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THE EFFECTS OF MOISTURE CONTENT ON THE PHYSICAL PROPERTIES OF SPRAY-DRIED NUTRITIONAL POWDER

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ABSTRACT

The relationship between moisture content (MC) and various physical attributes of spray-dried dairy-based nutritional powder was studied. There was no observed relationship between MC and particle size of the powder. However, MC was found to be inversely related to the glass transition temperature and directly related to the water activity of the powder. An increase caking index was also observed when MC increased. In particular, there was a sharp increase in the caking index when MC is higher than 3.5%. To minimize powder sticking and caking, it is recommended to keep the MC of spray-dried powderlower than 3.5%.

Keywords: *spray-dried nutritional powder, moisture content, physical properties, flowability, caking.*

I. INTRODUCTION

Nutritional supplement powder is one of the key functional foods with rapid growth in the past 20 years due to its market demand and health benefits. Dairy-based nutritional powder refers to the nutritional powder developed to deliver necessary nutrients to meet the additional nutritional needs of different consumers. It can be categorized into sport nutrition, medical nutrition, fortified food, and adult nutrient supplement powders. The matrices typically contain proteins (especially milk proteins), fats, carbohydrates, as well as various micro-nutrients such as minerals, vitamins, and other functional ingredients such as lutein and curcumin.Table 1 shows a summary of the macro- and micro- nutrients as well as the functional ingredients that are typically present and/or added to dairy-based nutritional powders.

Table 1:Macro-, micro-nutrients and functional ingredients in dairy based nutritional powders

	Classes	Ingredients
Macronutrients	Carbohydrates	Disaccharides (sucrose, lactose)
		Corn Syrup, maltodextrin
	Proteins	Milk protein (casein, whey)
		Vegetable protein (soy protein, pea protein etc.)
	Lipids	Vegetable oil (sunflower oil, soy oil etc.)
Micronutrients	Minerals	Major minerals (e.g. calcium, phosphorous etc.)
		Minor minerals (e.g. iron, zinc etc.)
	Vitamins	Fat soluble (Vitamins A, D, E & K)
		Water soluble (Vitamins C &Bs etc.)
Functional ingredients	Prebiotics	Inulin, fructo-oligosaccharides (FOS)
	Mono – or poly – unsaturated fatty acids	DHA, linoleic acid etc.
	Others	Lutein, curcumin, probiotics etc.

Fig. 1 shows the typical processing flow of the dairy-based nutritional product. Firstly, the ingredients are compounded to obtain a liquid blend. The blend undergoes preheating treatment and homogenization to achieve a fine emulsion. Homogenization is a mechanical process used to reduce the size of large fat globules and disperse them in the solution. The fine emulsion will go through Ultra high temperature short time treatment (UHTST) to kill off all the heat-resistance bacterial to ensure safety, and then evaporated to 40-50% total solid. The concentrated feed will be submitted for spray drying to achieve a powder microcapsule at a moisture ranging from 2% to 5%. The spray-dried powder could further dry blend with heat sensitive functional ingredients such as probiotics if needed. Finally, the finished product will be packed and dispatched [1].

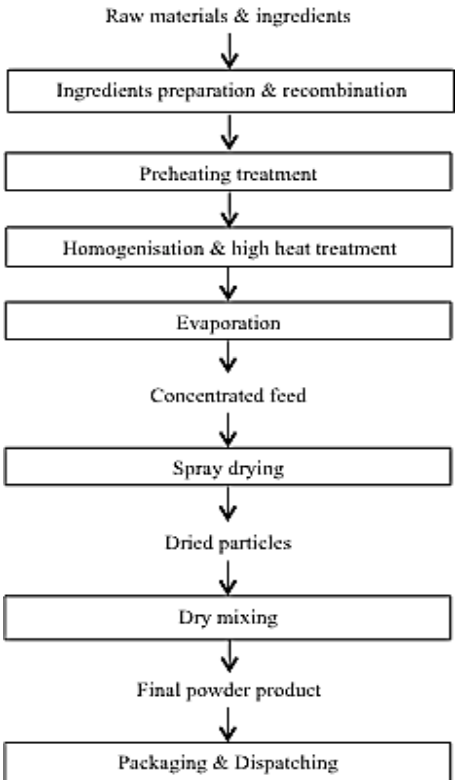

Figure 1:Processing flowchart of dairy-based nutritional powder

Spray drying is the dehydration method employed to produce powder from a liquid feed. It is the most common technology used to manufacture nutritional powder. The quality of the finished product is influenced through various factors including material used, processing, packaging, and storage conditions. The most known quality issues include poor powder flowability (lumpy, sticking and caking), poor wettability, dispersibility, and re-hydration, and oxidation etc.The industry needs the "right formula" and the "right processing" to lead to safe and high quality nutritional products in order to meet consumers' expectations.

