Review Liquid Bridge Adhesion Force and Dispersion of Aggregate Particles†

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Abstract
Many studies have been made on liquid bridges between particles, but the mechanism of liquid bridge formation and its adhesion force when the bridge is in an equilibrium condition with the surrounding humidity has not been theoretically clarified. A study has been made on this subject and a new theory is introduced in this paper. Next, a mechanism to disperse aggregate particles down to primary particles by the acceleration of an air stream is explained and a discussion is made on the dispersion force applied to aggregate particles and the adhesion force between particles (van der Waals force and liquid bridge force).

1. Introduction
Among the adhesion forces which act between particles or between particles and walls in air, the adhesion force arising from a liquid bridge is the greatest. This liquid bridge often makes handling of powdery materials difficult. In describing the liquid bridge, Laplace Young’s equation1,2 may be important. This equation gives the internal pressure inside a liquid surrounded by convex and concave surface. Many theoretical studies on liquid bridges use this equation to obtain the liquid bridge shape and the adhesion force of liquid bridges but these studies have not quantitatively related liquid bridge shapes and the adhesion forces to surrounding humidities. Another important equation in this connection is Kelvin’s equation3 which is used to explain the formation of liquid bridges by capillary condensation. However, this theoretical equation is erroneously used as explained later. Here, to simplify the discussion, the liquid bridge shape and the adhesion force acting between particles in an equilibrium condition with the surrounding humidity is explained for a liquid bridge formed between two spheres of identical size with 0° contact angle between the liquid and the particle. The equilibrium moisture contents in a powdery material and the strength of a powder bed are explained in relation with liquid bridges.

Next, a method and experimental results of the dispersion of aggregate particles into primary particles by acceleration of an air stream are introduced, and the mechanism of dispersion, the dispersion force acting on particles, and the adhesion force between particles are discussed.

2. Liquid Bridge Formation and Adhesion Force between Particles

2.1 Liquid Bridge Formation by Ideal Pure Water
As shown in Fig. 1, the negative pressure (capillary pressure) $P_L$ (taking negative pressure as a positive value) is obtained from the following Laplace-Young Equation.

$$P_L = \sigma \left(\frac{1}{r_1'} - \frac{1}{r_2'}\right)$$  \hspace{1cm} (1)

Where $\sigma$ is the surface tension of the liquid and $r_1'$, $r_2'$ are radii shown in Fig. 1. The capillary pressure $P_L$ is constant in a bridge and therefore the shape of the liquid bridge becomes a symmetry of rotation which satisfies Equation (1) with $P_L$ as a constant. The capital letters in Fig. 1 indicate the dimensionless values which are obtained on the basis of the particle radius $r_0$. On the other hand, the partial pressure of vapor at the surface of a liquid bridge is obtained using the following Kelvin equation4.

$$\frac{P_d}{P_{so}} = \exp \left[-\frac{M_s}{RT} \frac{\rho_i}{\rho} \frac{dS}{dV}\right] = P_{Kel}$$  \hspace{1cm} (2)

Where $P_{so}$ is the saturation vapor pressure, $M$ the molecular weight of the liquid, $R$ the gas constant, $T$ the absolute temperature, $\rho_i$ the density of the liquid, $s$ the vapor liquid interface surface area of the liquid bridge, and $v$ the volume. $P_{Kel}$ is explained.

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later. For water $P_d/P_{50}$ is equivalent to the surrounding relative humidity of the liquid bridge. A liquid bridge at a certain humidity satisfies Equation (2). Therefore $ds/dv$ for a liquid bridge which satisfies Equation (1) is obtained as the following approximate equation:

$$
\frac{ds}{dv} = \frac{1.5}{R_2} \text{ or } \frac{ds}{dv} = \frac{1.5}{r_2} \quad (R_2 = r_2/r_0) \quad (3)
$$

Where $R_2$ is the dimensionless neck radius shown in Fig. 1, and $r_0$ the particle radius. The relation of Equation (3) is valid not only for a liquid bridge between two identical spheres but also for a liquid bridge between different size spheres, and between a sphere and a flat plate. In this case, $r_0$ is the radius of the smaller sphere.

