

Improving the dispersion kinetics of a cocoa powder by size enlargement

Tuyet-Oanh Vu, Laurence Galet, Driss Oulahna, Jacques Fages

▶ **To cite this version:**

Tuyet-Oanh Vu, Laurence Galet, Driss Oulahna, Jacques Fages. Improving the dispersion kinetics of a cocoa powder by size enlargement. 7th Symposium on agglomeration, May 2001, Albi, France. p.739-748. hal-01809107

HAL Id: hal-01809107
https://hal.science/hal-01809107

Submitted on 27 Sep 2018

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Improving the dispersion kinetics of a cocoa powder by size enlargement

VU T.O., GALET L.,OULAHNA D., FAGES J.

Ecole des Mines d'Albi-Carmaux - Centre Poudres et Procédés - Albi - France

Abstract : The efficient dispersion of powders in liquids is required in many field. For this reason it is useful to understand the mechanisms and the physical properties of powders influencing the dispersion rate. Here we have chosen to work with a low-fat cocoa powder commonly used in food industry. Firstly we have determined the physical properties of the powder (densities, cohesion, flowability, size, surface energy and then measured the dispersion times of cocoa in two litres of water using a novel of an optical method. Further experiments, using the same technique, are performed using a larger grains obtained by granulating cocoa powder. It is found that the dispersion times are shorter for the granules and much shorter that the dispersion time for un-granulated powder. The dispersion times for granules increase with the size of granules.
Keywords : cocoa powder, dispersion, size, wet granulation, optical fibber bundle sensor

Résumé : La dispersion est un procédé très utilisé dans l'industrie, mais les paramètres gérant sa cinétique ont été peu étudiés. Nous avons voulu mettre en évidence les différents paramètres mis en jeu. Pour cela, nous avons choisi de disperser une poudre hydrophobe et insoluble. Notre choix s'est porté sur un cacao à faible teneur en beurre de cacao (10-12 %) mis en suspension dans de l'eau. Nous avons commencé par caractériser la poudre de cacao en terme de densités, taille, propriétés mécaniques, énergie de surface. Il a ensuite fallu faire le choix d'une méthode permettant de suivre de façon précise la dispersion. Un capteur à fibres optiques a été choisi. Les mesures de dispersion du cacao par cette technique ont montré que les temps de dispersion sont très longs. La mise en forme de la poudre par granulation en voie humide dans un mélangeur granulateur à haut-cisaillement permet d'améliorer les temps de dispersion. Nous avons donc pu montrer que l'augmentation de taille permet d'améliorer la dispersion.
Mots-clés : cacao, dispersion, taille, granulation, capteur à fibres optiques

INTRODUCTION

Powders are often used in industry in the form of a dispersion in a liquid. This is the case for cements, paints, pharmaceuticals and food powders. The different steps of which a powder becomes dispersed are : wettability, passing the surface, the sedimentation and the disaggregation. These different processes are affected by the way the operation is carried out : how the powder is poured into the liquid, how mechanical energy is used to agitate the liquid, also by the physical and chemical properties of the

dispersiong liquid (surface tension, density...)à and by the physical and chemical properties of the powder itself (particle size, density, surface). The latter can be modified by granulation and have a large effect on the dispersion kinetics.
Only few studies have dealt with the physico-chemical parameters involved in the dispersion of a powder in a liquid. In the food industry, different authors have began to study the use of granulation for obtaining instant powders [1, 2, 3, 4, 5]. but these studies often lack generality and do not deal with the importance of each parameter separately and how they interact together in dispersion kinetics. This study reported here is carried out in this context of studying the influence of some physical parameters on dispersion kinetics of powders in liquids.
The system chosen is to work cocoa powder in milk or water. This is an often encountered case of an insoluble, hydrophobic, but easy to formulate powder which is used is hard to disperse in milk or in water. We first present some physical characteristics of the cocoa powder, the experimental conditions to granulate it and the method used to follow the dispersion of cocoa particles in water. Finally we show the size influence of cocoa particles dispersion in water.

