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Melt pelletization in a high shear mixer. IX. Effects of binder particle size

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Abstract

Lactose monohydrate was melt pelletized in an 8-l high shear mixer. The meltable binders were polyethylene glycol (PEG) 3000, 6000, and 8000 used as flakes, coarse powder, and fine powder, and PEG 10000 and 20000 used as flakes and coarse powder. The distribution of the binder in different granule size fractions was dependent on the binder particle size. The binder particle size was reflected in the initial granule size at high binder viscosities. The fine powders gave rise to the lowest agglomerate growth rate, whereas a difference in growth rate between the flakes and the coarse powders was seen only at the highest viscosities.

Keywords: Binder particle size; Binder viscosity; Polyethylene glycols; Melt pelletization; High shear mixer; Agglomerate formation mechanisms

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1. Introduction

In wet agglomeration processes, the effect of an atomization of the binder liquid will depend on the forces acting in the granulation equipment. In fluidized bed granulators, the mechanical forces are low, because the particles are suspended in air. Therefore, the atomized droplets will keep their initial size and will bind particles together to nuclei, the size of which will depend on the droplet size (Schæfer and Wørts, 1978b). Consequently, the droplet size of the atomized binder liquid is an essential process variable in fluidized bed granulation (Schæfer and Wørts, 1978a).

When centrifugal and shearing forces are involved in a process, they will reduce the droplet size and will aid in distributing the binder liquid. This is why the droplet size was found to have only a slight effect on the agglomerate size in a rotary fluidized bed granulator (Wan et al., 1995)

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and in high shear mixers (Holm et al., 1983; Lindberg and Jönsson, 1985).

In melt agglomeration processes in high shear mixers, the binder is normally added as a powder or as flakes, and droplets of molten binder are formed when the melting point is reached. The initial size of these droplets will depend on the particle size of the solid binder. The high shearing forces in the mixer might reduce flakes to a powder during dry mixing before melting (McTaggart et al., 1984). After melting, a comminution of the molten droplets is assumed to occur by further massing, and this will reduce the effect of the particle size of the solid binder.

It was found in melt granulation experiments with PEG 6000 used as flakes, coarse powder, and fine powder that the granule size distribution became much more homogeneous when the PEG was used as a fine powder instead of flakes (Ukita and Murakami, 1994). Other authors (Schæfer et al., 1990) found a minor effect of the particle size of PEG 6000. This was probably due to a better distribution of the molten droplets because of a higher impeller speed in the latter experiments. Since the distribution of the molten binder is influenced by the binder viscosity too (Kinget and Kemel, 1985; Schæfer and Mathiesen, 1996b), it is possible that the effect of the binder particle size will depend on the viscosity.

The purpose of the present work was to elucidate the effect of the binder particle size by means of melt pelletization experiments with two or three different particle sizes of different types of PEG.

2. Materials and methods

2.1. Materials

The lactose 450 mesh (α-lactose monohydrate, DMV, The Netherlands) described in a previous paper (Schæfer and Mathiesen, 1996b) was applied in the present experiments.

Polyethylene glycol (PEG) 3000, 6000, 8000, 10000, and 20000 (Hoechst, Germany) were applied as meltable binders. PEG 3000, 6000, and 8000 were used as flakes, powder, and fine powder. PEG 10000 and 20000 were used as flakes and powder. The flakes were applied in previous experiments (Schæfer and Mathiesen, 1996b). The values of the true densities, the water contents, the melting ranges, and the molten binder viscosities of the powders and the fine powders were found to be either identical to or close to the values of the corresponding flakes, which were presented previously (Schæfer and Mathiesen, 1996b).

The particle size distributions of the PEG flakes and powders were estimated by a sieve analysis with a series of 12 ASTM standard sieves in the range of 75–4000 μm. A sample of about 100 g was sieved for 5 min at low vibration level (flakes) or 10 min at high vibration level (powders) by a Fritsch analysette 3 vibrator (Fritsch, Germany). The particle size distributions of the fine powders of PEG were determined by a Malvern 2601Lc laser diffraction particle sizer (Malvern Instruments, UK). The median particle diameters and the spans (the difference between the diameters at the 90 and the 10 percentage points relative to the median diameter) are shown in Table 1.

