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IN-SITU MEASUREMENT OF MICRO FLOW RATE USING NEAR INFRARED ABSORPTION METHOD

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ABSTRACT: *A novel technique is presented for measuring micro flow rate using the near infrared (NIR) absorption method. We obtained the water temperature in the tube in-situ condition using NIR absorption method. A calibration curve between temperature and the NIR absorption intensity in the range of 1500 nm – 1700 nm wavelength was obtained. For measuring flow rate in the tube, the tiny spot of water in the tube was heated using NIR laser (1450 nm) through the lens which was absorbed into the water. The temperature profiles along the tube were obtained using the NIR absorption method via laser heating for different flow rates. The flow rates were obtained from the temperature difference between the room temperature and the obtained temperature from the NIR method. The calibration curves between the flow rate and temperature obtained from the NIR absorption method was obtained in the two flow rates (1-20 mL/h and 40-100 mL/h). The error and uncertainty of the NIR absorption method for measuring flow rate were approximately 1.2 % and 1% at the 1-100 mL/min flow rate, respectively. Thus, we confirmed that the NIR absorption method quantitatively measures the flow rate with respect to the in-situ condition for the first time.*

1 General Introduction

The measurement and control of liquid flow rates is critical in several applications such as medical drug injection, semiconductor manufacturing processes, and chemical processes. The general flow detection methods include orifice, turbine, ultrasonic, Coriolis, and MEMS flow sensors[1-4]. Orifice, turbine, and ultrasonic flow meters cannot measure micro flow rates owing to their low sensitivity with respect to micro flow rates. Coriolis flow meter and thermal mass flow meters measure micro flow rates. Coriolis flow meters measure extremely low flow rates (maximum flow rate: 5 g/h) with extremely high accuracy ($\pm 0.2\%$). However, they exhibit a few limitations such as high cost and pressure resistance. The most widely used microflow sensor is the thermal mass flow sensor, and its advantages include small size, short response time, low power consumption, and a good interface with microfluidic devices [5]. The thermal mass flow sensor was commercialized by several companies and it measures extremely low flow rates in the range of nL/min with a 10% accuracy. The microthermal flow sensor typically consists of upstream and downstream temperature sensors and a heater located between the two temperature sensors. The operating principle is that a mass flow rate is measured via a temperature difference due to heat transfer between a heated sensor and fluid stream. The temperature profile of the sensor tube is symmetric at the zero flow rate. In the linear range, the temperature difference linearly increases with increases in the flow rate. This permits the measurement of the flow

rate by sensing the temperature difference. These temperature sensors are typically fabricated by using MEMS technology, and this makes a sensor expensive and complex. Additionally, it is necessary to install a temperature sensor in the tube. Hence, contamination due to a sensor may occur, and thus measurement in the in-situ condition is impossible.

Laser-based techniques for the measurement of flow include several advantages such as remote, nonintrusive, in situ, spatially, and temporally precise measurement [6-9]. The most commonly used flow measurement technique is particle image velocimetry (PIV) that measures the flow field by capturing two images of seeding particles in the fluid with a time gap. Although several studies use PIV to measure a flow field, there are a few limitations of PIV such as the requirement of particle seeding. Recently, techniques for measuring the flow field without seeding particles were developed by using fluorescence and Raman scattering [7, 8, 10]. In order to overcome the disadvantages associated with using particles for measuring flow, different dyes were used as markers [11]. A non-intrusive measurement technique based on spontaneous Raman imaging was proposed to investigate microscale flow structures [10] and spray flow [7].

Specifically, near-infrared (NIR) spectroscopy is a conventional method for measuring the molecular composition using a vibrational molecular band [12]. Water exhibits several absorption bands observed in an NIR region, and the spectral shape of each band varies with respect to temperature. The absorption band of water (O-H band, $\nu_1+\nu_3$) shifts to a shorter wavelength with increases in temperature [13]. Additionally, NIR spectroscopy is used for water temperature measurement in channels with sizes in the range of micrometers [14]. The water temperature in a microchannel was visualized by using the NIR absorption method with a high resolution (0.2 K)[14]. Furthermore, the NIR laser is a good source to heat water with a wavelength of 1450 nm, which corresponds to the O-H stretching band. The NIR laser heats a tiny spot in the order of a few micrometers by focusing and easily changing the heating position. The NIR absorption method exhibits high potential in terms of measuring the flow rate. However, it has not been applied to measure the flow rate to date.

