Effect of Operating Conditions on the Sessile Drop Oscillation of PEO Solutions in the Capillary Thinning Process

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(Received : November 30, 2020)

In this study, we investigate the sessile drop oscillation of aqueous PEO solutions after the transition from visco-capillary to elasto-capillary regime in the capillary thinning process of low-viscosity elastic fluids. Among various factors that affect oscillation, we focus on the effect of operating conditions that induce capillary thinning. The lifting velocity to lift the upper plate and the plate diameter to load the sample are selectively controlled. The generated oscillation is quantitatively analyzed in terms of oscillation frequency and decay ratio. The oscillation frequency and oscillation decay ratio are found to be independent of the lifting velocity. The oscillation frequency of all samples is about 140 Hz independent of the lifting velocity, and the decay ratio is not affected by the lifting velocity. On the contrary, the plate diameter affects the oscillation. The oscillation frequency increases to 210 Hz and the decay ratio shows a larger value (close to one) when a smaller plate is used. This study opens a way to improve the measurement of extensional properties which suffers from oscillation.

Key Words: Capillary thinning / Capillary breakup / Oscillation / Aqueous PEO solution

1. INTRODUCTION

Understanding the extensional properties of polymer solution is important in industrial processes such as coating,1-3) mixing,4) spraying1), spinning5), inkjet printing6), to list a few. The importance of extensional flow in these processes cannot be too much emphasized. Extensional flow is a strong flow and does not include rotation. In the extensional flow, the polymer chain is arranged in the flow direction and the information about the extensional property can be obtained through the flow-induced array of the polymer chain. One of the methods to characterize the extensional behavior of the fluid is capillary breakup extensional rheometer (CaBER)7,8). Capillary thinning is a phenomenon in which the liquid forms bridges when an extensional deformation is applied as the fluid is gradually thinned by the capillarity of the substance over time. It is dominated by the interplay of capillary, viscous, inertial and elastic forces. A well-known experimental technique that induces capillary thinning is to load a small amount of sample between two circular plates and then lift the upper plate to apply extensional deformation to the sample. The dynamics of capillary thinning process induced by the extensional deformation is determined by the balance between the capillary pressure to break up the liquid bridge formed between two circular plates and the inertia, viscous and elastic stress which resist capillarity. Capillary thinning of a Newtonian fluid shows a parabolic free surface as the elastic stress does not exist. However, the capillary thinning of a polymer solution forms a uniform filament due to the elastic stress generated from the stretched polymer chain. Various extensional properties such as extensional viscosity and relaxation time can be calculated through the change of filament diameter over time9,10).

Various instabilities and disturbances that occur during the thinning process of polymer solutions and particulate suspensions interfere with the formation of uniform surface and cause errors in extensional property measurement. Most typical instability is blistering, known as beads-on-a-string (BOAS), in which successive beads are formed in filament triggering free surface perturbation11-13). Blistering is the final part of the polymeric filament thinning where the polymer chain is fully stretched. The experimental observation of the blistering pattern was reported in detail by Oliveira et al.11). They visualized the thinning process of polymer solution by high-speed camera, and observed the formation and coalescence of iterated beads. Since then, Sattler et al.12,14) classified the formation of beads into four classes according to the position where the bead begins, and found that the bead

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formation occurs independently of solvent viscosity. In addition, they observed a breathing phenomenon in which the uniformity of the connection site between the filament and the upper and lower sessile drop is broken before the blistering pattern occurs. This is the instability that occurs in the early stage of polymeric filament thinning process. In connection with this, Ock et al. conducted a study on the breathing phenomenon and bead formation that occurs during capillary thinning with particulate suspension. They observed an acceleration of the transition from elasto-capillary regime to the blistering regime when the particles are added to the polymer solution. Furthermore, it was reported that the first bead formation is due to the local particle-concentration by visualizing the inside of the filament. The various instabilities that occur during capillary thinning process are the hurdles that make it difficult to precisely measure the extensional property.