Approximate estimations of the particle size distributions of the PEG flakes during the dry mixing with the lactose were accomplished by a sieve analysis with a series of 9 ASTM standard sieves in the range of 250–2000 μm. A sample of about 100 g of the mixture of the PEG and the lactose was sieved for 5 min at low vibration level, and the fractions smaller than 800 μm were sieved for an additional 5 min at high

Table 1
Median particle diameters (μm) and spans (in parentheses) of the particle size distributions of the PEGs

PEG	Flakes	Powder	Fine powder
3000	1640 (1.6)	304 (1.0)	38 (3.7)
6000	1550 (1.9)	324 (1.0)	57 (2.7)
8000	1280 (1.5)	258 (1.1)	64 (2.8)
10000	1360 (1.7)	294 (1.0)	—
20000	1780 (1.8)	263 (1.2)	—

Table 2
Approximate values of the median particle diameters and the spans (in parentheses) of the particle size distributions of the PEG flakes after comminution during dry mixing with lactose. Impeller speed: 1300 rev./min. PEG concentration: 23%

PEG	Mixing time (min)	Median size (μm) and span
3000	1.5	868 (1.0)
	3.5	791 (1.1)
	5.5	749 (1.1)
6000	8.0	757 (1.3)
8000	8.0	720 (1.2)
10000	9.5	789 (1.1)
20000	10.0	875 (1.0)

vibration level. The estimation was based on the assumption that all of the lactose and none of the PEG had passed the 250-μm sieve. The results are presented in Table 2.

2.2. Equipment

The 8-l laboratory scale high shear mixer (Pellmix PL 1/8, Niro A/S, Denmark), described in a previous paper (Schæfer et al., 1993), was employed in the experiments.

2.3. Mixing procedure

The load of the mixer was 1 kg of lactose in all the experiments. The PEG concentration is expressed as the percentage (% m/m) of the amount of lactose.

The lactose and the PEG were dry mixed at an impeller speed of 1300 rev./min. The impeller speed was increased to 1500 rev./min 2 min after the melting point of the PEG was observed as an inflection point on the recorded product temperature curve. This inflection point was defined as the start of massing time.

The power consumption of the impeller motor was measured by a power consumption meter, and the energy input was estimated from the power consumption curves as previously described (Schæfer et al., 1993).

The procedures for cooling of the agglomerates and for estimation of the adhesion to the bowl were the same as described in a previous paper (Schæfer, 1996).

2.4. Granule characterization

Size distribution, intragranular porosity, liquid saturation, and water content were estimated and image analysis was carried out as previously described (Schæfer and Mathiesen, 1996b). Photographs were taken by a scanning electron microscope (SEM) (Jeol JSM 5200, Japan).

The content of PEG in a granule size fraction was estimated indirectly on the basis of the true density of the ground size fraction, which was determined by an AccuPyc 1330 gas displacement pycnometer (Micromeritics, USA) using helium purge. The PEG content was calculated from the equation:

$$\frac{(1-x)}{\rho_l} + \frac{x}{\rho_b} = \frac{1}{\rho_a} \qquad (1)$$

where x is the fraction of PEG, and ρ_l, ρ_b, and ρ_a are the true densities of the lactose, the binder and the agglomerates, respectively.

2.5. Experimental design

A series of 96 factorially designed experiments was carried out with a PEG concentration of 23%. The five types of powders and the three types of fine powders included in Table 1 were used, and the massing time was varied at 1, 3, 6, 9, 12, and 15 min. All experiments were carried out in duplicate. The results of the present experiments are compared with results of previous experiments (Schæfer and Mathiesen, 1996b) with the PEG flakes mentioned in Table 1.

The results shown in this paper are mean values of two experiments unless otherwise stated. The data were analysed by analysis of variance.

3. Results and discussion

Dry mixing experiments with mixtures of the PEG flakes and the lactose were carried out in order to evaluate the comminution of the flakes before melting. The product temperature increases during the dry mixing, because of heat from the heating jacket combined with heat of friction caused by the impeller rotation. The dry mixing was stopped about 1 min before the melting point of the PEG was reached. This means that the mixing times in Table 2 had to be different for different PEGs since the duration of the dry mixing phase is dependent on the melting point of the PEG. Two further dry mixing experiments at shorter mixing times were carried out with the PEG 3000.

