

A study on friability, hardness and fiber content analysis of fiber enriched milk tablet

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Abstract. This study was performed to analyze the friability, hardness and fiber content of fiber enriched milk tablet derived from five different local fiber sources such as carrot, spinach, dragon fruit, mango and watermelon. Cow milk was mixed to complement with the tablet as a protein source. The powder were spray dried at 100°C, 120°C and 140°C and freeze dried at -60°C. The mixture of fruits and milk were made into equal ratio with the addition of 15% maltodextrin as a carrier. Tablets formed were used for friability and hardness test while dried powder were used for fiber content analysis. Dragon fruit tablet dried at 140°C have the highest friability with 11.42% of weight loss. The second highest friability was spinach tablet dried at 100°C and 120°C drying temperature with 9.30% and 9.28% respectively. The lowest friability was exhibited by carrot, mango and watermelon tablet at 100°C and dragon fruit at 120°C while carrot and spinach at 140°C. In contrast, none of the freeze dried tablets showed any weight loss hence they are not friable. For hardness test, all of the freeze dried showed to have higher tensile strength than spray dried, where carrot showed to be the highest at 2.27 Newton and the lowest were spray dried mango at 0.16 Newton. In fiber content analysis, freeze dried mango have the highest fiber content followed by freeze dried carrot and 140°C spray dried carrot. It can be concluded that the higher the spray dry temperature, the more friable is the tablet. While, high friability leads to lower hardness of tablets. In terms of fiber content, the higher the spray dry temperature, the lower the fiber content found.

1. Introduction

Recently, supplement is extensively developed to solve malnutrition which includes kids. Kids are prone to lack of fiber and protein which may retard their growth physically and mentally. Nowadays, kids have consumed unhealthy food like candy and junk food more than the food should be required like vegetables or fruits. Deficient of fiber has caused many gastrointestinal problems like constipation, diarrhea, inflammatory bowel disease and hemorrhoids. While the insufficient of milk has led to stunting, rickets, Kwashiorkor and even having lower IQs. Therefore, a more attractive way of approaching kids to have balanced food consumption is by introducing fiber enriched milk tablet. Previously, fiber enriched milk supplement has been invented to enhance the dairy products positive effects toward obese people. People are consuming dairy product like milk to control their body



weight[1]. The enrichment of fiber in the product has added more beneficial effects as it enhance the insulin sensitivity in obese people. Furthermore, fiber can also enhances the satiety of consumer due to its viscosity-producing and bulking effect which eventually helps people in controlling the body weight. A research has been proved that increasing dietary calcium of obese patients for one year resulted in a 4.9 kg loss of body fat[2].

On top of that, concerning on the advantages of fiber and milk itself, they promote a combination of useful effects toward kids in term of health and growth. Dietary fiber which comprised of soluble and insoluble form has individual roles in preventing Irritable Bowel Syndrome, inflammatory bowel disease, diabetes, heart disease, constipation, colon cancer, hemorrhoids and diverticulosis. Same goes with milk as it can helps prevent eye problems, promotes a healthy immune system, and essential for the growth and development of cells, mucous membranes, skin, bones and tooth health [3]. Fiber source for fiber enriched milk tablet could be derived from variety of plants or fruits. Different plants or fruits have particular content of fiber and it would not be the same. The problems arise in determining how the fiber content would vary between a source with another. Moreover, supplement made in tablet form has more benefits as it has longer shelf life, easy for transportation and storing. However, it is crucial to know how the different fiber sources would affect the structure of produced tablet like its hardness and friability. Therefore in this experiment, five different fiber sources are selected from vegetables and fruits to determine those parameters. Spray drying and freeze drying methods are used to transform the fiber and milk mixture liquid into powder form before tableting. Different temperatures of spray drying would also affect the tablet friability, tablet hardness and its fiber content. Therefore, analysis is done by varying the spray drying temperature to know how it would affect the resulting parameters and how would it be if compared with freeze drying technique.

