Observation of liquid infiltration process into closed-end holes by droplet train impingement

Toshiyuki SANADA*, Yuki FURUYA*, Shunsuke MURAKI* and Masao WATANABE**
* Department of Mechanical Engineering, Shizuoka University
3-5-1 Johoku, Naka-ku, Hamamatsu, Shizuoka 432-8561, Japan
E-mail: sanada.toshiyuki@shizuoka.ac.jp
** Division of Space and Mechanical Engineering, Hokkaido University
Kita 13, Nishi 8, Kita-ku, Sapporo, Hokkaido 060-8628, Japan

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Abstract
Wet cleaning methods using liquids are widely applied in many industrial fields. In such methods, it is first necessary to cover the object to be cleaned with the liquid. However, in structures with small holes, surface tension prevents the deformation of the gas–liquid interface, making it difficult to fill the object with the liquid. We have found that liquid infiltration into such small holes is promoted by the impingement of droplet trains, but the underlying mechanism has not yet been elucidated. In this study, we observed this liquid infiltration process through droplet train impingement into a closed-end hole, and compared the liquid column impact. The filling process was visualized with two high-speed video cameras. Our observations illustrate the importance of the oscillation and deformation of the gas–liquid interface inside the holes following droplet impingement. First, intermittent droplet impingement causes small droplets or large interface deformations to form, and then the gas column inside the hole becomes separated. This separated gas column is then gradually ejected. Therefore, the liquid infiltration can be increased by using a droplet train formed of a small-surface-tension liquid. Furthermore, we investigated the influence of the hole diameter and the uniformity of the droplet train frequency. The results show that droplet train impingement is effective for relatively large holes, although the uniformity of the droplet train frequency has little effect on the liquid infiltration.

Keywords: Cleaning, Liquid infiltration, Surface tension, Gas–liquid interface, Droplet impingement, Oscillation, Droplet formation

1. Introduction

The use of liquids for cleaning (so-called “wet cleaning”) is widespread in a range of manufacturing processes. There are many advantages to wet cleaning, such as the removal of part of the surface through the chemical action of etching, preventing the redisposition of impurities by controlling the pH or adding surfactant, and reducing the adhesion force of particles owing to capillary condensation (Kohli & Mittal, 2005). In such wet cleaning, the surface should be covered by the liquid prior to cleaning. However, in some cases, it is not easy to fully cover fine structures with the liquid (Spuller and Hess, 2003). This could be because surface tension prevents the interface from deforming as necessary (De Gennes et al., 2004), the material to be cleaned has poor wettability, or dead-end structures do not allow gas to escape. For these reasons, a number of trials have investigated gas dissolution (Olim, 1997), the use of supercritical fluids (Ota and Tutsumi, 2008), and the wettability and influence of dissolved gas (Ota and Tutsumi, 2007).

In previous work, we experimentally investigated the process of a liquid entering a micro tube that is sealed at the tip in a pressure vessel. We showed that liquid infiltration into a relatively large tube (about 1 mm in diameter) through pressurization was difficult, and the aspect ratio was important (Sanada et al., 2016). We also showed that the influence of liquid dissolution was limited, even when high pressures were applied. Furthermore, we prepared a plate with 1 mm holes and sealed tips, and showed that the liquid infiltration could be promoted by the impingement of a droplet train at
atmospheric pressure (Yamaguchi and Sanada, 2017). However, the underlying mechanism of this infiltration has not yet been clarified.

In this study, we used two high-speed cameras to visualize the liquid infiltration process as a droplet train impinged into closed-end holes. First, we investigated the effect of pressure for dissolving gas into liquid over different durations, and then observed the impingement of a droplet train or liquid column onto a plate with a series of holes. In addition, we investigated the influence of the liquid surface tension, hole size, and uniformity of the droplet train frequency on the liquid infiltration.