It appears from Tables 1 and 2 that the median particle size of the flakes is reduced during the dry mixing to approximately one half of the initial size, and that the size distribution is narrowed. The experiments with the PEG 3000 show that most of the comminution occurs early in the dry mixing phase. However, when the melting point is reached, the particle size of the flakes is still markedly larger than that of the powders. Further, a clear difference in particle size is seen between the powders and the fine powders (Table 1).

The effect of the binder particle size on the mean granule size is shown in Figs. 1 and 2. It appears from Fig. 1 that with the PEG 3000 and the PEG 6000, the fine powder results in a significantly smaller ($P < 0.001$) granule size than the powder and the flakes. No difference in effect is seen between powder and flakes. With the PEG 8000 (Fig. 1c), the effect of the binder particle size is unclear. The PEG 6000 results in a significantly smaller ($P < 0.001$) granule size than the PEG 3000 and the PEG 8000.

Fig. 2 shows that the flakes give rise to a larger granule size than the powder when the PEGs of the highest viscosities in the molten state are used. This effect is reflected in a significant interaction ($P < 0.001$) between the type of PEG and the binder particle size. The most marked effect of the particle size is seen with the PEG 20000 (Fig. 2b), which has the highest viscosity. It has to be considered, however, that with the PEG 20000 powder, one of the replicates at 12 min as well as 15 min of massing had to be interrupted owing to the formation of large balls caused by an uncontrollable agglomerate growth. Consequently, all the results with the PEG 20000 powder at 12 min and 15 min are results of single experiments. This indicates that the process is more difficult to control when the PEG 20000 is used as a powder instead of flakes.

A longer massing time results generally in a significantly larger ($P < 0.001$) granule size until 12 min of massing. The massing time was not found to have a significant effect between 12 and 15 min. This is because the agglomerate growth is counteracted by an evaporation of water of crystallization (Schæfer and Mathiesen, 1996a,b). No effect of the binder particle size on the evaporation of water of crystallization was found.

The granule size distributions were generally found to be in good agreement with the log-normal distribution except for a slight over-representation of larger agglomerates. The agreement with the log-normal distribution was not found to be affected by the binder particle size.

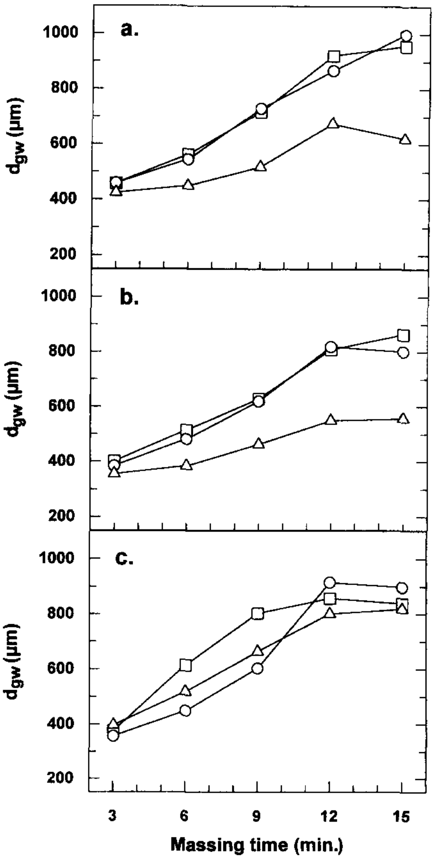

Fig. 1. Effect of binder particle size on the mean granule size during massing. Binder concentration: 23%. (□) flakes; (○) powder; (△) fine powder. Binder: PEG 3000 (a); PEG 6000 (b); PEG 8000 (c).

