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Analysis of microchannel flows with yield-stress fluid

미세 채널에서의 항복력 유체 흐름 분석

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박 정 원

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이 논문을 공학석사 학위논문으로 제출함

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Abstract

Analysis of microchannel flows with yield-stress fluid

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In this paper, the flow of a polymer microgel, e.g. Carbopol, in microchannel was analyzed in consideration of the effect of microgel size and slip on the wall of microchannel. From the previous flow visualization experiment, we proposed a new system that channels can move in the opposite direction of flow. This not only shows the velocity of particles but also the overall pathlines at microchannel after fully-developed flow.

With a new system and particle tracking velocimetry code, We conduct the study for two direction. First, we compare the velocity profiles obtained from flow visualizations and simulations in the mid-plane of the microchannel. Simulation with constitutive equation obtained from the rheological property did not coincide with velocity profile from visualizations. For example, the experimental results was slower than computation results near the wall. The reason for this phenomenon was explained by drawing pathlines of particles.

Additionally, we suggest the method to find parameters for constitutive equations from the small-gap rheometry. When we compared with the results of flow visualization, it was reasonable. velocity profile from flow visualization.

Second, we observe the effect of slip at wall on the velocity profiles. Unlike previous computation, which ruled out the effects of slip, the calculations proceeded with no slip condition. In our study, we consider both no slip condition and slip condition. For the fair comparison between experiment and computation results, we show that simulation with slip condition is close to the experimental results comparing with simulation with no slip condition. When we consider flow in the microchannel, we have to be careful on obtaining rheological property and considering slip condition.

Keywords: Flow visualization, Yield-stress fluid, Microchannel, Micro particle tracking velocimetry, Slip condition, Parameter estimation

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1. Introduction

Various products, such as Li-ion Battery, Flexible display, and Adhesive, are coated with the functional thin film through the slot coating process. Mainly, the Slot coating method is generally used when coating slurry to make battery electrodes. The coating solution consists of organic and inorganic particles and polymers. Because of microstructure which made with particles, the fluid shows yield stress.[1] A yield stress fluid is a liquid that has a solid-like property before it reaches certain stress and is then liquid-like when the stress exceeds certain stress. The specific stress at this time is called yield stress. There are several models to analyze this yield stress fluid, typical of the Bingham model and Herschel-Bulkley (HB) model.[2] Bingham model has constant viscosity after yield stress, while the viscosity growth rate of Herschel-Bulkley is gradually reduced because of shear-thinning. These models and fluid mechanics equations are fitted with a yield-stress fluid in a bulk region, which is a continuum. However, when carbopol with microgel is used in micro-processes, the process area becomes smaller, and the particle size remains the same. According to recent studies, experimental results are showing that the flow of fluid in the micro-area is not the same as the flow in the bulk area.[3-6] So, coating processed in micro-region is not guaranteed to be a continuum.

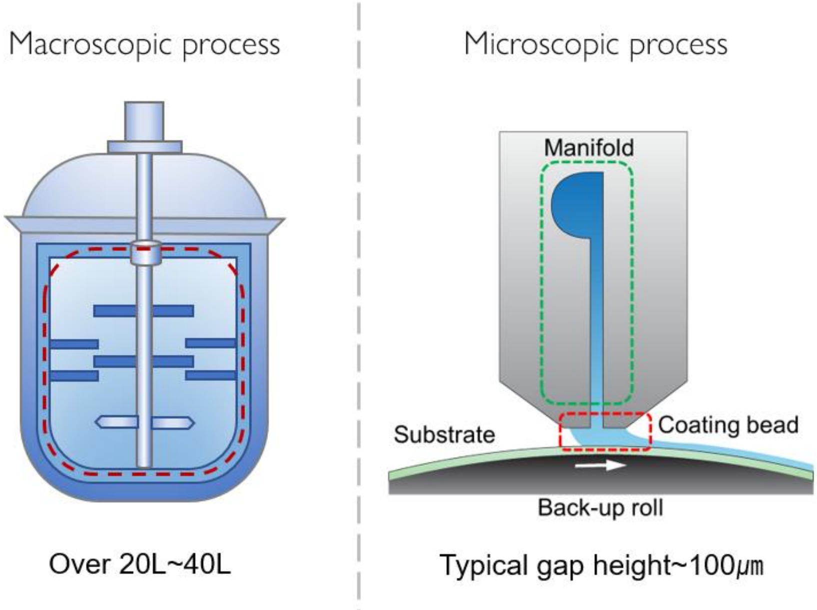


Figure 1. Macroscopic and Microscopic process at industry.

It is essential that find an appropriate model with a coating solution in micro-region because it is possible to understand the performance of the final product and the cause of the defect. Although it is best to experiment with the process, it is difficult to visualize the process due to the following reasons: A significant amount of solution, the Opacity of slurry, and the Inconvenient installation of visualizing equipment in the process.

In this study, a microchannel produced by polydimethylsiloxane (PDMS) imitates the flow in the micro-region of slot coating. Battery slurry is yield-stress and shear-thinning fluid, so Carbopol, which has similar properties to a coating solution, is chosen as model fluid. Carbopol is transparent so that we can visualize the flow inside the channel, and predict flow patterns in the slot coating.

Previous papers experiment that compares the flow in microchannel and the flow from calculating theoretical equation. Goyon et al. study the flow of emulsions in rectangular glass microchannel to neglect the effect of the top and bottom wall and observe the flow as two-dimension. Two glass microchannel was used in this study, one has smooth wall roughness, and the other has a rough surface. They observe that emulsion flows above a threshold volume fraction differ from flows at bulk-region.[3] Géraud et al. used rectangular rough glass channels to observe the flow of Carbopol ETD 2050. They also fabricate large aspect ratio microchannel so that the flow can be observed 2D flow at the half of its height.[4, 5] Liu et al. fabricate 50 to 500μm polydimethylsiloxane (PDMS) microchannel to visualize the flow of 0.14wt% Carbopol 940 solution.[6] All these papers identify that there is a difference between the flow of fluid used in each paper in the micro-region and that in the bulk-region, and they explain their results in terms of the non-local effect.

