EFFECT OF DRYING ON POWDER COATING EFFICIENCY AND AGGLOMERATION IN VIBRO-FLUIDIZED BED

Hiroyuki Kage, Keiichi Nishihara, Hiromichi Ishimatsu,
Hironao Ogura and Yoshizo Matsuno

Kyushu Institute of Technology, Dept. of Applied Chemistry
1-1, Sensui-cho, Tobata, Kitakyushu 804-8550 JAPAN

Key Words and Phrases: adiabatic process; atomization; binder; frequency; glass bead; silica; suspension; vibration

ABSTRACT

Glass beads of 43 μm were coated in a vibro-fluidized bed by atomizing a fine silica powder together with polyvinyl alcohol aqueous solution. The coating efficiency and weight fraction of the agglomerated particles were measured under various experimental conditions, and their dependencies on the frequency and the direction of vibration were investigated. The coating efficiency and the degree of the agglomeration among core particles correlated well with an index R. The index R was introduced to evaluate quantitatively the drying conditions in a fluidized bed.

Application of vertical vibration on the fluidized bed lowered the coating efficiency somewhat, while it prevented agglomeration. From the experimental results it was confirmed that coating with high quality and high efficiency, where few agglomerates were produced and silica powder was utilized efficiently, was possible in a vibro-fluidized bed with adequate vibration frequency and orientation of the vibration vector.

INTRODUCTION

Since Wurster [6] introduced the possibility of applying a fluidized bed to the granulation process, many investigators have reported about the particle treatment in fluidized bed. In practice, fluidized bed has been widely used in the pharmaceutical, food, and fertilizer industries. Two types of processes for particle growth, namely, agglomeration and coating can be operated in a fluidized bed, depending on the operational condition and treated materials. Main objectives of the coating are stabilization and desolubilization of the core materials owing to its separation from the external surroundings, addition of sustained-release characteristics, improvement of fluidity and adhesion, prevention of powder cracking and improvement of surface color and taste.

In the usual coating process in a fluidized bed, solution of the coating material is atomized on fluidizing core particles, and the coating material deposits on the particle surface to form the coating layer by vaporization of the solvent. Another coating technique is direct coating by powder. When fine powder suspended in a binder solution is atomized, the powder coats the surface of the fluidizing particles directly. In the pharmaceutical industries, the former with organic solution of coating material has been widely used until recently. However, aqueous powder suspension has become popular in many coating processes because of the disadvantages of using organic solvents, such as residual organic component, environmental pollution and toxicity to human health. Therefore, the former method has been gradually replaced by the latter. Furthermore, the smaller fluidizing particle is liable to agglomerate significantly and then it is almost impossible for conventional fluidized bed to coat the particles smaller than 100 μm. Then the development of the coating technique, by which the fine core particles smaller than 100 μm can be coated without the agglomeration, is strongly required.

The authors have coated glass beads larger than 100 μm by silica or nylon powder in a bubbling fluidized bed, and already reported the relationship between operational conditions and coating efficiency [1]. However, it was very difficult to prevent the agglomeration among core particles and to obtain the high quality products in the conventional bubbling fluidized bed, when fine particles smaller than 100 μm were used as the core. Therefore, we introduced vibration onto the fluidized bed [4] or use of a circulating fluidized bed [2, 5] to prevent particle agglomeration and to coat even small core particles.

In this investigation, glass beads of average diameter 43 μm and silica fine powder were chosen as model particles because of their spherical shapes. The glass beads were coated with silica in a vibro-fluidized bed. The effects of the direction and frequency of vibration on coating efficiency and agglomeration among core particles were investigated.

EXPERIMENTAL

A flowchart of the experimental apparatus is shown in Figure 1. The inner diameters of tapered fluidized bed were 100 and 210 mm at the distributor and at the freeboard, respectively. The column height was 1500 mm above the distributor plate. Air was supplied to the bed by a compressor after its temperature and humidity were controlled by a preheater and an air dryer. The suspension of coating material was atomized downward with a two-phase nozzle located in the freeboard. Its height was 370 mm above the distributor. It was confirmed that almost all suspension drops sprayed at the nozzle height arrived at the bed surface without direct entrainment by the fluidizing air. Glass beads of 43 μm average diameter (330-425 mesh, density: 2520 kg/m^3) and fine silica powder with narrow size distribution (1 μm, density: 2200 kg/m^3) were used as a core particle and the coating material, respectively. Polyvinyl alcohol (average molecular weight was 22000) was used as the binder. A couple of vibromotors were fitted on the steel plate or the box, attached directly to the bed column. The fluidized bed and the plate or the box were vibrated as a single. Vibration in vertical (θ=0°), oblique (θ=33°) and horizontal (θ=90°) direction were applied to the fluidized bed coater. Amplitude was kept constant for each vibration direction. Amplitude and frequency were measured by a digital vibration meter (Showa Sokki Corp., Digivibro Model-1332) and digital hand tachometer (Ono Sokki Co., Ltd., HT-441), respectively. In this paper, the term 'amplitude' is double the peak to centerline displacement.

After the fluidized bed was preheated by fluidizing gas up to a pre-selected temperature, silica powder was supplied from the nozzle together with the aqueous binder solution. The viscosity of suspension was 0.97 x 10^{-3} kg/m s. Small amounts of glass beads with silica were sampled from the top of the bed at intervals of 10 min through the coating experiment. The binder deposited on the sampled glass beads was dissolved in water and silica powder was separated from the core particle. The dry weight of silica was measured. As introduced later in Figure 3, its change with passage of time was well related by a straight line passing through the origin of the graph for all experimental runs. Both core particles and sprayed powder did not deposit on the bed wall.

The coating efficiency, E, was defined by the weight ratio of the powder used for the formation of coating layer to the powder supplied to the fluidized bed. The particles in the fluidized bed were sieved by a 330 mesh screen at the end of every coating experiment. The particles passed through the screen were regarded as single-core particles and their weight fraction, Y, was measured to evaluate the degree of agglomeration. The coating efficiency, E, and the weight fraction of single-core particle, Y, were defined as equation (1) and equation (2), respectively.

$$E = \frac{\text{(rate of powder deposition on core particles)}}{\text{(feed rate of powder supplied from spray nozzle)}} \tag{1}$$

$$Y = \frac{\text{(weight of particles recognized as single particles)}}{\text{(weight of all particles in the bed)}} \tag{2}$$

The experimental conditions tested are summarized in Table 1.

TABLE 1 Experimental Conditions

Weight of core particles	0.5 kg
Feed rate of suspension	$1.5 \sim 5.0 \times 10^{-8}$ m^3/s
Weight ratio of fine particle to binder solution	0.117 kg/kg-binder solution
Mass fraction of binder in binder solution	0.005 kg/kg-binder solution
Volumetric flow rate of fluidizing gas	$1.33 \sim 2.17 \times 10^{-3}$ m^3/s
Bed temperature	85 °C
Feed temperature of suspension	30 °C
Frequency of vibration	14.8 ~ 29.8 Hz
Amplitude of vibration	1.29 (Vertical), 2.53 (Oblique) 1.00 mm (Horizontal)
Vibrational acceleration	4.31 ~ 44.7 m/s^2

INDEX R FOR EVALUATION OF THERMAL OPERATING FACTORS

From coating results obtained in a fluidized bed without vibration under various drying conditions, we have already concluded that fluidizing gas is humidified adiabatically in the fluidized bed, and that the coating efficiency is determined by the moisture content in the coating layer formed on the core surface. The moisture content in the coating layer is directly affected by the thermal operating factors, such as temperature and humidity of inlet gas and feed rate of atomized suspension. Therefore, the role of these factors in the determination of the coating efficiency can be explained synthetically by the moisture content in the coating layer [1].

