work done,

F= force, and

x= corresponding displacement.

The Brazilian disk test was performed to determine the tensile strength of tablets produced in this study (Jamar, Anuar, & Tahir, 2019). This evaluation comprises analyzing the fracture resistance of the tablet and determining the force required to fracture or break the specimen along its diameter by applying a load on the tablet. The centre cylindrical axis of a tablet is crushed between two parallel platens. This test was also carried out using a universal testing machine (Model 5566, Instron Canton MA, USA) at a speed of 0.0116mm/s (Yaakub et al., 2018). The tensile strength of the tablet is calculated as:

$$\sigma_t = \frac{2P}{\pi D h} \tag{3.9}$$

Where,

σ_t = tensile strength,

P = breaking force,

D = tablet diameter, and

h = tablet thickness.

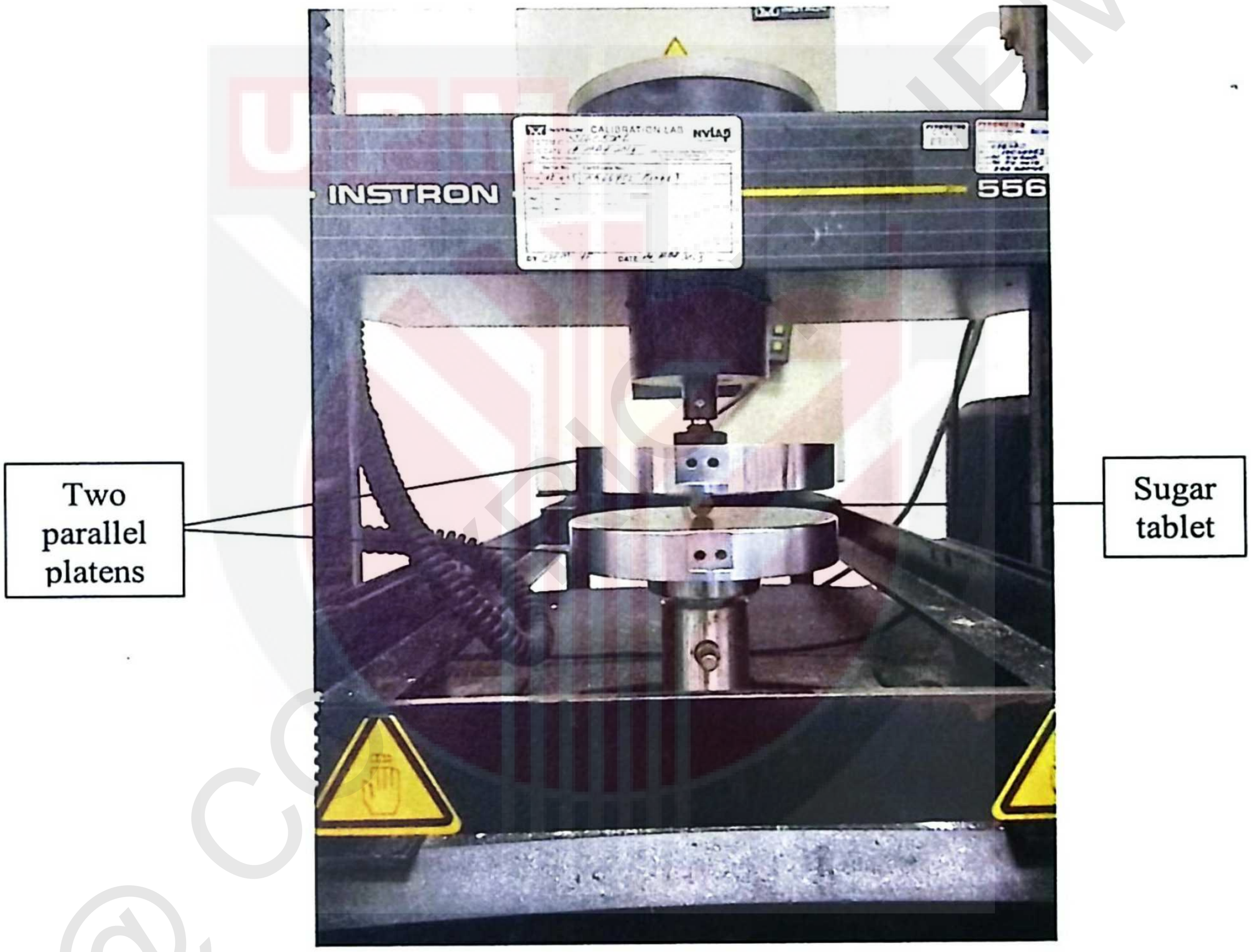


Figure 3-3: Indirect Tensile Strength Test: Brazilian Test using the universal testing machine (Model 5566, Instron Canton MA, USA)

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Physical Properties

Commercial soft brown sugar from different brands with different physical characteristic were tested in this research. Physical properties are an important factor in determining caking factors of sugar. In this research, moisture content, mean particle size, colour absorbance and density of soft brown sugar are investigated.

4.1.1 Moisture Content

The importance of maintaining a controlled amount of moisture in sugars can indeed be emphasized in terms of bacterial growth, inversion, fermentation, and sugar hardening during storage and was recognized that the addition of invert sugar in a sucrose product increased its ability to absorb moisture (Dittmar, 1935). The invert content of molasses is high as sucrose is digested by invertase into glucose and fructose (Yang & Montgomery, 2007). Since the molasses in brown sugar imparts the colour and taste of the sugar, it is safe to assume that the molasses content can be determined by the colour absorbance.

The chart in Figure 4-1 below shows that the moisture content and colour absorbance value is directly proportional since the increase of moisture shows an increase in colour absorbance. This confirms that the higher composition of molasses used to produce the soft brown sugar, making the sugar to have a higher colour absorbance, increases the moisture content of the sugar due to the hygroscopic properties of molasses that is discussed in 2.5.3, having the ability to absorb and retain moisture. The colour absorbance value is in Absorbance Unit (AU) and moisture content is expressed in percentage (%). The sample with the lowest value of colour and moisture is MSM sample, with the value 0 AU and 0.05% respectively and sample with the highest value is RWF sample, with colour and moisture of 0.71 AU and 1.93%.

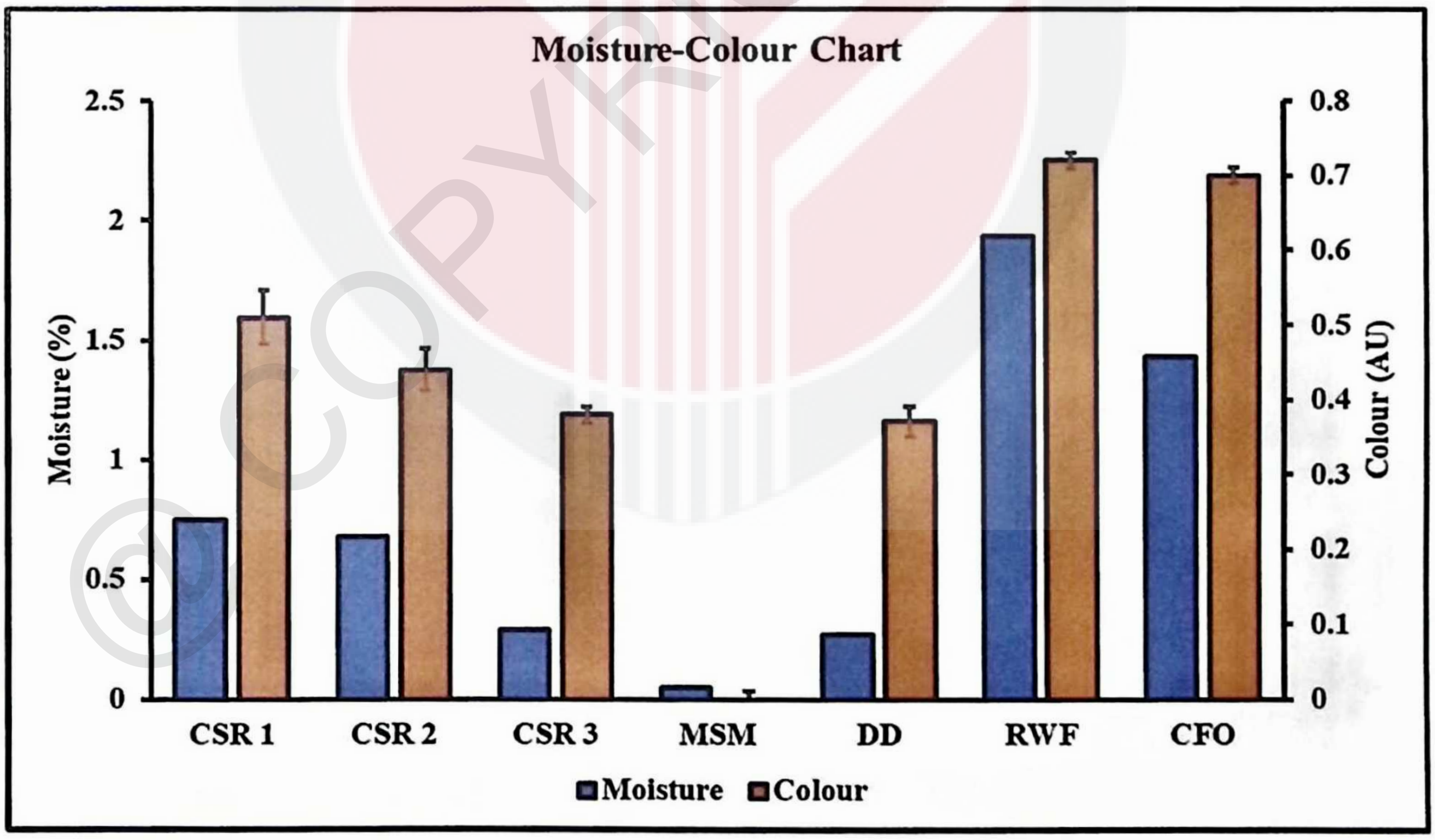
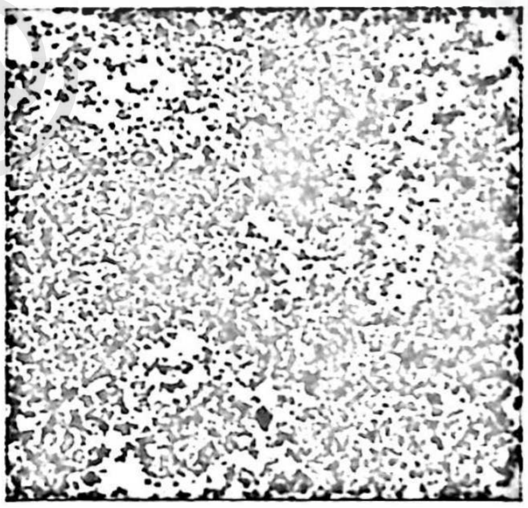


