



**UNIVERSITI PUTRA MALAYSIA**

***EFFECT OF PARTICLE SIZE AND COMPRESSIBILITY ON  
FLOWABILITY OF CUMIN, CORIANDER, CHILI AND TURMERIC SPICES***

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TURMERIC SPICES**

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## ABSTRACT

This research was aiming to investigate the effect of particle size and compressibility on the flowability of spice powders and to estimate hopper diameter for the respective spice powders. Commercial cumin, coriander, chili and turmeric spices were used as samples. The flow properties, physical and chemical properties of powder were measured for each spice. The flow properties which are cake and mean cake strength, cohesion, and powder flow speed dependency were measured using a texture analyzer whereas Jenike shear cell tests from a powder flow tester was used to measure the flow properties of spices based on their frictions values in order to be used as a benchmark to design hoppers such as the hopper half-angle and the minimum outlet diameter of the hopper. The powder physical properties such as particle size, moisture content, bulk, tapped and true densities, bulk porosity, Hausner ratio, and Carr index were determined. Besides, the compressibility of each spice was measured using uniaxial die compaction via a universal testing machine. The measured particle size and compressibility of spices used in this research were ranging from 80  $\mu\text{m}$  to 300  $\mu\text{m}$ , and  $5.6 \times 10^{-7} \text{m}^3$  to  $4.2 \times 10^{-7} \text{m}^3$  at highest applied pressure, respectively, where the flowability of the spice powders, as characterized by flow factor, Hausner ratio, and Carr index, varied from easy flow to very cohesive. Apart from particle size and compressibility, moisture and fat content of the powders were determined to identify their relationship with powder flowability, whether it will be easy-flowing or very cohesive. From the findings, it was found that the smaller the particle size, the lower the volume of the compact's spices, causing the effective angle of internal friction to be lower with a higher angle of wall friction. Turmeric powder with 81.0  $\mu\text{m}$  of median particle size was the lowest compared to chili powder, coriander powder and cumin powder which

have 114.3  $\mu\text{m}$ , 225.0  $\mu\text{m}$ , and 288.6  $\mu\text{m}$  of median particle size, respectively. Hence, it can be said that turmeric powder is more easy-flowing which required a smaller hopper half angle and minimum outlet diameter of the hopper in order to provide a smooth and consistent discharge of the spices from the hopper. Thus, this study provides equipment design such as hopper for spices industry in terms of storing, processing and handling of spices powder based on their flow measurements.



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## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 Background**

Food powders are widely used materials and are amongst the most difficult materials to characterize. Because of the complexity of the powders and the increased production by the industries, there is a need for information about their processing and handling characteristics. Moreover, food powders can be classified into different categories according to their handling properties where the properties of food powders are a function of physical and chemical properties of the material, as well as size and surface characteristics of the individual particles. Apart from that, powders are the least predictable of all materials in relation to the flowability of powder because of the large number of factors that can change their flow behaviors (Fitzpatrick, 2004; Iqbal & Fitzpatrick, 2006; Saifullah, Yusof, Chin, & Aziz, 2016).

Powder flow is defined as the relative movement of the bulk of particles among neighboring particles or along the container wall surface (Peleg, 1977). If a material has to be stored, removed from storage or transferred to some other places, no serious difficulties will be met if the material is a fluid. However, when the

material is a particulate solid, especially if it is made up of very small particles, cohesiveness, friction, and interlocking between particles are factors that could complicate the handling of these solids thus can lead to flow problems. Food powder compressibility is also useful in order to evaluate the extent of the intentional or unintentional compression that a powder suffers during transportation or production. The practical objective of powder flowability investigations is to provide both qualitative and quantitative knowledge of powder behavior, which can be used in equipment design and in equipment performance prediction (Sutton, 1976).

Besides, caking and cohesion, whereby free-flowing particles aggregates to form lumps, of food powders is a common problem which can contribute to reduced product quality and shortened shelf life since it is related to particle size, cohesiveness and moisture content of the food powder itself. It can occur during processing, handling or storage and could, therefore, be a problem both in the production lines and for the consumers. According to Larsson (2016), employees working in the spice factory describe difficulties when handling caked raw materials and additional process steps, in which the powder is de-lumped, is often needed before the raw material can be added into the silos or hoppers, thereby resulting in high cost of production as more energy was needed. In other words, the smaller the particle size, the higher the degree of contact area, the more compact the particles are and the stronger the intermolecular forces resulting to poor flowability (Landillon, Cassan, Morel and Cuq, 2008). Therefore, powder flow knowledge can help us to design the optimum industrial equipment for quality requirements.

Various food ingredients in the market including spices are supplied in a powdered form where spices are frequently used in cuisine, largely to improve flavor and to provide new tastes to the food. Hence, numerous research regarding handling and storage characteristics of bulk solids of flours, herbs, dairies, pharmaceuticals, and fruits powders have been conducted over the years, however, there were fewer studies on spices. Based on the exhaustive literature review, there was very limited information on the flow properties of spices. Hence, the present study was thus planned and carried out in order to fill the knowledge gap on flow characteristics of spices. Moreover, this research will facilitate the reliable information for the design of equipment such as silos and hoppers for handling, storage, processing, packaging and transportation of ground powder spices, which will, in turn, be economically helpful for powder processing industries. Such types of difficulties or flow problems due to the agglomeration of powder particles or powder sticking on the surface of processing equipment can be handled with the help of present studies. The interest spices that have been used in this research are the commercial spice powders from BABAS® brand which are cumin, coriander, chili and turmeric spices.

## **1.2 Problem Statements**

The flow properties measurements of food powders are very important because they reflect the powders' behavior during storage, handling, processing and even during discharge from the hopper in the food industry. In this research, different types of commercial spices in powdered form were chosen according to their different physical properties mainly the particle size, in order to measure their powder behavior and properties so that an evaluation on powder flowability can be identified.

Major problems that are related to a particle size such as caking, cohesion, effective angle of internal friction and angle of wall friction can contribute to poor flowability, reduced product quality, as well as functionality, especially during storage inside the hopper. Apart from that, issues can arise when powder flow properties have not been measured, thereby flow problems can occur. Hence, understanding the caking, cohesion and powder properties, as well as the required hopper half angle and outlet diameter of the hopper, are very crucial and important since most powders will be stored in hoppers or silos, or transported at some point in the production process. In other words, a powder that cakes easily and forms a strong cake may not discharge easily from a hopper when required as they can form arching or stagnant region inside the hopper.

### **1.3 Objectives**

With the problems that have been identified, this research builds up and come out to:

- 1) To investigate the effect of particle size and compressibility on the flowability of cumin, coriander, chili and turmeric spices.
- 2) To estimate hopper diameter for cumin, coriander, chili and turmeric spices.

## CHAPTER 2

### LITERATURE REVIEW

In this chapter, the established experimental flow characterization methods on powders as well as the existing literature on research into powder compressibility, powder flowability, physical and flow characteristics of powders, and evaluations on hopper design using shear cell techniques are reviewed.

#### 2.1 Spices

Spices were among the most valuable items of trade in ancient and medieval times. Since a spice mainly comes from a seed, fruit, root, bark, or other plant substance, spices are present in almost every food preparations, processed and cooked food, including ready-to-eat products where they are often used by the consumers to provide aroma, color, flavor, taste, or texture to food without further processing (Pilizota, 2014). Some examples of spices are chili, turmeric, coriander, star anise, cloves, fennel, cumin, ginger, and black pepper. In general, spices have a more pungent flavor than herbs. It is possible for one plant to provide an herb and a spice. For example, for the plant *Coriandrum sativum*, the leaves are used as the herb cilantro while the seed is used as the spice coriander. According to Collin

(2006), the distinction between the two sources is blurred but it has been suggested that herbs tend to be of leaf origin and spices of stem, bark and seed origin.

However, spices may be ground into a powder for convenience. In addition, many food ingredients in the market including spices are supplied in powdered form in order to assist in mixing of ingredients, facilitate use and processing, transportation, storage, as well as to improve the stability of the ingredients by reducing the amount of water and enhances the availability of flavoring components and nutrients because of increase in the total surface area (Bhupendra and Goswami, 2016; Sonal, 2017). Therefore, a large quantity of spices produced industrially is in the form of powder and mostly are used as a valuable raw material for numerous food applications (Bhupendra and Goswami, 2016). According to Tangirala et al (2014), the finer powder after grounding was obtained in hammer mill for cinnamon and coriander powder while in the case of pin mill was turmeric powder. Spices powders are widely used in food products such as syrups, jams, preserves, pickles, snacks, biscuits, and candies. Apart from adding color, flavor, and taste, consumption of spices provide infinite health benefits because they are comprised of unique biological compounds like antioxidants and phenols (Mian et al., 2018).

According to Dubey (2017), the major spices exported by India are chilies (40%), turmeric (10%), cumin (10%), coriander (9.5%), fenugreek (4.2%), peppers (4%) and others (19%). In Malaysia, BABAS® is a leading curry powder and spice brand, capturing in excess of 60% of the Malaysian market. Curry powders consist of a blend of spices commonly used to flavor East Indian-style dishes and most curry powders will include cumin, coriander, chili, and turmeric powder. According to BABAS® (2015), recent studies have shown at least one in the three main meals consumed daily by Malaysian contain at least one product from the BABAS® range.

In addition, they only use the finest grade ingredients being sourced from around the world. Therefore, to assess spice production characteristics, properties and behavior, these four commercial spices which are cumin, coriander, chili and turmeric in powder forms were chosen to be analyzed for its flowability as there were lacking information on their flow properties.

### **2.1.1 Cumin (*Cuminum Cyminum*) Powder**

Cumin is a spice made from the dried seed of a plant as shown in Figure 2.1 and known as *Cuminum cyminum*, which is a member of the parsley family and it is one of the earliest cultivated herbs in Asia, Africa, and Europe (Alfaro, 2019). According to The Epicentre (2019), the seeds come as paired or separate carpels, and are 3-6 mm long, brownish in colour, boat-shaped, tapering at each extremity, with tiny stalks attached. Besides, cumin is one of the most popular spices and is commonly used in Latin American, Middle Eastern, North African, and Indian cuisines, among many others. It is available both as whole seeds as well as in powdered form. The spice has a strong, sweet and spicy aroma with a slightly bitter and pungent taste, which is especially present in the ground spice.

Cumin is used mainly where highly spiced foods are preferred in which it is an ingredient of most curry powders and much savory spice mixture. It features in Indian, Eastern, Middle Eastern, Mexican, Portuguese and Spanish cookery. Indian cumin finds worldwide use in foods, beverages, medicines, perfumery, and others. According to Dubey (2017), it is a good source of iron and keeps the immune system healthy. While the use of this spice has declined since the height of its popularity in the Middle Ages, it is making a comeback, probably due to the renewed interest in ethnic dishes and spicy foods.



Figure 2.1 Cumin seeds

### 2.1.2 Coriander (*Coriandrum Sativum*) Powder

Coriander (*Coriandrum sativum L.*) is the seed of a small plant from the family of *Apiaceae*, typically found in the Mediterranean region. The seeds are almost spherical, one end being slightly pointed while the other slightly flattened as shown in Figure 2.2 where the length of the seed is 3-5 mm (The Epicentre, 2019). Seeds and the herb of coriander, both of which are used as a spice or a medicinal plant where the seeds are widely used in perfumery, tobacco products and liqueurs (Kiralan et al., 2009; Aumatell, 2012). In addition, mature fruits are used all over the world in whole, ground, and essence or oil form. The whole plant is edible but fresh leaves and dried seeds are usually used in cooking and the stem has little or no use (Wangensteen et al., 2004; Asghar et al., 2016). It is also used as a conventional treatment for diabetes, insomnia, indigestion, and renal disorders in Morocco (Aissaoui et al., 2008; Asghar et al., 2016).

Apart from that, grounded from coriander seeds, coriander powder is an essential condiment in Indian cuisine. As mentioned earlier, the finer coriander powder after grounding was obtained in a hammer mill (Tangirala et al., 2014). Very few dishes can be made without the touch of coriander powder as it has a pleasing aroma and savors, and often it gives aromatic flavors to every curry recipe added. Coriander from which the powder is made is one of the world's oldest spices

where the seeds are dry fried and grounded since this helps to retain the aromatic flavor of the seeds. According to Dubey (2017), it is good for coping with a sore throat, allergies, digestion problems, and can also be used externally on aching joints and rheumatism. Coriander powder is usually consumed with water as it alleviates gas and aids digestion. Besides, coriander powder is an essential condiment in barbecue and it is very beneficial in medical practices as well.



Figure 2.2 Coriander seeds

### 2.1.3 Chili (*Capsicum Annuum*) Powder

Chili is one of the most spices garnishing dishes where chili is the fruit of plants commonly known as chili pepper as shown in Figure 2.3 where it belongs to *Capsicum* genus and *Solanaceae* family. *Capsicum* is derived from the Greek word "Kapsimo" meaning "to bite". When eaten, it gives a burning sensation. According to Hultquist (2018), the peppers resemble long cayenne chili peppers that grow from an average of 50.8-88.9 mm in length but growing up to 152.4 mm long or longer and somewhat slender. The pods are somewhat wrinkly and twist as they grow. Ever since its introduction, chili powder has become a basic ingredient in Indian culinary. The chili powder used in South Asian countries is made by crushing or grounding the dried chili having chili flakes and chili pods to give

highly pungent red powder where the pungency in the chilly powder varies according to the chili used.

Apart from that, chili is an indispensable spice used as a basic ingredient in everyday cuisine all over the world as chili powder adds flavor to the food. Besides, it has many beneficial properties, making it an important part in Ayurvedic medicines to cure and fight diseases, destroys harmful toxins and stimulates gastric juices that help in digesting food (Ramdev, 2019). It also helps in clearing nasal congestion, relieves throat infection, and acts as a painkiller because when chili is eaten, it makes the brain to release endorphins which is a natural painkiller present in the body.



Figure 2.3 Chili peppers

#### 2.1.4 Turmeric (*Curcuma Longa*) Powder

Turmeric (*Curcuma Longa*) is the rhizome or underground stem of a ginger-like plant. The rhizome of turmeric as shown in Figure 2.4 is yellowish-brown with a dull orange interior that looks bright yellow when powdered. The main rhizome measures 25–70 mm length with a diameter of 25 mm, with smaller tubers branching off (The Epicentre, 2019). Turmeric is usually available ground, as a bright yellow, fine powder where the bright yellow powder is used in adding flavor and color to dishes, has properties that make it an effective ingredient in

medicines, cosmetics, and as a color dye, as well as used to heal wounds and rashes in a traditional medicine (Bhatia and Dahiya, 2015). As previously mentioned, according to Tangirala et al (2014), the finer turmeric powder after grounding was obtained in a pin mill.

Turmeric powder is the major constituent of curry powder used in confectionery industries for food seasoning and in the international market as a functional food due to its health-promoting properties (Akinpelu et al., 2012). Besides, the ground turmeric root has been used in Indian and Chinese cooking as well as traditional medicine for centuries (Macmillan, 2017). Turmeric is also used in the treatment of ulcers and liver disorders as herbal medicine since the antibacterial and antiseptic properties of turmeric are well known. According to Pujari et al (1986), on account of its flavor and medicinal properties, turmeric is also used in the preparation of cosmetics, soaps, ointments, face creams, and others.



Figure 2.4 Rhizomes of turmeric

Therefore, since most spice powders are grounded in order to reduce the size to obtain the powder, they are also facing challenges concerning powder flow properties as it can contribute to the risk of flow problems in the course of their industrial processing just similar to flour, herbal, pharmaceuticals, and fruit

powders which have been reported by numerous previous researchers (Teunou et al., 1998; Fitzpatrick et al., 2004; Xiu et al., 2008; Schulze, 2008; Yusof et al., 2009; Aulton, 2009; Juliano and Barbosa-Canovas, 2010; Yusof et al., 2010; Etti et al., 2014; Lecturia et al., 2014; Mohd Salleh et al., 2014; Slettengren et al., 2015; Saifullah et al., 2016; Jan et al., 2017; Nurhadi and Roos, 2017; Jan et al., 2018). Flow problems occur with cohesive powders of any nature, but may be more serious with food powders because they are commonly related to releasing sticky substances such as fat or to the presence of hygroscopic behavior, temperature and time of consolidation. Hence, a proper understanding on the flow behavior of spice powders is crucial and necessary in order to control or prevent problems from occurring such as stoppages or poor quality products that may result from the powder flowability where it generally depends on the properties of the powder itself. The practical objective of powder flowability investigations is to provide both qualitative and quantitative knowledge of powder behavior, which can be used in equipment design and in equipment performance prediction (Sutton, 1976). Thus, to guarantee a steady and reliable flow, it is crucial to accurately characterize the flow behavior of powders.

## **2.2 Properties of Powder**

Powders can be produced with a variety of different physical and chemical properties. Thus, the measurement of these two properties is crucial and important because both can help to define the powder as they intrinsically affect its behavior during storage especially in hoppers and silos, as well as handling and processing. The physical properties are particle size, bulk density, tapped density, true density, porosity, and moisture content whereas the flow properties are flowability along with its Hausner's ratio and Carr Index.

### **2.2.1 Particle Size**

Particle size is one of the most important parameters for the food powders in order to characterize the food since it has a major influence on powder flowability. Generally, the larger the particle size, the better the flowability of powders (Abdullah and Geldart, 1999). Besides,  $d_{50}$  is usually used to represent the particle size of a group of particles where it is known as the median diameter or the medium value of the particle size distribution. In other words,  $d_{50}$  is the value of the particle diameter at 50% in the cumulative distribution and it is one of an important parameter characterizing particle size. According to Teunou et al (1999) and Liu et al (2008), it is generally considered that powders with particle sizes larger than 200  $\mu\text{m}$  were free-flowing, while fine powders with particle sizes that fall below 100  $\mu\text{m}$  are subject to caking and cohesion, thus making their flowability to be more difficult. Apart from that, particles in the size range of 75-250  $\mu\text{m}$  may flow freely or cause problems, depending on the shape and other factors. With particles less than 100  $\mu\text{m}$  in size, flow is a problem. The reduction in flowability at smaller particle size is due to the increased surface area per unit mass of powder, thus, more surface area is available for cohesive forces, and frictional forces to resist flow (Jan, Ghoroi and Saxena, 2017). Therefore, according to Ferrari and Bell (1998), without careful engineering and selection of discharge equipment and hopper geometry, finer powders that have mean particle size at about 1000  $\mu\text{m}$  and below, frequently will not discharge reliably from the hoppers.

Apart from that, some previous researches done by Katikaneni et al., (1995); Teunou et al. (1999); Fitzpatrick et al, (2004); and Jan et al., (2018) showed that powder was cohesive when the particle size decreased. This happened because as mentioned earlier, the particle surface area per unit mass has increased, thus it tends

to increase cohesion behavior and resulting in more cohesive and difficult-flowing powders. In other words, the finer the particle size, the more contact area between particles, which leads to greater cohesive forces among the particles (Marinelli and Carson, 1992).

### **2.2.2 Density**

Density is the measurement of how much mass is contained in a given unit volume. The density of food material is used by the industry to adjust storage, processing, packaging and distribution conditions. In addition, density also relates to the movement of the particle and the porosity of the particle. Moreover, the density and the ability of a powder to increase its density are important parameters for storage, transportation, caking, and others. According to Yusof et al (2009), the density measurement is a very important means to characterize the compaction process for material in the powdered form. Therefore, density properties are used as an indication of powder compressibility and powder flowability (Barbosa-Canovas et al., 2005; Xanthakis et al., 2015) where it involved the measurement of bulk density, tapped density, and true density.

#### **2.2.2.1 Bulk Density**

Bulk density is a measure when the particle is simply poured inside a container without tapping the powder as we want to measure the volume or the height of the powders inside the container. It is used in determining the amount of powder that can fit in a confined space such as a hopper or a tablet filler. According to Barbosa-Canovas et al (1987), bulk density of a food powder is highly dependent on the particle size and its distribution where theoretically, the bulk density increased when the particle decreased, thereby will produce a powder with good flowability. In addition, bulk density has a relation with compressibility as well

where the relative increase of bulk density after compression of powder was an indicator that the powder had better compressibility and flowability of powders (Yusof et al., 2009; Yusof et al., 2010; Saifullah et al., 2016).

#### **2.2.2.2 Tapped Density**

Tapped density is the constant density after tapping several times. The tapped density is an increased bulk density attained after mechanically tapping a container or a graduated cylinder containing the powder sample for a defined period of time until little further volume change is observed. This density is required for measuring the cohesiveness of the powder by calculating the Hausner ratio and Carr index. The difference between the bulk and tapped densities determines the degree of consolidation and how much empty space will be left on top of a jar filled with product at the factory after it has been shaken through transportation (Coucoulas, 2003). Furthermore, tapped density is directly related to the classification on the flowability of powders because it corresponds to the Hausner ratio and Carr index values. Therefore, the higher the tapped density, the higher the Hausner ratio and Carr index, thus resulting in a cohesive and poor flowability of the powder (Xiu et al., 2008; Yusof et al., 2009; Yusof et al., 2010; Saifullah et al., 2016).