Flowability is an important attribute of powder and a high flowability is desirable. Flowability is known as the ability of powder particles to move freely without the formation of clusters, lumps or aggregates. It is dependent on the physical properties such as glass transition temperature (T_g), sticky-point temperature(T_s), moisture content (MC), powder composition and more [2]. T_g refers to the temperature where amorphous components of the powder change from "glassy" state where they have lower mobility and high viscosity of 10^{12} Pas·s to "rubbery" state where they have higher mobility and lower viscosity of 10^6 to 10^8 Pa·s. Various conditions like storage (e.g. relative humidity and temperature), processing (e.g. total solid content of feed), and compositions affect the T_g. When the temperature is above T_g, viscosity decreases that make lactose in powder sufficiently mobile to rearrange and initiate crystallization. Consequently, solid bridges formed amongst particles decrease the flowability of the powder [3].

Sticky-point temperature (T_s), where powder shows viscous flow and powder becomes sticky, is usually 10 to 20 ˚C above the T_g[3]. Above T_s, liquid bridges of sugar, fatty acid or dissolved materials in the powder are formed between neighboring particles and powder start to adhere to the wall of the spray dryer. This results in reduced flowability and even caking of the powder.

Moisture content (MC) is one of the important physical properties that affects the flowability of the powder. A study by Roos and Karel [4] shows that there is an inverse relationship between MC and T_g. This is because water has a good plasticizing property with low T_g of -135 ˚C and causes glass transition temperature depression. Hence, this depression of T_g increases the stickiness of powder and decrease its flowability. The effect of MC on T_{gm} of powder can be predicted using the following equation by Gordon and Taylor [5]:

$$T_{gm} = \frac{kw_w T_{g,\,w} + w_s T_{g,\,s}}{kw_w + w_s} \quad (1)$$

where $T_{g,w}$, and $T_{g,s}$are the glass transition temperatures of the water (-135 ˚C) and solid substance in the powder respectively. w_w, and w_s are the mass fractions of water and solid substance in the powder respectively.

Water activity (a_w) is a measure of the amount of free water present in food that can participate in chemical reactions and/or microbial activity. a_w can be determined using the following equation:

$$a_w = \frac{\text{partial pressure of water in food at x °C}}{\text{partial pressure of pure water at x °C}} \quad (2)$$

a_w is a physical property that is closely related to the MC of the products. When MC increases, the a_w of powder also increases. This relationship between a_w and MC at constant temperature and pressure is typically expressed using the moisture isotherm graph. a_w is an important property that affects the quality of spray-dried products. Stapelfeldt et al. [6] observed that for powder that has a_w between 0.11 to 0.23, the quality of the product is preserved after storage for two months at different temperatures (e.g. 25 and 45 ˚C).

The ingredients used, especially low molecular weight (LMW) carbohydrates and amino acids, affect the flowability of the powder. Referring to the ingredients commonly used in dairy-based nutritional powder shown in Table 1, LMW carbohydrates like lactose and corn syrup (contains LMW glucose and fructose) are used. Due to the rapidness of the spray drying process, these sugar molecules do not have sufficient time to arrange into crystal structures. Hence, these sugar molecules exist in the amorphous state and have their own T_g. The T_g increases when molecular weight increases. Hence, the amorphous sugars with LMW have low T_g. The presence of these components lowers $T_{gm}$of the powder. Particles are more likely to stick to one another and have lower flowability [7]. The influence of the amorphous components on the T_{gm} of powder can be predicted using the following equation [8]:

$$T_{gm} = \frac{\sum_1^n w_i \Delta c_{pi} T_{gi}}{\sum_1^n w_i \Delta c_{pi}} \quad (3)$$

where w_i is the mass fraction of an amorphous component, Δc_{pi} is the change is specific heat capacity during glass transition and T_{gi} is the glass transition temperature of the component.

