The Characterization of Microgranules Produced by Tumbling Fluidized Bed Granulator with Opposed Pulsed Jet Assembly[†]

Hiroyuki Tsujimoto, Toyokazu Yokoyama
Hosokawa Micron Corporation*

Isao Sekiguchi
Dept. of Applied Chemistry, Chuo Univ.**

Abstract

A new granulation method by a tumbling fluidized bed granulator equipped with opposed pulsed air-jet assembly applied to produce the microgranules having the size distribution in less than 100 μ m region was investigated. Powdered remedy material, consisting of diclofenac sodium as the main component was microgranulated by the granulator to investigate the effects of the pulsed air-jet on the microgranule properties, and also discuss its growth model. It was confirmed that the microgranules produced by the new method have not only the smaller granule size but the remarkably larger surface area, better solubility and higher compressibility compared with those of the granules made by conventional tumbling fluidized bed granulation. The microgranule growth mechanism was characterized by the following three steps. The first step was the formation process of smaller granules by the agglomeration of feed particles. The smaller granule size depended on the spray mist size. The next step was the successive coating process of the smaller granules. The final step was the coalescence of the smaller granules into final microgranules.

1. Introduction

A tumbling fluidized bed granulation¹⁾ is a modern technology which combines in both tumbling and agitating as granulation principles with batch fluidized bed granulation. Such granulation using the powdered materials less than 100 μ m in a mean particle size, generally produces the spherical granules with high bulk density having several 100 μ m average granule size.

Recently, the miniaturization of various kinds of granules has been strongly desired for the development in advanced composite materials. For examples, in the case of ceramics materials applied for several electronic components, the fine spherical microgranules less than 100 μ m in average with heavy high bulk density, which can not be attained by spray dry granulation, have been frequently required to realize the high density filling of the materials into tiny cells. The miniaturization of the core-particle²⁾ applied for remedy granules also has been examined.

Although the application of the tumbling fluidized bed granulation to above-mentioned demands has been expected, the miniaturization of granules by using the tumbling fluidized bed granulator has not been yet reported.

In this paper, we examined experimentally the miniaturization limit of granules produced by using a new tumbling fluidized bed granulator equipped with an opposed pulsed jet assembly abbreviated as PJ system hereafter^{3), 4)}. This PJ system is an advanced dispersion technology using particle to particle collision accomplished by focusing three or more compressed air jet nozzles at the center of a fluidized bed. The compressed air was spouted intermittently from the jet nozzles installed at the sidewall of a granulation chamber for restraining the turbulence of fluidization by the inflow of compressed air into the fluidized bed. The bed particles were partially and intermittently forced into intense movement by the strong shearing and mixing effects of the air jet. Thus, the tumbling fluidized bed granulator with PJ system was expected to apply for manufacturing the microgranules by restraining unwanted large granules generation^{3), 4)}.

Therefore, we attempted to produce the microgranules with granule size below 100 μ m to find out the advantages of the PJ system by comparing the new

* 1-9 Shoudai-Tajika, Hirakata Osaka 573-1132, Japan

** 1-13-27 Kasuga, Bunkyo-ku Tokyo 112-8551, Japan

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granulation using the PJ system with the conventional tumbling fluidized bed granulation. Physical properties of the microgranules obtained were also investigated to evaluate the new granulation method.

2. Experimental Apparatus and Procedure

2.1 Feed material and liquid binder

Table 1 shows the physical properties of feed particle. The particles shown in Fig. 1 were diclofenac sodium (hygroscopic and crystalline powder) widely

Table 1 Physical properties of feed particles

Feed particles		Diclofenac Sodium
Volume mean diameter of feed particles measured by Coulter Multisizer	[μm]	4.49
Surface area measured by BET method	[m^2/kg]	6200
True density	[kg/m^3]	1490
Bulk density	[kg/m^3]	240
Angle of repose	[deg.]	44.0
Ununiformity expressed by d_{90}/d_{10}	[—]	3.08

Fig. 1 SEM photograph of feed particles (←→; scale) (Diclofenac sodium)

