An Overview of Process Parameters and Spray drying agents involved in Spray drying of Herbal Extracts

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Abstract- Spray drying of herbal extract is important in pharmaceutical and food industries for obtaining powder with better physical properties, and stability of active ingredients. The inlet air temperatures, Feed flow rate and spray drying agent’s concentrations are the variables which affect percentage yield, moisture content, bulk density and hygroscopicity of the powder obtained by spray drying. Interdependence of spray drying process variables are discussed in this article for spray drying of herbal extracts. The stickiness of extracts, wall deposition and low percentage yield are major problems associated with spray drying process. Various ingredients like aerosol, maltodextrin are used as spray drying agents. Advantages and limitations of various spray drying agents used for herbal extracts are reviewed in this article. Powder can be obtained as per required specifications by understanding effect of additives on processing parameter of spray drying.

Keywords – Process parameters, Spray drying, Herbal, Temperature, Variables.

INTRODUCTION:
The development of spray drying equipment and techniques evolved over a period of several decades from the 1870s through the early 1900s. Spray drying comes of age during World War II, with the sudden need to reduce the transport weight of foods and other materials. Spray drying is a conventional method involves low cost in comparison to other method. Spray drying is a drying method that produces droplets of liquid feed into powdered products. The conversion involved atomization of liquid feed, undergoes heat treatment for rapid evaporation of moisture. The spray drying process converts liquids to powders with some but at an acceptable level of degradation and oxidation of volatile compounds. Spray drying is based on the preparation, homogenization, atomization, dispersion and subsequently dehydration of the solution. The gas generally used is air or more rarely an inert gas, particularly nitrogen gas. It increases powder stability and resistant to microbiological and oxidative degradation. Commercialization of spray drying process depends on parameters like process yield, end product characteristic and production cost. Few articles are published on the effectiveness of additives of spray drying optimization, in which there is no clear linear relationship between additives and spray drying parameters has been identified. The atomizing stage must create a spray for optimum evaporation conditions in order to achieve an economic production of the desired products. Spray-air contact is determined by the position of the atomizer in relation to the drying air inlet. In a co-current flow design, spray evaporation is rapid and as the drying air cools, accordingly the evaporation time is shortened. The product is not subject to heat degradation.

1. Basic steps in spray drying process
Basic steps in spray drying are shown in Fig.1.

1.1. Concentration of herbal extract
Generally, extract is concentrated before introducing into the spray dryer. The concentrated extract has increased in solid contents thereby reducing the amount of liquids which is evaporated in the spray dryer. In conventional large scale spray dryer normally extract is concentrated to 50%-60% before introducing to spray dryer. However, the
small scale laboratory spray dryer will have more diluted extract because it will be clogged easily if the feed have high viscosity. 

1.2. Atomization
Atomization refers to the conversion of bulk liquid into a spray or mist, often by passing the liquid through a nozzle. The liquid which sprayed through nozzle will increase the surface area of the liquid which later will be contacted to hot air and dried into a powder. The nozzle size may differ according to the size of spray dryer. Droplet size ranges from 20 μm to 180 μm and it depends on the nozzle. Smaller spray dryer occupies smaller nozzles and reverse in the industrial scale spray dryer. The aim of this stage is to create a maximum heat transferring surface between the dry air and the liquid, in order to optimize heat and mass transfers. The choice upon the atomizer configuration depends on the nature and viscosity of feed and desired characteristics of the dried product. The atomizers/sprayers are of three basic types.

a. Pressure atomizer / nozzles
The distribution and size of the particles are related to the size of droplets formed by the aspersion process. In the pressure atomizer, the material is pumped into the nozzle at a high pressure and it is forced to pass through a very narrow orifice. Therefore, the use of special high pressure pumps and abrasion resistant material are essential in the composition of the nozzle. From an energy point of view, the pressure atomizers are the most economic, however these tend to suffer from clogging.

![Diagram of spray drying process](image-url)
b. Pneumatic fluid / Double piston atomizer
In the pneumatic fluid atomizer a lower spray pressure is applied to the input. In this case, the liquid material is ruptured by the shearing force generated by the difference in speed between both fluids: air and product. This system has a higher energy demand. It is used due to its versatility, and high control of the size and uniformity of droplets. It is often used in small drying operations and when using a more viscous input material.

c. Centrifugal atomizer
The centrifugal atomizer, or rotating disc, is basically a disc that turns at the extremity of an axis, where the liquid material is injected and accelerates radially, pulverizing the input in the drying chamber. There are various variants of the disc project that provide a wide range of droplets sizes, therefore these are more widely used in industrial projects.

1.3. Droplet-air contact
The important component of spray dryer is the chamber; here the sprayed droplet is contacted with the hot air and the drying process begins. Air is heated by the heating element which situated before entering the chamber to a predefined temperature depending upon the characteristics of the feed fluid. The thermal energy of the hot air is used for evaporation and the cooled air pneumatically conveys the dried particles in the system. The contact time of the hot air and the spray droplets is only a few seconds, once the drying is achieved and the air temperature of air drops instantaneously. The nozzle increases the contact area of droplet and hot air influences in the huge heat transfer between droplet and hot air. The hot air evaporates moisture content in the droplet and changes into powder form. The hot air is brought in contact with the spray droplets in the following ways through the air distributor:
1. Co-current-Air and particles move in the same direction.
2. Counter-current-air and particles move in the opposite direction.
3. Mixed flow - particles are subjected to co-current and counter-current phase.
In co-current process the liquid is sprayed in the same direction as the flow of hot air through the apparatus, hot air inlet temperature is typically 150-220°C, evaporation occurs instantaneously and then dry powders will be exposed to moderate temperatures (typically 50-80°C) which limits the thermal degradations. In counter-current drying, the liquid is sprayed in the opposite direction of hot air flow for high temperature process. Thermo-sensitive products are usually restricted to in this process. However, the main advantage of this process is considered as more economic in term of consuming energy.

