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Operating Conditions for the Formation of Pellets

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The effects of the operating variables of the dish pelletiser on pellet formation and pellets so formed were studied. Pellet growth was found to be so sensitive to the amount of binding liquid incorporated, that a small variation of it could bring about changes in surface plasticity and ability to deform and coalesce through surface moisture bonds. Increasing the amount of binding liquid led to the formation of pellets with flattened facets instead of being well-rounded. Increase in agitation speed led to a reduction in the average pellet diameter. This could be due to increasing inertial forces which far exceeded the cohesive forces present in the nucleus of the pellet, thus resulting in split pellets. A longer residence time in the dish rendered the pellets more round and smooth but of lower strength. Increasing the load of charge in the pelletiser increased average pellet diameter, due to the size of the load pressing on the feed material. No definite relationship between angle of inclination of pelletiser and average pellet diameter was observed.

Keywords——pelletisation; binding liquid; agitation speed; residence time; load size; inclination angle; bulk density; flow property; crushing strength; friability

Introduction

Pellets are becoming as important as tablets or granules in the preparation of pharmaceutical dosage forms. Generally, pellets are spherical bodies formed from a mass of finely divided particles by a continuous rolling or tumbling motion.[1] The more important studies on pelletisation are found in the fertiliser[1-2] and iron ore industries.[3,4] The operating conditions of pelletisers have been shown to affect the formation of pellets.[5-9] The purpose of this study is to investigate the effects of the operating conditions of the dish pelletiser on the formation and properties of pellets. This paper reports the findings of the effects of binding liquid, load of feed material, residence time, agitation speed and angle of inclination on production of pellets and their physical characteristics.

Experimental

Equipment——A schematic representation of the inclined dish pelletiser (Erweka, Germany) is given in Fig. 1. It consists essentially of a shallow cylindrical dish, rotating about an inclined axis. The speed of rotation is variable. A scraper is located at the upper part of the dish to prevent build-up of material and to direct the flow pattern of the tumbling powder.

Materials——Maize starch (B.P. grade) was used to form pellets, with distilled water as the binding liquid. The particle size range of starch as determined microscopically was 2—10 μm and the bulk density was 0.6980 ± 0.054 g/ml.

Formation of Pellets——The charge of materials was placed in the pelletiser dish which was allowed to rotate at an angle. Binding liquid was sprayed at 0.33 ml/s onto the powder to form pellets which were then dried in an oven at 60 °C for about 4 h. The amount of binding liquid used was varied from 35% to 58.5% (w/w) and the feed charge was 200, 300, 400 and 500 g. The rates of rotation of the pelletiser employed at angles of inclination of 45, 50, 55, 60 and 65° were 20.33, 31.66, 42.0 and 48.0 rpm. The time the material was allowed to reside in the dish was for periods of 7, 10, 15, 30 and 60 min.

Pellet Size Analysis——A series of sieves (Endecotts, England) of aperture width 4.0, 2.8, 2.0, 1.4, 1.0, 0.71 and

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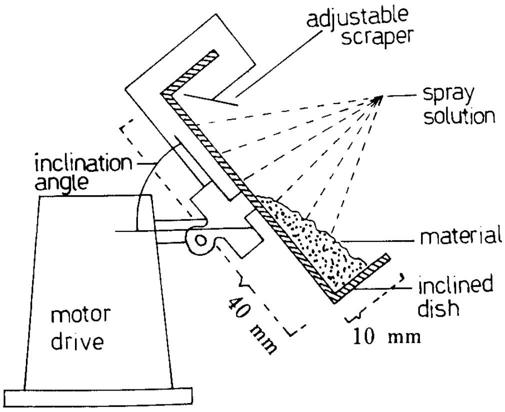

Fig. 1. Schematic Representation of the Dish Pelletiser

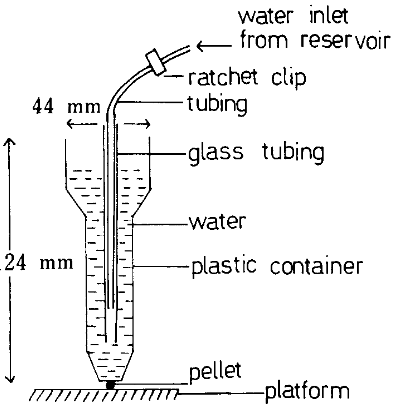

Fig. 2. Schematic Representation of Crushing Test Apparatus

0.375 mm were used for analysis, the end-point of analysis was taken when no more pellets passed through the sieve. A cumulative frequency curve is plotted, from which the median diameter by weight is obtained. The rate of pellet growth is the size change per unit dish revolution and is calculated as mm/number of revolutions. The percentage of binding liquid is expressed as: % wt = (mass of liquid/mass of solid) × 100.