Moreover, these components exhibit high hygroscopicity and absorb moisture from the environment. The absorption of water vapor causes a reduction in T_{gm}, which makes the powder susceptible to sticking and decreases the flowability [8]. Apart from the LMW carbohydrates and amino acids, fat content also affects the flowability of the powder. Powder that contains higher fat content will also have a higher free fat content on the surface of particles. When temperature fluctuation happens, particles form liquid bridges and these bridges transform into solid bridges consisting of crystalline or partially crystalline fat [9]. These bridges decrease the flowability of the powder.

The particle size is directly related to the flowability of powder. Fitzpatrick [10] studied the relationship between the mean particle size and flow index of whole milk and skimmed milk powders. The results showed that when the mean particle size increased, the flow index of the powder increased, indicating an increase in the flowability of powder. This observation could be attributed to the larger surface area that a smaller particle has as compared to larger particle. Hence, with higher surface area, the cohesive forces between the particle increase contributing to the low flowability of powder with small particle size.Flowability reduces when the inter-particle forces increase. Consequently, undesirable physical changes like sticking and caking can happen. These changes disrupt the processing and adversely affect the quality of the powder.

There is an inverse relationship between T_s and MC. Hence, when MC increases, stickiness increases, making sticking a challenge during the spray drying of powder with higher MC [11]. Sticking is an initial indication of caking. In the early stage of caking, the particles become sticky and start to adhere to one another. Then, a series of physical changes like formation of liquid and/or solid bridges, loss of open pores, and formation of amorphous melt, resulting in the formation of cakes.

Referring to Fig. 2, there are five distinct stages involved in caking. Particles first become sticky and start to adhere to one another. Then, particles form bridges with one another. These bridges thicken and increase in length. Particles then start to lose their shape and structure, resulting in the collapse of the particles. Open pores on the surface disappear and finally, an amorphous melt is obtained [12].

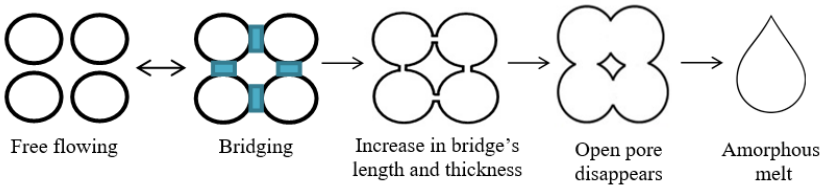

Figure. 2. Illustration of the caking processes

The occurrence of caking is closely related to the physical properties of powder (e.g. T_{gm}, powder composition and MC) and the external storage environment (e.g. relative humidity and storage temperature). For instance, amorphous sugar caking happens when the product is stored at a temperature near or higher than the T_g of the powder. Amorphous sugar molecules then begin to display viscous flow and become sufficiently mobile to initiate crystallization. Consequently, solid bridges formed amongst particles lead to caking [3]. This crystallization of lactose also increases the free fat on the surface of particles. This is because when lactose molecules form a crystal structure, protein and fat molecules are not included in the structure and are expelled to the surface of the particle. Hence, the increase in free fat content makes the powder more susceptible to caking caused by fat melting. Fat melting happens when the powder is exposed to the high-temperature environment and fat on the surface melts. Then, the particles stick to one another and form liquid bridges. When the powder is subsequently exposed to a low-temperature environment, fat molecules that form the liquid bridges crystallize, which result in solid bridges consisting of crystalline fats [13]. Hence, these bridges cause powder to be less flowable and cake.

Humidity caking occurs when the powder is stored in an environment of high relative humidity (RH), leading to moisture gain and Tg depression. This will also initiate lactose crystallization and eventually result in caking [14]. Moisture gain also causes proteins to aggregate through reactions like thiol-disulfide interchanges and formation of covalent bonds mediated by lysine residues [15]. This causes the sticking of particles leading to caking via protein sticking.