1.4. Droplet drying
At the stage of droplets - hot air contacts between the liquid and gas phases and balances the temperature and established the vapor partial pressure. Heat transfer is carried out from the air towards the product and thus induces the difference in temperature. Water transfer is carried out in the opposite direction due to the vapor pressure difference. Based on the drying theory, three successive steps can be distinguished. Just after the hot air - liquid contact, heat transfer majorly causes the increase of droplets temperature up to a constant value. After that, the evaporation of water droplet is carried out at a constant temperature and water vapor partial pressure. The rate of water diffusion from the droplet core to its surface is usually considered as constant and equal to the surface evaporation rate. Finally, when the droplet water content reaches a critical value, a dry crust is formed at the droplet surface and the drying rate rapidly decreases with the drying front progression and becomes dependent on the water diffusion rate through this crust. Drying is finished when the particle temperature becomes equal to that of the air. Each product has a difference of particle-forming characteristics such as expand, contract, fracture or disintegrate. The resulting particles may be relatively uniform hollow spheres, or porous and irregularly shaped.

1.5. Separation of dried particles
This separation is performed by a cyclone, placed outside the dryer that reduces product loss in the atmosphere. The dense particles are recovered at the base of the drying chamber while the finest ones pass through the cyclone to separate from the humid air. In addition to cyclones, spray dryers are commonly equipped with the filters, called “bag houses” that are used to remove the finest powder, and the chemical scrubbers remove the remaining powder or any volatile pollutants.

2. Advantages of Spray drying of Herbal Extracts
a. The cost of spray drying process is less. It is eight times more economical than freeze drying and four times more economical than vacuum drying.
b. The spray drying technique produce powders of a specific particle size and moisture content, irrespective of the dryer capacity.
c. In comparison to liquid extract, volume and weight of powder is less. It is easy for handling packaging and transportation.
The most common and economical way to carry out microencapsulation to retain and protect chemically reactive or flavor compounds in herbal extract is spray drying so that it is widely used in commercial scale. Powders produced by spray drying are resistant to microbiological and oxidative degradation. The dried dosage forms of medicinal plants are preferred than liquid presentations because of their higher stability. These powders have much longer shelf life.

The large surface obtained by the spraying assures pleasant conditions for the drying process. The technology is not very sensitive concerning the substance of the material, so that solutions, emulsions and pastes can be dried with this technology. It is applicable to both heat sensitive and heat-resistant materials. The short drying time allows the drying of heat sensitive materials.

It assures continuous operating conditions (continuous inlet and outlet in powder-form product). It is easy process which is fully automatically controlled with a quick response time.

Factors affecting spray drying process and its impact on spray dried powder properties are shown in fig.2.

4.1 Inlet Air Temperature
Temperature of the heated drying air is called ‘Inlet Air Temperature’. Temperature of the air with the solid particles before entering the cyclone is designated as ‘outlet temperature’. Usually, an inlet temperature of 150-220 °C and an outlet temperature of 50-80 °C is maintained. Powder properties such as moisture content, bulk density, particle size, hygroscopicity and morphology were affected by inlet air temperature.

4.1.1 Effect of Inlet air temperature on moisture content
Moisture content is decreased with the increase of drying temperature, due to the faster heat transfer between the product and drying air. At higher inlet air temperatures, there is a greater temperature gradient between the atomized feed and drying air and it results the greatest driving force for water evaporation.

4.1.2 Effect of Inlet air temperature on bulk density
An increase in temperature causes the reduction in bulk density. An increase in the inlet air temperature often results in a rapid formation of dried layer on the droplet surface and particle size and it causes the skinning over or casehardening on the droplets at the higher temperatures. This leads to the formation of vapor-impermeable films on the droplet surface, followed by the formation of vapor bubbles and, consequently the droplet expansion, the increase of drying air temperature generally causes the decrease in bulk, particle density and provides the greater tendency to the particles to hollow.

4.1.3 Effect of Inlet air temperature on particle size
The use of higher inlet air temperature leads to the production of larger particles and causes the higher swelling and thus increases particle size. When the inlet air temperature is low, the particle remains more smaller.

4.1.4 Effect of Inlet air temperature on particle shape
The particles exhibited the spherical shape and in various sizes, caused by materials produced in spray drying. When the inlet air temperature was low, the particles showed a shriveled surface, while increasing drying temperatures resulted in a larger number of particles with smooth surface. This is associated with the different drying rates, which
has the highest rate at higher temperatures, causing the faster water evaporation and then it led to the formation of smooth, porous and hard crust\(^4\).

### 4.1.5 Effect of Inlet air temperature on hygroscopicity

The powders produced at higher inlet temperatures were more hygroscopic due to the presence of lower moisture content in the powder\(^4\).

### 4.1.6 Effect of Inlet air temperature on yield

The increase of inlet temperatures has given the higher process yield and it was due to the greater efficiency of heat and mass transfer processes occurring when higher inlet air temperatures were used. Sometimes the increase of inlet air temperature has reduced the yield and it might be caused by melting of the powder and cohesion wall and therefore the amount of powder production and yield was reduced\(^4\).