Fig. 2 Size distributions of feed particles and spray droplets

used as paregoric-antiphlogistic remedy. Fig. 2 shows its size distribution measured by the Coulter Counter and its volume mean diameter was $4.5\mu\text{m}$. The feed particle having both a light bulk density ($\rho_b=240\text{ kg}/\text{m}^3$) and a high angle of repose ($\alpha=44.0\text{ deg.}$) showed a bad fluidity of 37 points expressed in term of the flowability index evaluated by Powder Tester (model PT-N, made by Hosokawa Micron Corp.). In addition, an aqueous solution of 5wt% hydroxypropylcellulose (HPC-SSL) was used as a liquid binder and was sprayed from a binary nozzle.

2.2 Experimental apparatus

2.2.1 Tumbling fluidized bed granulator

Fig. 3 shows a schematic diagram of the experimental apparatus employed. The main body of the tumbling fluidized bed granulator (AGM-2A-PJ, Hosokawa Micron) consisted of two parts made of acrylic fiber; a lower cylindrical granulation chamber with a capacity of $2 \times 10^{-3}\text{ m}^3$ and an upper conical-cylindrical chamber in the free board area. A distributor made of a sintered porous stainless steel plate was provided at the bottom of the granulation chamber. A rotor ① with the air slits in the radius directions and the agitator on the distributor was turning on a central axis for creating tumbling and agitating actions of the bed particles. The heated fluidizing air generated by an electrical heater ⑪ and a suction blower ② was blown from both the air slits and the gap between the periphery of rotor and the inner face of granulation chamber to create the tumbling fluidization. Fine particles accompanied by fluidizing air were entrapped at four bag-filters ⑫ and blown down by a pulsed air jet ⑮.

The flow rate of fluidizing air and its temperature were measured using an orifice meter ③ and two

Fig. 3 Schematic diagram of experimental apparatus

1. Rotor equipped with agitator, 2. Fan, 3. Orifice flow meter, 4. Thermometer, 5. Thermometer, 6. Jet nozzle, 7. Timer, 8. Electromagnetism valve, 9. Flow meter, 10. Spray nozzle, 11. Heater, 12. Filter bag, 13. Liquid binder, 14. Pump, 15. Jet nozzle

thermoresistances respectively. The thermoresistances were located at both a lower chamber under the distributor for measuring an inlet air temperature ④ and an upper part of fluidized bed for the bed particle temperature ⑤. The binary nozzle ⑩ located at 4×10^{-2} m high from the distributor was used for atomizing a liquid binder toward the center of fluidized bed.

2.2.2 PJ system.

As the PJ system, three air jet nozzles ⑥ focusing at the center of fluidized bed were installed in the granulation chamber in 120deg. arrangement as shown in Fig. 4. An electromagnetic valve ⑧ with the timer ⑦ (Fig. 3) controlled both the supplied time t_1 of pulsed air jet from these nozzles and its interval time t_2 . The total flow rate of these air jet was measured by a flow meter ⑨, and it was regulated at a constant flow rate of $6.67 \times 10^{-3} \text{ m}^3 \cdot \text{s}^{-1}$ with $5 \text{ kgf} \cdot \text{m}^{-2}$ in this experiment. When the air jet was injected intermittently, the bed particles were distributed partially as shown in Fig. 5.

2.3 Experimental procedure.

Table 2 shows the granulation conditions. The amount of feed particles was 0.5kg in every batch. Anhydrous-silica of 0.675 wt% against the feed particles was added beforehand to improve the fluidity of raw material. The preliminary mixing of feed particles for 700s was performed before the beginning of granulation for not only mixing the feed particles but also increasing the bed temperature. The drying operation after the granulation was pursued for 1200s. These operations involving the granulation were continuously accomplished in the same fluidized bed