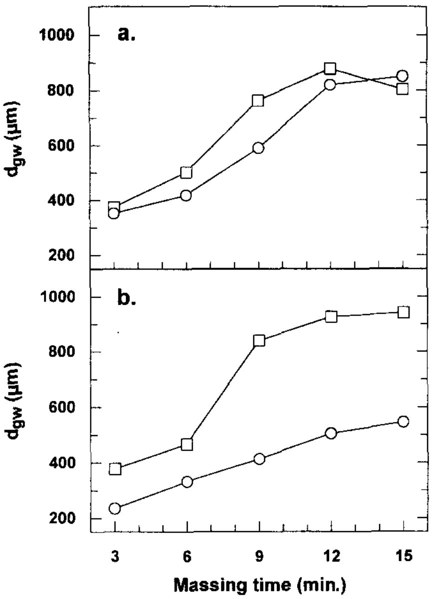

Fig. 2. Effect of binder particle size on the mean granule size during massing. Binder concentration: 23%. (□) flakes; (○) powder. Binder: PEG 10000 (a); PEG 20000 (b).

In the previous experiments with the PEG flakes (Schæfer and Mathiesen, 1996b), the granule growth kinetics was studied according to the work of Knight (1993). In the present experiments, regression analysis carried out on the linear correlation between time and mean granule size and between time and log mean granule size showed that neither a linear model nor an exponential model could be rejected except for PEG 20000 flakes, which gave rise to a rejection of both models. It is therefore not possible to decide if the growth kinetics is of zero order or of first order.

Table 3 shows the results of the regression analysis of the exponential model, which gave rise to a slightly better correlation than the linear model. The value, *a*, is the agglomerate size at time zero, and the agglomerate growth rate is expressed in %/min. The results confirm that a higher binder viscosity results in a smaller initial granule size owing to a more difficult distribution of the binder (Schæfer and Mathiesen, 1996b). It is further seen that the binder particle size has no clear effect on the initial granule size except for PEG 20000, with which the flakes cause a larger initial size than the powder. The fine powders are seen to give rise to a lower agglomerate growth rate than the corresponding flakes and powders in accordance with Fig. 1a and Fig. 1b. With PEG 10000 and 20000, the powder results in a slightly lower agglomerate growth rate than the flakes.

The agglomerate growth was previously found to be reflected in the power consumption of the impeller motor (Schæfer et al., 1992; Schæfer, 1996; Schæfer and Mathiesen, 1996b). This is confirmed in Fig. 3, which shows that the initial power consumption level is lower with the powder than with the flakes in the case of PEG 20000. It is further reflected in the power consumption curve that the powder results in a delayed agglomerate growth and a lower maximum level compared with the flakes. The fall in the power consumption after 10–11 min of massing is caused by an evaporation of water of crystallization (Schæfer and Mathiesen, 1996b).

A higher liquid saturation of the agglomerates will normally result in a larger agglomerate size. Fig. 4 shows that this is not the case in the present experiments. The fine powder gives rise to a significantly lower ($P < 0.01$) intragranular porosity

Table 3
The results of regression analysis of the linear correlation between time (t, min) and log mean granule size (d_{gw}, μm): $\log d_{gw} = \log a + t \cdot \log b$. Time interval: 3–12 min. The percentage increase in d_{gw} per min (%/min) is calculated on the basis of the constant b

PEG	*a* (µm)	%/min	Correlation coefficient
3000 F	357	8.1	0.991
3000 P	364	7.6	0.991
3000 FP	346	5.2	0.940
6000 F	319	8.0	0.993
6000 P	294	8.8	0.976
6000 FP	296	5.1	0.976
8000 F	319	9.5	0.916
8000 P	251	10.8	0.965
8000 FP	319	8.2	0.989
10000 F	284	10.3	0.960
10000 P	251	10.1	0.973
20000 F	268	11.5	0.960
20000 P	187	9.0	0.986

Binder particle size: Flakes (F), powder (P) and fine powder (FP).