#### **2.2.2.3 True Density**

True density is the density of the material when it does not include any interstitial pores. In other words, true density is represented by the mass of the particle divided by the volume excluding open and closed pores where helium gas will penetrate the open and closed pores, thus gives the true density value. There were several studies that have reported the concern on the increase in the true density under compression, of directly compressible materials (Blattner et al., 1986; Pedersen and Kristensen, 1994). Moreover, since most inorganic materials consist

of rigid particles, while most organic substances are normally soft, porous particles, true density of many food powders is considerably lower than that of mineral and metallic powders. Typical non-metallic minerals have true particle densities well over  $2,000 \text{ kg/m}^3$ , while some metallic powders can present true densities of the order of  $700 \text{ kg/m}^3$ . By contrast, most food particles have densities considerably lower of about  $1000\text{-}1500 \text{ kg/m}^3$  (Ibarz and Barbosa-Canovas, 2014). These trends can be seen in various previous works by Fitzpatrick et al., (2004); Yusof et al., (2009); Yusof et al., (2010); Mohd Salleh et al., (2013); and Saifullah et al., (2016).

### **2.2.3 Porosity**

Porosity characterizes the open structure of a material. It is the fraction of the empty volume (void fraction) and it is usually estimated from the bulk density and the true density of the material. The void is the space between the particles which results in porosity. If the particles were not uniform, the smaller particles will slip into the void spaces between the larger particles and decrease the void areas. According to Boukouvalas et al (2006), generally, food powders present a high porosity between 40% and 80% from internal, external, and interparticle pores. Bulk porosity can vary considerably due to mechanical compaction, the difference in particle sizes which is the concentration of fines, moisture content, and temperature (Boukouvalas et al., 2006). Low porosity tends to possess free-flowing properties. However, the porosity can be affected by the chemical nature of each constituent powder as well as from the process from which particles were originated. Changes in environmentally and time-dependent parameters such as moisture and temperature during storage can vary the interaction between particles, therefore affecting its density and porosity.

#### **2.2.4 Moisture Content**

Moisture content is one of the factors that are significant and can influence the food powder characteristic which is the flowability of powders. When the term moisture content is used in the food industry, it almost always refers to wet basis moisture content. Smaller particle size will increase the moisture content, thus leads to poor flowability due to the increase in liquid bridges and capillary forces acting between the powder particles (Scoville and Peleg, 1981; Teunou et al., 1999; Fitzpatrick et al., 2004; Jan et al., 2017). In addition, this may also lead to severe flowability problems due to powder caking. According to FMC Biopolymer (2009), one of the reasons for increasing moisture content of any powder product could be the hygroscopicity of powder, which facilitates powders to absorb moisture easily from the air. The moisture content of a food is normally expressed simply as the percent moisture in the food substances (Figura & Teixeira, 2007). Most dry powders are free-flowing and their handling does not represent a technical problem but wet or higher-moisture powders, on the other hand, will typically cause more transport, storage, and flow difficulties (Juarez-Enriquez et al., 2017).

#### **2.3 Compressibility**

Compressibility of powder is influenced by the flow properties where compressibility is used to predict whether the powder is free-flowing or cohesive. Food powder compressibility is the ability to reduce volume under pressure, therefore is useful to evaluate the extent of the intentional or unintentional compression that a powder suffers during transport or production (Grossmann, Tomas & Csoke, 2004; Yusof et al., 2009). According to Jan, Ghoroi and Saxena, (2017), high compressibility is often associated with high cohesivity of the powders in addition to other factors such as bulk density and packing structure. Thus, the

lower the compact's volume, the higher the density of the tablet and higher density obtained after compression indicate better compressibility (Ooi, 2008; Yusof et al., 2009; Yusof et al., 2010; Jan et al., 2018). Furthermore, according to Yusof et al (2009), the main characteristics to be investigated for compressibility usually were done by uniaxial die compaction

### 2.3.1 Uniaxial Die Compaction

In direct compression of powders, the dry powder blends must flow uniformly into the tablet dies in order to obtain a uniform product. According to Yusof et al (2009), uniaxial die compaction as shown in Figure 2.5 is a compression process of powder within a die cavity by the action of an upper punch at a constant velocity, while the lower punch does not move within the mechanical assembly. This process describes the volume reduction of powder due to the applied load in a confined space. When the powder is compressed, the air voids inside it is reduced and the powder particles are brought closer together to form a powder compact. This is significant in order to evaluate the compressibility of powder in order to measure the compact's volume after being compressed. Also, the uniaxial die compaction method was used to investigate the physical properties of food powders.

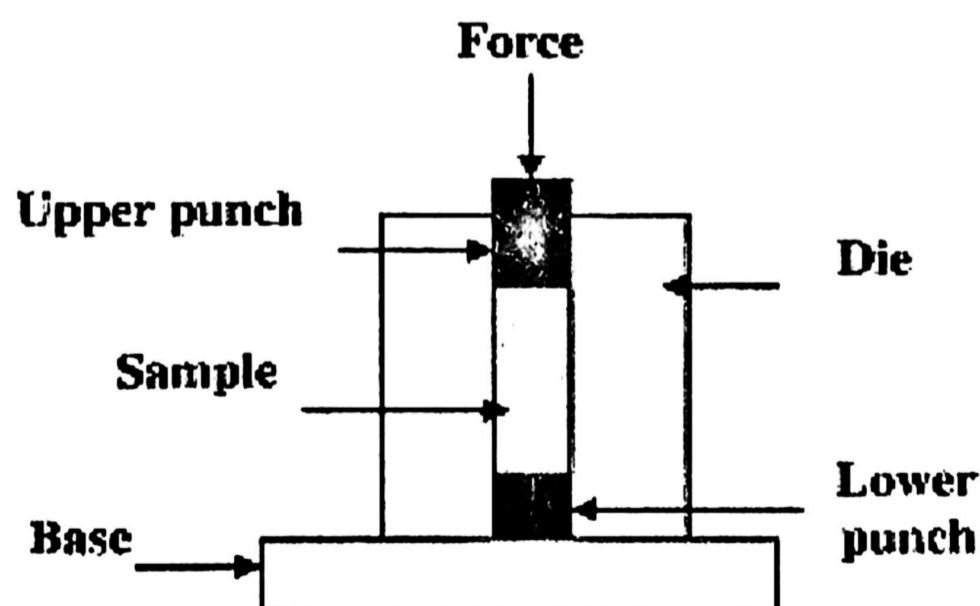


Figure 2.5 A schematic diagram of uniaxial die compaction (Yusof et al., 2009)

## **2.4 Flowability of Powders**

The knowledge about flow properties of powders is important in case of developing powder processes, storage, and handling procedures, as well as packaging practices and transporting the processed powders (Schubert, 1987; Meghwal and Goswami, 2015). Physical properties of the powder such as particle size, bulk density, and tapped density were found to have more impact on its flowability but somehow chemical properties such as moisture content as well as fat content of the powder composition can also affect the flowability of powders (Augestein and Hogg, 1978; Teunou et al., 1999; Fitzpatrick et al., 2004; Fitzpatrick et al., 2006; Nijdam and Langrish, 2006).

The powder flow properties such as compressibility, cohesion and caking strength, as well as friction result from collective forces such as the effective angle of internal friction, and angle of wall friction. The cohesive nature of fine powders is due to the presence of a cohesive component to their shear strength in addition to a frictional component. The cohesive component is enhanced by the presence of moisture as well as small particle size. Therefore, the smaller the particle size, the more cohesive the powder becomes, and the powder will increase in cake strength and becomes difficult to flow (Augestein and Hogg, 1978; Teunou et al., 1999; Fitzpatrick et al., 2004). The tendency of a powder to cake can give important data about the properties of the powder after storage and transportation. The formation of strong, high height cakes may lead to issues with the discharge of powders from storage hoppers or silos. Product settlement and cake formation may also impact on customer perception of products, as it may appear that the product is less voluminous than stated. Proper understanding of the caking characteristics of

powder may assist manufacturers in minimizing issues after storage or transportation (Stable Micro Systems, 2016).

Apart from that, Benkovic and Bauman (2009) have developed the powder categorization scale based on cohesion index of the powder as shown in Table 2.1 in order to determine the flow behavior of powders. In addition, poor powder flowability is a consequence of the combined effects of many variables including improper equipment design, particle size, densities, fat content, moisture content, and frictions (Bodhimage, 2006; Fitzpatrick et al., 2004).

Table 2.1 Powder characterization scale based on cohesion index (Benkovic and Bauman, 2009)

Cohesion index	Flow behavior
>19	Hardened, extremely cohesive
16-19	Very cohesive
14-16	Cohesive
11-14	Easy flowing
11	Free-flowing

#### 2.4.1 Hausner Ratio

The flowability and compressibility of powder were determined by the classification of flowability where mostly expressed using the Hausner ratio, which depended on the ratio of the tapped and bulk densities of the powder (Hausner, 1967).

$$HR = \frac{\rho_t}{\rho_b} \quad (2.1)$$

Where  $HR$  is Hausner Ratio,  $\rho_t$  is tapped density and  $\rho_b$  is bulk density. Hayes (1987) has defined the different ranges for the Hausner ratio for characterizing flowability where free-flowing powder means that the powder is a cohesive and difficult flowing powder as non-cohesive.

- $1.0 < HR < 1.1$  , free-flowing powder;
- $1.1 < HR < 1.25$  , medium-flowing powder;
- $1.25 < HR < 1.4$  , difficult-flowing powder;
- $HR > 1.4$  , very difficult flowing powder

#### 2.4.2 Carr Index

Compressibility is also one of the tests proposed by Carr for the assessment of powder flow properties (Carr, 1965). The Carr Index was calculated based on the equation below :

$$CI = \frac{\rho_t - \rho_b}{\rho_t} \times 100\% \quad (2.2)$$

Where  $CI$  is Carr Index,  $\rho_t$  is tapped density and  $\rho_b$  is bulk density.

Apart from that, the flow character of powder samples can be classified from excellent to very poor based on the values of Hausner Ratio and Carr Index obtained from the bulk and tapped densities of the powder. According to Lebrun et al (2012), the different ranges for the Carr index and the Hausner ratio have been classified as shown in Table 2.2 below.

Table 2.2 Flowability classification (Lebrun et al., 2012)

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

### 2.5 Hopper Design from Jenike Shear Cell Tests Measurements

One of the applications of flowability information is its use in the design of hoppers for mass flow. A test method that has gained interest in practice was the shear cell where Jenike pioneered the application of shear cell techniques as shown in Figure 2.6 for measuring the sliding friction between powder particles during flow which are effective angle of internal friction and angle of wall friction (Jenike et al. 1960; Teunou et al., 1999; Fitzpatrick et al., 2004; Mohd Salleh et al., 2013; Slettengren et al., 2015; Nurhadi and Roos, 2017). According to Mohd Salleh et al (2013), the smaller the particle size, the higher the angle of wall friction, thus the flowability will be reduced.

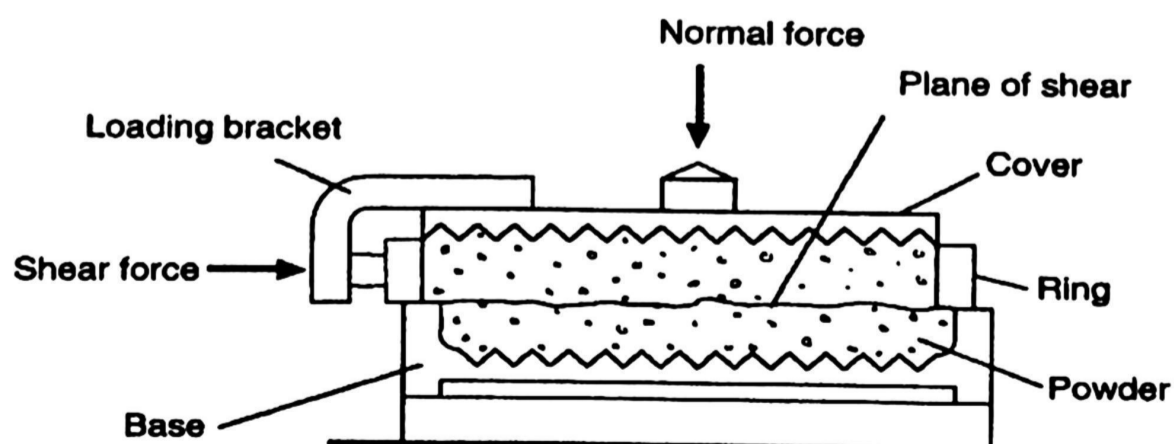


Figure 2.6 A schematic diagram of Jenike shear cell (Jenike, 1960)

Shear testers were used to evaluate how the unconfined yield strength (UYS) of the powder varies with major consolidation stress (MCS) (Schulze, 2008; Descamps et al., 2013) and the resulting data from these were called flow function which can be used to rate the powder flowability as shown in Figure 2.7 below according to the flow factor values that has been classified by Jenike (1960) in Table 2.3. The flow factor values were obtained by the ratio of the major principal consolidation stress (MCS) and unconfined yield strength (UYS). The lower the flow factor, the steeper the flow function line, thus the more difficult it is for the powder to flow, as the graph represents the strength that develops within the powder when it consolidates (Fitzpatrick et al. 2004; Mohd Salleh et al, 2013; Nurhadi and Roos, 2017). Apart from that, the lower the major principal consolidating stress, the higher the effective angle of internal friction (Barbosa-muriet Canovas et al., 2005; Slettengren et al., 2016; Nurhadi and Roos, 2017), thus the more free-flowing properties the powder has.

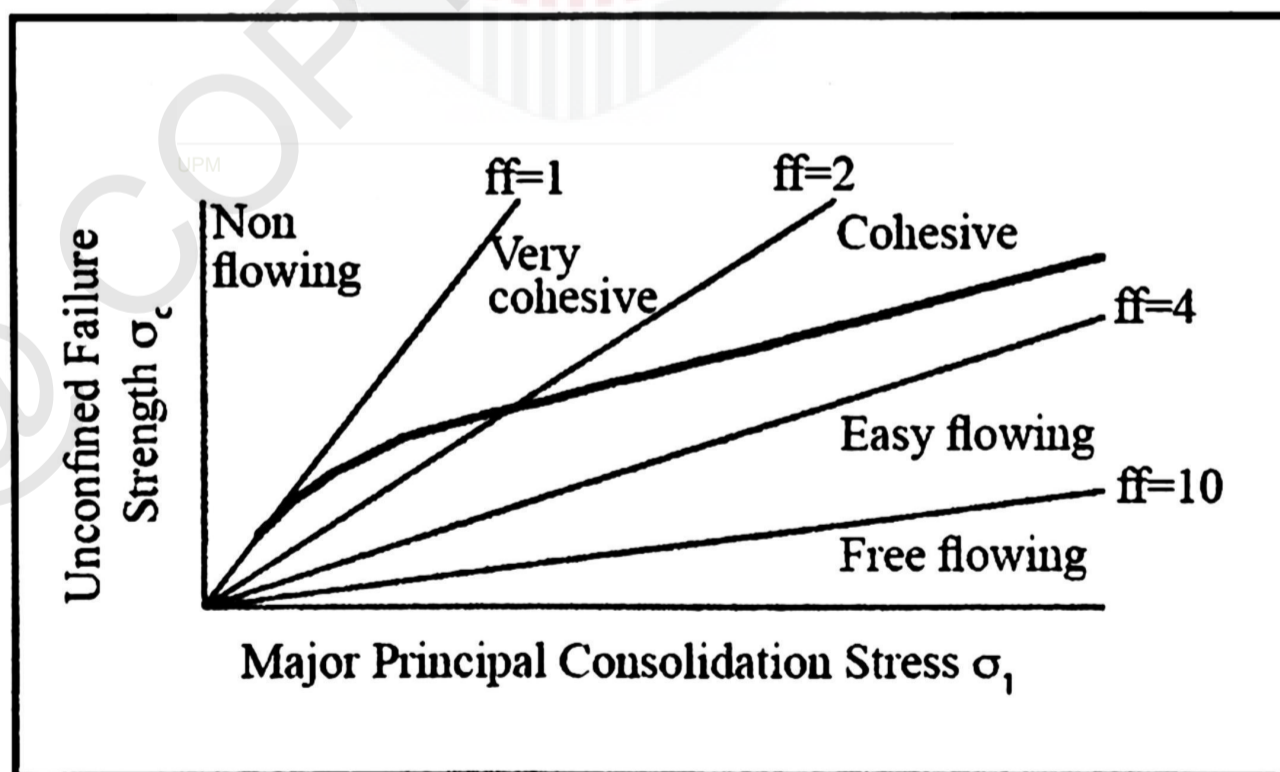


Figure 2.7 Powder flow function

Table 2.3 Jenike classification of powder flowability by flow factor ( $ff$ ) (Jenike, 1960)

Flow factor ( $ff$ )	Flowability
$ff < 1$	Non-flowing
$1 < ff < 2$	Very cohesive
$2 < ff < 4$	Cohesive
$4 < ff < 10$	Easy flowing
$ff > 10$	Free-flowing

Apart from that, according to Mohd Salleh et al (2013), this test is commonly used to determine the flow characteristic of powders such as cement (Schrämli 2001), soils, food (Kamath et al., 1993; Fitzpatrick et al. 2004), pharmaceutical drugs (Emery et al., 2008) and other materials (Schwedde, 2001; Ganesan et al., 2008). The flow function can also be used to assess the minimum hopper angle and hopper opening size for achieving uniform flow. Thus, Tim (2009) stated that selecting an appropriate outlet size and hopper half-angle will achieve the aim to consistently and steadily discharge the powder because hopper half angle strongly influences the flow mode or regime whether the mass flow or core (funnel) flow as shown in Figure 2.8 that develops within the silo and conical hopper. A steeper hopper wall required a smaller hopper half angles to encourage mass flow as opposed to core flow. Mass flow occurs when the cone is sufficiently smooth or steep to ensure particle movement along the wall whereas the core flow occurs in hoppers with a shallower cone angle, which allows a stagnant region to develop along the walls of the converging section.

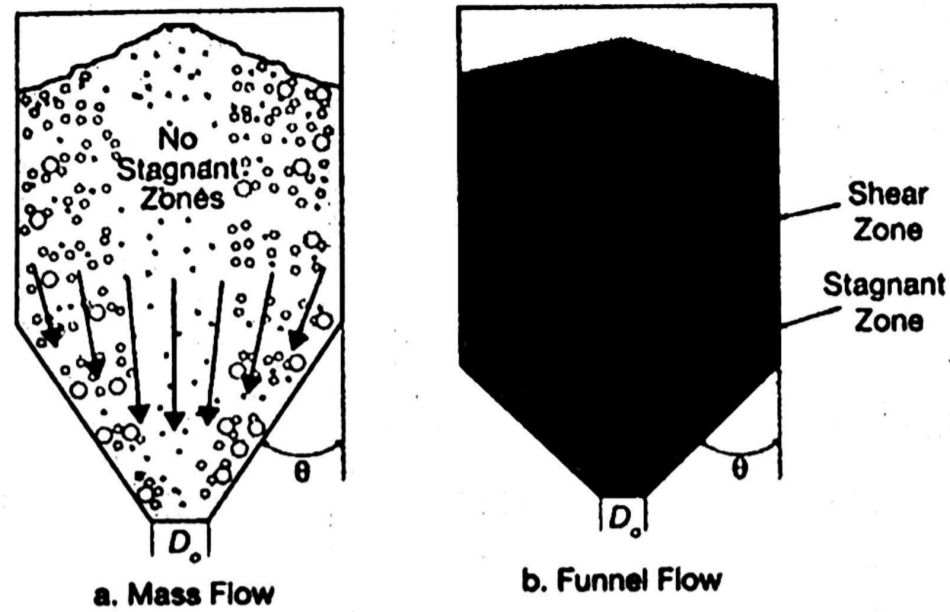


Figure 2.8 Mass flow and core flow that develops within silo and conical hopper

For a given powder there is a critical outlet dimension that must be exceeded to ensure reliable discharge of a mass-flow vessel (critical arching diameter,  $D_{\text{arching}}$ ) or core-flow (critical rat-hole diameter,  $D_{\text{RH}}$ ) as shown in Figure 2.9.