When Equations (2) and (3) are used, the size of a liquid bridge (represented by $r_2$ in Fig. 1) in an equilibrium condition with humidity can be obtained as the solid curve in Fig. 2, and under a room temperature and atmospheric pressure, the neck radius $r_2$ of the pure water bridge reaches about 0.15 nm (radius of water molecule) – 100 nm, regardless of the particle radius $r_0$. In the past, the liquid bridge shape was assumed to be a circular arc having a radius $r_1$ shown in Fig. 1 as constant ($= r_1$) and Equation (3) was expressed as follows:

$$
\frac{ds}{dv} = \frac{1}{r_1} - \frac{1}{r_2} \quad (4)
$$

When the separation distance between two spheres as shown in Fig. 1 is approximated to $h = 0$, this equation takes the following form.

The dashed curve of Fig. 2 shows the relation of Equation (5) when the particle radius $r_0$ is given as 1 μm and the value differs from the solid curve of Equation (3). In past studies, the bridge shape was approximated by a circular arc and therefore, the difference in Fig. 2 shows that Kelvin’s equation was not correctly used. When $r_0$ becomes larger than 1 μm, the difference between Equation (3) and Equation (5) becomes much greater.

Figure 3 shows the observation results of a liquid bridge between glass beads with diameters of 1 mm, with an optical microscope. Figure 3(a) shows a photomicrograph of ultra-pure water used for a bridge liquid, left for 24 hours at a humidity of 90%. Figure 3(b) shows tap water used for a bridge liquid left for 24 hours at a humidity of 40%. In Fig. 3(a), the existence of a liquid bridge could not be confirmed with a microscope, which supports the theory in Fig. 2. Figure 3(b) indicates that the inclusion of a small amount of soluble impurities in the tap water affects the formation of a liquid bridge. The reason for this is explained in detail in the next section. In past studies in which ultra-pure water was not available, liquid bridges which had grown large like the one shown in Fig. 3(b) were observed and it is thought that the results were compared with Equation (4).
2.2 Liquid Bridge Formation by Water Containing Soluble Impurities

Once particles produced in water are dried, soluble impurities contained in water remain on the particles’ surfaces as residue and if they are hygroscopic, they absorb the moisture contained in the air and form liquid bridges. Even in cases where soluble impurities are contained in a bridge liquid such as this, the following modified Kelvin equation can be applied\(^{(10,11)}\):

\[
\frac{P_d}{P_{s0}} = (1 - \gamma) P_{Kel}
\]  

(6)

Where \(P_{Kel}\) indicates the right side of Equation (2) and \(ds/dv\) in the equation is obtained from Equation (3). \(\gamma = \frac{n_s}{n_w + n_s}\), where \(n\) is the number of molecules, subscript \(W\) represents pure water, and \(s\) stands for the solute. Furthermore, \(i\) is van’t Hoff factor and differs depending on the type and concentration of the solute but with NaCl, H\(_2\)SO\(_4\), etc., it is about 2. \(P_d/P_{s0}\) is the ambient relative humidity. \(\gamma\) in equation (6) becomes as follows when the number of solute molecules, \(n_s\), divided by the single particle volume \(\nu_0\) is defined as \(N_5^{(10)}\).

\[
\gamma = \frac{iM_w}{\nu e}\left(\frac{1}{\nu_0} + \frac{M_s}{\nu_0 N_S} + \frac{iM_w}{\nu_0}\right)
\]  

(7)

Where \(M\) is the molecular weight, \(\nu\) is the density, \(\nu\) is the volume of the liquid bridge, subscript \(w\) represents pure water, and \(s\) the solute. The liquid bridge volume can be approximated by the following equation when particles of Fig. 1 come into contact with each other (\(H = 0\))\(^{(10)}\).

\[
\nu = \frac{(\pi/2)\nu_0^3 R_2^4}{N_5^{(10)}}
\]  

(8)

Figure 4(b) and (c) show the Kelvin effect (\(P_{Kel}\)) on a liquid bridge and the effect of the solute concentration (\(1 - \gamma\))\(^{(10)}\) for a CaCl\(_2\) water solution. The Kelvin effect shown in Fig. 4(c) satisfies the same relation as that of the solid curve of Fig. 2. The effect of solute concentration shown in Fig. 4(b) makes the liquid bridge (\(R_2\)) larger with an increase in solute contents \(N_S\). The dashed curve in Fig. 4(b) shows the range within which the solute (salts) recrystallizes with a decrease in humidity. Also, nearly the same results as those of Fig. 4(b) are obtained with solutes other than CaCl\(_2\)\(^{(10)}\). The adhesion force between particles shown in Fig. 4(a) is explained later.