Sticking and caking are undesirable deleterious change and they lead to a loss in functionality and lowered quality of the products [16]. A powder with good reconstitution property has a larger surface area and a higher porosity than the powder that has poor reconstitution property. As seen in Fig. 2, an amorphous melt of the powder is formed at the end of caking. The melt is an agglomeration of particles with a smaller surface area as compared to the original

powder. Also, caking causes pores on the surface of the particles to disappear, which decreases the porosity of powder. The decrease in surface area and porosity adversely affect the reconstitution of powder [17]. These cakes are unable to regain their original powdery state. Thepowder caking is considered as defect and have lost its quality and sales value [18].

The MC of the spray-dried powder has a significant impact on the final nutritional product from both economic and quality standpoints.In previous researches, only limited correlations have been built between the moisture content and physical properties of spray-dried nutritional powder. This study selected a mixed protein system of sodium caseinate and whey protein concentrate, aimed to investigate the relationship between MC of dairy-based nutritional powder, rate of caking and other changes in physical properties. A pilot scale experiment will be performed to reflect the industrial manufacture conditions.A recommendation will be made on the optimum moisture content of spray-dried nutritional powder for future commercial production.

II. METHOD AND MATERIAL

2.1. Materials

Sodium Caseinate (NaCas) was obtained from Fonterra Co. Group. Ltd in New Zealand, and Whey protein concentrate (WPC) was purchased from Leprino in the USA. The protein contents were tested by Kjeldahl method (N x 6.25). The protein contents are 90.2% in Sodium Caseinate and 76.6%in Whey Protein Concentrate. Corn syrup solid was supplied by Roquette in France. Soy oil was purchased from a local supermarket. All other reagents and chemicals purchased from Sigma-Aldrich (Missouri, USA) were of analytical grade.

2.2. Experimental design

In this study, the NaCas and WPC combination was served as wall materials. Total protein contents in the dried powders were constant at 20 % (w/w), and total fat contents were set at 40 % (w/w) for all samples. All the experiment was performed in pilot scale to mimic the industrial manufacturing conditions.

2.3. Preparation of oil-in-water (O/W) emulsions and spray-dried powders

Three liquid streams including the soy oil, protein blend (NaCas and WPC), and corn syrup solid solution were compounded at a temperature between 55 to 60°C using a high shear speed mixer (Essential Engineering, Singapore). Coarse emulsion was prepared by mixing the liquid mixtures for 30 min to ensure that all proteins were fully hydrated and no lumps were observed. Further homogenization was performed using a two-stage homogenizer (APV, Germany) to form a fine emulsion. All the prepared emulsions were set at total solid contents ranged from 30% to 33% (w/w).

The oil-in-water emulsions were then treated with an ultra-high-temperature-short-time (UHTST) process at 121°C for 5 s. The heat-treated samples were further evaporated to 45% total solids before spray drying, for the purpose of mimicking the industry conditions. After evaporation, the concentrates were spray-dried using a pilot scale spray dryer (SPX, Denmark). The inlet temperatures of the dryerwere set at180 °C. The outlet temperature was varied to obtain different samples of powder with MC from 2.5% to 5.0%.

2.4 Moisture content (MC) analysis

The MC was measured using a moisture analyzer (HR83 Halogen Moisture Analyzer, Mettler Toledo, Switzerland). About 3 g of sample was spread on the weighing pan. The sample was heated to 100˚C then held at that temperature until constant mass was achieved. Triplicated analyses were conducted for each sample. The equipment was reset and cooled to < 50˚C before the next analysis.

2.5 Water activity (a_w)analysis

The $a_w$was measured using a benchtop a_w meter (4TE AquaLab Water Activity Meter, Decagon Devices, Inc, Pullman, USA). The instrument was first calibrated using standard solutions with a_w of 0.250, 0.500 and 0.760. The sample cup was filled halfway with powder and the a_w readings at 20 ℃ were taken.

2.6 Glass transition temperature (Tg) measurements

The T_g of spray-dried powders were determined using a differential scanning calorimetry (DSC Q2000, TA Instruments, New Castle, USA). An amount of 5 mg of sample was placed in a hermetically sealed pan, against an empty pan as reference. Samples were first equilibrated at -50 °C, followed by a heating process with the ramping rate at 2 °C/min, until 140 °C. Every 80 seconds, an auto-modulation was conducted at ±1 °C. T_g was then analyzed and reported according to the DSC curves. Triplicated readings were taken for each sample.