Fig. 4 Location of pulsed air-jet nozzles

Fig. 5 Flow pattern of the bed particles when pulsed jet in use

Table 2 Granulation conditions

Granulation conditions	R-1	R-2	R-3	R-4	R-5	R-6
Pulsed air-jet	not used	used	used	not used	used	used
Supplied time of pulsed air-jet [s]	—	0.5	0.5	—	0.5	0.5
Interval time of pulsed air-jet [s]	—	15	7.5	—	6	3
Supplied number of pulsed air-jet [—]	—	928	1848	—	1095	2180
Feed weight [kg]	0.500	0.500	0.500	0.500	0.500	0.500
Granulation time [s]	13980	13920	13860	6600	6570	6540
Concentration of HPC in liquid binder [%]	5.00	5.00	5.00	5.00	5.00	5.00
Concentration of HPC in product [%]	10.39	10.39	10.39	4.76	4.76	4.76
Liquid binder weight [kg]	1.16	1.16	1.16	0.50	0.50	0.50
Averaged feed rate of liquid binder [kg/s]	8.30×10^{-5}	8.33×10^{-5}	8.37×10^{-5}	7.58×10^{-5}	7.61×10^{-5}	7.65×10^{-5}
Spray air flow rate [m^3/s]	9.5×10^{-4}	9.5×10^{-4}	9.5×10^{-4}	9.5×10^{-4}	9.5×10^{-4}	9.5×10^{-4}
Revolution of rotor [r.p.s.]	9.2	9.2	9.2	9.2	9.2	9.2
Inlet air temperature [K]	333	333	333	333	333	333
Outlet air temperature [K]	303~304	302~304	302~304	301~305	302~305	302~305
Fluidized air flow rate [m^3/s]	5.8×10^{-3}	5.8×10^{-3}	5.8×10^{-3}	5.8×10^{-3}	5.8×10^{-3}	5.8×10^{-3}

granulator for each batch.

The peripheral velocity of the rotor, the inlet fluidized air temperature and its flow rate in above series operations were set at 9.2 rps, 333 K and $5.83 \times 10^{-3} \text{ m}^3 \cdot \text{s}^{-1}$ respectively as fixed conditions. The PJ system was only used in the granulation operation for the batch tests, which examined the influence of the PJ system upon the granulation performance. In addition, the above-mentioned operational factors reached each set value within about 90s after setting in every batch. The bed temperature reached 304-305K at the end of preliminary mixing and it was raised to 314-316K at the end of the drying operation. The ratio of the additional liquid binder ⑬ to the feed particles C_b was set at 10.4wt% in the case for producing the microgranules having a heavy bulk density and granule strength. Moreover, C_b was set at 4.8wt% considering the balance of granules collapsibility and its fluidity for examining the miniaturization limit of granules. In each case, the liquid binder was continuously supplied at a constant rate by the binary nozzle with the roller pump ⑭ (Fig. 3). Besides, the feed rate of liquid binder in each batch was set at a suitable value that the bed temperature was maintained within $302 \pm 1\text{K}$. The amount of compression air for the binary nozzle was set at the maximum value ($9.5 \times 10^{-4} \text{ m}^3 \cdot \text{s}^{-1}$) for the generation of fine spray mists. Consequently, the median mean size of spray mists measured by a particle size analyzer using laser diffraction (2600 Particle Sizer, Malvern) was $7.5\mu\text{m}$ as shown in Fig. 2.

According to the above-mentioned granulation operation, the moisture content of granule product and the recovery ratio of granules in each batch were below 3.5 wt% (wet basis) and over 90 wt% (weight basis) respectively.

The other experimental procedures are described individually in the following part of experimental results.

3. Results and Discussion

3.1 Properties of fine granules at $C_b=10.39\%$

3.1.1 Granule size distributions

The granules size distribution was measured using the sieves (JIS standard sieves) and expressed as a cumulative pass percentage, D (%). Fig. 6 shows the granule size distributions obtained at $C_b=10.39\%$. The mean size of granules on mass basis prepared by the conventional granulation without using the PJ system of Run 1 was $54\mu\text{m}$. Moreover, the granule size at

Fig. 6 Size distributions of microgranules

$D=95\%$ became $80\mu\text{m}$ and all granules were below $100\mu\text{m}$. An uniformity of granule size distribution D_{90}/D_{10} expressed as the granule size ratio of $D=90\%$ to $D=10\%$ in cumulative pass percentage was 2.26. According to a previous report ²⁾, the uniformity of the core granules having a mean granule size of 116-181 μm prepared using ethenzamide particles in a similar tumbling fluidized bed granulator was from 2.1 to 3.0. As seen from these results, the granulation in this experiment was understood to have obtained the microgranules having the narrow size distribution in less than $100\mu\text{m}$ region which had been difficult previously.