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**Fig.2. Factors affecting Spray drying process and effect of spray drying process parameters on powder properties.**

### 4.2. Velocity of Inlet Air

Generally, the energy available for evaporation is varied according to the amount of drying air. The rate of air flow must be at a maximum in all cases. The movement of air is decided the rate and degree of droplet evaporation by inducing, the passage of spray through the drying zone and the concentration of product in the region of the dryer walls and finally extent the semi-dried droplets and thus re-enter the hot areas around the air disperser. A lower drying air flow rate causes an increase in the product halting time in drying chamber and enforces the circulatory effects. The increased residence time led to the greater degree of moisture removal. As a result, an increase of the drying air flow rate, decrease the residence time of the product in the drying chamber and it leads to have higher moisture contents. In addition, inlet air flow rate affects bulk density, moisture content and sticky nature of the
product. The higher moisture content in the powder leads to stick together and consequently leaving more interspaces between them and it results the larger bulk volume. Therefore, the raise of air flow rate leads to an increase of moisture content in the powder and decreased in powder bulk density. The effect of drying air flow rate on powder solubility depends on its effect on powder moisture content, as low moisture content seemed to be associated with the fast dissolution. The rising of air flow rate was led to the increased of powder moisture content and decrease in powder solubility.

4.3. Outlet air temperature
If outlet temperature is more, then the moisture content in dried powder is less and vice versa. The outlet air temperature controls the final moisture content of the powder.

4.4. Atomizer speed
The residual moisture content was decreased when increasing the atomizer speed. At higher atomizer speed, the smaller droplets were produced and more moisture was evaporated resulting from an increased contact surface. The higher atomizer speed resulted in a smaller particle size and quicker drying due to the larger surface area and consequently it prevented the “skinning” over the droplets. The increased of atomizer speed was spread the liquid into thin film layer and thus caused the smaller droplet and particle size.

4.5. Feed flow rate
Higher flow rates imply in a shorter contact time between the feed and drying air and making the heat transfer less efficient and thus caused the lower water evaporation. The higher feed flow rate showed a negative effect on process yield and that was resulting the decreased heat, mass transfer and the lower process yield. In addition, when higher feed rates were used, a dripping inside the main chamber was observed, when the mixture was passed straight to the chamber and that was not atomized and finally resulting the lower process yield.

4.6. Type of Spray drying agent
The addition of high molecular weight additives to the product before atomizing is widely used as an alternative way to increase Tg of powder. The use of carrier agents such as maltodextrins, gum Arabic, waxy starch, and microcrystalline cellulose, was influenced the properties and stability of the powder. Crystalline and amorphous forms of the same material powder show differences in particle size, particle shape, bulk density, physicochemical properties, chemical stability, water solubility and hygroscopicity. The common carrier agents used for fruit juices are maltodextrins and gum Arabic.

4.7. Concentration of carrier agent
The concentration of the carrier agent also affected the powder properties. Low concentration of carrier agent may obtain the stickiness powder.

4.8. Type of solvent used for preparation of extract
Closed cycle spray dryers should be used to handle flammable solvents, highly toxic products and oxygen sensitive products to avoid atmospheric pollution and/or to establish complete recovery of the evaporated solvent. The closed cycle system is based upon recycling and reusing the gaseous drying medium, which usually is an inert gas such as nitrogen. Drying chambers are incorporated with a cyclone/bag filter, solvent vapour condenser, exhaust drying medium particulate cleaning in wet scrubbers and indirect drying medium heating.

5.1. Types of Spray drying additives
Among commercially available additives that is used, major type additives that available for spray drying application are carbohydrates (hydrolysed starch, maltodextrin, dextran, cellulose and derived), gums (Arabic gums, agar, carrageenan), proteins (gluten, caseins, albumins and peptides), lipid (wax, paraffin, diglycerides and peptides) and biopolymers. Spray drying application using lipid and biopolymers are less significant. Table-1 shows various spray drying agents used for Herbal extract.

