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# Characterization of the Dispersion Behavior of Powders in Liquids

Carol Goalard\*, Arezai Samimi\*\*, Laurence Galet\*, John A. Dodds\*, Mojtaba Ghadiri\*\*

## Abstract

Powders dispersed in liquids to form suspensions are widely used as food products, medications, and occur in many stages in industrial processes, whether as a final or an intermediate product. To maximize the efficiency of a dispersion process, a sound understanding of the factors that influence and limit the formation of suspensions is necessary. These involve wetting characteristics based on surface energy and also the physical form of

the powders, such as particle size and the state of agglomeration. In this paper we examine the dispersion of talc powders either agglomerated in a conventional high-shear granulator or agglomerated using a combined method of electro-spray and freeze drying. The dispersion behavior of these agglomerates is studied as a function of the characteristics of the granules.

**Keywords:** dispersion, electro-spraying, freeze-drying, granules, high shear, wetting

## 1 Introduction

It is often necessary to disperse powders in liquids, for example in everyday life we disperse food products or medicines. Dispersion is also used in many stages of industrial processes, whether to give a final form to a product (e.g., a suspension) or as an intermediate production step. When the final product is a powder dispersed in a solvent (e.g., paint) the quality of dispersion defines the quality of the product. A sound understanding of the factors that can influence and limit the formation of dispersions is necessary in order to maximize the efficiency of a production process and the product quality.

The literature on dispersion is not extensive. Schubert [1] investigated modifications to powdered food products to promote rapid dispersion called “instantizing”. He studied the kinetics of dispersion of instant powdered milk in relation to the wetting characteristics such as the fat content of the milk and the state of agglomeration of the powder. Here we consider the effect of the structure of talc granules on their dispersion in aqueous media. Talc is chosen because it is widely used in many industrial sectors such as paper, paints, ceramics, cosmetics, and pharmaceuticals. It is a hydrophobic and lamellar powder that is hard to disperse in water and hard to granulate. A comparison is made of the dispersion characteristics of two types of talc granules: (a) Fragile highly porous talc granules made by a novel method combining electro-spray and freeze-drying. (b) Strong dense talc granules made with a conventional high-shear granulator. The wetting characteristics of both types of granule have been determined by contact-angle measurements, Washburn capillary-rise experiments, and vapor-sorption isotherms measured by DVS. These results will be reported elsewhere but are not considered further here as they do not differ significantly except in their PEG content.

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## 2 Agglomeration

### 2.1 Electro-spray Freeze-dry Agglomeration

A novel agglomeration process has been used to prepare fragile highly porous spherical granules (Samimi et al. [2]). In this method an electric field is applied between a nozzle and an annular counter electrode to control the size of liquid droplets which are rapidly frozen when dripped into a bath of liquid nitrogen. When a liquid emerges from a nozzle in air at a sufficiently small flow rate, drops are released individually from the tip of the nozzle. In this dripping mode, the application of an adequate electric potential to the nozzle increases the dripping frequency with a consequent reduction of the droplet diameter (Speranza et al. [3]).

Figure 1 shows a schematic drawing of the apparatus used to make the granules. The essential feature is a stainless steel nozzle (I.D. 1 mm, O.D. 1.48 mm) posi-

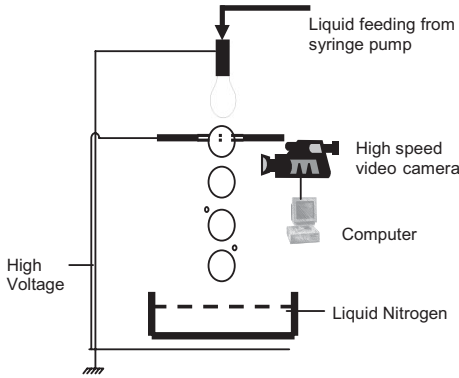


Fig. 1: Dripping arrangement.