Generally, a rough sandblast is used when measuring the property of Carbopol with a rheometer due to slip-issue.[7, 8] Wall slip of slurry also occur on smooth surfaces. The study of Liu et al. removed the effect of increasing flow rate due to a slip and proceeded computation with no slip condition.[6] When we obtain a velocity profile of yield-stress fluid through a microchannel, the slip velocity varies depending on the channel's vertical position. Therefore the effect of the slip on the velocity profiles varies for each height. In this study, the simulation with a slip condition was performed and compared with the experimental results.

In addition to the above papers, the points of view to be considered are as follows. The first is to see how the particles were moving inside the flow. As the flow area becomes small, the relative size ratio of microgel increases. In this respect, we expect that micro-region affects the laminar flow of fluorescence particles. In conventional experimental methods, the microscope observes only the fixed point of the channel. Therefore, the concept of relative velocity was used in this experiment by using motors to move channels in opposite directions of flow. This method has the advantage of being able to see fast-moving particles slowly and extracting the path of movement of particles in the channel. The second is to identify the velocity profile by including the slip-occurring effects on the channel wall at a high shear rate. Typically, no slip condition is specified in the calculation for accuracy reasons. The flow rates of Carbopol in the experiment, which Liu et al. conduct, are rather slow.[6] We wanted to verify that such a method mentioned in the paper of Liu et al. was reasonably applied even at high flow rates. The study was conducted in anticipation that slip would cause not only more flow but also affect the channel's velocity profile.

2. Experiment

2.1. Fabrication of microchannel

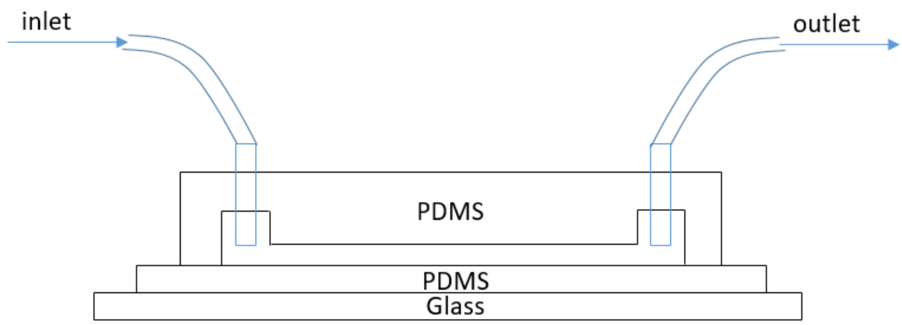


Figure 2. Schematic of microchannel composition. Unassigned area in the middle is empty.

Microchannel made of PDMS (Polydimethylsiloxane, Sylgard 184, Dow Corning) was fabricated to observe a flow of yield stress fluid in micro-size region.[9-11] The mold of upper part of the microchannel is made from uv photolithography of SU-8 100, an epoxy-based negative photoresist solution. To create PDMS microchannels with mold made by the method described above, the following steps are taken. Mix the PDMS base and PDMS elastomer in a ratio of 10:1. Remove air bubbles from the mixed PDMS with the Vacuum Chamber. We use petri dish to pour PDMS on the mold which is slightly larger than the mold. To prevent the PDMS from sticking to the glass, pour the mixed PDMS on the petri dish covered with aluminum foil. It is recommended that pour it on a horizontal plane. Make a bottom part of the microchannel the same process as creating the top of the channel. Instead, use a basic wafer instead of photolithography SU-8 wafer, and make a thin PDMS height not exceeding 2mm. Place these two petri dishes (top and bottom part of microchannel) in a level oven and cured at 65 degrees Celsius for 6 hours. Peel the cured PDMS and cut them into slide glass sizes. Using a puncher, drill holes in the inlet and outlet region for liquid flow. Place the cured PDMS parts and slide glass into the plasma cleaner and do plasma treatment (PDC-32G, Harrick) at 500 mTorr for 3 min. Attach bottom part and top part of microchannel on top of slide glass in order. Finally, Hard curing of microchannel conduct in oven at 180 degrees Celsius for 2 hours to enhance bonding of PDMS. When the two parts are well bonded, microchannel fabrication is complete.

2.2. Dissolving Carbopol

Carbopol 941(Lubrizol) was chosen to representative of yield-stress fluid, and used in PIV experiment.[4-6] The concentration of carbopol is 0.2wt%. We use stirrer (MISUNG SCIENTIFIC, BL 620D) to mix 2L of carbopol solution for 150rpm, 1 day. To prevent aggregation of Carbopol powder in water, Carbopol powder and deionized water was poured in alternately. When Carbopol powder was dissolved in deionized water, the pH of Carbopol solution is 3-4. The microgels of carbopol are most swelled at neutral pH. Therefore, add 20wt%

NaOH to raise pH of Carbopol solution 3 to 6-8. After addition NaOH, Carbopol solution was stirred for 3 hours and checked that pH comes in between 6-8 with pH meter (METTLER TOLEDO). To conduct of PIV experiment, put fluorescent particles (Polyscience, Fluoresbrite® BB Carboxylate Microspheres 0.50μm)[14] in the solution and mix them so that they spread homogeneous. The approximate solid volume fraction of beads is 0.0025%.

2.3. Rheology properties

Rheological properties of the Carbopol was measured with stress-controlled rheometer(TA instrument, Discovery HR-2). Geometry used for measured is 40mm sand-blast parallel plate. This geometry prevent slip occur. This geometry keeps the slip from happening. We obtained property data with Carbopol sample containing Fluorescent beads to consider even the possible effects of fluorescent particles on the channel when fluid flows. The sample is presheared at $50s^{-1}$ for 30s and have a relax time for 1 min. Through this process, we destroy the microstructure present in the carbopol sample to eliminate the history. After then, the sample go through Oscillation time process at 1% strain, 1 Hz frequency. Each point takes one minute and measures four times for one point to average. This is a process to ensure that the sample is stable. When the rheometer conduct a shear-rate sweep process, shear rate increase from $0.01s^{-1}$ to $1000s^{-1}$, and decrease on the contrary. The flow curve are plotted with data which obtained by decreasing the shear rate from $100s^{-1}$ to $0.01s^{-1}$. Comparing the data of upsweep and downsweep, the hysteresis was not seen. The parameters of Herschel-bulkley model were gotten from downsweep data. n = 0.436, K = 7.11, σy = 4.893. n is the power-law index, K is the consistency, σy is the yield stress.