<table>
<thead>
<tr>
<th>Sr. No</th>
<th>Herbal Extract</th>
<th>Spray drying additives used</th>
<th>Spray Dryer Model</th>
<th>Inlet Air Temperature</th>
<th>Outlet Air Temperature</th>
<th>Year of Publication</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Rose hip extract⁹</td>
<td>-</td>
<td>RSZL-10 type</td>
<td>153°C</td>
<td>65 °C</td>
<td>1994</td>
</tr>
<tr>
<td>2.</td>
<td>Camomile extract⁹</td>
<td>-</td>
<td>RSZL-10 type</td>
<td>145°C</td>
<td>66 °C</td>
<td>1994</td>
</tr>
<tr>
<td>3.</td>
<td>Lime blossom extract⁹</td>
<td>-</td>
<td>RSZL-10 type</td>
<td>150°C</td>
<td>64 °C</td>
<td>1994</td>
</tr>
<tr>
<td>4.</td>
<td>Faba bean (Vinca faba variety Dulce miel) seed extract¹⁴</td>
<td>-</td>
<td>Mini spray dryer (Buchi B-190 Laboratoriums-Technik, Flawil-Schweiz, Switzerland)</td>
<td>180°C</td>
<td>110°C</td>
<td>1998</td>
</tr>
<tr>
<td>5.</td>
<td>Tomato Pulp¹⁵</td>
<td>-</td>
<td>Pilot scale spray dryer (Buchi, B-191) with cocurrent operation</td>
<td>100-140°C</td>
<td>60-90 °C</td>
<td>2003</td>
</tr>
<tr>
<td>6.</td>
<td>Curcumin, obtained from the rhizomes of Curcuma longa L., Zingiberaceae (turmeric)¹⁶</td>
<td>PVP K30 (PVP)</td>
<td>Spray Dryer (Jay Instruments and Systems Pvt. Ltd., India)</td>
<td>60 °C</td>
<td>45 °C</td>
<td>2004</td>
</tr>
<tr>
<td>7.</td>
<td>Mango (Mangifera indica L.) bark extract¹⁷</td>
<td>Colloidal silicon dioxide</td>
<td>A mini spray-drier model LM MSD 1.0 (Labmaq do Brasil Ltda., Brazil)</td>
<td>100°C</td>
<td>60 °C</td>
<td>2009</td>
</tr>
<tr>
<td>8.</td>
<td>Tanacetum parthenium L. (Feverfew) hydroalcoholic extract¹⁸</td>
<td>Maltodextrin</td>
<td>Buchi, B-191 (Buchi Laboratoriums-Technik, Flawil, Switzerland)</td>
<td>100-140 °C</td>
<td>-</td>
<td>2010</td>
</tr>
<tr>
<td>9.</td>
<td>Concentrated orange juice¹⁹</td>
<td>Maltodextrin</td>
<td>Mini Spray Dryer Buchi B-290 (Buchi Labortechnik AG, Flawil, Switzerland)</td>
<td>170-185 °C</td>
<td>-</td>
<td>2010</td>
</tr>
<tr>
<td>10.</td>
<td>Acerola Pomace extract²⁰</td>
<td>Maltodextrin, cashew tree gum</td>
<td>Spray dryer (model BE1216, Bowen, Somerville, NJ)</td>
<td>150°C</td>
<td>100°C</td>
<td>2010</td>
</tr>
<tr>
<td>11.</td>
<td>Garcinia indica (Choisy fruits) extract²¹</td>
<td>Maltodextrin (DE 06, 19, 21, and 33)</td>
<td>K-Carrageenan</td>
<td>90-140°C</td>
<td>-</td>
<td>2011</td>
</tr>
<tr>
<td>12.</td>
<td>Morinda citrifolia L. (NoNI) fruit extract²²</td>
<td>Maltodextrin</td>
<td>Lab Plant spray dryer model SD-05 (pilot scale) with cocurrent spray.</td>
<td>110-130°C</td>
<td>-</td>
<td>2012</td>
</tr>
<tr>
<td>13.</td>
<td>Liquorice (Glycyrrhiza glabra) extract²³</td>
<td>Maltodextrin</td>
<td>Lab Plant spray dryer model SD-05 (pilot scale) with cocurrent spray.</td>
<td>90-140°C</td>
<td>-</td>
<td>2012</td>
</tr>
<tr>
<td>14.</td>
<td>Morinda citrifolia L. extract²⁴</td>
<td>Carrageenan and Maltodextrin</td>
<td>Lab Plant spray dryer model SD-05 (pilot scale) with cocurrent spray.</td>
<td>100-140°C</td>
<td>-</td>
<td>2012</td>
</tr>
<tr>
<td>No.</td>
<td>Product</td>
<td>Ingredients</td>
<td>Equipment/Technique</td>
<td>Temperature</td>
<td>Process Temp</td>
<td>Year</td>
</tr>
<tr>
<td>-----</td>
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</tr>
<tr>
<td>16.</td>
<td>Tomato (<em>Solanum lycopersicum</em> L.) pulp</td>
<td>Starch, Arabic gum and Maltodextrin</td>
<td>Acmefil make spray drier available at Gandhigram Trust, Gandhigram, Dindigul.</td>
<td>130°C</td>
<td>85°C</td>
<td>2013</td>
</tr>
<tr>
<td>17.</td>
<td>Red Pitaya (<em>Hylocereus polyrhizus</em>) Peel</td>
<td>Maltodextrin</td>
<td>Pilot-scale rotary atomizer-type spray dryer (Niro A/S, GEA, Germany)</td>
<td>155–175 °C</td>
<td>75–85 °C</td>
<td>2013</td>
</tr>
<tr>
<td>18.</td>
<td>Black tea (<em>Camellia sinensis</em>) extract</td>
<td>Gelatin, Gum Arabic, and Maltodextrin</td>
<td>Tall Type Spray dryer (Model No.IGO12X120, Mfd by SM Scientech Kolkata-29)</td>
<td>180-240°C</td>
<td>95°C</td>
<td>2014</td>
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<tr>
<td>19.</td>
<td><em>Gypsophilia Saponaria officinalis</em> (Soapwort) roots aqueous extract</td>
<td>Maltodextrin</td>
<td>Niro Automizer Mobile Minor Unit (Soeborg, Denmark)</td>
<td>110-160°C</td>
<td>50-80°C</td>
<td>2014</td>
</tr>
<tr>
<td>20.</td>
<td>Jucara (<em>Euterpe edulis</em> M.) pulp</td>
<td>Gelatin, Gum Arabic, and Maltodextrin</td>
<td>Mini Spray-Dryer-MSD1.0 (LabmaqdoBrasil, Ribeirao Preto, Brazil)</td>
<td>140-170°C</td>
<td>85-130°C</td>
<td>2014</td>
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<tr>
<td>23.</td>
<td><em>Psidium guajava</em> L. leaves extract</td>
<td>Maltodextrin, Colloidal silicon dioxide, Arabic gum and β-cyclodextrin</td>
<td>Benchtop spray dryer (model SD 05, Lab-Plant, UK)</td>
<td>150°C</td>
<td>100-110 °C</td>
<td>2014</td>
</tr>
<tr>
<td>24.</td>
<td>Grape Syrup</td>
<td>Maltodextrin</td>
<td>Pilot scale spray dryer (Maham Sanat, neishabour, Mashhad, Iran)</td>
<td>150-170°C</td>
<td>-</td>
<td>2014</td>
</tr>
<tr>
<td>25.</td>
<td>Tongkat Ali (<em>Eurycoma longifolia</em>) aqueous root extract</td>
<td>-</td>