Bulk Density——Twenty grams of the size fraction (0.71—1.0, and 2.0—2.8 mm) of pellets were poured gradually through a funnel into a graduated cylinder, tapped lightly 30 times on a laboratory bench and the volume measured. This procedure was repeated 5 times for each of 5 replicates. The bulk density was calculated as the quotient of the weight and volume of pellets.[10]

Angle of Repose——The angle of repose was determined by the fixed-height method.[11] The height was maintained at 20 mm and the average internal diameter of the funnel stem was 7.5 mm. Five determinations were made for each of 5 replicates.

Crushing Strength——The resistance of pellets to fracture was measured by placing a light plastic container on the pellet and applying a load by allowing a constant rate of flow of water into the container (Fig. 2). The load required to crush the pellet was taken as the crushing strength, in grams. Twenty individual pellets were taken for each of 5 replicates.

Friability——The strength of the pellets was assessed by determining the resistance to abrasion using a Roche Friabilator (Erweka, Germany). Ten grams of the pellets and 25 steel balls of average diameter 4 mm, and average weight 0.26 g were placed in the drum and rotated at 40 rpm for 3 min, the material was sieved and that remaining on the sieve was weighed.[12] The friability index is the loss in weight expressed as a percentage.

Results and Discussion

Effect of Binding Liquid

With a binding liquid content of 35%, there was a tendency for the feed material to adhere to the walls of the dish and form large weak lumps. A large proportion of the material was not wetted uniformly and on drying, these large pellets disintegrated easily. Increasing the binding liquid in 5% increments up to 50%, fine tumbling motion of the pellets was observed. At 55%, the starch pellets tended to stick to the dish base, whilst at 58.5%, large clusters developed and any further increase in binding liquid produced paste formation.

To initiate pelletisation of starch there must be a nucleus whose moisture content is not less than 40%. At the moisture range above 40%, the pellets could be considered to have excess of moisture at their surface to allow a certain degree of surface plasticity. This enabled partial deformation to occur when agglomerates were in collision. In the case of pellets prepared with 35% binding liquid, this lack of excess surface moisture meant that pellets would not be able to coalesce on collision with agglomerates. The number of liquid bridges formed would not be sufficient to withstand the breaking force produced by the tumbling action.[13]

As the amount of binding liquid was increased from 40% to 50%, the average pellet

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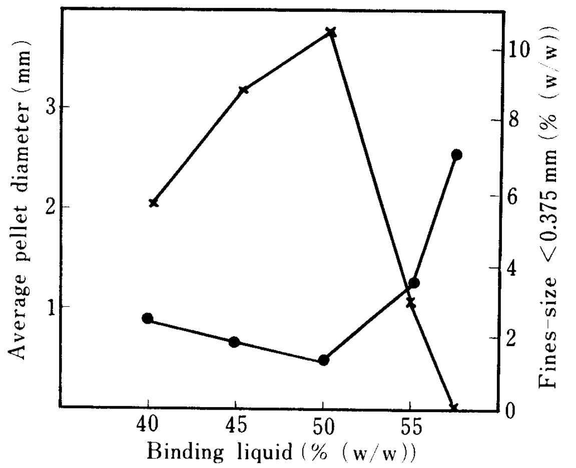

Fig. 3. Average Pellet Diameter (●) and Amount of Fines Produced (×) as a Function of Amount of Binding Liquid

Operating conditions: load, 200 g; speed, 20.33 rpm; ∠, 45°; residence time, 15 min.