2.7 Powder Particle size determination

For spray-dried capsules, powder injection cyclone accessory was used to inject the powder sample with compressed air. The particle size of spray dried powder was determined using a laser diffraction particle size analyzer (LS 13 320, Beckman Coulter, Inc., USA). The refractive index was set at 1.40.All measurements were repeated at least twice for all samples.

2.8 Powder rheology

Aeration study of each sample was conducted using a powder rheometer (FT4 Powder Rheometer®, Freeman Technology Ltd, UK) up to 40 mm/s air velocity. Triplicates were done for each sample. The aeration ratio was determined using the following equation [19]:

$$AR_{40} = \frac{\text{Basic flowability energy (BFE)}}{\text{Aerated energy at 40 mm/s } (AE_{40})} \quad (4)$$

where BFE refers to the energy at 0 mm/s air velocity and AE_{40} refers to the energy at 40 mm/s air velocity.

During the aeration study, air was introduced into the powder sample to mechanically overcome the cohesive forces (e.g. Van der Waal's forces of attraction) present amongst the particles. The aerated energy (AE) measures the strength of cohesive forces amongst particles. A high AE means that a high amount of energy is required to separate the particles, which implies the cohesive forces between the particles are strong.

The AR_{40} calculated using equation (4) is a measure of the flowability of powder. A high AR_{40} means that the sample has a high flowability, and vice versa. Generally speaking, an AR_{40} value greater than 4 is desirable as it implies that the powder has a good flowability.

2.9 Determination of caking index

A sample of powder from each batch was weighed then transferred to a sieve (pore size = 420 μm) where it was shaken for 5 min. The powder that passed through the sieve was weighed. The caking index was determined using the following equation

$$C = \frac{M_T - M_F}{M_T} \times 100\% \quad (5)$$

where C is the caking index, MT is the total mass of powder used and MF is the mass of powder that passed through the sieve

III. RESULT AND DISCUSSION

3.1. Effect of moisture content on particle size

Fig. 3 shows the mean particle size of dairy-based nutritional powder at different MC from 2.85 to 4.60%. The mean particle size ranged from 124 to 149 μm. A study by Buma [20] showed that the particle size of the spray-dried powder is not affected by drying temperature but the total solid content and the pressure used during the atomization process. In this experiment, only the outlet temperature was varied but the total solid content of the concentrated feed and pressure used were kept constant. Varying the outlet temperature to obtain powder with different MC has little impact on the particle size. Hence, there was no observable trend between the MC and particle size, as seen in Fig. 3.

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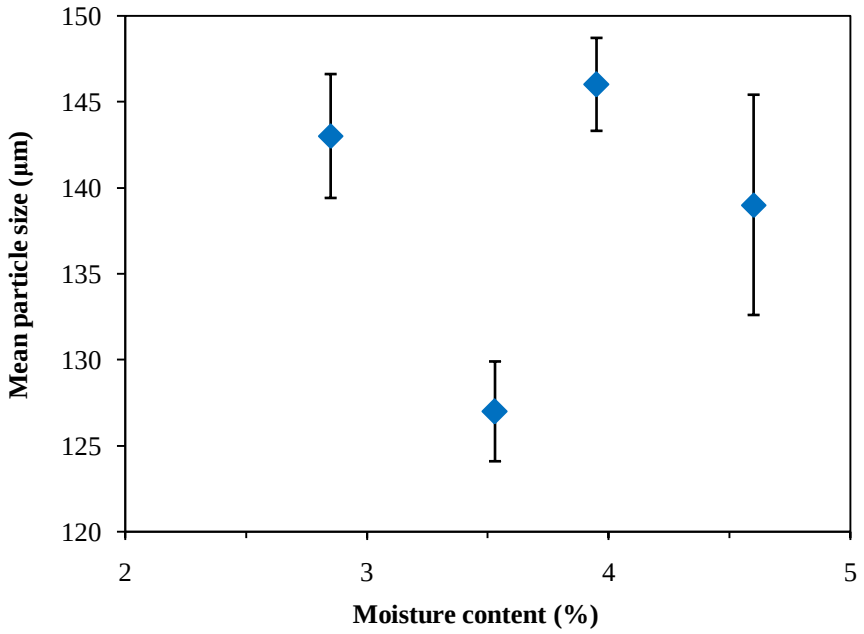

