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Chemical Engineering Communications

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gcec20>

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Published online: 03 Nov 2008.

To cite this article: C. R. F. Souza, M. W. Donida, S. C. S. Rocha & W. P. Oliveira (2008) THE ROLE OF COLLOIDAL SILICON DIOXIDE IN THE ENHANCEMENT OF THE DRYING OF HERBAL PREPARATIONS IN SUSPENDED STATE, *Chemical Engineering Communications*, 196:3, 391-405, DOI: [10.1080/00986440802359543](https://doi.org/10.1080/00986440802359543)

To link to this article: <http://dx.doi.org/10.1080/00986440802359543>

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The Role of Colloidal Silicon Dioxide in the Enhancement of the Drying of Herbal Preparations in Suspended State

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*Recently, some research groups have been developing studies aiming to apply spouted beds of inert particles for production of dried herbal extracts. However, mainly due to their complex composition, several problems arise during the spouted bed drying of herbal extracts such as bed instability, product accumulation, particle agglomeration, and bed collapse. The addition of drying carriers, like colloidal silicon dioxide, to the extractive solution can minimize these unwanted effects. The aim of this work was to study the influence of the addition of colloidal silicon dioxide on enhancement of the performance of the drying of hydroalcoholic extract of *Bauhinia forficata* Link on a spouted bed of inert particles. The physical properties of the herbal extract and of its mixture with colloidal silicon dioxide at several concentrations (20% to 80% related to solids content) were quantified by determination of the surface tension, rheological properties, density, pH, and contact angles with the inert surfaces. Drying performance was evaluated through determination of the elutriation ratio, product recovery ratio, and product accumulation. The product was characterized through determination of the thermal degradation of bioactive compounds and product moisture content. The results indicated that the rheological properties of the extracts and their preparations, the contact angle with inert material, and the work of adhesion play important roles in the spouted bed drying of herbal extracts. Higher concentration of the drying carrier significantly improved the spouted bed drying performance.*

Keywords Colloidal silicon dioxide; Drying; Herbal extract; Spouted bed; Stickiness

Introduction

The drying of pastes, solutions, and suspensions on the surface of inert particles in suspended state is a simple and well-recognized technique for non-sticky materials. This drying concept has some important advantages compared to other drying systems, such as higher capacity per unit volume of the dryer, lower energy, and reduced specific air consumption. The high drying efficiency results from the large contact

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area and from the large temperature difference between the inlet and outlet air (Zeljko et al., 2000).

The technique involves suspending a bed of inert bodies with warm air and spraying liquid material into the bed, thus coating the bodies with a thin film. The warm air dries the film, which then becomes friable and fractures due to the attrition produced by the colliding bodies. The fractured film is removed with the exit air to be collected as the product and the film-free bodies are recycled by the suspended bed to be recoated. The process has two components: drying of the layer of liquid material followed by removal of the dried film. The rewetting of the inert bodies without previous removal of dried film or accumulation of the feed material within the bed may be considered limiting factors for this drying method (Barret and Fane, 1989; Markowski, 1993).

The product properties and the conditions of stable operation that result in the removal of solids without agglomeration of the inert particles are dependent on the system configuration and several operating parameters such as the flow rates of the liquid material and of the drying gas and the physical and chemical properties of the solid and liquid materials (Spitzner-Neto et al., 2002; Maialle et al., 2002; Souza and Oliveira, 2002, 2005; Medeiros et al., 2002; Kudra, 2003; Daleffe and Freire, 2004).

In general, powdered products in the amorphous state (glassy) are generated by spouted bed drying, mainly due to the fast moisture removal. Solids in amorphous state have a very high viscosity ($>10^{12}$ Pas). With the increase of product temperature during drying, the viscosity may decrease to a critical value of around 10^7 Pa, and the material first becomes sticky (Goula and Adamopoulos, 2005). This critical viscosity is reached at temperatures 10° – 20° C above the glass transition temperature, T_g . The drying of sticky products is complicated. During the drying they may remain either as a syrup or stick to the surface of the dryer walls, leading to low product yield and operating problems (Jaya et al., 2002). Additionally, the stickiness property depends on the interfacial surface energy of contacting materials.

Recently, some research groups have been developing studies aiming to apply spouted beds for production of dried herbal extracts. However, the drying of herbal extracts on spouted beds is not a simple task. Herbal extracts have significant contents of organic acids, lipids, sugars, lignin, carbohydrates, proteins, resins, etc. (Chu and Chow, 2000). Most of these substances have low glass transition temperature and are highly hygroscopic and thermoplastic. These characteristics may be associated with some operating problems that arise during spouted bed drying of herbal extracts such as bed instability, product accumulation, adhesion to dryer walls, agglomeration, and spout collapse.