Another factor that causes a significant error in extensional property measurement is the oscillation that occurs throughout the sessile drop and filament. It has been reported that the oscillation occurs at the transition from visco-capillary to elasto-capillary regime in the thinning process of the low-viscous elastic fluid containing polymers. However, no research has been conducted on the factors and conditions that affect the oscillation so far. In the case of a Newtonian fluid, oscillation does not occur because it does not experience the transition during the capillary thinning process. But in the case of polymer solution, oscillation occurs during the transition process. Such an oscillation can also be observed in dripping-onto-substrate (DoS) rheometry, which loads a certain amount of sample on a substrate in the form of drop and induces capillary thinning. As with the instability such as blistering and breathing, the oscillation interferes uniform filament formation and causes difficulty in filament diameter measurement. Therefore, it is essential to analyze the various factors that affect the oscillation and to identify the mechanism of the oscillation for accurate measurement of extensional properties.

In this paper, we focus on the effect of operating conditions on the sessile drop oscillation that occurs during the transition from visco-capillary to elasto-capillary regime in the capillary thinning process of polymer solution. The relationship between the property of the sample and oscillation is also important, but prior to that, the relationship between the operating condition and oscillation should be investigated to screen the key factors. Among the operating conditions, the lifting velocity and plate diameter are changed. PEO with three kinds of molecular weight is used for reliability. From the height of the sessile drop over time, the frequency and decay ratio of the oscillation are analyzed.

2. EXPERIMENTS

2.1 Materials

Aqueous poly(ethylene oxide) (PEO, Sigma-Aldrich, Mw = 200,000 g/mol, 1,000,000 g/mol, 2,000,000 g/mol) solutions with three molecular weights were used. For a convenience, PEO solutions with the molecular weight of 200,000 g/mol, 1,000,000 g/mol, and 2,000,000 g/mol were named as 0.2 m PEO, 1.0 m PEO, and 2.0 m PEO, respectively. To eliminate the effect of difference in viscosity and elasticity, PEO solutions with three different molecular weights were prepared with five concentrations each, and they were adjusted to match the viscosity. In this way, only the effect of operating condition could be investigated. The viscosity of the PEO solution was measured using AR-G2 (TA instruments, USA), which is a stress-controlled rheometer. A 60 mm parallel plate was used and the temperature was kept at 25 °C. The extensional relaxation time (λe) of the PEO solution was measured through a change of the filament diameter over time. The filament diameter was calculated from the image taken during the capillary thinning process of the sample.

2.2 Experimental setup

In order to induce the capillary thinning process, a universal testing machine (UTM; LF Plus, Lloyd instruments, Inc., UK) was modified. A small amount of sample was loaded between two circular plates and the upper plate was lifted to apply the extensional deformation. After applying the step extensional deformation as shown in Fig. 1 (a), capillary thinning process and sessile drop oscillation were recorded using a high-speed camera (Photron Fastcam Ultima 512, Photron, Inc., USA). Figure 1 (b) shows the observing area. The region marked by a red dot box is the observing area. The
surface change of sessile drop in the lower plate where oscillation is clearly observed was recorded using the high-magnification lens (x10). One pixel in the image covered 1.97 μm. The high-speed camera captured 2000 frames per second at resolution of 256 × 512 pixels. During the process, backlight illumination was provided by a 250 W halogen lamp (BMH-250, Mejiro Precision, Japan). More detailed UTM setup is described in the reference. The initial gap between the two plates was set to 1 mm. In order to keep the initial configuration of loaded sample close to cylindrical without gravitational sagging, the tests were conducted under the condition \( h_0 / R_0 \leq 1 / \sqrt{Bo} \) where \( h_0 \) is the initial gap, \( R_0 \) is the plate diameter, and \( Bo \) is the Bond number. As the initial gap is narrower than the capillary length \( l_{\text{capillary}} = \sqrt{\sigma / \rho g} \approx 2.32 \) mm, \( \sigma \): surface tension of the fluid, \( \rho \): density of the fluid, gravitational sagging is suppressed. In order to analyze the effect of operating conditions on the oscillation, the lifting velocity of the upper plate and the plate diameter were changed. The lifting velocity was varied at 0.5 mm/s, 1.0 mm/s, and 1.5 mm/s at fixed plate diameter of 3 mm. The plate diameter was varied at 1 mm and 3 mm at fixed lifting velocity of 1.0 mm/s.