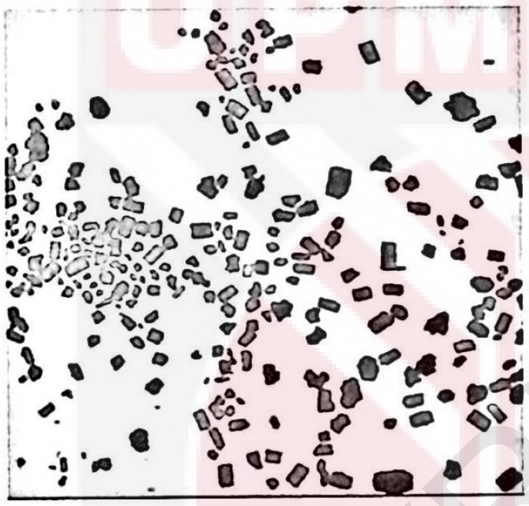
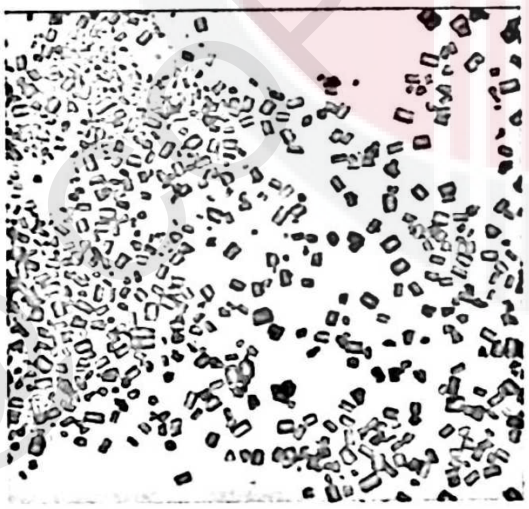
Figure 4-1: Bar chart representing the relation between moisture content and colour absorbance of samples

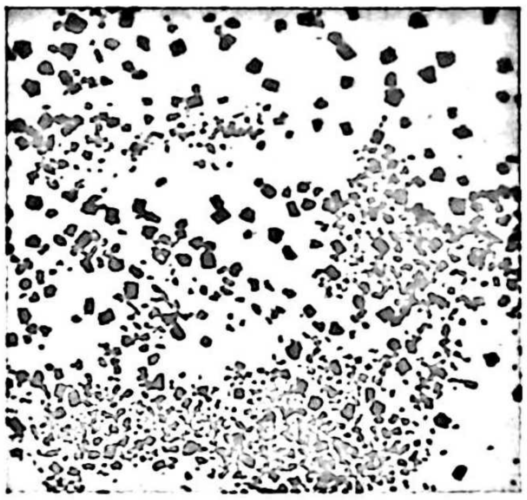
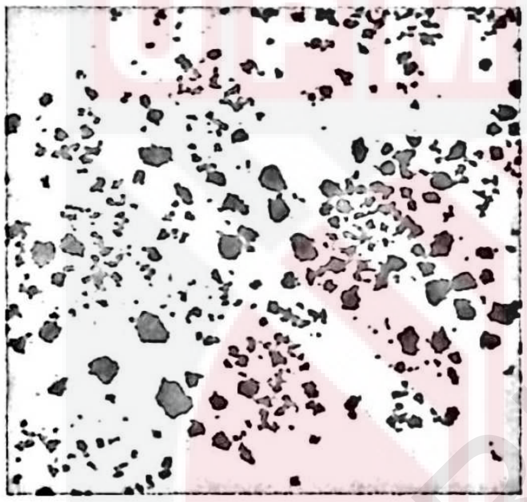
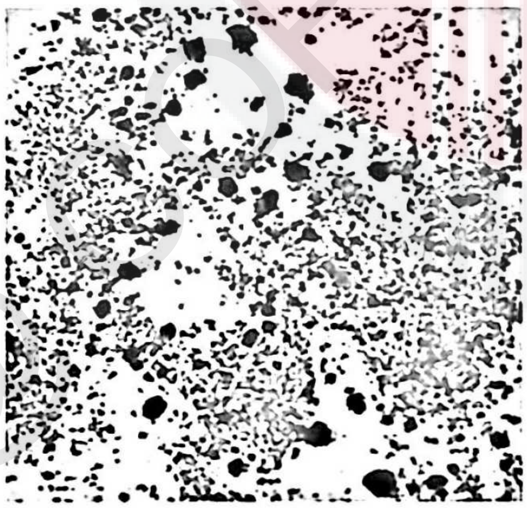
4.1.2 Mean Particle Size

The allowable particle size for soft brown sugar according to the quality assurance standard for industrial sugar is between 75µm to 750µm. According to Werner (1963), fine sugar crystals with a particle size of less than 250µm are the most hygroscopic because they absorb more water as relative humidity rises and function as the platform that triggers sugar caking and affects the flowability of sugar. Table 4-1 below shows the images of samples and the mean particle size of each sample. Sample CSR 1 and CSR 2 has a mean particle size that lies within the acceptable standard with approximately narrower particle size distribution, sample DD, RWF and CFO is quantitatively within the range but has a wider particle size distribution. Sample CSR 3 and MSM is out of range by 1347.04 µm and 845.92 µm and has a definite crystalline particle shape.

Table 4-1: Particle size of samples using image analysis

Sample Image	**Mean Particle Size**
	CSR 1 Area of particles: 11.223 mm Number of Particles: 81 Mean Particle Size: 138.56 µm

	CSR 2 Area of particles: 20.489 mm Number of Particles: 62 Mean Particle Size: 330.47 µm
	CSR 3 Area of particles: 66.005 mm Number of Particles: 49 Mean Particle Size: 1347.04 µm
	MSM Area of particles: 32.991 mm Number of Particles: 39 Mean Particle Size: 845.92 µm

	DD Area of particles: 7.823 mm Number of Particles: 17 Mean Particle Size: 460.18 μm
	RWF Area of particles: 27.387 mm Number of Particles: 40 Mean Particle Size: 684.68 μm
	CFO Area of particles: 14.442 mm Number of Particles: 43 Mean Particle Size: 335.86 μm

The graph in Figure 4-2 below shows the relationship between the mean particle size and the moisture content of sugar. The mean particle size value is in µm, and moisture content is in percentage unit (%). It can be seen that bigger mean particle size, Sample CSR 3 and MSM, that goes beyond the allowable range the lowest moisture content and the samples with small sizes that lies within the range has higher moisture, especially RWF and CFO which seems to have a wide particle size distribution.

This relation validates the Werner's (1963) findings that claims fine sugar crystals are the most hygroscopic since it absorbs more water when the relative humidity rises.

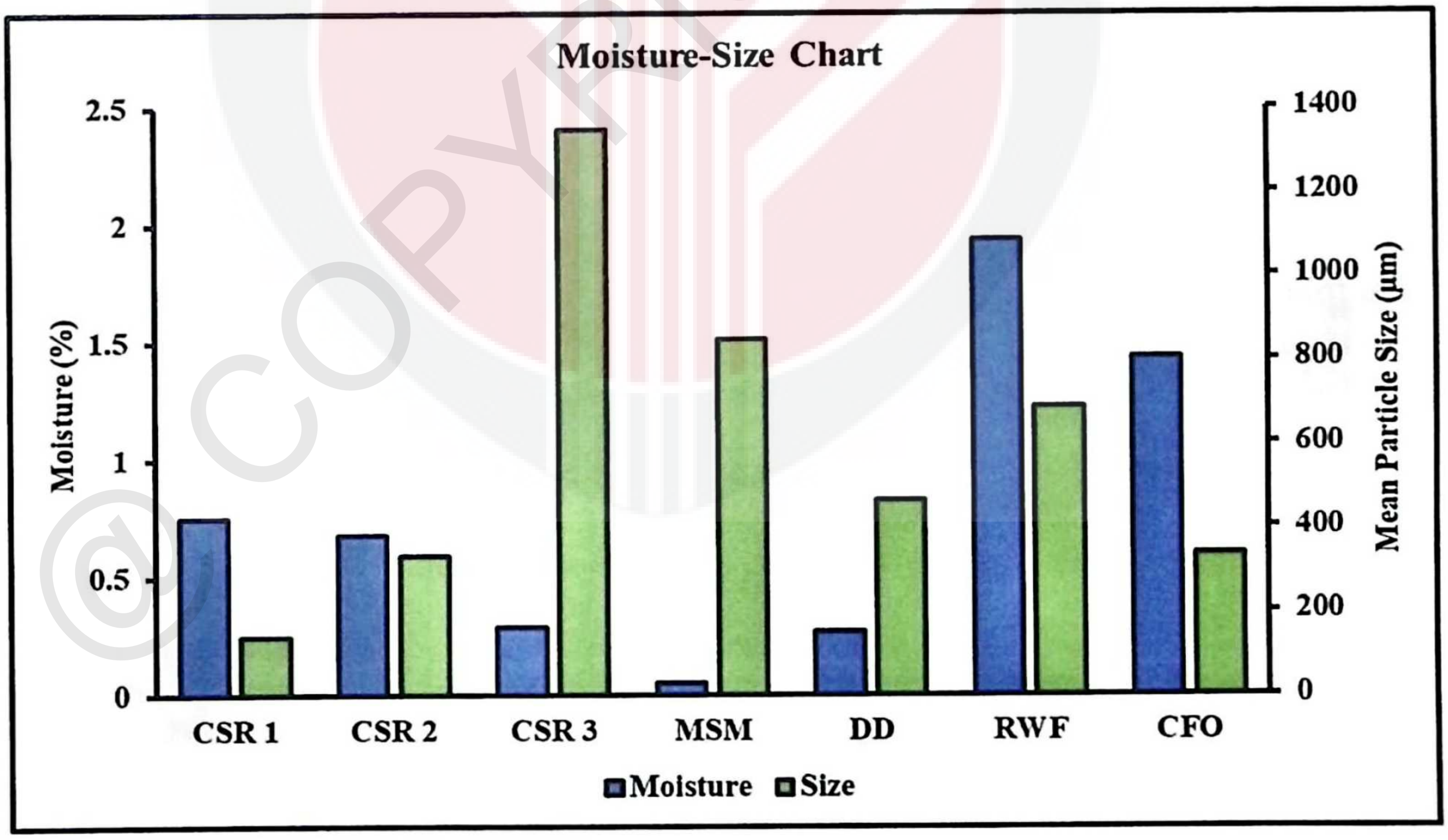


Figure 4-2: Relationship between mean particle size and moisture content of samples

4.2 Compaction Behaviour

4.2.1 Tablet Properties

For this analysis, the sugar samples were compacted into tablets at compaction pressure of 0.5kN to simulate the stacking positions of 50kg bags in storage warehouse. 2 ± 0.001g of each sample was used producing similar 6 mm thickness of tablets and 13mm in diameter since the punch die set used was of 13mm DIE (PT. No. 3000, Spepac, Britain). The compaction behaviour and fracture behaviour were studied using the force-displacement curve generated.