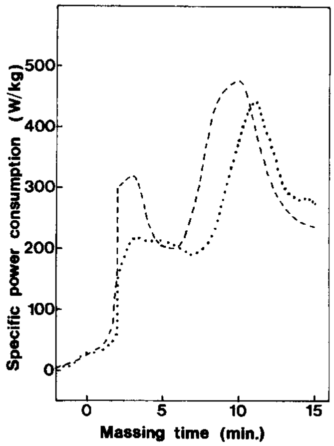

Fig. 3. Effect of binder particle size on the specific power consumption during massing. Binder: PEG 20000. Binder concentration: 23%. (- - -) flakes; (· · ·) powder.

and a significantly higher liquid saturation ($P < 0.001$) than the flakes and the powder. Nevertheless, the smallest granule size was obtained with the fine powder (Fig. 1a). The same effect, but less pronounced was found with the PEG 6000, whereas the binder particle size with the PEG 8000, the PEG 10000, and the PEG 20000 had no clear effect on the porosity and the liquid saturation. The rise in the porosity and the corresponding fall in the liquid saturation after 6 min of massing are due to the evaporation of water of crystallization (Schæfer and Mathiesen, 1996a,b). This fall in the liquid saturation explains the absence of a correlation between the liquid saturation and the agglomerate size.

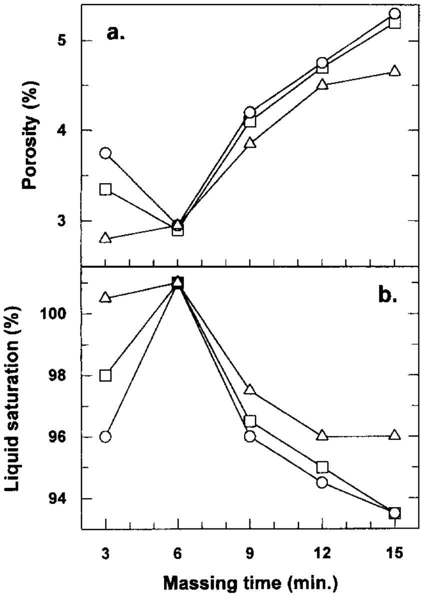

Fig. 4. Effect of binder particle size on the intragranular porosity (a) and the liquid saturation (b) during massing. Binder: PEG 3000. Binder concentration: 23%. (□) flakes; (○) powder; (△) fine powder.

The fine powder gave rise to a significantly larger ($P < 0.001$) geometric standard deviation (s_g) than did the flakes and the powder. However, the difference in s_g at the end of the process was only between 0.04 and 0.09. With the PEG 10000 and the PEG 20000, the powder resulted in s_g-values that were slightly larger ($P < 0.05$) than those obtained with the flakes until 12 min of massing, after which no effect of the binder particle size was found.

No clear effect of the binder particle size on the amount of lumps > 4 mm was found except for the above-mentioned uncontrollable ball growth seen in some of the experiments with the PEG 20000 powder. The final amount of lumps varied between 2% and 7% dependent on the binder viscosity (Schæfer and Mathiesen, 1996b).

With all the types of PEG, the adhesion of material to the bowl was found to be significantly larger ($P < 0.001$) with the powder than with the flakes and the fine powder. There is no obvious explanation for this effect. The adhesion observed with the powders varied between 8% and 12% at 15 min of massing, and this was 2–3% larger than the adhesion found with the flakes and the fine powders.

The shape of the pellets was characterized by the aspect ratio (ratio of length to width) estimated by the image analysis. The present experiments confirmed that the pellets become more spherical, the lower the binder viscosity (Schæfer and Mathiesen, 1996b). The binder particle size was found to have no significant effect on the sphericity.

The distribution of the binder is critical in the early stages of an agglomeration process (Ennis et al., 1991). This is confirmed in Fig. 5 showing the granule size distributions obtained with PEG