On the other hand, the humidity of the fluidizing gas and the moisture content of the coating layer surface equilibrate, if the atomized water has completely vaporized before the gas leaves the fluidized bed. This is true for the usual fluidized bed coater, because the fluidizing gas has sufficient drying ability. Consequently, the coating efficiency should depend on the humidity of the outlet gas because of its close relation to the moisture content in the coating layer. Then an index R was proposed to evaluate the influence of thermal operating factors upon the coating efficiency and defined by equation (3).

$$R = Ho/Hs = (Wi + Wp)/(Wi + Wt) \tag{3}$$

The drying condition in the bed directly reflects on the index R, and its higher value corresponds to milder drying in wetter bed.

We have already confirmed that the fluidizing gas is humidified adiabatically in the fluidized bed. Therefore, feed gas located at the point A in Figure 2 is heated up to the point B by preheater, and is subsequently cooled and humidified to the point C along the adiabatic cooling line by the atomization in the bed. The index R is easily calculated with the data of experimental condition. It was already reported that the coating efficiency was correlated well with the index R for the fluidized bed coating with and without vertical vibration [1, 3, 4].

The index R is also used in this paper to correlate the coating efficiency and agglomeration in the fluidized bed with vertical, oblique and horizontal vibration.

RESULTS AND DISCUSSION

Deposition of Powder on Core Particles

Figure 3 shows that silica powder deposited on the core particles increases by the passage of time. Horizontal vibration was added to the fluidized bed in Figure 3. The weight of the powder deposited on core particles increased and was almost in proportion to the coating time for all experimental runs. Successful growth of coating layer on the core particle in vibro-fluidized bed is confirmed from these results. In the figure, the effect of the suspension feed rate supplied from the nozzle on the coating is also indicated. The weight of the deposited powder increased with the suspension feed rate. However, it was not in proportion to the suspension feed rate and then the coating efficiency, E, increased with Fs.

Effect of Vertical Vibration Frequency on Coating Efficiency and Agglomeration

Figure 4 shows the relationship between the coating efficiency and the index R. The coating efficiency was correlated well with the index R. Large value of R corresponds to high humidity and low temperature of the inlet gas and high feed rate of the atomized water. As R increased, the coating efficiency became larger and the atomized silica was used more effectively for the formation of the coating layer regardless of the vibration frequency. From the comparison of E at the same value of R, it is found that higher frequency makes the efficiency lower and that the addition of vibration is not recommended for the excellent result in efficiency. The heavier attrition of the coating layer owing to frequent collisions among particles and between particle and bed wall might cause the coating efficiency to decrease, as the vibration frequency increases. Coating under the operating conditions where the index R is large is recommended for the achievement of high coating efficiency.

The relationship between the weight fraction of single-core coating products, by which the degree of agglomeration is quantified, and the index R is shown in Figure 5. As R increased and subsequently the humidity in the bed rose, Y kept constant at first and suddenly decreased at a certain value of R. The progress of agglomeration produces many multi-core coating products and particles in fluidized bed usually de-fluidize when Y becomes less than 0.2 or 0.3.

However, the agglomeration among core particles was prevented and Y kept large value in the wider region of R, as the vibration with higher frequency was applied.

As mentioned above, the application of vertical vibration is not recommended to obtain excellent coating efficiency, while it is sufficiently useful for the prevention of agglomeration among core particles. Then, for the comparison between the favorable effect of vibration on agglomeration and its unfavorable effect on efficiency, the relationship of $R_{E=80}$ and $R_{Y=80}$ is shown in Figure 6. Here, $R_{E=80}$ and $R_{Y=80}$ are the values of R where the coating efficiency, E, and the weight fraction of single-core particle, Y, equal to 0.8, as illustrated in Figures 4 and 5. These values were adopted as critical values of R, where the excellent coating efficiency was achieved and core particles do not agglomerate significantly. Therefore, in the region of vibration frequency where $R_{E=80}$ is less than $R_{Y=80}$, the coating products without agglomeration ought to be obtained efficiently. In Figure 6 it is found that the excellent coating without agglomeration is impossible, when the fluidized bed is not vibrated. However, it becomes possible by the addition of vertical vibration larger than 5 Hz. The same conclusion is also drawn by the comparison of Figures 4 and 5. When the vibration with high frequency is applied, the high coating efficiency is obtained and simultaneously the agglomeration is kept to be low ($0.52 < R < 0.58$ when f=24.8 Hz, $0.58 < R < 0.65$ when f=29.8 Hz). It was confirmed that excellent coating products were obtained effectively when the fluidized bed coater was vibrated vertically and the operational condition that prepared the adequate value of R was chosen.

The effects of amplitude of vertical vibration on the coating efficiency and agglomeration have already reported in the previous paper [4].

Effect of Oblique Vibration Frequency on Coating Efficiency and Agglomeration

The effect of the oblique vibration frequency on the coating efficiency and agglomeration are shown in Figures 7 and 8, respectively. Application of the oblique vibration with lower frequency did not affect both the coating efficiency and the agglomeration. However, the coating efficiency decreased and the agglomeration was prevented somewhat by the application of the highest frequency vibration. The comparison between $R_{E=80}$ and $R_{Y=80}$ for oblique vibration is shown in Figure 9. $R_{E=80}$ is slight larger than $R_{Y=80}$ except at f = 15 Hz. Then it was confirmed that the excellent coating with high efficiency and without agglomeration was almost impossible by the application of oblique vibration.

Effect of Horizontal Vibration Frequency on Efficiency and Agglomeration

Figures 10 and 11 show the effect of horizontal vibration on coating efficiency and agglomeration among core particles. When the vibration in horizontal direction was applied to the coater, the coating efficiency increased at lower frequency, however, decreased at higher frequency. Particle movement by fluidization might be weakened by the addition of vibration of lower frequency, because the direction of vibration and the particle movement by fluidizing gas were at right angles to each other. While, particle movement might not be principally governed by the fluidizing gas but by the strong horizontal vibration when the higher frequency vibration was applied. From Figure 11, it is clear that agglomeration progresses well by horizontal vibration of lower frequency, however, is reversely improved to go back to the former position by the highest frequency. When the core particles were coated effectively, agglomeration could not be prevented, even if the coater was vibrated in the horizontal direction.

The relationship between $R_{E=80}$ and $R_{Y=80}$ is also indicated in Figure 12. $R_{E=80}$ is larger than $R_{Y=80}$ everywhere in this figure. By the addition of horizontal vibration, high quality products were not obtained under any operational condition adopted in this investigation.