2. Nomenclature

| Symbol | Definition | Unit |
|--------|------------|------|
| $d_h$ | hole diameter | [mm] |
| $h_0$ | hole depth | [mm] |
| $h_w$ | liquid infiltration rate | [-] |
| $t$ | time | [s] |
| $u$ | impact velocity | [m/s] |
| $d$ | nozzle diameter | [mm] |
| $h$ | liquid infiltration height | [mm] |
| $L$ | distance between nozzle and plate | [mm] |
| $p$ | pressure | [kPa] |

3. Experimental setup, method, and conditions

First, to investigate the influence of gas dissolution, the liquid infiltration owing to liquid pressurization was tested using a pressure vessel, similar to the experiments of Sanada et al. (2016). A schematic diagram of the experimental apparatus used in this experiment is shown in Fig. 1(a). A test sample was fixed at the bottom of a water-filled container, and the entire experimental system was pressurized using a hand pump. The pressure change inside the chamber was measured with a pressure gauge (Keyence, AP-13S) and recorded (HIOKI, Memory Hi-coder 8870). The liquid filling process was observed through a high-speed camera (Vision Research, Phantom M 310) and a microscope (Leica, Z 16 APO).

The liquid infiltration due to jet impingement was then investigated. A schematic diagram of the experimental apparatus is shown in Fig. 1(b). In this experiment, liquid in a droplet train or column state was irradiated onto a sample containing microholes, as shown in Fig. 1(c). The liquid was injected from the pressure vessel with an air compressor using two kinds of micro-tube as a nozzle (polyether ether ketone tube and glass tube). The liquid behavior due to jet impingement inside the microholes was observed with a high-speed video camera (Photron, FASTCAM Mini WX 100). The droplet diameter and velocity were measured with another high-speed video camera (Vision Research, Phantom M310). In an experiment to control the droplet frequency, an audio speaker was attached to the nozzle as shown in Fig. 1(d). Sinusoidal waves with the same averaged frequency as the droplet train were used to actuate the speaker with a function generator (NF Corporation, WF 1974) and an amplifier (Showa Instruments, Model 4035-52).

Two kinds of pressure vessel experiments were performed. The first was a gradual pressurization experiment, in which the entire system was gradually pressurized from 0–700 kPa in increments of 100 kPa gauge pressure before the pressure was released. Each pressurize–release cycle took about 20 min, and was repeated three times for each experiment. The second was an instantaneous pressurization experiment in which pressurization from 0–700 kPa and subsequent depressurization took 10 s. This was also repeated three times. The frame rate of the high-speed video camera was 200 fps. In all experiments, the liquid infiltration depth $h$ of each pressure was measured and evaluated with the liquid infiltration rate $h_w = h / h_0$, where $h_0$ is the hole depth.

In the experiments investigating liquid filling by the impingement of a droplet train or liquid column, the rate $h_w$ was used to evaluate the liquid infiltration. We measured $h_w$ after 10 min irradiation. The recording rate of the high-speed video cameras was set to 5000 fps. To eliminate the influence of the initial impact, the hole was not initially positioned under the nozzle and the test sample was moved from 15 mm away to the nozzle bottom position at a speed of 15 mm/s.

Three nozzle inner diameters were used, $d = 0.34, 0.75$, and $1.0$ mm. The impingement velocity $u$ of the liquid column and droplet train varied from $1.0$ to $5.0$ m/s. The droplet diameter was randomly selected and the average value calculated. The droplet diameter was approximately 1.88 times the nozzle diameter $d$, similar to previous studies (Suzuki et al., 2015, Muraki et al., 2017). In the case of the liquid column, the mass of liquid ejected from the nozzle...
was sampled for 10 s, and the flow rate and averaged velocity were obtained. The mass measurements were repeated three times. The distance between the nozzle and the sample was changed to control the liquid column or droplet train. The distance $L$ for the liquid column was 10 mm and that for droplet train impact was 70 mm (water) or 90 mm (ethanol). The experiments were conducted for liquid temperatures of 18.5–26.2 °C. Further details are given in Muraki et al. (2017).

![Figure 1](image)

**Fig. 1** Experimental setup. (a) Setup for liquid infiltration experiments by applying pressure. Test samples were fixed in the pressure vessel, which was pressurized by a hand pump. Liquid infiltration process was visualized using a high-speed video camera with microscope. (b) Setup for droplet train impingement experiments. Liquid infiltration process was visualized using two high-speed video cameras. (c) Test sample. Three holes were drilled in a plate and the bottom was sealed with silicone rubber sheet. (d) Frequency control for the droplet train. In the droplet frequency control experiments, vertical oscillation was applied to the droplet generation nozzle using a speaker.