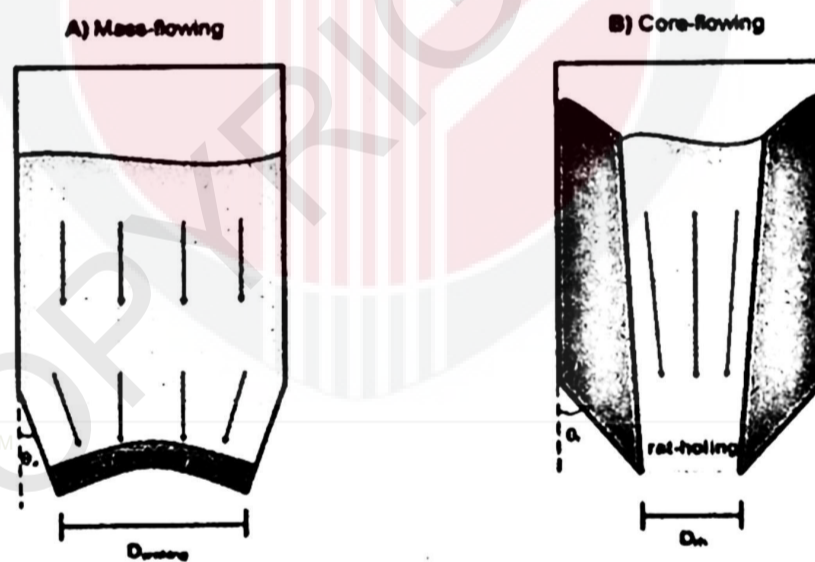


Figure 2.9 Critical outlet dimension for mass flow and core flow

Jenike's theory, based on the shear strength of the material, remains the basis for determining powder flowability, the design of silos for mass flow, and the sizing of silo outlets to prevent arching and ratholing. The shear strength is the resistance of a bulk material against failure or flow. According to Teunou et al (1999); Bell (1999); Fitzpatrick et al (2004); and Mohd Salleh et al (2013), Jenike's shear testing methods have proven to be valid and reproducible for a broad range of

powders. The Jenike shear cell provides measurements of powder cohesive properties that can be used to design a hopper to prevent arching and rathole formation (Behera et al., 2002). Therefore, Jenike shear cell was used in this research to calculate the critical minimum hopper outlet diameter by using Equation 2.3 below and hopper half angle for the design of a mass flow hopper for different bulk materials based on the results of effective angle of internal friction, angle of wall friction, flow factor, critical stress and bulk density from the shear cell test.

$$B = \frac{H(\theta)\sigma_{crit}}{\rho_b g} \quad (2.3)$$

Where  $B$  is the minimum outlet diameter,  $H(\theta)$  is a function of hopper angle,  $\sigma_{crit}$  is critical stress,  $\rho_b$  is bulk density and  $g$  is gravity.

## CHAPTER 3

### MATERIALS AND METHODOLOGY

#### 3.1 Materials

The materials that have been used in this research were four different types of commercial spices (BABAS® brand, Malaysia) in powder form which is cumin (*Cuminum Cyminum*) powder, coriander (*Coriandrum Sativum*) powder, chili (*Capsicum Annuum*) powder and turmeric (*Curcuma Longa*) powder as shown in Figure 3.1 until Figure 3.4. These four materials were bought from local supermarkets located in Sri Serdang, Selangor Darul Ehsan, Malaysia.



Figure 3.1 Cumin powder



Figure 3.2 Coriander powder

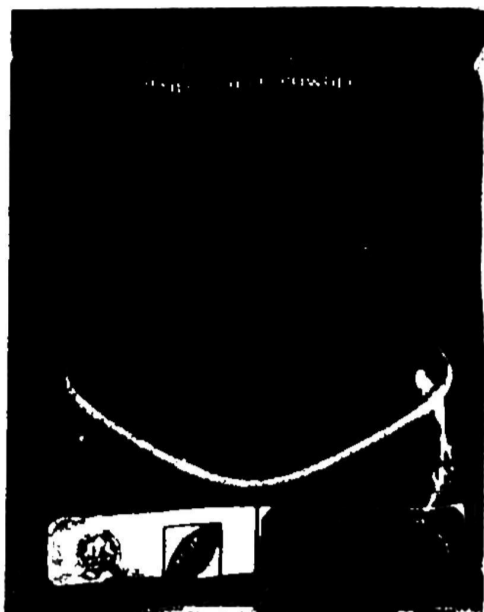


Figure 3.3 Chili powder

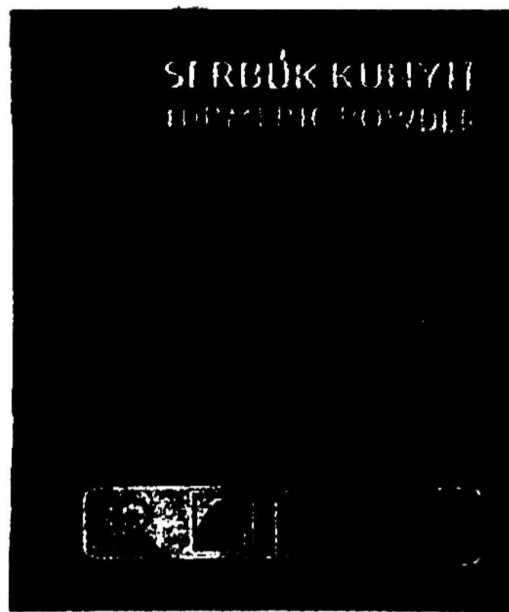


Figure 3. 4 Turmeric powder

### 3.1.1 Nutritional Composition

The nutritional composition for each spice used in this study was listed down in Table 3.1 below where the nutrient content was as labelled on each BABAS® packet.

Table 3.1 Nutritional composition of each spice (per 100 g)

Nutritional content	Cumin Powder	Coriander Powder	Chili Powder	Turmeric Powder
Fat (g)	25.4	21.8	13.5	2.7
Carbohydrate (g)	16.6	26.0	27.0	73.1
Protein (g)	15.5	21.1	10.4	6.3
Dietary fibre (g)	26.9	20.4	35.7	17.6
Calcium (mg)	892.0	606.0	0	0
Magnesium (mg)	281.0	212.0	203.0	0
Iron (mg)	11.2	8.1	25.0	16.0
Energy (kcal)	357.0	384.0	342.0	377.0

## 3.2 Powder Physical Properties

### 3.2.1 Particle Size

Each sample of cumin powder, coriander powder, chili powder, and turmeric powder was determined for its particle size distribution by using laser light diffraction equipment, Particle Size Analyzer (Malvern Mastersizer 2000, Worcestershire, U.K) as shown in Appendix A (Figure A.1). The instrument can analyze the median particle and size analysis. The particle size, as well as size distribution at  $d_{10}$ ,  $d_{50}$ , and  $d_{90}$ , were determined from the equipment. Each sample was tested in triplicate to obtain the average data.

The particle size of the population used must reflect the property or properties of the population, therefore median particle size or commonly known as  $d_{50}$  is often used since it is easily read from the cumulative distribution as the 50% size where the size which splits the distribution into two equal parts.

### 3.2.2 Bulk Density, $\rho_b$

Each sample was weighed approximately 2.5 g individually and put into a 10 ml glass measuring cylinder. Then, the volume of the powder sample was recorded without any preliminary tapping of the measuring cylinder. These were done in triplicate to each sample in order to obtain the average data. From the mass and volume data of the sample, the value of bulk density was then calculated by using the equation below :

$$\rho_b = \frac{m}{V} \quad (2.4)$$

Where  $\rho_b$  is bulk density,  $m$  is mass of powder sample, and  $V$  is the volume of powder sample inside the glass measuring cylinder without tapping.

### **3.2.3 Tapped Density, $\rho_t$**

The powder sample was weighed approximately 2 g and inserted into the sample chamber of 25.4 mm in diameter, then attached to the Envelop and Tap Density Analyzer equipment (Micromeritics GeoPyc 1360, USA) as shown in Appendix A (Figure A.2). The parameters set for tapped density were 3 cycles as the number of consolidations per analysis, and 51 N as the consolidation force to be applied according to the size of the sample chamber, before pressing the Run Key. Then, during the test, the sample chamber was rotated and agitated while the specified force was applied to the sample. The GeoPyc averaged the measurements from each consolidation and automatically calculated volume and density, and reported the results in  $\text{cm}^3$  and  $\text{g}/\text{cm}^3$ . Therefore, the average tapped density was obtained directly from the equipment for each spice.

### **3.2.4 True Density, $\rho_s$**

True density is a measurement of weight per unit volume of material excluding voids and inherent in the material tested. The powder sample was inserted into the medium size chamber ( $3.5 \text{ cm}^3$ ) gas pycnometer and the weight was recorded. The true density was obtained by using the Gas Pycnometer equipment (AccuPyc II 340; Pycnometer Micromeritics, USA) as shown in Appendix A (Figure A.3).

### **3.2.5 Hausner Ratio**

The bulk and tapped density obtained previously were used to calculate the value of Hausner Ratio using the equation below (Hausner, 1967) in order to determine the cohesiveness, compressibility, and flowability of the powders.

$$HR = \frac{\rho_t}{\rho_b} \quad (2.1)$$

Where  $HR$  is Hausner Ratio,  $\rho_t$  is tapped density and  $\rho_b$  is bulk density.

### 3.2.6 Carr Index

Carr Index or also known as Compressibility Index where it refers to the determination of the compressibility and flowability of powders. The bulk and tapped density obtained previously were also used in the Carr Index formula as shown in the equation below (Carr, 1965) in order to determine the flow characteristics of the powders.

$$CI = \frac{\rho_t - \rho_b}{\rho_t} \times 100\% \quad (2.2)$$

Where  $CI$  is Carr Index,  $\rho_t$  is tapped density and  $\rho_b$  is bulk density.

### 3.2.7 Porosity

The porosity of the powders was calculated from the bulk density and true density of the powders. It was calculated using the following equation (Abrahansen and Geldart, 1980; Boukouvalas et al., 2006) :

$$\varepsilon = 1 - \frac{\rho_b}{\rho_s} \times 100\% \quad (2.5)$$

Where  $\varepsilon$  is the porosity,  $\rho_b$  is the bulk density of the powder and  $\rho_s$  is the true density of the powder.

### 3.2.8 Moisture Content

Moisture content (wet basis) was measured by weighing 1 g of each sample before and after drying in an oven (Memmert, Germany) as shown in Appendix A (Figure A.4) at 105°C for 24 hours (Teunou et al., 1999; Fitzpatrick et al., 2004;

Mohd Salleh et al., 2014). The weight of plates and powder samples after drying were recorded and used to calculate the moisture content (wet basis) of each powder. Each test was carried out in triplicate. The percentage of the wet basis moisture content ( $M_{c,w}$ ) was calculated by using the equation below:

$$M_{c,w} = \frac{W_{initial} - W_{final}}{W_{initial}} \times 100\% \quad (2.6)$$

Where  $M_{c,w}$  is the percentage of moisture content of the sample,  $W_{initial}$  is the weight of the sample before drying and  $W_{final}$  is the weight of the sample after drying.

### 3.3 Compressibility

The compressibility of the powdered spices was carried out using the uniaxial die compaction method. A 0.5 g sample was poured into a cylindrical uniaxial die of 13 mm in diameter. The die set comprised of upper punch, lower punch, base, 13 mm diameter stainless steel die, and a holder. A universal testing machine (Instron Universal Testing Machine-5566, Canton MA, UK) was used to compress the powder samples into tablet form. The Bluehill software (Canton MA, USA) was the operating software of that particular machine, which helped to maintain a compaction speed of 0.1 mm/min at four different force settings of 4 kN, 6 kN, 8 kN, and 9 kN (Saifullah et al., 2016). After reaching the maximum force set for tablet formation, the tablet was ejected from the die and the thickness of the tablet was measured using a vernier caliper to calculate the compact's volume using Equation 2.7 and density of the powder in tablet form using Equation 2.4 in order to evaluate the compressibility and flowability of powders.

$$V = \pi r^2 h \quad (2.7)$$

Where  $V$  is the compact's volume,  $r$  is the radius of the tablet, and  $h$  is the thickness of the tablet.

### 3.4 Powder Flow Properties

#### 3.4.1 Caking Strength

The caking strength test is carried out to measure the potential of powder to form a cake and the force required to cut through the cake formed. The powder sample was inserted into the standard vessel approximately 180 ml which weighed around 70 g to 100 g of the sample. The test was done by using a texture analyzer (TA.XTplus Texture Analyzer Stable Micro Systems, London, U.K) as shown in Figure 3.5 and Appendix A (Figure A.5) where the standard vessel which contain the sample will be attached to the platform of the equipment and then run the test. During the test, the blade of the probe levels the top of the powder column and measures its height. The blade then moves down through the column and compacts the powder to a pre-defined force. When it has reached this force, the height of the cake was measured and the blade sliced up through the powder. This compaction cycle was repeated four more times. At the 5<sup>th</sup> time where the target force has reached, the blade sliced through the compacted cake of powder formed at the bottom of the vessel. Finally, the result of the cake and mean cake strength were obtained. Each sample was duplicated in order to obtain the average data.

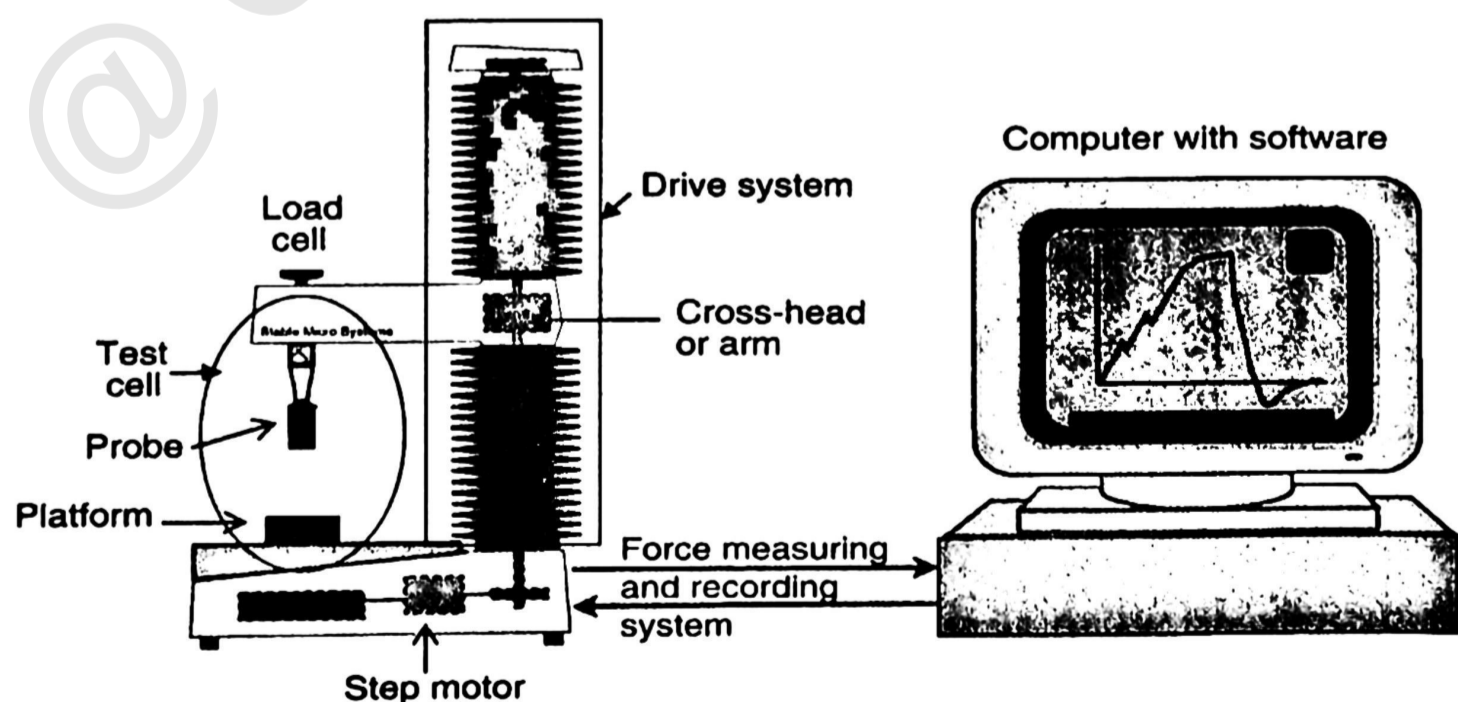


Figure 3.5 A schematic diagram of a texture analyzer (Rolle et al., 2011)

### **3.4.2 Cohesion Test**

The same equipment and procedure of the caking test was done in cohesion test as well but instead of caking test, cohesion test was chosen in the option before running the test. During the test, the powder blade moves down through the powder column using a cutting action to minimize compaction. The upward part of the cycle then lifts the powder and the force of the powder on the vessel base was recorded. A more cohesive powder will cling to itself and to the blade, therefore, reducing the force exerted on the base of the vessel. The result from the cohesion test was cohesion index where it can be used to evaluate the cohesiveness and flowability of powders. This quick analysis allows repeatable quantification of the cohesiveness of a bulk solid. Test results can be used to compare the sample being tested with the previously analyzed product, to assess if it is more cohesive or more free-flowing (Stable Micro Systems, 2016). Cohesiveness is the tendency for particles of powder to cling together and agglomerate (form larger clusters of particles). In other words, cohesion is the particles-particles interaction of the powder during the test.

### **3.4.3 Effective Angle of Internal Friction**

In order to evaluate the inter-particle friction of spices powder, the flow function test is carried out by using Jenike shear cell techniques as shown in Figure 2.6 in Section 2.5 from a powder flow tester (Brookfield, USA) as shown in Appendix A (Figure A.6). The Powder Flow Tester (PFT) comes with the trough, outer and inner catch tray, shaping blade and lid. The outer and inner catch tray was placed into position on the trough, and this will lock into place when properly seated. Next, the powder sample was placed in the trough whose annular shape by using the powder scoop which comes with the instrument and flattened with an

inner catch tray. Any spillage was conveniently captured by the inner and outer catch trays. When sufficient sample has been placed in the trough, the shaping blade with a curved profile was used and rotated on the inner catch tray to distribute the sample evenly around the trough as shown in Figure 3.6 below. It was rotated in a clockwise direction until the sample is completely distributed. Then, it needs to be rotated in a counter-clockwise direction to remove the excess sample.

After that, both catch trays were removed and the initial weight of trough and the weight after sample addition were recorded. Next, the sample trough was installed onto the PFT turntable. The outer catch tray can be installed on the trough as well to catch any spillage during the test. After that, the appropriate lid which is the vane lid was attached to the instrument and Standard Flow Function Test was set to run the experiment. During the test, the vane lid moved down to cover the trough. The trough then rotated while powder consolidated/compressed as the vane lid has 18 small compartments which trap the powder particles and cause them to shear against the powder particles in the trough. The trough was rotated until steady flow reached and then the shear stress and normal stress were recorded. Data on normal stress and shear stress were recorded for each consolidation stress. Besides, the standard flow function test data shows one curve based on the effective angle of internal friction for each consolidation setpoint and reports the effective angle of internal friction for the critical arching stress and critical rat-hole stress. The results were collected and recorded.

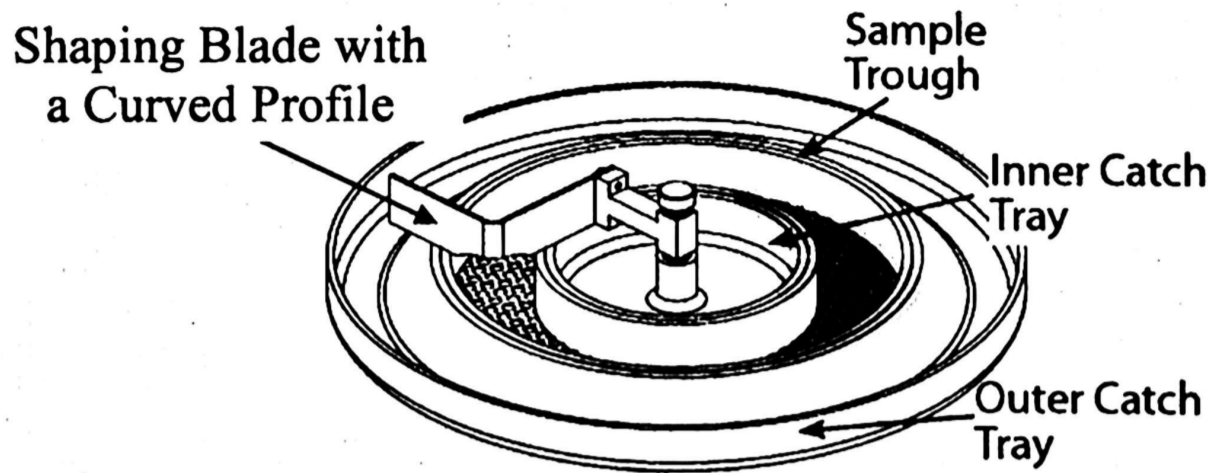


Figure 3.6 Inner catch tray with shaping blade inserted into the annular opening of the sample trough

#### 3.4.4 Angle of Wall Friction

The technique and procedure for standard wall friction test was the same as the flow function test used to measure the effective angle of internal friction in Section 3.4.3. However, the shaping blade and lid used in this test was not the same as flow function test where they need to be changed into the shaping blade with a flat profile and wall friction lid. The wall friction test was performed by using a powder flow tester (Brookfield, USA) as well. Then, the Standard Wall Friction Test was set to run the experiment.