On the other hand, the granules size distributions were shifted wholly to the fine size area corresponding to the increase of supplied number of air jet in the cases of Run 2 ($t_1=0.5\text{s}$, $t_2=15\text{s}$, $n=928$) and Run 3 ($t_1=0.5\text{s}$, $t_2=7.5\text{s}$, $n=1848$), using the PJ system based on the operating conditions of Run 1. As the result, these mean granule sizes decreased to $48\mu\text{m}$ and $45\mu\text{m}$ respectively. These uniformity of R-2 and R-3 became 2.73 and 2.68 respectively. The increases of uniformity by using the PJ system were caused by finer granules generation directly leading to the decrease of the value of D_{10} . In this way, at the granulation using the PJ system, the survival of feed particle and the generation of smaller particles by crushing and/or wearing of the granules by the dispersion effect of the air jet were suspected. Then, to examine the presence of these particles, Fig. 7 (a-1) and (b-1) show the electron microscope photographs of products passed through a $25\mu\text{m}$ sieve in Run 1 and Run 3. Especially, neither feed particles nor fine particles by crushing and wearing of the granules were observed in the product in Run 3 produced with higher ability of the PJ system as well as the product obtained in R-1 without the PJ system.

Fig. 7 SEM photographs of microgranules obtained in R-1 and R-3 (↔; scale)
 (a-1): R-1, Products under $25\mu\text{m}$ (b-1): R-3, Products under $25\mu\text{m}$
 (a-2): R-1, Products from 45 to $75\mu\text{m}$ (b-2): R-3, Products from 45 to $75\mu\text{m}$
 (a-3): R-1, Cross section photograph (b-3): R-3, Cross section photograph

3.1.2 Properties of granules

The photos (a-2) and (b-2) in **Fig. 7** show the surface photographs of granules from 45 to $75\mu\text{m}$ in R-1 and R-3 respectively. Besides the photos (a-3) and (b-3) similarly show the sectional photographs of granules obtained in R-1 and R-3 respectively, for examining the effect of difference in the granulation method on the granule properties. The granules (R-1) produced by the conventional granulation without the PJ system were recognized to have thicker binder component spread onto the surface of granules as compared with those (R-3) using the PJ system. Moreover, the sectional photograph of granules in R-1 indicated the dense agglomeration resulting in the formation of granules having high bulk density. On the other hand, in the case of R-3, the spreading of binder component onto the surface of granules was less as compared with R-1. The sectional structure of the granules, showing the loose agglomeration formed by primary granules below about $10\mu\text{m}$, obviously differed from the case of R-1. The granules in R-2 also indicated similar tendencies as in the case of R-3.

3.2 Lower limit of granule size at $C_b=4.76\%$

3.2.1 Granule size distribution

Fig. 8 shows the granule size distributions obtained

at $C_b=4.76\%$ for examining the miniaturization limit of granules. Here, the mean granule size and the uniformity of product (R-4) obtained with the conventional granulation became $45\mu\text{m}$ and 2.56 respectively. The cumulative undersize at $D=95\%$ was $75\mu\text{m}$. Both R-5 ($t_1=0.5\text{s}$, $t_2=6\text{s}$, $n=1095$) and R-6 ($t_1=0.5\text{s}$, $t_2=3\text{s}$, $n=2180$) used additionally the PJ system based on the operating conditions of R-4. Especially, the interval time of pulsed air jet t_2 in R-6 was set at 3s as a maximum condition. Because the formulation of stable tumbling fluidization for pursuing the granulation, became difficult when t_2 was set at shorter than 3s . Namely, the operating condition of R-6 was a limit condition to use stably the PJ system. The mean granule size in both R-5 and R-6 decreased to $36.0\mu\text{m}$ and

