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Detection of critical micelle concentration (CMC) using uniform and U-shaped optical fiber in sensing region

Faculty of Engineering, Shizuoka University, 3-5-1 Johoku, Hamamatsu 432-8561 Shizuoka, Japan

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ABSTRACT In this paper the critical micelle concentration (CMC) in surfactant solutions was detected by using an optical fiber. CMC detection is based on an adsorption effect in sample solution. The incident beam passing through the sensing region of the fiber reflected at the interface between the fiber core and the solution with repeating reflections. The change in adsorption condition leads to an effective change in the refractive index at the surrounding surface of the core. The output signal suddenly increases at the CMC due to the reflectivity change caused by adsorption of surfactant molecules onto the surface of the fiber. Plastic clad silica (PCS) fiber with a uniform sensing region was used to measure the CMC point in surfactant solution. It has been found that sensitivity of the sensor depends on the length of the sensing region, the launching condition of the light source and the optical fiber material. The higher the sensing length, the higher the sensitivity of the sensor. The U-shaped sensing region also tried to find the CMC point, and the experimental results indicate that the U-shaped sensing region is more sensitive than the uniform sensing region. Using this method we are able to detect the CMC of the surfactant solution very accurately and in real time.

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1 Introduction

Recently there has been considerable interest in the development of optical fiber evanescent wave adsorption sensors for measurements of critical micelle concentration (CMC) points for surfactant solutions. This is because they have several advantages such as long interaction lengths and rugged constructions [1–7]. Many kind of surfactants have been used in the food, chemical, cleaning and machining industries. Solutions containing surfactants exhibit drastic changes in their physical and chemical properties, such as detergency, conductivity, surface tension and change suddenly [8]. At this point the concentration of the surfactant are called critical micelle concentration (CMC). Detergency increases rapidly near CMC, after the CMC the detergent activity saturates and reaches a point where it stays constant even if the concentrations increase [4, 5, 7]. Hence the most economical concentration can be found by the detection of CMC.

✉ Fax: +81-53-478-1101, E-mail: cdsingh@yahoo.com

At low concentrations surfactant molecules disperse in the solution. They are gathering and scattering, and some of them intend to adsorb onto the core surface with the concentration. They are sparsely adsorbed onto the surface. At this time the output from the fiber reduces slightly (due to absorption and scattering effect, leaky rays, etc.). When the concentration of the surfactant solution reaches the CMC, the outputs from the fiber suddenly begin to increase due to the interaction between evanescent wave and surfactant molecules [5, 6]. Above CMC the surfactant molecules are adsorbed uniformly on the removed surface of the core in the solution. This is due to fact that after the surfactant solution becomes saturated, the reflectance at the interface between the fiber core and the surfactant solution begins to increase as surfactant molecules adsorb onto the surface of the sensing region (due to core surface smoothing, reduced of refractive index, less scattering effect, etc.). When the concentration reaches the CMC, the output suddenly increases. The CMC detection in this paper is performed using the rapid increase in output powers. It has been shown that the long interaction length and the U-shaped sensing region increase the sensitivity of the sensor.

2 Experiment

The experimental setup for design 1, in which the uniform sensing region of the fiber is used shown on the next page (see Fig. 1a). The beam from a He–Ne laser operating at 632.8 nm was focused using a beam splitter at the input end of a 1.0 mm core diameter plastic clad silica (PCS) fiber of NA equal to 0.45. The refractive index of the core and cladding are 1.450 and 1.380, respectively. The length of the fiber used in the experiment was 30 cm. A small length of the cladding from the middle portion of the fiber was removed. This unclad region of the fiber was the sensing region of the sensor. The unclad sensing region of the fiber was then inserted into a plastic cell and both the ends of the cell were sealed. The fluid used for the experiment was sodium dodecylbenzenesulfonate dissolved in deionized water. The value of CMC in the surfactant solution is considered to be $1.2-3.0 \times 10^{-3}$ mol/l from the obtained data depending on the location in bonding site of benzene and sulphonate in the group. The plastic cell was mounted on a rotation stage such that the input end of the fiber positioned on the axis of the rotation stage. Increasing the concentration of the sample solution, the light emerging from the output end of the fiber was measured by the power

FIGURE 1 a Experimental setup for design 1, **b** Experimental set up for U-shaped probe (design 2)

meter whose detector head was also attached to the rotation stage.

The experimental setup for design 2 is similar to design 1 except that in this case U-shaped sensing region was used (see Fig. 1b). To bend the unclad portion of the sensing region, we first exposed it to a flame and then slowly bent it until it became the desired U-shaped [10]. The temperature of the flame was in the range $800-1000$ °C. Proper mixing of liquid petroleum gas and oxygen controlled the temperature of the flame. The uniformities of the core diameter and the bending radius were checked using a traveling microscope. The experiments were performed only on those probes that were uniform in core diameter and where the bend was close to Ushape.