From the results obtained, it is concluded that the coating efficiency and the agglomeration among core particles significantly depend on the orientation of the vibration vector as well as its frequency.

CONCLUSIONS

Glass beads of 43 μm were coated by silica powder in a fluidized bed to which the vertical, oblique or horizontal vibration was applied. The coating efficiency and weight fraction of the agglomerated particles were measured and their dependencies on the direction and the frequency of vibration were investigated. The coating efficiency and the agglomeration were correlated well with the index R, on which the drying condition in the fluidized bed directly reflected.

It was confirmed that the excellent coating with high quality and high efficiency, where few agglomerates were produced and silica powder was utilized efficiently, realized in the fluidized bed coater by the application of vertical vibration and the choice of adequate operational condition which prepared the adequate value of R. Application of the oblique vibration with lower frequency did not affect both the coating efficiency and the agglomeration, however, the coating efficiency decreased and the agglomeration was prevented by the highest frequency. By the application of the horizontal vibration, the coating efficiency increased at lower frequency, however, reversely decreased at higher frequency. Furthermore, the agglomeration progressed well with lower frequency, but it shifted back to the former position by the highest frequency. In the fluidized bed coater with oblique or horizontal vibration, it was difficult to obtain effective coating and high quality products simultaneously because of the inverse relation between coating efficiency and quality.

NOTATION

a	amplitude of vibration, mm
E	coating efficiency, -
f	frequency of vibration, Hz
Hi	humidity of inlet gas, kg-H_2O/kg-dry air
Ho	humidity of outlet gas, kg-H_2O/kg-dry air
Hs	humidity of saturated gas, kg-H_2O/kg-dry air
Q	gas flow rate, m^3/s
R	index defined by Eq.(3), -
$R_{E=80}$	R at $E = 0.8$, -

$R_{Y=80}$	R at $Y = 0.8$, -
T	temperature, °C
t	time, min
Vc	weight of powder deposited on core particles, kg/kg-core particle
Wi	feed rate of water accompanied by inlet gas flow, kg/min
Wp	feed rate of water by spray, kg/min
Wt	additional maximum vapor content of inlet gas, kg/min
Y	weight fraction of single-core particle, -
θ	direction of vibration (angle from the vertical) , °

ACKNOWLEDGMENTS

We would like to express our gratitude to Catalysts and Chemicals Industry Co., Ltd., Japan, for the supply of silica powder.

REFERENCES

1. Kage, H., Takahashi, T., Yoshida, T., Ogura, H. and Matsuno, Y., 1996, Coating Efficiency of Seed Particles in a Fluidized Bed by Atomization of a Powder Suspension, Powder Technology, 86(3) pp. 243-250.

2. Kage, H., Katayama, K., Ishimatsu, H., Ogura, H. and Matsuno, Y., 1997, Application of Circulating Fluidized Bed to Powder Coating of Particles, pp. 382-387, in M. Kwauk and J. Li (ed) Circulating Fluidized Bed Technology V, Science Press, Beijing, China.

3. Kage, H., Takahashi, T., Yoshida, T., Ogura, H. and Matsuno, Y., 1998, The Coating Surface and Agglomeration of Seed Particles in a Fluidized Bed Coater, Advanced Powder Technology, 9(3) pp. 245-259.

4. Kage, H., Oba, M., Ishimatsu, H., Ogura, H. and Matsuno, Y., 1999, The Effects of Frequency and Amplitude on the Powder Coating of Fluidizing Particles in a Vibro-Fluidized Bed, Advanced Powder Technology, 10(1) pp. 77-87.

5. Kage, H., Dohzaki, M., Ogura, H. and Matsuno, Y., 1999, Powder Coating Efficiency of Small Particles and Their Agglomeration in Circulating Fluidized Bed, Korean J. Chem. Eng., 16(5) pp. 630-634.

6. Wurster, D. E., 1959, Air-Suspension Technique of Coating Drug Particles, J. Am. Pharm. Assoc., 48(8) pp. 451-454.

FIGURE CAPTIONS

FIGURE 1 Schematic diagram of experimental apparatus
1. Compressor, 2. Filter, 3. Rotameter, 4. Dryer, 5. Valve, 6. Pressure gauge, 7. Heater, 8. Vibromotor, 9. Distributor, 10. Vibration isolator, 11. Two-phase nozzle, 12. Tank, 13. Inverter, 14. Box

FIGURE 2 Adiabatic humidification of fluidizing gas

FIGURE 3 Growth of coating layer on core particles

FIGURE 4 Effect of vertical vibration on coating efficiency

FIGURE 5 Effect of vertical vibration on agglomeration

FIGURE 6 Relationship between $R_{E=80}$ and $R_{Y=80}$ for vertical vibration

FIGURE 7 Effect of oblique vibration on coating efficiency

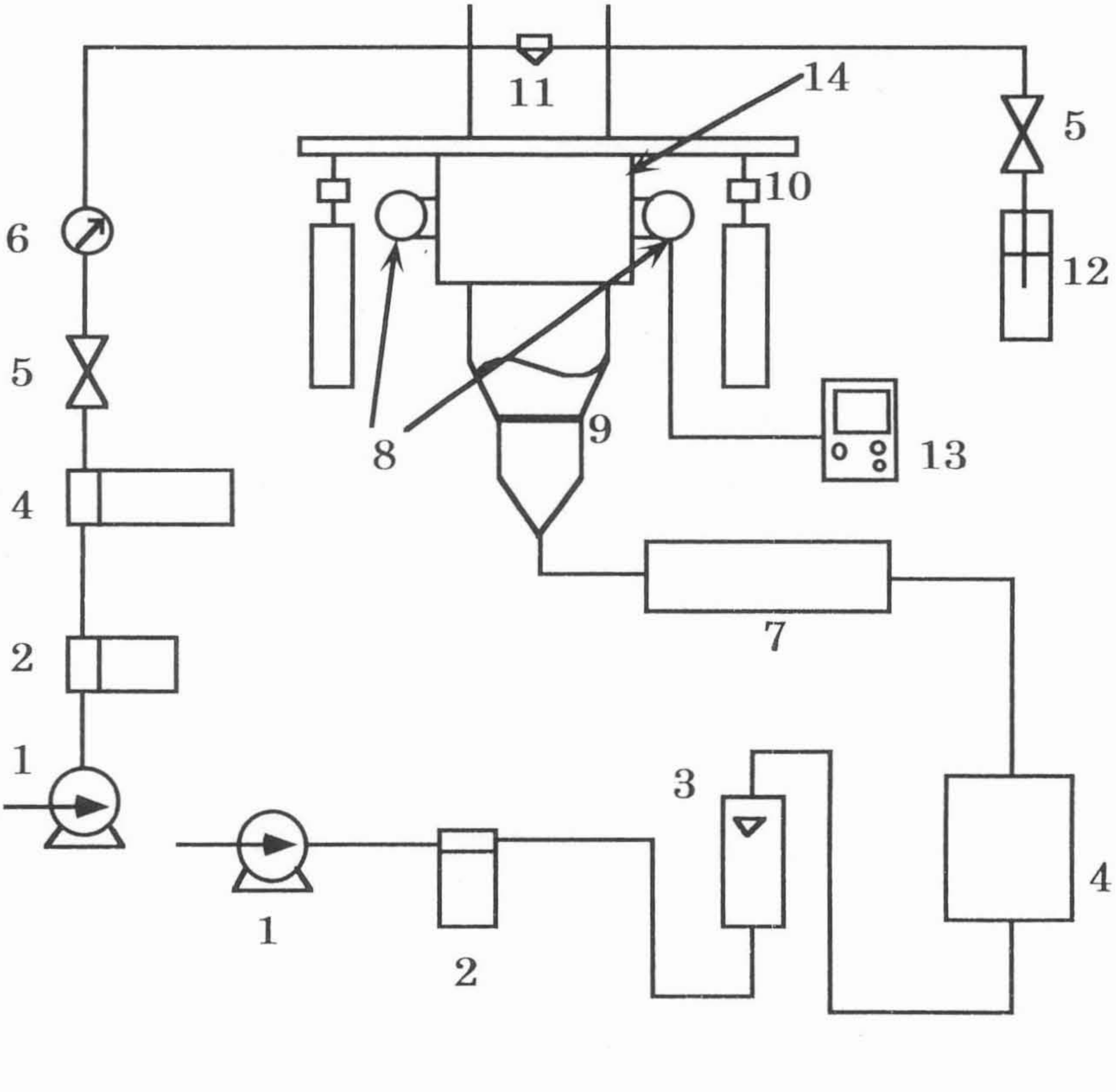
FIGURE 8 Effect of oblique vibration on agglomeration