According to several authors, factors such as physical properties of the feed composition and of contacting materials (surface tension, rheological properties, density, pH, and contact angle between them) may affect the performance of the drying/coating processes (Vieira et al., 2004; Donida et al., 2005; Bhandari and Howes, 2005). The properties of the feed composition can be modified with the addition of drying carriers such as maltodextrins, starches, and colloidal silicon dioxide.

In this work, the influence of the addition of colloidal silicon dioxide and drying conditions on enhancement of spouted bed performance during drying of hydroalcoholic extract of *Bauhinia forficata* Link was studied. The effect of the drying carrier (20% to 80% relative to the solids content) on physical properties of the herbal extract was quantified by determination of the rheological properties, surface

tension, density, pH, and contact angles as well as the work of adhesion on inert particles and stainless steel (dryer surface). Drying runs were conducted at static bed heights of 7 and 14 cm, feed flow rates of the herbal extract of 15% and 45% of the evaporative capacity of the dryer, and ratios of the drying gas flow rate to the gas flow at minimum spouting condition of 1.4 and 1.8. The drying temperature was maintained at 150°C.

B. forficata was chosen as a model herbal material due to its extensive use in folk medicine, mainly as a hypoglycemic, depurative, and diuretic (Yeh et al., 2003). Antioxidant properties (Damasceno et al., 2004; Sousa et al., 2004) and anticoagulant and antifibrinolytic activities against *Bothrops jaracussu* venom and its isolated thrombin-like serine protease enzyme induced by aqueous extract from this plant have been also reported (Oliveira et al., 2005).

Materials and Methods

Herbal Extract Preparation

Dried leaves of *B. forficata* Link were acquired from a Brazilian producer (Santa Rosa's Farm, Vinhedo, São Paulo, Brazil). The dried leaves were comminuted in a knife mill. Average particle size and size distribution was determined by sieving 200 g of the powdered product in a Bertel shaking device (Bertel Indústria Metalúrgica Ltda, Caieiras, São Paulo, Brazil) for 20 min, using standard sieves of 150, 300, 425, 500, and 600 µm. The experimental result of particle size distribution in a weight basis was fitted to the Rosin-Ramler-Benneth (RRB) model, according to the following equation ($R^2 = 0.994$):

$$X = 100 \cdot (1 - e^{-(dp/324.08)^{1.80}}) \quad (1)$$

The mean diameter of the powdered material was 265 µm, with 80% of the distribution having a diameter lower than 425 µm.

The dried and powdered leaves of *B. forficata* were placed in contact with five parts of ethanol:water solution (70:30 in weight) at temperature of 50°C in a cylindrical jacketed stirred vessel (height 340 mm, diameter 220 mm, and total capacity 12 L), connected to a heating circulating bath with temperature control (Brookfield TC-500). The extraction time was 60 min and the stirring rate was 200 rpm. After extraction, the crude extract was filtered in a vacuum system using filter paper (grade 80G) and concentrated three times in a rotary evaporator at 50°C and vacuum pressure of 700 mm Hg.

Drying Carrier

Several preliminary drying tests were carried out in order to evaluate the effect of the drying carriers, namely, cornstarch, manioc starch, maltodextrin (DE 14 and 20), and colloidal silicon dioxide on the enhancement of the drying performance. These drying aids resulted in low product recovery mainly due to adherence of the product to the surface of inert particles and drying chamber walls, which led to particle agglomeration and spout collapse in a relatively short processing time (data not shown). An exception was observed for colloidal silicon dioxide (Tixosil 333), which significantly improved the drying behavior. This behavior may be attributed to the

anticaking and free-flowing donating characteristics of colloidal silicon dioxide. Its most probable action mechanism is the formation of a monolayer around the herbal extract particles, which may keep the particles apart, reducing to a minimum the van der Waals adhesion forces between them. This material presents chemical inertness, innocuousness, thermal stability, and safety, which are required properties of a genuine pharmaceutical drying excipient. Thus, it was decided to investigate the role of colloidal silicon dioxide as a drying carrier in subsequent tests. This drying carrier (Tixosil[®] 333, Rhodia of Brazil, São Paulo, Brazil) at proportions of 20% to 80% relative to the solids content was added in two different ways: before extract concentration and after extract concentration. The effects of this carrier on the physical properties of the herbal extract were quantified by determination of the surface tension, rheological properties, density, pH, and contact angle between extract and inert material. The extracts added with carriers were submitted to the spouted bed drying.

Physical and Chemical Characterization of Extract and Carrier Preparations

The chemical composition of the concentrated extract was determined by the analytical methods reported in the literature, such as titratable acidity, high and low molecular weight carbohydrate, reducing sugar, lipids, and protein. These experiments were carried out in order to identify potential factors influencing the spouted bed behavior during drying of the herbal extract.