This is the first study to measure the micro flow rate in the in-situ condition by using the NIR absorption based thermal mass flow sensor[15]. The NIR laser heats a tiny spot in the transparent polymer tube through a lens. The water temperature in the upstream and the downstream is measured by the NIR absorption method. The temperature profile of the flowing tube with laser heating is obtained for different flow rates. The theoretical heat transfer was analyzed through the simulation of the temperature profile for different flow rates. The calibration curve for the temperature difference and the reference flow rate was obtained with a flow rate in the range of 1–100 mL/h. Thus, we confirmed that the NIR absorption method measures the micro flow rate in the in-situ condition with high accuracy in the 1–100 mL/h flow rate.

2 Experiment

The flow rate was measured by the NIR absorption method using FTNIR and a heating laser. The measured flow rate was calibrated according to the reference flow rate, which was obtained using a syringe pump and balance. Figures 1(a) and 1(b) show the experimental setup for the in-situ measurement of micro flow rate using the NIR absorption method and a magnification of the measurement position, respectively. DI water was flowed through the syringe pump (Chemyx, Nexus 3000) with a 50 mm diameter syringe at a constant flow rate. The syringe was connected to an IV set tube (transparent polymer tube), which is widely used in hospitals for drug delivery. The IV set tube has an internal diameter of 3 mm and optical transparency of 0.3 μm to 3 μm wavelength in the NIR region. The reference flow rate was obtained using a micro balance (Mettler-Toledo, XPE 206 DR) with

evaporation trap. The maximum capacity and readability of the balance were 81 g and 0.005 mg, respectively. All parts of the system were connected with silicon tubes. The tube was connected to the needle at the scale, and the needle was immersed into the water in the balance collecting glass vessel to avoid droplet formation and allow a continuous reading of the flow rate over time. The obtained reference flow rate was used for calibrating the measured flow rate. The flow rate was ranged from 1 to 100 mL/h using the syringe pump.

When the IR laser with 1450 nm wavelength is irradiated to the water, the laser is absorbed by the water due to vibration ($\nu_1 + \nu_3$) [13,14]. The IR diode laser (CNI, $1450 \text{ nm} \pm 15 \text{ nm}$) was focused on the center of the IV set tube through the lens (50 mm focal length) with 500 mW laser energy. The water was heated at a tiny spot region by the focused laser with a spot diameter of $12.5 \text{ }\mu\text{m}$. The temperature of the water around the heated region was obtained by the FTNIR method. Light from a halogen lamp (AvaLight-HAL), emitting broadband light with wavelength ranging from 360 nm to 2500 nm, was sent to the IV set tube through the optical fiber and collimator. The collimated light was absorbed by the water passing through the tube at a specific wavelength region depending on the water temperature. The absorbed light was sent to the FTNIR (AROptix) through the collimator and optical fiber. The FTNIR can measure broadband wavelengths from 900 nm to 3400 nm with a resolution of 1–5 nm. The optical fiber set (halogen lamp and FTNIR) was translated along the tube to measure the water temperature at the various measurement positions.