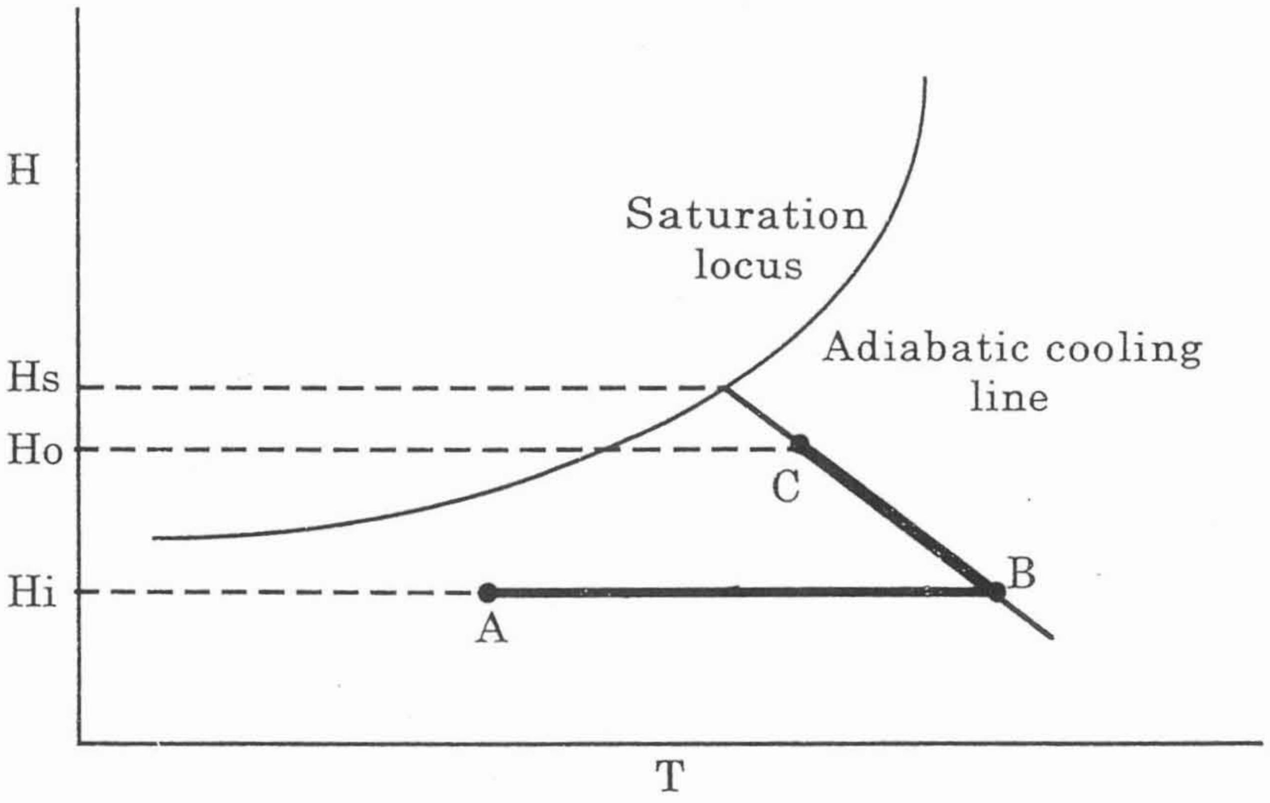
FIGURE 9 Relationship between $R_{E=80}$ and $R_{Y=80}$ for oblique vibration

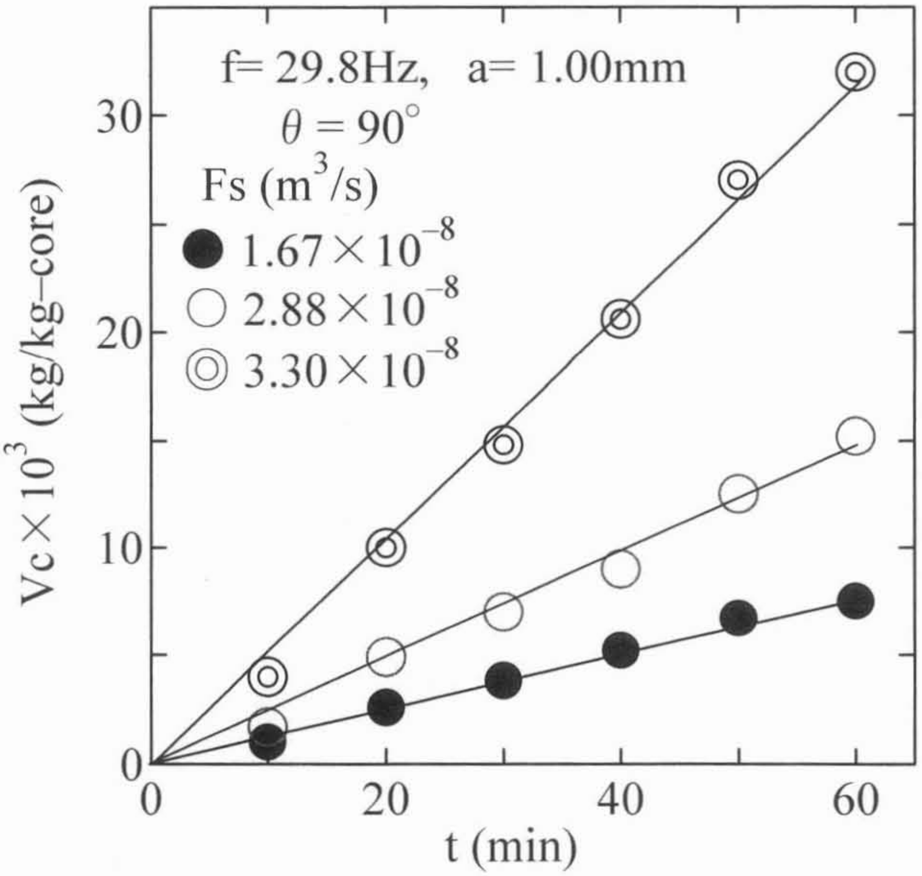
FIGURE 10 Effect of horizontal vibration on coating efficiency