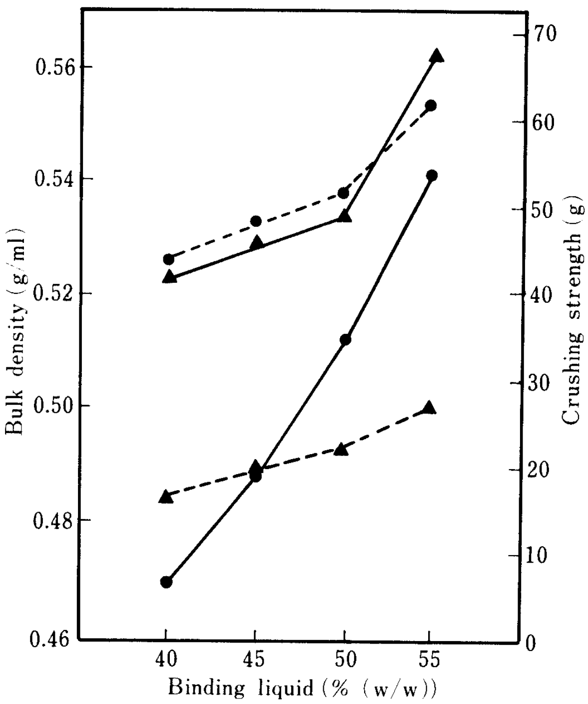

Fig. 4. The Change of Bulk Density (——) and Crushing Strength (----) of Pellets with Different Amounts of Binding Liquid

Operating conditions: load, 200 g; speed, 20.33 rpm; ∠, 45°; residence time, 15 min.
Pellet size: ▲, 0.71—1.0 mm; ●, 2.0—2.8 mm.

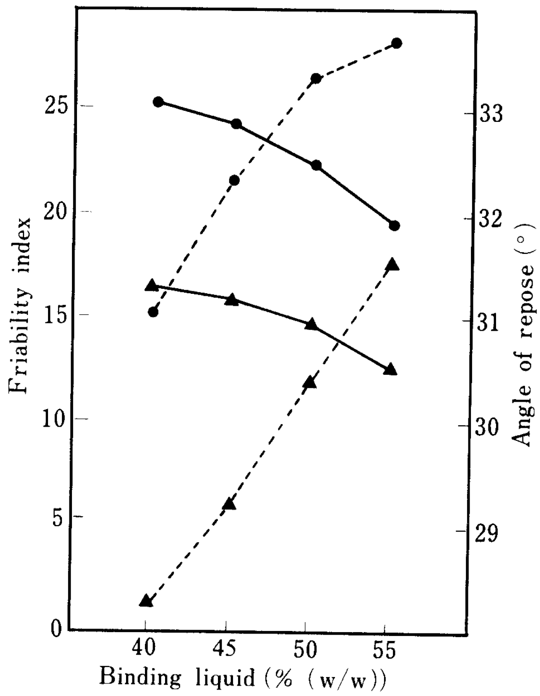

Fig. 5. Relationship of Friability Index (——) and Angle of Repose (----) of Pellets with Amount of Binding Liquid Added

Operating conditions: load, 200 g; speed, 20.33 rpm; ∠, 45°; residence time, 15 min.
Pellet size: ▲, 0.71—1.0 mm; ●, 2.0—2.8 mm.

diameter decreased followed by a sharp increase when the liquid content was increased further to 58.5% (Fig. 3). The amount of fines, *i.e.* pellets with size <0.375 mm increased initially followed by a decrease (Fig. 3). The reduction in size could be attributed to the lower strength of the pendular bonds, a state in which liquid is held at a point contact in the form of a bridge neck. Pellets in this form would not be strong enough to grow in size with increased liquid content and thus on tumbling in the pelletiser would break down, resulting in more fines (Fig. 3). The increase in average pellet diameter at >50% binding liquid could be due to the change from the pendular to the capillary state of wetting. This change from the pendular to the capillary state of wetting is accompanied initially by a gradual rise in bulk density followed by a rapid densification at >50% binding liquid (Fig. 4). There was also a gradual increase in the

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crushing strength[1,14] of pellets at 40% to 50% binding liquid, with a greater increase at >50% binding liquid (Fig. 4). This behaviour can be attributed to the larger number of liquid bridges being formed when there is a change from the pendular to the capillary states of wetting with higher liquid content. This correlates with the lower friability obtained with the pellets as shown in Fig. 5.

