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Spray Drying of Aqueous Extract of *Mangifera indica L (V*imang): Scale up for the Process

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Abstract: Vimang is the brand name of an aqueous extract of *Mangifera indica L* (mango) and is widely used as antioxidant, analgesic and antinflamatory agent. It is obtained from mango´s stem bark by decoction with water and further spray drying process. The aim of this study was to optimize the operation conditions of spray drying of Vimang to maximize recovery yield, minimizing cost and keeping the quality parameters of Vimang powder. Experimental 2^3 central composite factorial designs was used to investigate the influence of three parameters on the particle size, the moisture content, recovery yield and dimensional cost of spray-dried product. The obtained experimental results adequately fit to empirical quadratic models. Then, the optimal operating conditions were estimated from the empirical model and further experimentally verified. The process was successfully scale-up from laboratory equipment to industrial scale keeping the same optimal operating parameters.

Key words: Spray drying · Scale up · *Mangifera indica L*

INTRODUCTION

Natural products are important for pharmaceutical research and for drug development as a source of therapeutic agents. At present, the demand for herbal or medicinal plant products has increasing significantly.

Mango (*Mangifera indica* L.), belongs to the family of Anacardiaceaes and it is commonly found in tropical and sub-tropical regions and is one of the most popular edible fruits in the world.The chemical composition of this plant has been studied extensively over the past years and the extracts contain mainly triterpenes, flavonoids, phytosterols and polyphenols [1-3].

A novel technological scheme for obtaining an aqueous extract of this plant has been developed in Cuba at industrial scale [4]. The process involves the extraction of mango stem bark using water as a solvent. The extract is then concentrated by evaporation and dried in a spray dryer. The fine brown powder was obtained, which melts at 215–218°C with decomposition. Vimang is the commercial name of this new bioactive product, which is used as a nutritional supplement, cosmetic and phytomedicine [5]. The antioxidant [6-7], analgesic [8], antiiflamatory [9] and inmunomodulatory [10-12] properties of Vimang have been evaluated.

Nowadays, the development of new technologies to obtained standardized dried natural extracts is an important subject of the herbal processing industries. The advantages of the dried extract over conventional liquid forms are lower storage costs, higher concentration and stability of active substances [13]. Several techniques are implemented to obtain powder, including freeze drying; spray drying and spouted bed [14]. However, spray dryers being the most frequently used in food and protein processing industries.

Spray drying is extensively used in the pharmaceutical industry in the production of bulk (raw) drug and recipients in the microencapsulation process [15]. This technique transforms liquid feed into dry powder in a one step continuous particle processing operation and can be applied to a wide variety of materials [16]. Despite of the advantages of spray-drying process, operating variables must be well controlled to avoid any difficulties such as low yields, sticking and high moisture content [17-20].

The optimization of the spray-drying process involves the evaluation of parameters concerning both the spray-dryer and the solution feed [21-22]. Many operational variables associated to atomization and the drying operation play an important role on the

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characteristics of the dried product. For instance, increase in solid content of the liquid feed affects the evaporation characteristics and the products enhance both particle and bulk density [17]. On the other hand, drying temperature affects the properties of the final products. Higher inlet temperature increases the dryer's evaporative capacity at constant air rate. Higher inlet temperature means a more thermally efficient dryer operation. Higher temperature often causes a reduction in bulk density because the evaporation rate is faster and the dried products are more porous or have a fragmented structure. The higher in outlet temperature decreases the moisture content at constant airflow and the heat-input conditions. On the other hand, the operation at low outlet temperature produces a powder having high moisture content and it can be advantageous when agglomerated forms of powder are required.

The performance of a spray drying process involved the establishment of operating conditions that increase product recovery and produce an end product of a predefined quality specification.

The aim of this study is the evaluation of the effects of spray drying operating conditions on the recovery yield and the cost of Vimang spray-dried in order to obtain a recovery yield higher than 80% with a minimum cost. The quality parameters on the established range for the final Vimang powder have to be maintained as well. The experimental conditions obtained at laboratory equipment were tested at industrial scale.

MATERIALS AND METHODS

Materials: The stem bark of *Mangifera indica L.* was collected from plants grown in a fruit farm in Alquizar, Havana, Cuba and subsequently dried and milled to obtain particles of around 2-3 cm.

The extract was obtained by reflux of the vegetable material with water. Then, the aqueous extract was evaporated to obtain concentrations of solids between 7.5-22.5 w/v %.

Spray Equipment: The lab scale drying was carried out in a spray NIRO Mobil Minor (Niro Atomizer, Denmark), with concurrent flow regime. The main components of the apparatus were the feed systems of the drying material, constituted of peristaltic pump, rotary disk atomizer and air compressor. The feed system of drying gas constituted of a blower and an air filter. The dried gas temperature was controlled and the product was collected by a cyclone.