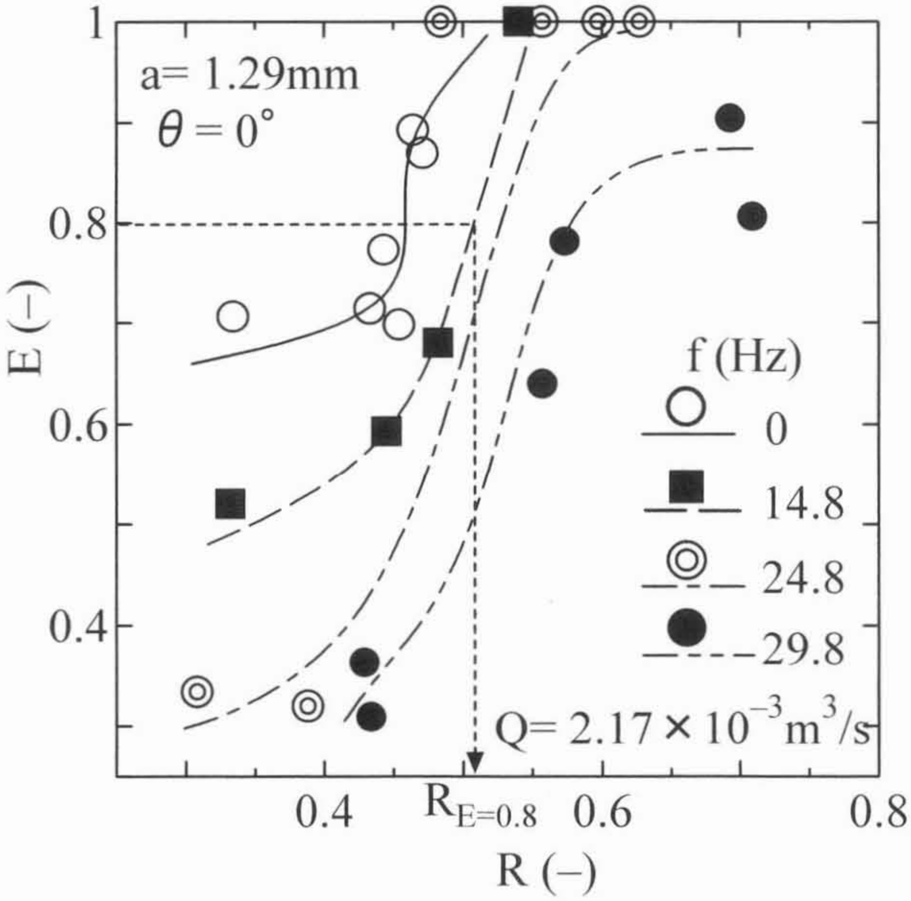
FIGURE 11 Effect of horizontal vibration on agglomeration

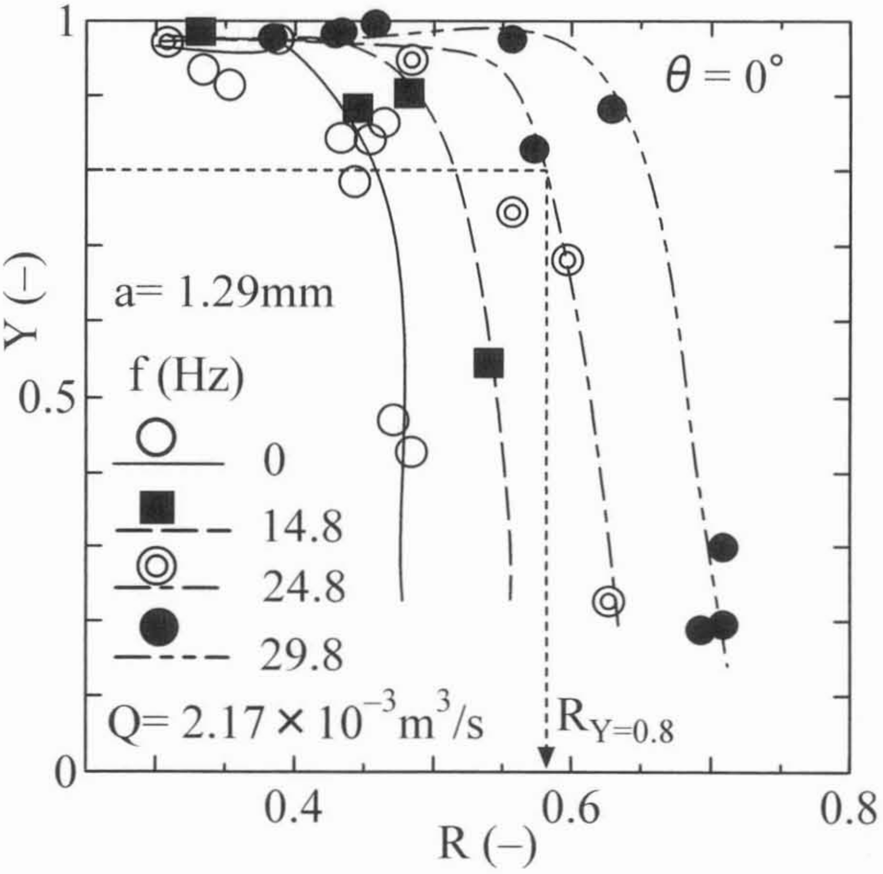
FIGURE 12 Relationship between $R_{E=80}$ and $R_{Y=80}$ for horizontal vibration