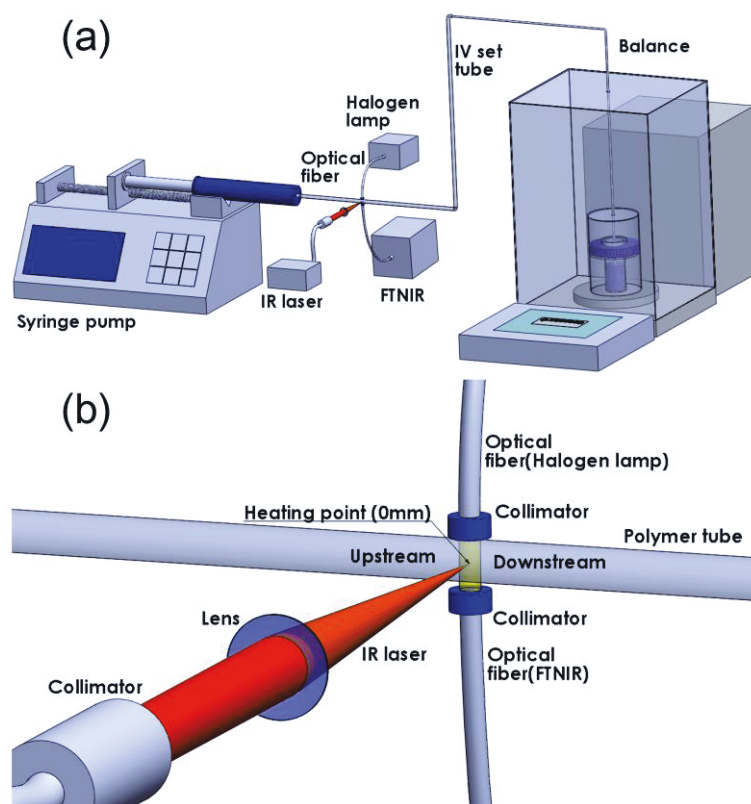


Fig. 1. (a) Experimental setup (b) Magnification of measurement position shown in Fig. 1(a)

3 Results

3.1 Temperature measurement using NIR absorption method

It is difficult to measure the water temperature using intrusive methods such as thermocouple in a small diameter tube. In addition, the thermocouple, which is fabricated using MEMS, can contaminate the fluid, and there are limitations for the installation of a thermocouple in a small diameter tube. The optical method can be good alternative for measuring water temperature in a small diameter tube. Therefore, the water temperature in the IV set tube was measured using the NIR absorption method. The temperature was controlled by a circulating bath (Lab Companion RW-0525G), which has a refrigeration and heating circulator. The circulating bath is connected to the IV set tube, which is covered by the insulator except for the measuring position. The reference water temperature in the tube was measured using PT 100 sensor with 2 mm diameter and temperature acquisition system (Lab tec). Figure 2 shows the absorption spectra for different temperatures obtained by the FTNIR. The reference spectra were obtained from the IV set tube without the water. The absorption spectra of the water in the tube were obtained by subtracting the spectra from the reference spectra. The first overtone of the O-H band is found around a wavelength of 1450 nm. The maximum of the O-H band (1450 nm) shifts towards lower wavelength with increase in temperature [13]. When the temperature of the water is increased, the absorption intensity of wavelengths from 1500 nm to 1700 nm is gradually decreased. Figure 3 shows the averaged absorption intensity of wavelengths from 1500 nm to 1700 nm according to the water reference temperature. The averaged absorption intensity of wavelengths from 1500 nm to 1700 nm decreases linearly with increasing temperature, as shown in Fig. 3. The red line in Fig. 3 indicates the linear fitting line whose slope and intercept are -0.00294×10^{-6} and 0.909, respectively, with 0.9996 R-square. We can measure the water temperature if the absorption intensity is obtained using this temperature calibration curve.