For the industrial scale up a NIRO Type P-6.3-R (Niro Atomizer, Denmark) for aqueous feeds spray dryer was used. The equipment has an evaporative capacity of 33 kg/h of water it is also provide of centrifugal disk like via the atomizing.

Spray Drying Experiments: A 2^3 factorial central composite design [23] was employed in order to investigate the influence of the inlet temperature (T: 155-185 °C), the atomization pressure (P: 0.5-3.5 bar) and the concentration of solution (C: 7.5-22.5 %w/v) over the average particle size (D_p), the moisture content (M_H), the recovery yield (Y) and the process cost (C_T). The atomized air flow rate was kept constant at 80 Kg/h.

The recovery yield of drying was calculated from the weight of the final product into the collector vessel of the spray dryer by the following equation:

$$Y = \frac{M}{V * C} * 100 \quad (1)$$

Where M is the mass (g) at the end of experiment and V is the volume of the feed (L).

Powder Product Characteristics: Residual moisture content and total content solids of the product were measured using the infrared humidity analyzer (Sartorius MA-40, Sartorius, Göttingen, Germany) on a 0.1 g sample at 105 °C. The particle size distributions of the different samples were obtained in the particle analyzer Coulter Counter model LS-630.

Evaluation of the Total Cost of the Spray Drying Process: The cost of the spray drying process (CT) was determine taking into account the cost of two processes related with the operation variables: the drying (CD) and the concentration (CC) of the solution for kg of final product:

$$C_T = (C_D + C_C)/V*C*Y \quad (2)$$

The cost of drying was conformed by the salary cost, depreciation of the equipment and the energy cost [24]. It was assumed that the energy cost for the drying operation is constant and independent of the operating parameters due to the small variations from one experiment to other. For the concentration stage, there are significant differences in the operation time of concentration because it depends on the required final value of the concentration of the suspension.

For the analysis, the cost was expressed as dimensionless function (C_r). It was calculated as: $C_r=C_T/C_l$, where C_T is the real cost of 1 kg of final product at each experiment and C_l, is the lowest cost obtained for the powder in a single experiment.

RESULTS AND DISCUSSION

Experimental Design: effects of spray-drying factors on recovery yield and total cost: Table 1 shows the results of the factorial design. In some experiments, the operating conditions fixed by the experimental design induce low recovery yield (*Y*) due to the existence of a large sticking in the drying chamber. This phenomenon is increased when the pressure was lower than 2 bar.

The recovery yield varied from 65 to 91 %. In parallel, moisture varied from 4.5 to 7.4. The average diameter of the particles was the parameter that more changed in the study. The mean diameter of particle varied between 23.1 and 134.8 µm. This variation could be attributed to the presence of fatty acids in the composition of the liquid feed, which provide sticky characteristics to the powder, affecting the recovery yield of the drying process. On the other hand, the Vimang is a suspension (not a solution), in which solid content is approximately of 6 g/L. In this case, suspended particles will be only dried while dissolved particles have to be first crystallized and then dried. For this reason, there will be, as minimum, two types of particles with different average diameter. In the specific case of the Vimang, hollow particles are obtained, because the atomized liquid feed is a suspension and not a true solution [25]. Finally, the poliphenol content was higher than 40 % in all experiments, accomplishing to the required quality of the final product.

Table 1: Results obtained from the drying tests. Experimental factorial design 2^3

N°	*T* (°C)	*C** (%)	*P* (bar)	M_H (%)	D_p (µm)	*Y* (%)	C_r
1	160	10	1	7.38	34.46	83.2	1.55
2	160	10	3	5.40	24.1	89.5	1.44
3	160	20	1	6.23	47.8	76.3	1.27
4	160	20	3	7.39	66.5	81.4	1.19
5	180	10	1	6.30	87.9	82.7	1.56
6	180	10	3	7.18	75.8	90.0	1.43
7	180	20	1	4.76	38.6	75.6	1.28
8	180	20	3	5.76	83.3	80.3	1.21
9**	170	15	2	5.01±0.39	34.4±11.47	85.5±0.8	1.06±0.01
12	155	15	2	4.65	50.4	78.7	1.15
13	185	15	2	5.39	107.3	80.2	1.13
14	170	7.5	2	5.54	65.4	85.3	1.84
15	170	22.5	2	5.22	134.8	72.5	1.29
16	170	15	0.5	4.59	99.2	65.9	1.38
17	170	15	3.5	5.70	65.4	90.8	1.00

* *concentration value ± 0.05* ***average of three experiment expressed as* $\bar{x}$*±SD.*

Table 2: Coefficients of the regression equation between response and operating variables

	M_H	D_p	*Y*	C_r
A: temperature	0.08	NS	0.89	0.04
B: pressure	-7.75	-45.30	9.88	-0.26
C: solution concentration	0.98	NS	NS	-0.28
AA	NS	0.008	-0.003	-0.0001
BB	0.32	NS	-1.25	0.05
CC	0.02	0.52	-0.03	0.008
AB	0.03	NS	NS	NS
AC	-0.001	-0.12	NS	NS
BC	0.08	-2.88	NS	NS

NS: Statistically not a significant parameter

Determination of Operational Parameters from the Analysis of the Cost Function: The experimental results were fit by regression equations correlating the moisture content, the mean particle diameter, the recovery yield and the operating cost to the inlet temperature of air, the pressure and the concentration of feed solution. Quadratic polynomial equations were generated to establish the relationship between the factors and the response variables. The coefficients of the regression equations linking the responses to the operating variables and their interactions are showed in Table 2.