4.2.2 Force Displacement Profile

Figure 4-3 illustrates the force-displacement curve which is used to measure the characteristics of sugar samples since it depicts the extrinsic properties of the material under test. The properties of sugar obtained from the curve are the breaking load, tensile strength and fracture energy.

During the test, the sugar tablet was observed until it reached its peak point in the graph generated such in Figure 4-3, which corresponds when the sugar tablet experience breakage and the test was manually stopped. The peak point from each curve representing the samples is denoted as the breaking load, which is the stress or tension steadily applied and just sufficient to break or rupture the material with CSR 3 sample, having the highest value of 3.25N followed by DD sample, CSR 2 and CSR 1 with values 2.45N, 2.11N, and 1.95N. RWF sample and CFO sample having similar breaking load value of 1.43N and 1.39N and MSM sample has the least force required to rupture of 1.24N. From the data, it is concluded that MSM sample is high in friability and this condition is desired since sugar tablets that needs higher compression force to break might already deform and the compression is irreversible, and breaking the compressed sugar cake might result in quality deterioration.

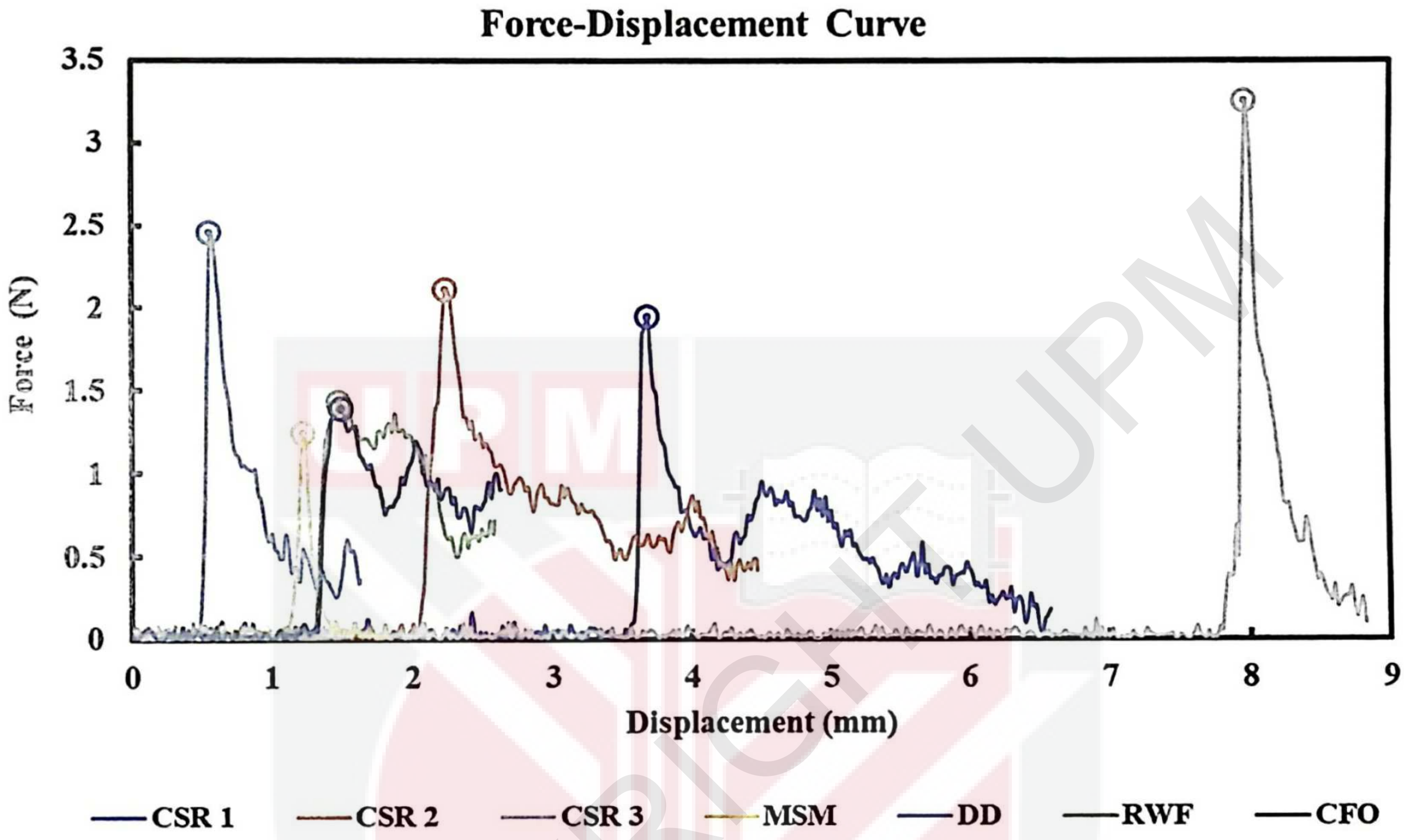


Figure 4-3: Force-Displacement Profile of sugar tablets.

4.2.3 Tensile Strength

The breaking load values was used to calculate tensile strength based on Equation (3.9)(3.42), and the results is as shown in Figure 4-4. The tensile strength, which is the ability of a material to resist tearing of the samples ranges from 10.11kPa to 26.56kPa. CSR sample 3, with the highest breaking load, have the highest tensile strength of 26.56kPa and can hold the most stress.

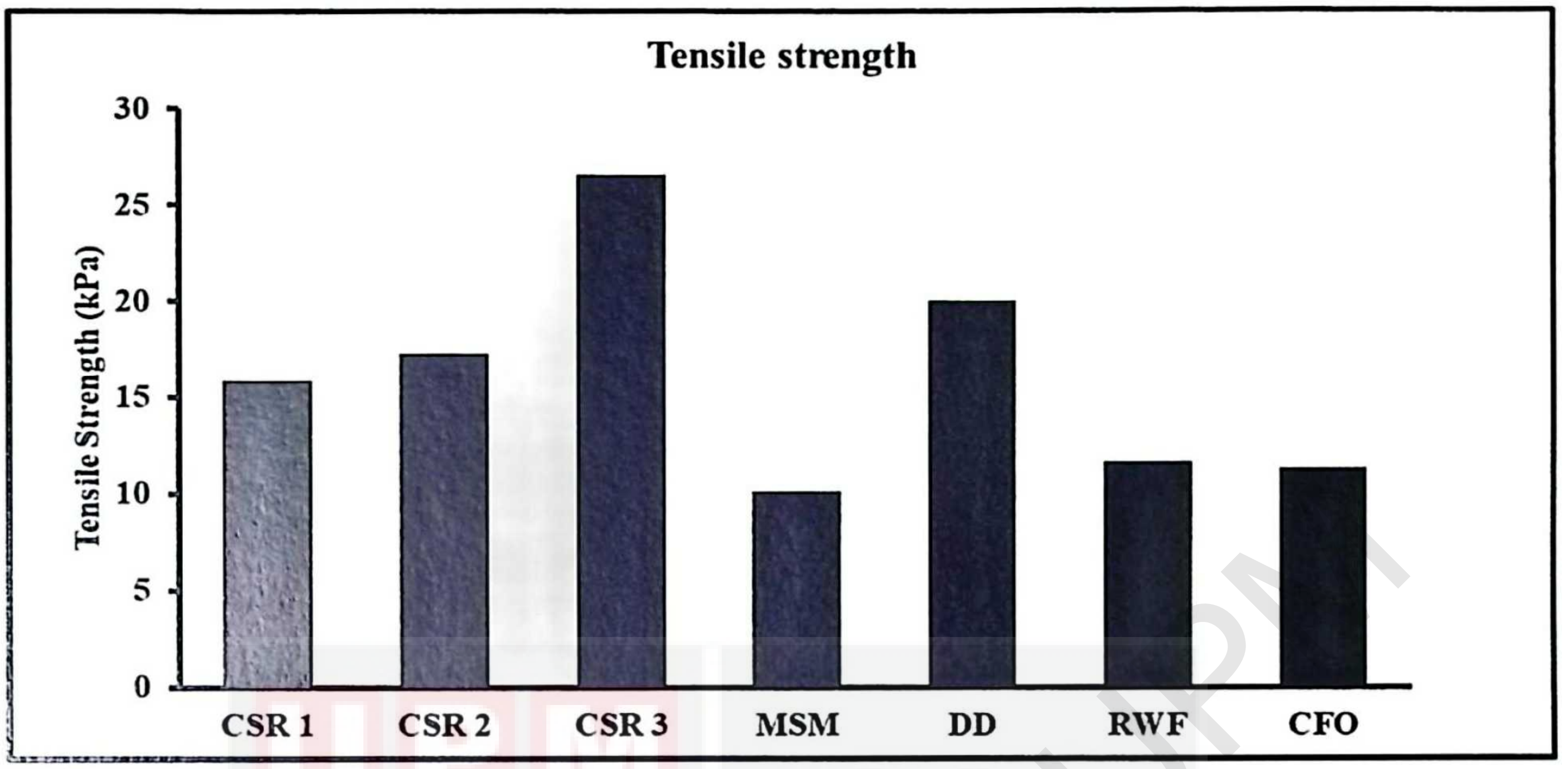


Figure 4-4: Tensile strength of sugar tablets

The moisture content curve was added to the chart in Figure 4-5 shows that CSR 3 sample, MSM sample and DD sample tensile strength values were in tune with its moisture level. MSM sample behaves the best as it has a low moisture content level and a low tensile strength, consistently.