<td>Laboratory scale spray dryer Lab Plant TM SD-06 (UK)</td>
<td>100-220°C</td>
<td>-</td>
<td>2015</td>
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<tr>
<td>29.</td>
<td>Mango Pulp</td>
<td>Maltodextrin</td>
<td>Laboratory-scale</td>
<td>150°C</td>
<td>-</td>
<td>2017</td>
</tr>
<tr>
<td>No.</td>
<td>Product Description</td>
<td>Stabilizer</td>
<td>Spray Drying System</td>
<td>Temperature Range</td>
<td>Other Conditions</td>
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<tr>
<td>-----</td>
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</tr>
<tr>
<td>31.</td>
<td>Laurel clock (Thunbergia laurifolia Lindl), Bamboo grass or Ya-nang (Tiliacora triandra) Diels and Asiatic pennywort or Bai-Bua Bok (Centella asiatica (L.) Urb.) extract</td>
<td>Maltodextrin (DE 10)</td>
<td>Mini Spray dryer (Buchi, B-191, Laboratory-Techniques Ltd., Flawil, Switzerland)</td>
<td>120-160°C</td>
<td>90°C</td>
<td>2018</td>
</tr>
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<td>32.</td>
<td>Lemongrass (Cymbopogon citratus L.) leaf extract</td>
<td>Maltodextrin and gum Arabic</td>
<td>Lab Plant SD06 spray dryer (Keison, Chelmsford, UK)</td>
<td>110-150°C</td>
<td>-</td>
<td>2018</td>
</tr>
<tr>
<td>33.</td>
<td>Liquorice (Glycyrrhiza glabra Linn.) roots extract</td>
<td>Aerosil</td>
<td>Laboratory scale Labultima UV222 spray dryer</td>
<td>110-130°C</td>
<td>-</td>
<td>2018</td>
</tr>
<tr>
<td>34.</td>
<td>Mulberry (Morus alba Linn.) leaves extract in 70% ethanol</td>
<td>Maltodextrin</td>
<td>Laboratory-scale Mini Spray Dryer Model LU-122 Advance, Labultima</td>
<td>60°C</td>
<td>55°C</td>
<td>2019</td>
</tr>
<tr>
<td>35.</td>
<td>Passion Fruit (Passiflora Edulis) fruit juice</td>
<td>Maltodextrin, Arabic Gum, Modified Starch, Whey Protein concentrate</td>
<td>Laboratory spray dryer provided with rotary atomizer, pressure nozzle, pneumatic nozzle and sonic nozzle</td>
<td>120-150°C</td>
<td>-</td>
<td>2019</td>
</tr>
<tr>
<td>36.</td>
<td>Palmyra Palm (Borassus flabellifer) Juice</td>
<td>Arabic Gum, Maltodextrin</td>
<td>Laboratory spray dryer</td>
<td>120-150°C</td>
<td>75-90°C</td>
<td>2019</td>
</tr>
<tr>
<td>37.</td>
<td>Fallopia multiflora extract</td>
<td>Maltodextrin</td>
<td>Laboratory spray dryer</td>
<td>145-175°C</td>
<td>75-90°C</td>
<td>2019</td>
</tr>
<tr>
<td>38.</td>
<td>Watermelon (Citrullus lanatus) juice</td>
<td>Arabic Gum, Maltodextrin</td>
<td>Laboratory spray dryer</td>
<td>140-170°C</td>
<td>75-90°C</td>
<td>2019</td>
</tr>
<tr>
<td>39.</td>
<td>Black Carrots (Pusa asita) extract</td>
<td>Maltodextrin (20DE), Gum Arabic and Tapioca Starch</td>
<td>Laboratory Spray Dryer (LU–228, Labultima Pvt. Ltd., Mumbai, India)</td>
<td>150°C</td>
<td>53°C</td>
<td>2019</td>
</tr>
<tr>
<td>40.</td>
<td>White horehound (Marrubium vulgare L.) extract</td>
<td>Maltodextrin</td>
<td>Pilot scale spray dryer (APV Anhydro AS, Soborg, Denmark)</td>
<td>130 ± 5°C</td>
<td>75-80°C</td>
<td>2019</td>
</tr>
<tr>
<td>41.</td>
<td>Basil (Ocimum Basilicum L.) extract</td>
<td>Maltodextrin</td>
<td>Anhydro lab scale spray dryer (APV Anhydro AS, Denmark)</td>
<td>120°C</td>
<td>80°C</td>
<td>2019</td>
</tr>
<tr>
<td>42.</td>
<td>Anthocephalus cadamba leaves extract</td>
<td>Aerosil</td>
<td>Laboratory scale Labultima UV222 spray dryer</td>
<td>110-140°C</td>
<td>-</td>
<td>2019</td>
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</table>
5.1. Carbohydrates
High molecular weights carbohydrate additives help to increase the transition temperature of the spray drying process. Maltodextrin is available in the form of white powder, made from corn starch hydrolysed by acids of enzymes. Maltodextrin is non-sweet, neutral smell additives and used in spray drying production due to its low cost and bulking properties. The variable nature of maltodextrin can be found through the degree of hydrolysis of starch polymer. The significant higher level of dextrose equivalent (DE) in the hydrolysed starch has lower average molecular weight and has low permeability to oxygen and water. Maltodextrin with dextrose equivalent between 10 and 20 have higher flavour retention. Otherwise, usage of lower DE with higher molecular weight can improve the transition temperature of the product and it increases percentage yield and decreases caking problem. Maltodextrin has a significant effect on the solubility of powder, however, has shown contradicting results in hygroscopicity properties of the powder. Maltodextrin is also used in combination of other additives like Arabic gum, modified starch and whey proteins.
Sucrose, glucose and fructose are not suitable with high temperature of spray drying which can cause formation of caramel. Non-hydrophobic properties of starch have high viscosity value. Modified starch has found their way into commercial spray drying production, where modified starches have high retention of volatiles and stability. However, maltodextrin is commonly added with modified starches to improve against oxidation during storage of product. Cellulose has been used as complementary additives in spray drying to produce partial crystalline surface, reduce stickiness and caking problem.