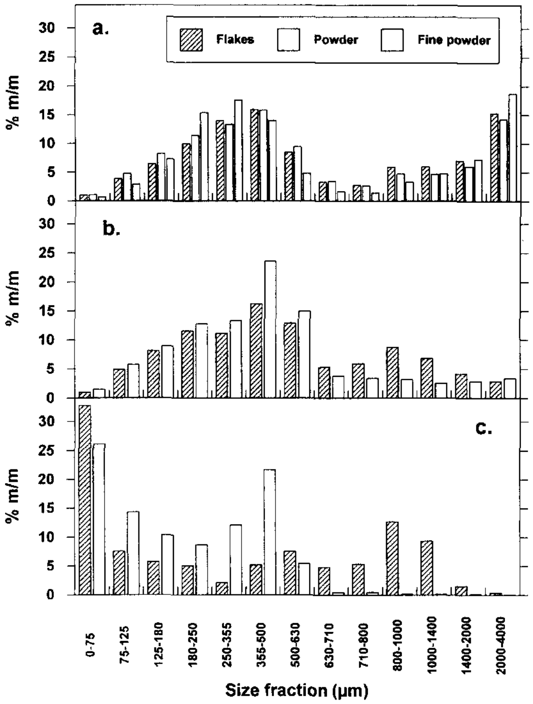

Fig. 5. Effect of binder particle size on the granule size distribution at 1 min of massing. Binder concentration: 23%. Binder: PEG 3000 (a); PEG 10000 (b); PEG 20000 (c).

3000, PEG 10000, and PEG 20000 after 1 min of massing. It was assumed that the larger droplets of molten PEG caused by the flakes would give rise to a formation of granules larger than those formed with the powders and the fine powders. This is seen with the PEG 10000 (Fig. 5b) and particularly with the PEG 20000 (Fig. 5c). Fig. 6 compares the size distribution of the PEG 20000 after 10 min of dry mixing (cf. Table 2), i.e. 1 min before the melting, with the granule size distribution obtained 1 min after the melting. It is clearly seen that the size distribution of the PEG 20000 is mirrored in the granule size distribution.

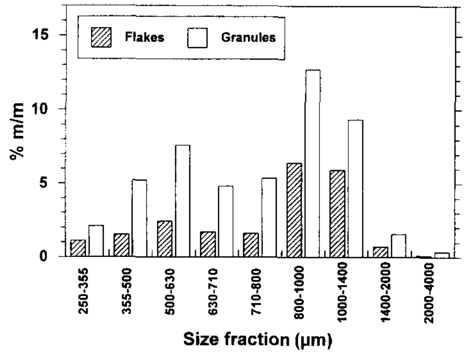

Fig. 6. Comparison between the particle size distribution of PEG 20000 flakes at 1 min before melting and the granule size distribution at 1 min of massing.

The binder particle size is not mirrored in the granule size distribution in the case of the PEG 3000 (Fig. 5a). On the contrary, the fine powder results in a slightly higher content of larger granules than the flakes and the powder. The same effect of the binder particle size was seen with the PEG 6000 and the PEG 8000. When the binder viscosity is low, the high shearing forces from the impeller will cause a rapid distribution of the molten binder, and the effect of the binder particle size on the initial granule size distribution, therefore, is slight.

At higher binder viscosities (PEG 10000 and PEG 20000), the distribution of the binder becomes difficult. This is illustrated by the high content of ungranulated particles seen with the PEG 20000 (Fig. 5c). Granule formation will primarily occur by adsorption of lactose particles to the highly viscous droplets of the molten PEG. The initial granule size distribution, therefore, is markedly affected by the binder particle size. This explains the effects of the binder viscosity and the binder particle size on the initial granule size in Table 3. Fig. 7 shows that the shape of the flakes of the PEG 20000 is reflected in the platelike shape of the agglomerates, whereas the powder gives rise to more rounded agglomerates. This illustrates that the deformability of the agglomerates is low when the binder is highly viscous.

The results mentioned above document that two different mechanisms of agglomerate formation will be active dependent on the binder particle size and the viscosity of the molten binder (Fig. 8). Distribution of the molten binder on the surface of the solid particles will occur when the molten binder droplets are smaller than the solid particles or are of the same order of size. Subsequently, agglomerates will be formed by coalescence between the wetted particles. Immersion of the solid particles in the molten binder will occur when the molten binder droplets are larger than