The rheology of the herbal extract preparations with and without the presence of drying carrier at varied concentrations was characterized by the determination of the shear stress, τ , and apparent viscosities, μ , at varying shear rates, $\dot{\gamma}$, using a Brookfield rheometer, model LV-DVIII (coaxial cylinder geometric system with small sample adapter, Brookfield Engineering Laboratories Inc., USA), connected to a circulating bath TC-500. The measurements were made at temperatures of 25° and 75°C.

The surface tension of the herbal extract preparations was measured with a Fisher Scientific Surface Tensiomat 21 (USA), using the ring method at controlled room temperature (25°C). The assays were carried out in quadruplicate.

Density measurements were carried out by picnometry (in triplicate). The pH was measured by a Micronal pH meter, model B474 (Brazil). The results were expressed by averaging three determinations.

The wettability between the herbal extract preparations and the solid surfaces (Teflon and stainless steel) was quantified by contact angle measurements (Tantec contact angle meter, Lunderskov, Denmark). Samples of 15 μ L were used in all measurements performed. Sessile drops (sitting drops that rest on a substrate below or bubbles that rise up against a substrate from below) were formed on the surface of the test materials (Teflon and stainless steel). The results are expressed by the average and standard deviation, according to the procedure found in Vieira et al. (2004).

Detachment tests of the extract and carrier preparations were also performed on small surface plates (Teflon and stainless steel). The plates, previously submitted to heat treatment at 105°C for 1 h to remove residual water from the surface, were positioned in a support. A thin layer of the concentrated herbal extract and of its preparations with drying carrier (20% to 80% of Tixosil) were uniformly spread ($\cong 0.10$ mm) over the surfaces and placed in a circulation oven at $75.0 \pm 0.5^\circ\text{C}$. The drying and the film detachment were visually monitored and registered by digital pictures, following the procedure presented in Collares et al. (2004).

Spouted Bed Drying

The drying experiments were carried out in a home-made conical-cylindrical spouted bed, with conical base with an internal angle of 40° and inlet orifice diameter of 33 mm. Connected to the conical base is a cylindrical column with a diameter of 150 mm and height of 400 mm. The upper part of the equipment is composed of another cone and a Lapple type cyclone with a diameter of 95 mm, having a cut diameter of about $4.1 \mu\text{m}$ (for the experimental conditions used). All parts are made of stainless steel. Figure 1 shows a schematic diagram of the dryer setup and its accessories. Concave cylindrical Teflon particles with a mean diameter of 5.45 mm and density of 2160 kg/m^3 were used as inert material. The main components of the system are a 7.5 HP blower, a flow meter, an electric heater, and a temperature controller. The extract feed system consists of a double fluid atomizer with internal mixing (0.8 mm hole), a peristaltic pump, and an air compressor.

The drying operation started with the introduction of a given load of inert material into the equipment. Spouting occurred when air was injected at the base of the bed at the appropriate flow rate. When the spout was established the spouting gas was heated to the temperature of 150°C . Once this temperature was reached, feeding of the mixture extracts and drying carrier (20% to 80% of the solids content) at preset values of flow rate and atomizing air was started. The extract flow rate was fixed at 15% or 45% (8.0 and 33.2 g/min) of the evaporative capacity of the dryer. Data of evaporative capacity of the spouted bed dryer were presented elsewhere (Souza and Oliveira, 2002). Table I shows the operating parameters of the spouted bed dryer. Measurements of the outlet gas temperature, T_{go} , were taken at regular intervals in order to detect the moment when the process attained steady state ($\pm 15 \text{ min}$). Once the steady state was attained, the dried extract was withdrawn over a period of 30 min. The samples were used for physical and chemical characterization of the dried product.

The powder properties were characterized through determination of the thermal degradation of marker compounds, D_F , and the product moisture content, X_p . The