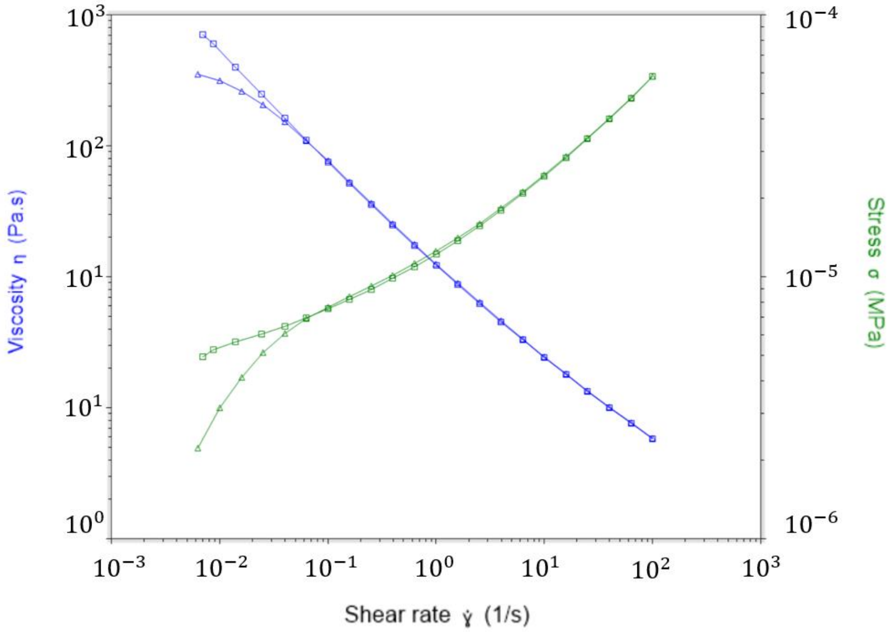


Figure 3. Rheological curve of Carbopol 941 0.2wt% measured at bulk.

2.4. Micro particle tracking velocimetry

The experiment which is necessary to observe the flow of fluid, consists of three parts.[3-6, 11, 12] Syringe pump pushes the carbopol, inverted microscopy detect fluorescent beads in fluid which flow in microchannel, and Capture a series of images with a high-speed camera. Syringe pump deliver Carbopol to microchannel with a flow rate of 800ul/h.

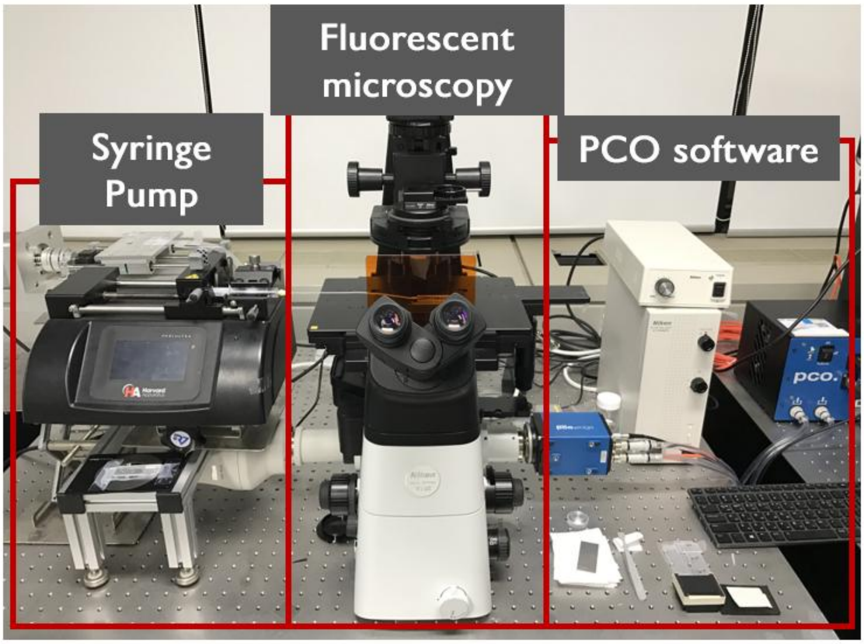


Figure 4. Experimental setting of particle tracking velocimetry.

Syringe size used in this experiment is 5ml. Excitation of fluorescent beads is 360nm, so Dapi filter is mounted. Place the microchannel on the microscope (Nikon, Inverted microscope ECLIPSE Ti2) and observe the flow of fluorescence particles in the fluid. The images of the beads were obtained with an exposure value of 10μs and 499.87 fps (frame per second) using a High-speed camera (PCO edge 4.2). The spatial resolution of the images observed through a microscope and a high-speed camera is 1.5pixels/μm when using a 20x objective lens. In addition to the common experimental methods, a new system has been proposed to extract not only the velocity of particles but also the pathlines from images. The conventional method is to capture fluorescent particles passing through the observation area of the microscope, so we can get the velocity profile. If the microscope is made to observe along a set of fluorescence particles in the fluid, it can be seen how the particles travel throughout the microchannel. However, the microscope cannot move, so the microchannel should be moved. The concept of relative velocity was used by making the channel move in the opposite direction of fluid flow. This allows for relatively slow detection of fast-moving particles while also identifying the path of particles movement. Motor(PI, N-381 NEXACT linear actuator) was used to construct these systems.

