The Role of Compaction in Agglomeration in Liquid and Formulation of Scale-up Equation*

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The effects of agitation intensity and amount of bridging liquid on agglomerate formation in agglomeration in liquid were experimentally investigated. An increase in agitation intensity advanced the compaction of agglomerates, reduced the porosity and changed the saturation degree which is the ratio by volume of the bridging liquid to the pores of an agglomerate. It was observed that the process of agglomerate growth depended on the changes in porosity. Based on this observation, the effects of agitation intensity on the process of compaction and changes in porosity were quantitatively elucidated and a scale-up equation was formulated. In addition, model agglomerates of the zirconia powder were cold isostatically pressed to investigate the influence of green agglomerate porosity on the density of sintered agglomerates. The results showed that the agglomerates produced by agglomeration in liquid must have porosities less than 0.51 in order to achieve high density and strength after sintering. To achieve this objective, the formulated scale-up equation was utilized to design a batch-type horizontal agglomerator that can provide the agitation energy required to produce agglomerates with porosities below 0.51. The newly designed agglomerator was successfully tested in the production of highly dense microspherical agglomerates of zirconia.

KEY WORDS: Compaction, Scale-up, Agglomeration in Liquid, Zirconia Microspheres

1. Introduction

High performance micro-spheres offer a number of industrial applications. Mixing, dispersing and ultrafine grinding of fine ceramics, super conducting materials, etc. are some applications that necessitate the use of micro-balls. However, conventional methods are hardly capable of producing micro-spheres with diameters less than 500 µm. The authors previously reported that it is possible to produce high performance dense micro-spheres of zirconia by the methods of agglomeration in liquid¹². In the production of microagglomerates, understanding the mechanism of agglomeration formation is very important. This knowledge is a key to the development of a scale-up equation that can be utilized to design large capacity agglomerators. Until now, scale-up of agglomerators have been based mainly on the experience of engineers and references are seldomly available. It is known that compaction is important for agglomerate formation as reported by Hira-jima et al.,³ Kawashima et al.,⁴ and Capes et al.,⁵. However, the effects of bridging liquid and the agitation intensity on agglomerate formation based on the compaction process have never been quantified which prevented their application in the development of a scale-up equation. In this paper, agglomeration tests are conducted to elucidate the effects of agitation intensity and the amount of bridging liquid on the process of agglomeration in liquid. The results are theoretically discussed based on the compaction process. The results from the agglomeration experiments are used in the derivation of a scale-up equation and agglomerate porosity equation. The formulated equation is applied in the scaling-up of agglomerator.

The final porosity of the green agglomerates affects the density and strength of sintered agglomerates. The porosity of a green agglomerate needed to ensure high density and strength after sintering is quantified by sintering model agglomerates shaped as disks. The result is used to determine the required agitation intensity and amount of bridging liquid to produce highly dense agglomerates. The calculated amount of bridging liquid and impeller rotational speed are used during the testing of the newly designed agglomerator.

2. Experimental

2.1 Agglomeration test

The starting material used was a commercially available zirconia stabilized with 2.6 mole % of Y₂O₃. A typical lot analysis (weight percent) of this material was 4.58 Y₂O₃, 0.02 CaO, 0.03 Na₂O, and the balance was ZrO₂. The equilibrium moisture content of this material was approximately 0.3%. The specific surface area was 6.76 m²/g and the average particle size was 0.45 µm. The size distribution of this powder fits the Rosin-Rammler distribution with absolute size constant \( D_e = 0.635 \) µm and the distribution constant \( n = 1.075 \). The suspending liquid used was an aliphatic hydrocarbon.