With increasing moisture content, the surface of the pellets becomes more plastic and on tumbling they can be easily deformed, giving rise to irregularly shaped pellets, as evident by the increase in the angle of repose (Fig. 5). With moisture content, >58.5%, paste formation was observed, a state in which the pellets are presumed to be saturated with water as has been reported previously.[15]

Effect of Loading

The effect of increasing mass of material on pellet forming showed that the average pellet diameter became larger with bigger loads (Fig. 6). With small loads, 200 and 300 g, it was observed that the pellets formed exhibited a regular and even tumbling motion on the surface of the dish. There was efficient usage of dish surface, more of it was traversed by the pellets and less chance of collision, thus leading to smaller size pellets being produced (Fig. 6). The size distribution of largest pellets obtained were in the range of 8—10 mm in diameter. With large loads, 400 and 500 g, the tumbling motion was replaced by a cascading motion, this resulted in a larger number of collisions, thereby producing larger size pellets and a broader size distribution of pellets when compared with a 200 or 300 g load. The size of the load pressing on the feed material during tumbling is important. If excessive loads are introduced, larger pellets are formed and these pellets exert a pulverising action to the smaller ones. These can result in fragmentation of the smaller pellets into fines and subsequently bring about layering of the fines into larger pellets.

Figures 7 and 8 show the physical properties of pellets formed with different feed loads. It is believed that with increased loads, pellets tend to rotate through a greater angle in the bulk of the material before approaching again into the tumbling region.[16] Since the pelletisation process was carried out for a fixed duration of time, there is a greater hold up at the base of the dish and this restricts particle rearrangement.

It is also of importance to note that with increased loads, there is a larger amout of material pressing down on the remaining material during the tumbling motion which is able to deform/fracture the pellets at the base of the dish. This can explain the reduction in bulk density and crushing strength as well as increase in the angle of repose and in friability. There

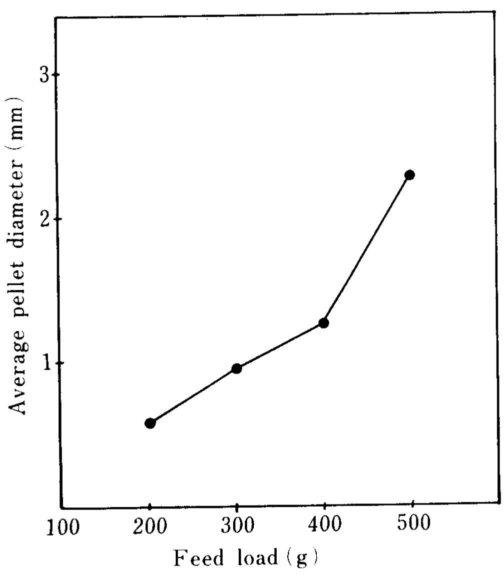

Fig. 6. Average Pellet Diameter as a Function of Feed Load

Operating conditions: speed, 20.33 rpm; ∠, 45°; residence time, 15 min; binding liquid, 50% (w/w).

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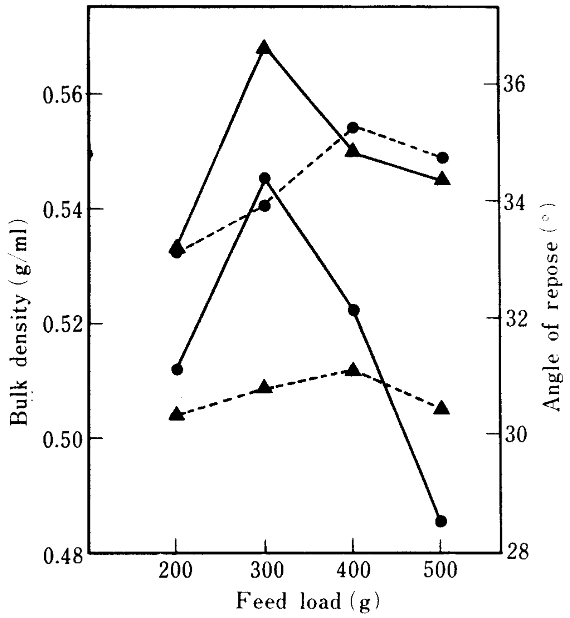

Fig. 7. The Change of Bulk Density (——) and Angle of Repose (- - - -) of Pellets with Different Loads of Feed Material

Operating conditions: speed, 20.33 rpm; ∠, 45°; residence time, 15 min; binding liquid, 50% (w/w). Pellet size: ▲, 0.71—1.0 mm; ●, 2.0—2.8 mm.

