Journal of Academia Vol. 12, Issue 2 (2024) 205 – 217

SPRAY-DRIED ENCAPSULATION OF BERGAMOT FLAVOUR BY BETA-CYCLODEXTRIN INCLUSION COMPLEXES

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Abstract
In the field of the food industry, a diverse range of flavours has been utilised in numerous food products, one of which is the bergamot flavour that has been employed to boost the infusion of both black and green tea, thereby introducing a delightful intricacy to the overall flavour profile of the tea. Nevertheless, due to its characteristic of having a low boiling point and being susceptible to degradation when subjected to heat, oxygen, and light, it becomes crucial to employ the technique of encapsulating the flavour through methods such as spray drying. This study focused on the analysis of spray-dried bergamot powder which evaluate its process yield, solubility, moisture content, hygroscopicity, bulk density, and tapped density. Furthermore, the objective of this study is to ascertain the best spray drying parameters for the encapsulation of taste that determines the ideal ratio of maltodextrin (MDs) and β-cyclodextrin (CDs), as well as the feed flow rate. The study's findings unveiled formula that was deemed the best powder which was run number 2 of 25% maltodextrin and 2% β-cyclodextrin, at a 10% feed flow rate. The percentage of the process yield, and the solubility of the powder were found to be 61.71% and 100%, respectively. The powder exhibits the lowest moisture content, measuring at 0.69, and also demonstrates the lowest hygroscopicity, with a value of 0.0409. Hence, the use of encapsulation by the spray drying technique is intriguing for the development of powdered particles with those mentioned ideal attributes whereas these particles can be utilised across several domains within the food processing sector.

Keywords: bergamot, flavour encapsulation, maltodextrin, β-cyclodextrin, spray drying

Article History:- Received: 1 July 2024; Revised: 3 July 2024; Accepted: 10 July 2024; Published: 31 October 2024

Introduction
Bergamot or *Citrus bergamia Risso et Poiteau*, is a member of the *Rutaceae* family (*Esperidea* subfamily) of plants which is a citrus fruit with a greenish yellow skin that is quite acidic and has a pleasant, sophisticated fragrance. Its primary production is bergamot essential oil, which is obtained by cold processing or steam distillation from the pericarp and a portion of the mesocarp of the fresh fruit (Berliocchi et al., 2011). Bergamot essential oil finds its primary application in the global perfumery industry, where it serves both as a fixative for fragrances' aromatic bouquets and an additive to other essences. Bergamot essential oil is widely used as a flavouring agent in foods and beverages, including teas, candies, and carbonated beverages. Linalool, citral, and linalyl acetate are responsible for most of the citrusy aroma and flavour of bergamot essential oils, while limonene and pinene contribute a small amount and degrade quickly in elevated temperatures and light (Navarra et al., 2015). On the other hand, the non-volatile portion of bergamot essential oil, constituting around 4-7% of the total composition, is composed of coumarins and furocoumarins (Dugo et al., 2000; Berliocchi et al., 2011). Thus, it is

essential for these compounds to go encapsulation process to extend the shelf life of it.

Encapsulation is a process of coating one substance or material over one material whereby it serves the purpose as a protection and preservation of bioactive, volatile, and easily degradable compounds due to biochemical and thermal deterioration (Saifullah et al., 2019). According to Sultana et al. (2018), encapsulation has the potential to boost the stability of taste in relation to heat, light, and oxygen. Additionally, it can also improve the control and flow properties of liquid flavour. This study focuses on the method of spray drying as a prominent technique for flavour encapsulation, among other methods such as freeze-drying, fluidized bed, and coacervation. Spray drying is a highly efficient and extensively utilised method for improving the stability of flavour and mitigating degradation by safeguarding flavouring compounds against oxidative and thermal degradation throughout the production stage (Premjit et al., 2022). Through undergoing this process, the bergamot flavour can exhibit an extended shelf life because of enhanced stability, rendering it suitable for utilisation in various food processing applications, including baking. Furthermore, the efficacy of the product is augmented using encapsulation, a process that facilitates the more convenient manipulation of variables and the regulated or stimulated release of flavours under specific circumstances, as shown by (Kim et al., 2019). Moreover, it is anticipated that the use of maltodextrin and β-cyclodextrin as wall materials will yield a powder with more advantageous characteristics, as compared to alternative combinations such as maltodextrin and gum Arabic, according to their respective qualities.

Spray drying has been used in many industrial processes for decades to obtain dehydrated materials in the form of fine powders and it is the most used encapsulation method in the food industry. According to Balci-Torun & Ozdemir (2021), the most common method for flavour encapsulation is spray drying because it can be used continuously, is simple to use, inexpensive, offers a variety of carrier materials, effectively retains volatile components, and produces stable products. By atomizing an emulsion into a hot gas chamber with a nozzle to produce a powder of particles, this technique ensures high-quality microparticles (Pellicer et al., 2019). Additionally, Saifullah et al. (2019) mentioned in a study that spray-dried flavour powder is utilised in a variety of food products to increase consumer acceptance of the product. The process of spray drying is illustrated in Figure 1 in which preparation of feed solution is the initial stage of this process. The preparation of feed solution is prepared by dissolving core materials in the wall materials to form an emulsion in which the wall materials can be a single or combined wall material such as a combination of maltodextrin and β-cyclodextrin. Since only water-based dispersions are effective for spray drying, the dispersed wall materials must be water-soluble. To form a coarse emulsion, core materials and wall materials can be mixed using high-speed mixing or high-shear emulsification using colloid mills. The emulsion can then be processed further using various mechanical methods such as high-pressure homogenization, micro fluidization, and ultrasonic emulsification. As the feed solution is ready, the process is continued to the three main stages whereby the first stage is the atomization of feed sample, followed by drying of liquid droplets within drying chamber in stage two and lastly the powder collection through cyclone separator as mentioned by Saifullah et al. (2019). All these stages including the operational parameters are directly affect the characteristics of the final product which is the spray-dried powder.