2. Methodology

2.1. Materials

Fiber source like carrot, spinach, dragon fruit, mango and watermelon were purchased at Giant, Seksyen 7, Shah Alam, Selangor. Local fruits and vegetables selections are based on different colours as follow:

Table1. Colours represented by five different fiber sources

Fruits/Vegetables	Representative for colour
Spinach	Green
Watermelon	Peach
Mango	Yellow
Carrot	Orange
Dragon fruit	Purple

2.2. Spray Drying

500 g carrots are cut into smaller pieces and blended with 500 g milk (equal ratio) and mixed with 15% of maltodextrin using blender Pensonic PB-325. The mixture is transferred into beaker and filtered by filtering cloth to eliminate any solid material that might cause clogging through the spray dryer nozzle. Then, it is dried with spray drier (SD-Basic Lab Plant) with inlet air temperature of 100°C, 120°C and 140°C with compressed air pressure of 0.10 Mpa [4]. The powder produced is collected and stored in freezer before tableting. Repeat all the steps for different fiber source.

2.3. Freeze Drying

500 g carrots are cut into smaller pieces and blended with 500 g milk (equal ratio) and mixed with 15% of maltodextrin using blender Pensonic PB-325. The mixture is transferred into a freeze dryer round bottomed flask and covered with parafilm. Then, it is stored in freezer overnight before dried with freeze drier (SASTECH) with -60 C and vacuum pressure. After 2-3 days when it is sufficiently dried, the powder produced is collected and punched by mortar to reduce the particle size. Then, it is stored in freezer before sent to tableting. Repeat all the steps by different fiber source A subsection. The paragraph text follows on from the subsection heading but should not be in italic.

2.4. Tableting

The powder made from spray dry and freeze dry are sent to Faculty of Pharmacy of University Malaya for tableting. It used direct compression machine called GlobePharma MTCM-1 model which produced averagely 0.7 gram tablet with 6.3 mm thickness and diameter of 12.7 mm. The pressure used is 3000 atm.

2.5. Friability

Friability test was run at Faculty of Pharmacy at Universiti Teknologi Mara (UiTM). The equipment used is Electrolab Friabilator which set with 60 rotations. Every tablet was cleaned with brush and weighted by weighing balance before inserted into the friabilator. Then the equipment will rotate 60 times and once it finished, the tablet is collected. The tablet is cleaned again by brush and weighted by the same weighing balance. The percentage of weight loss is calculated as formulae follow:

$$\text{Percentage of weight loss} = \frac{\text{initial weight of tablet} - \text{final weight of tablet}}{\text{initial weight of tablet}} \times 100 \quad (1)$$

2.6. Hardness

Hardness test is run at Faculty of Pharmacy in Universiti Teknologi Mara (UiTM). The equipment used is Pharmatest. Once the tablet is held vertically on the machine platform, it will be compressed against the wall to measure the weight, diameter and thickness. Then the tablet will slide and brought horizontally before being compressed once again until it break to measure the force needed to break the tablet. All the reading is recorded for every tablet. The hardness of the tablet is measured by knowing its tensile strength which is calculated as following formulae.

$$\text{Tensile strength} = \frac{2F}{\pi dv} \quad (2)$$

F = force needed to break the tablet (Newton)

d = tablet diameter

v = tablet thickness

2.7. Fiber Content

Association of Official Agricultural Chemists (AOAC) method [5] 985.29 is a standard method invented by United States Department of Agriculture is used to study the content of total dietary fiber. The sample of fiber enriched milk powder is tested with three different enzymatic digestions which are alpha-amylase to gelatinize the solution, protease to remove protein, and amyloglucosidase to remove starch. Next is precipitation using ethanol to precipitate the residue. Then the precipitated residue is filtered by vacuum filter and washed by three different chemical

which are 78% ethanol, 95% ethanol and acetone. Next, one set of duplicate of residue was tested protein analysis and another set of duplicate was tested for ash analysis. The total fiber content will be calculated by subtracting the initial weight residue with weight of (protein + ash)[6].