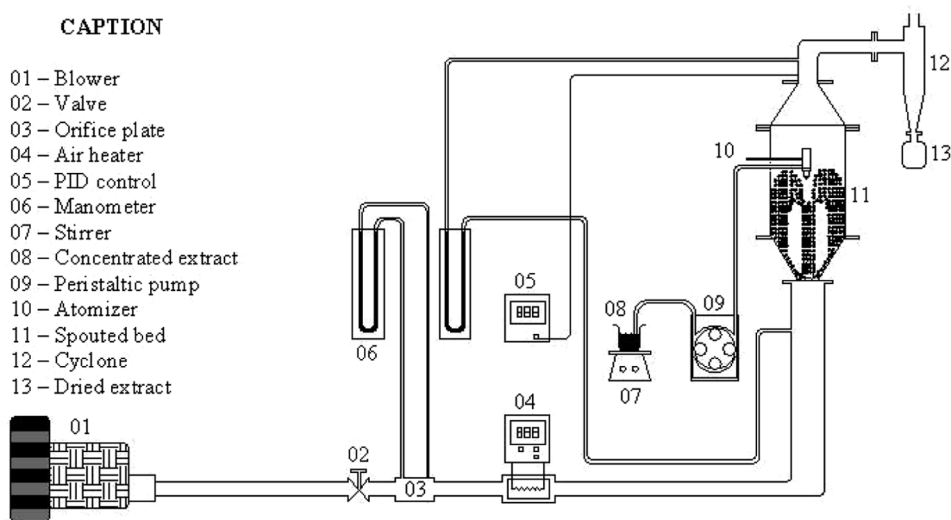


Figure 1. Schematic diagram of the dryer setup and its accessories.

Table I. Drying conditions

Drying parameter	Value
Inlet gas temperature (T_{gi}), °C	150
Drying gas flow rate (W_g), kg/s	0.0340 and 0.0437
Q/Q_{ms}	1.4 and 1.8
Atomizer system position	Top spray
Feed atomizing air, L/min	15
Pressure atomizing air, kgf/cm ²	2.0
W_s , g/min	
(15%)	8.0
(45%)	33.2
Static bed height (H_0), cm	7 and 14
Mass of inert material (M_i), g	498.0 and 1585.3

evaluation of thermal degradation during processing stages was performed by UV spectrophotometry. The analytical methodology for quantification of the marker compounds (total flavonoids) was presented by Souza (1997). Absorbance was measured at 425 nm using an HP 8453 spectrophotometer running the software HP Chem-Station[®]. Total flavonoid was selected as chemical marker since *Bauhinia* species are characterized by the accumulation of flavonoids (glycosides and aglycones) in the leaves (Jorge et al., 2004). The flavonoids represent a group of organic compounds that have an important role as antioxidants, presenting high reactivity to O₂. Phytochemical and pharmacological studies carried out with this plant species have demonstrated the presence of several classes of organic compounds of medicinal interest including the flavonoids (Silva and Cechinel-Filho, 2002). Product moisture content was determined by the oven drying method (World Health Organization, 1998).

To evaluate the system performance, the mass of product accumulated on the inert material and in the drying chamber and the product collected by the cyclone were measured. These data were used for the determination of the elutriation ratio, E, the product recovery ratio, R, and the product accumulation, Ac, which were estimated by mass balance in the dryer using Equations (2) to (4) presented in Table II. In these equations, $W_s C_s \theta_p$ is the total solids mass introduced into the dryer; M_c is the total mass collected by the cyclone and M_i and M_f are, respectively, the total bed mass at the beginning and at the end of the process.

Table II. Parameters used for evaluation of the spouted bed drying performance

Equations	
$R = \frac{M_c(1-X_p)}{W_s C_s \theta_p} 100$	(2)
$E = 100 - Ac - R$	(3)
$Ac = \frac{(M_f - M_i)(1 - X_p)}{W_s C_s \theta_p} 100$	(4)

Results and Discussion

The extracts and preparations were characterized through the determination of chemical composition, rheological properties, surface tension, density, pH, and contact angle with Teflon and stainless steel. These measurements were carried out in order to identify the properties that could play important roles in the drying performance. Table III presents the results of the chemical properties of the concentrated extract. These results confirm that the herbal extract is a complex composition of organic acids, carbohydrates, reducing sugars, lipids, and proteins. The knowledge of the chemical composition of the extract becomes important due to the fact that sugars, lipids, organic acids, proteins, and carbohydrates may have positive or negative effects on drying. Table III shows that the herbal extract presents high LMW carbohydrates (13.64 ± 0.42 mg/g of concentrated extract). The high hygroscopic and thermoplastic natures of these compounds are associated with problems such as adhesion to dryer walls, difficult handling, and caking during storage. Thus, the use of drying carriers to facilitate the drying and to improve the transport and storage properties of the herbal dried extracts is practically unavoidable (Bhandari et al., 1993).

Benali and Amazouz (2002) studied the efficiency of the jet spouted bed dryer with inert particles for the processing of meat-rendering slurry to obtain a dry marketable product. The authors suggest that the presence of fat may increase the predisposition of the dry powder to stick on the dryer wall and inert particles, mainly due to adhesion and cohesion phenomena.