If the particle radius \(r_0\), the type of the solute and its \(N_S\) content are known, the size of the liquid bridge (\(R_2\)) can be determined so that the ordinate product \((1 - \gamma) P_{Kel}\) of Fig. 4(b) and (c) becomes equal to the atmospheric humidity. When absolutely no solute is contained in the liquid, then \(\gamma = 0\), and \(R_2\) is determined by Fig. 4(c) only. On the other hand, when the particle radius \(r_0\) is large and the solute amount \(N_S\) is large, the effect of the solute concentration becomes dominant and Kelvin’s effect becomes negligible (\(P_{Kel} \approx 1\)). Thus the size of the liquid bridge (\(R_2\)) is determined only by the effect...
of the solute concentration shown in Fig. 4(b). Most fine particles used in industrial processes satisfy this condition\textsuperscript{12}.

\begin{figure}
\centering
\includegraphics[width=0.5\textwidth]{fig4.png}
\caption{Change in liquid bridge force $F_L$ and neck radius $R_2$ with relative humidity $(1-\gamma) \cdot P_{Kel}$.}
\end{figure}

2.3 Equilibrium Moisture Contents in Powdery Materials

If it is to be assumed that all moisture contents in powdery materials form liquid bridges between particles and that the bridges are of a consistent size with uniform distribution, the equilibrium moisture contents (dry basis) $M_f$ can be expressed by the following equation.

$$M_f = \frac{k_p \varrho_l \nu}{2 \varrho_p \nu_p}$$

(9)

Where $\varrho_p$ is the density of a particle, $\nu_p$ is the volume of a particle, $k_p$ is the coordination number of particles in the powder bed and can be calculated from the porosity $\varepsilon$ of the powder bed by using Ridgway-Tarbuck's equation\textsuperscript{13} ($k_p = 13.8 \left(1 - \sqrt{1.21\varepsilon - 0.297}\right)$). $\nu$ is the volume of one liquid bridge and if the separation distance between particles in the powder bed is $h \approx 0$, $\nu$ can be approximated by Equation (8). From Equation (9), the volume of one liquid bridge $\nu$ and the moisture contents $M_f$ in a powder bed can be mutually converted. The equilibrium moisture contents for various packed beds of submicron particles were observed, and a relation between Equation (7) and Equation (9) was found to be valid\textsuperscript{12}. Figure 5 gives one of the examples. In this figure, after immersing a certain amount of iron oxide particles in CaCl\textsubscript{2} water solution, it was left at various humidities until equilibrium was reached and the equilibrium moisture contents $M_f$ was measured. The abscissa of Fig. 5 indicates the normalized volume of one liquid bridge converted by Equation (9) from the equilibrium moisture content $M_f$. The results roughly coincide with the theoretical line of Equation (9) in which CaCl\textsubscript{2} concentration was high and Kelvin's effect was neglected. When ultra-pure water without CaCl\textsubscript{2} was used (\(\circ\) mark), a liquid bridge of about same size when $1.3 \times 10^2$ mol/m\textsuperscript{3} of CaCl\textsubscript{2} is added was formed. This is presumed that some kinds of soluble impurities corresponding to this were attached to the particle from the beginning.

\begin{figure}
\centering
\includegraphics[width=0.5\textwidth]{fig5.png}
\caption{Relation between volume of a liquid bridge in powdery materials and relative humidity}
\end{figure}

Small amounts of soluble impurities are usually attached to fine particles which are used in industrial processes and this is often the cause of the formation of liquid bridges. By sufficiently washing the particles and eliminating soluble impurities as much as possible, the moisture content of a particle bed and the adhesion force of liquid bridges can be reduced.