The initial phase of spray drying, as previously indicated by Saifullah et al. (2019), involves atomization. The emulsion, which has been established to possess stability, is afterwards employed as a feed sample for introduction into the drying chamber by atomization (Saifullah et al., 2019). The process of atomization can be achieved through the utilisation of several atomizers, including pneumatic atomizers, pressure nozzles, and fluid nozzles. Among these atomizers, the high-pressure nozzle and centrifugal wheel are the most frequently employed varieties (Estevinho & Rocha, 2017). The atomization process entails the breakdown of the fluid supply (emulsion) into droplets of consistent size, hence facilitating the even distribution of heat and mass transfer during the drying phase. The process of atomization also serves to enhance the surface area of the feed and facilitate uniform dispersion of the feed within the drying chamber (Mohammed et al., 2020). After the liquid feed is broken down into small droplets by the atomizer, the small droplets are sprayed into hot air in the drying chamber. The heat from the hot air will be transferred to the droplets as it is converted to latent heat during the evaporation of moisture content (Mohammed et al., 2020). The size of the powder particle is determined by the pore size of the atomizer. In the final stage, the encapsulated dry particles are separated by a cyclone separator and deposited in a receiver. The uses of cyclone separator in the final step as in dairy industry is intended for gas-solids separation, gas-liquid separation, and solid-liquid separation. Gas-liquid separation is seen to separate product fines from leaving the spray dryer into the air thus increasing the yield and reduce air pollution (Gupta et al., 2016). Additionally, Mohammed et al. (2020) stated the final product of spray drying is obtained as the liquid emulsion is transformed into fine powder that is in the range of 10 to 100 micrometres.

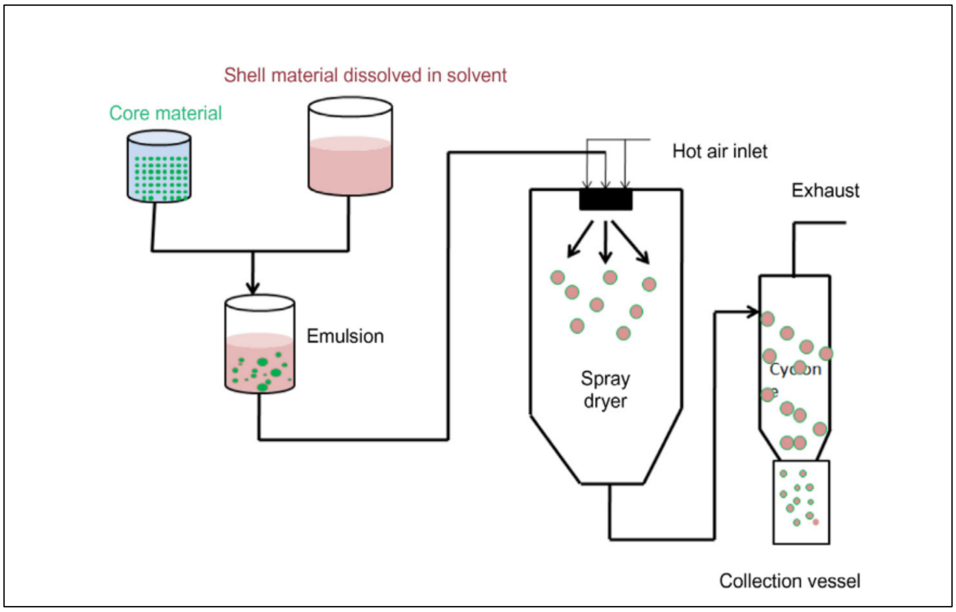

Figure 1. Spray drying process.