### 4. Results and discussion

#### 4.1 Liquid infiltration by applying pressure

Figure 2 shows the relationship between applied pressure $p$ and liquid infiltration rate $h_w$ in the case with hole inner diameters $d_h = 1$ mm and 0.1 mm. The solid line in the figure is the estimated value from the equation of state of the ideal gas in the isothermal process. As shown in Fig. 2(a), in the case of $d_h = 1$ mm, the predicted value indicated by the solid line and the experimentally obtained liquid infiltration rate were in good agreement. The difference from the
theoretical result corresponds to the gas dissolution into the liquid. In this case, about 25% of the liquid was filled by gas dissolution after three cycles of pressurization. When \( d_h = 0.1 \) mm, complete liquid infiltration was achieved on the first pressurization. These results are the same as those reported by Sanada et al. (2016). In a tube of about 1 mm diameter, liquid infiltration cannot be achieved despite high pressure. For the smaller-diameter tube, the gas dissolves into the liquid at high pressure and the liquid fills the tube.

![Figure 2: Liquid infiltration height \( h_w \) by pressurization.](image)

Next, we discuss the influence of liquid dissolution for short-time pressurization and its repetition. Figure 3 shows the relationship between time \( t \) and liquid infiltration rate \( h_w \). Applied pressure \( p \) is also shown. The broken line is the predicted value of \( h_w \) estimated from the equation of state of the ideal gas, as in Fig. 2. As shown in Fig. 3, good agreement with the predicted value was achieved with both \( d_h = 1 \) mm and \( d_h = 0.1 \) mm. The dimensionless gas dissolution, obtained by dividing each gas dissolution amount by the hole volume, was 0.014 for \( d_h = 1 \) mm and 0.11 for \( d_h = 0.1 \) mm. This result indicates that, in the present experiment, the movement of the gas–liquid interface accompanying the pressurization and depressurization operation produced relatively little liquid infiltration through gas dissolution. Thus, the non-negligible infiltration owing to gas dissolution seems to occur with small holes and long-time pressurization.

![Figure 3: Liquid infiltration during instantaneous pressurization process.](image)
The above results indicate that the effect of gas dissolution in the holes considered in this study is limited to pressures of several hundred kPa. As discussed in the next section, the pressure generated by droplet impingement is estimated to be several kPa (Yamaguti and Sanada, 2016). Therefore, in this paper, we ignore the effect of gas dissolution into the liquid under intermittent pressures of several kPa occurring over the short time scales of droplet train impingement.

4.2 Liquid infiltration rate by impingement of droplet train

First, we discuss the influence of the droplet (or liquid jet) impingement velocity on the liquid infiltration rate \( h_w \) into \( d_h = 1 \) mm holes. The relationship between impingement velocity \( u \) and \( h_w \) is shown in Fig. 4. The liquid infiltration rate \( h_w \) increases with an increase in impingement velocity for both nozzle diameters. Higher values of \( h_w \) were achieved through the impingement of the droplet train than the liquid column over the whole velocity domain. In addition, as shown in Fig. 5, the interface behavior inside the hole differs greatly for the droplet train and the liquid column impingement. For the droplets, the air column inside the hole oscillates while repeatedly compressing and expanding, and the interface becomes deformed. These features were not observed during the collision of the liquid column, and the interface remained relatively unperturbed during the impingement.

![Fig. 4](image-url) Liquid infiltration rate \( h_w \) after 20 min impingement by droplet train or liquid column. Droplet train impingement produces higher liquid \( h_w \) than the liquid column.

![Fig. 5](image-url) Gas column behavior inside the holes during impingement. (a) Droplet train, (b) liquid column. Image height is 10 mm. White parts indicate the liquid. Bubble oscillation can be observed during droplet train impingement.
In this experiment, the increase in $h_w$ through the impingement of droplets occurred with discontinuous gas discharge, and $h_w$ did not continuously increase. An example of the gas discharge process is shown in Fig. 6. First, some of the liquid intruded into the air column (Stage I). This process repeated, then the liquid part started necking, as shown by the red line. The necking portion of the liquid gradually grew as the liquid repeatedly intruded into the air column (Stage II). Eventually, the air column became separated from the necking portion (Stage III). The separated column of air gradually rose while repeatedly oscillating, and the air column finally discharged (Stage IV). Thus, to obtain a high liquid infiltration rate, it is important to generate the air column breakup from Stage II to Stage III. Therefore, we focus on the air column breakup process in the rest of this section. Note that almost no air column breakup was observed when the liquid column collided with the gas in the holes.