Several drying runs at varied operating conditions and concentrations of drying carriers (Tixosil 333) were conducted. Table IV presents the results obtained. They show that the drying conditions as well as the concentration of the drying carrier have significant impact on the spouted bed drying behavior. The increase in the concentration of Tixosil 333 enhances the drying performance, reducing significantly the product accumulation in the bed with consequent enhancement of the product recovery ratio. Significant parcels of the dried product were carried away (elutriated) by the exhaust gas. However, the powder losses can be easily handled using more efficient powder separation systems, such as, for example, bag filters and/or electrostatic precipitators.

Table III. Chemical characterization of the concentrated herbal extract

Assay	Results	Reference
Titrateable acidity (% v/w)	0.92 ± 0.11	Instituto Adolfo Lutz (1985) method 13.6.2
Carbohydrate ^a HMW (mg/g _{ext})	0.24 ± 0.01	Chu and Chow (2000)
LMW	13.64 ± 0.42	
Reducer sugar (% w/w)	4.07 ± 0.04	Instituto Adolfo Lutz (1985) method 4.13.2
Lipids (% w/w)	14.66 ± 0.51	Instituto Adolfo Lutz (1985) method 4.10
Protein (mg/mL _{ext})	8.71 ± 0.09	Bradford (1976)

^aHigh molecular weight (HMW) and low molecular weight carbohydrate (LMW). Results related on solid contents (dry base).

Table IV. Drying parameters and experimental results obtained

Variables					Experimental results							
T _{gi} (°C)	Q/Q _{ms} (-)	W _s (g/min)	H ₀ (cm)	Carrier (%)	T _{go} (°C)	T _{amb} (°C)	UR (%)	R (%)	Ac (%)	E (%)	D _F (%)	X _p (%)
150	1.4	32.9	14	20	113.3	24.2	62.4	13.80	76.38	9.82	54.6	10.48
150	1.4	33.8	14	40	116.8	25.6	66.3	45.42	28.71	25.87	22.0	6.03
150	1.4	33.8	14	60	115.4	25.1	63.9	45.14	26.69	28.17	9.0	5.92
150	1.4	33.6	14	80	114.0	25.9	68.7	45.90	16.33	37.17	0.1	4.67
150 ^a	1.8	8.1	7	20	116.6	25.2	73.5	20.57	59.31	20.12	9.2	12.58
150 ^a	1.8	7.7	7	40	118.3	26.0	70.7	49.80	19.06	31.14	10.0	8.13
150 ^a	1.8	8.1	7	60	121.4	23.4	74.7	75.40	9.66	14.94	7.0	6.13
150 ^a	1.8	8.0	7	80	119.4	26.3	66.4	70.05	12.25	17.70	6.7	6.78
150	1.8	8.1	7	20	116.7	26.6	48.6	22.35	46.16	31.49	7.9	16.60
150	1.8	8.2	7	40	120.9	23.6	54.2	52.84	24.63	22.53	14.7	12.52
150	1.8	8.3	7	60	127.9	24.5	50.9	67.04	15.27	17.69	7.3	8.06
150	1.8	8.4	7	80	129.2	25.4	47.8	65.98	13.50	20.52	8.0	5.98

^aExtract and drying carrier concentrated together.

The favorable results obtained may be attributed to the anticaking and free-flowing donating properties of this drying carrier, which improves the powder flow inside the dryer and reduces the attractive forces between the extract particles and Teflon beads and equipment wall. It can be also observed in Table IV that the increase of the concentration of the drying carrier has a protective effect on the active marker compounds (total flavonoids), reducing their degradation. In the same way, the product moisture content shows a tendency to decrease conversely with the amount of colloidal silicon dioxide added.

According to several authors, factors such as rheological and surface properties of the liquid material influence the performance of the drying/coating processes (Vieira et al., 2004; Donida et al., 2005; Bhandari and Howes, 2005). The rheology, surface tension, density, pH, contact angles, and the work of adhesion on Teflon and stainless steel were determined for the concentrated extracts and their preparations with drying carrier (Table V) in order to evaluate the effects of these properties on the spouted bed performance during drying of the herbal extract.

Figures 2(a)–(c) present the rheological behavior of the herbal extract and its preparations with the drying carrier ((a) Tixosil added to herbal extract before concentration, measurement at 25°C; (b) Tixosil added to extract after the concentration step, measurement at 25°C; and (c) Tixosil added to extract after the concentration step, measurement at 75°C). The temperature of 75°C was used in order to simulate the rheological characteristic of the herbal extract at high temperatures, like those prevailing inside the spouted bed dryer. The mean viscosity represents the average values obtained between the shear rates of 50 and 300 s⁻¹. Figures 2(a) and 2(b) show that the alterations in the rheological behavior of the herbal extract preparations were more significant with the addition of the drying carrier before the concentration stage. These differences may be due to the thermal treatment suffered by the herbal preparations containing colloidal silicon dioxide during concentration, when the

