Development of a Flowmeter for Sub Micro Liter per Second Order Unsteady Flow Rate Based on the Pressure Difference in a Capillary

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Abstract
In order to measure unsteady flow rate of the order of less than 1 µl/sec, a new flowmeter consisting of a capillary and a pressure gauge has been developed. When a target flow passes through the capillary, the measured pressure loss in the capillary gives the flow rate according to the Hagen-Poiseuille equation, which indicates the flow rate is proportional to the pressure loss. Investigating prototype flowmeter characteristics in the case of steady flow of water, we confirmed that the flow rate given by the Hagen-Poiseuille equation derived from the measured pressure loss accords with the flow rate estimated by the gravimetric technique. The accuracy is within ± 1 %. When the flow rate decreases gradually, the measured flow rate accords with the theoretical value at each moment. This flow meter enables to measure time varying flow rate. In the case that the flow rate abruptly changes, the measured pressure has a time delay because of the property of the pressure gauge used in this study. Compensation for this time delay is demonstrated.

Key words: Flowmeter, Flow Measurements, Unsteady Flow, Capillary, Microfluidics, Micro Pumps, Micromechanics

1. Introduction

Recently, development of microfluidic devices has been significant and several kinds of micro pump also have been developed for practical use(1)–(5). The flow rate generated by a micro pump is generally too small to be measured by ordinary measurement equipment, so it is difficult to assess pump performance accurately. For instance, a commercial microsyringe pump used in this study is supposed to be able to pump with a flow rate less than 1 µl/sec, in principle, however, it is difficult to verify the actual flow rate with high time resolution by measurement. Some researchers(6)–(9) have developed measurement methods for such low flow rate based on phenomena caused by heating the fluid. These methods are complex to treat and require long time to measure and are not applicable to unsteady flow rate.

In order to measure unsteady flow rate of the order of less than 1 µl/sec, we have developed a new flowmeter system based on pressure loss when the fluid goes through a capillary. Advantages of this flowmeter are a simple structure, which consists of only a capillary, a pressure gauge and pipe arrangements, and application of the Hagen-Poiseuille law. In this paper, the characteristics and the capability of the flowmeter are examined using water as the working fluid and also measurement results of unsteady flow rate are examined.
2. Nomenclature

- $d$: inner diameter of the capillary
- $g$: gravity
- $H$: head difference between the buffer tanks
- $l$: length of the capillary
- $p$: ball screw pitch of the microsyringe pump
- $P$: pressure difference between the inlet and the outlet of the capillary
- $P_h$: applied pressure by the head difference
- $P_m$: measured pressure
- $P_\infty$: measured pressure when time enough elapses after the syringe pump starts
- $P^*$: non-dimensional pressure difference $P_m/P_\infty$
- $Q$: target flow rate
- $Q_c$: flow rate in the capillary
- $Q_g$: apparent flow rate through the pressure gauge
- $Q_h$: theoretical value of the flow rate generated by the head difference
- $Q_m$: flow rate given by the Hagen-Poiseuille equation adopting $P_m$
- $Q_m'$: compensated flow rate
- $Q_p$: flow rate generated by the microsyringe pump
- $Q_p'$: time mean of $Q_p$
- $Q_w$: flow rate given by the gravimetric technique
- $s$: inner cross-sectional area of the microsyringe
- $S$: cross-sectional area of the small buffer tank
- $t$: time
- $v$: stage speed of the microsyringe pump
- $\bar{v}$: time mean of $v$
- $x$: stage position of the microsyringe pump
- $\alpha$: proportional coefficient of the relation between $P$ and $\Delta V$
- $\Delta V$: capacity increment of one of two compartments divided in the pressure gauge
- $\mu$: viscosity of the working fluid
- $\nu$: kinematic viscosity of the working fluid
- $\rho$: density of the working fluid
- $\omega$: motor rotational frequency of the microsyringe pump