The wall friction lid will descend to a position slightly above the sample in the trough and the instrument will perform an autozero. Then, the lid will make contact with the sample and the test begins. The standard wall friction lid has a smooth bottom surface which act as the weights and slides over the powder particles in the trough during the test as shown in Figure 3.7, thereby measuring the friction of powder against the surface material of the lid. The results were collected and recorded. A wall friction test will be able to give an approximate assessment of whether a given hopper geometry will support mass-flow. For an exact calculation

of the hopper half angle and minimum outlet diameter of the hopper, both wall friction and flow function tests must be conducted.

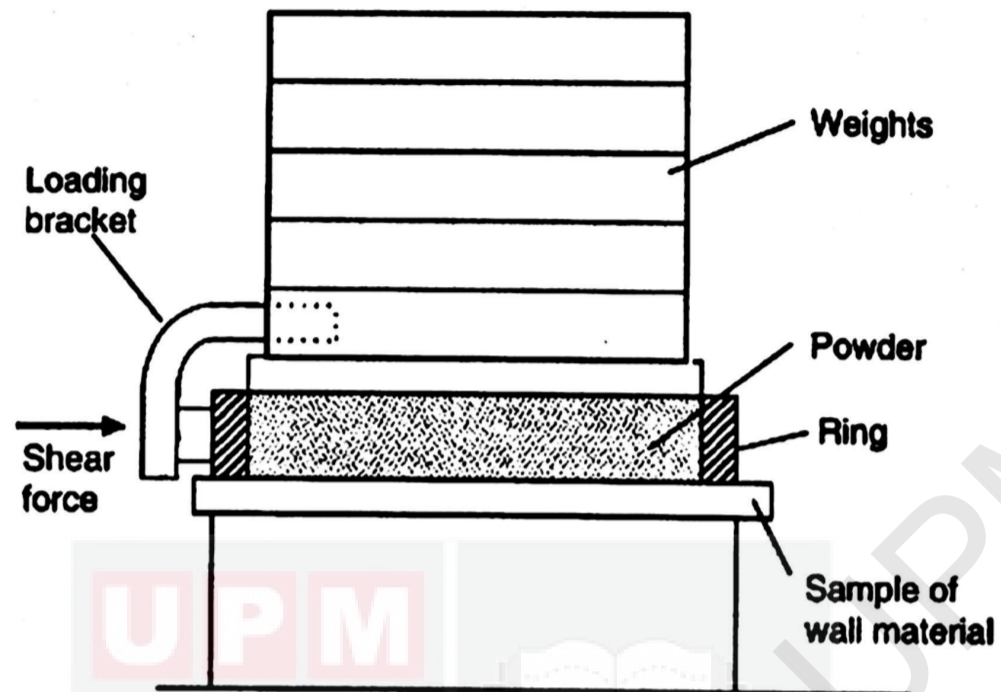


Figure 3.7 Apparatus for the measurement of angle of wall friction,  $\phi_w$  (Rhodes, 2008)

## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### **4.1 Physical and Flow Properties of Spice Powders**

Design, optimization, and performance of spice powder products are influenced by the physical and flow properties of the spice powders. The physical properties are particle size, bulk density, tapped density, true density, and moisture content whereas the flow properties are Hausner's ratio, Carr Index and flowability as shown in Table 4.1.

Table 4.1 Physical properties of powders

Powder Properties	Chili Powder	Coriander Powder	Cumin Powder	Turmeric Powder
Median particle size, $d_{50}$ ( $\mu\text{m}$ )	114.3 $\pm$ 2.1	225.0 $\pm$ 2.2	288.6 $\pm$ 3.7	81.0 $\pm$ 1.1
Bulk density, $\rho_b$ ( $\text{kg}/\text{m}^3$ )	289.6 $\pm$ 7.0	258.3 $\pm$ 2.7	395.1 $\pm$ 6.2	404.6 $\pm$ 19.9
Tapped density, $\rho_t$ ( $\text{kg}/\text{m}^3$ )	761.6 $\pm$ 6.3	668.6 $\pm$ 4.6	768.8 $\pm$ 7.3	825.4 $\pm$ 6.7
True density, $\rho_s$ ( $\text{kg}/\text{m}^3$ )	1361.9 $\pm$ 0.5	1306.8 $\pm$ 1.0	1313.9 $\pm$ 0.4	1520.1 $\pm$ 0.2
Bulk porosity, $\varepsilon$ (%)	79.0	80.0	70.0	73.0
Hausner Ratio, $HR$ (Hausner, 1967)	2.6	2.6	1.9	2.0
Carr Index, $CI$ (%) (Carr, 1965)	62.0	61.4	48.6	51.0
Flowability (Lebrun et al., 2012)	Very, very poor	Very, very poor	Very, very poor	Very, very poor
Moisture content, $M_{c,w}$ (wet basis) (%)	12.1 $\pm$ 1.0	10.7 $\pm$ 5.3	8.4 $\pm$ 1.2	11.9 $\pm$ 0.3
Moisture content, $M_{c,d}$ (dry basis) (%)	13.8 $\pm$ 1.2	12.2 $\pm$ 6.8	9.2 $\pm$ 1.6	13.5 $\pm$ 0.4

#### 4.1.1 Particle Size

Table 4.1 shows the particle size distribution of each spice where it can be seen that the median particle size,  $d_{50}$  of turmeric powder was the finest, followed by chili powder, coriander powder and cumin powder. This is because as reported by Ortega-Rivas (2012) for the approximate ranges of the median sizes of some common food powders along with the terms recommended by the British Pharmacopoeia for use with powders, when the median particle sizes are in the range of 210-300  $\mu\text{m}$  and 53-75  $\mu\text{m}$ , the range for British standard meshes is 52-72

and 200-300, respectively. Thus the powder particles are categorized as moderately fine and fine, respectively.

Particle size distribution is directly related to the behavior of the material and the physical properties of products. Also, some previous researches done by Fitzpatrick, Barringer, and Iqbal (2004); Katikaneni, Upadrasha, Rowlings, Neau, and Hileman (1995); and Teunou et al. (1999) showed that when the particle size decreased, the powder is known as a cohesive powder because it favors a greater number of contact points for interparticulate bonding and additional interactions where it tends to increase cohesion behavior as the particle surface area per unit mass increases, thus, resulting in more cohesive and less free-flowing powders. Marinelli and Carson (1992) also stated that the finer the particle size, the more contact area between particles, which leads to greater cohesive forces among the particles. Apart from that, bulk density, compressibility, and flowability of a food powder are highly dependent on the particle size and its distribution (Barbosa-Canovas et al., 1987).

#### **4.1.2 Densities**

As shown in Table 4.1, the values for bulk density, tapped density, as well as true density of turmeric powder was the highest compared to others and it was the lowest for coriander powder. This happened due to fine particle size occupied less volume in a bulk, thus, higher bulk density was obtained. In addition, the bulk density increased when the particle decreased, thereby will produce a powder with good flowability (Barbosa-Canovas et al., 1987). However, Xiu et al (2008); Yusof et al (2009); Yusof et al (2010); and Saifullah et al (2016) have reported that the higher the tapped density, the higher the Hausner ratio and Carr index, thus resulting in a cohesive and very poor flowability of the powder. Therefore,

according to result from densities value, chili powder was cohesive and more-difficult flowing powder compared to others.

As for true density for each spice, the results were in agreement to classification on true density of common food powders classified by Ibarz and Barbosa-Canovas (2014) as well as having the same trends as the previous works by Fitzpatrick et al (2004); Yusof et al (2009); Yusof et al (2010); Mohd Salleh et al (2013); and Saifullah et al (2016), where the true densities obtain for each spice were in the ranges of 1000-1500 kg/m<sup>3</sup>.

#### **4.1.3 Porosity**

As shown in Table 4.1, the bulk porosity of coriander powder was the highest while turmeric powder was the lowest. This happened due to the true density obtained from each spice, where the lower the true density of spices, the higher the porosity. Therefore, coriander powder will possess difficult-flowing properties due to high porosity obtained. In addition, as reported by Boukouvalas et al (2006), apart from true density, bulk porosity can vary considerably due to mechanical compaction, the difference in particle sizes as well as moisture content.

#### **4.1.4 Moisture Content**

According to Table 4.1, the moisture content of the samples varies from one sample to another. For both wet and dry basis, chili powder has the highest moisture content while the lowest moisture content was cumin powder. This was probably because chili powder was high in hygroscopicity compared to others. Moisture content always related to hygroscopicity of the material where hygroscopicity is the tendency of a solid substance to absorb moisture from the surrounding atmosphere. Therefore, the hygroscopicity of powder is one of the crucial causes that lead to

high the moisture content in any powder product where it promoted powders to absorb moisture easily from the air (FMC Biopolymer, 2009).

#### **4.1.5 Flowability of Spice Powders**

By referring to Table 4.1, even though turmeric powder was the smallest particle size compared to others, however, chili powder that was the second smallest particle size has the highest Hausner ratio along with its Carr index, whereas the lowest was cumin powder. Even though particle size may significantly affect flowability, some other factors such as bulk density, tapped density, and moisture content can affect the Hausner ratio and Carr index value of each spice thus has a significant impact on its flowability too where the lower the bulk density and the higher the moisture content, the higher the Hausner ratio. According to Scoville and Peleg (1981); Fitzpatrick et al (2004); and Jan et al (2016), high moisture content leads to reduced flowability or resist powder flow since the liquid bridges and cohesive forces acting between the powder particles are increasing and will lead to the interaction which results in powder caking and cohesive flow. As for Carr index, a higher Carr index will be obtained when the difference of tapped density and bulk density were higher along with its lower value of tapped density. Since chili powder has satisfied all the characteristics above, thus, the highest Hausner ratio and Carr index were obtained from it. Hence, chili powder has a very, very poor flowability followed by coriander, turmeric and cumin powder.

Apart from that, according to the flowability classification that has been classified by Lebrun et al. (2012), the spice powders used in this research have a very, very poor flowability since their Hausner ratio and Carr index are higher than 1.60 and 38%, respectively. Hausner ratio and Carr index are measured in order to determine the flowability of powdered materials where the flowability was usually

depending on the moisture content as well as the physical properties of powder which is the particle size, bulk density, and tapped density. According to Jan et al. (2018), smaller particle size will reduce the flowability of the powder. Hence, flowability problems can occur such as failure of powder to be discharged consistently from the hopper.

#### 4.2 Compressibility of Spice Powders

Hence, the applied pressure-volume relationship has been plotted as shown in Figure 4.1, where it can be seen clearly that the higher the applied pressure, the lower the volume of each spice tablets because the thickness of the tablets was reduced after compression. It has been measured that chili tablet has the highest thickness which leads to the highest volume of the compact compared to others, while turmeric tablet has the lowest thickness and volume of the compact. Thus, the lower the compact's volume, the higher the density of the tablet and higher density obtained after compression indicate better compressibility, where the results obtained are comparable to the result found by Ooi (2008) and Yusof et al (2010) of *Ficus deltoidea* (*F. deltoidea*) and *Andrographis paniculata* (*A. paniculata*) powders into tablet, respectively. The result from their researches showed that *A. paniculata* was more difficult to compress compared to *F. deltoidea* since the obtained density of *A. paniculata* after compression was lower than *F. deltoidea*.

According to Yusof et al (2009), the variation of the compact's volume was related to the flowability characteristic of the powders, which may be classified from the Hausner Ratio and Carr Index that has been presented in Table 4.1. Even though both chili and turmeric powder are classified as very, very poor flowability, however, turmeric powder has lower Hausner ratio and Carr Index's value than chili powder. Therefore, better compressibility is determined by the lower the compact's

volume as well as the lower the Hausner ratio and Carr Index values. In other words, these values will lead to difficult flow of the powder where flowability is directly related to cohesiveness, in which the flowability of powder will increase when the cohesiveness decrease (Teunou et al., 1999). But since turmeric powder has low fat content, therefore the flowability of turmeric powder was better than chili powder.

Therefore, the compressibility is used to evaluate and predict the behavior of powder materials whether they are free-flowing or cohesive. This evaluation is done by the measurement of bulk property that determines the volume change of a conditioned powder when it is slowly compressed into a tablet form (Jan et al., 2018). Besides, previous research done by Yusof et al (2009) has stated that the main characteristics to be investigated by uniaxial die compaction are presented in the form of applied pressure-volume relationship.

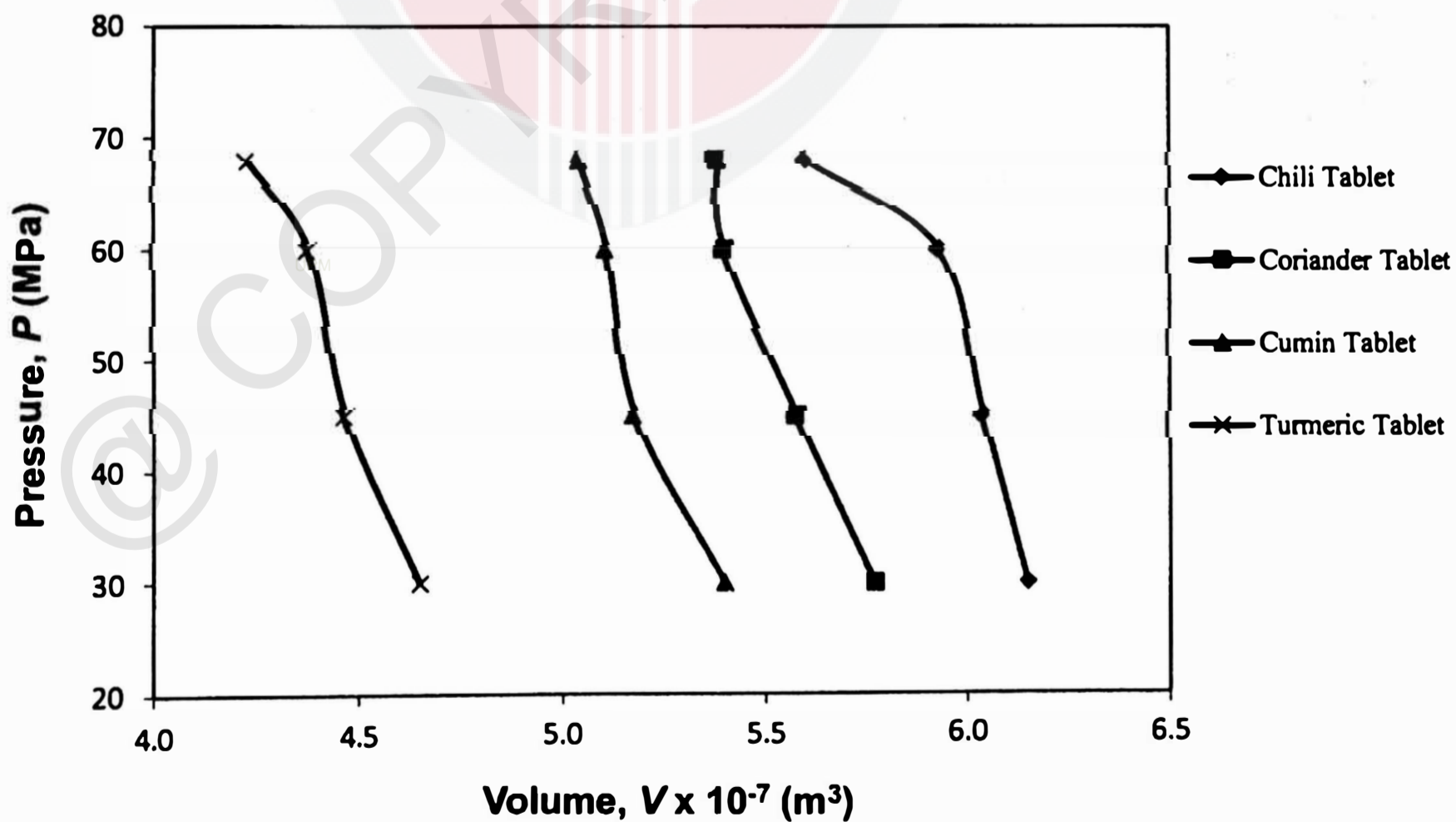


Figure 4.1 Applied pressure-volume relationship of powders

## **4.3 Flow Measurements**

### **4.3.1 Cake Strength of Spice Powders**

The result of cake strength of each spice powders used in this research is shown in Figure 4.2. By referring to Figure 4.2, it can be seen that the highest cake strength was coriander powder followed by cumin, chili, and finally, the lowest was turmeric powder. Theoretically, cake strength can be influenced by particle size, moisture content, and particle to particle interaction which make them stick together and form a lump. However, the results showed that even though chili and turmeric powder have lower particle size compared to coriander and cumin powder, the powders which are lower in particle size produced lower cake strength. Augestein and Hogg (1978); Teunou et al (1999) and Fitzpatrick et al (2004) have reported that the smaller the particle size, the more cohesive the powder becomes, thereby making the powder to increase in cake strength and becomes difficult to flow.

The obtained result is opposite to what has been stated by Fitzpatrick et al (2004) and this probably happened due to the nutritional value of cumin, coriander and chili powder which contain high-fat content compared to turmeric powder as shown in Table 3.1 in Section 3.1.1. The fat content will affect the stickiness and agglomeration of particles, thus will influence the particle size distribution, bulk density, and flowability of the powder (Augestein and Hogg, 1978; Teunou et al., 1999; Fitzpatrick et al., 2004; Nijdam and Langrish, 2006).

Hence, cake strength of any powder is important in order to know whether the powder has high tendency to form a cake or caking that can lead to the formation of lump or agglomerate of powder especially during storage inside its containers, silos or hoppers at some point in the production process. Once the

powder has formed a lump inside a hopper, it tends to reduce the flowability of powder during the discharge operation. The tendency of a powder to cake is closely related to its cohesiveness and generally, a powder that is cohesive will also form a cake during the caking test.

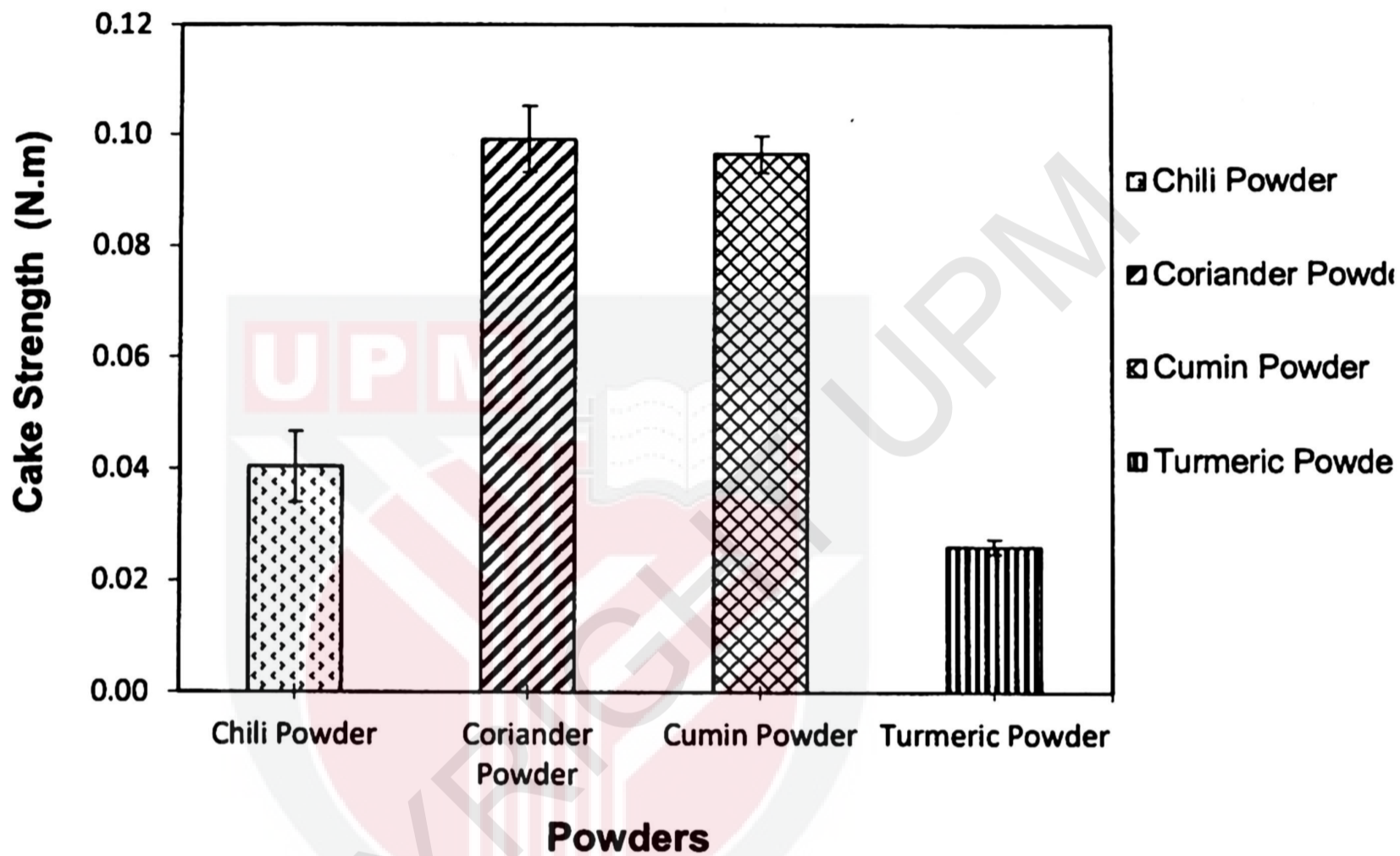


Figure 4.2 Cake strength of powders

#### 4.3.2 Mean Cake Strength of Spice Powders

Mean cake strength is defined as a mean of evaluation of proneness to caking, and are shown in Figure 4.3. It can be seen that coriander powder has the highest mean cake strength while the mean cake strength for turmeric powder was the lowest. This is due to the relation on cake strength of powders where the higher the cake strength, the mean cake strength will be higher too where fat content has a significant effect on the results. Thus, mean cake strength is directly proportional to cake strength since Benkovic and Bauman (2009) reported that a powder that has a high tendency to cake is likely to have a high cake and mean cake strength.