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In other way, the selection of wall materials plays a vital effect for the whole encapsulation process as the wall materials are supposed to protect the core material from external influences by acting as a barrier and prevent it from reacting with any other component. Balci-Torun & Ozdemir (2021) also mentioned the selected wall material must have greater emulsion stability, lower viscosity at high concentrations, the ability to create films and the release of flavour at the right time and place. These wall materials which are maltodextrin, Arabic gum, modified starch, whey protein and cyclodextrins are the common wall materials used in encapsulation. A combination of maltodextrin and β-cyclodextrins were used as the wall materials in this study to accommodate the desired properties of wall material. Maltodextrin is a common wall material used in flavour encapsulation due to its properties that can provide excellent encapsulant for the core material. However, maltodextrin cannot be used as a single wall material due to its low emulsifying ability hence it is combined with another wall material such as β-cyclodextrins (Pellicer et al., 2018). The property of β-cyclodextrins which can stabilise emulsion of fats and oils in can complement that lacking aspect of maltodextrin as mentioned by Premjit et al. (2022). Other than that, β-cyclodextrin is chosen due to its capabilities to stabilise and protect certain sensitive colours and unique flavours during the food processing steps such as microwaving and thawing thus greater extent and longer period of these qualities than α-cyclodextrin and γ-cyclodextrin (Zhou et al., 2022). A unique structure of cyclodextrin with a hydrophobic cavity and a hydrophilic outer surface allows them to form a stable non-covalent inclusion complex with flavour molecules and can be by co-precipitation, kneading, slurry complexation, spray-drying and freeze-drying techniques (Xiao et al., 2019).

This study aims to examine the integration of bergamot flavour encapsulation within the framework of the Sustainable Development Goals (SDGs), with a specific focus on SDG 9, which pertains to Industry, Innovation, and Infrastructure. Based on the findings of the Sustainable Development Report (2022), Malaysia is making progress towards achieving the Sustainable Development Goals (SDGs), with indications of moderate improvement in this pillar. Hence, the encapsulation of bergamot flavour emerges as a significant factor in driving the positive trends observed in SDG 9. This method aims to enhance the shelf life, stability, efficiency of bergamot flavour, thereby offering substantial benefits to the food industry. Consequently, it is anticipated that there will be an increased interest in the encapsulation of this flavour.

Flavo Blitz Sdn. Bhd., a leading company in the flavour manufacturing industry, is currently facing a challenge in determining the optimal physical attributes of its bergamot flavour that align with the preferences of their clients. Moreover, there exists a lack of data regarding the efficacy of the combination of wall materials, specifically maltodextrin and β-cyclodextrin, in the process of encapsulating bergamot flavour, as well as the characteristics of spray-dried bergamot flavour. Hence, the present study was conducted in collaboration between Flavo Blitz Sdn. Bhd. and UiTM (Universiti Teknologi MARA) Kuala Pilah to assess the efficacy of different combinations of wall materials on the stability of spray-dried bergamot flavour. Additionally, the study aimed to identify the best ratio of wall materials that would yield the desired characteristics of spray-dried bergamot flavour.

Methods

Material

The 1 L packages of synthetic liquid bergamot flavour and β-cyclodextrin (β-CD) were acquired from Flavo Blitz Sdn. Bhd. in Pulau Pinang, Malaysia. Additionally, maltodextrin (MD) with dextrose equivalent (DE) of 20 was procured from local chemical supplier namely EvaChem in Selangor, Malaysia for the purpose of serving as coating materials in the encapsulation process.

Mixture formation

A mixture of maltodextrin (DE=20) and β-cyclodextrin was formed according to the ratio suggested by Response Surface Methodology (RSM) of Box Behnken design in Design Expert 13 tabulated on Table 1. The combination of maltodextrin (DE=20) and β-cyclodextrin were completely dissolved in 200 mL distilled water, which is 60% of whole solution. Then, the solution was added with 20 ml of synthetic bergamot flavour (10% of dry weight of wall materials). The obtained mixture was homogenized at 350 rpm for 3 minutes by an ultrasonic homogenizer (Pro, SC-250, USA).

Table 1. Ratio of maltodextrin concentration and β-cyclodextrin

Run	Feed flow %	MD concentration %	β-CD concentration %
1	10	20	2
2	10	25	2
3	40	20	2
4	40	22.5	3
5	10	22.5	3
6	25	22.5	2
7	25	25	3
8	10	22.5	1
9	25	22.5	2
10	40	22.5	1
11	25	20	3
12	40	25	2
13	25	25	1
14	25	20	1
15	25	22.5	2

Spray drying process

The entire process of converting the solution into powder was performed by using a laboratory scale spray dryer (BUCHI Mini Spray Dryer, B-290, Switzerland). The homogenized mixture was pumped into the main chamber of spray dryer through a peristaltic pump in which the pump rotation speed was controlled as the flow rate of the mixture. As for this process, the pump rotation speed applied was expressed in percent (%) whereas the speed used were 10%, 25% and 40%. For the inlet temperature, the temperature was controlled at 175°C and the outlet temperature was dependent on the inlet temperature as well with the temperature at the surrounding, which the range was in between 93℃ to 118℃.

Process yield

The obtained powder from the spray drying process was calculated for the process yield determination for each run. According to Pellicer et al. (2019), the process yield was calculated by dividing the weight of obtained powder to the total solid content used in the mixture, by using the following equation:

$$Yield\ (\%) = \frac{weight\ of\ obtained\ powder\ (g)}{total\ solid\ content\ used\ in\ mixture\ (g)} \times 100\%$$

Moisture content

The moisture content of 15 samples of bergamot powder were determined by using an infrared moisture analyser as stated by Sultana et al. (2018) in which an amount of 1 g of the bergamot powder was spread on an aluminium dish and was analysed for 13 minutes for each run. For each sample, the readings were duplicate to get the mean reading of the moisture content.