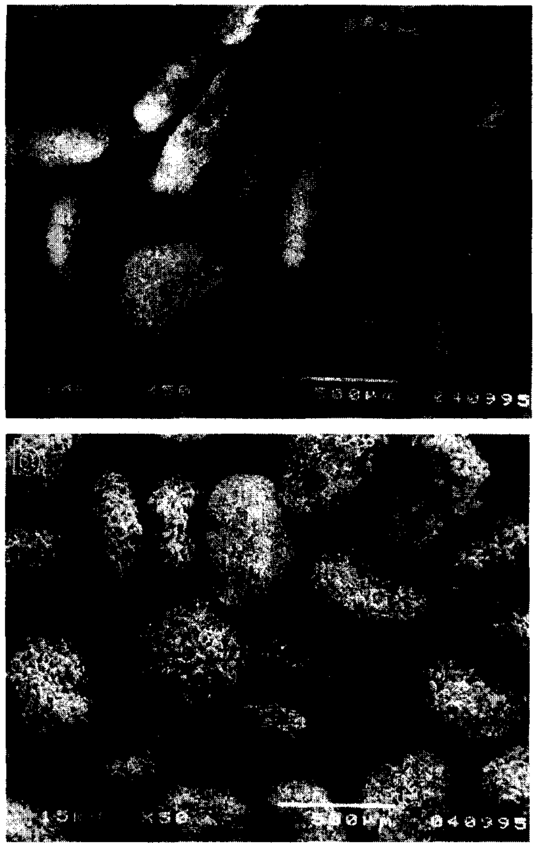

Fig. 7. SEM photographs of agglomerates at 1 min of massing. Size fraction: 355–500 μm. Binder concentration: 23%. Binder: PEG 20000 flakes (a); PEG 20000 powder (b).

the solid particles. This mechanism controls the granule formation in fluidized bed granulation (Schæfer and Wørts, 1978b; Ennis et al., 1991).

By melt agglomeration in high shear mixers, both mechanisms will be active simultaneously, because the binder droplets become comminuted by the high shearing forces. Normally, one of the mechanisms will be dominant. The distribution mechanism is promoted by a small particle size of the solid binder, by a low binder viscosity and by a high impeller speed. The immersion mechanism is promoted on the other hand by using the meltable binder as flakes, by a high binder viscosity and by low shearing forces during the process.

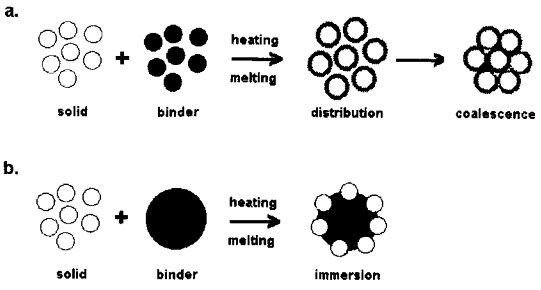

Fig. 8. Granule formation mechanisms in melt agglomeration. (a) Distribution mechanism. (b) Immersion mechanism.

The distribution mechanism gives rise to a more uniform distribution of the binder within the agglomerates than does the immersion mechanism. Therefore, the fine powder results in a significantly lower ($P < 0.001$) initial porosity with the PEG 3000 (Fig. 4a) as well as with the PEG 6000 and the PEG 8000. The fine powder is able to cause a formation of large agglomerates early in the process (cf. Fig. 5a), because the binder liquid is present on the surface of the particles.

In the case of immersion, a further distribution of the binder occurs by a densification of the agglomerates at prolonged massing. This densification is difficult at a high viscosity, and this is why the initial intragranular porosity was found to be high with the PEG 20000 (Schæfer and Mathiesen, 1996b). At the time when binder liquid is squeezed to the surface of the agglomerates owing to the densification, the surface plasticity becomes so high that agglomerate growth by coalescence can occur.

Figs. 9 and 10 show examples of the distribution of the PEG in selected granule size fractions. The theoretical content of PEG in the granules is 18.7%. The binder distribution could not be estimated at massing times longer than 3 min, because the density of the lactose in Eq. (1) becomes changed owing to evaporation of water of crystallization.

With the PEG 3000 (Fig. 9), the distribution of the binder between the size fractions appears to be rather uniform owing to the low binder viscosity. There is a tendency to a higher binder content in the larger size fractions in accordance with previous results from wet granulation experiments (Kokubo et al., 1993). The binder distribution is further seen to be slightly more uniform with the fine powder than with the flakes. This is because