3. Theory and Experiments

3.1. Schematic of the flowmeter

In the new flowmeter, a liquid flows through a capillary. The fully developed flow in the capillary is laminar because the Reynolds number of the flow is up to 1. Assuming that the effect of the inlet and the outlet of the capillary is negligible, the relation between the pressure loss $P$ and the flow rate $Q$ in the capillary is given by the Hagen-Poiseuille equation as

$$Q = \frac{\pi \cdot d^4 \cdot P}{128 \cdot l \cdot \mu}, \tag{1}$$

where $d$, $l$ and $\mu$ are the inner diameter and the length of the capillary and the viscosity of the working fluid, respectively. $d$ and $l$ are specified and constant, $\mu$ is given by the kind and the temperature of the working fluid. The measurement value of $P$ gives the flow rate according to Eq. (1), which indicates that $P$ is simply proportional to $Q$. In this study, it is verified that the pressure measurement is possible and the flow rate given by Eq. (1) adopting the measured pressure loss accords the actual flow rate.

Figure 1 shows a diagram of the flowmeter system. The flowmeter consists of a block containing a capillary and a pipe arrangement with a pressure gauge connected to the block. In practice, the block is also connected to an inlet pipe, an outlet pipe and a valve for relief of the pressure difference. The inner diameters of the pipes excepting the capillary are so large that the pressure losses in the pipes are negligible.
The inner diameter and the length of the capillary are \( d = 0.46 \text{ mm} \) and \( l = 60 \text{ mm} \), respectively. The measurement range of the pressure gauge (Tsukasa Sokken Co., Model SDP-11) is (0-20) Pa. The measurement flow rate range is within about 360 nl/sec assuming that the working fluid viscosity \( \mu \) as \( 1 \times 10^{-3} \text{ Pa} \cdot \text{sec} \), Eq. (1).

In this study, degassed distilled water is used as the working fluid. A thermocouple touching the capillary outer wall is installed and it gives the wall temperature. The fluid temperature outside the capillary is also measured. It is confirmed that both measured values of the temperature are identical each other and the values should be the fluid temperature, which gives the viscosity \( \mu \) and the density \( \rho \) of the fluid.

3.2. Flow generation by a head difference

For assessment of the flowmeter faculty, a reference flow, which is of sub \( \mu \text{l/sec} \) order and accurately known, is necessary. A head difference is applied to generate this flow. As shown in Fig. 2, the inlet and the outlet are connected to the buffer tanks opened to the atmosphere. In order to control the head difference \( H \) between the tanks, the height of the inlet tank could be changed by an automatic \( z \)-stage on which the tank is laid. The stage is automatically driven and be able to change the height in increment of 2.5 \( \mu \text{m} \). The theoretical value of the applied pressure \( P_h \) in the capillary is written as

\[
P_h = \rho g H,
\]

where \( \rho \) and \( g \) are the fluid density and the gravity, respectively.

For generating a steady flow, tanks with large cross-sectional areas are connected to both the inlet and the outlet, so that the displacement of the water levels in the tanks are negligible regardless of inflow or outflow to the tank, i.e. change of the head difference \( H \) during a measurement is negligible. The steady flow rate can be measured by a gravimetric technique. The weight increment time series of the working fluid in the outlet tank is estimated by an electric scale, on which the tank is laid. For instance, time series of the measured fluid weight increment in the tank are shown in Fig. 3. The tank is in a possibly airtight box and it had been confirmed that the weight of the evaporating water during the required time to measure
the flow rate is negligible, so the time derivative of the weight and the fluid density give the flow rate $Q_w$.