For the moisture content, the pressure of the centrifugal disk is the more significant parameter in the mathematical model. Increasing the pressure in the centrifugal disk diminishes the moisture content, especially at low inlet temperature. The inlet temperature also affects moisture content. A higher inlet temperature promotes a decreasing of residual moisture because it improves the water evaporation rate.

The Vimang powder was essentially influenced by the pressure of the centrifugal disk and the dryer inlet temperature. An increase in inlet temperature improves yields only when feed flow rate is high. High feed flow rates required more thermal energy than low flow rates, but the complete evaporation of the solvent was granted [17].

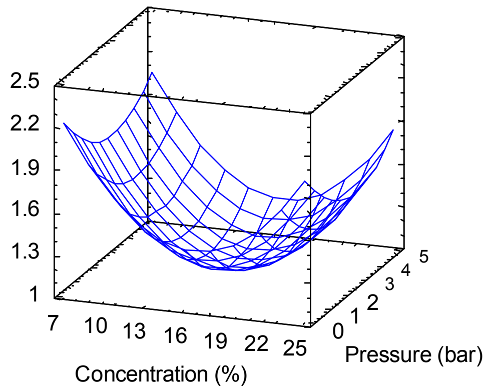

Fig. 1: Estimated response surfaces for cost variable. T= 171 °C.

Table 3: Experimental data obtained from industrial scale dryer

	M_H (%)	D_P (μm)	Y (%)
Experimental value	6.4 ±0.3	35.24±3.46	84.1±2.4
Estimated value	5.9	44.38	90
Error of estimation (%)	8.4	25.9	6.8

Finally, the total cost of the process is affected for different variables such as: the pressure of the centrifugal disk, the initial solution concentration, the inlet temperature in the dryer and their quadratic interactions. Higher inlet temperature indicates that the thermal energy supplied in the dryer is higher and then the cost of the process can be increasing. Lower feed concentrations requires less energy thermal because there is less suspending solids and generate lower values of the cost. The statistical analysis of the results, concerning both recovery yield and total cost, using the response surface methodology (Fig. 1), allows us the choice of the optimal operating conditions such as: inlet temperature of air T=171 °C, the rotation of centrifugal disk P=3 bar (approximately 21700 r/min) and the feed concentration C=17 % w/v. These parameters maximize recovery yield and reducing the total cost at the same time.

Spray Drying of VIMANG Extract at Industrial Scale: The defined conditions at laboratory scale could be validated at industrial level. However, it was not possible to work at 17 % w/v of solution concentration due to obstructions problems into the distributor of the centrifugal disk. For this reason, it was necessary operate at concentration of the feed solution between 11-12 %. 270 liters of suspension volume was processed in the first experiment and 330 liter in the second one. Table 3 shows the estimated and experimental values of moisture content, the mean diameter particles and the recovery yield.

The error of estimation was calculated as:

$$\text{Error of estimation: } \frac{\text{Value}_{\text{experimental}} - \text{Value}_{\text{estimatedl}}}{\text{Value}_{\text{experimental}}} * 100 \qquad (3)$$

The results at industrial scale (Table 3) have a good agreement with the predicted value from empirical model, taking place the most sensitive variation in the diameter of particles, similar to the obtained results at the laboratory level. The moisture content, the mean particle diameter and the recovery yield are similar to those calculated by the models obtained from the laboratory studies. The recovery yield however is smaller than those obtained at laboratory scale. But it can be considered as a satisfactory result because it is in the range of reported values for industrial process using similar equipment [26]. The Vimang powder was easily recovered at the end of the cyclone. The lower product lost can be explained by the existence of sticking on the chamber walls.

CONCLUSIONS

The results reported in this article demonstrate the significant impact of the spray drying conditions on final product properties. In this work were optimize the operating conditions; inlet temperature of air, the concentration of solids of the feed and the speed of rotation of the centrifugal disk, at laboratory level for obtaining a novel process of drying of the Vimang powder. The scale up of the process was done from the laboratory equipment to the industrial level. The operating parameters permitted obtained average recovery yield higher at 84.1 % and guarantee the specifications quality the active ingredient Vimang.

NOMENCLATURE

C feed concentration (w/v %)
C_T process cost
D_p average particle size (μm)
M weight of the final product (kg)
M_H moisture content (%)
P pressure of the centrifugal disk (bar)
T inlet temperature of air (°C)
V volume of the feed (L)
Y recovery yield (%)

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