Figure 3. Particle size of spray-dried dairy-based nutritional powder at different MC

3.2. Effect of moisture content on a_w and T_g

Fig. 4 shows how the a_w and T_{gm} changed with the moisture content of powder,from 2.85 to 4.6%. when the MC of powder increased, a_w also increased.However, an inverse relationship was observed between MC and $T_{gm.}$For a food, an increase in MC is accompanied by an increase in a_w, and this relationship can be described by the water vapour sorption isotherm. The results are aligned with the previous studies [21][11]. As mentioned earlier, water has a low T_{gm} and has good plasticizing property. Powder with higher MC contained more moisture. This caused T_{gm} depression of the powder, and consequently, a decrease in T_{gm} when MC increased [22]

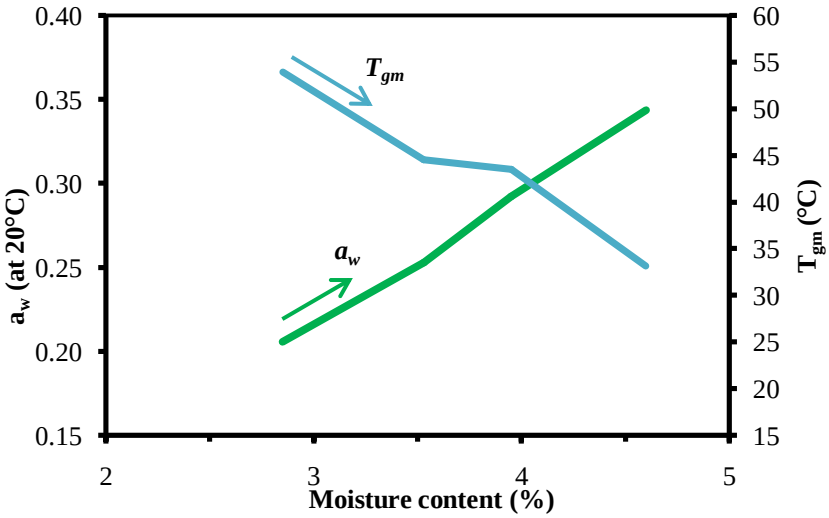

Figure 4. Effect of MC on a_w and T_{gm} of dairy-based nutritional powder

3.3. Aeration studies of powder with different MC

Aeration ratio serves as a measure of the flowability of the powder. During an aeration study, air is continually supplied to powder at increasing velocity to overcome cohesive forces and separate neighboring particles. Powder that is more flowable has weaker cohesive forces amongst particles. Hence, there will be lesser resistance to air flow and greater reduction in AE_{40}. The value of AE_{40} would be smaller for a flowable powder that has weaker cohesive forces and the AR_{40} calculated will be higher. The flowability is directly related to AR_{40}.

Fig. 5 shows the relationship between AR_{40} and MC of powder. The AR_{40} of the samples with different MC tested ranged between 2 to 5. Apart from the sample with MC of 3.53%, the AR_{40} of the other three samples were lesser than 4, which implies that the three samples had strong cohesive forces and did not have good flowability. There was no observable trend between MC and AR_{40} of the samples tested.