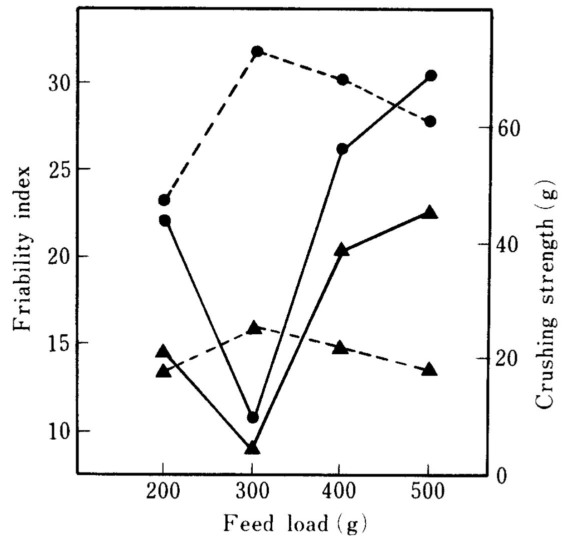

Fig. 8. Relationship of Crushing Strength (- - - -) and Friability Index (——) of Pellets with Feed Load

Operating conditions: speed, 20.33 rpm; ∠, 45°; residence time, 15 min; binding liquid, 50% (w/w). Pellet size: ▲, 0.71—1.0 mm; ●, 2.0—2.8 mm.

appears to be a maximum load which can produce pellets of desired characteristics, 200 or 300 g.

Effect of Residence Time

With a residence time of 7 min, very large pellets were already formed. These were weak and in the form of loose, fluffy agglomerates. This accounted for the big average pellet diameter (Fig. 9). These pellets, on further tumbling, deagglomerated[17] to smaller ones which serve as nuclei for coalescence to occur. As a consequence of deagglomeration and subsequent formation of nuclei, the average pellet diameter determined at a residence time of 10 min were smaller when compared with those formed at the 7 th minute. Coalesced pellets formed at the 10 th minute are generally irregular in shape, rough and resist fragmentation, because of their greater strength as evident from greater crushing strength and corresponding low friability and the larger angle of repose (Figs. 10 and 11).

These pellets also undergo a consolidation period in which excess liquid is exuded at the surface which gives it a certain degree of plasticity and deformation, which in turn favours further coalescence. Hence, as tumbling continued, pellets grew in size as shown by the increase in average pellet diameter from the 10th to 15th minute (Fig. 9). Pellets formed at the 15th, 30th and 60th minute were increasingly smooth and spherical as demonstrated by the fall in the angle of repose at these times (Fig. 11). It was also observed that the average pellet diameter increased but reduced in growth rate (Fig. 9), with increasing residence time. It is thought that layering predominates at residence time >15 min, where the larger pellets (due to their heavier weight) have an abrasive action on the smaller pellets and the fines produced can then layer on to the larger pellets. Hence pellet size increases with time (Fig. 9).

As a result of increasing sphericity of pellets and consolidation during tumbling, the bulk density was greater with a larger residence time (Fig. 11). The forces producing densification are capillary forces and the gentle repacking of particles are due to rolling.[14] Eventually a close-packed system is obtained at residence greater than 30 min as shown in the bulk density

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measurements (Fig. 11). Since the process of pelletisation was carried out under standardised conditions, the continual tumbling of the pellets may drain the liquid from the interstitial spaces[18,19] and the capillary forces present in the nucleus become insufficient to retain the liquid phase and thus bring about a lowering of crushing strength and an increase in friability (Fig. 10) at residence time greater than 15 min.

Effect of Agitation Speed

Agitation speeds of 20.33, 31.66, 42.0 and 48.0 rpm, using angles of inclination of 40, 45, 50, 55, 60 and 65°, were employed. Rotation of the dish pelletiser at 40°, yielded pellets which were powdery and at 65° it was not practical to operate the pelletiser due to spillage of the feed material from the dish.