2.3 Oscillation characterization

2.3.1 Image processing

The height change of the oscillation over time was tracked by analyzing the captured image of the surface of the sessile drop. Figure 2 (a) is an image in which the surface of the sessile drop is tracked over time through a custom MATLAB-based image processing code. It is the image sequence of 1.0 m PEO solution and filament thinning process is recorded after the transition to elasto-capillary regime. \( h \) is the height of the sessile drop, and \( h_{\text{inf}} \) is the height of the sessile drop after the filament is completely broken. Since the height of the sessile drop depends on the position being measured, the height of the sessile drop of all samples was measured 100 pixels away from the center of the filament. Although the height of the sessile drop was dependent on the measurement position, the frequency and decay ratio of oscillation were independent of the position being measured. Figure 2 (b) shows the change of calculated height over time. \( h - h_{\text{inf}} \) is used to unify the height criteria for all the samples.

2.3.2 Oscillation frequency (\( \omega \)) and decay ratio (\( \zeta \))

In order to analyze the oscillation of sessile drop quantitatively, the frequency and decay ratio of the oscillation were calculated. For each sample, the same experiment was repeated 10 times or more to guarantee reproducibility. The oscillation frequency (\( \omega \)) was calculated through the reciprocal of the period from Fig. 2 (c). Since the period between each peak was almost constant, the period up to the third oscillation was averaged to get the frequency. Figure 2 (d) indicates the way to calculate the oscillation decay ratio (\( \zeta \)). The oscillation decay ratio was defined as the ratio of the first peak difference (A) (the difference between the first maximum and the first minimum peaks) to the second peak difference (B) (the difference between the second maximum and the second minimum peaks). That is, the closer to zero, the stronger the damping. Similarly, the closer to one, the weaker the damping.

3. RESULTS AND DISCUSSION

3.1 Capillary thinning and oscillation

In order to investigate the effect of lifting velocity and plate diameter on the oscillation that occurs during the capillary thinning process, PEO with three different molecular weights was used. First, the shear viscosity and the extensional relaxation time of each polymer solution were measured. Figure 3 is the flow curve of the PEO solution for each molecular weight. In the case of 0.2 m PEO in Fig. 3 (a), the viscosity was independent of the shear rate at all concentrations. On the other hand, the 1.0 m PEO and 2.0 m PEO in Fig. 3 (b) and (c) showed weak shear thinning behavior. The
zero shear viscosity of each sample was matched so that the PEO with each molecular weight has the same viscosity in order to eliminate the effect of molecular weight.

Figure 4 (a) is a capillary thinning process of 0.45 wt% 2.0 m PEO and 0.89 wt% 2.0 m PEO. Both samples show typical capillary thinning behavior of the polymer solution. Regime ① of each sample is the visco-capillary thinning stage where the minimum neck diameter decreases linearly over time. In this stage, the stretching of the polymer does not occur and a parabolic-shaped surface is observed like the capillary thinning of Newtonian fluid. That is, the elasticity of the polymer does not affect the thinning dynamics in this regime. After the visco-capillary regime, it transits to regime ②, which is the elasto-capillary thinning stage. At this stage, the elastic stress which originates from the extension of the polymer chain balances against the capillary pressure to break the filament. Hence, the uniform filament is obtained and the diameter of the filament exponentially decays as shown in Eq. (1)\(^{20}\).