5.1.2. Proteins
Gelatin, casein, whey protein concentration are used for multiple feed products. These additives show ability to combine with a different type of feed products through their molecular chain diversity and functional properties. Protein additives have rapid film-forming properties, large molecular weight and expanded functional properties such as high solubility and viscosity. Proteins can be used to improve spray drying of high sugar content powder. Small concentration of protein is capable of forming film properties around the surface spray-dried particles, prompting a thin protein-rich film to resist particle-to-particle cohesive and particle-to-wall stickiness.
High-protein concentration powders have substantially different structure and stability compared to low-protein concentration powders, considering the type of protein-additives and concentration of feed-additives. Whey powder has the capability of retaining flavoring compound. Protein additives are commonly used in spray drying for milk protein and gelatins. Spray drying efficiency and preservation of milk fat yield is greater without the use of additives. New studies have found that vegetable proteins additives have additional functional advantages like biocompatibility, biodegradability, emulsifying and foaming capacity.

5.1.3. Gums
Acacia gum (Gum Arabic) consists of a combination of complex carbohydrates and protein (D-glucuronic acid, L-rhamnose, D-galatose and L-arabinose in the proportion of 4:2:2:1). The protein component of the acacia gum improves the emulsification properties. It is used for its surface activity and film forming activity. Acacia gum has a produces stable emulsion in wide range of pH, so it can be used for lipid-based products. Product obtained using acacia gum does not have crystalline configuration and bulk density is less. Arabic gum is associated with high cost, limited availability and impurities due to prone to variability in supply and quality.

5.2. Factors affecting Selection of additive/s in Spray Drying Process
While selecting an additive for spray drying, physiochemical properties of the additive, molecular weight, glass transition temperature, concentration of additives are to be considered. The type of feed material used together with added additives has a significant impact on the properties of extract obtained.

5.2.1. Molecular weight of additives
The molecular weight of additives represents the molecular size of the additives which plays important role in spray drying process. Shorter chain molecules of additives have low transition temperature than longer chain additives. Maltodextrin (DE 36) with a molecular weight of 500 has transition temperature of 100°C and maltodextrin (DE 5) has a significantly higher transition temperature of 188°C.
Increasing transition temperature also contributes to powder stability and reduce caking and stickiness problem. Gum acacia has higher molecular weight in comparison to maltodextrin. As the transition temperature increase with the increase in molecular weight, the addition of gum acacia has a higher Tg compared to the addition of maltodextrin. The larger molecular weight of maltodextrin has a direct relationship with faster rehydration and reabsorption of powdered particles, due to the higher surface area over volume ratio exposed to moisture.
5.2.2. Concentration of additives
High concentration additives used in feed material produce a difference in the yield percentage, transition temperature, hygroscopicity and other physicochemical properties. Multiple published papers have shown that increase of maltodextrin concentration has resulted in an increase of yield product percentage. The yield product percentage can be supported by the fact that surplus level of maltodextrin increases the total solid and reduced the level of total water for evaporation.

Higher concentration of maltodextrin added produces powder with lower moisture content, but the concentration of maltodextrin added does not affect its rehydration ability to absorb moisture. The concentration of protein additives has inconsistent results towards physiochemical properties of spray dried powder. Addition of protein additives at certain concentration increase yield production but doubling the concentration yield insignificant results. The ratio of protein-carbohydrate additives has proven contradicting results in term crystallization process.

5.2.3. Combination of additives
Maltodextrin is also used in combination of other additives like Arabic gum, modified starch and whey proteins.

5.2.4. Feed Material Properties
Liquid properties relevant to spray drying are: solids content, density, surface tension and viscosity. High concentration of solutes in the liquid is desirable to increase dryer thermal efficiency. Liquid components should be thermally stable to withstand thermal treatment in the dryer. Type of feed materials varies in properties like sugar and fat content, viscosity, and transition temperature. Understanding of spray drying of various feed material on its wide range of processing parameter and quality parameter of the powder attained requires through knowledge.