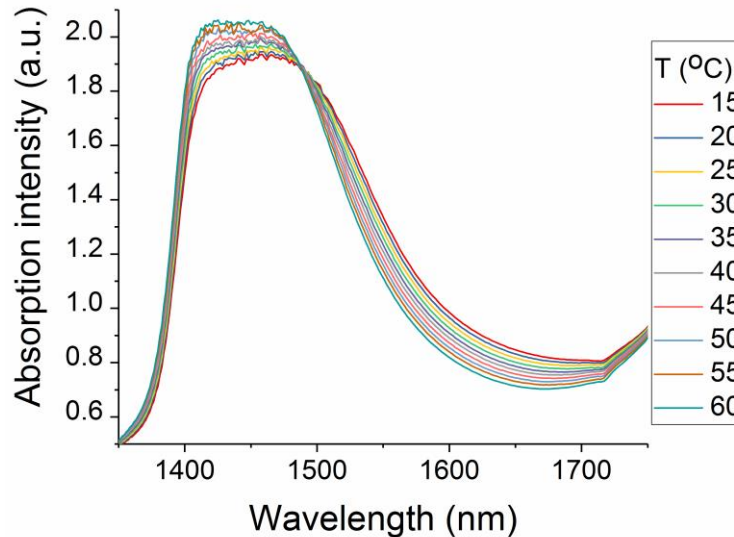


Fig. 2. Absorption spectra for different temperatures obtained by FTNIR

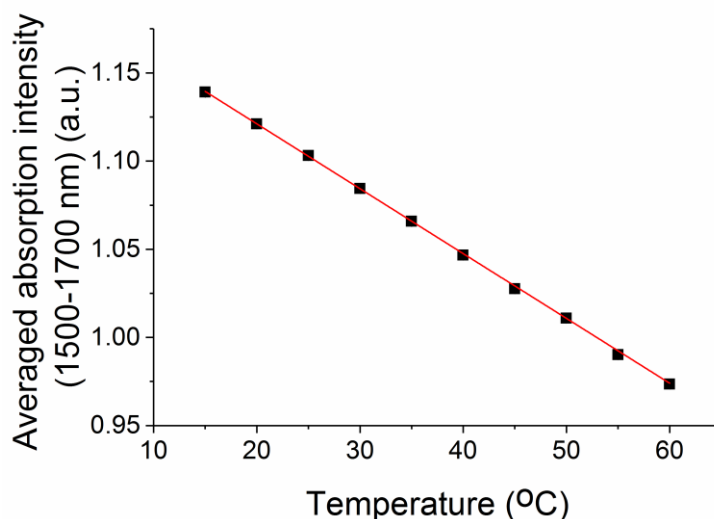


Fig. 3. Absorption intensity obtained from FTNIR according to the DI water temperature

3.2 NIR Laser heating in flowing tube

Laser is a good local heating source, since it can be focused at a very tiny spot (tens of μm) and controlled very easily under in-situ conditions. NIR laser ($1450\text{ nm} \pm 15\text{ nm}$ wavelength) is absorbed by the water due to the O-H band. Therefore, we heated the water at a tiny spot in the IV set tube using focused NIR laser with a 50 mm lens. Figure 4 shows the absorption spectra for different flow rates at the 0-mm position with IR laser heating. The absorption intensity for laser heating at 1450 nm did not exhibit a change for different flow rates from 1 to 100 mL/h. This implies that the heating value from the laser absorption is fixed with the same laser energy and position irrespective of the flow rate difference. However, the absorption spectra from 1500 nm to 1700 nm changed for different flow rates owing to the temperature change. The intensity of absorption spectra from 1500 nm to 1700 nm decreases when the flow rate decreases. Thus, we obtain the fluid temperature by using the intensity of absorption spectra from 1500 nm to 1700 nm from the calibration curve (Fig. 3) between the temperature and the absorption spectra with changes in the flow rate.

Figure 5 shows the temperature profile according to the position of the tube for different flow rates when the laser is heated at the 0 mm position. The temperature was obtained from the NIR spectra through the calibration curve shown in Fig. 3. When the flow is low, the temperature profile is symmetric according to the positions. However, the temperature profile shifts toward the downstream position with increasing flow rate. This is because the heated water is moved to the downstream. The temperature profile was almost similar to that of the thermal mass flow meter [16]. The temperature profile exhibits a significant change when the flow rate is low (1-20 mL/h) and a relatively low change when the flow rate is high (40-100 mL/h). Additionally, the change in the temperature profile in the upstream region exceeds that in the downstream region in the low flow rate (1-20 mL/h). Conversely, in high flow rates (40-100 mL/h), the change in the temperature profile is relatively higher in the downstream region when compared to that in the upstream region. This tendency is caused by the heat transfer characteristics of different flow rates. The conductive and convective heat transfers dominantly influence the temperature change in the low and high flow rates, respectively.

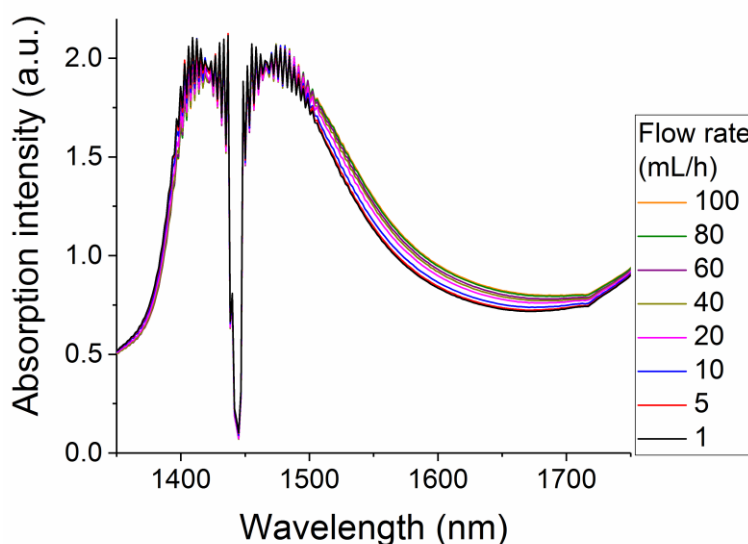


Fig. 4. Absorption spectra for different flow rates at the 1 mm position with IR laser heating.