Hygroscopicity

An even spread of 1 g of bergamot powder in a petri dish was put in a desiccator that containing 10 ml nitric acid (HNO_3). The samples were weighed after one week, and the hygroscopicity was quantified as grammes of absorbed moisture per 100 g of dry solids (g/100 g) (Yamashita et al., 2017).

Powder solubility

According to Balci-Torun & Ozdemir (2021), 50 ml of distilled water was mixed with a 0.50 ± 0.01 g powder sample. The mixture then was magnetically agitated for 5 minutes at 600 rpm. The solution was centrifuged by using a centrifuge (KUBOTA, 2420, Japan)) for a total of 5 minutes at 3000 g. After that, 25 mL of the supernatant from centrifugation was put onto a petri dish and kept at 70°C until the weight is constant. By calculating the difference in weight, the particle solubility in percent was obtained.

Bulk and tapped density

Bulk density (ρb) and tapped density (ρt) was determined using the weight/volume ratio and results are presented in kg/m^3 (Balci-Torun & Ozdemir, 2021). An amount of 2 g of bergamot powder was put in a 10 ml graduated cylinder and was beaten 20 times on a hard surface. The volume of powder before and after beaten were taken and the bulk and tapped density was calculated.

Statistical Analysis

The experimental sequence obtained through Box Behnken Design was randomized to minimize the effects of the uncontrolled parameters. The results were analysed through the Design Expert 13 of response plots and analysis of variance (ANOVA).

Result and Discussion

Process yield

The process effectiveness in the industrial sector, particularly regarding manufacturing cost, often relies on process yield as a significant criterion (Balci-Torun & Ozdemir, 2021). The yields of bergamot-flavoured powder exhibited a range of 41.54% to 74.20%, as indicated in Table 2. Furthermore, 50% of the 15 experimental trials resulted in yields surpassing the 50% threshold. Based on the findings of Balci-Torun & Ozdemir (2021) it is established that a laboratory scale spray dryer must achieve a minimum yield rate of 50% to be considered successful. Consequently, it can be inferred that the bergamot spray drying process under study meets the criteria for success, thereby indicating the viability of employing a spray drying technique for the encapsulation of bergamot flavour.

Table 2. Process yields of bergamot flavoured powder

Run	Feed flow %	MD concentration %	β -CD concentration %	Process yield %
1	10	20	2	70.23
2	10	25	2	61.71
3	40	20	2	46.80
4	40	22.5	3	41.54
5	10	22.5	3	74.20
6	25	22.5	2	54.36
7	25	25	3	56.39
8	10	22.5	1	68.51
9	25	22.5	2	54.27
10	40	22.5	1	49.10
11	25	20	3	53.10
12	40	25	2	47.89
13	25	25	1	47.47
14	25	20	1	45.54
15	25	22.5	2	47.34

According to the data presented in Table 2, the maximum yield rate achieved is 74.20% when employing a combination of 22.5% maltodextrin and 3% β-cyclodextrin, along with a feed flow rate of 10%. Conversely, the minimum yield rate observed is 41.54% when utilising the same proportions of maltodextrin and β-cyclodextrin, but with a pump setting of 40%. Based on the obtained results, it was observed that run 5 exhibited the highest yield of 74.20%, whereas run 4 demonstrated the lowest yield of 41.54%. Both experimental runs were conducted using a mixture of 22.5% MD and 3% β-CD at varying feed flow rates. This observation implies that the flow rate may have had an impact on the overall process yield in the context of the spray drying procedure. Insufficient atomization can result from the utilisation of a higher feed flow rate, resulting in the adhesion of powder particles within the cyclone and consequently limiting the quantity of powder that can be collected. This study has shown that utilising a feed flow rate within the range of 10% to 25% results in an increased process yield than the 40% flow rate. It is evident that nearly all the experimental runs that employed the mentioned flow rate necessitate a process yield beyond 50%. Barbieri et al. (2018) provided support for this assertion, positing that the diverse process yield can be influenced by the composition of the wall material, particularly the core ratio and drying temperature. As evidenced by previous research, it has been observed that a minimum of 10% of MD can result in a yield exceeding 50%, and a greater proportion of MD can further enhance the yield of the process. However, it should be noted that a high proportion of MD, specifically at levels of 30% or 50%, can lead to a decrease in flavour perception (Bhandari, 1997; Balci-Torun & Ozdemir, 2021).

Moisture content

According to Sanchez-Reinoso et al. (2017), the presence of high humidity can lead to various problems during storage, such as particle adhesion, clumping, and oxidation of encapsulated powder. Therefore, the moisture content of powders plays a crucial role in determining their organoleptic quality and shelf life. The powder infused with bergamot flavour exhibits a moisture content that varies between 0.69 and 3.75%, as depicted in Table 3. Based on the findings, it can be observed that the moisture content of the bergamot spray-dried powders fell below the microbial spoilage threshold typically observed in dried powders utilised in the food industry, which typically ranges from 3% to 4%. This aligns with the results of a different encapsulation study conducted by Sanchez-Reinoso et al. (2017), which also identified a moisture content range of 1.96% to 2.54% as being within the safe parameters. The moisture content of the powder exhibited a positive correlation with the feed flow rate, indicating that as the feed flow rate increased, the moisture content of the powder also increased.