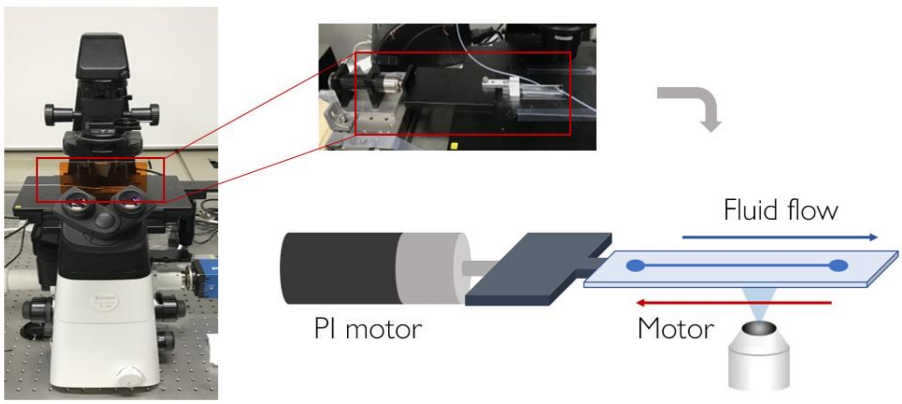


Figure 5. The motor-composed system with relative velocity concept

The schematic diagram for this system is shown in Figure 5. Parts that can adjust angles were made through a 3D CAD program to parallel the motor and microchannel.

A code was written in Matlab to determine the travel distance of fluorescent particles by comparing successive image pairs. Approximately three hundred trace particles are captured in one image, and the blurry particles which is out of focus cannot represent the speed of the target plane, so they are excluded through post-processing. The applied technique is detectORBFeatures, and after recognizing the fluorescent beads through perceive the edge of them, determine how much it has moved to vector. Velocity profile according to channel width is drawn from Orthogonal projection of displacement vector on axis of flow direction. It was created through image analysis of 500 frame pairs and measured at 10 positions of height that divided into nine equal parts from the channel wall to the center of the channel. The slip velocity at the sidewalls of each height positions can be obtained from tangent lines of the velocity profile near the walls. These points were plotted to obtain a slip velocity profile through fitting of the function.

2.5. Computation

Computation programs for meshing, simulation, and post-processing which used in this research are Trelis(Csimsoft, American Fork, UT), Goma[15], and Paraview[16]. These software programs were released by Sandia National Laboratories. Mesh generation was conducted according to the length specifications of the channel. For the appropriate number of mesh, we made the channel in trelis with square inlet and outlet plane, $200*200\mu m^2$. It is divided into 15 parts in the width and height, but elements near the walls were made more finer than middle of the channel (dual bias 1.2 in trelis). Because the amount of variation is greater in velocity near the wall. For simulation, parameters of fluid are required. The following parameters were used to calculate water. Viscosity = 0.00089, Density = 0.0001, Surface tension = 72. In the case of Carbopol, Calculation is carried out using the herschel-bulkley model embedded in Goma with parameters obtained from rheometer properties graph. Simulation with boundary condition of no slip, was conducted with the flow rate excluded as much of its more in-flow due to slip. The methods to do this were referred to in the paper of Liu et al[6]. To process the simulation with boundary condition of slip condition, Put the function of slip velocity Vs(h) at the sideset of vertical sidewalls. The input of the calculations is pressure and this simulation is steady state. The Simulation was processed until residual norm decrease below $5*10^{-7}$.

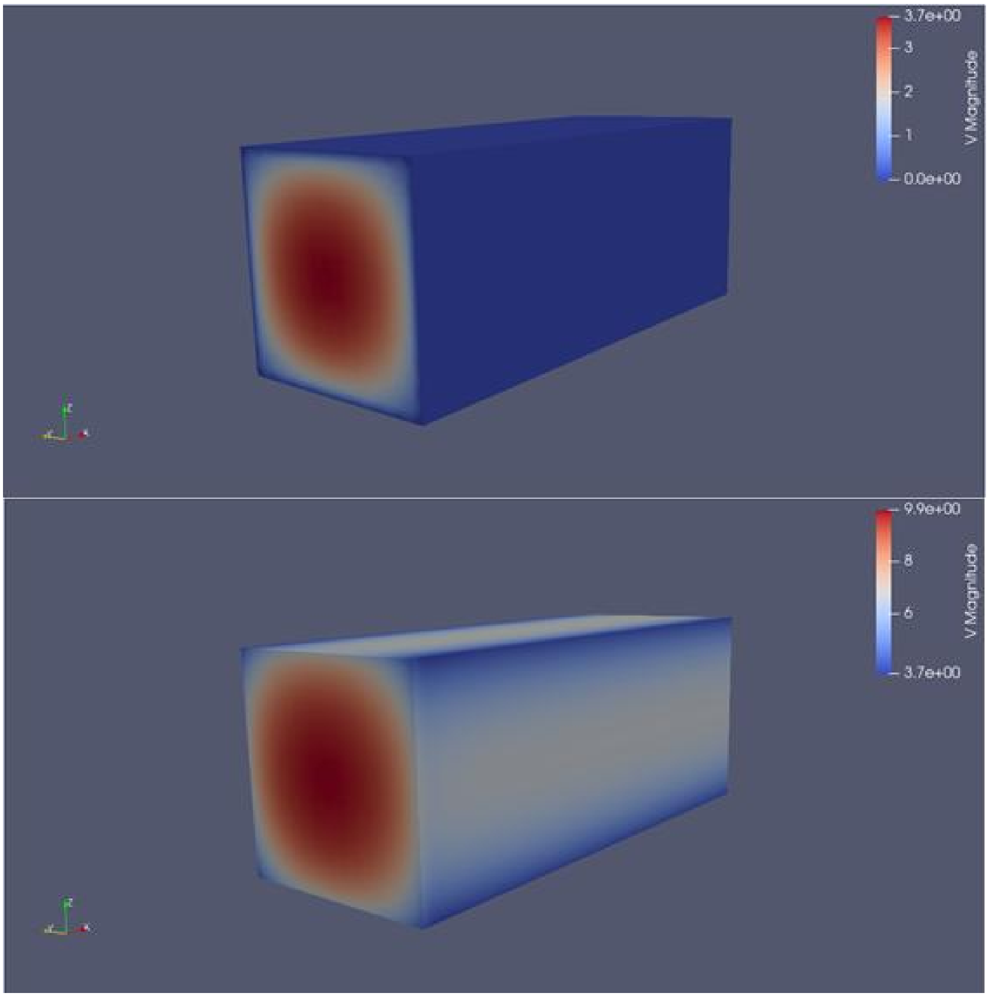


Figure 6. Computation with no slip condition (top), and with slip condition (bottom).

