다이오드 레이저 비색 분광기를 이용한 Cetylpyridinium Chloride의 농도분석

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Application of a Diode Laser Colormetric Spectrometer to Determination of Cetylpyridinium Chloride

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요 약 본 논문은 다이오드 레이저와 광 다이오드를 사용한 이중 빗살형 다이오드 레이저 비색 분광기를 개발하여 항균 성분의 양이온 계면 활성제로 널리 사용되고 있는 cetylpyridinium chloride(CPC)의 농도를 측정하였다. 분광기의 안정도는 광원의 세기, 감도, 재현성을 측정한 예비 실험을 통하여 검증이 되었다. 또한 상용화된 UV/VIS 분광기와의 비교 결과를 나타내었다. 다이오드 비색 분광기는 3×10^{-5} M에서 1.1×10^{-4} M의 CPC 농도 범위에서 0.9635의 상관계수를 나타내었다. 이러한 결과는 CPC의 농도 분석을 위한 간편한 다이오드 레이저 비색 분광기 개발의 가능성을 나타내었다.

1. Introduction

Cetylpyridinium is classified as a cationic surfactant and is one of the very important surfactants that is widely used in industrial and pharmaceutical substances, especially in manufacturing of dermal ointments, drugs and cosmetics. The formation of ternary chelate compound of CPC with bromopyrogallol red (BPR) and strontium is accompanied by a marked increase in the absorbance and a bathochromic shift in the maximal absorption of the complex from 555 nm to 627.5 nm. On the basis of the color change of CPC from red to blue, the colometric quantification of CPC was achieved [1].

The visible diode laser of 630-670 nm wavelength has been recently and widely used in optical spectroscopy because the light source in the region has a long lifetime, power stability, low power consumption, small size, and low price. Even though output power of a diode laser is usually limited to 3-30 mW, is comparable to the power of a xenon arc radiation passing through the monochromator in a con-

Since the emitting wave-length is agreed with the absorption maximum of blue colored CPC, ternary chelate compound of CPC with BPR and strontium, the developed system was applied to the analysis of CPC.

In the paper, we describe the structure of the compact and simplified spectrometer with a diode laser of 633-nm radiation, double beam system, and photodiode detector. And also evaluate its performances, comparing data of the developed system (Diode Laser-Double Beam Colometric System, DL-DBCS) with those of the UV/VIS spectrometer.

2. Experimental

2.1 Instrumentation

The system is depicted in Fig. 1. This system features a diode laser, a double beam system, and a photodiode detector. The 633 nm diode laser (LDM 145/633/5, Imatronic Co., Ltd., USA) was used. The laser beam was focused by a convex lens (F.L.=6 cm). The beam splitter separated a signal beam and

ventional UV/VIS spectrometer [2]. Therefore, a diode laser could replace the radiation source and a monochromator in the conventional UV/VIS system, and simplify the development of a practical and specific analytical instrument [3-8].

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a reference beam from laser beam. A simple photodiode (Pin-6DP, UDT Sensors Inc., USA) was used for detection. The signal was amplified and acquisition to the RS232C port of notebook-PC using A/D converting system(Home Made). In order to compare the results obtained from conventional method, a UV/VIS spectrophotometer (UV-2101PC, Shimadzu, Japan) was used. The line profile of the diode laser was measured by using a conventional grating and a CCD detector (SD-1000, Ocean Optics, USA).

2.2 Reagents and Procedure

Cetylpyridinium chloride, bromopyrogallol red and strontium(II) were from Aldrich (Analaytical grade) and used without further purification. A 1×10^{-2} M cetylpyridinium chloride stock solution was prepared by dissolving 895.0 mg of cetylpyridinium chloride in distilled water. The required cetylpyridinium chloride standard solution was prepared daily by dilution of the stock solution. A strontium (II), bromopyrogallol red and buffer solution were prepared respectively 1×10^{-2} M, 1×10^{-3} M and 1×10^{-2} M. In order to obtain a calibration curve, 4 ml of each standard solution, 0.4 ml of strontium solution, 0.4 ml of bromopyrogallol red, and 0.2 ml of buffer solution were vigorously mixed. The blue color formation of ternary chelate compound was completed in 20 minutes.