Table 3. Moisture content of bergamot flavoured powder

Run	Feed flow %	MD concentration %	β-CD concentration %	Moisture content (%)
1	10	20	2	0.83
2	10	25	2	0.69
3	40	20	2	3.04
4	40	22.5	3	3.39
5	10	22.5	3	0.99
6	25	22.5	2	2.92
7	25	25	3	1.76
8	10	22.5	1	1.04
9	25	22.5	2	2.35
10	40	22.5	1	3.15
11	25	20	3	1.58
12	40	25	2	3.75
13	25	25	1	1.59
14	25	20	1	1.77
15	25	22.5	2	3.13

The blend consisting of 25% maltodextrin and 2% β-cyclodextrin demonstrated the lowest moisture content (0.69) when subjected to a feed flow rate of 10%. Conversely, the blend comprising of 25% maltodextrin and 2% β-cyclodextrin exhibited the highest moisture content (3.75) under a feed flow rate of 40%. The efficiency of heat transfer is negatively affected by a high rate of feed flow due to a reduction in the duration of contact between the feed and the drying air. This reduction in contact time also influences the intensity of evaporation, leading to a decrease in the temperature of the inlet air and an increase in the moisture content of the powder (Pui & Saleena, 2022). Furthermore, the moisture content is affected by the proportion of maltodextrin and β-cyclodextrin utilised, particularly when maltodextrin is employed in significant quantities. This study utilised a combination of high maltodextrin with varying percentages of β-cyclodextrin as wall materials. It was shown that combining maltodextrin with 1% and 2% of β-cyclodextrin resulted in lower moisture content. The pattern is not adhered to when it comes to the combination of 3% of β-cyclodextrin. Figure 2 shows the effect of maltodextrin and feed flow rate on moisture content. Higher maltodextrin deployed lower moisture content at low feed flow rate as explained by Xiao et al. (2022) in a study that using high maltodextrin can lead to lower moisture content in powder because of its low hydrophilic groups, which bind fewer water molecules. In a previous study done by Nadali et al. (2022), it has been suggested that a lower DE of maltodextrin is a more effective binding agent than a higher one thereby validating the obtained result.

As illustrated in Table 4, the Model F-value of 18.59 indicates that the model is statistically significant. There is a low probability of 0.25% that such a large F-value could be attributed to random noise. Thus, the statistical analysis revealed that the feed flow rate had a significant impact ($p < 0.05$) on the moisture content of the powder. However, this study did not yield any significant findings related to the other aspects being tested *i.e.* process yield, hygroscopicity, powder solubility as well as bulk and tapped density.

Table 4. ANOVA table for moisture content using Box Behnken Design

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	14.49	9	1.61	18.59	0.0025	significant
A-Feed flow rate	12.03	1	12.03	138.93	< 0.0001	
B-Maltodextrin	0.0392	1	0.0392	0.4527	0.5309	
C-Beta-cyclodextrin	0.0028	1	0.0028	0.0325	0.8641	
AB	0.1892	1	0.1892	2.19	0.1994	
AC	0.0028	1	0.0225	0.2599	0.6319	
BC	0.0342	1	0.0342	0.3953	0.5571	
A^2	0.0592	1	0.0592	0.6842	0.4458	
B^2	1.30	1	1.30	15.05	0.0116	
C^2	1.02	1	1.02	11.38	0.0185	
Residual	0.4329	5	0.0866			
Lack of Fit	0.1161	3	0.0387	0.2442	0.8612	not significant
Pure Error	0.3169	2	0.1584			
Cor Total	14.92	14				