tioned vertically above an annular stainless steel disk. A high-voltage DC power supply is connected to the nozzle, while the stainless steel disk (internal hole 30 mm, external diameter 100 mm) is earthed. The liquid is pumped to the nozzle using a syringe pump SP 200 IZ series, which allows to feed the liquid with a wide range of flow rates. The internal diameter of the Monojet syringe is 38.4 mm and its volume is 140 ml. The optimum liquid flow rate feed in this work is 2 ml/min for which the mode of flow from the nozzle was dripping. The experiments were conducted at room temperature. The electric field was supplied by a high-voltage source Hartley Measurements model 421. The applied DC voltage had a positive polarity and was varied from 0 to 10 kV.

Figure 2 shows the change in drop characteristics caused by applying an electric field to the nozzle. The drops are pulled down and the main droplet diameter is reduced while the dripping frequency is increased. The main

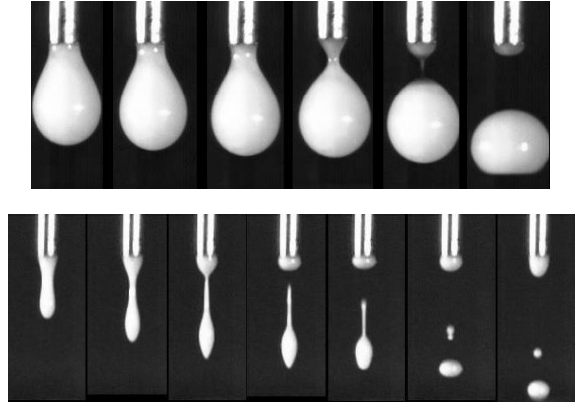


Fig. 2: Formation of drop without and with electrical field.

drop is accompanied by one or more satellites due to the rupture of the liquid filament between the main drop and the liquid meniscus left attached to the nozzle. The diameter and the size distribution of the main droplets depend on the voltage applied, the liquid flow rate, the liquid surface tension, the conductivity, and the viscosity of the liquid. The droplets produced in the dripping mode are collected in liquid nitrogen where they are rapidly frozen. The collected droplets are then freeze-dried in a SB4 laboratory freeze drier thus conserving the spherical shape of the granules.

Seven different suspensions of talc particles ( $d = 6 \mu\text{m}$ ,  $\theta = 2.38$ ) in polyethylene glycol and water have been used giving granules between 1 and 1.2 mm. The density of the granules was measured using a Micrometrics AccuPyc 1330 helium pycnometer. The BET specific area was determined using a Micromeritics Asap 2010 with argon. The porosity was measured with a mercury porosimeter Micromeritics AutoPore III. The granule characteristics are given in Table 1.

Table 1: Characteristics of electro-spray freeze dried granules.

PEG (%)	Talc (%)	Density	Porosity	$S_{\text{BET}}$
5	15	1.94	90	6.5
5	30	2.31	84	5.5
10	10	1.83	84	4.1
10	15	1.82	86	5.1
10	20	1.98	84	4.8
10	30	2.17	80	3.6
20	15	1.64	79	3.9

Figure 3 shows different ESEM views of electro-spray granules: A) a whole granule (10 % PEG, 30 % Talc); B) the external surface (10 % PEG, 20 % Talc); C) a fractured granule (10 % PEG, 30 % Talc); D) a granule with a scratched surface (5 % PEG, 30 % Talc); E) a detail of a broken granule (10 % PEG, 10 % Talc); F) detail obtained using a back-scattering electron (BSE) detector (5 % PEG, 15 % Talc).