3. Results

3.1. Rheometer data

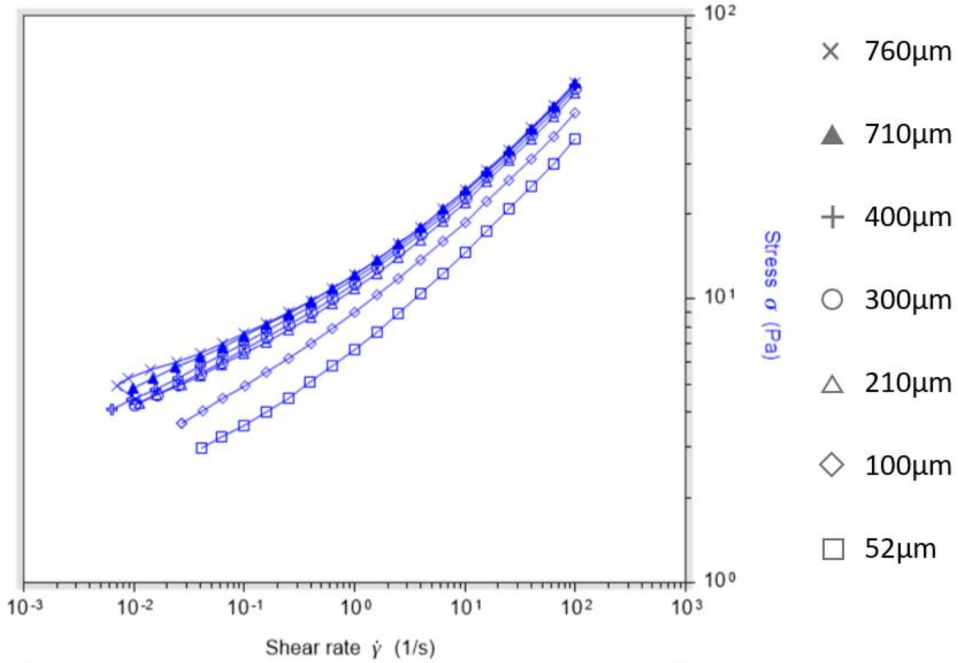


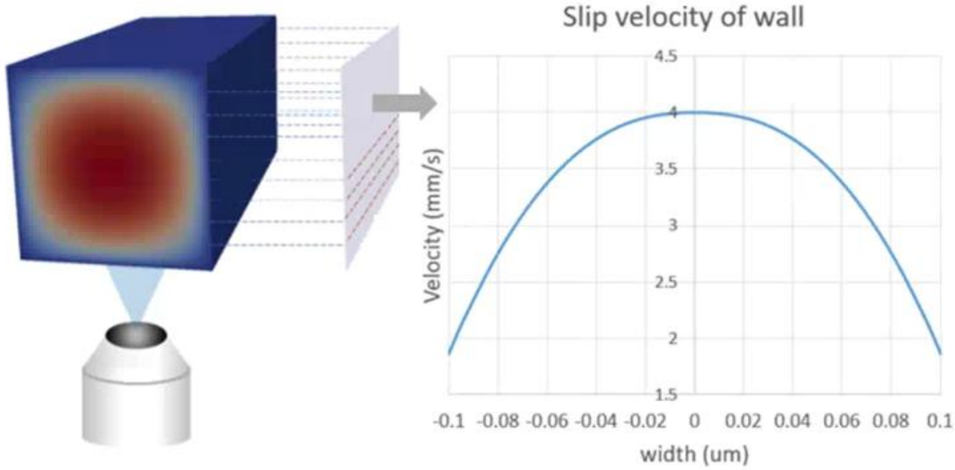
Figure 7. Transition of curve form according to the gap. Carbopol 941 0.2wt% used in this measurement.

To confirm the change of rheometer data of Carbopol 941 when the gap of geometry is getting smaller, we measured rheological curves with rheometer at various gap height. The geometry gap range is from 760μm to 52μm. The protocol measuring the rheological curve is the same procedure, which is described in part of 2.3. When we get curves with a rheometer, curves of 760㎛ to 210㎛ are comparably matched. However, below 210㎛, rheological curves differ from the bulk one. These results are coincidence with the results of Géraud et al.[5] We can notice from these data that Carbopol flow in micro-region is also different from that in the bulk area of a rheometer.

3.2. Slip velocity function at wall

With the code described in part of 2.4, We obtained velocity profiles at each height position of a microchannel. This verified code obtained the moving speed from the carbopol flow visualization images and confirmed the slip existence. The method to determine slip velocity is cited from the study of Liu et al.[6] The slip velocity was obtained from the tangent line near the walls. We notice that the slip velocity varies depending on the vertical position of the channel. With the data points of slip velocity at each height, Slip velocity function can be fitted, as shown in Figure 8.

Vs is a slip velocity, Vm is a maximum velocity of slip, a and b are constants of function. The reason for using these functional shapes was that a gradient at the center of the velocity profile is almost zero, which could not be expressed as parabolic. This function was used for simulation with a slip condition.



$$V_s = V_m(1 - a|x|^b)$$

Figure 8. Slip velocity function at verticle wall. The function was derived through fitting of the slip velocity points.