6. Effect of Spray Drying Parameters on Powder Properties
The selection of additives is heavily supported by understanding the properties of both spray drying feed and the additives used, as different additives have different physical and chemical structure. Gum acacia is a highly ramified structure that contains shorter chains and hydrophilic groups, while protein additives have many different functional advantages such as biocompatibility, biodegradability, emulsifying and foaming capacity, water and fat absorption, gelation and film-forming properties.

6.1. Bulk density
There is no clear relationship between additives capabilities and bulk density. Additives addition into feed solution has a different effect on the bulk density. Addition of maltodextrin shows additive effect that changes the bulk density of powdered particles. Skin forming nature of additives increases the volume of air trapped in the particle and decreases the bulk density of powders.

6.2. Glass transition temperature (Tg)
Glass transition temperature (Tg) is a property of an amorphous material, where it defined as the temperature of product amorphous system interchanging between a glassy and a rubbery state. During spray drying the particles will undergo the transition from a glassy crystalize temperature to rubbery state matter depending on their water activity level and molecular weight. Similarly, transition temperature (Tg) is highly associated with changes in various physical properties such as boiling and melting point and appearances. Increasing temperature above Tg of a material increase rate of deteriorative and stickiness. Spray drying of extract containing of low-molecular weight molecules cause a stickiness problem during spray drying. To overcome low glass transition temperature of feed material, adding additives have shown to increase the molecular weight of molecules, therefore increasing feed’s transition temperature. By this application, the extension of shelf life of the powder can be extended. High stability of powder is associated with high transition temperature and the risk of caking, and crystallization can be reduced.

6.3. Particle size
The size of particle formed during spray drying is strongly related to feed viscosity, as higher liquid viscosity, the larger the droplets formed during atomization produces larger particles obtained during spray drying. The addition
of additives, especially an increase of maltodextrin concentration has proven to increase the particle size of spray
dried powder2.

The lack of research on additives is towards the microscopic point of view, there is substantial evidence showing
how the additives reacted on different powders products2.

6.4. Colour index
The physical appearance of the powders especially the color index depends on the type of feed material and
concentration of additives used. As the function of additives used in spray drying may increase the droplet size of
atomized feed as viscosity increases, there are no reports stating that additives improved the physical appearance of
end powder products. The increase in concentration of additives dilutes the colour of the feed solution and the end
product significantly as shows colour similar to additive. Overall, there are insufficient studies done on the effect of
additives on the powder colour index, as major studies focused on yield production, moisture content and other
parameters2.

6.5. Solubility index
Solubility is an important key factor for evaluating wettability and dispersibility of powder in aqueous solution. The
solubility index of spray dried powder is affected by the raw materials, and additives used. The solubility index also
depends on the properties of the powder (moisture content and size of particles). During spray drying, crust
formation occurred during rapid heat exchange environment in the chamber, where the least soluble substance
started to precipitate and forming crust at the droplet surface. Therefore, the formed crust is mainly constituted of
maltodextrin that is highly soluble in nature. If hard surface layer is formed over the powder particle, preventing the
diffusion of water molecules, then the wettability and solubility of the particle are reduced2.

6.6. Percentage yield
In the spray drying process, an increase of additive concentration increases percentage yield. The addition of
additives increases the total soluble solids content, resulted in an increase in efficiency yield. Sometimes, the
addition of additives in a spray drying operation led to a less efficient system, where high residual moisture content
found in powder products. Water molecules have difficulty in escaping from maltodextrin due to their large size and
film coating abilities. Furthermore, the increase of additives would produce a higher viscosity feed which reduces
the product yield of powder2.

6.7. Hygroscopicity/ Moisture content
In the spray drying process, an increase of additive concentration decreases moisture content in powder obtained and
it increases hygroscopicity of powder. The powder is more hygroscopic, if it contains less quantity of moisture2.

6.8. Flavor retention
The losses of both hydrophilic and hydrophobic flavors during the spray drying were reported in each step of
process. The losses of flavors were occurred first during the atomization step. The flavors and water are evaporated
with the hot inlet air at initial period of drying. The loss of flavors especially the high volatile flavors (with low
boiling point temperature) was expected in this period with the temperature of droplet increase. In the second step
with the constant drying periods, the formation of crust was occurred. The crust was the selective membrane allows
the higher diffusion rate of water than the flavors resulted the higher remaining of flavor in the droplet. However,
the high solubility flavor as well as hydrophilic flavor was lost in the higher amount than in the hydrophobic flavor.
The dissolved flavors were loss during the drying as the water molecule. After the solid droplets were formed, the
loss of flavors was expected to occur with the change of powder morphology. However, the losses in this step were
less amount comparing to the first and second steps7.

6.9. Powder flow properties
The powder obtained by spray drying show generally show poor flow properties because of the small size of its
particles. The small size results in problems related to the powder’s transport on an industrial scale, where uniform
mass flow out of bins and other vessels is required. In the case of dry plant extracts, the interactions between the
powder particles are often so strong that they do not pour at all. This phenomenon may also generate problems
related to obtaining proper mass uniformity in solid dosage forms. In addition, the tablet manufacturing process
requires the application of high pressure, which in turn results in tablets that are too hard and give prolonged
disintegration times89.
7. Sticking problem associated with Spray drying

When spray drying technology is used to manufacture products, powder handling difficulties can arise for some products due to adhesion of powder to equipment surfaces. This is generally considered to result from the formation of liquid bridges containing amorphous sugar or other dissolved material conferring high surface tension, or perhaps lipid material in some cases, between particles and equipment surfaces\(^7\).