2.4 Adhesion Force Arising from a Liquid Bridge

The liquid bridge adhesion force $F_L$ can be obtained as follows by integrating Laplace-Young’s equation\textsuperscript{14, 15}.

\begin{figure}
\centering
\includegraphics[width=0.5\textwidth]{fig5.png}
\caption{Relation between volume of a liquid bridge in powdery materials and relative humidity}
\end{figure}
Where the right side first term is the capillary force of an arbitrary cross section vertical to the x axis of the liquid bridge shown in Fig. 1, the second term expresses the component of x axis direction of surface tension at the vapor liquid interface of the same cross section, and it is shown that the sum of the two forces is constant \( F_L \) regardless of the position of the x axis. Equation (10) is not limited to a liquid bridge between two identical spheres but is valid between two different size spheres or between spheres and plates. Besides this, equations expressing the adhesion force of liquid bridges include Pietsh’s equation and Fisher’s equation for two identical spheres, and McFarlane’s equation for spheres and plates but Pietsh’s equation does not accurately express the adhesion force because it approximates a liquid bridge shape to a circular arc. Also the results obtained in the two latter equations are equivalent to the result of Equation (10) but in a strict sense, their derivation is not accurate.

Fig. 4(a) shows the relation of Equation (10) for liquid bridges between two identical spheres. As mentioned before, when a liquid bridge size \( R_2 \) in equilibrium with the surrounding humidity is obtained from Figs. 4(b) and (c), adhesion force \( F_L \) between particles can be obtained from Fig. 4(a) for each distance between particles \( H (= h_{l0}) \). In a condition where there are no other external forces other than the liquid bridge adhesion force, the separation distance between particles approaches \( h \approx 0.4 \text{ nm} \) under the effect of the adhesion force of the liquid bridge and the surface free energy of the liquid bridge.

2.5 Strength of a Powder Bed

It is assumed that the powder bed tensile strength \( \sigma_t \) can be expressed by the following Rumpf’s equation,

\[
\sigma_t = \frac{k_p (1 - \varepsilon)}{\pi d_p^2} F_L
\]

(11)

Where \( \varepsilon \) is the porosity, \( d_p (= 2 r_0) \) is the diameter of particles composing the powder bed, and \( F_L \) is the liquid bridge force acting between two particles. Using this equation, the adhesion force \( F_L \) acting between two particles shown in Fig. 4(a) can be converted into powder bed strength \( \sigma_t \). If values other than the particle diameter \( d_p \) are constant, the liquid bridge adhesion force \( F_L \) is proportional to \( d_p \) as shown in Fig. 4(a). Therefore the powder bed strength \( \sigma_t \) is inversely proportional to \( d_p \) and as the particle diameter becomes smaller, \( \sigma_t \) becomes larger. Therefore as the particle size becomes smaller, handling of powdery materials becomes more difficult.

3. Dispersion of Aggregate Particles by Acceleration in an Air Stream

3.1 Theoretical Considerations

When an aggregate consisting of two particles, one large and one small, is placed in a high-speed air stream, a separation force arises during acceleration because the drag force and the inertial force acting on the respective particles are different. The dispersion of aggregate particles into primary particles by acceleration in an air stream utilizes this mechanism and as a disperser, an ejector structured to rapidly accelerate an air stream is used.

Using a model aggregate consisting of one large and one small particle as shown in Fig. 6, the dispersion force \( F_d \) can be derived as follows when the aggregate is instantaneously introduced into an uniform fluid flow.

\[
F_d = \left\{ 0.119 \eta / u_t \right\} A \left[ d_{V2}^2 D_V^2 \left( \sigma_A - \sigma_B \rho_A / \rho_B \right) + 2.07 \eta / u_t d_{V2}^3 D_V \left( \sigma_A - \sigma_B \rho_A / \rho_B \right) \right] \left[ 9.05 \eta u_t d_{V2} D_V \left( \sigma_A - \sigma_B \rho_A / \rho_B \right) \right] / \left( D_V^3 \rho_A / \rho_B + 1 \right)
\]

(12)

Where subscripts A and B relate to the two particles composing the aggregate shown in Fig. 6, \( \eta \) is the density of the fluid, \( \eta \) is the viscosity of the fluid, \( u_t \) is the relative velocity between the particle and the fluid, \( D_V = d_{V2}/d_{Vp} \), \( d_{Vp} \) is the volume equivalent diameter, \( \rho_p \) is the particle density, and \( \chi \) is the dynamic shape factor which is described later. The fluid density is related to this equation and therefore, even though the fluid velocity \( u_t \) reaches the critical velocity (sound velocity), a further large dispersion force can be obtained by increasing the fluid density by applying a high pressure.