Table V. Physicochemical properties of the concentrated extract and its preparations

Properties	EC	EC20	EC40	EC60	EC80
Carrier (%)	0	20	40	60	80
μ_{ap} (cp)	3.73 \pm 0.34 25°C ^a 3.73 \pm 0.34 25°C 1.29 \pm 0.18 75°C	4.36 \pm 0.22 4.35 \pm 0.23 2.09 \pm 0.41	5.75 \pm 0.32 4.08 \pm 0.18 4.48 \pm 1.63	7.28 \pm 0.42 5.95 \pm 0.34 6.48 \pm 1.78	9.09 \pm 0.43 5.28 \pm 0.29 8.96 \pm 1.70
θ (°)	54.9 \pm 1.9 Teflon 12.9 \pm 2.3 Stainless steel 30.6 \pm 0.1 0.938 \pm 0.012	52.4 \pm 1.9 21.1 \pm 2.2 29.9 \pm 0.1 0.954 \pm 0.007	54.9 \pm 2.3 19.9 \pm 2.2 30.5 \pm 0.2 0.966 \pm 0.002	57.2 \pm 2.5 27.1 \pm 3.6 30.3 \pm 0.3 0.980 \pm 0.011	58.1 \pm 2.9 21.2 \pm 2.4 29.8 \pm 0.3 0.978 \pm 0.014
W_{ad} (dyn/cm)	5.92 \pm 0.04 PH 48.2 Teflon 60.4 Stainless steel	5.99 \pm 0.05 48.1 57.8	5.85 \pm 0.02 48.0 59.2	5.60 \pm 0.07 46.7 57.3	5.58 \pm 0.07 45.5 57.6

^aExtract and drying carrier concentrated together.

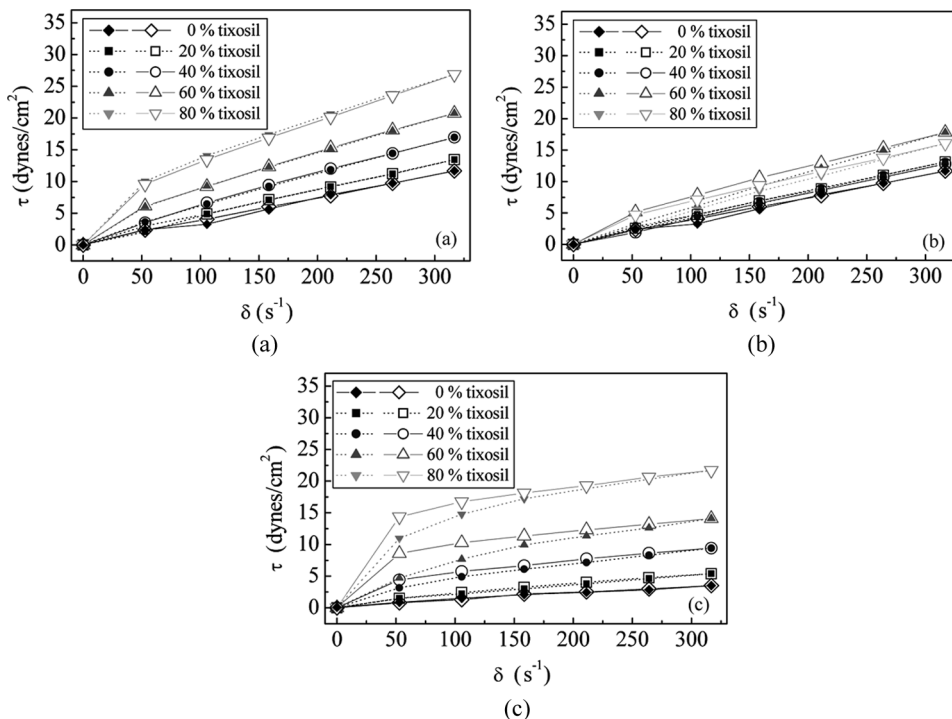


Figure 2. Rheological behavior of the herbal extract and its mixtures with the drying carrier; (a) Tixosil added before the concentration, measurement at 25°C, (b) Tixosil added after the concentration, measurement at 25°C, (c) Tixosil added after the concentration, measurement at 75°C.