3.3. Mid-plane velocity profile in microchannel

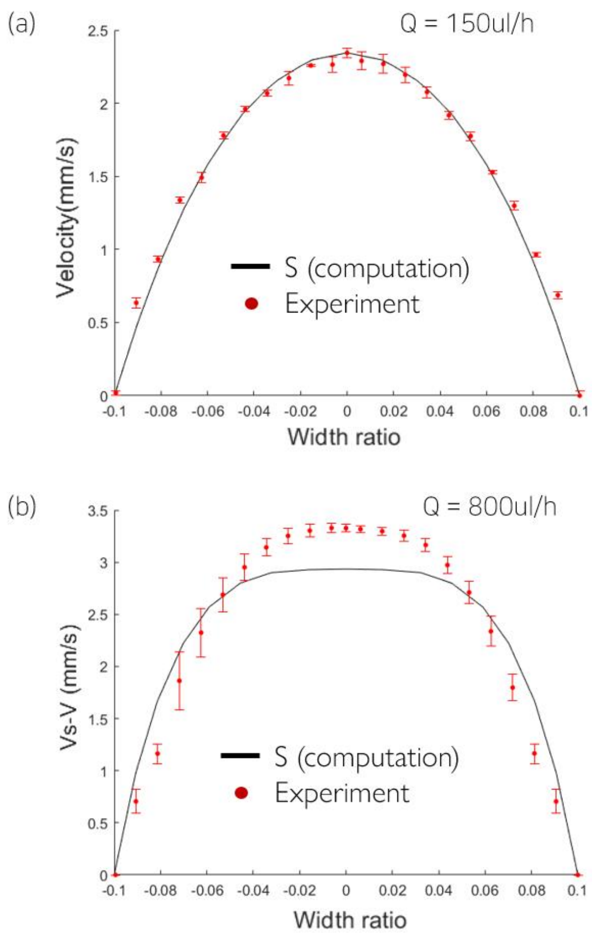


Figure 9. Comparison of Experiment results and simulation. (a) Velocity profiles of DI water. (b) Velocity profiles of 0.2wt% Carbopol mixed at 150rpm. Q is experiment flow rate controlled by syringe pump.

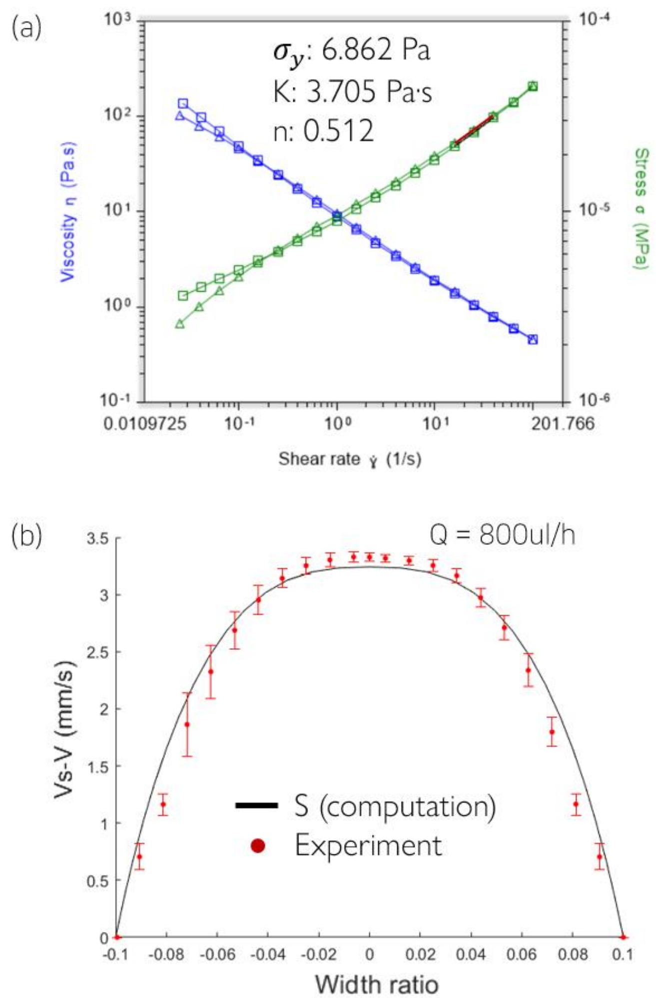


Figure 10. (a) Rheometer curve that measured at 100μm gap. Fitting is conducted at shear rate range in microhannel. (b) The velocity profile that calculated with parameter from (a). The model used at simulation is power-law model.

Before the slip velocity profile was obtained by the code we made, We verified the code's accuracy with distilled water. Figure 9(a) is the velocity profile of water that we gain at experiment and simulation. The inlet flow rate was 150μl/h. The slip at the wall in the flow of water was unobservable. As shown in Figure 9(a), the simulation results and the velocities obtained by the experiment match.

However, the velocity profiles of carbopol that obtained from visualization, in Figure 9(b), did not coincide with the simulations. The inlet flow rate of carbopol was 800μl/h. Computation conducted with parameters that were written in part 2.3. with the Herschel-Bulkley(HB) model. This model is expressed as an equation (1) and is a representative model of yield-stress fluid at bulk-region.

$$\tau = \tau_y + k(\dot{\gamma})^n \qquad (1)$$

The discrepancy between experiment and calculation means that flow does not follow the HB model. Therefore, we proceeded with the process written below to match the experiment and calculation.

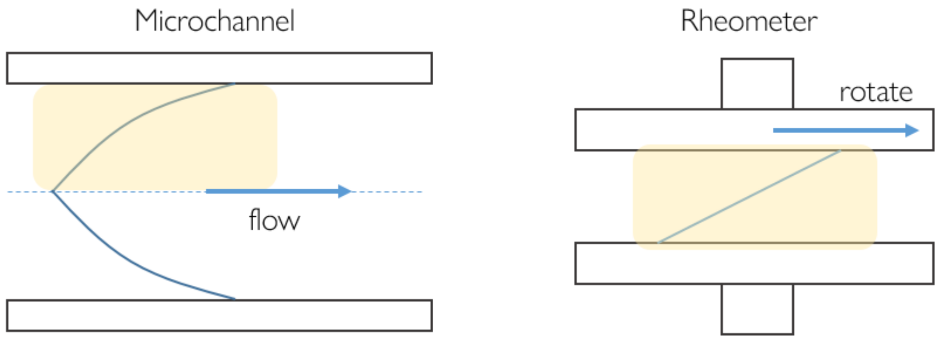


Figure 11. Schematic figure of shear rate in microchannel and rheometer. Shear rate graphs are similar at yellow box.

Figure 11 shows the shear rate graph in the microchannel and rheometer. At microchannel, When the middle of the channel is the center axis, the top and bottom are symmetrical. Therefore, the appropriate parameters were determined when measured with a rheometer gap at half the height of the channel. We measured rheological property at 100μm height. The curve graph is in Figure 10(a). In previous papers, when a carbopol flows in a microchannel, even a carbopol solution made with the same protocol, and the same concentration was not fitted with the same parameter. In other words, parameters that come from curve fitting the entire data, measured at a low gap, do not represent flow in micro-channel. Therefore, the parameters obtained by fitting only the data near the shear rate in this experiment were used for calculation. The parameters obtained by fitting, drawn with a red line in the figure, are n=0.512, K=3.705, σ_y=6.862.