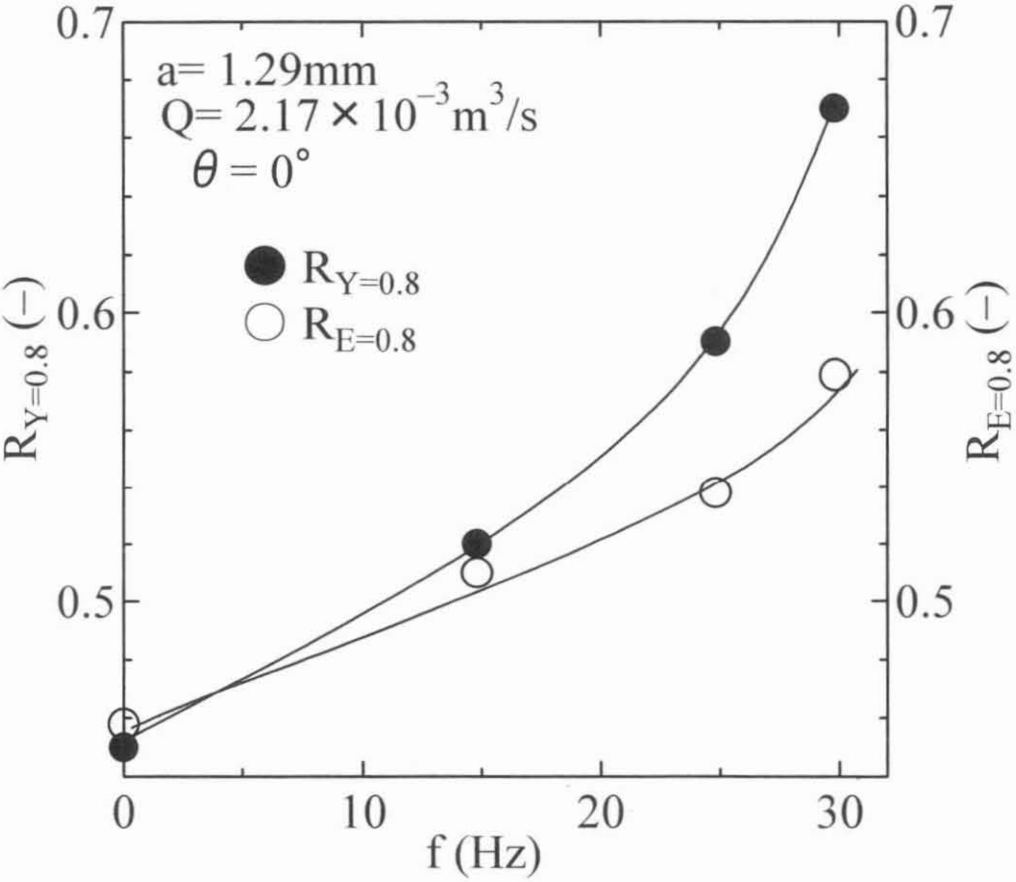

14
11
5
10
6
12
5
9
8
13
4
2
7
1
3
4
1
2

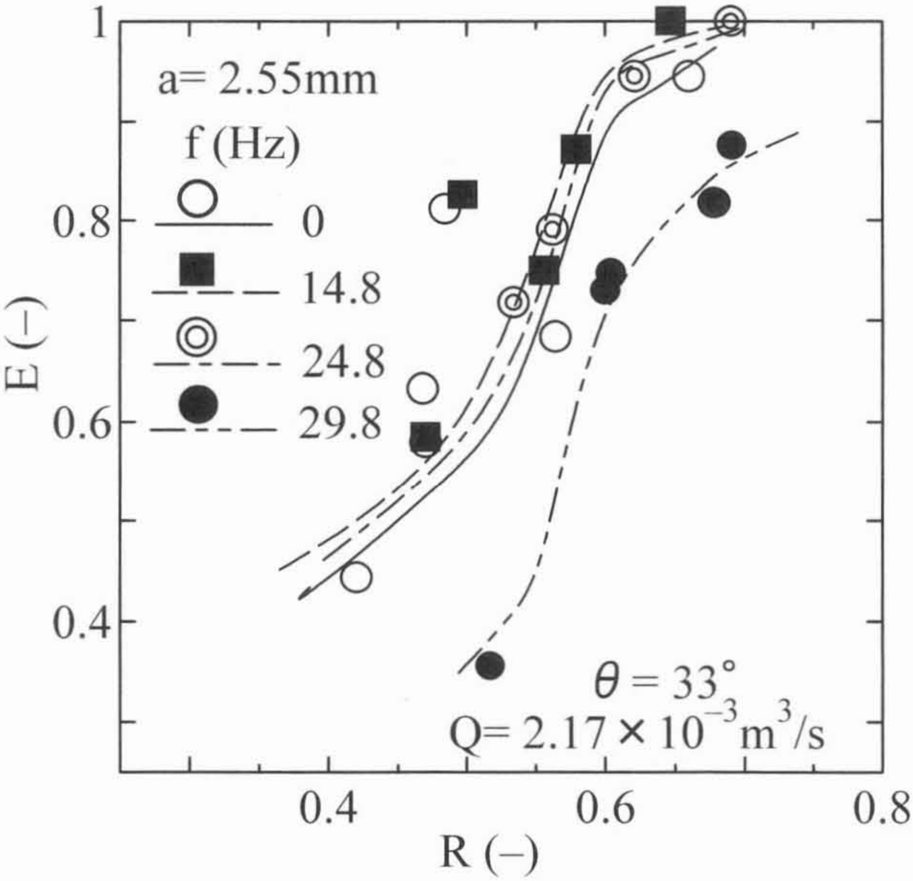

H
Saturation
locus
Adiabatic cooling
line
Hs
Ho
C
B
Hi
A
T

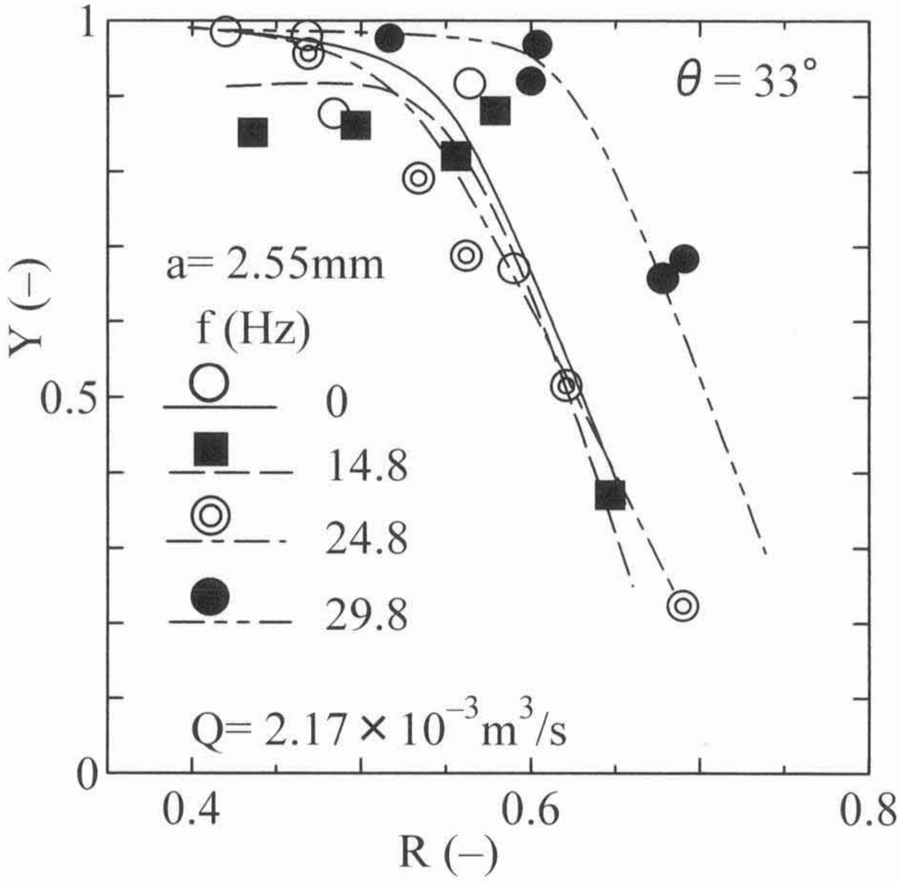

f= 29.8Hz, a= 1.00mm
θ = 90°
Fs (m³/s)
● 1.67×10⁻⁸
○ 2.88×10⁻⁸
◎ 3.30×10⁻⁸
Vc×10³ (kg/kg-core)
0
10
20
30
0
20
40
60
t (min)