I. MATERIAL AND METHODS

I.1. The cocoa powder

Physical properties
The cocoa powder used is characterised in term of particle size, true, aerated , and packed density, flowability, porosity and surface energy. The size distribution was measured with a MALVERN 2000 laser diffraction instrument as a liquid dispersion in water. The true solid density is measured by means of a MICROMERITICS Accupyc helium pycnometer. Aerated and packed densities are measured using a HOSOKAWA PTN, a powder tester. The porosity is measured with a MICROMERITICS mercury porosimeter.

Surface energy
Stevens's method [6] was used to determine the surface energy of cocoa powder. This method was used by Cordier [7] to measure the surface energy of powder before granulation. It consists in sprinkling the powder on the surface of known surface tension solutions. These solutions are composed with different water-isopropanol ratios. The powder surface energy value is the surface tension of the mix for which the sprinkled powder is wet in less than one minute.

Results
Table 1 shows the characteristics of the cocoa powder.
With the aerated and packed densities ($D_{aerated}$ and D_{packed}), we can calculate Carr and Hausner indexes (I_{Carr} and $I_{Hausner}$) [8]. I_{Carr} and $I_{Hausner}$ characterise the flowability and the cohesive forces of a powder.

$$I_{carr} = \frac{D_{packed} - D_{aerated}}{D_{packed}} \quad (1)$$

$$I_{hausner} = \frac{D_{packed}}{D_{aerated}} \quad (2)$$

Tab. 1. Physical properties of cocoa powder

true density	$1.44 \pm 0{,}01$ g/cm^3
aerated density (Daerated)	$0.34 \pm 0{,}01$ g/cm^3
packed density (Dpacked)	$0.67 \pm 0{,}01$ g/cm^3
porosity	no intra-particular porosity
medium size	$d_{50} = 16$ µm $(d_{90}-d_{10})/d_{50} = 2.2$
I_{carr}	49 % very bad flowability
$I_{hausner}$	1,96 very cohesive
surface energy	$40 \leq \sigma \leq 50$ mN.m^{-1}

There is no intra-particular porosity in cocoa grains. The media particle size is quite low (16 µm), but the size distribution is large (span = 2.2). The powder is very cohesive and the flowability is bad. The surface energy is less than surface tension of water (72 mN.m^{-1}). The cocoa powder can't be wet by water.

I.2. Formulation

Wet granulation

The cocoa powder was agglomerated by wet granulation in a high-shear mixer granulator DIOSNA. The experimental conditions are given in table 2.

Tab. 2. Wet granulation conditions

mass of cocoa powder	200 g
water ratio (mass water/mass powder)	65 %
water flow	0.62 g.s^{-1}
mixing axe speed rotation (rpm = rotation per minute)	300 rpm
water addition time	210 s
granulation temperature	25°C

The binder water drip fed. After granulation, granules are dried in an oven at 45°C during five hours and separated in five granulometric classes by sieving.

Characteristics of the granules

We calculate the mass ratio of each particle size class and we measure the intra-granular porosity with a mercury porosimeter. The results are given in table 3. Wet granulation increases the size of the powder. All particle size classes are found to have the same porosity. The cohesion is low, and the flowability of each class is good.

Tab. 3. Size, mass fraction and granules porosity

class	size (µm)	mass fraction (% ± 0,1 %)	porosity (%± 1 %)	flowability	cohesion
1	d < 200	30.0	21	good	low
2	200 < d < 630	22.1	17	good	low
3	630 < d < 1000	13.3	20	good	low
4	1000 < d < 2000	24.6	20	good	low
5	2000 < d	9.9	21	good	low

I.3. Dispersion time measurement

Why an optical method ?
Several different methods are currently used in laboratories to measure dispersion times [1]. However cocoa suspensions are opaque making it difficult to observe the different phenomena involved in the dispersion. Crawley [9] used a turbidimetric measurement, but the cocoa particles are absorbent and this method is impossible. So we have chosen to use an novel optical method based on an optical fibber bundle sensor which allows determination of the fluctuations of volume concentrations and size in a suspension which can be interpreted in terms of dispersion quality.