The case when aggregate particles are composed of spherical primary particles of the same density \( \rho_A = \rho_B \) of Equation (12) was considered. Here, the volume equivalent diameter \( d_{Vp} \) of the aggregate particle is obtained using the following equation.
Ur
Fig. 6 A model aggregate

\[ dv = N^{2/3} d_{pl} \]  

(13)

Where \( N \) is the number of primary particles composing the aggregate particle and \( d_{pl} \) is the primary particle diameter. The dynamic shape factor \( x \) can be obtained approximately, within the range where \( N \) is small, using the following equation:

\[ x = 0.862 N^{1/3} \quad (2 \leq N \leq 10) \]  

(14)

With aggregates composed of a large number \( N \) of primary particles, \( dv_B \) is large and therefore the dispersion force \( F_d \) of Equation (12) the particles are more easily dispersed. With doublets consisting of two identical spheres, \( x_A = x_B = 1 \), \( Dv = dv_A/dv_B = 1 \) and the dispersion force of Equation (12) becomes \( F_d = 0 \). This means that the dispersion is not initiated by acceleration. Therefore, attention was focused on triplets composed of 3 primary particles and their dispersion force and adhesion force between particles were considered. Figure 7 shows the dispersion force \( F_d \) (solid curve in the Figure) obtained using Equation (12) when triplets disperse into one primary particle and one doublet. \( P_1 \) in the Figure is the air pressure supplied by an ejector mentioned later, \( \rho \) is the density of air which has reached critical pressure at the throat section of the ejector and \( \nu_r \) is the flow velocity (critical velocity) at the throat section. On the other hand, the dashed line in the Figure indicates van der Waals force as the adhesion force \( F_a \) between the particles when dry PSL (polystyrene latex) particles are used. This \( F_a \) becomes larger in proportion to the number of contact points between the primary particles in separation (namely \( F_{a1} \), \( F_{a2} \) in the Figure).

By comparing the adhesion force between the particles in this Figure with the dispersion force, for particles of primary particle diameter of 5 \( \mu \)m and 2 \( \mu \)m can be estimated that almost all particles will be dispersed under the pressure of \( P_1 = 1 \) MPa and 5 MPa respectively. The alternate long and short dash line in the Figure indicates the liquid bridge adhesion force obtained from the previous Fig. 4(a) and this will be explained later.

3.2 Experimental Results

Aggregate particles composed of freeze-dried monodispersed PSL particles were dispersed by an ejector (made by Nisshin Flour Milling Co., Ltd.) shown in Fig. 8, allowed to settle on a slide glass placed at the bottom of the sampling chamber shown in Fig. 8 by gravitational settling, then observed through an optical microscope as shown in Fig. 9. The results are given in Fig. 10. The ordinate in Fig. 10 shows the ratio of the number of aggregate particles consisting of \( N \) primary particles (including \( N = 1 \)) to the total number of aggregates and primary particles after dispersion. \( P_1 \) denotes the pressure of nitrogen gas supplied to the ejector. In Fig. 10, the results predicted in Fig. 7 were obtained, such
as doublets do not disperse much, and with an increase in nitrogen gas pressure or when the primary particle diameter becomes large, dispersion readily takes place. In this way, by raising the critical pressure of the air stream and increasing the gas density, it became possible to disperse aggregate particles composed of submicron particles with diameters of around 1 \( \mu m \) in air which was believed to be difficult in the past.