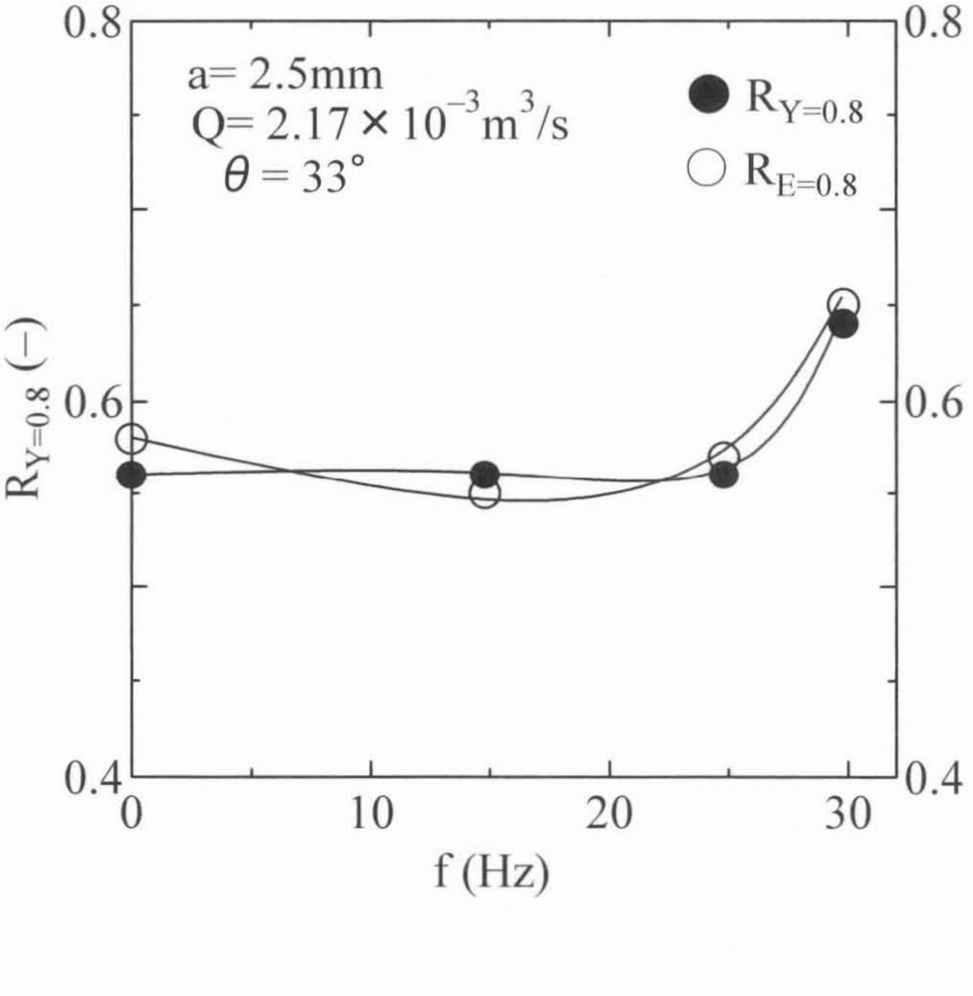

a= 1.29mm
θ = 0°
1
0.8
0.6
0.4
E (–)
0.4
$R_{E=0.8}$
0.6
0.8
R (–)
f (Hz)
0
14.8
24.8
29.8
Q= $2.17 \times 10^{-3} m^3/s$


1
0.5
0
Y (–)
0.4
0.6
0.8
R (–)
θ = 0°
a= 1.29mm
f (Hz)
0
14.8
24.8
29.8
Q= 2.17 × 10⁻³m³/s
R_{Y=0.8}

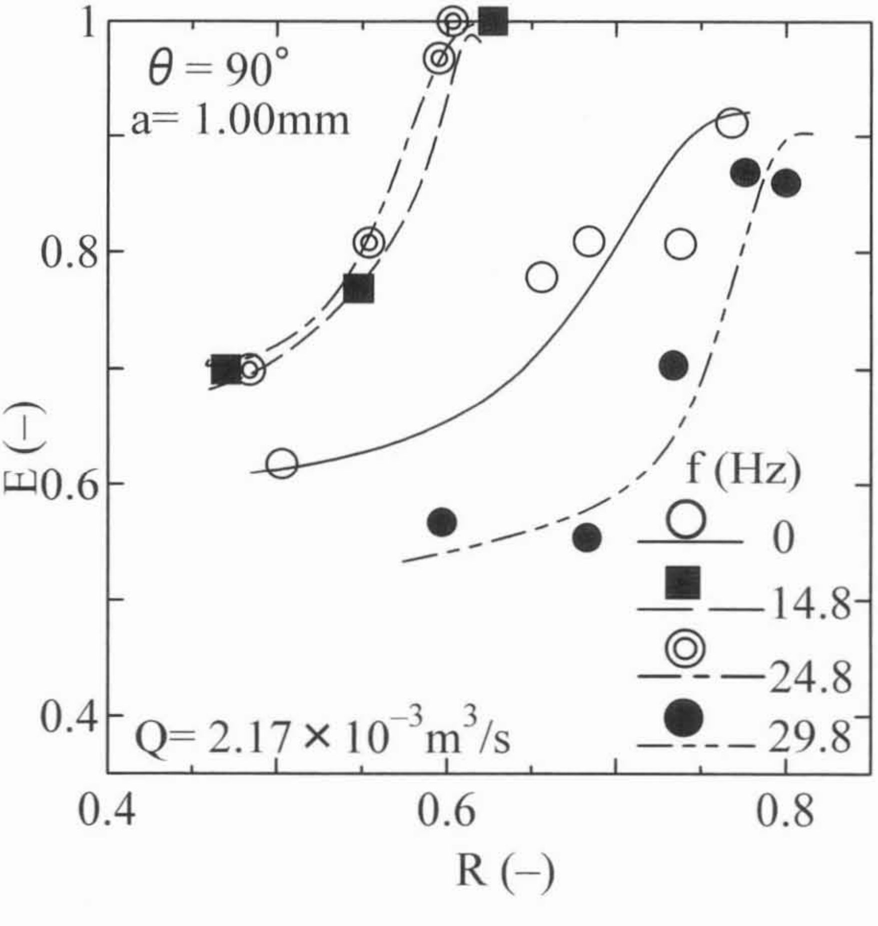

a= 1.29mm
Q= 2.17 × 10^{-3} m^3/s
$\theta = 0°$
● $R_{Y=0.8}$
○ $R_{E=0.8}$
$R_{Y=0.8}$ (−)
$R_{E=0.8}$ (−)
0.7
0.6
0.5
0
10
20
30
f (Hz)