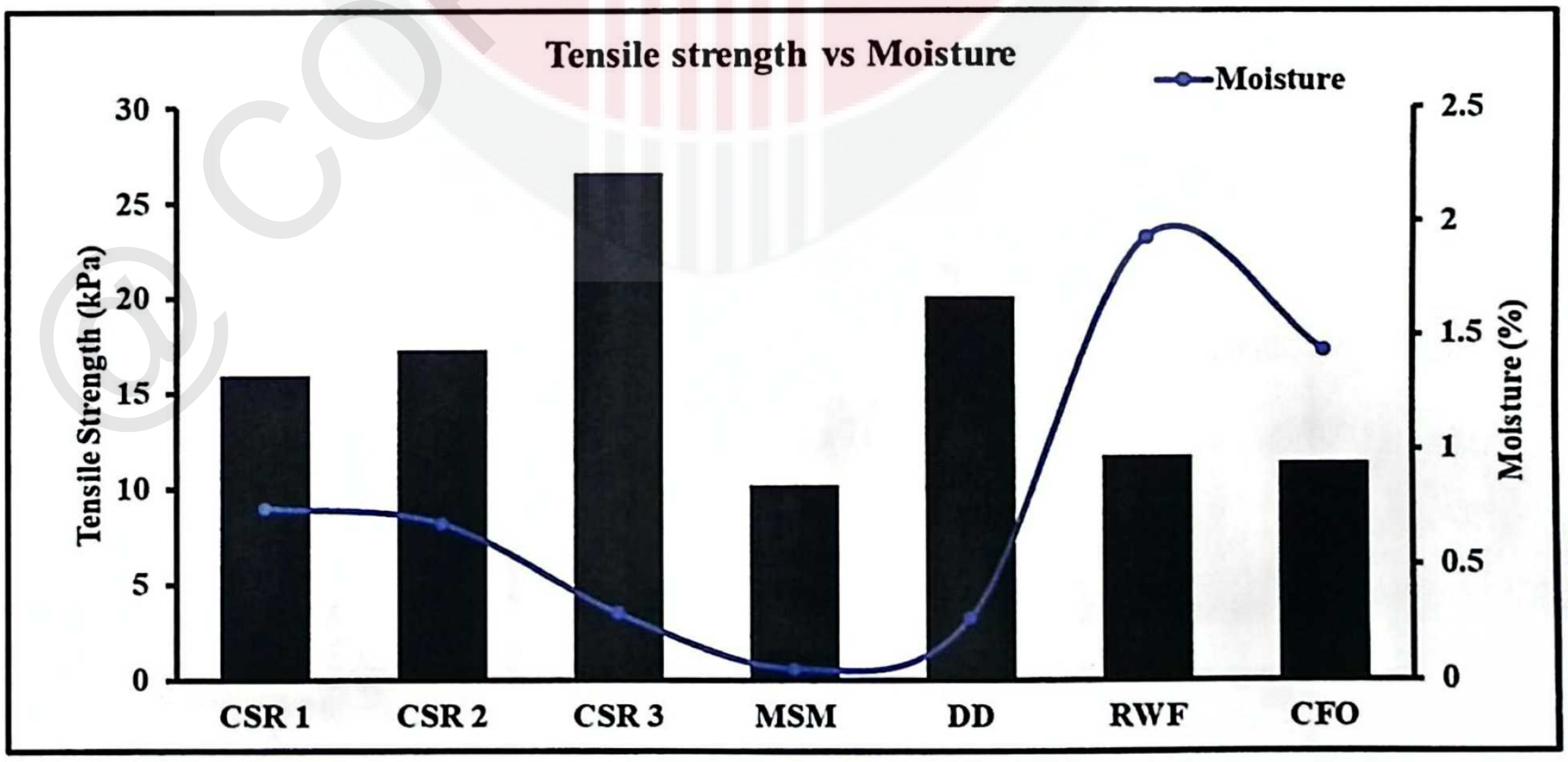


Figure 4-5: Tensile strength of Sugar Tablets based on breaking load with comparison to the moisture content

The tensile strength was put in comparison to the mean particle size in Figure 4-6 and it reveals that the sugar tablet sample, CSR 3, with the highest tensile strength has the largest mean particle size while others with lower strength have smaller particle sizes.

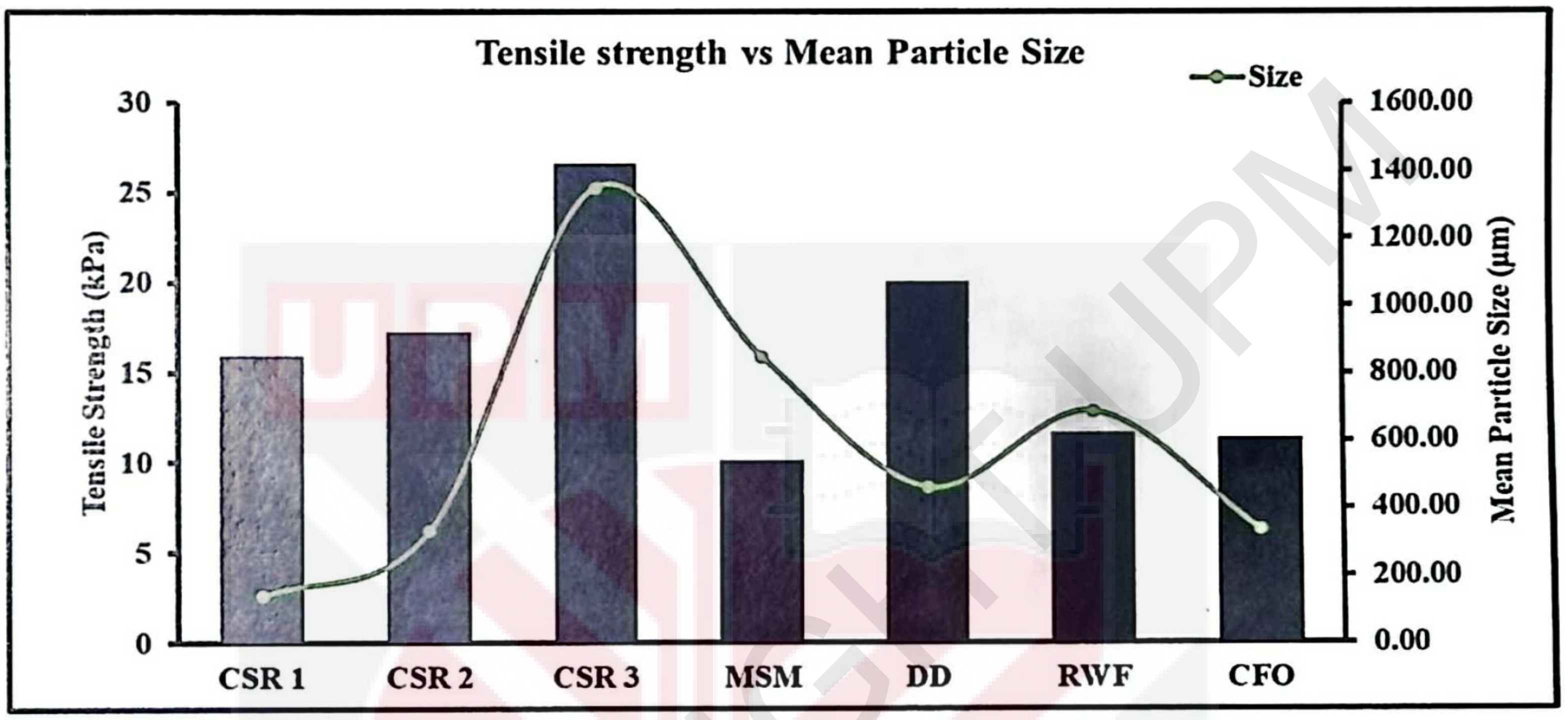


Figure 4-6: Tensile strength of sugar tablets based on breaking load with comparison to the mean particle size

4.2.4 Fracture Energy

Fracture energy was then calculated from the whole area under the force-displacement curve in Figure 4-3, up until the peak point of breaking load. The error causing fluctuations from the background is not included in the calculation of area until the parallel platens acting as load touches the sample. Results of the fracture energy of each sugar tablets are as shown in Figure 4-7.

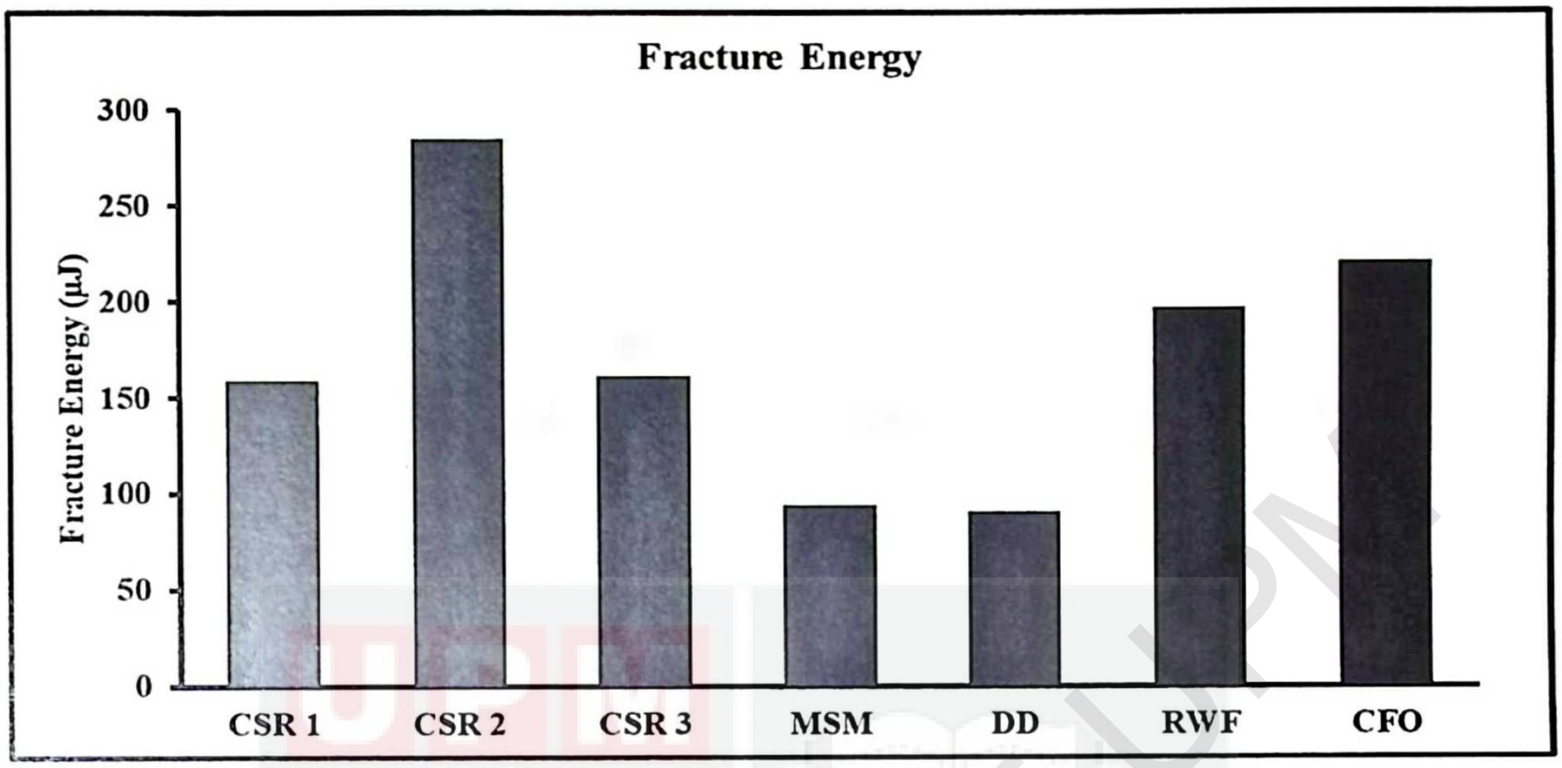


Figure 4-7: Fracture energy of sugar tablets calculated by the area under the graph based on force-displacement profile

Moisture content effect on fracture energy is such that higher moisture will involve higher fracture energy due to the stickiness property of the sugar that the particles uphold, as displayed in Figure 4-8.