Note: df: Degree of freedom, ANOVA: Analysis of variance

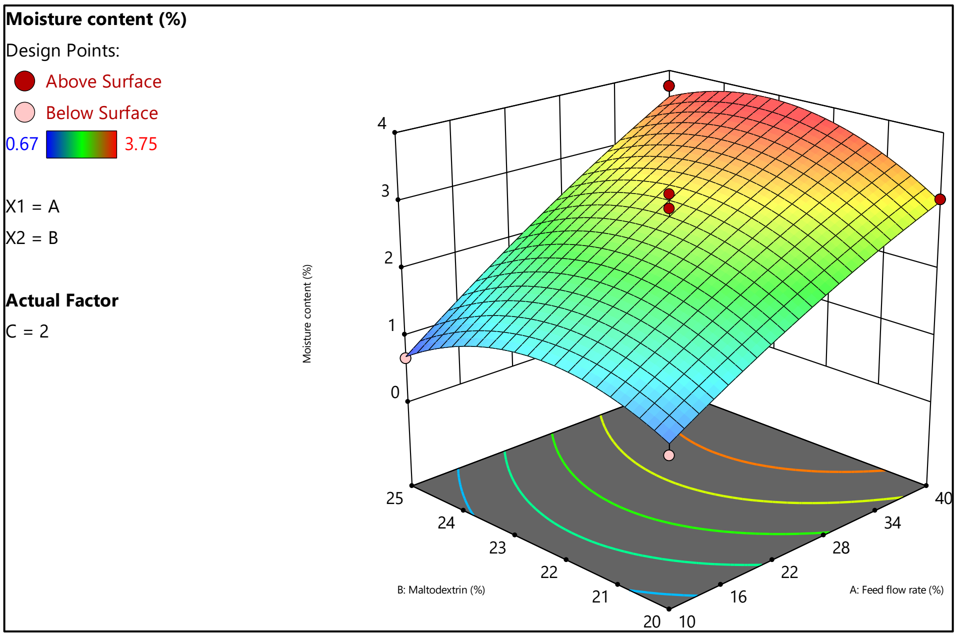

Figure 2. 3D surface plot of moisture content

Hygroscopicity
Table 5 presents the hygroscopicity of spray-dried bergamot powder. It is observed that the hygroscopicity of the powder changes, which is a common phenomenon in spray-dried powders. Specifically, it is noted that the hygroscopicity tends to rise with longer storage durations under higher humidity conditions, as reported by Bashir et al. (2023).

Table 5. Hygroscopicity of bergamot flavoured powder

Run	Feed flow %	MD concentration %	β-CD concentration %	Hygroscopicity
1	10	20	2	0.0416
2	10	25	2	0.0409
3	40	20	2	0.0437
4	40	22.5	3	0.045
5	10	22.5	3	0.041
6	25	22.5	2	0.0429
7	25	25	3	0.0422
8	10	22.5	1	0.0412
9	25	22.5	2	0.0427
10	40	22.5	1	0.0431
11	25	20	3	0.042
12	40	25	2	0.0439
13	25	25	1	0.0413
14	25	20	1	0.0426
15	25	22.5	2	0.0425

In this study, a powder with low hygroscopicity was produced, exhibiting a range of 0.0409 to 0.045. The powder's low hygroscopicity was attributed to the use of maltodextrin with a dextrose equivalent (DE), while maltodextrin with a DE of 20 was employed as the encapsulant. The findings of Ghani et al. (2017) and Xi et al. (2020) align with this conclusion, as they assert that spray drying processes exhibit advantageous characteristics when employing high DE values. These values promote the formation of homogeneous matrices during the drying process, thereby enhancing the retention rate and yielding a powder with reduced hygroscopicity. These factors collectively contribute to the stability of the core components. Furthermore, the presence of maltodextrin in the powder was found to be a contributing factor to its hygroscopic nature. Specifically, when a 10% maltodextrin concentration was used, the hygroscopicity of honeydew melon powder decreased by approximately 30% from 29.51 ± 1.42% to 20.56 ± 0.79% (Chang et al., 2022). The obtained results were a result of the properties exhibited by maltodextrin which is a substance known for its low hygroscopic nature. Consequently, maltodextrin has been extensively employed as a carrier agent during the spray drying procedure. The researchers noticed that the bergamot particles exhibited limited moisture absorption from the ambient humidity generated by nitric acid (HNO_3) solutions. As a result, these powders were classified as low-hygroscopic.

Powder solubility
The solubility of a powder in water is a fundamental parameter that must be considered when evaluating the behaviour of a powdered product in an aqueous solution. The solubility of a substance is a measure of its ability to dissolve and form a solution or suspension (Balci-Torun & Ozdemir, 2021). Consequently, the complete solubility of the bergamot spray-dried powder in water can be attributed to the use of maltodextrin and β-cyclodextrin as the encapsulating agents during the spray drying procedure. Based on the findings of Alvarenga et al. (2017) and Balci-Torun & Ozdemir (2021), it has been demonstrated that carbohydrate-based encapsulants, such as maltodextrin and Arabic gum, have a propensity to enhance solubility. Conversely, protein-based carriers, such as gelatine and soy protein isolate, have been observed to impede resolution.

Table 6. Solubility of bergamot flavoured powder

Run	Feed flow %	MD concentration %	B-CD concentration %	Solubility (%)
1	10	20	2	100
2	10	25	2	100
3	40	20	2	100
4	40	22.5	3	100
5	10	22.5	3	100
6	25	22.5	2	100
7	25	25	3	100
8	10	22.5	1	100
9	25	22.5	2	100
10	40	22.5	1	100
11	25	20	3	100
12	40	25	2	100
13	25	25	1	100
14	25	20	1	100
15	25	22.5	2	100

Maltodextrin, which was used as the wall material in this study is recognised for its notable attribute of solubility in aqueous solutions, owing to the hydroxyl group present in its molecular structure, which facilitates its interaction with water upon contact. According to a study conducted by Ningsih et al. (2019) the inclusion of high-loading groups in amino acids enables these groups to exhibit solubility in polar solvents like water and ethanol. Consequently, this characteristic contributes to the complete solubility of bergamot spray dried powder for all runs as presented in Table 6. The study conducted by Archaina et al. (2018) demonstrated that a high degree of solubility, specifically 94.25%, was achieved in blackcurrant spray-dried powder when 40% w/w of MD was utilised. This conclusion aligns with the results obtained in another study involving spray-dried powder. Numerous studies have demonstrated that the solubility of spray-dried powder can be enhanced through the manipulation of various factors, including the product feed rate, atomization size, and air intake and outlet temperatures. According to Muzaffar et al. (2015); Balci-Torun & Ozdemir (2021) the solubility of the product is inversely proportional to the moisture content, which can be reduced by increasing the product feeding rate, decreasing the atomization speed, and increasing the air inlet temperature.