\[
\frac{d(D_0)}{dt} = \left(\frac{G}{\eta_0 \sigma} \right)^{1/3} \exp \left(-t/3\lambda_e\right)
\]

(1)

where \(G\) is the elastic modulus, \(\sigma\) is the surface tension of the fluid, \(\lambda_e\) is the extensional relaxation time, and \(D_0\) is the initial diameter of the exponentially decaying filament. The extensional relaxation time can be calculated through the above equation, and Fig. 4 (b) shows the relaxation time of all samples. The measurement of relaxation time was reproducible with little error. The relaxation time increased as the molecular weight and concentration of polymer solution increased. After the elasto-capillary regime, it transits to regime ③, which is the blistering stage. The instability of bead-on-a-string structure is observed. It should be noted that the deviation of the filament diameter occurs in the transition from regime ① (visco-capillary regime) to regime ② (elasto-capillary regime) in both samples of Fig. 4 (a). The transition point is where the elastic stress begins to get involved in the thinning dynamics. This point coincides with the time when the oscillation occurs in the upper and lower sessile drop of the plate. In other words, the deviation of the filament diameter is caused by the oscillation generated during the transition from visco-capillary to elasto-capillary regime.

### 3.2 Effect of lifting velocity on sessile drop oscillation

Among the various factors that affect the oscillation occurring in the transition of the capillary thinning, we first changed lifting velocity to Fig. out the effect of operating conditions. The lifting velocity was varied at 0.5 mm/s, 1.0 mm/s, and 1.5 mm/s with a fixed plate diameter of 3 mm. Figure 5 shows the oscillation frequency (\(\omega\)) of the PEO solutions with different lifting velocity. It is noteworthy that there was a sample condition in which the oscillation was not observed. In the case of 2.0 m PEO in Fig. 5 (c), the oscillation was observed for all \(\eta_0\) covered in this study and the oscillation frequency at each concentration could be measured. However, in the case of 0.2 m PEO and 1.0 m PEO, there was a concentration range where the oscillation was not observed and the oscillation frequency above the certain concentration could not be calculated. In Fig. 5 (a) and Fig. 5 (b), no oscillation was observed in the range of \(\eta_0\) above 40 cP and 60 cP, respectively. Given that there exists a certain viscosity range in which no oscillation occurs in polymer solution of the same molecular weight and that the occurrence of oscillation depends on molecular weight even under the same viscosity condition, the oscillation is affected by the viscosity as well as the molecular weight of the polymer solution. More research on the relationship between the properties of polymer solution and the oscillation could be conducted later.

There was no difference in the oscillation frequency of the three molecular weight PEO solutions according to the lifting velocity. The oscillation frequency has a constant value of about 140 Hz in all samples when oscillation occurs. This is because the lifting velocity cannot change the Rayleigh time scale (\(t_R\)), which is the characteristic time of the capillary thinning process. The Rayleigh time scale is defined as Eq. (2)\(^{16}\).

![Fig. 4](image.png)  
Fig. 4 (a) Evolution of the filament neck diameter at different PEO (2,000,000 g/mol) concentration of 0.45 wt% and 0.89 wt%, (b) extensional relaxation time (\(\lambda_e\)) as a function of zero shear viscosity (\(\eta_0\)).

![Fig. 5](image.png)  
Fig. 5 Oscillation frequency (\(\omega\)) with different lifting velocity: (a) 200,000 g/mol PEO, (b) 1,000,000 g/mol PEO, and (c) 2,000,000 g/mol PEO.
where \( \rho \) is the fluid density, \( R_0 \) is the characteristic length, and \( \sigma \) is the surface tension. The plate diameter is fixed at 3 mm. The effect of adding the polymer on the solution density could be ignored because the amount of polymer added was very small. In addition, the surface tension is \( 59.6 \pm 0.5 \) mN/m which is an almost constant value. Therefore, the \( t_R \) of the capillary thinning process under the condition where the plate diameter is fixed is constant. For the measured value of \( \sigma \), \( \rho \), and \( R_0 \), Eq. (2) gives \( t_R \approx 21 \) ms. Chandrasekhar et al.\(^{21} \) clarified that the relation between the fluctuation period of the low viscosity fluid drop and \( t_R \) during jetting process follows Eq. (3).