Results and Discussion

3.1 Radiation Profiles of the Diode Laser

In Fig. 2, solid line is a radiation profile of the

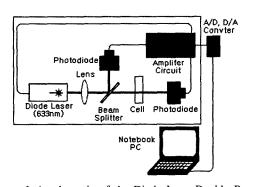


Figure 1. A schematic of the Diode Laser-Double Beam Colorimetric system (DL-DBCS)

diode laser between 450 nm 750 nm wave-length obtained by conventional grating and CCD detector. Doted line is the absorption spectrum of ternary chelate compound measured by conventional UV/VIS spectrophotometer.

The solid line forms a Gaussian shape whose peak is located at 633 nm and whose width of the half maximum is 29 nm. The wavelength at the peak of the diode laser is well agreed the narrow bandwidth is included in the broad absorption spectrum of colored ternary chelate compound. These characteristics suggest that the diode laser can be applied to molecular spectroscopy of detecting ternary chelate compound.

3.2 Stability of the DL-DBCS

In Fig. 3, solid line is the trace of absorbance of the DL-DBCS according to time and dots is that of the UV/VIS spectrometer when the sample cells have the standard solution. In the conventional system, the base line is stable and located at zero absorbance. On the other hand, in the DL-DBCS, the base line is slightly unstable and oscillates. It is believed that this slight unstableness probably occurs by a change of hole-electron pairs according to temperature changes in the diode laser. The noise, however, is smaller in the DL-DBCS than in the conventional system and the oscillation is within the noise level of the conventional system. Consequently, the base absorbance by the system is

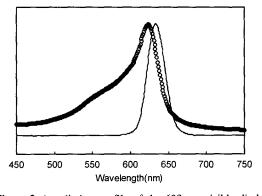


Figure 2. A radiation profile of the 633 nm visible diode laser (−) and absorption spectrum of ternary chelate compound of CPC (○) between the wavelength of 450 nm and 750 nm

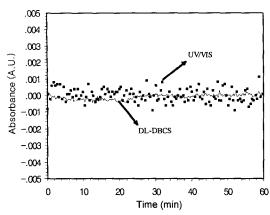


Figure 3. Stability comparison between DL-DBCS (−) and conventional UV/VIS (■)

expected to give no effect on measuring the absorption of ternary chelate compound.

3.3 Calibration and Evaluation of the Instruments

Fig. 4 shows the calibration curves of ternary chelate compound for conventional UV/VIS and the DL-DBCS. In the DL-DBCS, with the increment of the concentration of cetylpyridinium chloride from 3.1×10^{-5} M to 1.1×10^{-4} M, the absorbance increases linearly with a slope of 7785.2 AU/M and intercept of 1.153×10^{-1} , and an correlation coefficient of 0.9635. While, for the conventional UV/VIS spectrophotometer, slope, intercept, and correlation coefficient is 16219.6 AU/ M, 1.539×10^{-1} , and 0.9906 respectively.

From these results, we anticipate that the developed system has enough possibility to be utilized in practice.

4. Conclusions

In spite of its simple and inexpensive structure, the Diode Laser-Double Beam Colorimetric System reveals the good performances to determine the

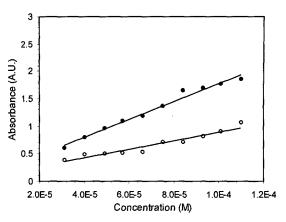


Figure 4. Calibration curve for CPC using DL-DBCS(○) and conventional UV/VIS(●)

cetylpyridinium. This instrument also can be introduced to detection of other chemical compound if blue color develops in the solution of chemical reaction. In addition, other chemical species could be detected by the colorimetry, if the laser diode was replaced by other laser diode whose radiation wavelength matches up to the color of the compounds. These studies are in progress in our laboratory.

References

- [1] M. Benamor, M. Aquersaf, MT. Draa, J. Pharm. Biomed. Anal., 26: 151, 2001.
- [2] Kaneta, T., and Imasaka, T., Anal. Chem. 67: 829, 1995.
- [3] Imaska, T., Spectrochimica Acta Rev., 15: 329, 1993.
- [4] Niemax, K., Zybin, A., Schn rer-Patschan, C., and Groll, H., Anal. Chem., 68: 351A, 1996.
- [5] Mank, A. J. G., Lingeman, de NIJS, Lingeman, H., Brinkman, U.A. Th., Velthorst, N. H., and Gooijer, C., Appl. Spec., 50: 28, 1996.
- [6] Imaska, T., Anal Sci., 9: 329, 1993.
- [7] Kim, S. H., Shin, C. M., and Yoo, J. S., *Bull. Kor. Chem. Soc.*, 17: 536, 1996.
- [8] Kim, S. H., Hong, S., Instru. S & T, 29: 101, 2001.