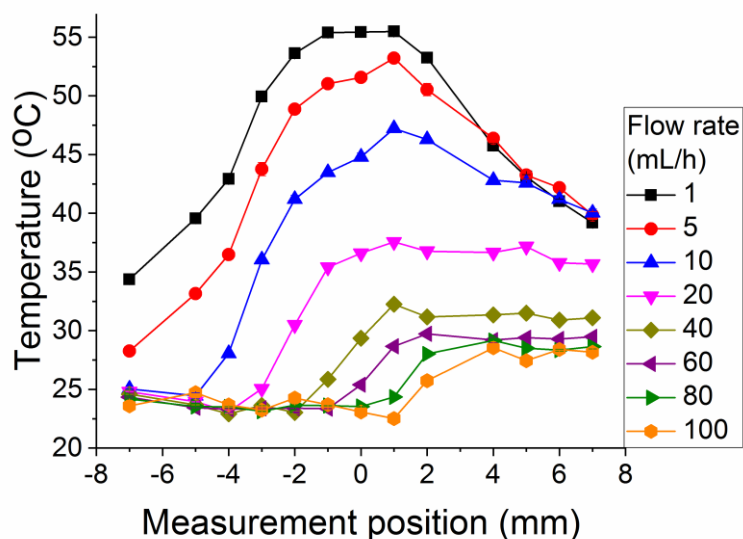


Fig. 5. Temperature profiles according to the position of tube for different flow rates (1–100 mL/h) with IR laser heating at the 0 mm position

3.3 Quantitative flow rate measurement using NIR absorption method

The flow rates were related to the temperatures obtained from the NIR absorption method with laser heating. Figures 6(a) and 6(b) show the difference in room temperature (T_{room} , 22.5 °C) and the temperature (T) obtained from the NIR absorption method based on the flow rate for different positions (a) upstream and (b) downstream with IR laser heating at the 0-mm position, respectively. With respect to the -1-mm measurement position, an increase in the flow rate linearly increases the difference in temperature up to approximately 20 mL/h as shown in Fig. 1(a). However, the slope of the temperature difference gradually decreases when the flow rate exceeds 20 mL/h, and the slope of the temperature

difference finally corresponds to '0' at a flow rate of 100 mL/h. When the flow rate is small, the effect of conduction is dominant, but as the flow rate increases, the influence of convection. Additionally, as the measurement position moves away from the laser irradiation position toward the downstream direction, the flow rate at which the temperature difference slope is zero decreases and the temperature difference at the lower flow rate also decreases. The reason for this is that the effect of conduction decreases when the measurement position moves away from the laser irradiation point. To improve the sensitivity of the low flow rate measurement, it is necessary to measure the same at a close range of upstream laser irradiation.