\[
T_{osc} \equiv \frac{2\pi}{\omega_{osc}} \approx \frac{(\pi/\sqrt{2})t_R}{\omega_{osc}}
\]

(3)

where \( T_{osc} \) is the oscillation period and \( \omega_{osc} \) is the frequency of oscillation. For the calculated \( t_R \), Eq. (3) gives \( \omega_{osc} = 135 \) Hz in good agreement with the experimental observation. In other words, the constant value of the oscillation frequency is due to the constant Rayleigh time scale.

3.3 Effect of plate diameter on sessile drop oscillation

Among the various factors that affect the oscillation occurring in the transition of the capillary thinning, we conducted an experiment by changing the plate diameter. The plate diameter was 1 mm and 3 mm at fixed lifting velocity of 1 mm/s. Figure 7 shows the oscillation frequency (\( \omega \)) of the PEO solution with different molecular weight depending on the plate diameter. Higher oscillation frequency was measured when using a 1 mm plate compared to the 3 mm plate. The frequency was about 145 Hz for 3 mm plate, while it was 210 Hz for 1 mm plate. This is because \( t_R \) decreases when 1 mm plate is used and \( \omega_{osc} \) changes inversely to this. In addition, the range of \( \eta \) where the oscillation occurs becomes wider when 1 mm plate is used. As can be seen in Fig. 7 (a) and (b), when the 3 mm plate is used, the oscillation did not occur in the range of \( \eta \) above 40 cP and 60 cP, respectively. However, when using 1 mm plate, the oscillation was not observed for \( \eta \) larger than 60 cP at 0.2 m PEO. In the case of 1.0 m PEO, the oscillation was observed in all the viscosity range covered in this study. In other words, the oscillation occurring in the transition of the capillary thinning process on a smaller plate shows a higher frequency for wider range of \( \eta \).

Figure 8 is the oscillation decay ratio (\( \zeta \)) with different plate diameter. The 1 mm plate has higher oscillation decay ratio that the 3 mm plate. As mentioned before, the decay ratio was calculated from the ratio of the first peak difference to the second peak difference. Thus, the closer the decay ratio to one, the slower the decay progresses. Therefore, when using a plate with a small diameter, the decay progresses slowly than that of a large diameter plate. This is because the amount of the polymer solution to be loaded on the plate varied depending on the plate diameter. In a general mass-spring-damper system, the energy loss per cycle is proportional to

![Fig. 6 Oscillation decay ratio (\( \zeta \)) with different lifting velocity: (a) 200,000 g/mol PEO, (b) 1,000,000 g/mol PEO, and (c) 2,000,000 g/mol PEO.](image1)

![Fig. 7 Oscillation frequency (\( \omega \)) with different plate diameter: (a) 200,000 g/mol PEO, (b) 1,000,000 g/mol PEO, and (c) 2,000,000 g/mol PEO.](image2)

![Fig. 8 Oscillation decay ratio (\( \zeta \)) with different plate diameter: (a) 200,000 g/mol PEO, (b) 1,000,000 g/mol PEO, and (c) 2,000,000 g/mol PEO.](image3)
the damping coefficient of the system. The damping coefficient is proportional to the square root of the mass$^{22}$, That is, when the 3 mm plate is used, the mass of the loaded sample is larger, thereby increasing the damping coefficient and energy loss. As a result, when a larger plate is used, the oscillation decay ratio is closer to zero. As the concentration of the polymer solution increases, the decay ratio decreases. Even with smaller plates, the decay ratio becomes smaller as the viscosity of the polymer solution increases, which is due to the increase in energy dissipation.