Optical method description [10]
The optical method uses a central optical fibber emitting light (Fig. 1). This light is back-scattered by the particles in the suspension and received by a six-fibber crown around the central one. An electronic system converts the light intensity into a tension. A data acquisition scores the tension so the dispersion in function of time.

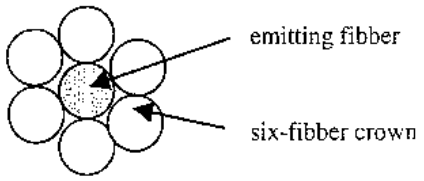


Fig. 1. Optical fibber bundle sensor

Measurement conditions
The apparatus must be calibrated with different volume ratios of cocoa. This is done by introducing a known amount of cocoa in two litres of water. This is thoroughly mixed by mechanical agitation until complete dispersion is obtained. We then measure the corresponding tension.
The second step is the measurement of dispersion time of unprocessed cocoa powder. The mass of cocoa is poured in one movement onto the water surface without sieving. This measurement is performed for different cocoa to water ratios at room temperature (22°C±1°C). The signal is observed to decrease as the powder becomes dispersed and we stop the acquisition when a steady level is reached. The agitation is made by an pale agitator.

An example of acquisition curve is given on figure 2 using 30 grams of cocoa in 2 litres of water (1.03 $\%_v$) at 470 rpm agitation. The initial level corresponds to the water signal. The signal decreases during the dispersion until final level corresponds to the maximum dispersion in laboratory conditions. We calculate the medium for each level to have $\overline{V}_{water}$ and $\overline{V}_{final}$. We calculate $V_{x'}$ with equation 3.

$$V_{x'} = \frac{(V_X - \overline{V}_{water})}{(\overline{V}_{final} - \overline{V}_{water})} \qquad (3)$$

We have drawn on figure 3 $V_{x'}$ versus time. 100 % is the difference $(\overline{V}_{final} - \overline{V}_{water})$ and corresponds to the total dispersion. The dispersion time is considered as the time between 90 and 10 % of $(\overline{V}_{final} - \overline{V}_{water})$.