Bulk and tapped density

The bulk and tapped density of bergamot spray-dried powder are presented in Table 7. The powder achieved its highest bulk density which is 387.63 when composed of 20% maltodextrin and 2% β-cyclodextrin, with a feed flow rate of 10%. Conversely, the lowest bulk density which is 304.77 was observed under conditions of 22.5% maltodextrin and 3% β-cyclodextrin, with a feed flow rate of 40%. Specifically, it was observed that an increase in the feed flow rate resulted in a decrease in the bulk density compared to a lower feed flow rate. Powder particles possessing a higher bulk density have the advantage of being transportable in smaller volume packaging in comparison to particles characterised by a lower bulk density. Simultaneously, it has been mentioned by Balci-Torun & Ozdemir (2021) that smaller packaging materials may accommodate a greater number of powder particles. Nevertheless, the present study observed much greater bulk and tapped density compared to the previous investigation conducted by Balci-Torun & Ozdemir (2021). Specifically, the bulk and tapped density values for the spray dried flavour powders in their study were reported as 306 and 350 kg/m3, respectively. The elevated values found in this study could potentially be attributed to the limited expansion experienced by these particles during the drying process, as evidenced by their rough surface texture. This roughness contributes to their increased weight, resulting in a bigger overall mass of the powders (Alvarenga et al., 2017).

Table 7. Bulk and tapped density of bergamot flavoured powder

Run	Feed flow %	MD concentration %	β-CD concentration %	Bulk density	Tapped density
1	10	20	2	387.63	430.69
2	10	25	2	358.7	409.94
3	40	20	2	309.1	381.79
4	40	22.5	3	304.77	372.5
5	10	22.5	3	374.3	415.34
6	25	22.5	2	344.52	413.43
7	25	25	3	329.06	381.06
8	10	22.5	1	350.76	391.49
9	25	22.5	2	312.45	367.59
10	40	22.5	1	306.61	371.64
11	25	20	3	357.98	417.65
12	40	25	2	307.83	384.78
13	25	25	1	351.05	421.26
14	25	20	1	345.87	413.43
15	25	22.5	2	316.97	369.81

Conclusion

This study aimed to discover the best spray drying conditions for the encapsulation of taste, with a specific focus on the ratio of maltodextrin (MDs) and β-cyclodextrin (CDs). The optimal spray drying conditions were identified through comprehensive evaluations, encompassing process yield, moisture content, solubility, hygroscopicity, bulk density, and tapped density. The spray drying parameters that resulted in the highest efficiency were found with concentration of 25% MDs, 2% β-CDs, and a feed flow rate of 10% which was found at run number 2. The powder exhibited excellent solubility, achieving a process yield of over 50%. It possessed a minimal moisture content, displayed hygroscopic properties, and indicated optimal values for both bulk and tapped density.

Acknowledgement/Funding

The authors wish to express their profound gratitude to Universiti Teknologi MARA Kuala Pilah and Flavo Blitz Sdn. Bhd. for their generous provision of financial support, which has made this groundbreaking project possible. This remarkable endeavour has been made feasible through the esteemed University Research Grant Scheme (100-TNCPI/PRI 16/6/2 (022/2023) and 600-IRMI from CRSF2022.

Author Contribution

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Conflict of Interest

Authors declare no conflict of interest.

References

Archaina, D., Leiva, G., Salvatori, D., & Schebor, C. (2018). Physical and functional properties of spray-dried powders from blackcurrant juice and extracts obtained from the waste of juice processing. *Food Science and Technology International*, *24*(1), 78–86. https://doi.org/10.1177/1082013217729601

Balci-Torun, F., & Ozdemir, F. (2021). Encapsulation of strawberry flavour and physicochemical characterization of the encapsulated powders. *Powder Technology*, *380*, 602–612. https://doi.org/10.1016/j.powtec.2020.11.060

Barbieri, N., Sanchez-Contreras, A., Canto, A., Cauich-Rodriguez, J. V., Vargas-Coronado, R., & Calvo-Irabien, L. M. (2018). Effect of cyclodextrins and Mexican oregano (Lippia graveolens Kunth) chemotypes on the microencapsulation of essential oil. *Industrial Crops and Products*, *121*, 114–123.

https://doi.org/10.1016/j.indcrop.2018.04.081

Berliocchi, L., Ciociaro, A., Russo, R., Gilda, M., Cassiano, V., Blandini, F., Rotiroti, D., Antonio, L., & Tiziana, M. (2011). Toxic profile of bergamot essential oil on survival and proliferation of SH-SY5Y neuroblastoma cells. *Food And Chemical Toxicology*, *49*(11), 2780–2792. https://doi.org/10.1016/j.fct.2011.08.017

Chang, L. S., Ooi, Y. W., & Pui, L. P. (2022). Production of enzymatic hydrolysed spray-dried honeydew melon (Cucumis melo L.) powder. *Journal of Agriculture and Food Research*, *10*, 100364. https://doi.org/10.1016/j.jafr.2022.100364