i. Alpha-Amylase digestion

Duplicate of 1 gram of each fiber source powder is weighted and recorded as initial weight before put into 250 mL conical flask. 10 mL phosphate buffer (pH 6.0) is added into the beaker and check pH with pH meter. Adjust if pH does not equal 6.0 ± 0.1 . 0.1 mL heat-stable alpha-amylase solution is added. The sample is then transferred into boiling tube. The tube is covered with aluminium foil and placed in boiling water bath. The tube must be left at $95-100^\circ\text{C}$ for 15 minutes and is shaken gently at every 5 minutes intervals. Then, the solution is cooled to room temperature.

ii. Protease digestion

The pH is first adjusted to 7.5 ± 0.1 by adding drops of 0.275 M NaOH solution. pH is checked with pH meter. 0.1 mL of protease solution is added. The conical flask is covered with aluminium foil and incubated at 60°C with continuous agitation for 30 minutes. Then, the solution is cooled to room temperature.

iii. Amyloglucosidase digestion

Drops of 0.325 M HCl solution are added to adjust pH to 4.5 ± 0.2 . pH is checked with pH meter. 0.3 mL amyloglucosidase is added, then covered with aluminium foil, and incubated for 30 minutes at 60°C with continuous agitation. Then let it cool.

iv. Precipitation of residue by Ethanol

60 mL of 95% EtOH is first pre-heated to 60°C (measure volume before heating) then added into the solution from previous step. Let precipitate form at room temperature for 60 minutes.

v. Filtration of Residue

Weigh 1 gram of celite to nearest 0.1 mg, then wet and distribute bed of celite in fitted crucible by using stream of 78% ethanol. Celite efficiently separates the fiber from the fritted glass of the crucible, to allow easy removal of the crucible contents. By using vacuum pump, suction is applied to draw celite onto fritted glass as even mat. The suction is maintained and quantitatively transferred the precipitate from enzyme digest to crucible. The residue is washed successively with three 20 mL portions of 78% Ethanol, two 10 mL portions of 95% Ethanol, and two 10 mL portions of acetone. In some cases, gums might form during filtration, trapping liquid in residue. If so, break surface film with spatula to enhance filtration.

vi. Drying of Filtered Residue

Residue is transferred into ceramic crucible and dried overnight in 105°C air oven. Cooled in desiccator and weigh to nearest 0.1 mg. Subtracted (crucible + celite + residue) with initial (crucible + celite) weights to determine weight of residue. The residue is analysed from one sample of set of duplicates for protein content and the other set of duplicate for ash content. Protein analysis is done by Kjeldahl Method, where the sample is get by cautiously scraping the celite and the fiber mat onto a proper piece of filter paper and kept it for Kjeldahl method.

vi. Fiber Content Calculation

Incinerate residue from second sample of duplicate for 5 hours at 500°C. The samples in the ceramic crucible s are arranged in the furnace and label first before started the incineration. After 5 hours, the furnace is left for another 5 hour for the temperature to cool before take out the samples. When ready, the samples are weighed to 0.1 mg. and recorded the reading. Subtract the reading with crucible and celite weights to determine ash.

vi. Ash Analysis

The fiber content was calculated by the equation below[5]:

$$\text{Total dietary fiber} = \left(\frac{\frac{R_1+R_2}{2}}{\frac{W_1+W_2}{2}} - P - A \right) \times 100 \quad (3)$$

R1= residue weight of first duplicate R2 =

residue weight of second duplicate P =

Protein weight

A = Ash weight

W1 = weight of initial sample of first duplicate, W2 =

weight of initial sample of second duplicate

vi. Kjeldahl Method

a) Digestion. The sample from previous procedure is placed into a digestion flask, along with 12 ml of concentrated sulfuric acid (H₂SO₄). 7 grams of potassium sulfate is added and a catalyst, usually copper to increase the boiling point. The digestion flask is heated to 390 C until white fumes can be seen, and then continue the heating for about 60 minutes. The flask is cooled and cautiously adding 250 ml of water.