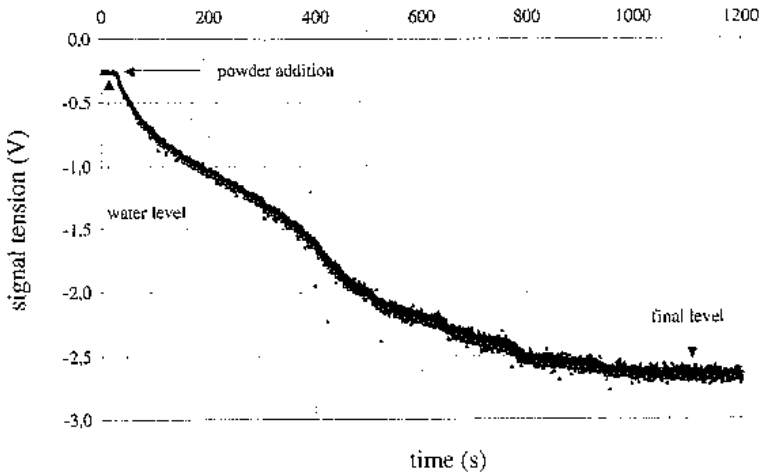


Fig.2. Data acquisition curve for C_v = 1,03 %v at 470 rpm

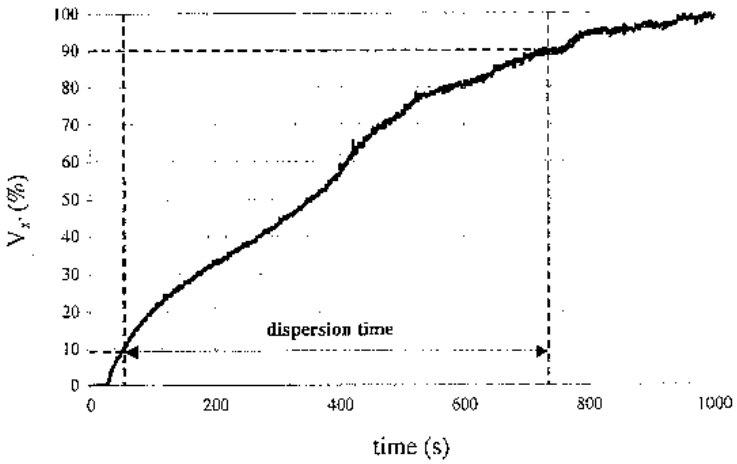


Fig.3. Exploitation curve

II. RESULTS

II.1. Calibration curve

The calibration curve tension (V) versus cocoa concentration (%) obtained by successive additions of cocoa is given in figure 4.

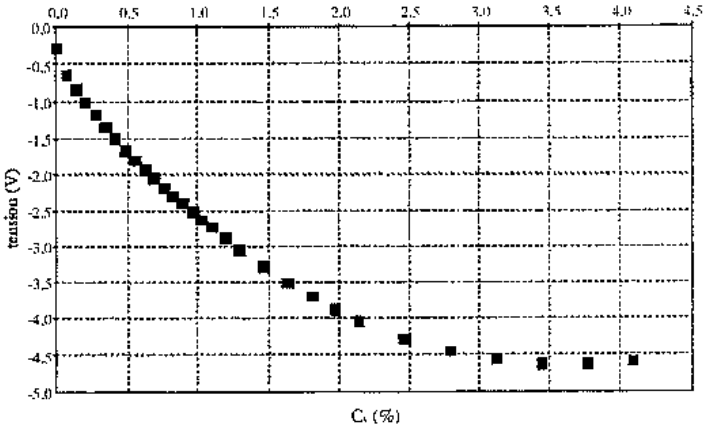


Fig. 4. Calibration curve for cocoa powder in 2litres of water

It can be seen that the signal decreases as the cocoa water ratio increases as light is back-scattered by the cocoa particles. After a critical amount of powder, the signal is saturated and the tension no longer changes with the amount of cocoa. For the rest of our study we have worked with a concentration less than $3\%_v$.

II.2. Unprocessed powder

We have measured the time and the rate of dispersion for different concentrations of cocoa (0.35 to 2.03 %) and for two agitation speeds. The first agitation is medium (470 rpm) and the second is a high agitation (1000 rpm). All the results, the dispersion time and the dispersion rate are shown in table 4. The time dispersion (s) and dispersion rate ($g.s^{-1}$) curves versus cocoa concentrations (%) are shown in figures 5 and 6. The mean dispersion rate (in $g.s^{-1}$) is calculated with equation 4.

$$\bar{r} = \frac{m_{cocoa}}{t_{dispersion}} \qquad (4)$$

Tab. 4. Dispersion time and dispersion rate as function of concentration (470 and 1000 rpm, 2 litres of water)

cocoa powder		dispersion time (s)		mean dispersion rate (10^{-2} $g.s^{-1}$)	
mass (g)	C_v (%)	470 rpm	1000 rpm	470 rpm	1000 rpm
10	0.35	378	48	2.7	20.9
20	0.69	616	62	3.3	32.3
30	1.03	671	80	4.5	37.5
40	1.36	760	154	5.3	26.0
50	1.70	∞	16	$\rightarrow 0$	312.5
60	2.03	∞	12	$\rightarrow 0$	500.0