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containing isoparaffin. Some important properties of this organic liquid are: density: 0.75 g/cm³, interfacial tension: 43.01 dyne/cm, and carbon number: 7 to 12. Suspension consisting of 4.5 grams of zirconia powder and 150 cm³ of organic liquid was placed in a cylindrical container with a diameter of 60 mm and a height of 60 mm. The suspension was agitated at a certain speed by using a high speed motor fitted with an impeller and desired amount of bridging liquid, i.e., demineralized distilled water, was added by using a microsyringe. The apparatus set-up and the configurations of the impellers used are depicted in Fig. 1. Agitation was stopped when all the suspended powders had formed to agglomerates and the suspension became transparent. The time when agitation was stopped is defined as the agglomeration time, $T_A$. The total amount of electrical power consumed for the agitation of the suspension for each impeller configuration and speed was measured by using a power meter, Hioki Hi-Tester model 2503. All power data refer to the power consumed for agitating the suspension minus the power consumed for rotating the impeller in air. In this paper, agitation intensity is used synonymous to energy dissipation which is proportional to the agitation power divided by the volume of suspension.

2.2 Quantification of the effect of presintered agglomerate porosity on sintered density

The zirconia powder was formed into compacts shaped as disks or tablets. A desired amount of the powder was placed in a cylindrical mold with a diameter of 16 mm and height of 50 mm and uniaxially pressed at a load of 500 kgf/cm² with a crosshead speed of 2 cm/min. The powder compact was carefully withdrawn from the mold, sealed in a vacuum pack and isostatically pressed at pressures ranging from 500 to 4,000 kgf/cm² in a cold isostatic press, Dr. CIP by Kobe Seiko. The pressed compacts were weighed and their dimensions measured by using an image analyzer, Luzex II by Nireco Corp. The powder compacts were sintered at 1,450°C for two hours. Porosity of the green powder compacts was calculated based on their weight and dimensions. Archimedes method was utilized to determine the density of the sintered compacts.

3. Results and Discussion

3.1 Effect of compaction process on agglomerate formation

The effects of bridging liquid, impeller configuration and agitation speed on the agglomeration time ($T_A$) are shown in Fig. 2. The amount of bridging liquid is expressed as a ratio by weight of the bridging liquid added to the solid particles in suspension. The titles in the legend refer to the impeller type and agitation speed, e.g. A3000 means impeller type A rotated at 3,000 rpm. Some of the curves were extrapolated from experimental data because even after 120 minutes of agitation only partial agglomeration was attained. In general, immediately after the addition of bridging liquid, the initial particles are observed to form agglomerates and flocs. With the passage of time, some flocs consolidate to form discrete agglomerates and the remaining flocs coalesce with existing agglomerates by snow balling mechanism. Since $T_A$ refers to the time when the suspension became clear, it indicates the time when all the flocs had formed to agglomerates. For all impeller configurations, when $T_A$ was kept constant, increasing the impeller speed resulted in the reduction of the necessary amount of bridging liquid to form agglomerates. For constant bridging liquid addition, $T_A$ decreased with an increasing impeller speed. When the impeller speed was 3,000 rpm, larger amount of bridging liquid was required to obtain a certain $T_A$ for the impeller of type A configuration compared to types B and C. When types B and C were used at an impeller speed of 3,000 rpm, curves 5 and 10 coincided. In other words, doubling the width of the blade produces the same result as increasing the number of blades. The results shown in Fig. 2 indicate that increasing agitation intensity enhances compaction and reduces the amount of bridging liquid. In addition, it is possible to manipulate agitation...
The role of compaction in agglomeration in liquid and formulation of scale-up equation

Intensity through the impeller speed, blade width, number of blades, etc.

The compaction process of an agglomerate in a drum granulator was theoretically studied by Ouchiyama and Tanaka\(^6\) and given as:

\[
\frac{d\varepsilon}{dr} = -\left(1 - \frac{\varepsilon}{\varepsilon_0} (1 - \varepsilon)^m / K_C\right) \quad \text{I} \quad \text{equation (1)}
\]

where \(l\) and \(m\) are constants approximately equal to 1 and 2, respectively, \(\varepsilon\) is porosity, \(r\) is dimensionless time and \(K_C\) is compaction rate. They elaborated that compaction of agglomerates in a drum granulator is caused mainly by the impact of agglomerates to the walls of the drum, hence, \(K_C\) is a function of the square of the maximum velocity of agglomerate before impact (\(v_{\text{max}}^2\)). Other factors that influence \(K_C\) are: particle diameter, friction, interfacial tension of the bridging liquid, contact angle and the saturation degree \(S\). Saturation degree refers to the volume of bridging liquid divided by the volume of pores of an agglomerate.