Fig. 8 Size distributions of microgranules

Fig. 9 SEM photographs of microgranules obtained in R-4 and R-6 (↔; scale)
(c-1): R-6, Products under $25\mu\text{m}$
(c-2): R-6, Products from 45 to $75\mu\text{m}$
(c-3): R-6, Products from 45 to $75\mu\text{m}$
(d-1): R-4, Products from 45 to $75\mu\text{m}$

$33.5\mu\text{m}$ respectively as compared with R-4, and the cumulative undersize at $D=95\%$ attained to the fine region less than $50\mu\text{m}$. Moreover, the uniformity also decreased to 2.33 and 2.32 respectively as compared with R-4. Using the PJ system contrary to the results of granulation at $C_b=10.39\%$ described in 3.1 led to the decreases of uniformity. The reasons were presumed that the D_{90} value depending on the generation of coarse granules was extremely suppressed by reducing the amount of liquid binder (HPC) by half, and increasing the supplied number of the air jet as well. In addition, the photo (c-1) in **Fig. 9** shows the electron microscope photograph of granules under $25\mu\text{m}$ of R-6 produced using the maximum ability of the PJ system. The existence of the ungranulated particles also was not observed here. In this way, it was confirmed that the tumbling fluidized bed granulation in the region of microgranules under $50\mu\text{m}$ without ungranulated particles was realized by using the PJ system as shown in **Fig. 8** for R-6.

3.2.2 Properties of microgranules

The photo (c-2) in **Fig. 9** shows the granules from 45 to $75\mu\text{m}$ at $C_b=4.76\%$ to examine the influence of the PJ system on the surface properties of granules. This photograph indicated the decrease of the binder component spread onto the surface of granules reducing the C_b by half. Moreover, the formation of porous granules (secondary agglomerated particles) made by soft agglomeration of the fine granules (primary agglomerated particles) from 3 to $10\mu\text{m}$ using the PJ

system was observed from the enlarged photograph shown in photo (c-3) of **Fig. 9**. On the other hand, photo (d-1) of **Fig. 9** shows the enlarged photograph of granules in R-4 produced without the PJ system. In this case the granules agglomerated more compactly than those of photo (c-3) of **Fig. 9** were observed.

3.3 Growth model of microgranules

Fig. 10 shows the growth model of microgranules in accordance with the foregoing observation results. The first step was the formation process of smaller granules (primary agglomerated particles) from 3 to $10\mu\text{m}$ formed by the agglomeration of feed particles. The size of primary agglomerated granules produced in this process nearly corresponds to the spray mist size. It was presumed that the primary agglomerated granule size was prescribed by the spray mist size. The next step was the successive coating process onto the surface of primary agglomerated particles. The final step was the formation process of final microgranules formed by the coalescence of primary

Fig. 10 Schematic diagram of microgranule growth mechanism

agglomerates. Namely, the granulation process of microgranules was categorized as the following three steps, agglomeration/coating/coalescence.

3.4 Other granules properties

The others granules properties such as bulk density, angle of repose, specific surface area, dissolution-disintegration and compression-tension characteristics of microgranule bed were evaluated for examining the effectiveness of the PJ system.

The test samples used for the measurement of both the bulk density and the angle of repose were the final granule product without the sieving. On the other hand, the granules from 25 to 45 μ m were used for the other tests for eliminating the influence of granule size distributions on the measurement results.

3.4.1 Bulk density and angle of repose

Fig. 11 shows the measurement results by using the powder characteristic tester (PT-N). The bulk density and the angle of repose showed the tendency of decrease and increase respectively corresponding to the increase of the supplies number of the air jet. Moreover, the granules produced at $C_b=4.76\%$ showed low bulk density and high angle of repose comparing with that of $C_b=10.39\%$. That is, the bulk density became high in consequence of porous agglomeration in the case of both increasing the supplied number of the air jet and decreasing the added liquid binder as comprehended from the above-mentioned Photographic observation. Furthermore, it was presumed that the increase of the angle of repose was resulted from the increase in the sliding friction between granules due to the increase of the holes of granule surface in the operation using the PJ system.