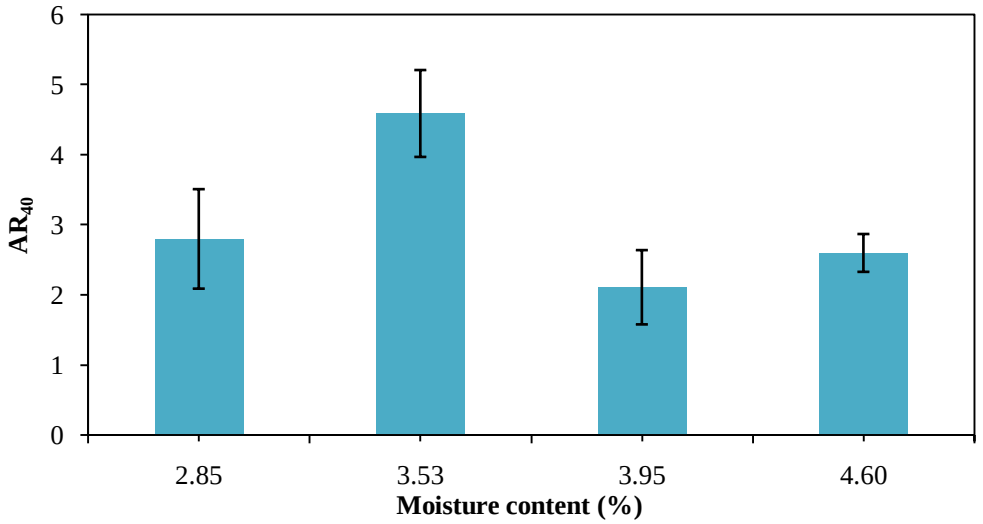

Figure 5. Aeration ratio of dairy-based nutritional powders with different moisture contents based on aeration studies conducted up to 40 mm/s air velocity

3.4. Caking indices of powders with different MC

The caking index is a measure of the extent of caking. A higher caking index implies that the powder has undergone a greater extent of caking as compared to powder with a lower caking index.Fig. 6 shows how caking index changed with MC of the sample. When MC increased, the caking index also increased. The caking index increased when MC increased because moisture causes the T_{gm} of powder to decrease. This would cause amorphous components to be stickier and powder to be more susceptible to caking [16].

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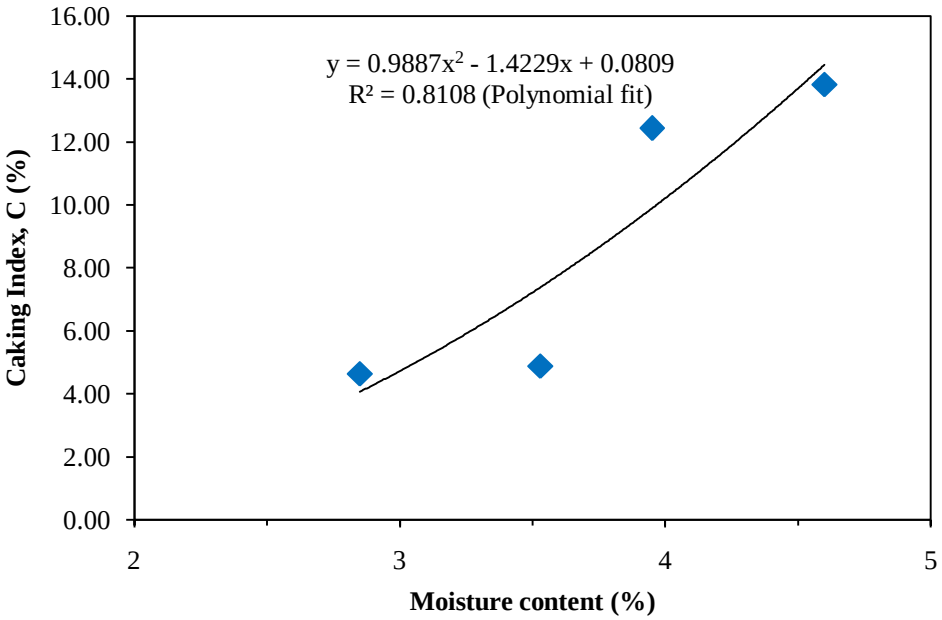

Figure 6. Effect of MC on caking index of dairy-based nutritional powder

Based on the experimental results, when MC increased from 3.53 to 3.95%, there was a drastic increase in the caking index. This implies that the optimal MC of the dairy-based nutritional powder is between 2.85% and 3.53% in this study. Otherwise, powder with MC greater than 3.50% will have a greater caking tendency, which is undesirable.

IV. CONCLUSION

In the present work of spray-dried nutritional powder, the effect of moisture content on various physical propertiesincluding particle size, glass transition temperature, water activity, flowability,and the caking index was studied. MC had little impact on the particle size. The increase in MC led to an increase in $a_w$and a decrease in T_{gm}. Above 3.50% MC, there was a drastic increase in the caking index. Hence, for the processing of the mixed NaCas and WPC protein nutritional product, it is recommended that the optimal MC to achieve should be less than 3.5% to maintain physical qualities of powder to prevent extensive caking of the powder.MC could also affect the rate of auto-oxidation of unsaturated lipids present in powder [6]. Future works should focus on the potential impact of MC on different chemical reactions such as lipid oxidation and non-enzymatic browning of spray-dried nutritional powder.

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