In a recent report, the Ouchiyama-Tanaka equation was adapted to describe the compaction process of the agglomerates in the agglomeration in liquid by using the Kolmogorov theory to describe the velocity of agglomerates\(^3\). The relation between \(\tau\) and real time (\(t\)) was expressed as:

\[
\tau = (13/256)q_o STvT/V \quad \text{equation (2)}
\]

where, \(ST\) is the total surface area of the vessel and impeller available for impact with the agglomerate, \(V\) is the volume of suspension, \(v\) is the mean velocity of agglomerate and \(q_o\) is a proportionality constant.

Hirajima et al. reported the influences of saturation degree (\(S\)) in the process of agglomeration in liquid. They have shown that in a heterogeneous agglomeration system, where flocs and agglomerates coexist, disappearance of flocs and complete agglomeration are attained upon reaching about 0.70–0.75 \(S\) value. This range of \(S\) value may change depending on the agglomeration mechanism which is affected by the method of bridging liquid addition, size distribution of feed particles, etc. However, for similar agglomeration mechanism, the required range of \(S\) for complete agglomeration is almost the same. \(T_A\) shown in Fig. 2 refers to the time when all the flocs disappeared and can be considered to indicate the time when \(S\) attained the required range for complete agglomeration. The saturation degree \(S\) is expressed as:

\[
S = \frac{1 - \varepsilon}{\varepsilon_0} \frac{\rho_p}{\rho_l} \frac{\omega}{\varepsilon} \quad \text{equation (3)}
\]

where \(\omega\) is the amount of bridging liquid (liquid weight/solid weight), \(\rho_p\) and \(\rho_l\) are the densities of solid particles and the bridging liquid, respectively. By assuming \(S\) to be equal to 0.7 when all the flocs disappeared and assigning values for \(\omega\), porosity can be calculated from Equation 3. Let us assume that this calculated porosity \(\varepsilon_{\text{min}}\) attainable in a given agitation environment.

By equating \(d\varepsilon / dr\) of Equation 1 to zero, the relation between \(\varepsilon_{\text{min}}\) and \(K_C\) can be derived:

\[
K_C = -\left(1 - \varepsilon_{\text{min}}^3\right) / \varepsilon \quad \text{at} \quad \varepsilon = \varepsilon_{\text{min}} \quad \text{equation (4)}
\]

The calculated \(K_C\) was plotted against the amount of bridging liquid \(\omega\) and shown in Fig. 3. The straight line in Fig. 3 can be expressed as:

\[
\omega = 0.064K_C^{-0.47} \quad \text{at} \quad \varepsilon_{\text{min}} \quad \text{equation (5)}
\]

It was generally observed that the compaction process is characterized by an initial rapid decrease in porosity followed by a transition stage and equilibrates to the minimum attainable value\(^3\). Based on this observation and the results shown in Fig. 2, it can be assumed that in the proximity of 100 minutes agglomeration time the porosity of agglomerates approaches the minimum because complete agglomeration was attained with the least amount of bridging liquid. In other words, the least porosity possible under the existing operating condition is attained at \(T_A = 100\) min or \(\varepsilon\) approaches \(\varepsilon_{\text{min}}\). The amounts of bridging liquid (\(\omega\)) needed to form agglomerates at \(T_A = 100\) min were obtained in Fig. 2. The plot of \(\omega\) against the agitation intensity \(\xi\) is shown in Fig. 4. The data can be fitted to the expression:

\[
\omega = 0.225\xi^{-0.094} \quad \text{when} \quad \varepsilon \rightarrow \varepsilon_{\text{min}} \quad \text{equation (6)}
\]

Increasing agitation intensity will decrease the amount of bridging liquid necessary to form agglomerates. Now, equating Equation 5 to Equation 6 gives:

\[
K_C = 0.069\xi^{0.199} \quad \text{at} \quad \varepsilon_{\text{min}} \quad \text{equation (7)}
\]

This empirical relation shows that the compaction rate is directly proportional to \(0.199\) power of the agitation intensity.