### 3.4 Conditions for oscillation occurrence

As discussed in Section 3.2 and 3.3, the range of $\eta_0$ where the oscillation occurs changed depending on the lifting velocity and the plate diameter. For 2.0 m PEO, the oscillation was observed at all viscosity range regardless of the operating conditions used in the experiments. On the other hand, in the case of 0.2 m PEO and 1.0 m PEO, the range of $\eta_0$ where the oscillation occurs changed depending on the plate diameter. Figure 9 (a) and (b) show the occurrence of oscillation according to the plate diameter for 0.2 m PEO. In Fig. 9 (a), the oscillation occurred up to 40 cP. In the case of smaller plate diameter, however, the oscillation was observed up to 60 cP as shown in Fig. 9 (b). A similar trend can be seen in Fig. (c) and (d), which indicates the oscillation of 1.0 m PEO. When 3 mm plate was used, oscillation did not occur even at 100 cP. However, the oscillation occurred under the same viscosity condition when using 1 mm plate. Thus, it can be confirmed that the oscillation occurs in a wider viscosity range when the plate with a smaller diameter is used. In other words, the occurrence of oscillation depends on the characteristic length of the system. On the other hand, no change in viscosity range due to lifting velocity was observed. Therefore, in the case of the sample with severe oscillation, it is possible to measure the extensional properties in a state where the oscillation is reduced by using a larger plate diameter. On the other hand, the difference depending on the molecular weight was also observed. The oscillation occurred under a wider range of $\eta_0$ for PEO solutions with higher molecular weight. Researches on the effect of rheological properties (viscosity and elasticity) on the oscillation could be investigated in the future.

### 4. CONCLUSIONS

In this study, we investigated the effect of lifting velocity and plate diameter on sessile drop oscillation as a first step to quantitatively analyze the oscillation generated in the capillary thinning process. For this purpose, the surface of the sessile drop was observed using high-speed camera and high-magnification lens. Then, the oscillation was quantified in terms of the oscillation frequency and decay ratio. PEO solutions with three different molecular weight were used and the viscosity of the PEO solution was matched to each other.

The PEO solution showed a typical capillary thinning behavior. The oscillation occurred in the upper and lower sessile drop during the transition from visco-capillary to elastocapillary regime except for some conditions. The deviation of the filament diameter was observed in the transition point and it coincides with the time when the oscillation begins.

The lifting velocity did not affect the oscillation frequency. The plate diameter was fixed at 3 mm. The oscillation frequency of all polymer solutions was almost constant. It can be interpreted such that a change in lifting velocity does not affect the Rayleigh time scale, which is the characteristic time of the capillary thinning process. Also, the lifting velocity used in this study did not affect the decay ratio. Since the dynamics of the capillary thinning depends only on the material properties of the sample, the decay ratio related to the energy loss was not affected by the lifting velocity. On the other hand, the diameter of the plate influenced the oscillation frequency. When using a plate with smaller diameter, the frequency increased. This could be attributed to the fact that the oscillation frequency shows negative correlation for the change in Rayleigh time scale. The plate diameter also affected the decay ratio. When using a smaller plate, slower progress of decay was observed. This is because the amount of the polymer solution loaded according to the plate diameter affects the energy dissipation.

An interesting result is that the range of viscosity occurring the oscillation was varied depending on the lifting velocity.
velocity and the plate diameter. In addition to the operating conditions, the area where the oscillation does not occur may be affected by the rheological properties of the sample. Study on the effect of rheological properties (viscosity and elasticity) on the oscillation needs to be carried out further. A practical use of this study is that we can induce capillary thinning process without oscillation by adjusting the operating conditions properly. In conclusion, the significance of this study is the possible improvement of the extensional property measurements of polymer solutions in which the oscillation occurs severely.

ACKNOWLEDGMENT

This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIT) (No. NRF-2018R1A5A1024127).

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