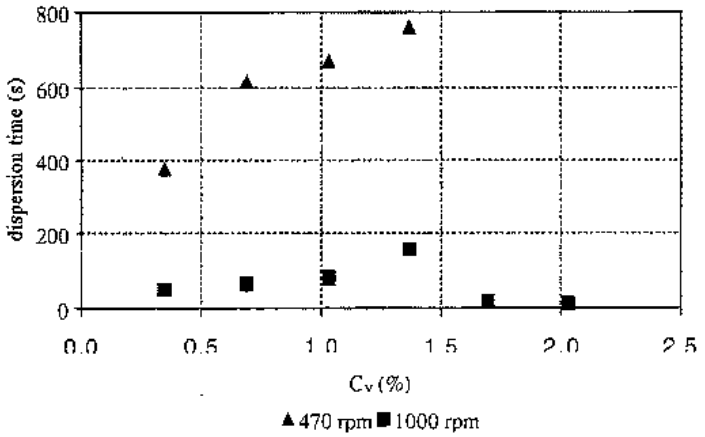


Fig.5. Dispersion time vs. concentration for 470 and 1000 rpm

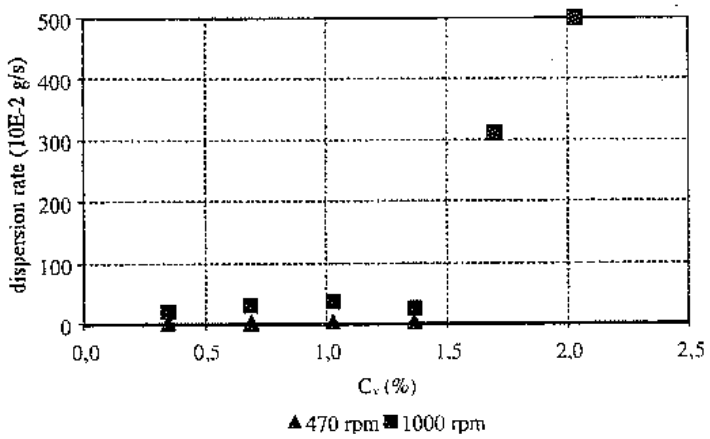


Fig.6. Dispersion rate vs. concentration for 470 and 1000 rpm

At medium agitation (470 rpm) the dispersion times are high. Several minutes are required to disperse only 10 grams of cocoa (0.35 %). From 0.35 to 1.35 %, the dispersion rates are very low, less than 5.3 10^{-2} $g.s^{-1}$. We observe that the dispersion rate is not linear with the cocoa concentration. For the two higher concentrations the powder quantity was not all wetted by water and some stayed on the surface during an "infinite" time (more than 30 minutes). A steady level is not attained, dispersion is not complete, and no dispersion time can be attributed.
With a high-speed agitation (1000 rpm) the dispersion times are much lower than with a medium agitation. A dispersion time can be attributed for all concentrations. For this agitation the dispersion time and rate both increase with all different concentration as we observed at 470 rpm except for the results at 1.36 $\%_v$. At high concentration of cocoa powder, we observe a very short dispersion time as the whole quantity of cocoa powder penetrates directly into the water without staying on the surface.

II.3. Formulated cocoa powder

Each particle size class of cocoa granules (see I.2.) are dispersed in water at 1.03 $\%_v$ (30 grams in 2 litres). The dispersion time and dispersion rate are measured for each granule size class and are given in table 5. It should be noted that all the granules have the same intra-granular porosity.

Tab. 5. Dispersion of granules of cocoa, C_v = 1.03 %, 470 rpm

class	size (µm)	dispersion time (s)	dispersion rate (10^{-2} $g.s^{-1}$)
1	d < 200	88	33.8
2	200 < d < 630	599	10.1
3	630 < d < 1000	266	11.3
4	1000 < d < 2000	464	6.5
5	2000 < d	861	3.5

The dispersion times determined for yhe five particle size classes are shown on figure 7. It can be seen that the dispersion time all the greater the greater the granule. Dispersion time is very short for small granules (about 90 seconds are required to disperse 30 grams of granules less than 200 µm) and quite long for big granules (about 460 seconds are required to disperse 30 grams of granules of size greater than 2000 µm). However there is an improvement with respect to the unprocessed powder for all classes except class 5 (d > 2000 µm). It should be noted that no undispersed granules remain in the

suspension at the end of the experiment. Figure 8 shows that for granules there is a regular change in dispersion rate with the concentration.