The relationship between fracture energy values and the particle sizes is compared in Figure 4-9 and it can be observed that the three samples with lowest particle size values have the largest three fracture energy, and the larger the particle size with lower fracture energy such in sample CSR 3, MSM, DD, and RWF. This shows that smaller sized particles are easier to be compacted and affects its flowability as shown by the Figure. This shows that sugar with smaller particle size requires more energy to break the cake compact.

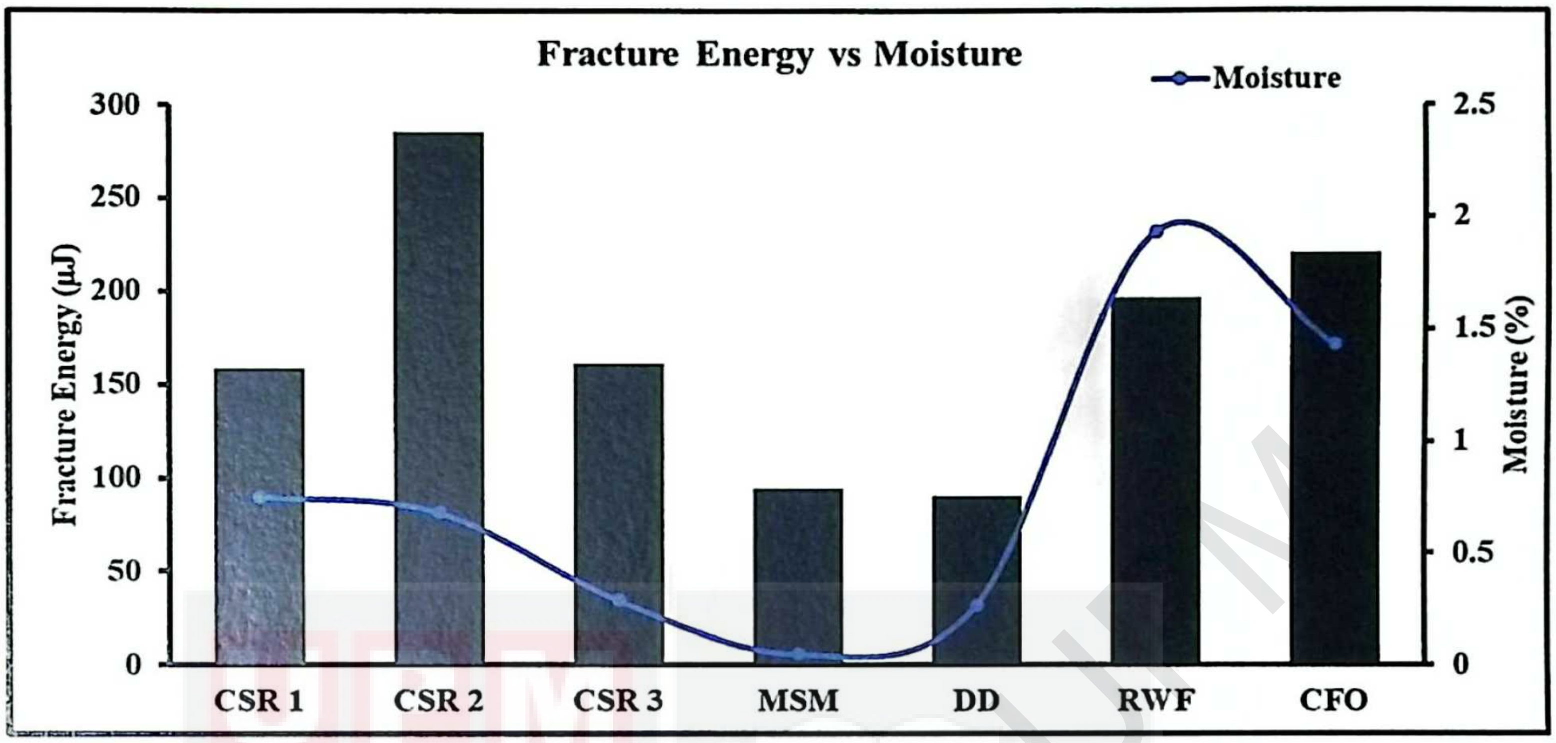


Figure 4-8: Relation between fracture energy and moisture content of sugar

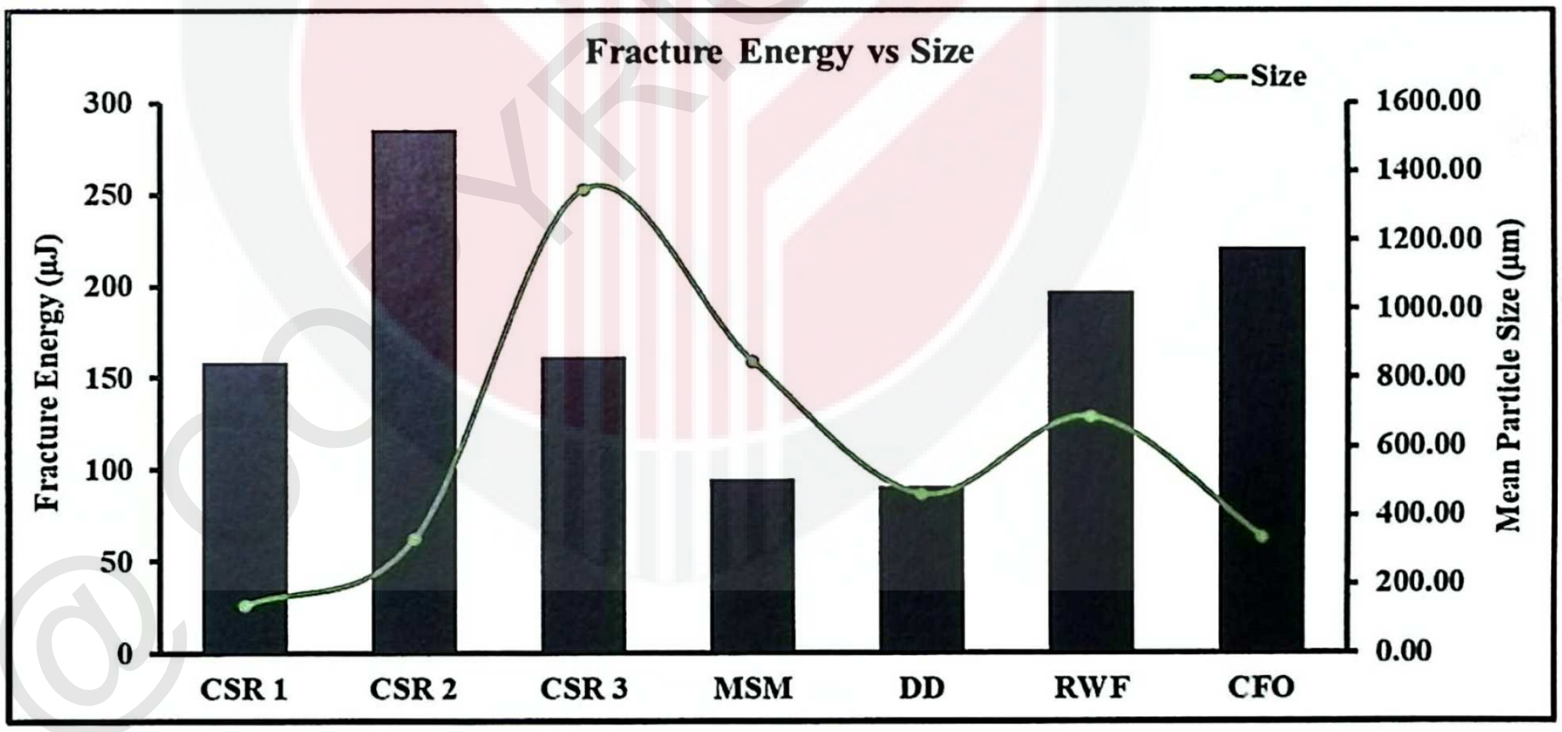


Figure 4-9: Relation between fracture energy and mean particle size of sugar

4.3 Flowability Behaviour

4.3.1 Flowability

Based on the bulk density and tapped density values identified, the flow type according to the table in

and Table 2-3 is determined based on the Carr's Compressibility Index and Hausner Ratio as shown in table below. The Carr's Index and Hausner Ratio is calculated using equation (3.53) and (3.6).

Expressing the samples flowability in ranking, MSM sample has the best flowability properties and ranked as first followed by CSR sample 3, DD, RWF, CSR 2, CFo and finally CSR 1.

Table 4-2: Sugar samples flow type based on bulk density and tapped density determined

Sample	Bulk Density $\left(\frac{kg}{m^3}\right)$	Tapped Density $\left(\frac{kg}{m^3}\right)$	Carr's Index	Hausner Ratio	Flow Type	Rank
CSR 1	515	887	42	1.72	Extremely Poor	7
CSR 2	623	942	34	1.51	Very Poor	5
CSR 3	772	878	12	1.14	Good	2
MSM	878	904	3	1.03	Excellent	1
DD	771	900	14	1.17	Good	3
RWF	613	911	33	1.49	Very Poor	4
CFO	597	912	35	1.53	Very Poor	6

As presented in Table 4-3, ranking of moisture is arranged in ascending order with the smallest value of MSM sample as number 1 followed by DD, CSR 3, CSR 2, CSR 1, CFO and lastly RWF. In contrary, the ranking for mean particle size is lined-up in descending order such that CSR sample 3 with the largest mean particle size is ranked number 1 followed by MSM, RWF, DD, CFO, CSR 2 and finally CSR 1.

Table 4-3: Ranking of moisture content and mean particle size

Sample	Moisture (%)	Moisture Rank	Mean Particle Size (µm)	Mean Particle Size Rank
CSR 1	0.75	5	138.56	7
CSR 2	0.68	4	330.47	6
CSR 3	0.29	3	1347.04	1
MSM	0.05	1	845.92	2
DD	0.27	2	460.18	4
RWF	1.93	7	684.68	3
CFO	1.43	6	335.86	5

The ranking of moisture content and mean particle size studied previously is compared with the ranking of flowability in Figure 4-10 and Figure 4-11 and it shows that the flowability is corresponding to the moisture content with only three ranks in difference at most. MSM sample with the lowest moisture content has the lowest flowability but not for sample with highest moisture. This demonstrates that moisture plays an important role in determining the flowability of soft brown sugar.

A relation between mean particle size and the flowability properties was compared on Figure 4-11 established that the mean particle size is a factor affecting flowability of soft brown sugar with only one rank in difference at most. This validates the study made by Mohamed Mathlouthi, (2014) that fine sugar particles have the worst flowability and have a relatively larger and disordered structure which makes these particles play a big role as a cement of cohesion in the case of caking.