Two patterns of gas column separation were observed in this experiment. The first was caused by the generation of droplets, as shown in Fig. 7(a). In this column separation, the gas–liquid interface oscillated in the vertical direction because of the volumetric oscillation due to the impulse of the impingement of droplets. A droplet was then generated from the oscillating gas–liquid interface in the hole. The droplet intruded into the gas column, as shown by the red circle, and adhered to the wall of the hole. This process was repeated, and air column separation occurred once the necking portion had grown sufficiently. Hereinafter, this air column separation is referred to as Pattern 1. In the case of liquid column collision, Pattern 1 was barely discernible, whereas for droplet train impingement, it was observed under all conditions. During liquid column collision, pressure was applied continuously, but the droplet train collision caused the pressure application and release to be repeated. We consider this intermittent pressure application to be important. The volume of the air column thus oscillates continuously, unlike the case of the liquid column collision, and the droplet formation is a factor in obtaining the high liquid infiltration rate given by the collision of droplets.

The second pattern of air column separation is shown in Fig. 7(b). In this separation, the gas–liquid interface is greatly deformed by the impingement of droplets that directly intruded into along the wall of the hole, and a necking portion forms. Hereinafter, this air column separation is referred to as Pattern 2. Pattern 2 is observed at relatively high impingement velocities and at the top region of the hole. We consider that the liquid inertia to increase because of the increase in collision speed, and then the interface becomes greatly deformed. Therefore, as shown in Fig. 4, the liquid infiltration rate and droplet collision velocity are correlated, because the air column separation in both Pattern 1 and Pattern 2 occurs in the domain of high impingement velocity.

Note that the liquid column collision led to some infiltration, although very little air column separation occurred. This is because of the jet collision method used in the experiment. As described in the previous section, the test sample was moved into position for the liquid jet or droplet train to impinge on the holes. In all experiments, a liquid film formed on the sample and the droplets did not directly impact on the dry solid surface. However, some of the gas inside the holes was discharged at a very early stage, before reaching the jet collision site, and so a small $h_w$ was observed in the case of liquid column collision. This liquid infiltration was observed in both the droplet train and the liquid column collision. In addition, from Fig. 4, $h_w$ when $d_b = 0.75$ mm is greater than when $d_b = 1.0$ mm at high liquid column collision speeds. As the diameter of the liquid column was almost the same as the nozzle inner diameter and less than the inner diameter of the sample holes, the gas inside the hole may have been easily discharged by a small-size liquid column collision.

![Fig. 6 Gas discharge process. First, some of the liquid entered the gas column (Stage I). The process of Stage I repeated, then the liquid started necking (Stage II). The gas column separated from the necking point (Stage III). With the oscillation caused by droplet impact, the upper part of the gas column was discharged (Stage IV).](image-url)
4.3 Influence on surface tension for liquid infiltration rate

We now discuss the influence of surface tension on the liquid infiltration. Figure 8 shows the relationship between impact velocity $u$ and liquid infiltration rate $h_w$ for both water and ethanol. In this case, a test sample with an inner diameter $d_h = 1.0$ mm and a nozzle of inner diameter $d = 0.75$ mm were used. The ethanol droplet train produced higher $h_w$ values than the liquid column, and a similar situation was observed for water. In addition, the $h_w$ values given by ethanol droplets were higher than those for water at all collision velocities.