composition is heated to 50°C for a long time period. This thermal treatment would promote a better dispersion between the Tixosil and water-insoluble substances like the plant oleoresins. The results presented in Figure 2(a) are similar to the ones shown in Figure 2(c), obtained at a temperature of 75°C, and indicate a remarkable alteration in the rheological properties of the preparations containing 60% and 80% Tixosil, which present higher values of the average viscosity (see Table V). Interestingly, the increase in the mean viscosity was followed by a tendency of reduction in product accumulation, suggesting the existence of a critical value for this parameter ($\cong 4.5$ cp) below which the spouted bed drying operation becomes challenging (Figure 3). The results presented in Figure 3 reveal that the change of the feed composition viscosity caused by the addition of the drying carrier together with the thermal treatment plays an important role in the spouted bed drying behavior. This increment of the viscosity leads to a reduction of the surface area wetted by the atomized extract during drying (higher resistance to flow). The contact area reduction would reduce the formation of liquid bridges between the dryer wall/product (adhesion) and product/product (cohesion). If the contact area is very small and the drying continues, then the dried particles may be carried away from the equipment surfaces (dryer wall and Teflon beads). The increase in the viscosity of the herbal preparation was followed by a slight increase of the contact angle, as can be observed in Table V. The increase in the contact angle could also contribute to

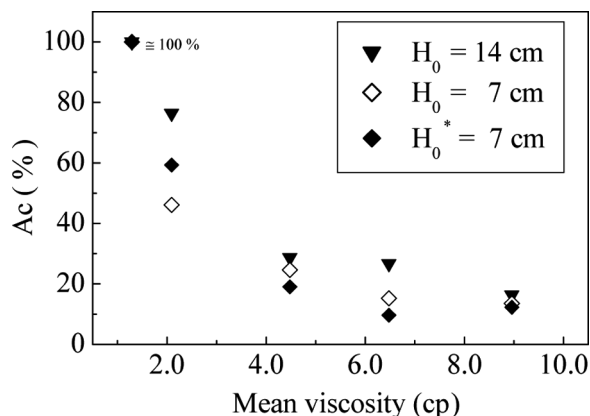


Figure 3. Product accumulations during spouted bed drying as a function of the mean viscosity of the extract preparations at 75°C.

the weakening of the adhesive bonds between the atomized drops and the inert material and dryer wall surfaces. As expected, the data of surface tension presented in Table V show that the extract preparations were not affected by the drying carrier concentration and by the thermal treatment applied. The values of surface tension were between 29.8 and 30.6 dynes/cm.

Adhesion is the physical phenomenon that attracts atoms together to form molecules and molecules together to form liquid and solids. Surface forces and surface energy are important factors influencing the adhesive property of material. In liquid systems the surface tension plays a fundamental role in adhesion. The wetting of the solid surface is related to the energy of adhesive and substrate (also called adherent). If the liquid wets the surface it will spread out on the solid surfaces. If the adhesive has low energy levels or low surface tension, high-energy level solids will attract it; the contact angle decreases and the wetting is effective. If the surface of the solids has lower energy, the contact angle is high and the wetting is poor. In other words, to achieve wetting, the surface energy of the solid material should be higher than that of the liquid (Bhandari and Howes, 2005). Using the experimental values of the contact angle and surface tension presented in Table V, the work of adhesion for the pairs of herbal extracts and their preparations with colloidal silicon dioxide/inert material and herbal extract preparations/dryer wall surface could be estimated through the following equation:

$$W_{ad} = \sigma \cdot (1 + \cos.\theta) \quad (5)$$

A tendency of reduction of the work of adhesion between the Teflon and herbal extracts preparation with the increment of the concentration of the drying carrier can be seen in Table V. This trend was also followed by a decrease in product accumulation on Teflon beads. A lower work of adhesion implies weak adhesive bonds, contributing to the improvement of drying performance. Nevertheless, the close values of surface tension and contact angles for the different herbal extract preparations gave similar values of the work of adhesion, making it difficult to detect significant effects of the liquid-solid wettability on the spouted bed performance during drying of herbal extracts with and without drying carrier.

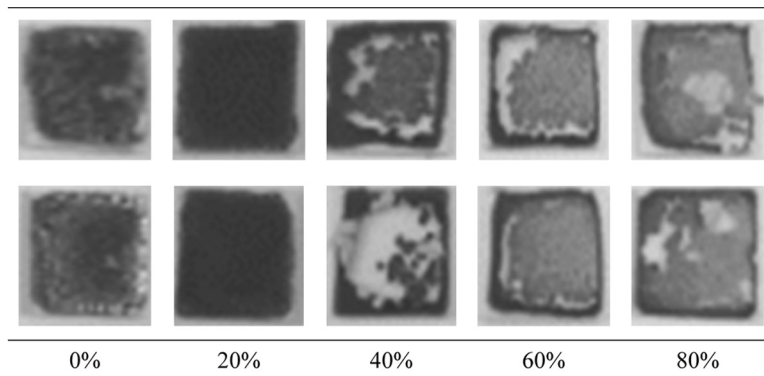


Figure 4. Detachment of the dried extract films containing different concentrations of colloidal silicon dioxide from Teflon surfaces.