b) Distillation. The pH of the mixture is raised using sodium hydroxide (45% NaOH solution). This has effect of changing the ammonium (NH₄⁺) ions which are dissolved in the liquid to ammonia (NH₃), which is a gas. The nitrogen is separated away from the digestion mixture by distilling the ammonia (converting it to a volatile gas, by raising the temperature to boiling point) and then the distilled vapors is trapped in a special trapping solution of about 15 ml hydrochloric acid in 70 ml of water. The trapping flask is removed and the condenser is rinsed with water so as to make sure that all the ammonia has been dissolved.

c) Titration. 5 drops of phenolphthalein is added to the trapping solution. The dye turned into colorless indicating significant amount of the original acid still exists. A standard solution of NaOH is put into the burette, and slowly is added to the acid solution. The point at which the dye turns pink, indicate the "endpoint" has reached which all the acid has been neutralized by the base. The volume of the neutralizing base is recorded.

d) Calculation of Protein Content. One mole of ammonia coming from the digestion mixture will neutralize exactly one mole of acid in trapping flask. Therefore, the number of ammonia moles which equivalent to moles of nitrogen is found by steps as follow:

1. The number of moles of acid in the trapping flask originally (before any ammonia was trapped) is calculated as: moles of acid = molarity of acid x volume used trapping flask (moles_A = M x V)
2. The number of moles of base (NaOH) that were added from the burette to neutralize the remaining acid (that NOT neutralized by the ammonia) is calculated as:
Moles of base = molarity of base x volume added from burette (moles_B = M x V)

3. The number of "moles of ammonia" is calculated by subtracting mole base by mole of acid.
4. The number of "moles of ammonia" are coming from the protein is similar to the "moles of nitrogen"
5. The number of nitrogen (gram) in the original sample of protein is calculated as:
Grams of nitrogen = moles nitrogen x atomic mass
6. The percentage of nitrogen found in the original sample can now be calculated as:
%nitrogen =(gms nitrogen / gms sample) x 100%
7. Crude protein , CP = %nitrogen x 6.25 (6.25 as conversion factor)