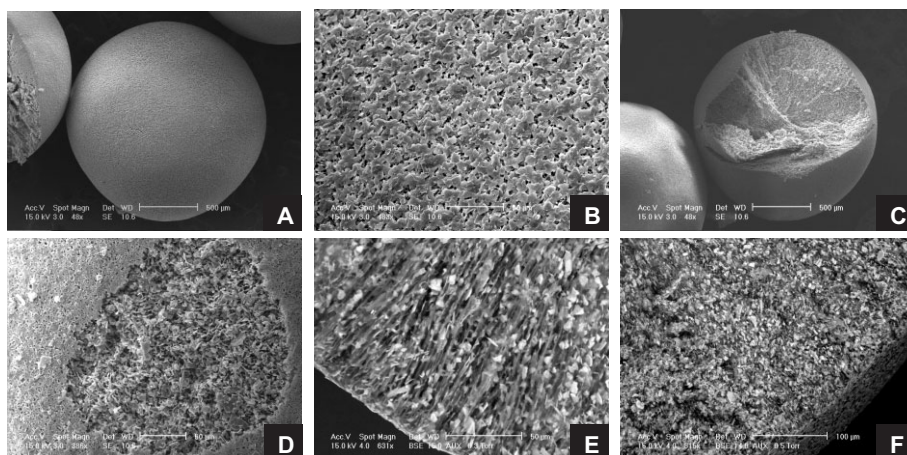


Fig. 3: Environmental Scanning Electron Microscopy images (ESEM) of the granules obtained by electro-spray and freeze-drying.

These photos show that the apparent smooth surface does have some detail and that the inside of the granule seems to have a lamellar structure below a surface skin. Finally, 3-F shows that the talc particles are uniformly and randomly distributed in the granule which is not the case for the PEG binder.

## 2.2 High Shear Granulation

Talc has been agglomerated in 200 g lots in a conventional 2 kW Diosna high-shear granulator with a 2 liter bowl and using a solution of 5 % polyethylene glycol in water as a binder (Chateau [4]). A preliminary study with a Caleva Mixer Torque Rheometer was carried out and led to the choice of a wetting ratio of 0.2 ml/g and a three-phase program for the speed of rotation. Phase I: 5 mins with a stirrer at 300 rpm and a chopper at 200 rpm. Phase II: 3 mins with a stirrer at 800 rpm and a chopper at 500 rpm, Phase III: 5 min with a stirrer at 500 rpm and a chopper at 200 rpm.

The resulting granules were sieved in 5 size classes. Observations by ESEM (see Figure 4) show that the talc granules produced by the Diosna high-shear granulator are roughly spherical and rather compact. The characteristics of the granules did not permit mercury porosimetry to be used and a measure of the porosity of the largest size class was determined by counting and weighing certain number of granules. This gave a value of 40 % porosity and was taken to be characteristic of the other size classes.

## 3 Dispersion

Two methods have been used to investigate the dispersion behavior of granules in liquids. The first method is based on a novel optical fiber sensor which allows the

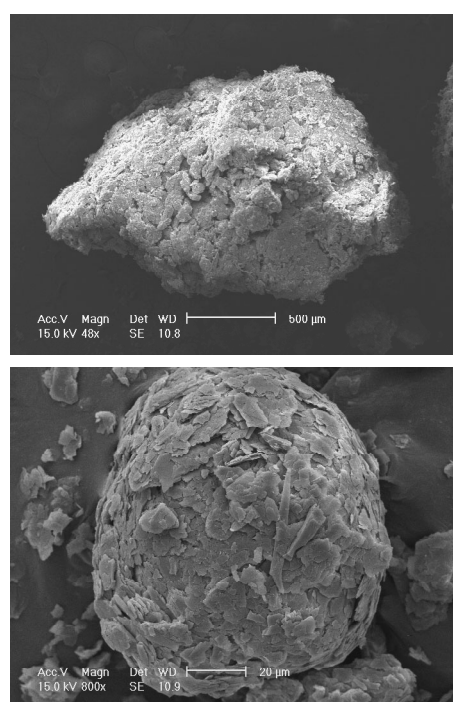


Fig. 4: Granules produced with the Diosna high-shear granulator.

increase in concentration of the particles to be followed as the dispersion proceeds. The second method uses an Insitex laser diffraction instrument in a Spraytech bench to follow the changes in particle-size distribution as dispersion proceeds.