The positive effect of addition of colloidal silicon dioxide on the detachment of the dried material from the drying surfaces (Teflon and stainless steel) could be verified through a simple assay. A thin layer of herbal extract and its preparations with drying carrier (at proportions of 20% to 80% relative to the solids content) were uniformly spread ($\cong 0.10$ mm) on clean and smooth surfaces of Teflon and stainless steel laminas and placed in a circulation oven at $75.0^\circ \pm 0.5^\circ\text{C}$ until dryness. The drying of the films was monitored visually and recorded by digital pictures according to the procedure presented by Collares et al. (2004). Figures 4 and 5 show typical pictures of the dried films on Teflon and stainless steel surfaces. Figure 4 shows that the detachment of the dried films containing colloidal silicon dioxide (at concentrations $\geq 40\%$) from Teflon surfaces occurred nearly spontaneously. On the other hand, in the stainless steel plates (Figure 5), the films of dried extracts remained adhered due to the low contact angle and consequently higher adhesion work. In practical

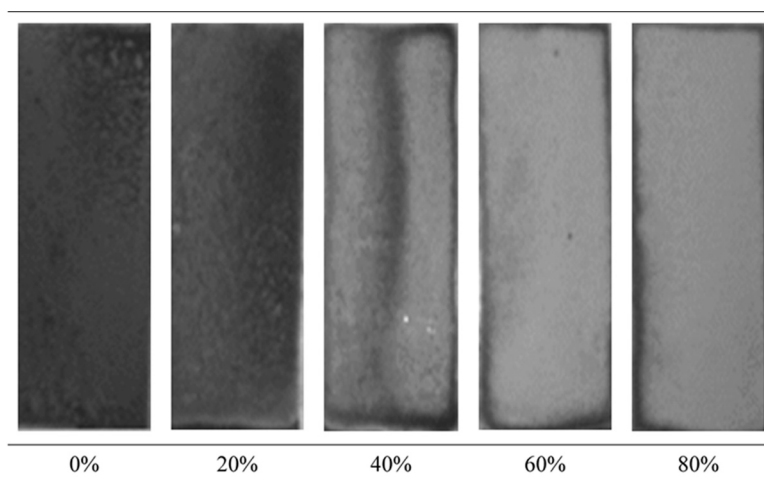


Figure 5. Detachment of the dried extract films containing different concentrations of colloidal silicon dioxide from stainless steel surfaces.

situations, impact and friction forces between the solid particles in the spouted bed dryer probably will enhance the removal rate of the dried product from the dryer surfaces.

Conclusions

Herbal extracts and their preparations present significant contents of organic acids, lipids, sugars, lignin, carbohydrates, proteins, and resins. This complex composition makes the drying of these products challenging. Adhesion and stickiness are common problems during the drying of these products. In this work the effect of the addition of colloidal silicon dioxide on the enhancement of the spouted bed drying behavior was studied. The experimental results show that the drying conditions as well as the concentration of the drying carrier used have significant impact on the spouted bed drying behavior. Increase in the concentration of colloidal silicon dioxide improves the drying performance, reducing significantly the product accumulation in the bed. Increase of the concentration of the drying carrier also showed a protective effect on the active substances, reducing their degradation during drying. In the same way, the product moisture content shows a tendency to decrease with the amount of the colloidal silicon dioxide added. These results have relations with physical, chemical, and rheological properties of the feed composition as well as the drying conditions used.

Acknowledgments

We gratefully acknowledge The State of São Paulo Research Foundation (FAPESP) and The National Council for Scientific and Technological Development (CNPq) for financial support.

Nomenclature

Ac	accumulation ratio, %, w/w
C_s	solids content, g/g
DE	equivalent dextrose
D_F	flavonoids degradation ratio, %, w/w
E	elutriation ratio, %, w/w
EC	concentrated extract
H_0	static bed height, cm
M_c	mass recovery by the cyclone, g
M_f	final bed mass, g
M_i	mass bed load, g
Q	gas flow rate, m ³ /min
Q_{ms}	gas flow rate at minimum spouting, m ³ /min
R	product recovery ratio, %, w/w
T_{gi}	inlet gas temperature, °C
T_{go}	outlet gas temperature, °C
UR	relative humidity, %
W_{ad}	work of adhesion, dyn/cm
W_g	spouting gas flow rate, kg/s

W_s	feed flow rate, g/min
X_p	moisture content, %, w/w

Greek Letters

$\dot{\gamma}$	shear rate, s^{-1}
θ	contact angle, $^\circ$
θ_P	processing time, s
μ	mean viscosity, cp
ρ	density, g/cm^3
σ	surface tension, dyn/cm
τ	shear stress, dynes/cm ²

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