The amount of bridging liquid needed to form agglomerates at \(T_A = 100\) min as a function of energy dissipation \(\xi\).

Fig. 3 Relationship between the calculated \(K_C\) and the amount of bridging liquid \(\omega\).

Fig. 4 The amount of bridging liquid needed to form agglomerates at \(T_A = 100\) min as a function of energy dissipation \(\xi\).
Fig. 5 Agglomerate porosity $\varepsilon$ as a function of dimensionless time $\tau$.

The $K_C$ value was calculated by using the $\xi$ from agitation experiments as inputs to Equation 7. Using some of these $K_C$ values and assuming that the initial porosity $\varepsilon_o = 0.9$, Equation 1 was solved numerically. The calculated $\varepsilon$ was plotted against $\tau$ and shown in Fig. 5. The value of $\varepsilon_0$ was arbitrarily chosen to demonstrate the qualitative variation of $\varepsilon$ with time as a function of $K_C$. These curves show that with the passage of time, the compaction process is characterized by an initial rapid decrease in porosity followed by a transition stage and equilibrates to a minimum value. Fig. 5 also shows that with constant time, increasing $K_C$ or increasing agitation intensity results to a reduction in the attainable minimum porosity.

It is important to establish the relation of $\tau$ with the real agglomeration time $t$. From the works of Ouchiyama and Tanaka, $K_C$ is a function of the square of $v_{\text{max}}$ and from the empirical relation of Equation 7, $K_C$ is proportional to the $\xi$ raised to the 0.199 power. Deduction from these relations will show that $v_{\text{max}}$ is a function of the 0.1 power of $\xi$. If we can assume that the mean velocity included in Equation 2 is a function of $v_{\text{max}}$, then the relation between $\tau$ and $t$ can be simplified as:

$$t = K_1 \tau^{-0.1} \tag{8}$$

where $K_1$ is a constant. Published reports in hydrodynamics have shown that the mean velocity of particles in a turbulent environment is proportional to $1/3$ power of $\xi^{3/7}$. If this is adapted, the mean velocity will be proportional to $\xi^{1/3}$ and the relation between $\tau$ and $t$ will be:

$$t = K_2 \tau^{1/3} \tag{9}$$

where $K_2$ is a constant.

By using either Equation 8 or 9, the results of the compaction process simulation, i.e. Fig. 5, where replotted against $t$ and shown in Fig. 6 where a) $K_1 = 42$ and b) $K_2 = 104$ were arbitrarily used to investigate qualitatively the compaction process. From Fig. 6 it is evident that when $t$ is kept constant increasing the compaction rate by increasing agitation intensity results in the reduction of porosity. The time needed to achieve constant porosity decreases with an increasing agitation intensity. These results indicate that increasing agitation intensity enhances compaction. Let us say our objective agglomeration time is 20 minutes. The porosity of agglomerates at this specified time was obtained from the compaction curves in Fig. 6 and the amount of bridging liquid ($\omega_C$) required to attain $S = 0.70$ was calculated from Equation 3. The calculated $\omega_C$ was noted to be decreasing with increasing agitation intensity. Thus, by providing enough agitation intensity, even with less amount of bridging liquid it is possible to attain a saturation of 0.70 and achieve complete agglomeration. From the results of agglomeration test shown in Fig. 2, the experimental value $\omega_{20}$ when $T_A$ is 20 min...
was obtained, plotted against the calculated $\omega_c$ and shown in Fig. 7. The values of $\omega_c$ obtained by using Equation 8, i.e. $t \alpha x^{-0.1}$ show closer linearity than those obtained when Equation 9, $t \alpha x^{-1.3}$ was used. The good agreement between the calculated and measured data confirms the validity of the assumptions that $S$ is constant at the time when complete agglomeration occurred and particle velocity is a function of energy dissipation, i.e. agitation intensity. The results of the compaction process simulation provide a vivid picture of the mechanism of agglomerate growth and formation. Immediately after the addition of bridging liquid, agglomerates and flocs are formed. Some of the flocs are consolidated during agitation and formed agglomerates, while some are gathered by existing agglomerates by snowballing mechanism. The snowballing mechanism is enhanced by an increase in the agitation intensity since the bridging liquid inside the pores of the agglomerates are squeezed out to the surface. The flocs coalesce easily with the surface wet agglomerates. At the time when flocs disappeared and all particles have formed into agglomerates, the saturation degree is constant and approximately equal to 0.70.