3. Results and Discussion

3.1. Friability Test

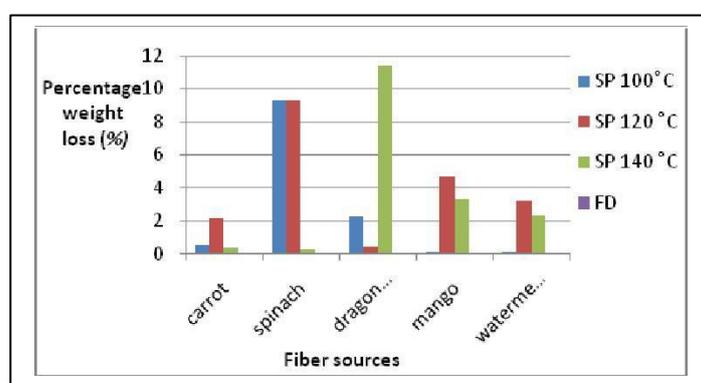


Figure 1. Percentage weight loss (%) vs Fiber sources

During the rotations, the surface of the tablet will gradually rupture and the tablet will lose some of its weight due to hitting force. Thus, this test will measure the friability of the tablet by determining the percentage weight loss. The higher the percentage of weight loss, the higher the tablet friability. Based on figure 1, at spray drying inlet air temperature of 100 C and 120 C, the spinach tablet shows the highest percentage of weight loss which around 9.3%. It shows quite big gap when compared with other fiber source tablet. This is because spinach has the lowest carbohydrate content, but high fiber content. Initially during the preparation for spray drying, the mixture of spinach and milk is filtered to eliminate any solid substance like pulp. A fully liquid suspension is obtained to prevent any clogging in the spray drier and also to get high quality of powder. The filtration somehow has eliminated most of fiber contained in the spinach [7]. Same goes to some of carbohydrate content which had left behind, along with the fiber during filtration. Basically, carbohydrate is comprised of sugar and starch [8]. Both substance acts as natural binding agent which increase the hygroscopicity and interparticulate bonding strength within the tablet [9]. Therefore, the higher the carbohydrate content, the higher the hardness of tablet. When more fiber is removed, carbohydrate content will also be removed. Therefore, based on figure 2, the spinach shows quite low value of tensile strength which is 0.52% at spray drying temperature of 100 C and 120 C. This is due to the fact of the higher the friability, the lower the tensile strength and thus the hardness.

At inlet air temperature of 140 C, dragon fruit tablet shows the highest percentage of weight loss which around 11.5%. It shows great gap when compared with inlet air temperature of

100 C and 120 C and even with other tablet at the same temperature. This is because at high inlet a temperature, the residual moisture content within powder particle is reduced. When the moisture content reduced, there will be less water to bind powder particle together by its surface tension or known as water holding capacity [10]. This give rise to reduced internal cohesion and shear strength between particles in the tablet. Shear strength is defined as the ability of solid particle to resist friction [11]. This is because the existing solid particle of powder tends to flow against each other when external force is applied and thus cause internal friction. When the shear strength is reduce, the powder particle become less cohesive. As a result, the flowability of the particle will increase(A-sun 2015). Flowability is defined as the ability of powder to flow[11]. If moisture content is decreases, the flowability of the particle will increase[12]. As the powder particles are free to flow, the tablet will exhibit a friable characteristic. Therefore, it can be concluded that the tablet becomes more friable at higher air inlet temperature.

While at inlet air temperature of 120 C and 140 C; mango, watermelon and carrot tablet are ranked the second, third, and fourth of decreasing friability respectively. At 120 C, the percentage of weight loss of mango is 4.66%, watermelon is 3.21% and carrot is 2.18%. At 140 C, the percentage of weight loss of mango is 3.35%, watermelon is 2.35% and carrot is 0.36%. Mango has the second highest friability because it has high sugar and fiber content. Since, a lot of fiber has separated out during filtration, it has caused the high amount sugar to left behind as well as the amount fiber. Therefore, there is lack of remaining sugar content which make the mango tablet more friable than watermelon and carrot. While, watermelon has moderate friability because it has moderate sugar content and least fiber content. Therefore, even the sugar content is moderate, the fiber content is too low to drag along much of sugar to be separated during filtration. Thus, the watermelon is less friable than mango. Next, carrot tablet is less friable than mango and watermelon because it contains more fiber that has been separated through filtration, but the remaining sugar content is being concentrated instead of drag along with fiber. This is due to the structure of carrot sugar which in form of dilution instead of mango and watermelon sugar which is present in pulp or semi liquid. Hence, the carrot sugar will pass through the filter medium more efficiently while mango and watermelon sugar are partially passed through it due to its semi liquid structure. It is even trapped by the filter aid. Thus, the carrot tablet is less friable than mango and watermelon.

The figure also shows that the friability of most of tablet is lower at inlet air temperature of 100 C, than at 120 C and 140 C. This may due to larger moisture content that is still present in the powder. Moisture content is aid in cohesiveness of the powder particle especially when it contains sugar. The water will bind more tightly with carbohydrate rather than lipid, protein or fiber. Therefore, it can be seen that the spinach in contrast has the most friable because it contain least sugar. It is followed by dragon fruit, carrot, mango and watermelon. The ranking is however does not justifying the rank of sugar content for each of fiber source. This may due to size of fruit particle itself which has different effect towards the friability of the tablet. Particle size is majorly affects the cohesiveness of the tablet. Lower particle size leads to lower degree of size distribution of particle. So, the particle will be arranged between them compactly where they are in close form and there are fewer spaces between particles [13]. As a result, the bulk density will be increased. This indirectly has reduce the flowability because there will be more internal friction that cause the particle hard to flow. Basically, particle size would not be affected by the inlet air temperature, otherwise after added by high concentration of carrier such as maltodextrin like 30% and above.