At low speed of agitation, the feed material experienced a smooth tumbling motion and the resultant pellets were large and resistant to breaking up into smaller particles. But at low speed, the content in the dish did not have sufficient momentum to reach the height of the rim wall and roll. This was overcome by providing friction between the wall of the rim and the feed material by spraying a very small quantity of binding liquid on the rim to provide adhesion. However, at high speeds, the pellets were small. The centrifugal force was so great that the dish content was continuously held against the surface of the dish and were not able to roll. This was particularly evident at 42.0 and 48.0 rpm at angles of inclination of 55 and 60°.

The average diameter of pellets decreased with increase in agitation speed for each of the angles of inclination studied (Fig. 12). This may be explained by the manner by which pellet growth is brought about. Two types of forces are involved, natural and applied. Natural forces arise mainly from the cohesive forces such as interfacial and capillary attraction between particles due to the presence of liquid. The tumbling motion generates forces which cause collisions between individual particles and agglomerates so that the natural forces can bring about pellet growth. To survive and grow, the cohesive forces (natural forces) present in

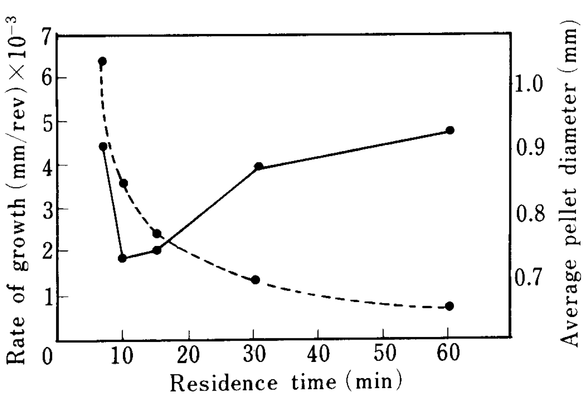

Fig. 9. Average Pellet Diameter (——) and Rate of Pellet Growth (– – – –) as a Function of Residence Time

Operating conditions: load, 200 g; speed, 20.33 rpm; ∠, 45°; binding liquid, 50% (w/w).

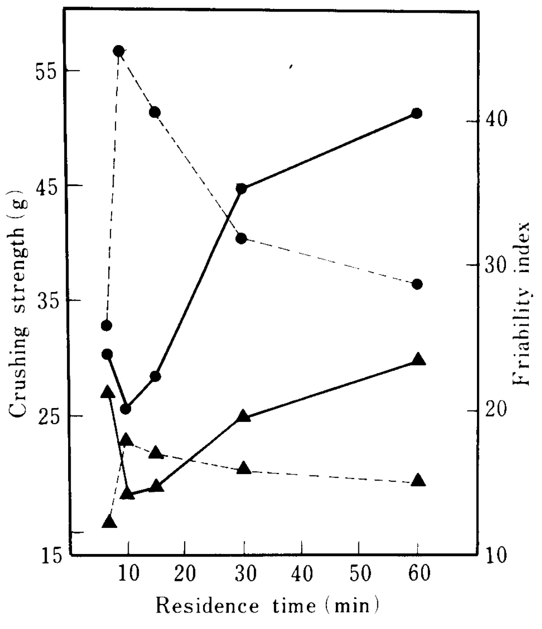

Fig. 10. Relationship of Crushing Strength (– – – –) and Friability Index (——) of Pellets with Residence Time

Operating conditions: load, 200 g; speed, 20.33 rpm; ∠, 45°; binding liquid, 50% (w/w).
Pellet size: ▲, 0.71—1.0 mm; ●, 2.0—2.8 mm.

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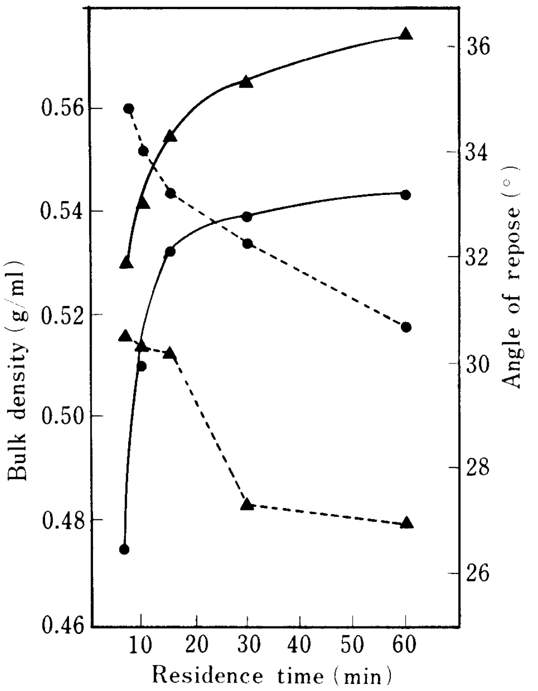