3.4.2 Specific surface area

Fig. 12 showed the specific surface area obtained by a BET method using liquid nitrogen as an adsorbate at 343K as the deaeration temperature. A rapid surface area analyzer 2200 (Shimadzu Seisakusho) was used as an analyzer. The values of specific surface area were increased corresponding to the increase of the supplied number of the air jet. Especially, the specific surface area obtained at $C_b=4.76\%$ were from 100 to 500 times larger as compared with the result at $C_b=10.39\%$. It was thought that the measurement results at $C_b=10.39\%$ mainly corresponded to the surface area of secondary agglomerated particles and the measurement results at $C_b=4.76\%$ relied on the surface area of primary agglomerated particles.

Fig. 11 Bulk density and angle of repose of microgranules

Fig. 12 Specific surface area of microgranules from 25 to 45 μ m
• Feed particles; $S_w=6200\text{m}^2/\text{kg}$

Fig. 13 Mass percentage of residue of microgranules from 25 to 45 μ m in dispersibility test, R_w
• Feed particles; $R_w=22.1\%$

3.4.3 Dissolution and disintegration

When the produced granules were put into agitated water, a great deal of granules were dissolved easily, but a part of granules formed a comparatively coarse aggregates over 500 μ m. Fig. 13 shows the dissolution and disintegration ability of granules in water by evaluating the above aggregates. The granules of 1.5g were put into 100ml water agitated by a stirrer in a beaker of 200ml. The water temperature was fixed at 308K. The dispersed water containing the sample was

passed through the sieve having 500 μm opening size after 10s from putting the granules into the water. The weight of the aggregates remaining on the sieve was measured after drying and it was expressed as mass percentage of residue of microgranules in **Fig. 13**. The results were improved in proportion to the specific surface area as shown in **Fig. 12**.

3.4.4 Properties of compaction and tension of microgranule bed

The properties of microgranule bed was measured by a full automatic testing system for tensile strength of compressive powder beds (Hosokawa micron, AGR)⁵⁾. The microgranules were filled in a stainless steel split cell having 15mm inner diameter and 40mm length to be split to upper and lower parts. The compression test was performed by compacting the bed with the compression piston at a fixed speed (0.1 mm $\cdot\text{s}^{-1}$) till attaining the set compression stress ($1.1 \times 10^{11} \text{ N}\cdot\text{m}^{-2}$) for measuring both the displacement of the bed caused by the compaction and its compaction stress. After maintaining the compression state at a set stress for 5 minutes, the tensile breakage force, σ_T was measured by raising the upper cell vertically at a fixed speed (0.64 mm $\cdot\text{s}^{-1}$).

The symbol L_s shown in **Fig. 14** represents the logarithmic strain expressed using both the initial bed height L_0 and the bed height at maximum compression state L_1 .

$$L_s = -\log (L_1 / L_0)$$

The microgranule bed at $C_b=4.76\%$ exceeded 0.7 in this L_s value indicating the high compressibility of the bed. On the other hand, the logarithmic strain L_s in the case of $C_b=10.39\%$ showed a low value of about 0.3. It was attributable to the production of the microgranules having heavy bulk density with high granule

Fig. 14 Log-strain and tensile strength of microgranules bed in the compressive-tensile test at $\sigma_p=1.1 \times 10^{11} \text{ N/m}^2$
 • Feed particles; $L_s=0.852$, $\sigma_T=4.38 \times 10^4 \text{ N/m}^2$ at $\sigma_p=2.04 \times 10^6 \text{ N/m}^2$

strength corresponding to the added liquid binder. In each liquid binder condition, the microgranules formed more porous structure due to the increase of the supplied number of the air jet. Consequently, the compressibility of microgranules and the logarithmic strain L_s were increased. Next, the tensile breakage stress of microgranule bed, σ_T at $C_b=4.76\%$ in spite of small amount of added liquid binder indicated a higher value than at $C_b=10.39\%$. The σ_T closely depended on the compaction state of microgranules at the breakage of bed. Namely, the formability by compression of microgranule bed at $C_b=4.76\%$ was improved.