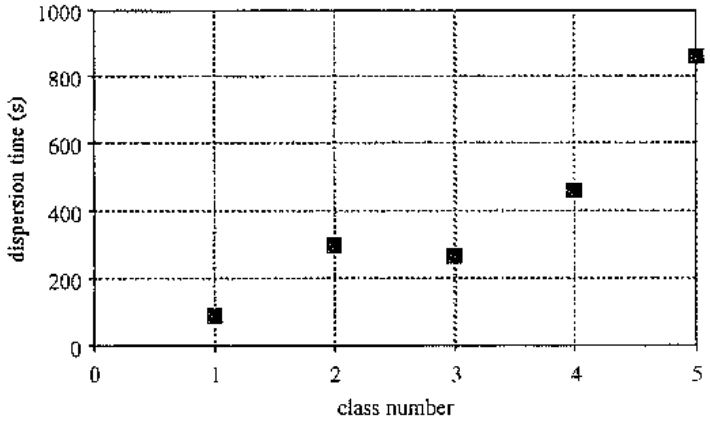


Fig. 7. Dispersion time of cocoa granules, Cv = 1,03 %, 470 rpm

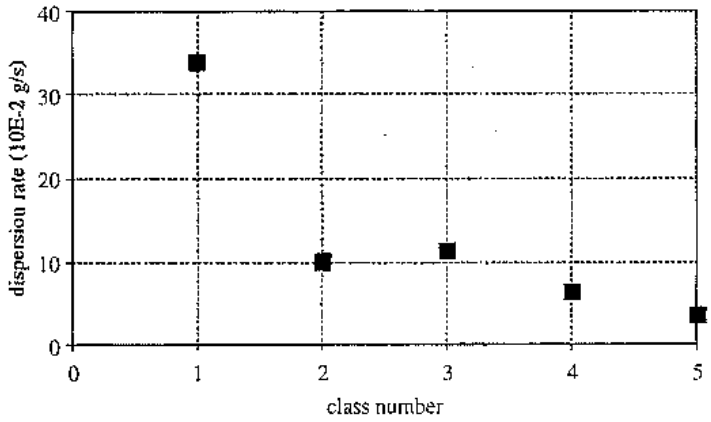


Fig. 8. Dispersion rate of cocoa granules, Cv = 1,03 %, 470 rpm

III. DISCUSSION

III.1. Unprocessed powder

Medium agitation speed (470 rpm)

Figure 4 shows that the dispersion times are very long for cocoa powder (more than 6 minutes for 0.35 % and more than 12 minutes for 1.37 %). At the start the experiment at 1.27 %, the dispersion time can not be measured. In fact the mechanical agitation at 470 rpm is insufficient to disperse cocoa powder. A bed of powder remains on the surface of water, with a viscous layer stopping capillary flow in the inter-granulate porosity. The agitation of the is not sufficient to make the cocoa layer penetrate into the bulk liquid. This bad dispersion behaviour is due to different phenomena. One is the hydrophobic nature of cocoa, the fat-content being 10-12 % in cocoa powder and the surface energy of the powder is lower than the surface tension of water.

Another is the size of the grains which has an influence on the quality of dispersion [3]. The smaller are the particle the more difficult is the dispersion. No sedimentation occurs even though the solids density is greater that the density of because it is the aerated and packed densities (0.34 and 0.67 $g.cm^{-3}$) of the powder bed which are acting. In this these

values densities are lower than the water density. The powder bed does not naturally sediment and the system requires an important mechanical energy.
Another reason of the bad dispersion is the bad flowability and the cohesive forces of powder (I_{Carr} and $I_{Hausner}$). It seems that this cohesion causes solid agglomerates formation in the powder and the shear forces are no longer sufficient to break the cohesive forces between grains. These considerations indicate the interest in another experiment with a high-shear in the suspension that is a higher agitation speed (1000 rpm).