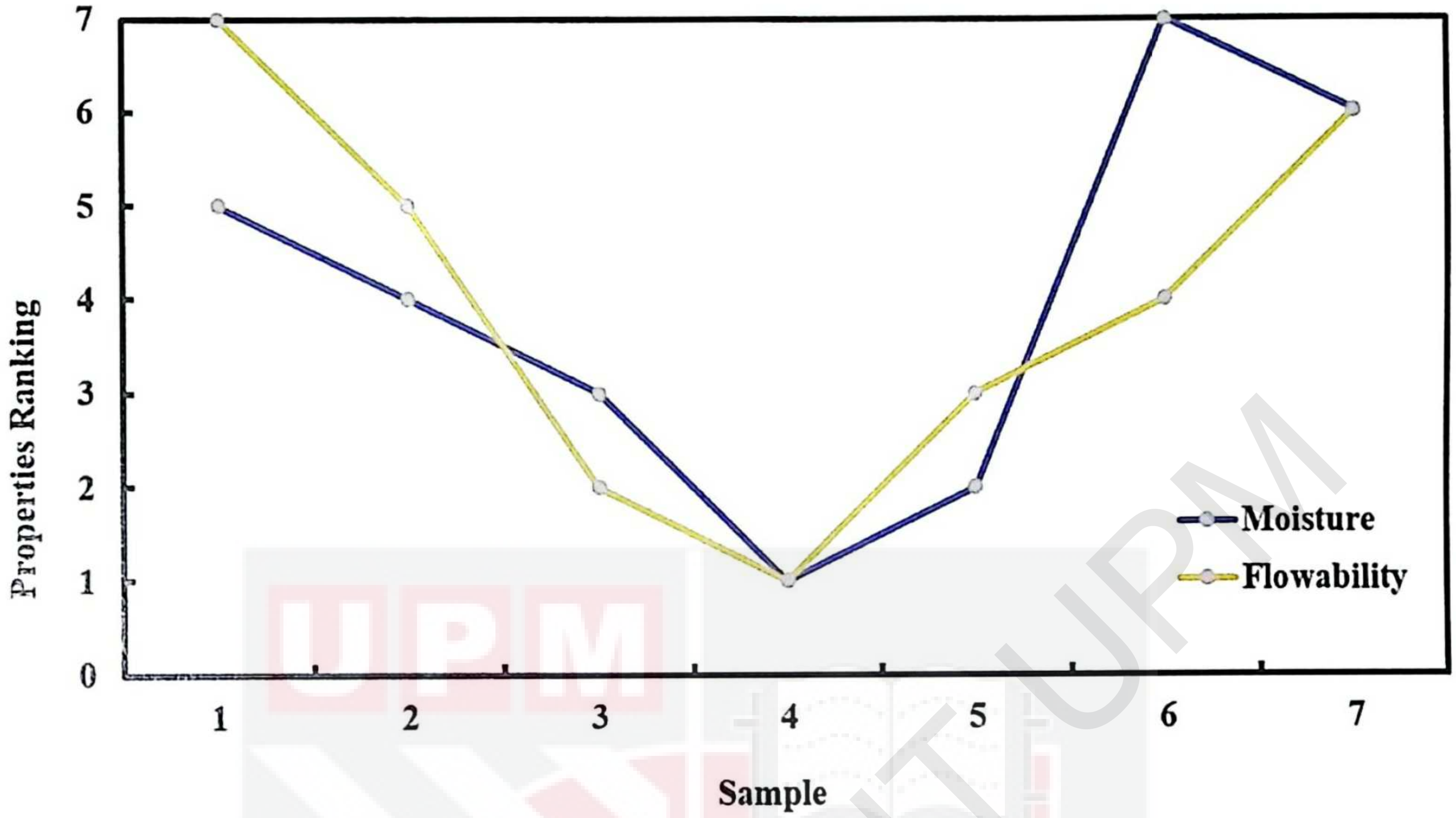


Figure 4-10: Relation between moisture content and flowability in terms of properties ranking

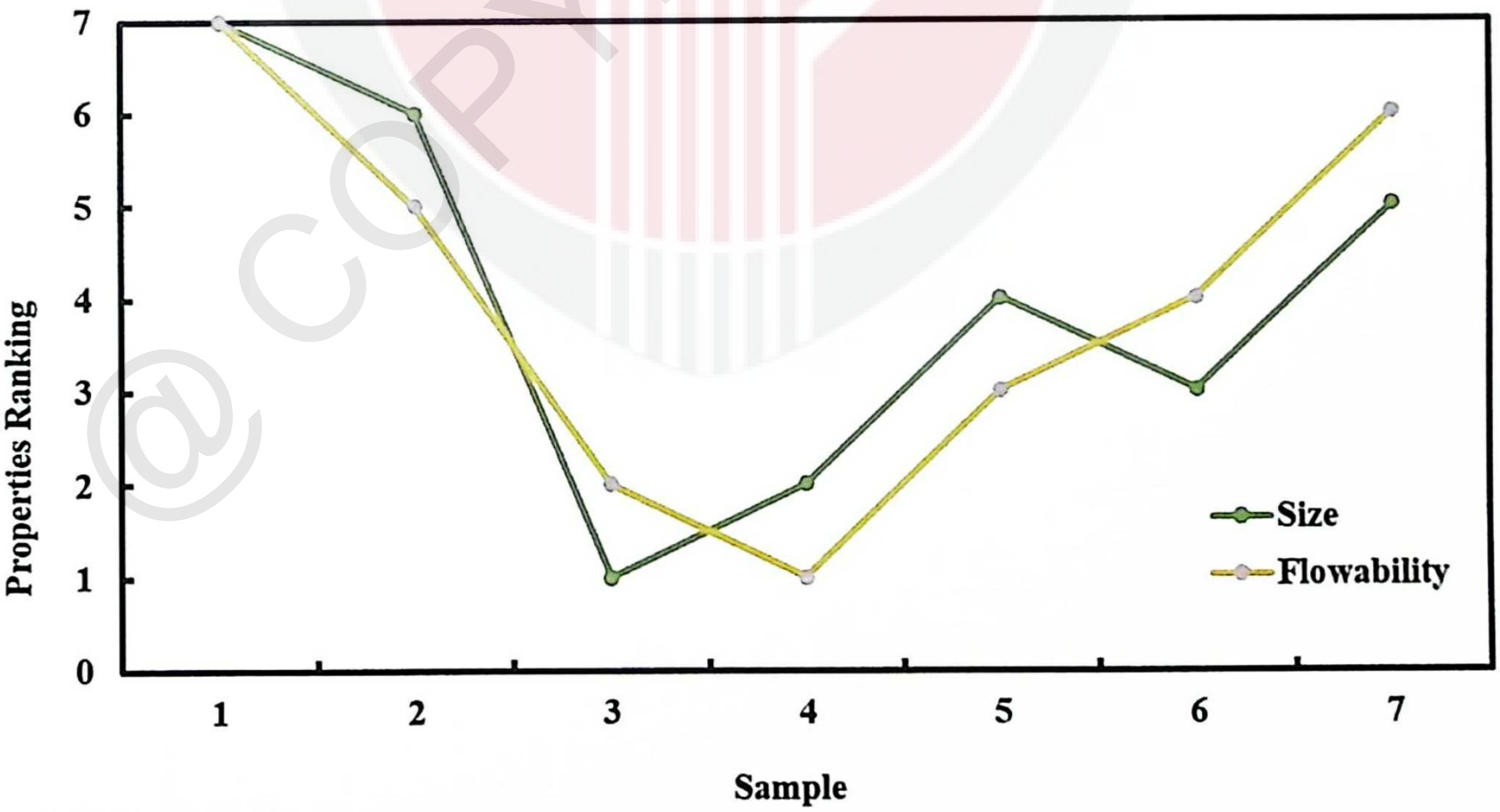


Figure 4-11 Relation between mean particle size and flowability in terms of properties ranking

The initial angle of repose of samples were measured with the forming of three different heaps by different part of sample from the packaging, and two width measurement were taken for each heap. All samples are characterized having 'good' flow type property which lies between 31-35° except for CSR 2 sample that was characterized as 'fair', having the angle of repose between 36-40° and has a large standard deviation.

Table 4-4 Initial angle of repose readings of three heaps

Sample	Angle of Repose (°)						Mean	Std Dev
	1st	2nd	3rd	4th	5th	6th		
CSR 1	34.48	33.31	37.57	35.26	35.54	35.15	35.22	1.40
CSR 2	42.44	40.86	36.22	32.38	34.78	32.66	36.55	4.22
CSR 3	30.32	31.05	34.99	34.72	33.42	33.69	33.03	1.93
MSM	32.44	32.20	33.47	32.38	33.69	34.28	33.07	0.85
DD	32.99	31.77	32.74	32.28	32.76	32.76	32.55	0.45
RWF	33.31	33.12	34.50	34.50	35.01	34.56	34.17	0.76
CFO	33.69	32.74	35.43	34.92	32.34	32.24	33.56	1.36