7.1. Factors affecting Sticking

Factors affecting sticking are shown in Fig.3.

![Diagram showing factors affecting sticking in spray drying process]

**Fig.3. Factors affecting Sticking**

7.1.1. Material of wall for drying chamber

The properties of the wall of the dryer in which deposition occurs also play a significant role in the mechanism of product deposition. Recent experiments showed that nylon exhibited a lesser deposition rate due to the non-sticky nature of the nylon compared to stainless steel. A teflon surface with less surface energy has a less deposition flux compared to a stainless steel surface that has a higher surface energy. A higher wall temperature also increases the deposition flux\(^3\).

7.1.2. Geometries or spray dryer types

The chamber geometry directly changes the air flow pattern and affect the behavior and flow pattern of the particle within the dryer. Several researchers have studied possible chamber geometries such as pure conical, lantern and hour-glass geometry, horizontal configuration and parabolic geometry as the spray drying chamber. It should be noted that not only the cylinder geometry but the duration of the particle residence in the drying chamber as well as overall chamber volume can impact drying performance\(^3\).

7.1.3. Swirling flow patterns

Swirling flow patterns in a spray dryer can be of two types: inlet vane induced swirls and atomizer-induced swirls. Experimental measurements and observations have been undertaken for inlet-induced swirls which highlighted the unsteady and oscillatory behavior of such flows. Experimental data indicated that the wall deposition was minimized for no swirl, but that evaporation was still adequate\(^3\).

7.1.4. Spray Dryer Wall Temperature

Particles deposit on the wall by sticking to it, which is due to the sticky particle which occurs above the glass transition temperature, T\(_g\). Spray drying produces dry amorphous powders that are thermo- plastic due to heating or exposure to high humidity, resulting in water sorption and thermal plasticization of the particle surfaces. At temperatures above T\(_g\), amorphous structures are in a rubbery state where the polymer molecules become softer and more flexible because of greater molecular mobility. The temperature of the surface of the product such as amorphous sugar should not reach more than 10–20\(^\circ\)C above T\(_g\) during spray drying to avoid substantial product...
stickiness, especially in low molecular weight carbohydrates. This is due to the greater molecular mobility of the amorphous components in a highly viscous flow between the particle surfaces, making the powder more cohesive. At temperatures below $T_g$, the amorphous parts of the materials are in a glassy state where the polymer molecules have no segmental motion but vibrate slightly. When the wall temperature is below the sticky point temperature, there is less wall deposition\(^3\).

**7.1.5. Effect of Feed flow rate**

When the feed flow rate increases, larger droplets are formed and the evaporation rate is lower. This is caused by the larger amounts of water introduced into the dryer. Increase in feed flow rate result in higher residual moisture content. There was a corresponding visual increase in deposits on the wall of the drying chamber. The deposition in this case is high because of the higher feed flow rate. The rate of adhesion of the first layer was different compared to that of other layers when particles cohere to the adhered particles. At a constant air inlet temperature, increasing the feed flow rate increased the wall deposition. When more feed was atomized into the chamber, the residence time of the particles was shorter and the drying time was reduced, resulting in wetter particles. In this condition, the particles were more cohesive which caused the deposition rate to increase and the yield to decrease\(^3\).

**7.1.6. Effect of spray drying agents**

The effect of additive as a spray drying agent is more significant in reducing the deposition fluxes. The additives added to the sugar-rich feed, increased its molecular weight and hence its glass transition temperature $T_g$, which reduced the particle stickiness and wall deposition in the spray drying\(^3\).

**7.1.7. Nature of Feed component**

Wall deposition is affected by the residence time and distribution of the particles. An important factor in determining residence times with high wall deposition rates was the time taken by the particles to slide down the conical wall of a spray dryer. The sticking of particles to the walls and to each other, and the sliding of wall deposits are therefore important issues. The presence of lactose and protein on the surface could have made the particle surface more rigid due to the high glass transition point. On the other hand, another possibility is that the presence of lactose and protein could have made the particle surface more hydrophilic. Presence of a high proportion of protein on the surface, however, led to significant reduction in the adhesion rate at the cone of the spray dryer. The rate of adhesion of the first layer might be different compared to that of other layers when particles cohere to the adhered particles\(^3\).

**CONCLUSION:**

Medicinal plants are used by 80% of the world population as primary health care and the phytomedicine market is growing exponentially. Currently, the production of phytopharmaceuticals with proper efficacy, safety and consistent quality constitutes a relevant challenge. The spray drying technology can be used to produce dried extracts from medicinal plant liquid extracts\(^8\).

Published reports that signified the effect of additives on the properties of the powder, it is shown that different powder has shown different characteristic under the influence of different additives. However, a similar trait has appeared that additives improve product yield through the manipulation of transition temperature. Added to that, usage of combination additives proved positive results towards better yield, solubility and bulk density. Optimization of spray drying requires an evaluation of both spray drier parameter and feed formulation, as the modulating of spray drying must be controlled to avoid low yield, moisture content and sticking problem. Effective additives can optimize the performance of spray drying parameters. The efficiency of spray drying parameters such as inlet temperature, feed flow rate, outlet temperature and nozzle pressure can be enhanced with the use of additives. Use of additives is cost reducing and high productivity through the functionality of additives of manipulating the transition temperature, total soluble solids and viscosity of the solution.

**CONFLICT OF INTEREST:**

The authors declare no conflict of interest.

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