3.2 Formulation of scale-up equation

For direct scale-up it is necessary to establish an index of conditions required to give an acceptable process result. The previous section provided a clear understanding of the influence of agitation intensity and bridging liquid on agglomerate formation. This knowledge will be used in the derivation of a scale-up equation and estimation of product porosity. It is considered that energy dissipation or power per unit volume can provide the basis for scale-up. The experimental values of $\omega_{20}$ for all impeller configurations were plotted against their corresponding $t$ and shown in Fig. 8. The relation can be expressed as;

$$\omega_{20} = 0.242 \xi^{-0.104}$$

This represents the bridging liquid needed to form agglomerates under a certain agitation intensity and when $T_A$ is 20 minutes. Equation 10 shows that increasing $\xi$ will reduce $\omega_{20}$ or with large $\omega_{20}$ the required $\xi$ will be lower. If it is desired to maintain a constant $\omega_{20}$, it is imperative to keep a constant $\xi$. Using the relation expressed in Equation 3, Equation 10 can be transformed to one with $\xi$ as variable;

$$\xi = \frac{1.382 \omega_{20}^{-0.104}}{S + 1.382 \omega_{20}^{-0.104}}$$

This expression is represented in a plot of $\xi$ versus $\omega_{20}$ shown in Fig. 9, where $S$ equal to 0.7 was used. Increasing $\xi$ will effectively decrease porosity. With a known agitation intensity the porosity of agglomerates formed in 20 minutes can be estimated from Equation 11.

Early investigators considered that impeller power should be a function of the geometry of the impeller and the tank, the properties of fluids, i.e., viscosity and density, the rotational speed of the impeller and gravitational force. Since only one type of organic liquid was used in our experiments, the kinematic and dynamic similarity of the system for all experiments is maintained and only the impeller geometry and rotational speed can be considered as variables. Agitation power $P$ in turbulent flow is expressed as;

$$P = N \pi D_f^4 \omega n_b$$

where $D_f$ is the impeller diameter, $\omega$ is the blade width, $n_b$ is the number of blades multiplied by the number of impellers divided by 4 and $N_f$ is the impeller rotational speed in rps.

The energy dissipation or the electrical energy consumed per unit volume of the fluid, i.e. $\xi$, should be proportional to the agitation power divided by the volume of fluids in the vessel ($V \propto \pi D_v^2 L_v/4$, $D_v$ and $L_v$ are the vessel's diameter and length) or;

$$\xi = P/V$$

By letting $k$ to be a proportionality constant and combining Equations 10, 12 and 13, an expression for the relation of $\omega_{20}$ with impeller diameter and rotational speed can be obtained;

$$\omega_{20} = 0.242 \left( \frac{kN \pi D_f^4 \omega n_b}{V} \right)^{-0.104}$$

If it is desired to form agglomerates in 20 minutes using a constant dosage of bridging liquid, the agitation intensity can be adjusted by manipulating the geometry of the vessel and impeller or the rotational speed of the impeller. Thus, Equation 14 is a scale-up equation that can be utilized for designing an agglomerator.