Based on the figure 1, all of freeze dried tablet is found to have no percentage of weight loss. This is because the powder formed exhibit flaky or irregular shape particle [14].This can be observed during the powder collection where the powder structure is obviously different from spray dried powder. The flaky structured is observed when the powder is being punched by mortar to reduce the particle size into finer. The freeze drying mechanism has caused sublimation of solid into gas without entering liquid phase. Eventually, in effect of the collapse and shrinkage of the product is prevented thereby resulting in a porous dried material which make the particle formed to be flaky [15]. Flaky particles have a high surface to volume ratio and as a result, the particles have high inter-particle friction. This has led to a decrease in flowability and become less or not friable at all.

3.2. Hardness Test

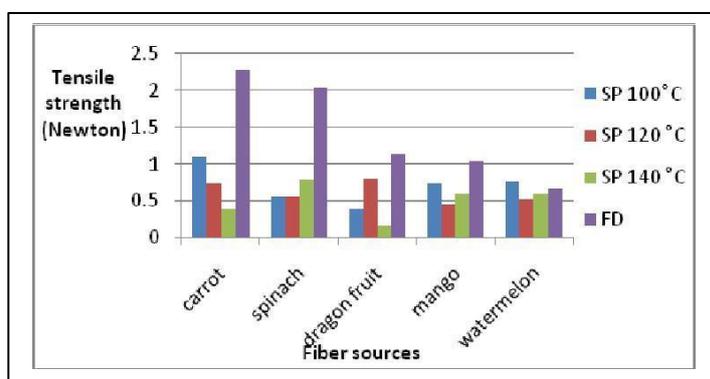


Figure 2. Tensile strength (N) of tablets vs Fiber sources

Hardness is largely influenced by the friability of tablet. The higher the friability, the lower the hardness of the tablet. As stated earlier, flowability is the ability of powder to flow under applied pressure. When the powder particle is more cohesive toward internal friction, the tablet will be less friable and eventually exhibit higher hardness. When comparing figure 1 and 2, the hypothesis earlier is justified. At inlet air temperature of 140 C, the highest percentage of weight loss exhibited by dragon fruit in figure 1 shows the lowest tensile strength in figure 2 which is 0.16%. Same goes with the lowest weight loss percentage exhibited by spinach shows the highest tensile strength in subsequent figure which is 0.78%. While, the very low weight loss percentage of carrot, mango and watermelon at inlet air temperature of 100 C shows a higher tensile strength in the subsequent figure respectively.

Meanwhile, there is no percentage of weight loss exhibited by most of freeze dried tablet except for spinach which account only tiny amount. Therefore, the hardness or tensile strength of all of freeze dried tablet is seen drastically high at figure 2 when compared with spray dried tablet. This is because the irregular shape of the powder formed where the more irregular the shapes of the particles, the poorer its flowability [14]. This may due to the compression during the tablet formation, where the irregular shape of the particles forming a high compaction and inter-particle friction. Thus, flowability is reduced and high in cohesiveness. Eventually, the tablet gain higher tensile strength and become harder than spray dried tablet.