Fig. 11. Relationship of Angle of Repose (- - - -) and Bulk Density (——) of Pellets with Residence Time

Operating conditions: load, 200 g; speed, 20.33 rpm; ∠, 45°; binding liquid 50% (w/w).
Pellet size: ▲, 0.71—1.0 mm; ●, 2.0—2.8 mm.

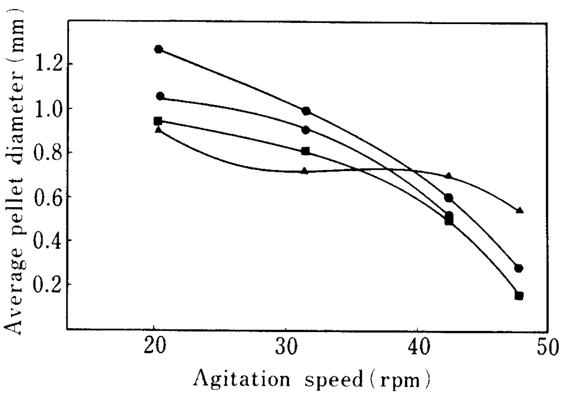

Fig. 12. Average Pellet Diameter as a Function of Agitation Speed

Operating conditions: load, 200 g; residence time, 15 min; binding liquid, 50% (w/w).
Angle of inclination: ●, 45°; ■, 50°; ▲, 55°; ●, 60°.

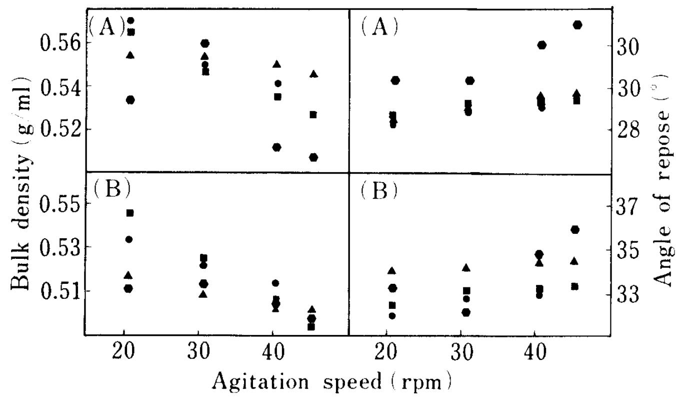

Fig. 13. Relationship of Bulk Density and Angle of Repose of Pellets of Size 0.71—1.0 mm (A) and 2.0—2.8 mm (B) with Agitation Speed

Operating conditions: load, 200 g; residence time, 15 min; binding liquid, 50% (w/w).
Angle of inclination: ●, 45°; ■, 50°; ▲, 55°; ●, 60°.

a nucleus which are responsible for pellet formation must be able to withstand the destructive agitation forces (applied forces). Once the pellet is formed, the final size attained by the agglomerates represent a balance between these two forces. Hence with increasing agitation speed, the destructive forces exceed the cohesive forces, pellet growth is severely restricted and thus the rate of pellet growth is limited and the average pellet diameter decreases.

It is known that the mechanical strength of a pellet will be dependent on the moisture content. With increasing agitation speeds, pellets undergo greater compacting stresses as a result of impacts on the rim of the dish while tumbling, also, there are greater collisions between pellets and this may remove/force water held by capillary forces to extrude. This will set up a suction potential in the pellet. This potential will increase continuously up to a point when air begins to enter the pore spaces, this is known as entry suction.[1]

Thus, with increasing agitation speed, the cohesive forces holding the particles together