Estevinho, B. N., & Rocha, F. (2017). A Key for the Future of the Flavors in Food Industry: Nanoencapsulation and Microencapsulation. In *Nanotechnology Applications in Food: Flavor, Stability, Nutrition and Safety*. Academic Press: pp. 1-19. https://doi.org/10.1016/B978-0-12-811942-6.00001-7

Gupta, S., Khan, S., Muzafar, M., Kushwaha, M., Yadav, A. K., & Gupta, A. P. (2016). Encapsulation: entrapping essential oil/flavors/aromas in food. In *Encapsulations*. Academic Press: pp.229-268. https://doi.org/10.1016/b978-0-12-804307-3.00006-5

Kim, E. H. J., Paredes, D., Motoi, L., Eckert, M., Wadamori, Y., Tartaglia, J., Green, C., Hedderley, D. I., & Morgenstern, M. P. (2019). Dynamic flavor perception of encapsulated flavors in a soft chewable matrix. *Food Research International*, *123*, 241–250. https://doi.org/10.1016/J.FOODRES.2019.04.038

Mohammed, N. K., Tan, C. P., Manap, Y. A., Muhialdin, B. J., & Hussin, A. S. M. (2020). Spray Drying for the Encapsulation of Oils—A Review. *Molecules*, *25*(17), 3873. https://doi.org/10.3390/molecules25173873

Nadali, N., Pahlevanlo, A., Sarabi-Jamab, M., & Balandari, A. (2022). Effect of maltodextrin with different dextrose equivalents on the physicochemical properties of spray-dried barberry juice (Berberis vulgaris L.). *Journal of Food Science and Technology*, *59*(7), 2855–2866. https://doi.org/10.1007/s13197-021-05308-w

Navarra, M., Mannucci, C., Delbò, M., & Calapai, G. (2015). Citrus bergamia essential oil: From basic research to clinical application. *Frontiers in Pharmacology*, *6*, 36. https://doi.org/10.3389/fphar.2015.00036

Ningsih, R., Sudarno, & Agustono. (2019). The Effect of Maltodextrin Concentration on the Characteristics of Snappers' (Lutjanus sp.) Peptone. *IOP Conference Series: Earth and Environmental Science*, *236*(1), 012127. https://doi.org/10.1088/1755-1315/236/1/012127

Pellicer, J. A., Fortea, M. I., Trabal, J., Rodríguez-López, M. I., Carazo-Díaz, C., Gabaldón, J. A., & Núñez-Delicado, E. (2018). Optimization of the microencapsulation of synthetic strawberry flavour with different blends of encapsulating agents using spray drying. *Powder Technology*, *338*, 591–598. https://doi.org/10.1016/j.powtec.2018.07.080

Pellicer, J. A., Fortea, M. I., Trabal, J., Rodríguez-López, M. I., Gabaldón, J. A., & Núñez-Delicado, E. (2019). Stability of microencapsulated strawberry flavour by spray drying, freeze drying and fluid bed. *Powder Technology*, *347*, 179–185. https://doi.org/10.1016/j.powtec.2019.03.010

Premjit, Y., Pandhi, S., Kumar, A., Rai, D. C., Duary, R. K., & Mahato, D. K. (2022). Current trends in flavor encapsulation: A comprehensive review of emerging encapsulation techniques, flavour release, and mathematical modelling. *Food Research International*, *151*, 110879. https://doi.org/10.1016/J.FOODRES.2021.110879

Saifullah, M., Shishir, M. R. I., Ferdowsi, R., Tanver Rahman, M. R., & Van Vuong, Q. (2019). Micro and nano encapsulation, retention and controlled release of flavor and aroma compounds: A critical review. *Trends in Food Science and Technology*, *86*, 230–251. https://doi.org/10.1016/j.tifs.2019.02.030

Sultana, A., Tanaka, Y., Fushimi, Y., & Yoshii, H. (2018). Stability and release behavior of encapsulated flavor from spray-dried Saccharomyces cerevisiae and maltodextrin powder. *Food Research International*, *106*, 809–816. https://doi.org/10.1016/j.foodres.2018.01.059

Xiao, Z., Hou, W., Kang, Y., Niu, Y., & Kou, X. (2019). Encapsulation and sustained release properties of watermelon flavor and its characteristic aroma compounds from γ-cyclodextrin inclusion complexes. *Food*

Hydrocolloids, *97*, 105202. https://doi.org/10.1016/j.foodhyd.2019.105202

Xiao, Z., Xia, J., Zhao, Q., Niu, Y., & Zhao, D. (2022). Maltodextrin as wall material for microcapsules: A review. *Carbohydrate Polymers*, *298*, 120113. https://doi.org/10.1016/j.carbpol.2022.120113

Yamashita, C., Chung, M. M. S., dos Santos, C., Mayer, C. R. M., Moraes, I. C. F., & Branco, I. G. (2017). Microencapsulation of an anthocyanin-rich blackberry (Rubus spp.) by-product extract by freeze-drying. *Lwt*, *84*, 256–262. https://doi.org/10.1016/j.lwt.2017.05.063

Zhou, J., Jia, J., He, J., Li, J., & Cai, J. (2022). Cyclodextrin Inclusion Complexes and Their Application in Food Safety Analysis: Recent Developments and Future Prospects. *Foods*, *11*(23), 3871. https://doi.org/10.3390/foods11233871