## 3.1 Optical Fiber Sensor

This technique has been presented in detail elsewhere [5]. Essentially the particles are poured on the surface of the liquid in a baffled stainless steel vessel stirred by an impeller. Two mechanisms occur: The first is immersion

where the granules cross the liquid surface. The second is erosion or break-up where the talc particles become dispersed in the liquid. The signal from the optical fiber sensor gives curves as shown in Figure 5 which are fitted to a two-parameter exponential model.

$$X(t) = A(1 - \exp(-kt)) \quad (1)$$

$X(t)$  is the fraction of dispersed particles at time  $t$ .  $k$  ( $s^{-1}$ ) is the dispersion rate of the particles which includes the immersion and dispersion. The greater  $k$  is, the faster is the dispersion. The constant  $A$  corresponds to the final stage of the dispersion and  $A$  equals 1 when all the particles are dispersed in the liquid.

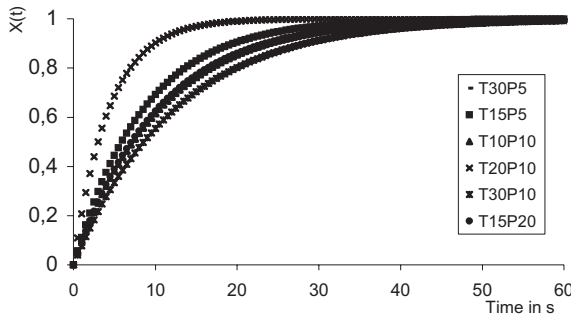


Fig. 5: Dispersion kinetics (Talc 30 %, PEG 5 %).

### 3.2 Insitec

The second method used for following the dispersion of the granules in liquids is with an Insitec EPCS in-line laser diffraction instrument fitted in a Malvern Spraytec bench holding a rectangular glass vessel (50 mm, 100 mm). This system gives a real-time continuous measurement of the particle-size distribution ( $d_{10}$ ,  $d_{50}$  and  $d_{90}$ ) and light obscuration related to the concentration of the dispersed suspension. In these experiments a constant mass of granules contained in a wire mesh holder is introduced into 1 liter of distilled water with minimal magnetic agitation to avoid the sedimentation of the particles. This mode of introduction means that there is no resistance to immersion and the only kinetic effect is that due to granule erosion and break-up.

Figure 6 shows that the particle size in suspension increases for the first 10 s after which there is a slow decrease until they stabilize at the particle size of the primary talc particles when disintegration is completed. The decrease in obscuration has been fitted by:

$$\frac{O(t)}{O_{(\infty)}} = \exp(-k't') \quad (2)$$

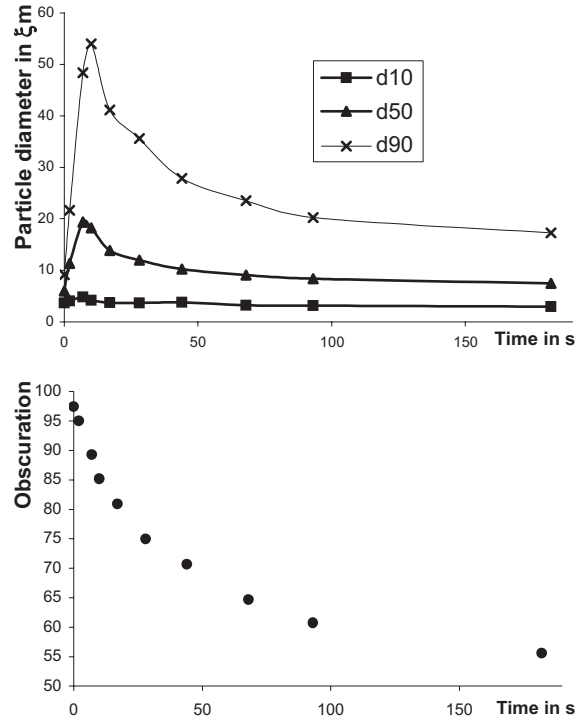


Fig. 6: Particle diameters and obscuration Insitec during the dispersion of granules (Talc 30 %, PEG 5 %).

where  $O(t)$  is the obscuration at time  $t$ ,  $O_{\infty}$  is the minimal obscuration at the end of disintegration.  $k'$  is the dispersion rate that does not include immersion.