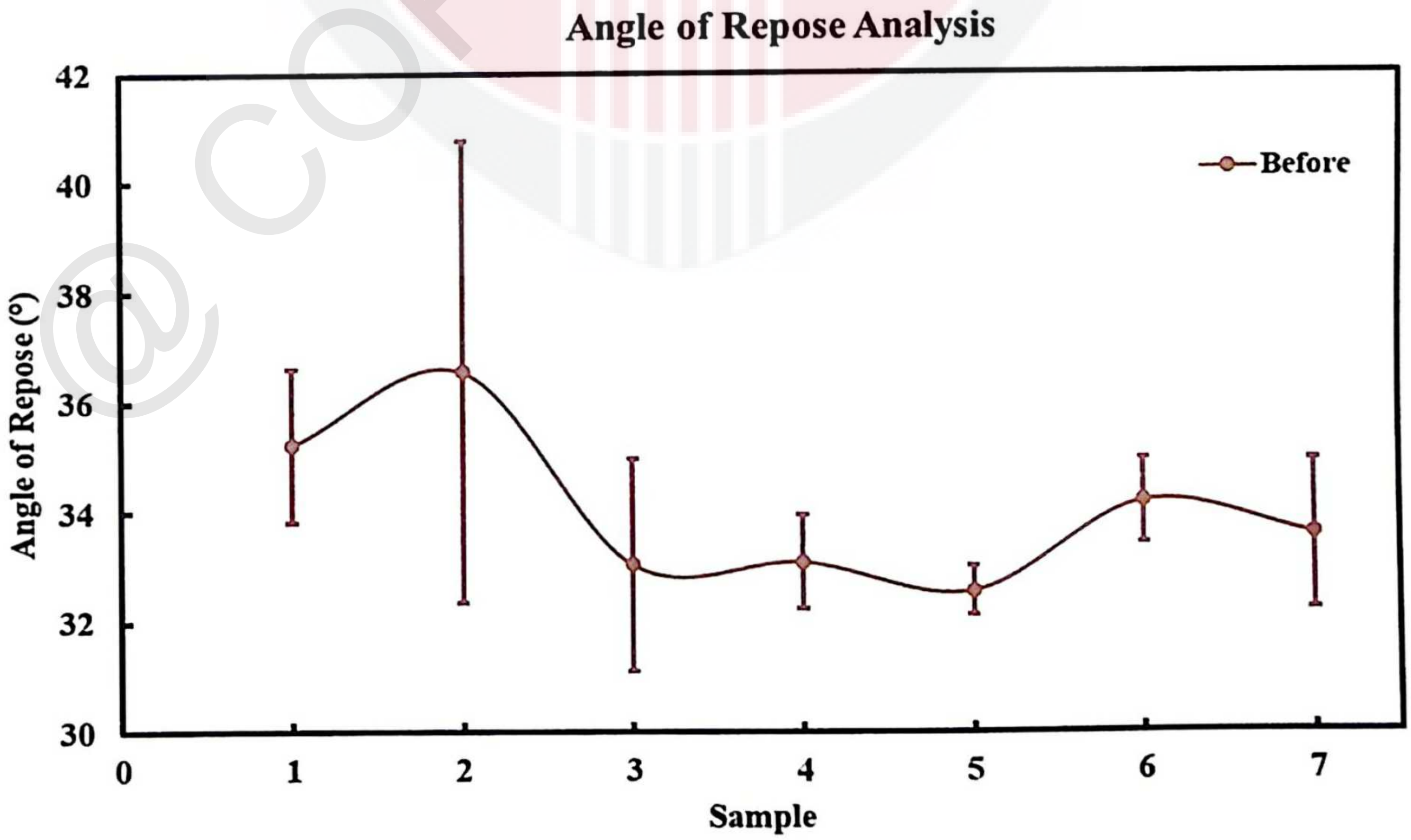


Figure 4-12: Line graph of initial angle of repose of samples

4.3.2 Effect of Humid Storage Condition

After measuring the angle of repose of samples which is used as an indication of flowability, samples were left under open air for six days in a controlled humidity and temperature environment. The chosen timeframe was concluded to be the best representation of industrial storage period. The samples were tested again using the same method of angle of repose to measure the difference in flowability. The average humidity and temperature recorded throughout the six days of storage was 68% and 28.86°C respectively.

From the graph in Figure 4-13, it appears that CSR 2 sample is the most affected to the storage condition being exposed to open air as represented by the large differences between the grey and orange lines on condition that it has a huge standard deviation. Based on the flow property classifications according to Table 2-1, CSR 1 had a negative change from a 'good' flow property to 'fair' flow property and CSR 2 had positive change from 'fair' to 'good'. Other samples had no flow property change and remained as 'good'.

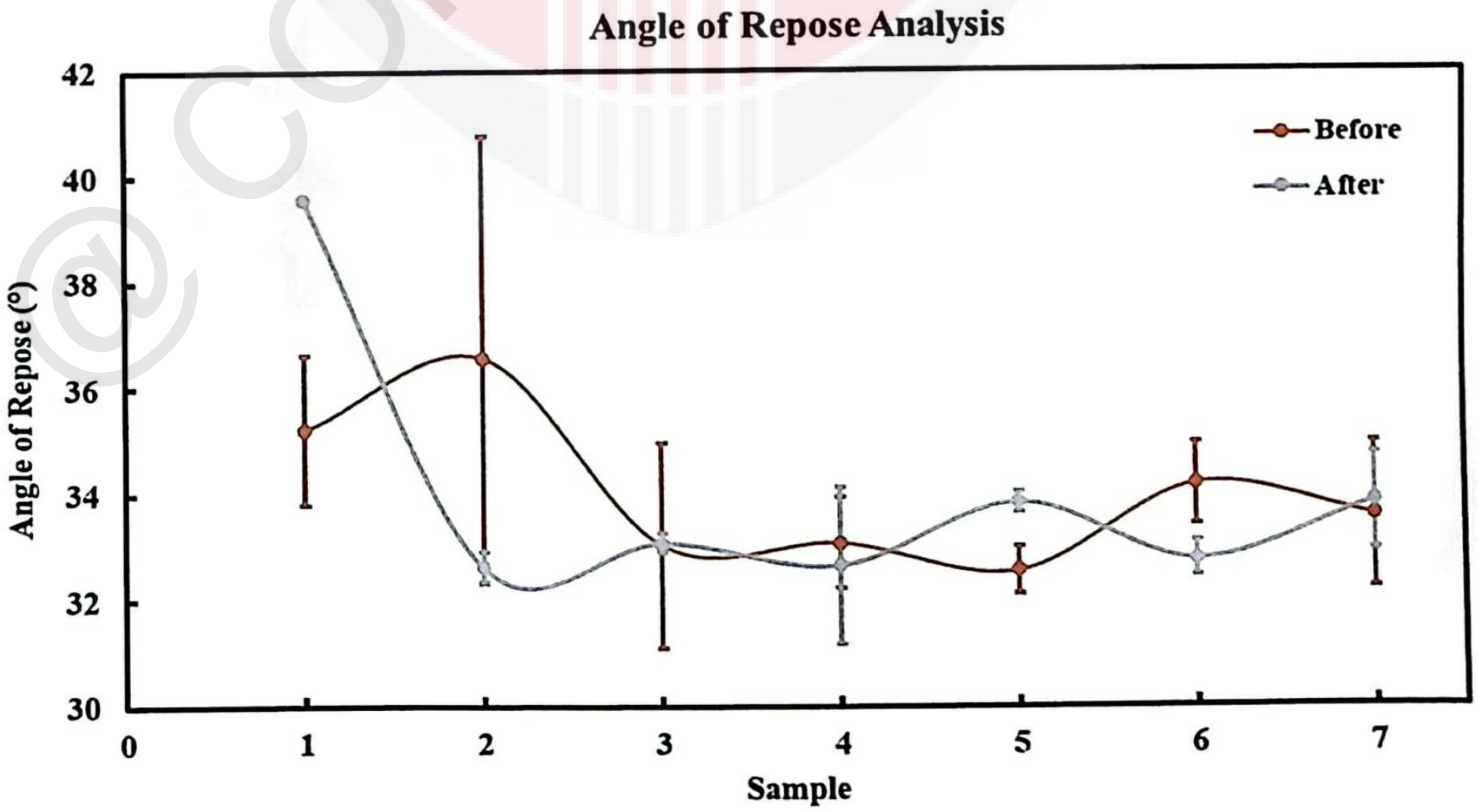


Figure 4-13: Angle of repose change indicating flowability of sugar after 6 days left

CHAPTER 5

CONCLUSIONS AND RECOMMENDATION

5.1 Conclusion

This research proves that storage condition of sugar has a significant effect on caking formation of soft brown sugar with the level of hygroscopicity that differentiate the degree of caking.

From the physical properties' tests of moisture content and colour absorbance to indicate the molasses content, both properties were directly proportional and concluded that the composition of molasses influences the moisture content. Upon relating to the sample particle size, samples of soft brown sugar with the largest mean particle size have the lowest moisture content and vice versa.

Since sugar with fine particle size is the most hygroscopic and has the most moisture content, the flowability property of sugar with fine particles is extremely bad for it has a larger surface area per unit mass to interact cohesively between particles. The flowability distinguished by the Carr's Compressibility Index and Hausner Ratio determined by the value of bulk density and tapped density shows that the degree of flow corresponds to the moisture content.

From the aspect of flowability analysis in outdoor storage, only CSR 1 and CSR 2 had changes as the other sample remained as "good" in terms of its flow type. Moisture migration is presumed to happen between the layer of moisture on the surfaces of sugar particles and the surrounding humidity. This causes the flowability, measured attributed by the angle of repose, to change after six days.

Additionally, the compaction strength identified as a result of stacking of bags in storage is proven to be another factor of caking formation. The compaction of sugar into tablets is to imitate the stacks of particles, which allows bond formation between particles. The indirect tensile strength test was carried out to deduce the extrinsic properties of samples which includes breaking force, tensile strength, and fracture energy. All extrinsic energy can be established from the force-displacement profile from the indirect tensile strength test curve.

As a whole, the overall results in correlating flowability properties shows significant correlations and eliminating the factors would be a great solution in fixing caking problems in soft brown sugar.

In conclusion, moisture content plays a big role in caking formation and the storage conditions act as a trigger for sugar cake to form. Caking of soft brown sugar is an undesirable phenomenon in food manufacturing industries as it interferes the handling, storage, transportation and processing. It is also is disruptive to the company since it might lead to a loss of functionality and a product of less quality.

5.2 Future Recommendation

A potential solution to the formation of caking is to reduce the molasses content to minimize the hygroscopicity properties of sugar, as well as sieving off fine particles that acts as base in binding the particles into caking. In terms of storage conditions, a non-stacking, closed and controlled humidity area is proposed as a way of reducing moisture migration and compressive force acting on sugar.

The results of this study appear to only consider the storage conditions. Since the manufacturing is working on a larger scale and there is no pilot scale to imitate the manufacturing process, the access is limited. Further research in this area would benefit from wider factors identification conducted over a better solution possibility. Due to limitations of laboratory use adhering to the Covid-19 SOP, it is only possible to get data from one run. A repetitive run should be considered, each test should be evaluated for at least a total of three times to assess the degree of repeatability. Additionally, having the information of molasses composition used in the making of soft brown sugar for every sample would establish a better correlation for the moisture content, colour, mean particle size and tensile strength and used as a measure of hygroscopicity. Lastly, establishing a linear regression for modelling the relationship between the caking degree and factors of caking from the data determined in this study will aid the process of predicting and preventing caking formations.