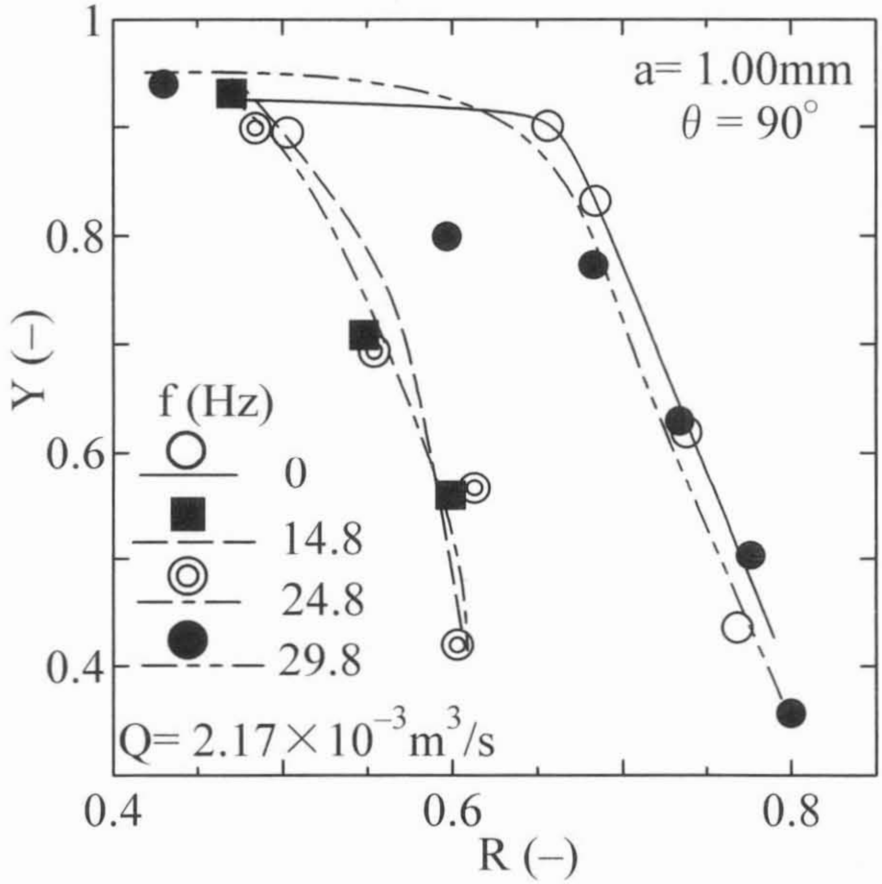

1
0.8
0.6
0.4
E (–)
0.4
0.6
0.8
R (–)
a= 2.55mm
f (Hz)
0
14.8
24.8
29.8
θ = 33°
Q= 2.17 × 10^{-3} m^3/s

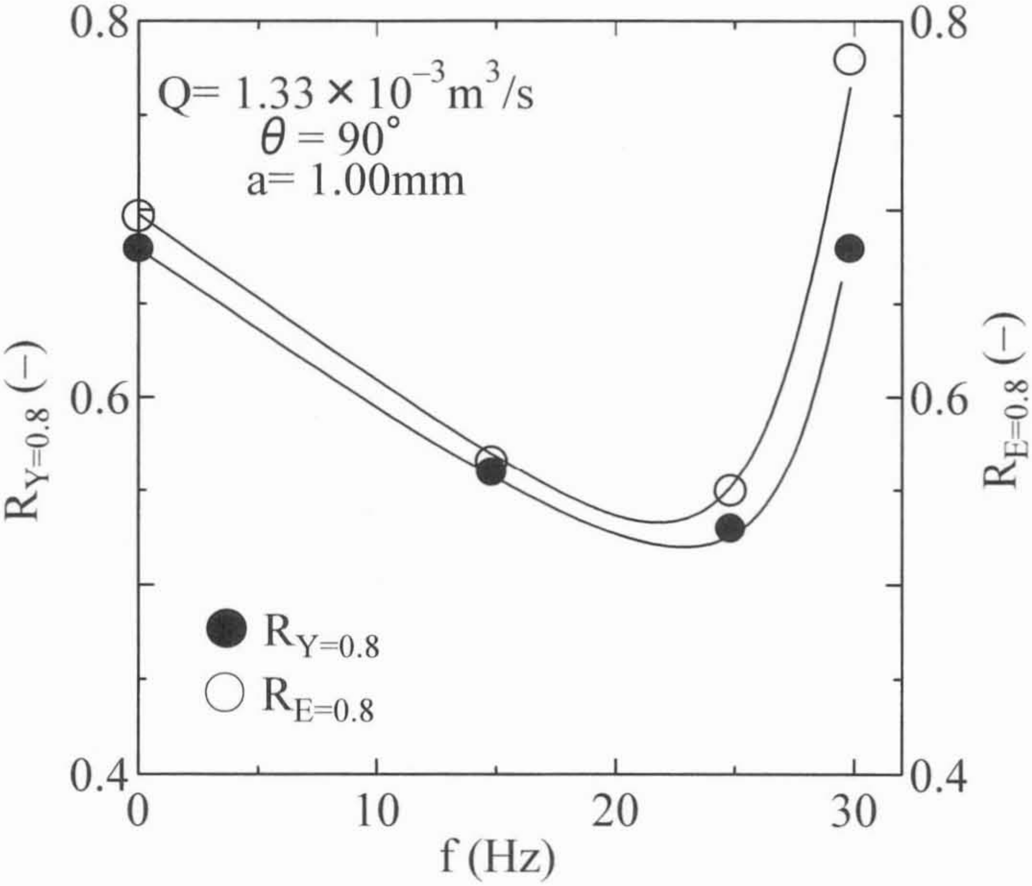

1
0.5
0
Y (–)
0.4
0.6
0.8
R (–)
θ = 33°
a= 2.55mm
f (Hz)
○ 0
■ 14.8
◎ 24.8
● 29.8
Q= 2.17 × 10⁻³m³/s



0.8
0.6
0.4
0.8
0.6
0.4
0
10
20
30
f (Hz)
$R_{Y=0.8}$ (–)
$R_{E=0.8}$ (–)
a= 2.5mm
Q= 2.17 × 10^{-3} m^3/s
θ = 33°
● $R_{Y=0.8}$
○ $R_{E=0.8}$


θ = 90°
a= 1.00mm
Q= 2.17 × 10^{-3}m^3/s
f (Hz)
0
14.8
24.8
29.8
E (–)
R (–)
1
0.8
0.6
0.4
0.4
0.6
0.8


a= 1.00mm
$\theta = 90^\circ$
Y (–)
1
0.8
0.6
0.4
R (–)
0.4
0.6
0.8
f (Hz)
○ 0
■ 14.8
◎ 24.8
● 29.8
Q= 2.17×10^{-3}m^3/s



0.8
0.6
0.4
0.8
0.6
0.4
0
10
20
30
f (Hz)
$R_{Y=0.8}$ (−)
$R_{E=0.8}$ (−)
Q= 1.33 × 10^{-3} m^3/s
θ = 90°
a= 1.00mm
● $R_{Y=0.8}$
○ $R_{E=0.8}$