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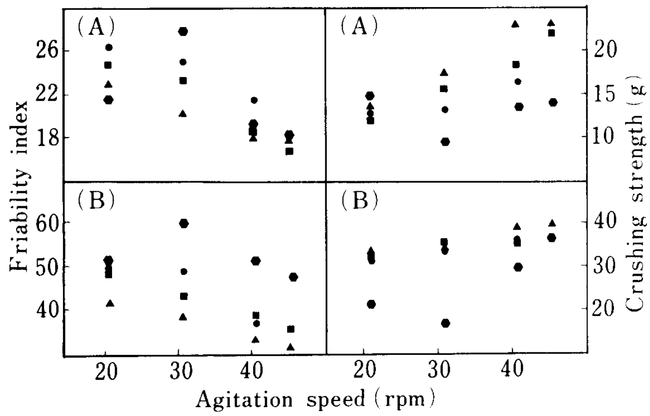

Fig. 14. Relationship of Crushing Strength and Friability Index of Pellets of Size 0.71—1.0 mm (A) and 2.0—2.8 mm (B) with Agitation Speed

Operating conditions: load, 200 g; residence time, 15 min; binding liquid, 50% (w/w).

Angle of inclination: ⬣, 45°; ■, 50°; ▲, 55°; ●, 60°.

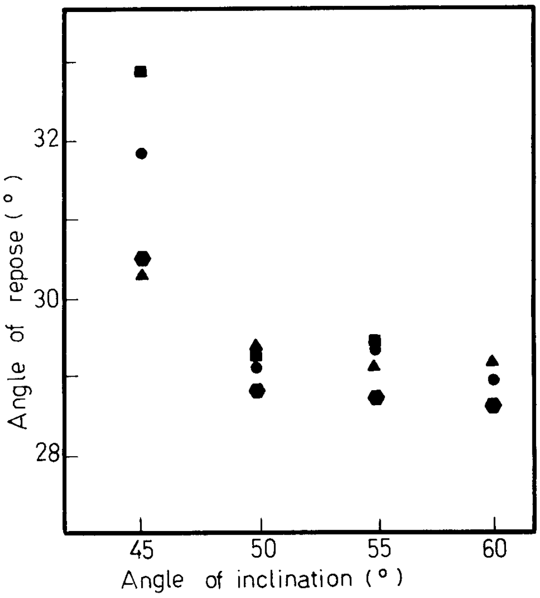

Fig. 15. Relationship between Angle of Repose of Pellets and Angle of Inclination of Pelletiser

Operating conditions: load, 200 g; residence time, 15 min; binding liquid, 50% (w/w).

Agitation speed (rpm): ⬣, 20.33; ▲, 31.66; ●, 42.0; ■, 48.0.

will be less than the inertial forces of the pelletiser, hence, this gives rise to the formation of weak and split pellets. The presence of air in the pellet makes it less dense and particle rearrangement in the pellet is then minimal. Consequently, as the agitation speed becomes faster, the pellets formed are more irregular, less dense (Fig. 13) and they lack sufficient mechanical strength (Fig. 14).

Effect of Angle of Inclination

It was observed that increasing the angle from 45 to 60°, the friction between the powder and the dish became less and the spray of the binding liquid was directed onto the dish surface to provide adhesion. There was no definite relationship between average pellet diameter and angle of inclination (Fig. 19). In addition, the physical properties determined did not bear any definite relationship. However, pellets of size fraction 0.71 to 1.0 mm appeared to be more spherical as the angle is increased (Fig. 15).

Conclusion

The results of this study show that in the pelletisation of starch, pellet growth is brought about by three mechanisms. First is the nucleation stage, where the pellets formed are large, weak and easily deagglomerated. The second mechanism is coalescence, which if it predominates, the pellets formed are rough and irregular. In the third case, where layering is the main mechanism operating, fines and feed particles layer onto the large agglomerates and the resultant pellets are smooth and round. Generally, both coalescence and layering occur simultaneously and it is usual for one of these mechanisms to predominate.

For pelletisation, it is essential to control operating variables as these influence the size and properties of pellets. The amount of binding liquid added should be adequate to ensure formation of pellets of desired properties. Underwetting and overwetting produce rates of growth that are too low or too high respectively.

A long residence time favours production of pellets which are smooth and round but of

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lower strength.

High loading of feed material enlarge pellet size but the pellets are weak.

Slow agitation speeds and greater angles of inclination promote the formation of pellets with uniform shape, particularly those pellets of size 0.71—1.0 mm.

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