To investigate the difference in the liquid infiltration process, the $h_w$ values 1 s after collision for both water and ethanol are shown in Fig. 9(a). From this figure, we see that ethanol has a high $h_w$, even just after the droplet collision. If we focus on the results for $u = 4.5$ m/s, complete liquid infiltration was achieved with ethanol, but only about 50% $h_w$ was observed with water. As shown in Fig. 8, perfect liquid infiltration was achieved with both ethanol and water after 10 min irradiation. These results indicate that the ethanol can quickly fill holes. Figure 9(b) shows a continuous photograph of the liquid behavior observed in the holes when using ethanol. Multiple droplets are generated, as indicated by the red circles in the figure. We believe that this is because ethanol has a lower surface tension and it is easier to atomize liquid droplets through the inertial force generated by the movement of ambient liquid accompanying air column oscillations. The increased droplet formation enhances the air column separation, and the liquid infiltration rate may increase. Typical capillary wetting phenomena include capillary rise, whereby large surface tension liquids have a greater driving force, but the opposite result has been obtained here.
4.4 Influence on hole size for liquid infiltration rate

The results discussed above relate to a sample hole inner diameter $d_h = 1.0$ mm with 10 min irradiation. In this section, smaller hole diameters of $d_h = 0.3$ mm and 0.1 mm are discussed. When the inner hole diameter becomes smaller, $h_w$ exhibits different temporal behavior. Figure 10 shows the temporal transition of $h_w$ for $d_h = 1.0$ mm and 0.1 mm with nozzle diameter $d = 0.75$ mm and impact velocity $u = 4.5$ m/s. Basically, $h_w$ increases with time when the inner hole diameter is large. However, $h_w$ increases and decreases repeatedly in the case $d_h = 0.1$ mm. We believe that this is because entrained bubbles form when the droplet impinges on the liquid film, and these bubbles enter into the holes. Even when $d_h = 1.0$ mm and 0.3 mm, entrained air bubbles were observed, but gas discharge was the dominant factor in the temporal change of $h_w$. However, in the case of $d_h = 0.1$ mm, the original gas column has a small volume and the influence of the entrained air bubbles on $h_w$ becomes large, leading to $h_w$ increasing and decreasing repeatedly. Therefore, for $d_h = 0.1$ mm, $h_w$ was measured every 30 s for 10 min irradiation, and the maximum value was evaluated as the liquid infiltration rate $h_w$. 

Fig. 8 Effect of surface tension for the liquid infiltration rate $h_w$. Ethanol droplets, which have lower surface tension than water, enhanced the liquid infiltration.

Fig. 9 Effect of surface tension on liquid infiltration. (a) Liquid infiltration rate $h_w$ 1 s after irradiation, (b) Formation of multiple droplets once the gas–liquid interface began to oscillate.
Figure 11 shows the relationship between the liquid infiltration rate $h_w$ and the impact velocity $u$ for samples with different inner diameters. A nozzle of $d = 0.75$ mm was used. For small-hole samples, it was difficult to visualize the bottom part, and approximately 15% of the hole bottom could not be observed in the case of $d_h = 0.1$ mm. Therefore, $h_w$ was calculated assuming no liquid filling in the bottom 15%. There is a possibility that liquid infiltrated this 15% region, and so the error bars are set in the positive direction of $h_w$.

The collision of the liquid column produced no significant difference in $h_w$ for $d_h = 1.0$ mm and 0.3 mm (Fig. 11(a)). In contrast, the collision of the droplet train caused $h_w$ to decrease as the hole diameter became smaller (Fig. 11(b)). We consider this to be because the amplitude of the interface oscillation decreases as the hole diameter becomes smaller, making droplet generation less likely to occur. These results and the gas dissolution experiments suggest that gas dissolution is more efficient than the collision of droplets for small-hole liquid infiltration, because small holes are affected by entrained bubbles.

![Graph showing the relationship between liquid infiltration rate and impact velocity](image)

Fig. 10 Effect of hole size on liquid infiltration rate $h_w$ under droplet train impingement.

![Graphs showing the relationship between liquid infiltration rate and impact velocity](image)

Fig. 11 Effect of hole size on liquid infiltration. (a) Liquid column, (b) Droplet train impingement. Droplet train impingement enhances the liquid infiltration, but the enhancement effects decrease as the hole size becomes smaller.