3.3 Effect of green agglomerate porosity on density of sintered agglomerates

In the production of zirconia microspheres that can be...
used as media for grinding, mixing and dispersing, it is important to reduce the porosity of green agglomerates to enhance density and strength of sintered agglomerates. From the previous section it was shown that porosity was a function of the agitation intensity and decreased with an increase in agitation intensity. It is therefore essential to determine the optimum porosity of green agglomerates that will yield high density of sintered agglomerates. The effect of the porosity was quantified by using model agglomerates shaped as disks. The porosity of each disk was varied by using different pressures during cold isostatic pressing (CIP).

Shown in Fig. 10 is the plot of CIP pressure versus the porosity of green powder compacts. The straight line is the statistical fit which clearly shows that the amount of void spaces in the powder compacts decreases with an increasing CIP pressure. Fig. 11 shows the relationship between the porosity of green powder compact and the relative density of sintered compacts. Relative density refers to the percentage of density of sintered compact measured by the Archimedes principle against the theoretical density of zirconia assumed as 6.1 g/cm³. The maximum relative density attained was in the neighborhood of 97.7%. This plot illustrates the dependence of final sintered density on the porosity of green powder compact prior to sintering. Generally, decreasing the porosity increases the relative density of sintered compact. However, at about 0.51 porosity, the relative density began to level off at its maximum value. With porosities less than 0.51, the voids can be sealed during the process of sintering whilst beyond this limit the voids are probably too large to close.

This indicates that the porosity of agglomerates must be reduced below 0.51 in order to obtain highly dense agglomerates.

The agglomerates formed with agitation intensity ranging from 2 to 143 kW/m³ and bridging liquid additions ranging from 0.150 to 0.222 were sintered and densities were measured. As expected, the relative densities of sintered agglomerates were noted to be below 97.7% and in the range of 89.4% to 95.2%. The porosities of these agglomerates were estimated from the relative density-porosity curve shown in Fig. 11. The estimated porosities were plotted against the amount of bridging liquid added and shown in Fig. 12. The line in Fig. 12 is obtained by using Equation 3 with $S = 0.70$. The closeness of the measured data to the line indicates that the assumption, that $S$ is approximately equal to 0.70 when complete agglomeration is attained, is valid. The fluctuation in the measured data can be understood as errors arising either from the estimation of porosity from Fig. 11 or from TA which was judged based only on ocular observation of the slurry. As can be seen in Fig. 9, the calculated porosity of agglomerates when $T_A = 20$ min and $S = 0.7$ for different agitation intensities, the porosity reduction is only in the range of 0.65 to 0.55. It is apparent that in order to produce dense agglomerates, higher degree of agitation and compaction is necessary to reduce the porosity to less than 0.51 with $T_A = 20$ min.

The required energy dissipation to produce dense agglomerates with an agglomeration time of 20 minutes was calculated by using $\xi = 0.51$ as input to Equation 11 and the result was $\omega_{20} = 0.128$ as the desirable amount of bridging liquid. The point where $\xi = 463$ kW/m³ and $\omega_{20} = 0.128$ was extrapolated in Fig. 8 and marked by $\Rightarrow$.

Using the relationship expressed in Equation 14 and the desirable bridging liquid calculated previously, $\omega_{20} = 0.128$, yields 7,826 rpm as the required speed for impeller type A and 6,212 rpm for types B and C. Agitation at these impeller speeds is rather unsafe for the previously described system.