Another method to improve the dispersion process is shown in the following\textsuperscript{25} . Submicron particles (PSL particles) to be dispersed were mixed with larger dispersing media (glass beads with a diameter of about 100 \( \mu m \)) so as the PSL particles to coat on the glass bead surfaces, and then they were fed to the ejector shown in Fig. 8. As shown in Fig. 11(a), the PSL particles were attached thinly to the surface of the glass beads prior to dispersion. As shown in Fig. 11(b), the PSL particles were separated from the glass beads which have passed through the ejector. Figure 12 shows the experimental results. With PSL particles having a particle diameter of about 0.5 \( \mu m \), singlets (primary particles) were obtained of 85\% of the particles. As expressed by Equation (12), when the particle diameter \( d_{\text{P}} \) of particles \( P \), and the particle density \( \rho_{\text{P}} \) become

![Fig. 8 Experimental set-up for dispersion of aggregate particles](image1)

![Fig. 9 Photomicrograph of dispersed PSL particles](image2)

![Fig. 10 Experimental results of dispersion of dry PSL aggregate particles](image3)
large, dispersion force $F_d$ increases. This method utilizes this fact. The mixture of PSL particles and 100 μm glass beads has good flowability and therefore a stable feed is possible. Since the terminal settling velocity of 100 μm glass beads is high (0.5 m/s), separation of beads from PSL particles is easy after passing through the ejector.

3.3 Evaluation of Adhesion Forces between Particles in Air
The above concerns the dispersion of particles coagulated by van der Waal's force but when particles coagulate together due to the liquid bridge adhesion force, the adhesion force between particles becomes stronger by about one order of magnitude than the van der Waals force as shown by the alternate long and short dash line in Fig. 7 and therefore it becomes difficult for aggregate particles to disperse. Figure 13 shows the experimental results. The experimental setup is the same as that of Fig. 8 and the feed gas pressure $P_1$ to the ejector is 10 MPa. The ordinate of Fig. 13 is the same as that

Fig. 11 Photomicrographs of glass beads ($d_{p2} \approx 100 \mu m$) coated with PSL particles ($d_{p1} = 0.493 \mu m$) before and after dispersion

Fig. 12 Experimental results of dispersion of PSL aggregate particles ($d_{p1} = 0.493 \mu m$)
of Fig. 9. Since the dispersion force applied to the aggregate particles is the same, the wet aggregate particles which form the liquid bridge do not disperse so much compared with dry aggregate particles. In this way, when the adhesion force between particles becomes strong, it becomes difficult for particles to disperse but depending on the magnitude of the dispersion force.

**Figure 14** is a plot of the dispersion results obtained using an ejector, of PSL aggregate particles for both adhesion forces between particles of van der Waal force and liquid bridge adhesion force. The abscissa of the figure represents the percentage of singlets against the total number of aggregates and singlets after dispersion while the ordinate of the figure represents the ratio of the dispersion force to the adhesion force between particles when triplets are dispersed. The number of contact points $n_k$ of primary particles in separation of triplets is one or two but the adhesion force value between particles in the figure was assumed to be $n_k = 1$. The $\bigcirc$ mark in the figure shows the case of van der Waal’s force while the $\Delta$ mark shows the case of liquid bridge adhesion force which correlate well with the ratio of the dispersion force to the adhesion force between particles. In this figure, the slope of the line has a smaller gradient up to a point where the line bends and the value of the ordinate is unity and this shows that when the dispersion force becomes smaller than the adhesion force between particles, particle dispersion becomes difficult. If a dispersion experiment is conducted using an ejector with a known dispersion force applicable to aggregate particles and the results are plotted as shown in **Fig. 14**, then the adhesion force between the primary particles which compose the aggregate can be roughly estimated.
4. Conclusion

The adhesion among submicron particles changes the behavior of aggregates or powder beds and is a cause of trouble in industrial particle processing. The adhesion force arising from a liquid bridge changes depending on the humidity of the surrounding in which the particles are placed. If trouble should occur in processing of particles, it is thought that some of the problems can be solved by considering the cause following the line of thought described here. Demands to handle fine particles as single particles are increasing in industry and the dispersion of aggregates has also become an important issue. It is expected that dispersion phenomena may be further clarified by conducting studies focusing on individual particles.

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