For instance of a measurement of time varying flow rate, a tank having small cross-sectional area is connected to the inlet and the outlet is connected to a large tank. Because the fluid flows from the inlet tank via the capillary to the outlet tank, the water level in the inlet tank and the flow rate decrease with time. The time derivative of the head difference $dH/dt$ gives the flow rate $Q_h$ as

$$Q_h = -S \frac{dH}{dt},$$

where $S$ is the cross-sectional area of the small tank. When $Q = Q_h$ and $P = P_h$, Eqs. (1), (2) and (3) give the relation between the pressure $P$ and the time $t$ as follows.

$$\frac{\pi}{128} \frac{d^4}{l \mu} P_h = -S \frac{d}{dt} \left( \frac{P_h}{\rho g} \right),$$

Assuming that the temperature of the fluid is constant, i.e. $\mu$ and $\rho$ are constant,

$$P_h = C \exp \left\{ -\frac{\pi}{128} \frac{d^4}{l \nu S} t \right\},$$

where $\nu \equiv \mu/\rho$ is the kinetic viscosity and $C$ is an integral constant. Defining the value of $P$ when $t = 0$ as $P_0$, the above equation is written as

$$P_h = P_0 \exp \left\{ -\frac{\pi}{128} \frac{d^4}{l \nu S} t \right\}.$$

Adopting Eq. (5) into Eq. (1), the theoretical flow rate $Q_h$ is given as

$$Q_h = \frac{\pi}{128} \frac{d^4}{l \mu} P_0 \exp \left\{ -\frac{\pi}{128} \frac{d^4}{l \nu S} t \right\}.$$

### 3.3. Microsyringe pump

The flow rate generated by a commercial microsyringe pump is measured using the flowmeter. As shown in Fig. 4, the pump consists of a microsyringe and a linear motion stage driven by a ball screw with a stepping motor. The fluid is pumped by displacement of
the syringe piston driven by the stage. The generated flow rate $Q_p$ is given by the product of the syringe inner cross-sectional area $s$ and the stage speed $v$;

$$Q_p = sv. \tag{7}$$

In order to confirm the stage speed $v$, time series of the stage position $x$ is measured using an electric micrometer. The measured stage speed $v$ is given by the time derivative of $x$. Figure 5 shows the relation between $x$ and the ratio of the measured speed $v$ to the time mean speed $\overline{v}$. The configuration rotational frequency of the stepping motor is constant, however, $v$ includes a fluctuation. The value of $v/\overline{v}$ at any stage position $x$ is identified regardless of $\overline{v}$. $v$ is given by the product of the ball screw pitch $p$ and the motor rotational frequency $\omega$, i.e. $v = p \times \omega$. If $\omega$ fluctuates with time, the fluctuation does not depend on the position $x$. If $p$ includes an error, its value is inherent in $x$ along the ball screw. Figure 5 shows that the speed fluctuation ratio depends on $x$ and is mainly due to the error of $p$. It is supposed that the pumping flow rate $Q_p$ is also fluctuating.

4. Results and Discussion

4.1. Assessment of the flowmeter in the case of steady flow rate

When steady flow is generated by a hydrostatic head, the pressure difference $P_m$ between the inlet and the outlet of the capillary is measured. Figure 6 shows that the measured pressure $P_m$ and the theoretical value of the applied pressure $P_h$ are identical. Thus, the pressure gauge gives a precise value of the pressure difference.

In parallel with the measurement of $P_m$, the flow rate $Q_m$ is estimated by the gravimetric technique. $Q_m$ is defined as the flow rate given by Eq. (1) adopting the measured pressure $P_m$. In Fig. 7, $Q_m$ and the difference ratio to $Q_w$ are depicted. The black circles indicate the results in the case that the capillary inner diameter $d$ is given as 0.460 mm. $Q_m$ is around 4 %
smaller than $Q_w$ in the whole range. The cause of the characteristic difference is believed to be due to be the errors of the capillary sizes, especially the inner diameter $d$, the biquadrate of which is proportional to the flow rate as illustrated by Eq. (1). The measurement value of $d$ is 0.46 mm in the accuracy of 0.01 mm, so the true value is between 0.455 mm and 0.465 mm. Assuming that $d = 0.464$ mm, $Q_m$ is re-calculated and plotted with the white circles in Fig. 7. The characteristic difference between $Q_m$ and $Q_w$ is settled and it is reasonable to adopt 0.464 mm to $d$ for estimation of $Q_m$. Random difference between re-calculated $Q_m$ and $Q_w$ remains and the value of the difference is ±1% (equivalent to ±2 nl/sec), which should be the measurement accuracy of the flowmeter in the region between 40 nl/sec and 350 nl/sec. As Eq. (1) specifies, a flowmeter with a narrower and/or longer capillary enables us to measure smaller region flow rate.