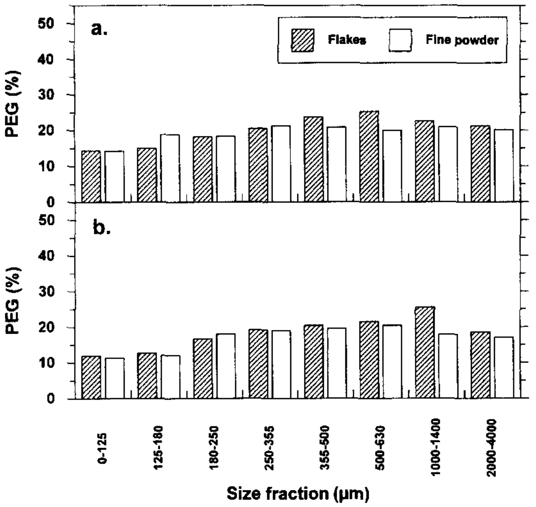

Fig. 9. Content of PEG 3000 in selected granule size fractions. Massing time: 1 min (a); 3 min (b).

the distribution mechanism (Fig. 8a) is slightly more dominant in the case of the fine powder. It is surprising that the binder distribution becomes less uniform after 3 min of massing (Fig. 9b). The flakes are seen to result in a higher binder content in the large agglomerates than the fine powder. A similar binder distribution was observed with the PEG 6000 and the PEG 8000.

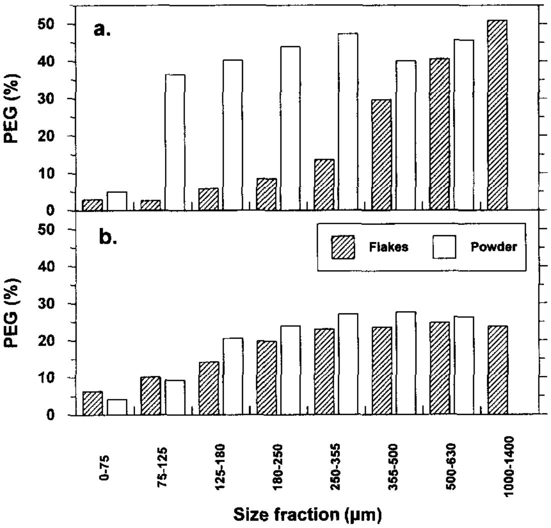

Fig. 10. Content of PEG 20000 in selected granule size fractions. Massing time: 1 min (a); 3 min (b).

After 1 min, the distribution of the PEG 20000 is very inhomogeneous (Fig. 10a), because the immersion mechanism (Fig. 8b) dominates. The binder content is very low in the size fractions that are smaller than the initial particle size of the powder and the flakes respectively, because practically no comminution of the molten binder droplets has occurred. By further massing, more of the ungranulated lactose particles become adsorbed to the molten droplets, and the binder distribution becomes more uniform (Fig. 10b). With the powder, the distribution mechanism is counteracted by the high viscosity and the immersion mechanism by the rather small particle size. This is assumed to be why the powder results in a less uniform binder distribution than the flakes.

4. Conclusions

The effect of the binder particle size on the agglomerate formation interacts with the binder viscosity. At low binder viscosities, the binder particle size has only a slight effect on the agglomerate formation, and the dominant mechanism is the distribution of the molten binder on the surface of the solid particles. When highly viscous binders are used, the binder particle size is reflected in the initial agglomerate size, because the agglomerates are formed by immersion of the solid particles in the molten binder droplet.

The subsequent agglomerate growth by coalescence is dependent on the binder particle size too. At low viscosities, a fine powder results in a lower agglomerate growth rate than coarse powder or flakes. At high viscosities, a coarse powder gives rise to a lower agglomerate growth rate than flakes. These effects cannot be explained by differences in the liquid saturations of the agglomerates, but are assumed to be due to differences in the deformability of the agglomerates caused by an effect of the binder particle size on the intragranular distribution of the binder.

Flakes are preferable for melt pelletization compared to a coarse or fine powder, because flakes give rise to less adhesion than the coarse powder and cause a narrower size distribution and a larger agglomerate size than the fine powder.

These conclusions are based upon experiments with an impeller speed that was very high in order to avoid an uncontrollable agglomerate growth at high viscosities (Schæfer and Mathiesen, 1996b). The effects of binder particle size, therefore, might be larger at a lower impeller speed, because the binder distribution is assumed to be affected by the shearing forces involved in the process.

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