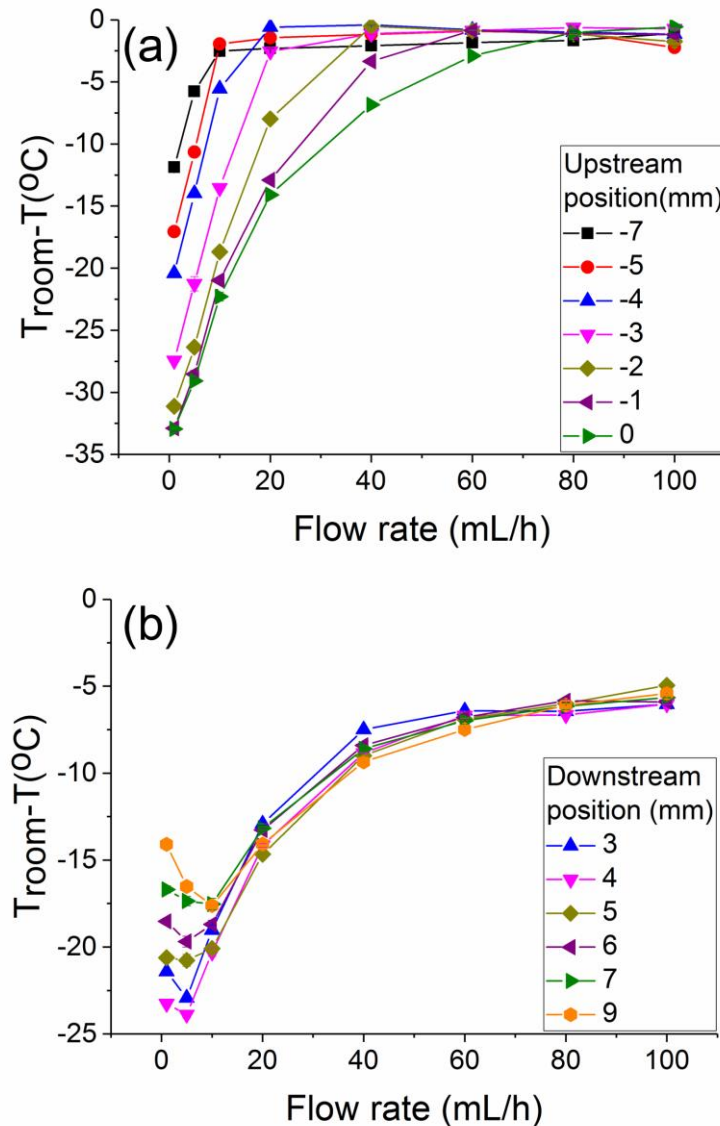


Fig. 6. Difference between room temperature (T_{room} , 22.5 °C) and temperature (T) according to the flow rate for different positions (a) upstream and (b) downstream with IR laser heating at the 0 mm position

In the case of the downstream position, an alternative tendency is observed as shown in Fig. 6(b). It is observed that the temperature difference varies based on the measurement position prior to 20 mL/h

due to the conduction effect in Fig. 6(b). After 20 mL/h, the temperature difference reaches the equilibrium state and exhibits a similar value based on the flow rate irrespective of the measurement position. At this regime, the convective heat transfer is a dominant factor. The slope of the temperature difference decreases when the flow rate increases. The downstream position (1 mm and 2 mm) near the laser heating position was affected by both conduction and convection heat transfer, and the temperature beyond the 3 mm downstream position was mainly affected by convection heat transfer. So the temperature at 1 mm and 2 mm and the temperature difference after 3 mm exhibit different tendencies depending on the flow rate.

Figure 7 shows flow rate according to the difference in room temperature and temperature (T) obtained from the NIR absorption method at 1 mm position with exponential curve fitting for different flow rate ranges (1-20 mL/h and 40-100 mL/h). The temperature difference exhibits two exponential curves based on the flow rate ranges (1-20 mL/h and 40-100 mL/h). The two flow rate ranges (1-20 mL/h and 40 mL/h) are significantly influenced by conduction and convection, respectively as shown in Fig. 9. The sensitivity of the flowrate measurement decreases with increasing flow rate. This is because the temperature difference is lower at high flow rates than at low flow rates. The two fitting curves are used for the flow rate calibration for the difference in flow rate ranges. We measure the quantitative flow rate by using the calibration curve.

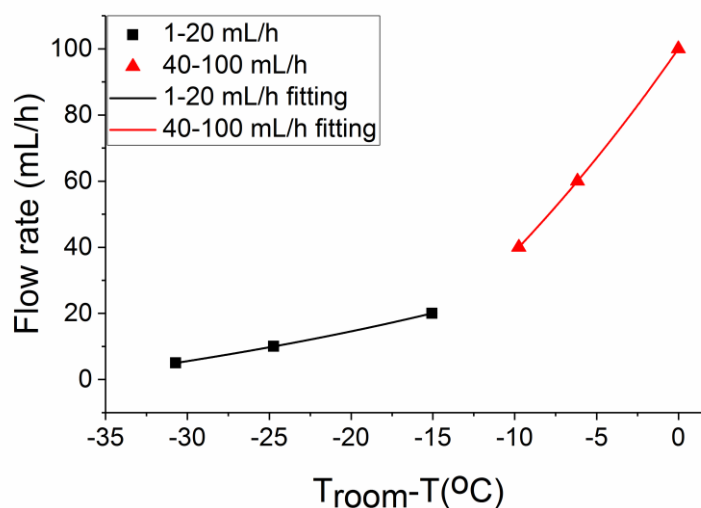


Fig. 7. Flow rate according to the difference of room temperature (T_{room} , 22.5 °C) and temperature(T) at 1 mm position with exponential curve fitting for different flow rate ranges (1-20 mL/h, 40-100 mL/h)