However, when calculating with the Herschel-Bulkley model, the graph shape did not change even if any parameters were used. For this reason, we conduct simulation with power-law model parameters from Figure 10(a). The result of this simulation is drawn in Figure 10(b). Consequently, simulation with the power-law model comparably was matched with visualization results than the simulation with the HB model.

3.4. Computation with slip condition

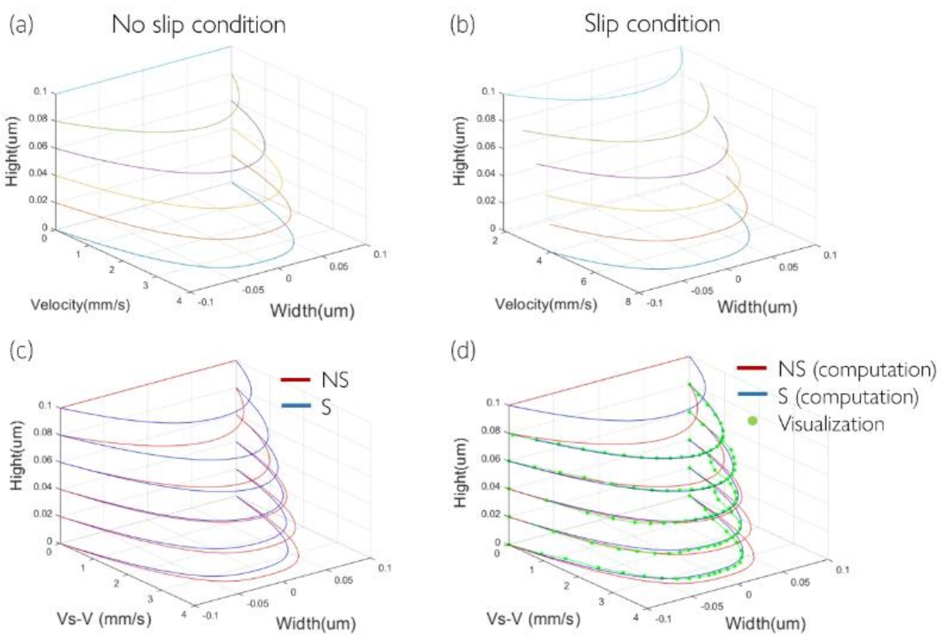


Figure 12. Simulation with no slip and slip condition of carbopol flow. (a) No slip condition simulation, (b) Slip condition simulation, (c) Comparison of simulation with no slip and slip condition. y-axis is velocity that substract slip velocity. (d) Comparison result of (c) with visualization velocity profile.

Computation with slip condition was conducted with the slip velocity function that we obtained by flow visualizations. When we compared simulation results with no slip and slip, velocity near the wall did not match. The experimental velocity profile was also compared to determine which calculations were more appropriate for the actual flow. Therefore, when we consider flow in the microchannel, we have to be careful about obtaining rheological property and considering slip condition.

4. Final Remark

In previous papers, they focused on explaining why flow visualization results and simulation results do not coincide at micro-region. Almost all the papers mention that the reason is the non-local effect.[3-6]. In this study, it developed in a different direction than just explaining the reason. First, we propose the new system that can get information not only velocity but also pathlines of fluorescent beads for a longer distance than the previous PTV method. We also propose the Matlab code to conduct PTV, which is optimized for obtaining a velocity profile. Second, with our new system, we analyze why visualization results are slower than simulation near the wall. Finally, we suggest the method to conduct simulation more coincide with the actual flow of yield-stress fluid in microchannel.

Velocity profiles of Carbopol and water were obtained using the Particle tracking velocimetry (PTV) method. There are many techniques for detecting features in Matlab. Among them, the detectORBfeature, which is used in this research, detected each particle well. When the analysis of movement proceeds with the code we have written, we can check whether the particles are well detected in the image pair. This code ensures the accuracy of the velocity profiles. Newtonian fluid, such as water, is a good match between theoretical and experimental values in micro-region.[6] The test was conducted with water to verify the code written. The simulation results and the velocity profiles of water obtained by the experiment were found to match.

We analyze why visualization results are slower than simulation near the wall through microgel size and pathline. In the study of Geraud et al., all data points show that experimental velocity was faster than the simulation data.[5] In the study of Liu et al., experimental velocity data points at the center were faster than simulation, but that points near the wall were slower than simulation.[6] Geraud et al. made a 1wt% Carbopol solution. Mixing speed is very fast at 2100rpm, and the structure size measured by the authors is 2.4㎛. Yang Liu et al. made a Carbopol solution with 0.14wt% concentration at 50rpm. Authors of this paper expected that carbopol particle size is tens of μm order. We made 0.2wt% Carbopol solution, and the mixing speed is 150rpm. These papers construed the reason for these results as confinement and non-local effect. Our experiment showed the same tendency as the paper of Liu et al. We analyze why experimental results were slower than computation results near the wall, unlike previous work of Geraud et al., by drawing pathlines of particles.