High speed agitation (1000 rpm)
It can seen on figure 4 that at 1000 rpm the dispersion times for all amounts of cocoa are very low and smaller than for 470 rpm. There is no longer a cocoa bed on the surface whatever the concentration and the dispersion time increases with the concentrations as for 470 rpm. The dispersion rate increases (Fig.5) with concentration as the mechanical energy is sufficient to break the cohesive interaction between grains. The point at 1. 36% does not follow this reasoning but for the moment remains without explanation.

II.2. Granulated powder

The wet granulation increases the granules size. Each particle size class has the same porosity of about 20 %. The dispersion time is improved when the size is greater than the particle size of unprocessed cocoa and less than 2000 microns. The gravity effect is applied on a granule, which is bigger than a grain and has an apparent density for a granule is about 1.16 $g.cm^{-3}$. This is more effective in promoting sedimentation in water and the powder wets instantly in contrasts to the behaviour of unprocessed powder. The granules penetrate into the bulk liquid and are broken by the mechanical agitation. The dispersion time is improved because we eliminate one step on the dispersion process.
The granulated cocoa powder has a good flowability and the granules are not cohesive. So the cohesive forces between granules are low and shear forces at the suspension surface are sufficient to separate the granules without requiring a high mechanical energy.
For the largest particle size class, the same phenomenon occurs but the breakage of these large agglomerates is more difficult and takes longer. Water penetrates in the intra-particular pores to the heart of the granule but as the granule radius is greater this step takes longer and harder to do. The capillary flow into the granules is slow and breakage is not complete. The dispersion time is no longer improved by large granule size.
This hypothesis is checked by the final tension level which is higher than the expected value indicating that dispersion is not complete and the finale granule size in the dispersion is higher than the unprocessed grain size.

CONCLUSIONS

A novel optical method has been used to measure the dispersion times is shown to give interpretable results suited for a cocoa powder/water system. The method is reliable for these opaque suspensions and the measurements are accurate.
It is found that dispersion is hard for cocoa powder but can improved bu a high-speed agitation. Dispersion rates of cocoa can be improved by wet granulation to increase the size of particles. Further work is in progress to measure the effects of granule porosity

and pore size on dispersion kinetics and on the dispersion rates for cocoa-sugar mixtures.

Acknowledgement
The authors would like to thank Phoscao (Chateuneuf/Loire, France) for providing the cocoa samples and Miss Laurence Bergougnoux (IUSTI, Marseille, France) for her assistance in performing the optical measurement. We specially thank Laurent Devriendt (EMAC, Albi, France) for the measurements of mechanical properties of cocoa powder.

References
[1] S. Hogekamp, H. Schubert and S. Wolf, Powder Technol., 86 (1996)
[2] J. Due Jansen, The Manufacturing Confectionner, (1973)
[3] J. Due Jansen, Food Technol., 29 (1975)
[4] P. Kyaw Hla, Intern. J. of Food Sc. and Technol., 34 (1999)
[5] R.K. Niven, N. Khalili and D. Brynn Hibbert, Chemical Eng. Sc., 55 (2000)
[6] P. Stevens, L. Gypen and R. Jennen-Bartholomeussen, Powder Technol., 82 (1974)
[7] F. Cordier, Etude de la granulation humide de poudres cosmétiques par assiette granulatrice et mélangeur à haut-cisaillement, phD, Ecole des Mines de Paris (2000)
[8] B. Castel, *in* : Techniques de l'Ingénieur, J3380, p.6
[9] G.M. Crawley, M. Cournil and D. Di Benedetto, Powder Technol., 91 (1997)
[10] I. Bellino, L. Bergougnoux and J. Misguich-Ripault, Powder Technol., in press