Figure 8 shows the error of NIR absorption method according to the flow rate with measurement uncertainty. The reference flow rate was obtained from the gravimetric flow standard system in KRISS in table 1. The error is calculated using below equation.

$$\text{Error} = \frac{(q_{\text{NIR}} - q_{\text{REF}})}{q_{\text{REF}}} \times 100 \quad (5)$$

q_{NIR} and q_{REF} are the flowrates obtained from the NIR absorption method and the gravimetric micro flow standard system, respectively. An error of 1.12 % (0.05 mL/h) was observed at a flow rate of 5 mL/h and 0.39 % (0.39 mL/h) at a flow rate of 100 mL/h. The error according to the flow rate is less than 1.2 % and the uncertainty is less than 1 % at the 1-100 mL/h flow rate.

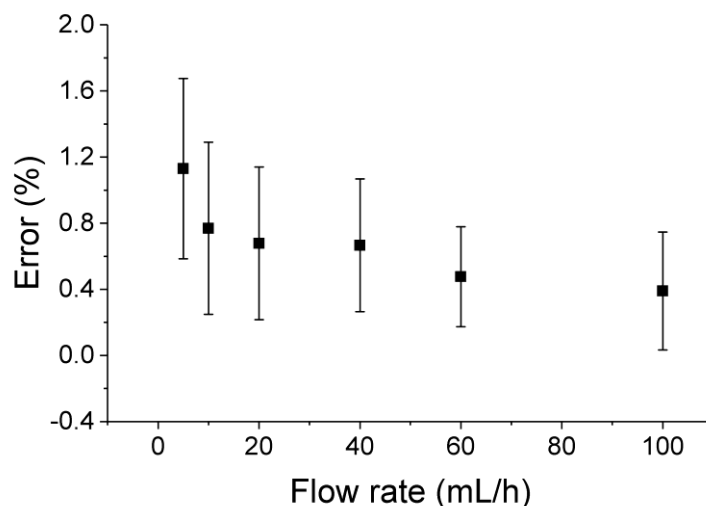


Fig. 8. Error of NIR absorption method according to the flow rate with measurement uncertainty

To date, this is the first study that measures the flow rate in-situ by using the NIR absorption method. The flow rate is measured by irradiating water with an IR laser to heat it, and then measuring the water temperature by using FTNIR. The developed flow measurement method measures a wide range of flow rates from 1 to 100 mL/h. It is possible to measure a low flow rate (1–20 mL/h) with higher resolution when the measurement position is upstream of the laser irradiation position, and it is more suitable to measure a large flow rate (20–100 mL/h) when the measurement position is downstream of the laser irradiation position. The calibration curve for temperature difference was obtained based on different flow rate ranges (1–20 mL/h and 40–100 mL/h). Hence, the results confirmed that the NIR absorption method quantitatively measures the flow rate in the in-situ condition. The developed method can be used in various fields including the medical field (monitoring drug injection) and semiconductor field by measuring the flow rate using only an external light source without having to cut the pipe and without having to install a flowmeter separately.

4 Conclusion

In-situ measurement of micro flow rates was conducted using the NIR absorption method. We used the IR absorption method to measure the temperature under in-situ conditions instead of using a contact temperature sensor. The calibration curve between the temperature and the NIR absorption intensity was obtained. An NIR laser (1450 nm) was irradiated through a convex lens to heat water in a tube. The temperature profiles were obtained by the NIR absorption method via laser heating for different flow rates. The shape of the temperature profile changes resulting from conduction and convection heat transfer were presented based on the range of flow rates. The temperature difference between room temperature (T_{room} , 22.5 °C) and the temperature (T) obtained from the NIR absorption method according to the flow rate showed different tendencies upstream and downstream of the heating

position. The calibration curves between the flow rate and the difference in room temperature (T_{room} , 22.5 °C) and the temperature (T) obtained from the NIR absorption method exhibit two exponential curves based on the flow rate ranges (1–20 mL/h and 40–100 mL/h). The error and the uncertainty of the NIR absorption method were approximately 1.2 % and 1% in the 1–100 mL/min flow rate range, respectively. Thus, we confirmed for the first time that the NIR absorption method quantitatively measures the flow rate in the in-situ condition.

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