### 3.3 Comparison

Tables 2 and 3 give the fitting parameters from the dispersion experiments where the  $k$ ,  $t$  and  $A$  values are determined with the optical fiber sensor and the  $k'$  and  $t'$  values are determined in the experiments with the Insitec. The  $t$  and  $t'$  values are those corresponding to 90 % of the final state of dispersion. Examining the dispersion times  $t'$  and  $t$ . For the electro-spray granules, the greater the talc content, the longer is the dispersion time  $t'$ , which does not include immersion. It may be that the  $t'$  values are also linked to the high PEG content, but the variability of the results does not allow us to confirm this. For the high-shear granules the dispersion time  $t$  (including immersion) passes through a minimum at a granule size of 500–630  $\mu m$ . It seems that granules smaller than this size do not cross the liquid surface as easily as heavier granules, and that granules larger than this optimum size take longer to break up and disperse. Similar observations were made previously by Schubert [1]. This observation can not be made for the electro-spray granules as they were all the same size. In general



Table 2: Dispersion parameters for electro-spray granules.

PEG (%)	Talc (%)	$k$ (s <sup>-1</sup> )	$k'$ (s <sup>-1</sup> )	$t$ (s)	$t'$ (s)
10	10	0.096	0.045	23	51
5	15	0.117	0.054	19	42
10	15	0.046	0.040	49	57
20	15	0.098	0.071	23	32
10	20	0.231	0.035	9	66
5	30	0.093	0.021	24	107
10	30	0.081	0.017	28	135

Table 3: Dispersion parameters for high shear granules (PEG 5 %).

Size (μm)	A	$k$ (s <sup>-1</sup> )	$k'$ (s <sup>-1</sup> )	$t$ (s)	$t'$ (s)
250–315	0.58	0.13	0.0022	17.3	1063
315–500	0.68	0.59	0.0103	3.9	222
500–630	0.59	0.62	0.0111	3.7	206
630–800	0.72	0.054	0.0089	42.3	257
800–1	0.67	0.052	0.0039	44.2	589

the electro-spray granules have higher  $t$  values than the high-shear granules but this does not take into account the size effect. Also for the electro-spray granules it can be seen that the higher the talc content, the lower is the  $k'$  value, meaning the longer the dispersion takes. It should be noted that those electro-spray granules which have a higher talc content are heavier, which facilitates immersion and affects the  $k$  values. The  $A$  values for the electro-spray granules were all equal to 1 suggesting that they all became fully dispersed to primary particles. This is not the case for the high-shear granules where  $A$  is much less than 1.

#### 4 Conclusion

The experiments with various sieve fractions of high-shear granules confirm that there is an optimum size of granule for rapid dispersion. The electro-spray granules with a very low bulk density and a high porosity are found to disperse completely into primary particles whilst, in the stirring conditions used here, the high-shear granules only reduce to small agglomerates (50 μm). This suggests that to achieve the best immersion and dispersion highly porous granules but also heavy granules are required.

#### 5 Nomenclature

$A$	–	constant in Eq. (1) corresponding to the quality of final dispersion
$k$	s <sup>-1</sup>	dispersion rate
$k'$	s <sup>-1</sup>	dispersion rate determined by Spraytec not including immersion
$O(t)$	–	obscuration at time $t$
$S_{BET}$	m <sup>2</sup> /g	specific surface by the BET method
$t$	s	time
$t'$	s	time in Spraytec experiments, not including immersion time
$X(t)$	–	fraction of particles dispersed at time $t$

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