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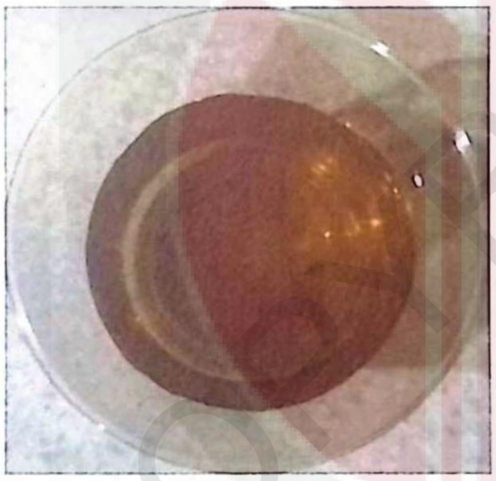
APPENDICES

APPENDIX A: SAMPLES SOLUTION TO MEASURE COLOUR

Appendix A3: Sugar solution CSR 1

Appendix A4: Sugar solution CSR 2



Appendix A5: Sugar solution CSR 3

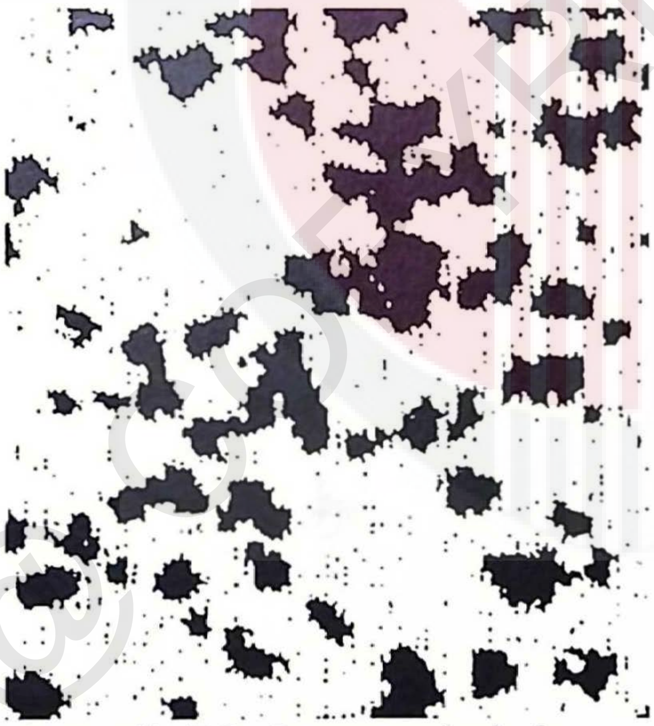
Appendix A6: Sugar solution MSM

Appendix A2: Sugar solution DD

Appendix A1: Sugar solution RWF

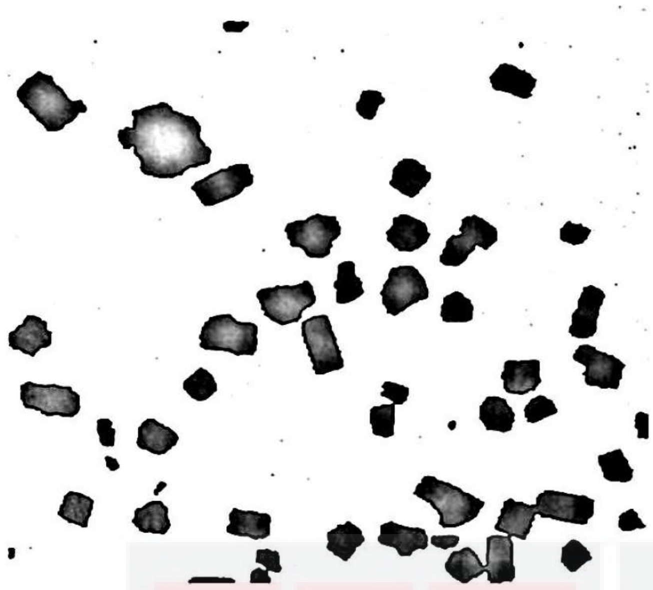
Appendix A7: Sugar solution
CFO

APPENDIX B: SAMPLES IMAGE ANALYSIS



Appendix B8: Image analysis for determination of particle size of CSR 1

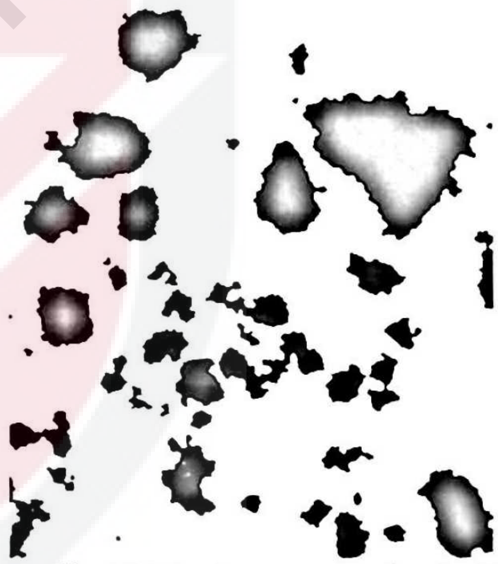
Appendix B9: Image analysis for determination of particle size of CSR 2



Appendix B10: Image analysis for letermination of particle size of CSR 3

Appendix B11: Image analysis fo determination of particle size of MSM

Appendix B12: Image analysis for determination of particle size of DD



ppendix B13: Image analysis for determination of particle size of RWF

Appendix B14: Image analysis for determination of particle size of CFO

APPENDIX C: SUGAR TABLETS

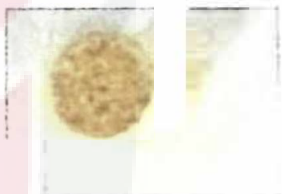
Appendix C15: Sugar tablet CSR 1

Appendix C16: Sugar tablet CSR 2

Appendix C17: Sugar tablet CSR 3

Appendix C18: Sugar tablet MSM

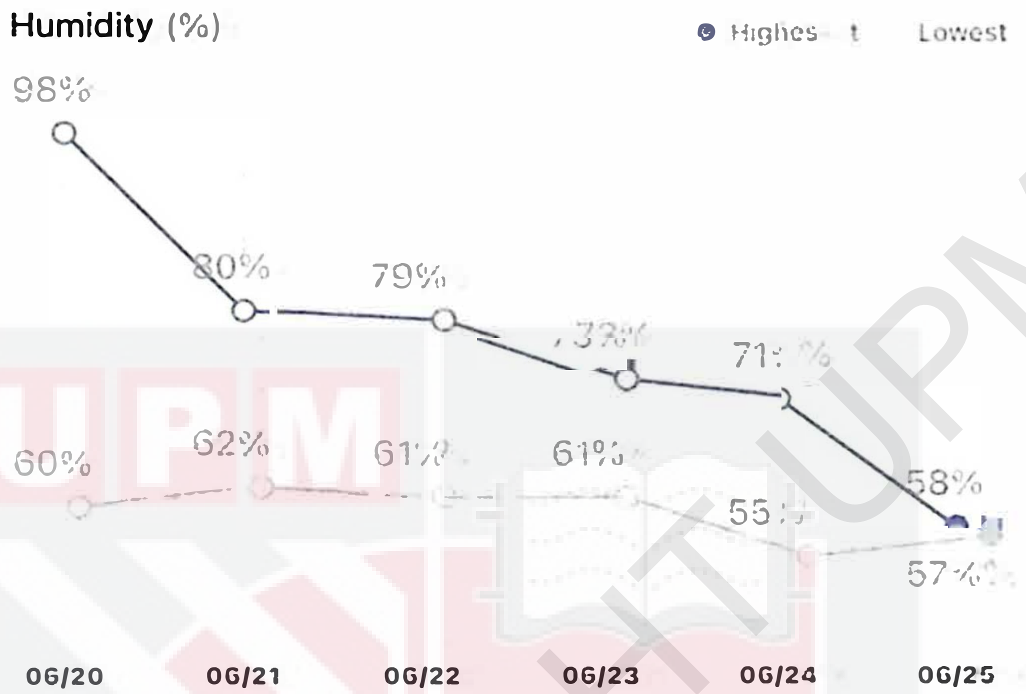
Appendix C19: Sugar tablet DD



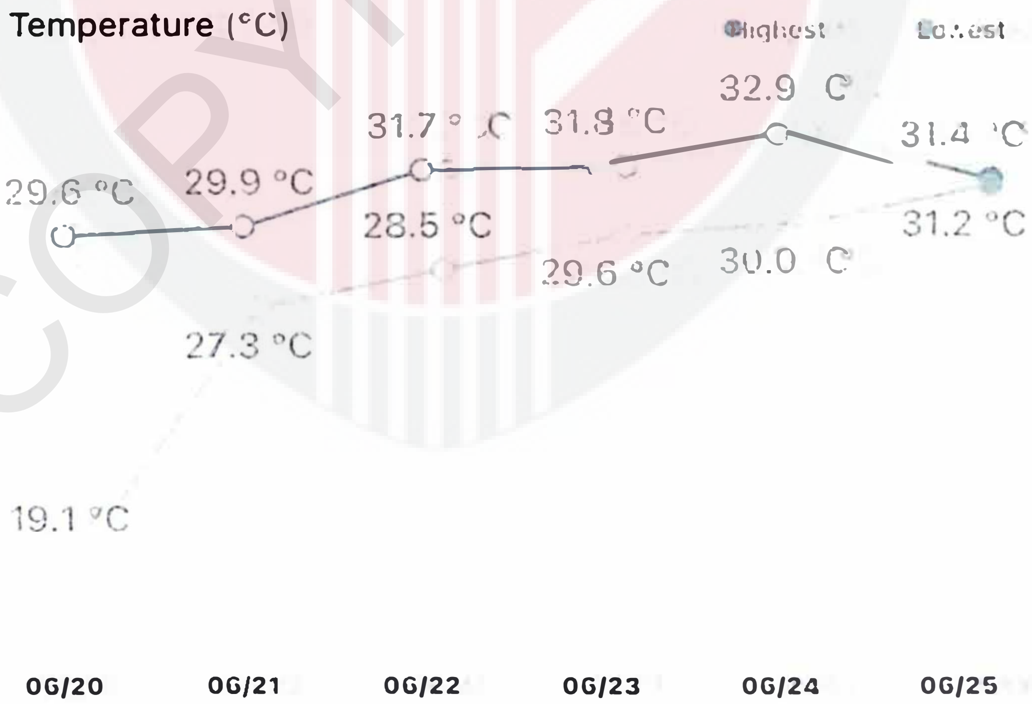
Appendix C20: Sugar tablet RWF

Appendix C21: Sugar tablet CFO

APPENDIX D: OPEN AIR STORAGE



Appendix D22 : Daily maximum and minimum humidity for open air storage analysis



Appendix D23: Daily maximum and minimum temperature for open air storage analysis

APPENDIX E: SUMMARY DATA

Sample	Moisture (%)	Colour (AU)	Mean Particle Size (μm)	Flow Type
CSR 1	0.75	0.48	138.56	Extremely Poor
CSR 2	0.68	0.42	330.47	Very Poor
CSR 3	0.29	0.37	1347.04	Good
MSM	0.05	-0.01	845.92	Excellent
DD	0.27	0.36	460.18	Good
RWF	1.93	0.71	684.68	Very Poor
CFO	1.43	0.69	335.86	Very Poor

Appendix E24: Summary for physical properties and flowability analysis

Sample	Angle of Repose (°)		Tensile Strength (Pa)
	Before	After	
CSR 1	35.22	39.58	15896.34
CSR 2	36.55	32.62	17239.94
CSR 3	33.03	33.07	26564.87
MSM	33.07	32.65	10108.31
DD	32.55	33.83	20015.03
RWF	34.17	32.76	11659.23
CFO	33.56	33.83	11334.24

Appendix E25: Summary for storage analysis