3 Results and discussion

The variation of output power with the concentration of the surfactants for design 1 is shown in Fig. 2 using different sensing lengths i.e., 1 cm, 6 cm and 10 cm of the

120 115 Sensing length 110 Output (arb. unit) 10_{cm} 105 6 cm 1_{cm} 100 95 90 10^{-3} 10^{-2} 10^{-5} 10^{-1} 10^{-4} Concentration (mol/l)

FIGURE 2 Output power vs. surfactant concentration for PCS fiber with sensing length of (a) 10 , (b) 6, (c) 1 cm long

PCS fiber. It can be seen from Fig. 2 that the change in output power in case of 1 cm is lower than 6 cm and 6 cm is lower than 10 cm sensing length. This is due to the micelle formation surrounding the fiber core. The roughness finish of the core surface is improved and the refractive index of the sensing region is decreased due to effect of the micelle formed by surfactant solution near the fiber core in the sensing region. This result implies that the sensor based on the large sensing length is more sensitive than the sensor based on the small sensing length. To see the effect of the U-shape probe and its bending radius on the sensitivity of the sensor, we carried out experiments with three different bending radii of U-shaped i.e., 1.5, 2.5 and 3.0 cm. The experimental results obtained for U-shaped designs are shown in Fig. 3. Two points may be noted from the figure. First, the value of output power slightly decreases or remains nearly constant as the concentration of the surfactant solution increases. The measured output power increases drastically at 1.3×10^{-3} mol/l for all three cases. This is due to the fact that after the surfactant solution becomes saturated, the reflectance at the interface between the fiber core and the surfactant solution begins to increase as excluded molecules adsorb onto the surface of the sensing region. At this point the con-

FIGURE 3 Output power vs. surfactant concentration for different bending radii of the U-shape probe

centration of the solution should be the CMC. It means that CMC point in the case of sodium dodecylbenzensulfonate is 1.3×10^{-3} mol/l. Second, Fig. 3 also implies that the sensor based on the low bending radius of the U-shape probe is more sensitive than the sensor based on the high bending radius because change in the output power is more in the case of the low bending radius after the CMC point. After the CMC point, for a given concentration, the decrease in the bending radius increases the value of output power and hence the sensitivity of the sensor. We have also found that the change in the fiber loss maximum near the critical angle of the fiber in the case of the uniform core sensing region and the selected rays launching closer to the critical angle of the fiber gives three times higher sensitivity as compared to all bound rays. In [5, 6] they have done experiments for the cationic (Dodecyltrimethyl-ammoniumchloride) and nonionic (Heptaethylene-glycol-dodecyl-ether) surfactants and found that the principals are true for the both cases. This shows that for higher sensitivity and effective CMC measurement, U-shaped probes which make the launched light angle closer to the critical angle of the sensing region should be used.

REFERENCES

- 1 T. Takeo, H. Hattori: Jpn. J. Appl. Phys. **21**, 1509 (1982)
- 2 J.Y. Ding, M.R. Shahriari, G.H. Sigel, Jr.: Electron. Lett. **27**, 1560 (1991) 3 G.E. Badini, K.T.V. Grattan, A.W. Palmer, A.C.C. Tseung: Springer Proc. Phys. **44**, 436 (1989)
- 4 M. Ogita, K. Yoshimura, M.A. Mehta, T. Fujinami: Jpn. J. Appl. Phys. **37**, 85 (1998)
- 5 M. Ogita, K. Yoshimura, T. Fujinami: Measurement of concentration and adsorption phenomenon using optical fiber In: Proc. 23rd Chem. Sensor Symp. **12**, 45 (1996)
- 6 M. Ogita, T. Hasegawa, M.A. Mehta, T. Fujinami, Y. Hatanaka: Industrial utilization of the adsorption effect of optical fibers for detection of critical micelle concentration. In: Proc. IECON 701 (2000)
- 7 M. Ogita, Y. Nagai, M.A. Mehta, T. Fujinami: Sens. Actuators B **64**, 147 (2000)
- 8 M.J. Rosen: *Surfactants and Interfacial Phenomenon* (Wiley, New York 1987) p. 83
- 9 M. Archenault, H. Gagnaire, J.P. Goure, N. Jaffrezic-Renault: Sens. Actuators B **8**, 161 (1992)
- 10 B.D. Gupta, H. Dodeja, A.K. Tomar: Opt. Quantum Electron. **28**, 1629 (1996)