4. Conclusions

The production of microgranules using the new tumbling fluidized bed granulator equipped with the PJ system was examined. Properties of the microgranules obtained were evaluated. The conclusions are as follows;

- 1) The extremely fine granules, having 45-48 μm and 33.5-36 μm in a mean granule size at the condition of $C_b=10.39\%$ and $C_b=4.76\%$ respectively, were produced using the PJ system. By means of the PJ system, the granule size distributions shifted wholly to finer size area compared with the conventional granulation without the PJ system. It was clearly found that the new granulation proposed here was a effective method for the miniaturization of granules.
- 2) The agglomeration of primary agglomerated particles, which leads to the formation of secondary agglomerated particles as a product, was restrained by injecting the air jet. Consequently, the new granulator using the PJ system produced the advanced microgranules having various features such as large specific surface area, good dispersibility and high compressibility.
- 3) By the observation of the electron microscope photographs, the granulation process of microgranules in this study using fine feed particles was characterized by the following three steps; Agglomeration/Coating/Coalescence.

Nomenclature

- C_b : concentration of solid binder (HPC) in feed particles [%w.b.]
 D : cumulative mass percentage of under size [%]
 D_p : particle or microgranule size [μm]
 D_{10} : 10% particle diameter on a mass basis [μm]

D_{90}	: 90% particle diameter on a mass basis	$[\mu\text{m}]$
L_s	: log-strain of compressive microgranule bed	$[-]$
N	: supplied number of pulsed air-jet in granulation	$[-]$
R_w	: mass percentage of residue of microgranules in dispersibility test	$[\%w.b.]$
S_w	: specific surface area	$[\text{m}^2/\text{kg}]$
t_1	: supplied time of pulsed air-jet	$[\text{s}]$
t_2	: interval time of pulsed air-jet	$[\text{s}]$
α	: angle of repose	$[\text{deg}]$
ρ_b	: bulk density	$[\text{kg}/\text{m}^3]$
σ_T	: tensile strength of pre-compressed microgranules bed	$[\text{N}/\text{m}^2]$
σ_P	: pre-compressive stress in tensile strength test	$[\text{N}/\text{m}^2]$

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Author's short biography

Hiroyuki Tsujimoto

Hiroyuki Tsujimoto received the master degree in chemical engineering from the Chuo University in 1988. His M.S. thesis focused on the research of agglomeration of polymeric powder in a hot fluidized bed granulator. Since his graduation he has been working mainly at R&D section of Hosokawa Micron Corporation, where he developed "Tumbling Fluidized Bed Granulator with Opposed Pulsed Jet Assembly" in 1994. His primary research interest is in new granulation technology of "Direct Granulation Method of Liquid Material in a Fluidized Bed Granulator".

Toyokazu Yokoyama

Toyokazu Yokoyama graduated in Chemical Engineering from Kyoto University and received M.S. in 1975. Then he spent six years in Europe to study powder technology at Karlsruhe University and to work as an engineer for Hosokawa Europe Ltd. in Cologne and near London. After another six year work in the engineering division and laboratory of Hosokawa Micron Corp. in Osaka, he was engaged in research at Nagoya University, where he received Ph.D. on the subject of fine wet grinding by ball media mills. Since 1992, he is a manager of Hosokawa Micromeritics Laboratory. His major interests are particle design and processing for advanced functioning by mechanical composing and granulation methods as well as fine grinding and particle characterization.

Isao Sekiguchi

Dr. Isao Sekiguchi graduated from the Department of Industrial Chemistry, Chuo University in 1958, and obtained the Doctor degree of Engineering from Tohoku University in 1975. Since 1977, he is a professor in the Department of Applied Chemistry, Chuo University. His main research in the field of granulation techniques is concerned with fundamentals and extended application which cover three areas: fluidized bed granulation, tumbling granulation and spray drying or prilling. Additional research topic being pursued at present is a new development in the use of microcapsuled asphalt powder as an auxiliary in soil stabilization.