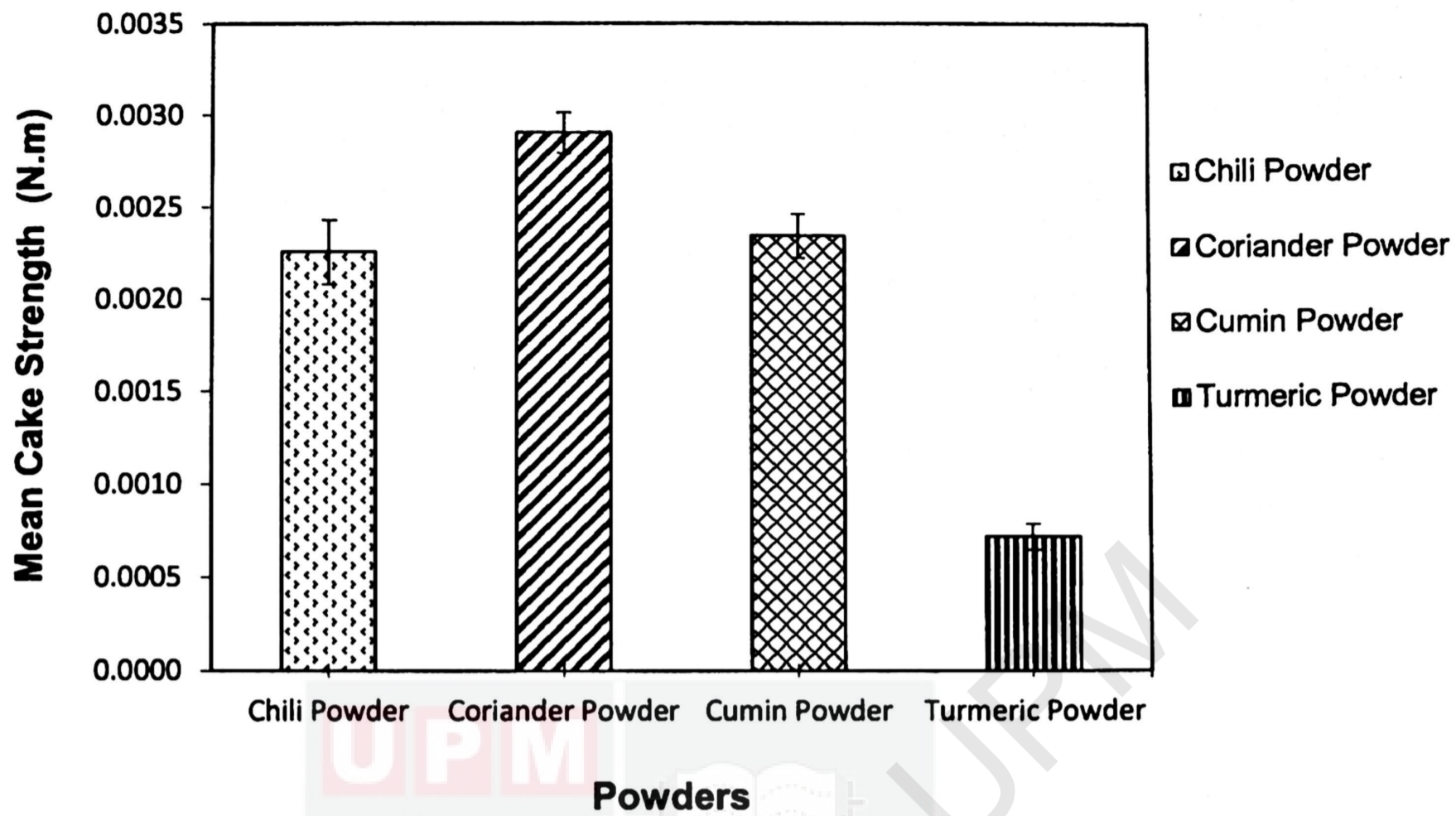


Figure 4.3 Mean cake strength of powders

#### 4.3.3 Cohesion Index of Spice Powders

The test results from the cohesion test were cohesion index as shown in Figure 4.4, which used to assess if the powder is more cohesive or more free-flowing. According to Figure 4.4, the cohesion index for turmeric powder was slightly higher than cumin powder whereas chili powder has the lowest cohesion index among other spices. Landillon et al (2008) reported that low cohesion index represents a non-cohesive free-flowing powder while high cohesion index indicates a cohesive poor flowing powder. However, by referring to powder categorization scale based on cohesion index (Table 2.1 in Section 2.4) that has been classified by Benkovic and Bauman (2009), the flow behavior for all spice powders used in this research was categorized as hardened and extremely cohesive flowing powders since the values of cohesion index were all has exceeded 19.

This cohesive nature of the powder based on cohesion index categorization scale was in line with the powder properties shown in Table 4.1 where the Hausner ratio and Carr index values for all spice powders were all higher than 1.6 and 38% respectively, thus have proved that the powders to be very difficult to flow. The difficult flow nature of many powders was because of smallness in powders particle sizes (Fitzpatrick et al., 2004) and cohesion was expected to increase with low fat content (Fitzpatrick et al., 2007), hence that is the reason for turmeric powder to result in high cohesion index since it has the smallest particle size and lowest fat content compared to others.

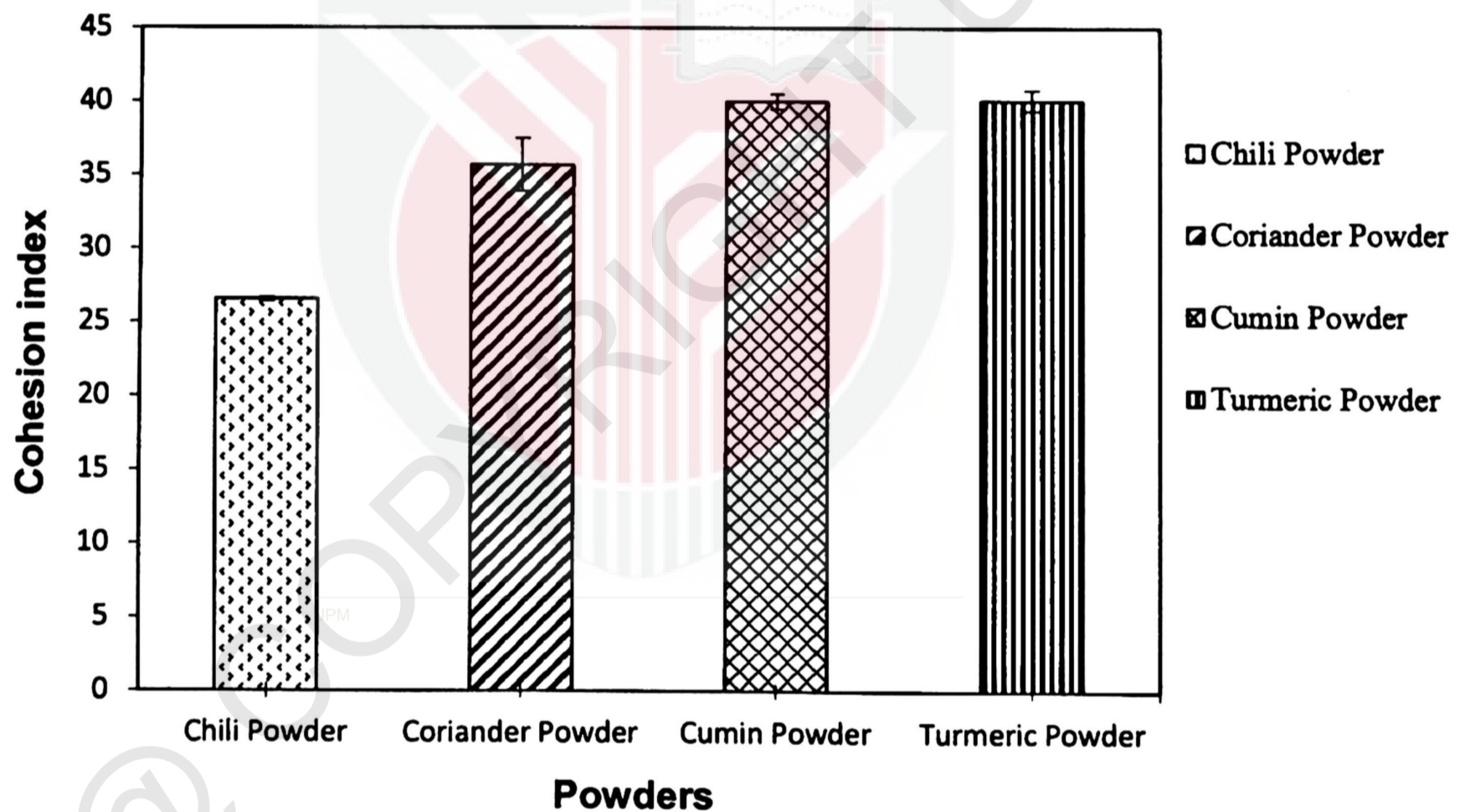


Figure 4.4 Cohesion index of powders

#### **4.3.4 Powder Flow Speed Dependency (PFSD) of Spice Powders**

According to Figure 4.5, the flow stability index of chili powder was slightly closer to 1 while turmeric powder was slightly greater than 1. However, all spice powders used in this research were considered as stable as the values of stability index are reaching to 1 due to the powder did not significantly alter during the test. Etti et al (2014) have stated that once the flow stability index value is less than or greater than 1, it indicates that the powder has undergone changes during the test while if the flow stability index value near to 1 means that the powder did not significantly alter while the test was in progress. These changes may be due to attrition of the powder particles themselves or the breaking down of agglomerates Benkovic and Bauman (2009).

PFSD test quantifies the resistance of a powder sample as the measured flow is imposed at a different speed. As shown in Appendix B (Figure B.6 until Figure B.9), cumin and turmeric powder showed an increased in compaction coefficient when the test flow speed increased, and this indicates an increase resistance to flow by the cumin and turmeric powder which means that these powders are flow speed dependent. However, chili and coriander powder showed a contrast result from cumin and turmeric powder. This is because there was a slight decreased in compaction coefficient after 20 mm s<sup>-1</sup> of test flow speed. When the compaction coefficient decreased with increasing flow speed, this indicates that the powders have become to be more free flowing (Etti et al (2014)). Therefore, this is the reason why the flow stability index of cumin and turmeric powder were slightly lower and higher than 1, respectively since both have increased in resistance to flow.

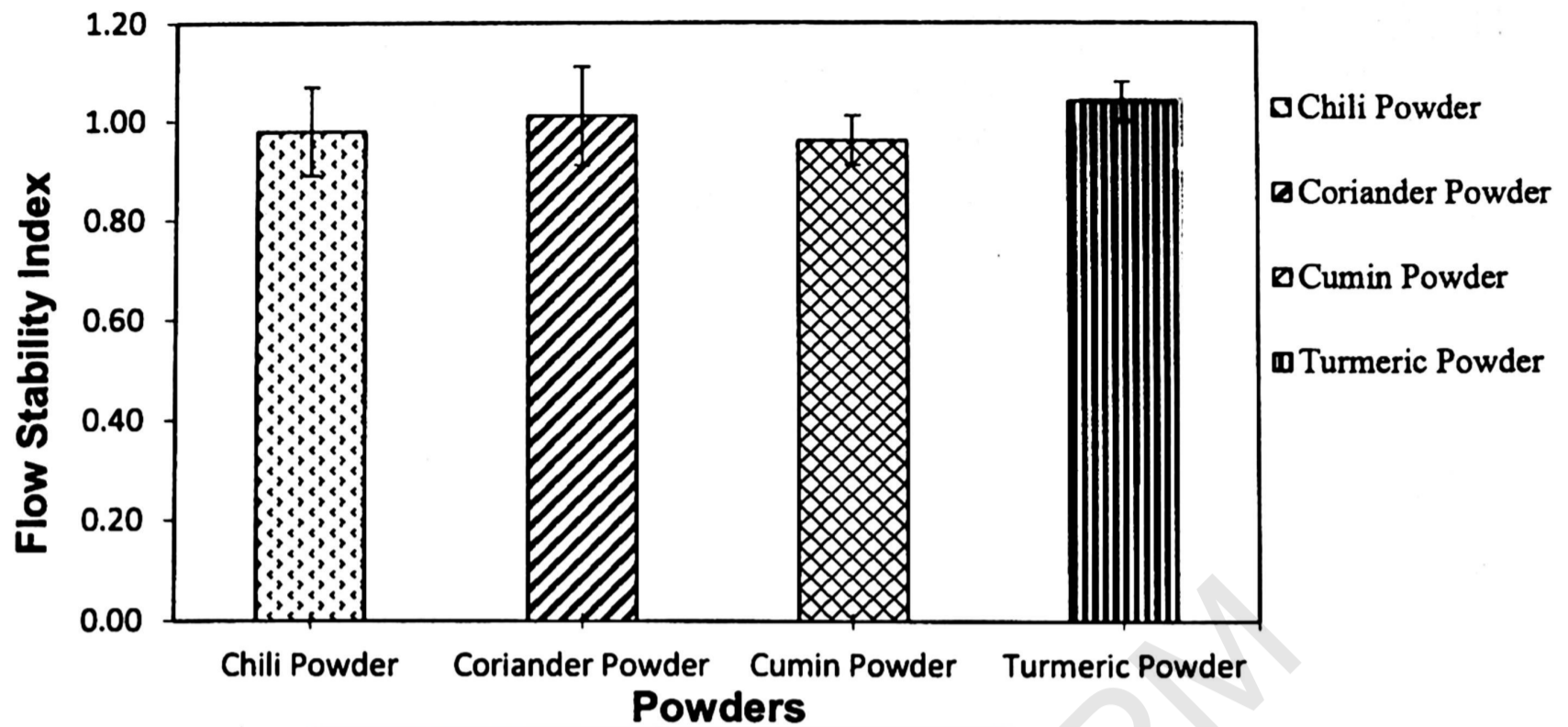


Figure 4.5 Powder flow speed dependency (PFSD) of powders

#### 4.3.5 Flow Properties Measurement by Shear Cell Tests

The properties measured by the shear cell test can be used directly in the design of hoppers using the well known Jenike design method. The results on evaluation for a cylindrical hopper geometry (Figure 2.8 and Figure 2.9 in Section 2.5) using Jenike's method were presented in Figure 4.6 and Table 4.2 where it includes the flow function graph and values of flow index, flow factor, effective angle of internal friction, angle of wall friction, hopper angle, bulk density from shear cell test, and the minimum outlet diameter of hopper.

Table 4.2 Evaluation for a cylindrical hopper using Jenike's method

Powders	Flow index	Flow factor, $ff$	Effective angle of internal friction, $\delta$ ( $^{\circ}$ )	Angle of wall friction, $\phi_w$ ( $^{\circ}$ )	Hopper half angle, $\theta$ ( $^{\circ}$ )	Bulk density from shear cell, $\rho_b$ ( $\text{kg/m}^3$ )	Minimum outlet diameter, $B$ (m)
Chili Powder	$0.56 \pm 0.02$	$1.21 \pm 0.26$	55.7	30.5	14.0	$432.20 \pm 2.12$	0.26
Coriander Powder	$0.44 \pm 0.01$	$1.51 \pm 0.40$	53.1	22.0	19.0	$377.85 \pm 2.90$	0.37
Cumin Powder	$0.45 \pm 0.03$	$1.47 \pm 0.38$	51.8	21.0	25.5	$461.95 \pm 2.12$	0.22
Turmeric Powder	$0.32 \pm 0.00$	$1.84 \pm 0.67$	49.0	30.5	14.0	$513.40 \pm 10.75$	0.16

The measured flow functions of each spice were presented in Figure 4.6 where it showed that only chili powder was classed into the very cohesive region while the others were classified into the cohesive region since the flow index of chili powder was the highest among others, thus making the flow factor to be the lowest. This is because when the major consolidating stress increased, the unconfined yield strength of chili powder was higher than the other powders, thereby the flow function of chili powder to be steeper and falls into very cohesive region since it was related to low fat content, small particle size, high Hausner ratio and Carr index as well as high flow index and lower flow factor as shown in Table 3.1 (Section 3.1.1), Table 4.1 (Section 4.1) and Table 4.2 (Section 4.3.5), respectively. As a result, the steeper the flow function line, the more difficult it is for the powder to flow, as the graph represents the strength that develops within the powder when it consolidates (Mohd Salleh et al, 2014; Fitzpatrick et al. 2004).

Besides, cumin and coriander powder have similar flow functions since their flow index as shown in Table 4.2 were kind of similar, thereby both were categorized as cohesive powders. It can be seen that at first, both cumin and coriander powder behave as a very cohesive but as the major consolidating stress increased, both flow function lines of cumin and coriander powder showed a better flowability were from very cohesive, they changed into a cohesive flow. This can be explained by the fat content in cumin and coriander powder as shown in Table 3.1 in Section 3.1.1 since Fitzpatrick et al (2007) reported that fat content has a significant effect on powder cohesion. Even though cumin powder has a much greater fat content than coriander powder, however, its cohesiveness was similar. This may be explained by the fact that it is the composition at the surface of the powder particles that led to cohesiveness of the coriander powder and make it to have a similar flow function with cumin powder where the results obtained were and in agreement with work presented by Fitzpatrick et al (2007) research on whole-milk powder (WMP) and high fat powder (HFP).

The same goes for turmeric powder where it can be seen that the trend line of its flow function was the same as cumin and coriander powder, and it also was classified into the cohesive region. However, the flow function line was lower than cumin and coriander powder since the slope of the line of turmeric powder flow function was lower and getting near to the easy flowing region and this might be because of the lowest fat content in the turmeric powder compared to others as shown in Table 3.1 in Section 3.1.1. Apart from that, Nurhadi and Roos (2017) reported that the lower the slope, the more free-flowing properties of the powder obtained.