3.3. Fiber Content Analysis

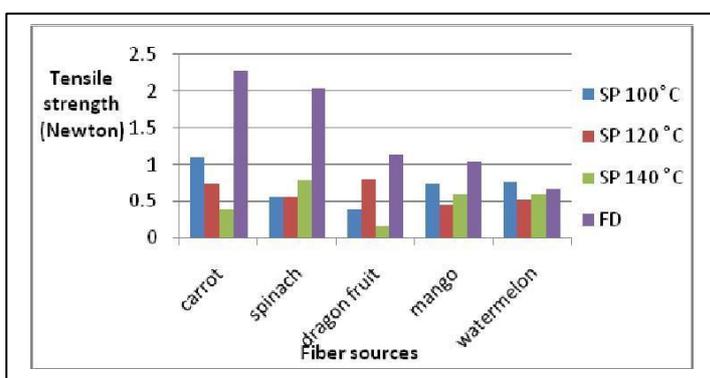


Figure 3. Fiber Content (%) vs Fiber sources

Theoretically, the highest fiber content of those five fiber source tablet is carrot, followed by mango, spinach, dragon fruit and watermelon. However, the results produced are not justifying the theory. This may be due to the procedure used in spray drying which the mixture of fiber and milk is filtered first before sucked by the equipment for powder formation. Based on figure 3, there is an inconsistent trend of fiber content through the increasing inlet air temperature of spray dryer for each of the fiber source powder. Only mango powder is justifying the theory of decreased fiber in increased inlet air temperature. Basically, total dietary fiber contains insoluble and soluble fiber. Insoluble fiber is insignificantly affected by the changes of inlet air temperature. Soluble fiber instead, is significantly changed as its amount decreases in increased temperature. This is because soluble fiber is comprised majorly of mucilage around 93% [16]. Mucilage will be degraded as early as 45 C as it is entering the glass transition temperature. Glass transition temperature is where an amorphous material transforms from hard and relatively brittle state into molten and rubber-like state in increasing temperature [17]. In other words, the mucilage started to degrade and disintegrate at its own temperature. Therefore, the fiber content of mango powder is decreasing at increasing inlet air temperature. Higher temperature can also reduce total dietary fiber as it disrupts the cellular matrix of the fiber source or it is called as thermal degradation [18].

For the other fiber source powder, like carrot it shows the lower fiber content at inlet air temperature of 120 C than 100 C and 140 C. While, spinach and dragon fruit shows increasing fiber content in increased inlet air temperature. Watermelon on the other hand shows quite insignificant changes among those temperatures. These trends are not justifying the previous theory maybe due to the age of the fiber source used. The content of soluble fiber decreases with age but insoluble fiber increases instead [16]. This shows how the alteration of fiber content which has been affected by the maturity of the fiber source used. For every inlet air temperature of spray drying procedure, the fiber source used are slightly not in the same maturity, therefore the final total dietary fiber would be varied between them. The fiber content gets somehow different for freeze dried method. Since freeze drying is not introducing high temperature toward the sample, hence no thermal degradation is occurred. As a result, the fiber content is found higher than spray dry method. This is shown obviously by mango and carrot powder in figure 3 which have 9.86% and 9.57% respectively. Dragon fruit and spinach have shown moderate and the lowest fiber content with 8.75% and 4.75% respectively. While watermelon shows insignificant changes between spray dry and freeze dry method with average 7.34%. In conclusion, there are many other factors that would not have been known that has significant impacts towards the findings of friability, hardness and fiber content analysis. Hence further studies need to be performed. The flowability of powder could be further analyzed with equipment like angle of repose, compressibility index, flow rate through an orifice and shear cell. Thus, these will prove and strengthen the support towards the findings of friability and hardness test. Same goes with the details fiber content of vegetables and fruits used. It is important to know how the different composition of elements under dietary fiber would affect the final fiber content.

4. Conclusion

In a nutshell, fiber enriched milk powder and tablet has varying characteristics in terms of friability, hardness and fiber content depending on the properties of the fiber itself. There is a huge range of knowledge about vegetables and fruits that need to be studied. Different fiber sources have different fiber structures, sugar content, carbohydrate content and moisture content. These relate with final fiber content, hygroscopicity, flowability and particle size of powder or tablet formed. Eventually it links with cohesiveness and interparticle strength within the powder. Based on findings, for freeze dried tablet, carrot is the hardest while for spray dried tablet, dragon fruit is the most friable at inlet air temperature of 140 C. On top of that, dragon fruit shows the highest fiber content at 140 C inlet air temperatures while mango is the highest fiber content for the freeze dried tablet.

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