**3.4 Design and testing of a 3 liter agglomerator**

The limitations of the small agglomerator previously described necessitated the design and construction of an
agglomerator with larger capacity and capable of higher agitation intensity. Based on Equation 14, a horizontal three liter agglomerator was developed. The internal volume of the cylindrical vessel minus the volume of the shaft-impeller assembly is 3,000 cm³. The vessel consists of lower and upper parts constructed from stainless steel. The lower portion is fitted with a cooling jacket to prevent overheating and control the temperature during agglomeration. The agglomerator is also fitted with circulation tubes to facilitate visual observation of the progress of agglomeration and to ensure homogeneity of mixing. The combined internal volume of the circulation tubes is 400 cm³. To serve as baffles, grooves are machined in the internal surface of the cylindrical vessel. A sketch of the newly designed agglomerator is illustrated in Fig. 13.

The relation between impeller speed and agitation power was determined by agitating 3,200 cm³ of the organic liquid in the newly designed agglomerator. The relation of to is shown in Fig. 14. It is evident that the agitation power in a turbulent flow is proportional to the impeller speed raised to the third power and confirms Equation 12.

The above agglomerator was tested using 100 grams of the zirconia powder and 3,200 cm³ of organic liquid. In section 3.3 it was shown that if it is desired to reduce below 0.51, to obtain high sintered density, the required must be larger than 463 kW/m³. In this test, was 553 kW/m³, equivalent to an impeller speed of 1,840 rpm, to assure that the green agglomerates will have a porosity less than 0.51, i.e. 0.505. With this , the required amount of bridging liquid added was calculated from Equation 10 to be 0.125. Complete agglomeration was attained after 17 minutes. The agglomerates exhibited good spherical shape. The mean diameter of the sintered agglomerate was 670 μm and the density was 5.95 g/cm³ or 97.5% theoretical density.

The good results of the 3 liter agglomerator paved the way to the design of commercial-scale agglomerators which are currently operational for the production of highly dense zirconia microspheres 9).

4. Conclusion

The roles of agitation and bridging liquid in agglomerate formation were elucidated. Theoretical considerations based on compaction process support the results of the agglomeration experiments. The good agreement between the results of simulation and agglomeration experiments evinces the assumption that saturation degree needed for complete agglomeration has a value close to 0.7. Increasing agitation either accelerates agglomerate formation with a constant dosage of bridging liquid or reduces the amount of bridging liquid needed to form agglomerates. Likewise, increasing bridging liquid addition either shortens agglomeration time with a constant agitation intensity or lowers the agitation intensity needed to form agglomerates. Agitation facilitates compaction and the reduction of porosity causes the saturation to increase to the level needed for agglomerate formation even with small dosage of bridging liquid. With large dosage of bridging liquid, the required saturation degree is attained even with lesser compaction and lower agitation.

The relation of bridging liquid to agitation intensity was used in the formulation of a scale-up equation for agglomerators and green agglomerate porosity equation. The green agglomerate porosity must be reduced below 0.51 in order to achieve high density and strength after sintering. The agitation intensity must be higher than 463 kW/m³ and the amount of bridging liquid needed must be ω = 0.128 in order to form dense agglomerates within 20 minutes. The 150 cm³ agglomerator is unsafe to this level of agitation creating a need for an agglomerator with higher agitation capability. The scale-up equation was used to design a 3 liter agglomerator. The newly designed 3 liter agglomerator, using calculated operating conditions, was successfully tested in the production of highly dense microspherical agglomerates.

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液中造粒における造粒体の圧密過程の役割とスケールアップ則の導出

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本論文では、液中造粒における造粒体形成に及ぼす攪拌強度および実験体の影響、造粒体の圧密過程について検討し、スケールアップ則を導き、これを用いて高密度造粒体を得るための造粒装置を設計、製作した。造粒体の空隙に占める実験体の体積割合を飽和密度にすると、攪拌に伴う造粒体の圧密が進行し、飽和度も変化する。造粒体の成長過程はこの飽和度の変化と密接に関係している。この関係に着目して、圧密過程、飽和度に及ぼす攪拌強度の影響を定量化的に把握することにより、スケールアップ則を導いた。また、シルコニア粉末を冷間圧延圧制成型で作成したモデル造粒体について、空隙率と焼結後の密度との関係を調べた。

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