4.2. Measurement of unsteady flow rate generated by the head difference

When the small tank was connected to the inlet of the flowmeter and a head difference was applied, the time series of the pressure difference was measured. Figure 8 (a) shows the time series of the measured pressure $P_m$. The time $t$ is defined as zero when $P_m$ is 20 Pa, i.e. $P_0 = 20$ Pa. $P_h$ given by Eq. (5) is also shown in Fig. 8 (a) and it is confirmed that $P_m$ corresponds to Eq. (5). In order to estimate the accuracy of the flow rate $Q_m$ given by Eq. (1) adopting $P_m$, the difference between $Q_m$ and $Q_h$ given by Eq. (6) is shown in Fig. 8 (b). The difference between the measurement value and the theoretical value is within the measurement error derived in §4.1. Therefore, this flowmeter system is suitable for time varying flow rate measurement.
4.3. Measurement of the pumping flow rate of the microsyringe pump

We commenced by zeroing both flow rate and pressure difference, and started to drive the fluid using the microsyringe pump when $t = 0$. The set value of the pumping flow rate was constant, however, the actual flow rate is supposed to fluctuate because of the non-uniformity of the piston drive speed. The time series of the measured pressure $P_m$ is illustrated in Fig. 9. $\bar{Q}_p$ is the time mean value of the pumping flow rate $Q_p$. The variation components of $P_m$ are classified into two categories. One is smaller time scale fluctuation due to the fluctuation of $Q_p$ given by Eq. (7) with the measured speed $v$. The other is larger time scale variation. Initially, $P_m$ increases and after that $P_m$ is constant neglecting the smaller time scale fluctuation.

The initial period $t_d$, during which $P_m$ increases with time after the pumping starts, arises because the pressure gauge output follows the discontinuous flow rate increasing. As discussed in §4.2, the flowmeter can measure smoothly time varying flow rates, however, in the case that the flow rate instantaneously changes $P_m$ does not follow the step change of rate at once.

This time delay $t_d$ can be caused by the measurement mechanism of the pressure gauge used in this study. The measurement principle of the pressure gauge is based on a diaphragm deformed by the pressure difference. We cannot know the detail of its internal structure because it is commercial. In order to compensate the time delay, a basic internal model of the pressure gauge shown in Fig. 10 is considered. The inside of the pressure gauge is divided into two compartments by a diaphragm, deformation of which gives the measured pressure difference $P_m$. When the pressure is applied, the capacity of the higher pressure side compartment increases because of the diaphragm deformation. It is assumed that the capacity increment $\Delta V$ is proportional to the pressure difference $P_m$ as

$$\Delta V = \alpha P_m,$$

where $\alpha$ is a constant value given by the specification of the pressure gauge. When $P_m$ in-
creases because of increase of the flow rate $Q$, $\Delta V$ also increases, so that a part of the fluid flows into the upper stream side compartment in the pressure gauge and the same volume fluid flows out from the other compartment as if there the fluid flows through the pressure gauge. This apparent flow rate going through the pressure gauge is denoted by $Q_g$ that is given by the time derivative of $\Delta V$ as

$$Q_g = \frac{d\Delta V}{dt} = \alpha \frac{dP_m}{dt}. \tag{9}$$

Therefore, the flow rate in the capillary $Q_c$ should be the remains of $Q$;

$$Q_c = Q - Q_g. \tag{10}$$

Assuming $P = P_m$ and adopting Eqs. (1) and (9) to Eq. (10), the following relationship is obtained.