### 4.5 Influence on uniformity of droplet train frequency for liquid infiltration rate

In the case of liquid infiltration by droplet train impingement, it is important to generate volumetric oscillations of the air column by the intermittent application of pressure. Finally, we discuss the influence of the frequency of pressure application, i.e., the generation frequency of the droplet train. A speaker was attached to the nozzle to control the
frequency of the droplet train. Figure 12(a) shows the relationship between the collision speed $u$ and the liquid infiltration rate $h_w$ for both a uniform droplet train, in which the droplet diameter and velocity are controlled, and a non-uniform droplet train. Figure 12(b) shows example images of the droplet train with and without this control process. The droplet diameter and velocity are made uniform by applying vibration to the nozzle. In these experiments, the inner diameter of the sample hole was $d_h = 0.3$ mm and the inner diameter of the nozzle was $d = 0.314$ mm. A nozzle with a smaller inner diameter was used because the diameter of the generated droplet was large in the case of $d = 0.75$ mm and 1.0 mm, and the extremely small controllable range makes control very difficult. From the figure, it can be seen that, when the droplet diameter and velocity are uniform, the liquid infiltration rate tends to be slightly lower.

Examining the inside of the hole during the collision, we can observe a slight difference in the oscillation of the interface between the uniform and non-uniform droplet trains. In the case of the uniform droplet train, the interfacial oscillation had a smaller amplitude. No significant difference was observed in the appearance of the air column when $u = 5.0$ m/s, but there was a clear difference during the collision when $u = 3.5$ m/s. A photograph is shown in Fig. 13. In the general droplet train, the air column inside the hole separates into multiple gas columns, whereas the uniform droplet train causes the air column to separate only once, and only the gas around the top of the hole is discharged. The liquid infiltration using the droplet train suggests that the intermittent application of pressure and the collision cycle and timing of the droplet are important factors. In surface cleaning techniques using droplet collision, there are cases where the diameter and speed of the droplet are controlled to equalize the physical action. However, this result suggests that the uniformity of the droplet train is another important factor in the liquid filling rate.

![Fig. 12](image1.png)  
**(a)** Non-uniform droplet train  
**(b)** Uniform droplet train

**Fig. 12** Effect of uniformity of droplet frequency on the liquid infiltration rate $h_w$. (a) Relation between $h_w$ and impact velocity $u$. (b) Droplet train before impingement (left: without control, right: with control). Non-uniform droplet train enhances the liquid infiltration.

![Fig. 13](image2.png)  
**(a)** Non-uniform droplet train  
**(b)** Uniform droplet train

**Fig. 13** Gas column breakup during droplet train impingement. (a) Non-uniform droplet train, (b) uniform droplet train. Multiple gas columns appear during non-uniform droplet train impingement.
5. Conclusion

We experimentally investigated the liquid filling process of closed-end holes through the impingement of droplet trains. First, we evaluated the effect of gas dissolution for liquid filling. Over an experimental duration of 20 min, the gas dissolution was limited, even with high external pressures, especially for relatively large tubes of 1.0 mm diameter. For small tubes, the gas did not dissolve into the liquid under instantaneous pressurization over several seconds. However, perfect liquid infiltration was achieved under the 20 min pressurization process because of gas dissolution.

Second, we visualized the liquid infiltration process due to droplet impingement. High-speed photography clarified the liquid filling process. The gas–liquid interface inside the tube was observed to oscillate under droplet impingement, and droplets formed from the interface and attached to the tube wall. In the high-speed droplet case, the interface was directly deformed and attached to the tube wall. These processes repeated and the attached liquid volume increased. The liquid then connected across the cross-section of the tube and the gas column separated into two distinct gas regions. The separated gas column oscillated with droplet impingement and was finally discharged. The droplet formation and gas–liquid interface deformation are more likely to occur with small-surface-tension liquids, and so the liquid infiltration rate increased when ethanol was used instead of water. In all experiments, the impingement of a droplet train produced higher liquid infiltration rates than the application of a liquid column.

Furthermore, the influence of hole size and uniformity of the droplet train frequency were investigated. With small-size tubes, the liquid infiltration rate initially increased under the impingement of the droplet train, but in some cases the rate decreased over time because of the entrapment of bubbles. In addition, the infiltration rate decreased when the frequency of the droplet train was controlled. These results indicate that droplet train impingement without frequency control is effective for liquid filling into closed-end holes of millimeter size. However, gas dissolution under pressurization is more useful for smaller-size tubes.

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