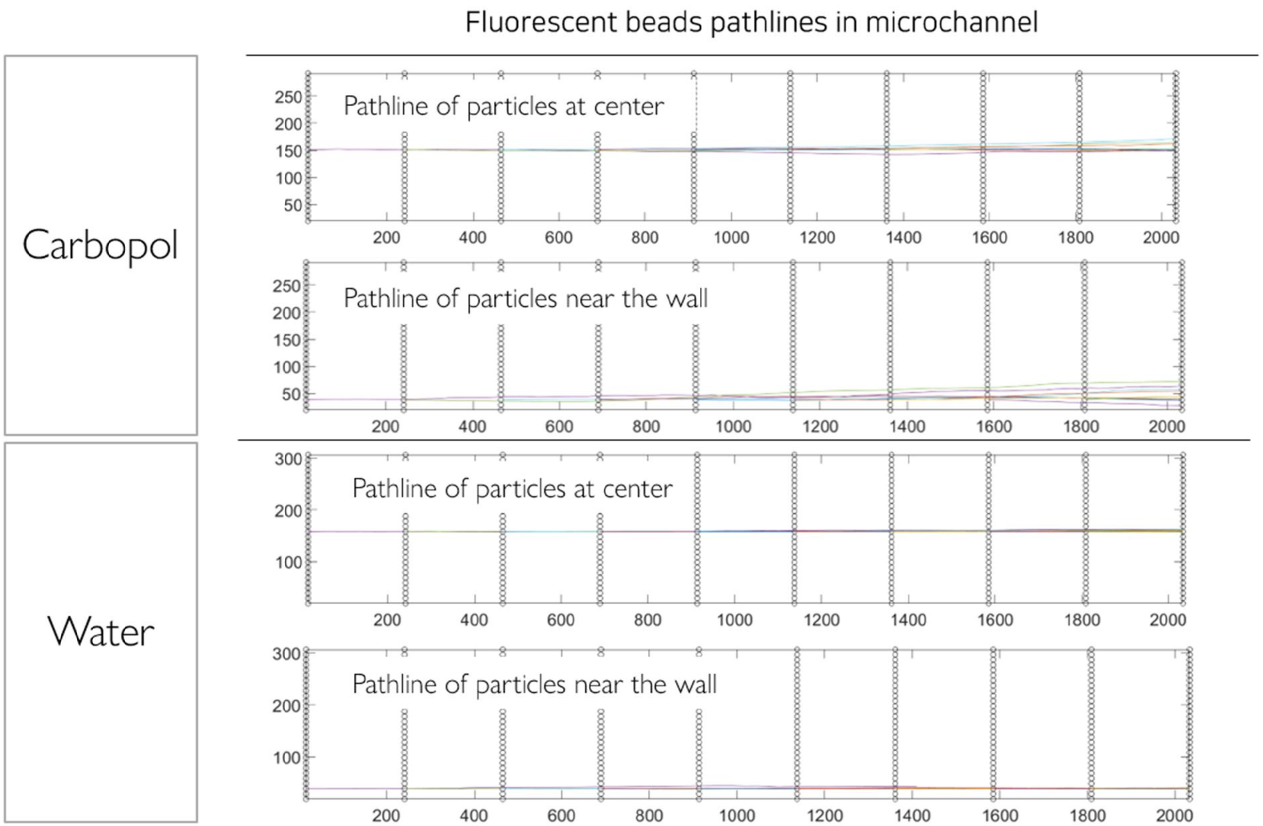


Figure 13. Pathlines of fluorescent beads in the flow of Carbopol and water

First, we draw the pathline of fluorescent particles in the flow of water compared with Carbopol as Figure 13. Fluorescent particles in water move in a straight line both center and near the wall.

However, in the case of carbopol, the flow is unstable near the wall of the channel. As it flows through the microchannel, the particle size ratio has increased when the microgel size is large. Therefore, the unstable flow, which occupied a small portion in the bulk area, occurs near the wall appears to be maximized. As a result, we guess that the velocity near the wall was measured slower than the theoretical value.

Based on our study, we suggest the two methods to simulate more accurately. Conduct simulation with slip velocity function appropriate for each situation. Obtain parameter at half of the microchannel height gap and proceed curve fitting with the local points, representing the shear rate of the channel. Of course, tests should be carried out at diverse concentrations and flow rates, but this study suggests a way to obtain parameter values through rheometer data without eye fitting.

The following studies should be conducted for further experiments. In this study, we tried to stabilize the new experiment system. Thus, with this system, the above content should be verified with various concentrations or different types of fluids. Furthermore, Instead of dispersing fluorescence particles into fluids, it would also be meaningful to tag fluorescent material on polymers to determine the flow and movement patterns.

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국문초록

이 연구에서는 벽에서의 슬립 현상과 미세 영역에서의 마이크로젤의 크기 비율로 인한 효과를 고려하여 마이크로 채널에서의 카보풀과 같은 고분자 용액의 흐름을 분석하고자 한다. 이전의 흐름 시각화 연구들에서 더 나아가, 마이크로 채널을 흐름의 반대 방향으로 움직일 수 있도록 새로운 시스템을 구축하였다. 이 시스템은 입자의 흐름뿐만 아니라 마이크로 채널의 전반적인 내부 입자 이동경로 또한 관찰할 수 있다.

새로운 시스템과 입자 추적 속도 측정 코드를 가지고 두 가지 방향으로 연구를 진행했다. 첫 번째로, 기존 실험들과 같이 채널 중앙에서 흐름 시각화를 통해 구한 속도 프로파일과 시뮬레이션 결과를 비교하였다. 이 두 결과는 일치하지 않았고, 벽 근처에서의 실제 흐름 속도가 이론적으로 계산된 결과보다 느렸다. 이 현상의 이유를 채널 내부 흐름 속 형광 입자의 이동경로를 통해 설명하였다. 추가적으로, 시뮬레이션을 통해 흐름 시각화의 속도 프로파일에 맞도록 피팅할 때, 적합한 파라미터를 레오미터 데이터로부터 찾아낼 수 있는 가능성을 제시하였다. 두 번째로, 벽면에서의 슬립으로 인해 속도 프로파일의 분포에 주는 영향을 보았다. 기존 연구에서는 슬립으로 인해 더 흘러간 유량만큼을 제하고 no slip condition으로 계산하였다. 각 높이에서의 속도를 구한 후, 시뮬레이션 결과와 흐름 시각화 속도 프로파일을 높이별로 비교해보았다. 이를 통해 슬립을 반영한 시뮬레이션 계산결과가 실제의 속도 프로파일을 비교적 알맞게 예측할 수 있다는 것을 파악했다. 작은 마이크로채널에서의 흐름을 예측하기 위해서는 작은 갭에서 구한 레오미터 파라미터를 활용하고 슬립을 고려해야한다.

주요어: 흐름 시각화, 항복력 유체, 마이크로채널, 마이크로 입자 추적 속도 측정법, 슬립 조건, 파라미터 예측

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