$$\frac{\pi}{128} \frac{d^4}{l \mu} P_m = Q - \alpha \frac{dP_m}{dt} \tag{11}$$

$$\therefore P_m = \frac{128l\mu}{\pi d^4} Q - C \exp \left( -\frac{\pi d^4}{128l\mu} \frac{t}{\alpha} \right), \tag{12}$$

where $C$ is an integral constant. Adopting the conditions that $P_m = 0$ when $t = 0$ and $Q$ is constant when $t \geq 0$,

$$P_m = \frac{128l\mu}{\pi d^4} Q \left( 1 - \exp \left( -\frac{\pi d^4}{128l\mu} \frac{t}{\alpha} \right) \right). \tag{13}$$

The second term of the right side expresses the effect of delaying and the value approaches to zero with time. Here, $P_m$ when $t = \infty$ is denoted by $P_\infty$ and a non-dimensional pressure $P^+$ is defined as

$$P^+ \equiv \frac{P_m}{P_\infty} = 1 - \exp \left( -\frac{\pi d^4}{128l\mu} \frac{t}{\alpha} \right). \tag{14}$$

In Fig. 11, the experimental results of $P^+$ are depicted. Neglecting the small fluctuation, the time series of $P^+$ does not depend on the time mean pumping flow rate $\bar{Q}_p$, and is indicated by an unique approximate line, which gives $\alpha$ in Eq. (14) as $2.47 \times 10^{-11}$ m$^3$/Pa. According to this result, the hypothesis of the pressure gauge inside model is available. The stabilization period $t_d$ defined as the time to be $P^+ = 0.99$ in Eq. (14) is 6 sec.

The time delay can be compensated using Eq. (11). The compensated measured flow rate $Q'_m$ is derived as

$$Q'_m = Q_c + Q_g = \frac{\pi}{128} \frac{d^4}{l \mu} P_m + \alpha \frac{dP_m}{dt} \tag{15}$$

This equation indicates when $P_m$ gradually varies, as like the case in §4.2, $Q_g$ is so small that the time delay on the measured flow rate $Q_m$ is negligible. In Fig. 12, $Q_m$ given by Eq. (1) and $Q'_m$ compensated by Eq. (15) are compared. The initial period of $Q'_m$ is shortened.
After the initial period, $Q_m'$ has a fluctuation similar to that of the pumping flow rate $Q_p$. Note that $Q_p$ is not a certain value but an estimated value given by Eq. (7). For calculation of $Q_p$, a measured piston drive speed $v$ is adopted, however, the syringe inner cross-sectional area is assumed as uniform and any other disturbance is not considered. Therefore, $Q_p$ does not necessarily express the actual flow rate and thus the incomplete accordance between $Q_m'$ and $Q_p$ is reasonable. It is supposed that the flow meter system demonstrates the fluctuation of the flow rate of the microsyringe pump.

5. Conclusions

In order to measure unsteady flow rate of the order of sub µl/sec, we have developed a new flowmeter system based on the pressure loss in a capillary. We employed a capillary, whose inner diameter $d$ and length $l$ are $d = 0.46$ mm and $l = 60$ mm, and a pressure gauge, whose measuring scale is (0-20) Pa. The characteristics of the flowmeter were investigated using water as the working fluid and the following main results were obtained.

(1) In the case of a steady flow rate, the flow rate $Q_m$ given by the Hagen-Poiseuille equation adopting the measured pressure loss $P_m$ agrees with the flow rate $Q_w$ estimated by the gravimetric technique. The accuracy is within 1% in the region between 40 nl/sec and 350 nl/sec.

(2) When the flow rate generated by the head difference decreases gradually, the measured flow rate $Q_p$ accords with the theoretical value $Q_h$ at each moment. This flow meter enables us to measure continuous unsteady flow rate.

(3) It takes a time delay $t_d$ for the measured pressure $P_m$ indicated by the pressure gauge used in this study to follow a step flow rate variance. The effect of this time delay on the measured flow rate can be compensated by allowance for the diaphragm deformation in the internal model of the pressure gauge.

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References