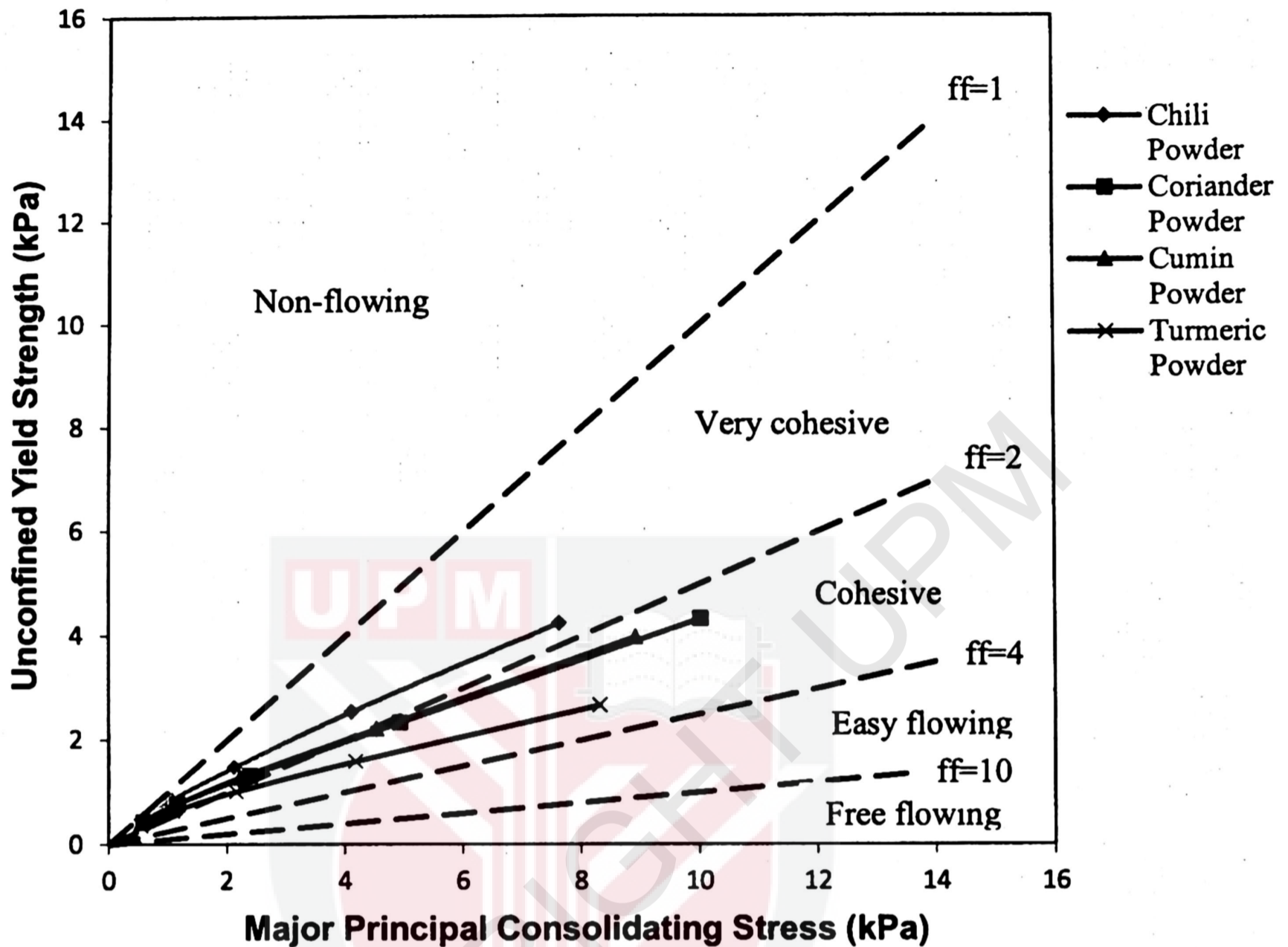


Figure 4.6 Flow function of spice powders

According to Table 4.2, the highest effective angle of internal friction was chili powder while turmeric powder has the lowest effective angle of internal friction. This is due to the relation on the flow function of spice powders shown in Figure 4.6 where turmeric has the lowest flow function line where it almost near to the easy flowing region. Besides, according to Appendix B (Figure B.1), it can be seen that the lower the major principal consolidating stress, the higher the effective angle of internal friction. As reported by Barbosa-muriet Canovas et al (2005); Slettengren et al (2016); and Nurhadi and Roos (2017), the lower the value of effective angle of internal friction, the lower the particle-particle interactions that can lead to cohesiveness, thus the more free-flowing properties the powder has.

As for flow factor values shown in Table 4.2, turmeric powder has the highest flow factor while the lowest was chili powder. This showed that the higher the flow factor, the better the flowability of spice powders when we relate it to the flow function of spice powders shown in Figure 4.6. However, when referring to the Jenike standard classification of powder flowability that has been classified by Jenike (1960) as shown in Table 2.3 in Section 2.5 and also was reported on Barbosa-muriet Canovas et al (2005); and Nurhadi and Roos (2017) works, the flow factor of all spice powders were categorized as very cohesive flow. But still, this was in agreement with the flow function graph where it can be seen that at the starting of each flow function line, they were all in the very cohesive region. Flow factor was the inverse of the slope of the best fit linear line between major consolidating stress and unconfined yield strength. Thus, as discussed earlier, the flow factor was higher when the slope was lower, thereby the more free-flowing properties of the powder exist (Nurhadi and Roos, 2017).

As shown in Table 4.2, both chili and turmeric powder has the same highest angle of wall friction while cumin powder was the lowest. This is due to the particle size of the powders where the smaller the particle size, the higher the friction occurred between the particles and the wall of the hopper, thus the flowability will be reduced. These results was in agreement with Mohd Salleh et al (2014) research which found that *F. deltoidea* had the highest friction compared to others and lesser flowability upon contact with stainless steel wall surface by applying the Jenike shear test experiment. Apart from that, as the normal stresses were reduced during the wall friction test, the higher the angle of wall friction will be obtained as shown in Appendix B (Figure B.5). Therefore, the smaller the particle size, the larger or greater its surface area, providing a higher adhesive force that reduces the

flowability of the powders on the wall surface. In addition, as the angle of wall friction increases, the more difficult it is to move the powder along the surface of the hopper or silo walls (Fitzpatrick et al. 2004) and so the hopper walls must be steeper for the powder to flow. Steeper hopper walls mean that the hopper will need a smaller hopper half angles in order to encourage mass as oppose to funnel flow (Freeman, 2009). This wall friction test evaluated on how easy the powder will flow over the inner surface of the container or hopper.

Hopper half angle for each spice was shown in Table 4.2 where it showed that chili and turmeric powders have the lowest hopper half angle while cumin powder was the highest. This is because hopper half angle was related to the angle of internal friction as mentioned previously. Thus, the higher the angle of wall friction, the lower the hopper half angle in order to encourage flowability inside the hopper. These results were similar to previous research done by Teunou et al (1999) and James et al (2015).

From Table 4.2, the minimum outlet diameter for each spice has been obtained as well. The minimum outlet diameter for coriander powder was larger than others while turmeric powder was the smallest. This is because the minimum outlet diameter was affected by the values of critical stress, bulk density from shear cell test and function of hopper angle. Hence, the higher the critical stress and function of hopper angle with low values of bulk density, the larger the minimum outlet diameter will be obtained. This explained why coriander powder has the largest value of minimum outlet diameter compared to others. The critical stress for each spice was obtained from the intersection of the flow function line to the flow factor line as shown in Figure 4.7 below and Appendix B (Figure B.2 until Figure B.4).

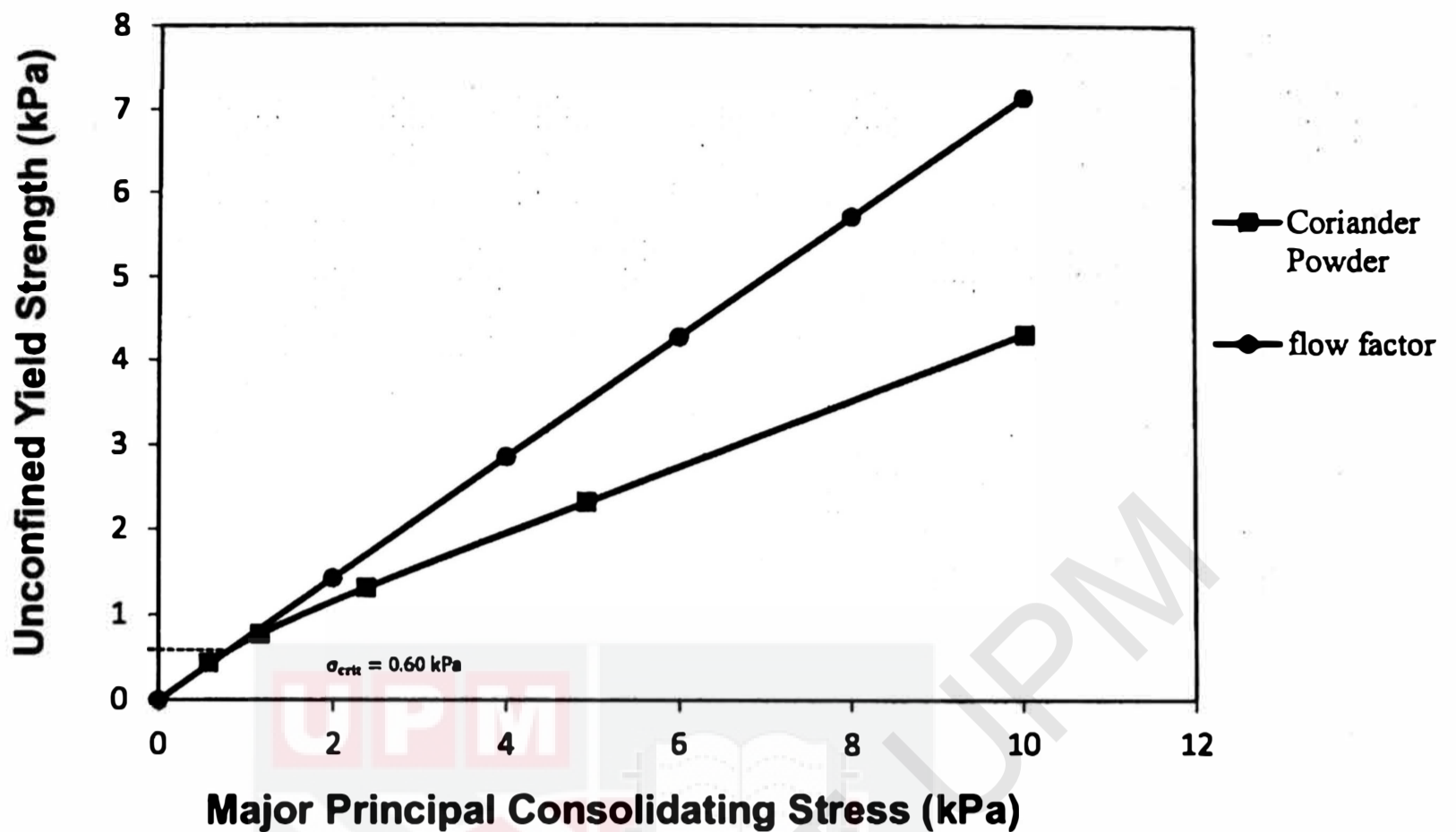


Figure 4.7 Determination of critical stress ( $\sigma_{crit}$ ) of coriander powder

As stated by Mohd Salleh et al (2014), more difficult-flowing powders would require a greater opening diameter of the design hopper, while powders with easier flow properties require a smaller opening diameter. This has been proven by the smallest minimum outlet diameter obtained from turmeric powder where it has better flow properties when referring to the flow function in Figure 4.6. However, chili powder which was the difficult-flowing powder has the second smallest of minimum outlet diameter since the critical stress and bulk density from the shear cell test was higher than turmeric powder. As for coriander and cumin, even though both flow function line for these powders were kind of similar, cumin powder has smaller minimum outlet diameter compared to coriander powder because the bulk density and critical stress of both powders are vice versa to each other, thus resulting in difference minimum outlet diameter as obtained in Table 4.2.

## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

In conclusion, the median particle size of turmeric powder, chili powder, coriander powder, and cumin powder were 81.0  $\mu\text{m}$ , 114.3  $\mu\text{m}$ , 225.0  $\mu\text{m}$ , and 288.6  $\mu\text{m}$ , respectively. This showed that turmeric powder has the smallest particle size compared to others. It can be said that the smallest particle size provides better compressibility since the particles will pack together closely during compression whereby the volume of the compact will be reduced, thus resulting in high cohesivity of the powder, but better flowability could be obtained because of low fat content of the powder.

Other factors that also contribute to the flowability of spices are moisture content, bulk density, and fat content. It has been found that chili powder with 114.3  $\mu\text{m}$  of median particle size has higher Hausner ratio and Carr index value compared to turmeric powder which has a smaller median particle size than chili powder. This is due to low bulk density, high fat and moisture content of chili powder which result in high values of Hausner ratio and Carr index. Thus, it can be said that although all spices used have a very, very poor flowability, however, chili powder was categorized as a very cohesive powder whereas the others were only cohesive.

Apart from that, smaller particle size will result in a lower effective angle of internal friction and higher angle of wall friction. Hence, a steeper hopper design with smaller hopper half angle and a smaller minimum outlet diameter as the opening of the hopper are required to be designed in order to ensure a consistent and smooth powder flowing during the discharge operation from the hopper without any problems. Based on the result, it has been found that turmeric powder has the smallest minimum outlet diameter compared to others, which is 0.35 m. Hence, these results and findings are very useful in designing equipment for spices powder storage, filling, compacting and packaging.

As for the future works and recommendations, in-depth research should be carried out on various foods or spice powders such as powder flowability analysis based on their fat content and viscosity as according to Larsson (2016), decreased viscosity will increase the molecular mobility and favor inter-particle interaction, hence leading to increased cohesion, formation of lumps and decreased flowability of the powder. Moreover, comparison of a commercial anti-caking agent with the natural anti-caking agent added in food powders with different range of particle sizes should be studied in order to evaluate the particle interactions and powder flowability as well as hopper design.

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## APPENDIX A

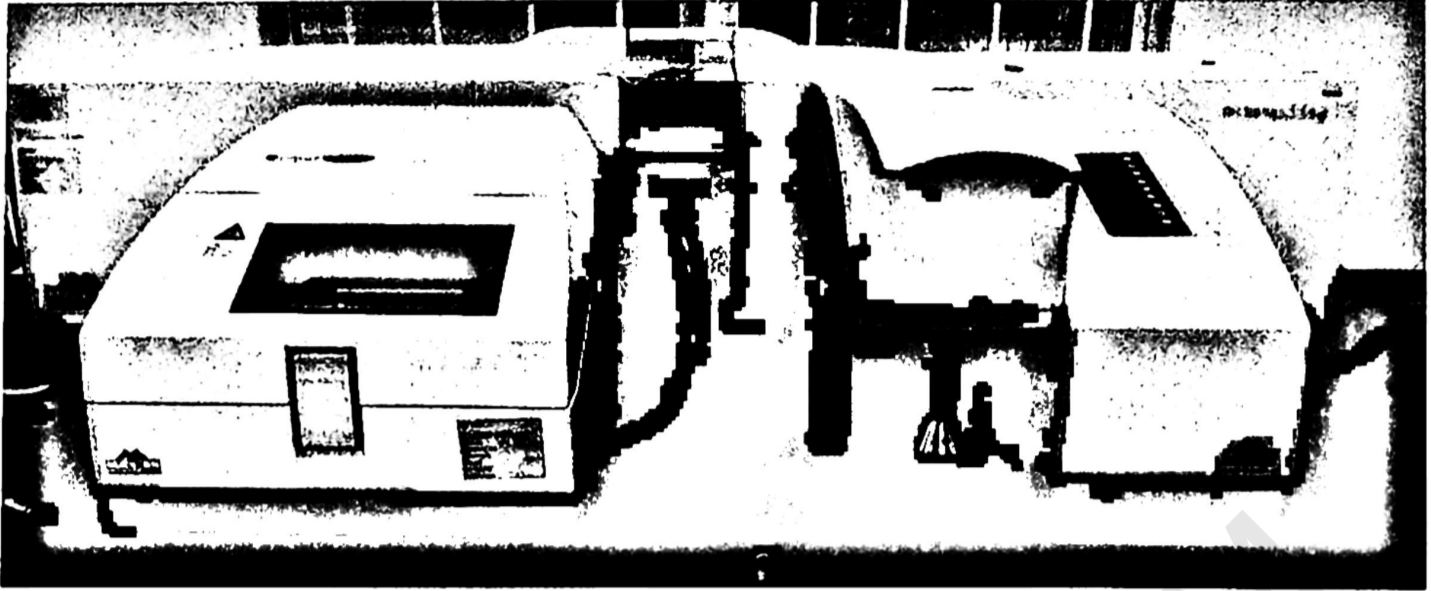


Figure A.1 Particle Size Analyzer (Malvern Mastersizer 2000, Worcestershire, U.K)



Figure A.2 Envelop and Tap Density Analyzer (Micromeritics GeoPyc 1360, USA)

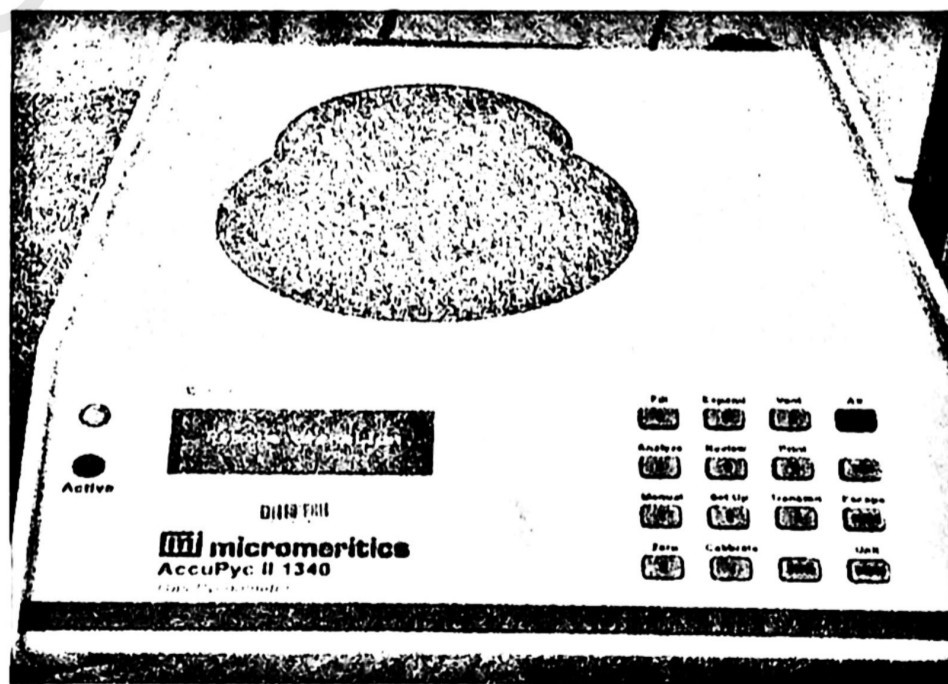


Figure A.3 Gas Pycnometer (AccuPyc II 340; Pycnometer Micromeritics, USA)

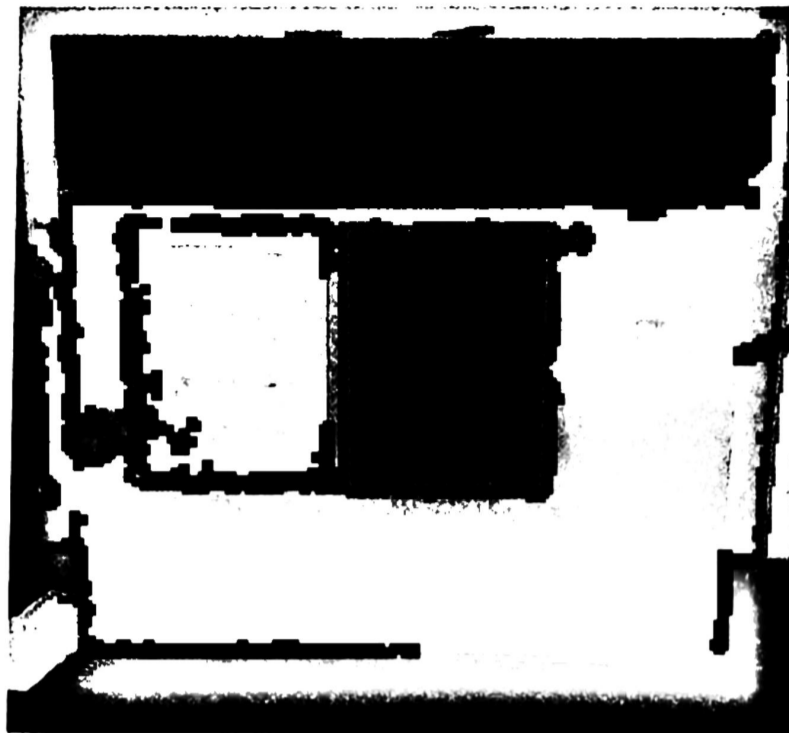


Figure A.4 Oven (Memmert, Germany)

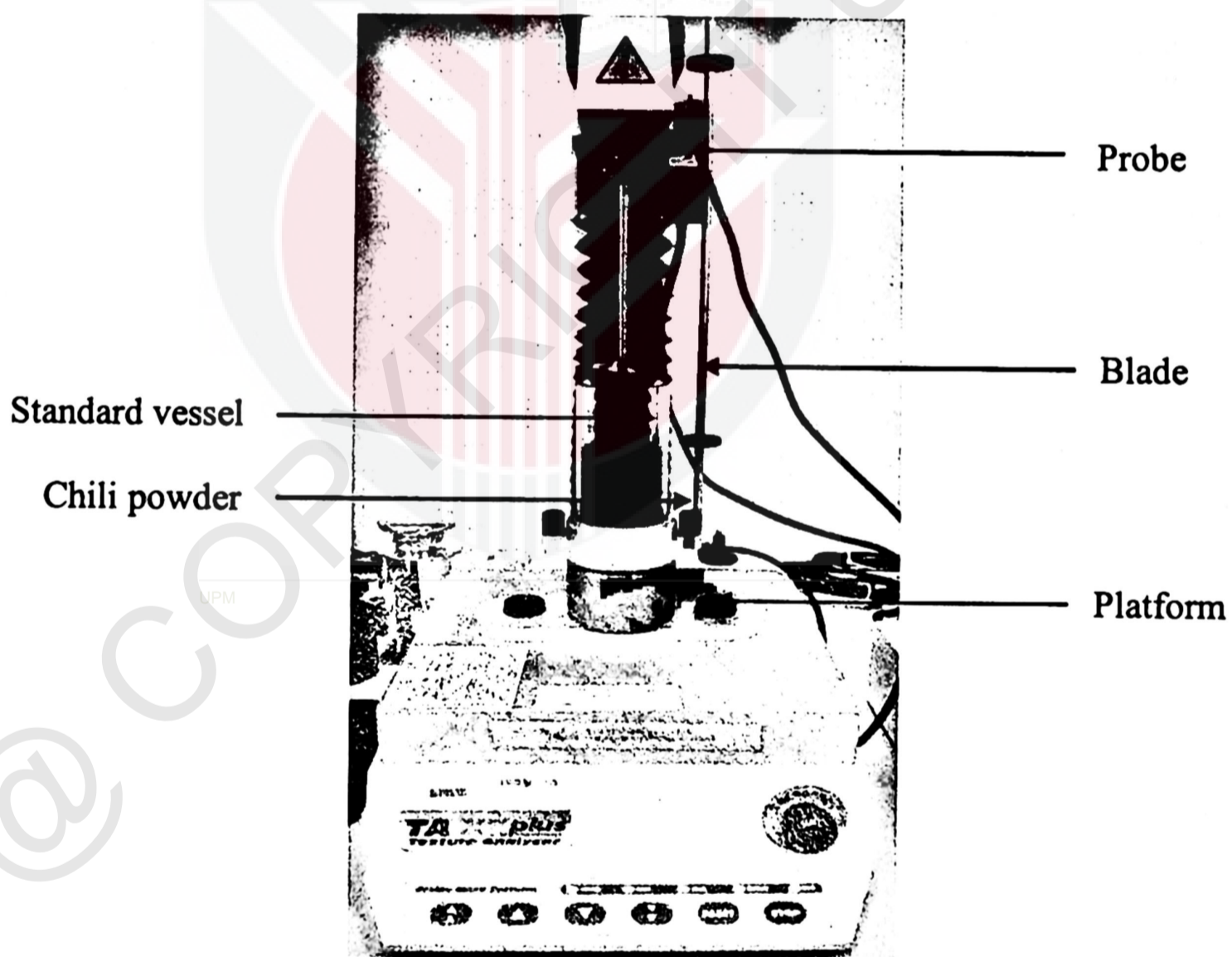


Figure A.5 TA.XTplus Texture Analyzer Stable Micro Systems (London, U.K)

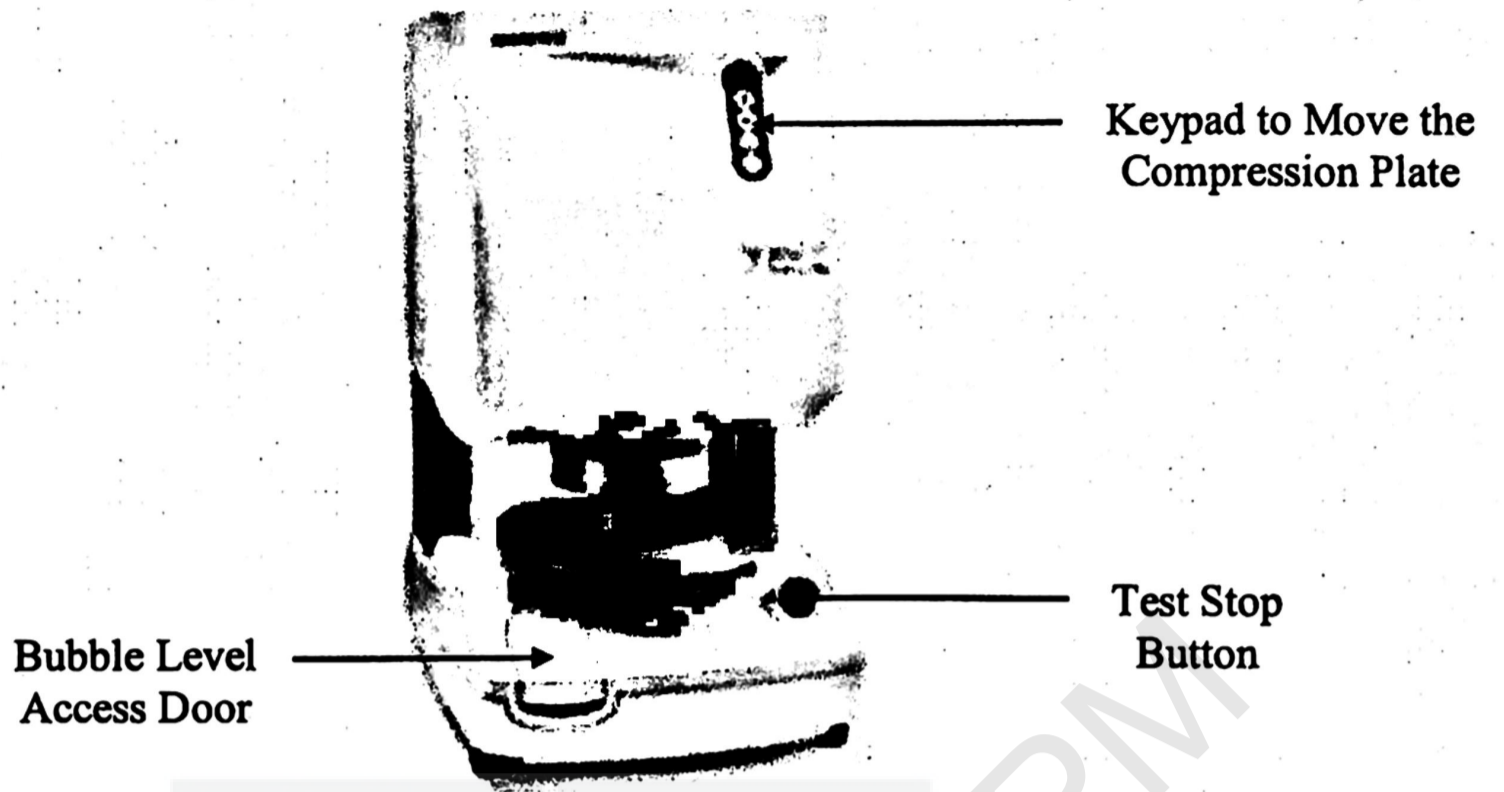


Figure A.6 Powder Flow Tester (Brookfield, USA)

## APPENDIX B

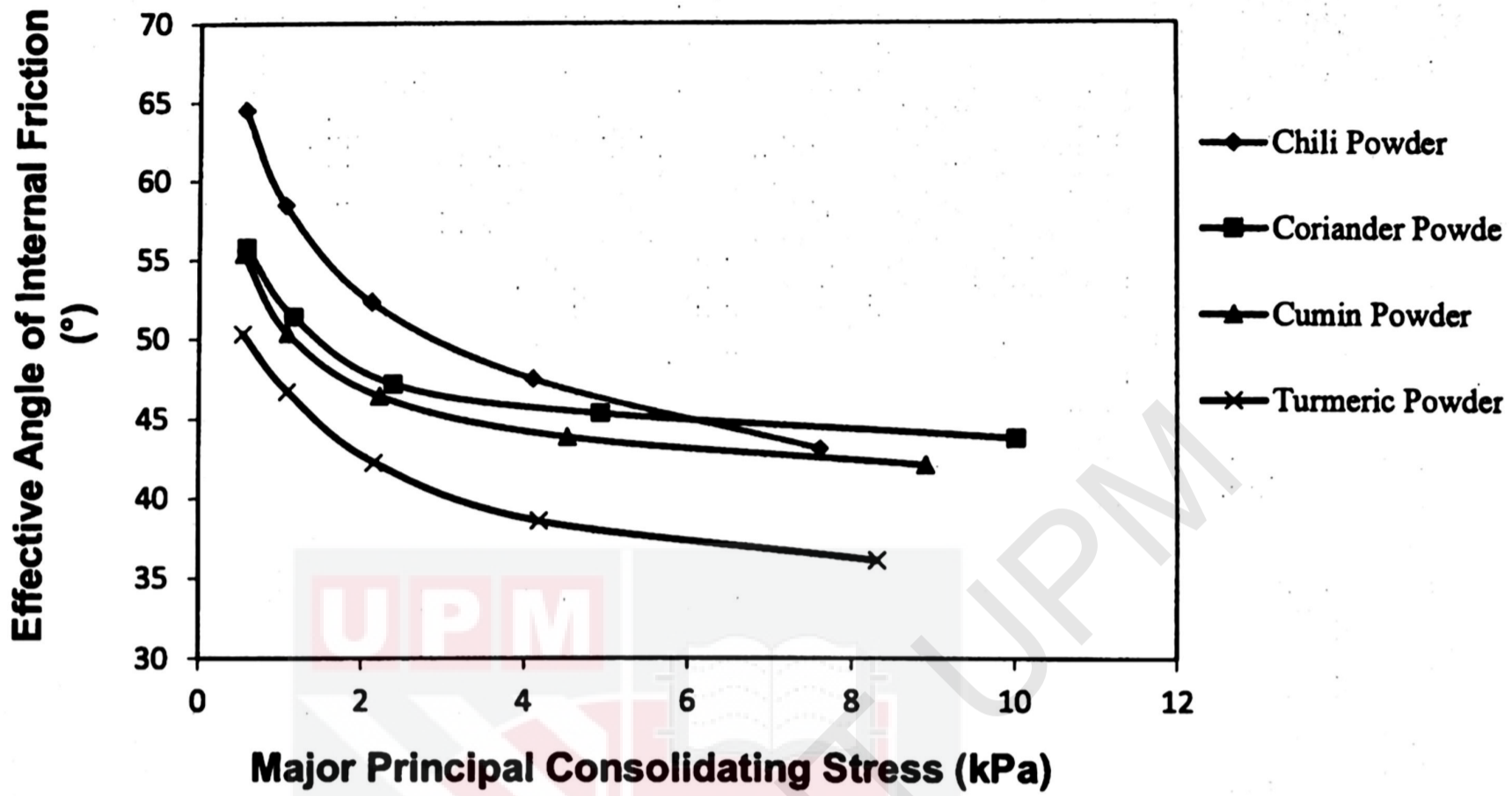


Figure B.1 Effective angle of internal friction of powders

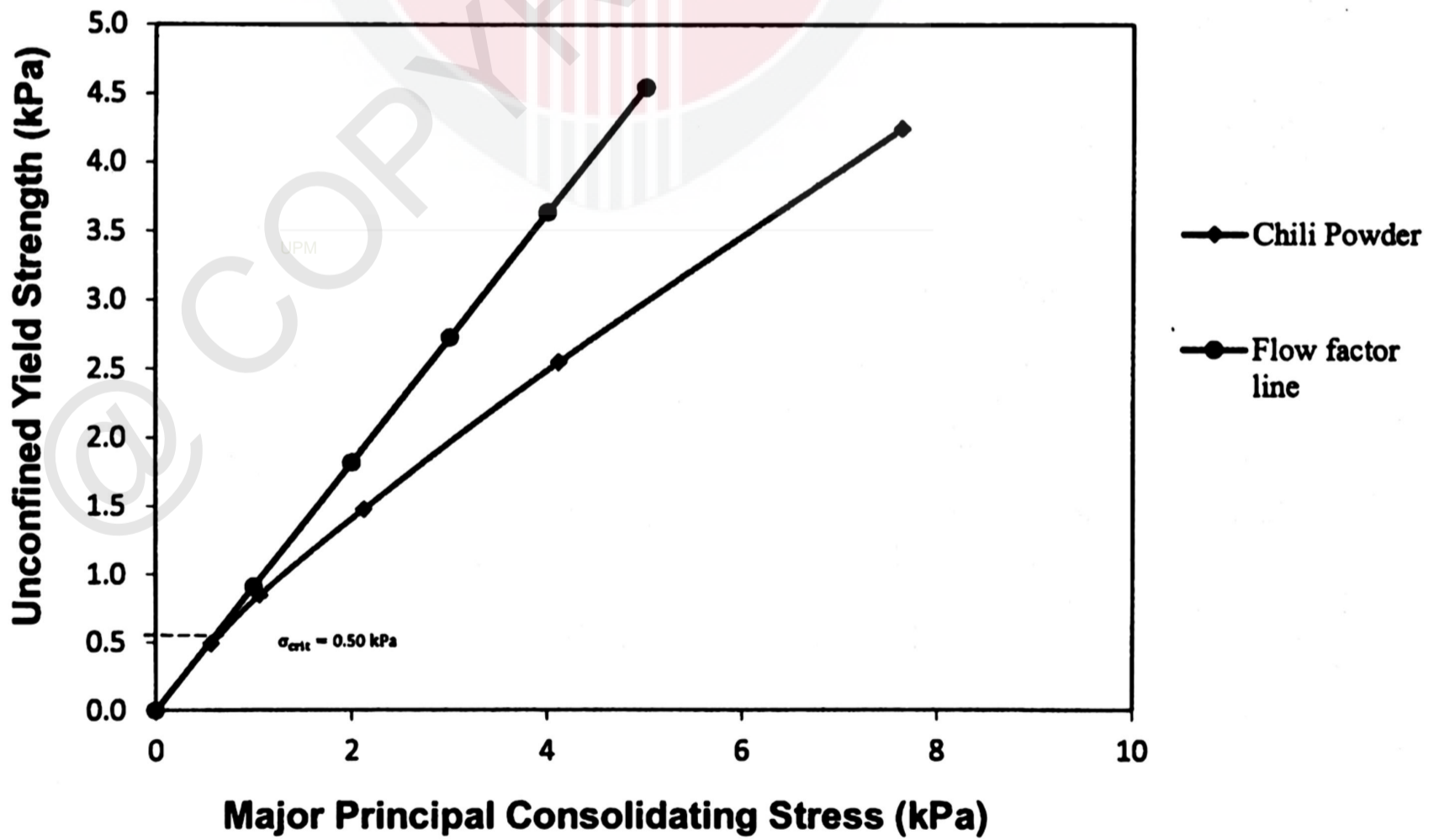


Figure B.2 Determination of critical stress ( $\sigma_{crit}$ ) of chili powder

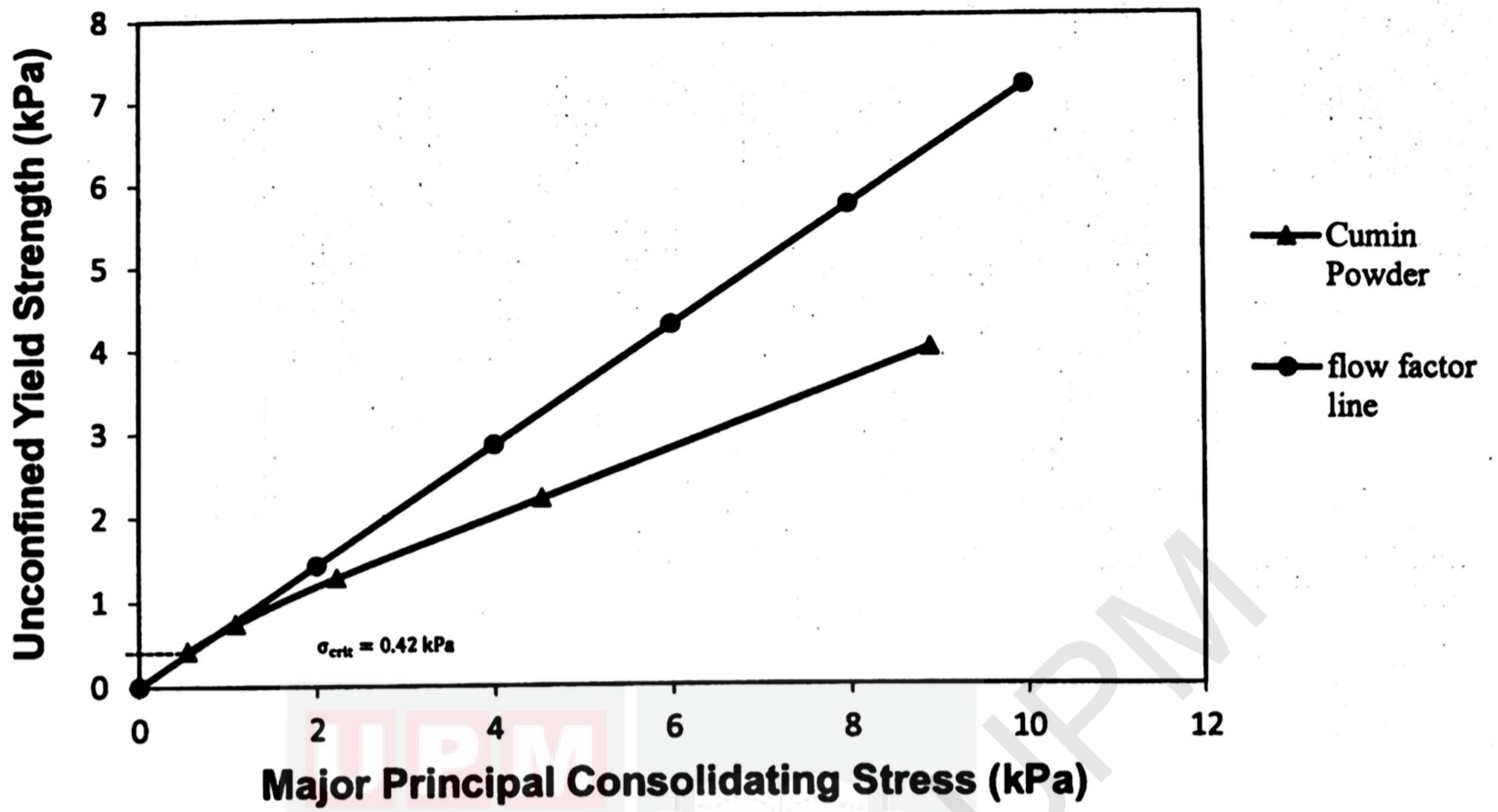


Figure B.3 Determination of critical stress ( $\sigma_{crit}$ ) of cumin powder

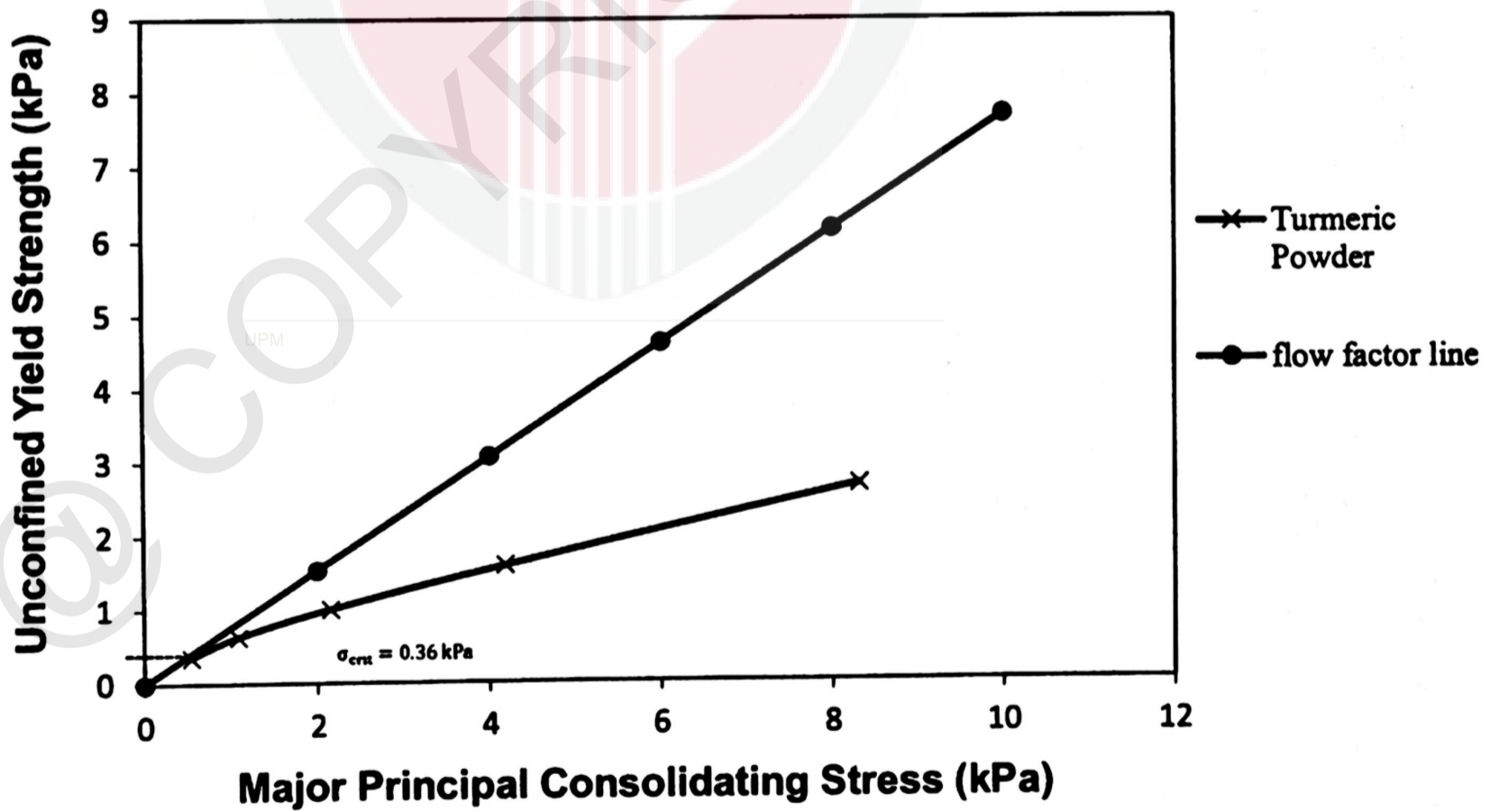


Figure B.4 Determination of critical stress ( $\sigma_{crit}$ ) of turmeric powder

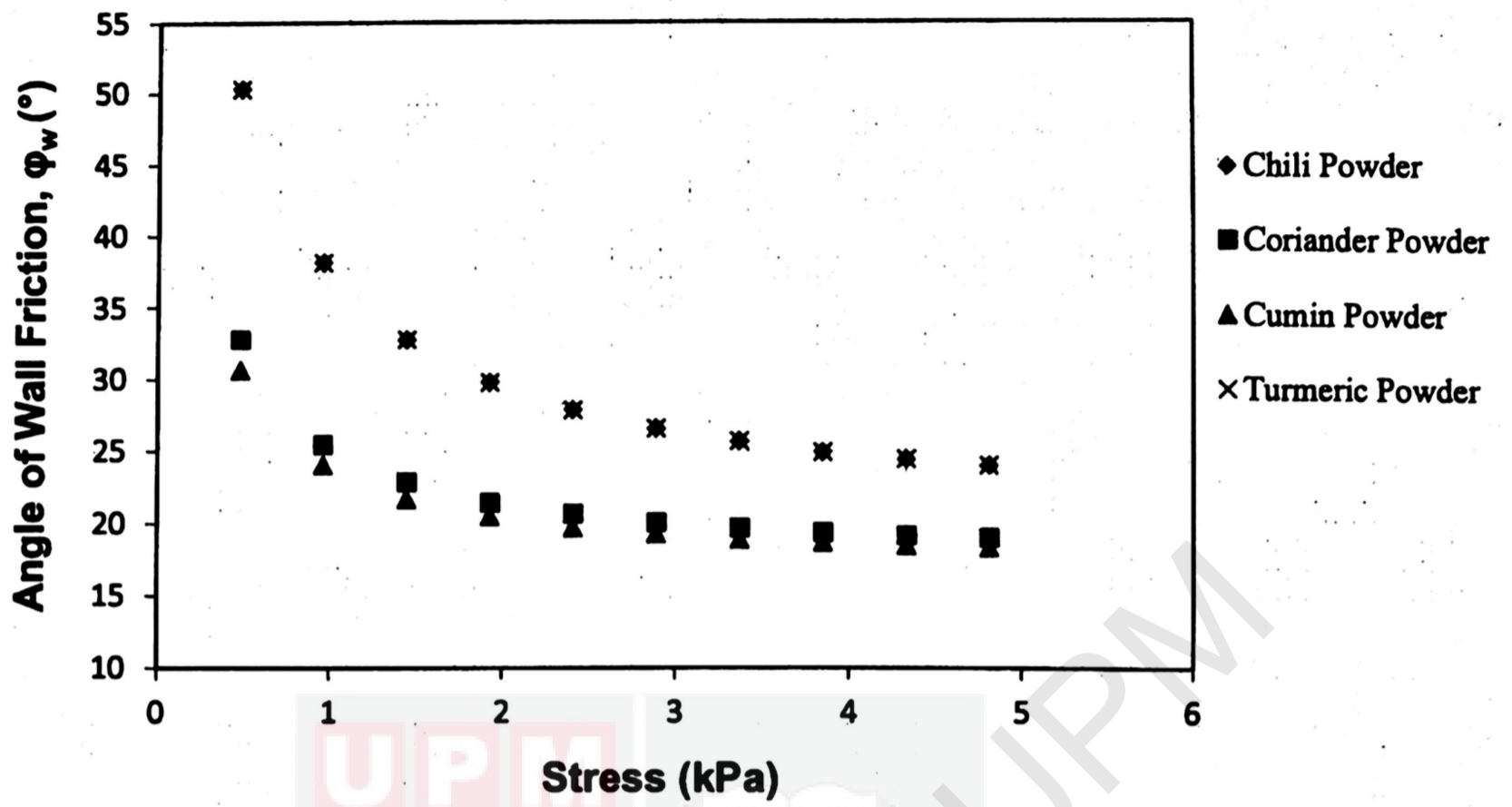


Figure B.5 Angle of wall friction of powders

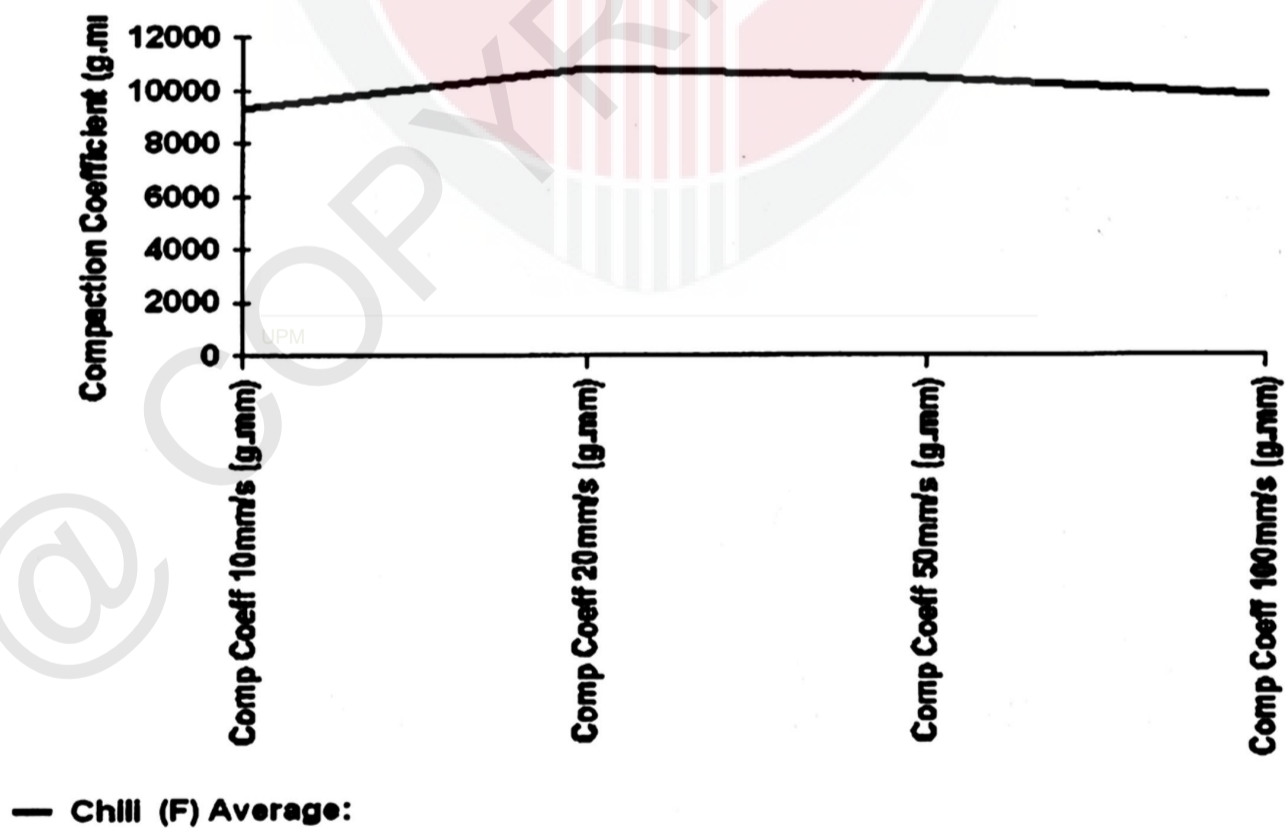


Figure B.6 Compaction coefficient-speed relationship of chili powder

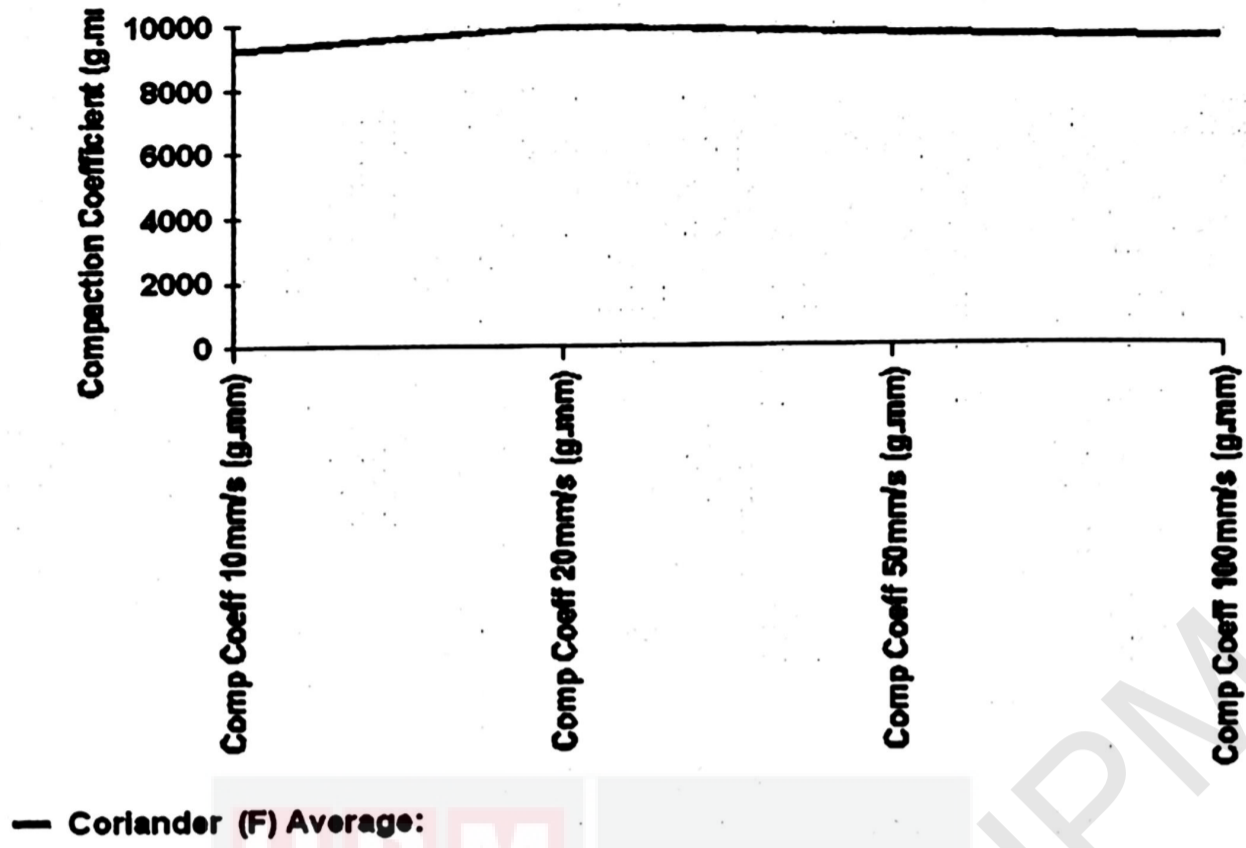


Figure B.7 Compaction coefficient-speed relationship of coriander powder

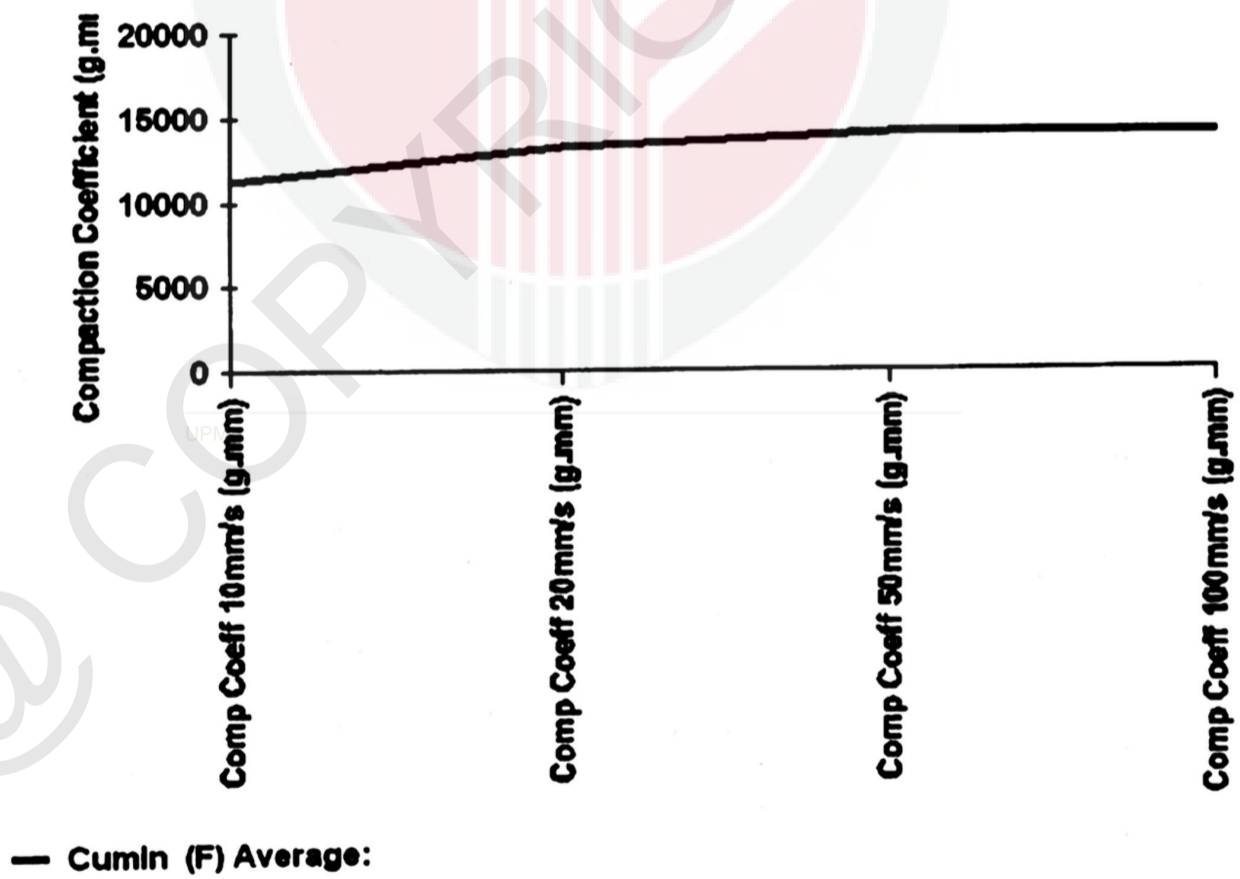


Figure B.8 Compaction coefficient-speed relationship of cumin powder

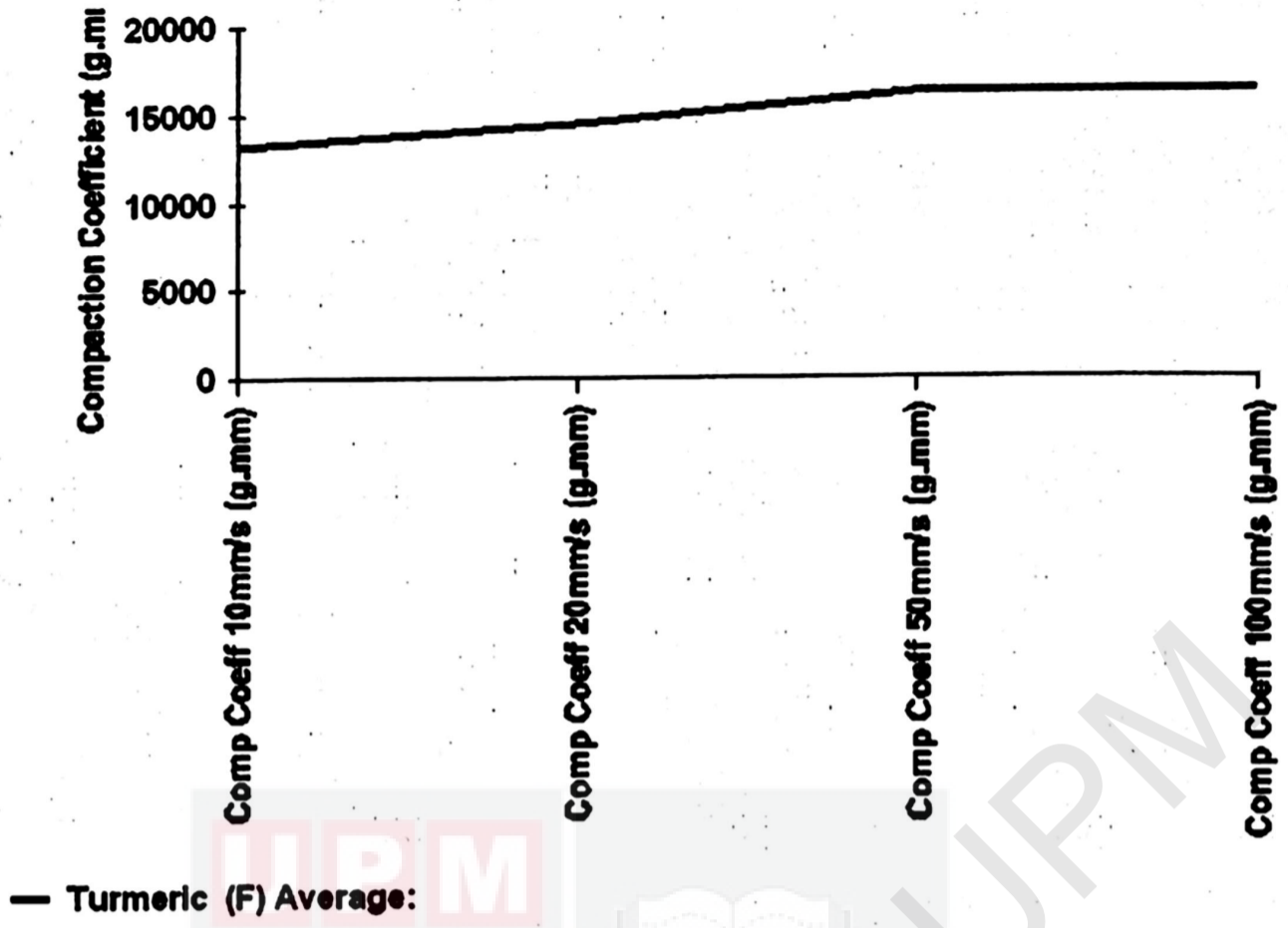


Figure B.9 Compaction coefficient-speed relationship of turmeric powder