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Simultaneous Measurement of Multi-solute Concentrations in Mixed Solution Using Fiber Fabry-Perot Resonator Interference Transmission Wavelengths*

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Abstract: In order to simultaneously detect the multi-solute concentrations in mixed solution, system for measuring the multi-solute concentrations in mixed solution using optical Fiber Fabry-Perot Resonator (FFPR) is proposed. The relation between FFPR interference transmission wavelength and concentrations of the mixed solution is analyzed theoretically. The measuring system consists of an InGaAs LED, fiber coupler, FFPR sensor, opto-electrical convertor, amplifier and spectrometer. The analytical equation between concentrations and interference transmission wavelengths is standardized by measuring the samples contain ethanol and glycerin. The action script 2. 0 is used as the programming language according to the mathematical relation. The real-time variation of the concentration change is monitored by the PC.

Key words: Mixed solution; Concentrations of multi-solute solution; Fiber Fabry-Perot Resonator (FFPR) sensor; Interference transmission wavelength; Spectrometer

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0 Introduction

The concentration by mass of the solution is a key parameter of the physical property of the solution. In the industrial field, the concentrations must be monitored very precisely. Using the relation between concentration and its refractive index, the concentration can be measured by optical means^[1-4]. The rapid development of fiber technique gives the fiber sensor a boom in many application field^[5-7], as well as the applications in concentration measurements^[8-10]. The previous mono-solute reports are on concentration measurements, but the need for measuring the multi-solute concentrations is required in many chemical industries. In this paper a measurement of multi-solute concentrations in mixed solution using FFPR was proposed. The measuring system consists of a InGaAs LED, fiber coupler, FFPR sensor, Opto-electrical convertor, amplifier and The analytical equation between spectrometer. concentrations and interference transmission wavelengths was standardized by measuring the samples contain ethanol and glycerol. The degree of accuracy can be within the magnitude of 0.001%.

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1 Principle and measuring system

1.1 Principle

The refractive index of a solution can be expressed as the function of the concentration c, temperature T and wavelength $\lambda^{[11]}$

$$n = N(c, T, \lambda) \tag{1}$$

Thus the variation of the refractive index in Eq. (1) can be written as

 $\Delta n \cong (\partial N/\partial c) \Delta c + (\partial N/\partial T) \Delta T + (\partial N/\partial \lambda) \Delta \lambda$ (2) The temperature usually has little influence on refractive index, and the typical $\partial n/\partial T$ value is approximately 1×10^{-4} °C⁻¹. If the temperature variation ΔT during concentration test is about 1 °C ~2 °C, it can be ignored, thus Eq. (2) can be approximated as

$$\Delta n \cong (\partial N / \partial c) \Delta c + (\partial N / \partial \lambda) \Delta \lambda \tag{3}$$

As we derive from the principle of the F-P interferometer^[12], the interference transmission wavelength satisfies

$$2nd = k\lambda$$
 (4)

where *n* is the refractive index, *d* is the cavity length of the F-P resonator, and *k* is the interference order. According to Eq. (4), when *d* is invariant, the interference transmission wavelength λ is the variable of the solution refractive index *n*, thus also is the variable of the concentration *c*.

The relation between variations of refractive index and of interference transmission wavelength is

$$\Delta n = (k/2d) \Delta \lambda \tag{5}$$

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Substitute Eq. (5) into Eq. (3), we get

 $\Delta c = (k/2d - \partial N/\partial \lambda) (\partial N/\partial c)^{-1} \Delta \lambda$ (6) Integrate both sides of Eq. (6), we derive the relation between concentration and wavelength

$$c(\lambda) = \int_{c_0}^{c} \Delta c = \int_{\lambda_0}^{\lambda} (k/2d - \partial N/\partial \lambda) (\partial N/\partial c)^{-1} d\lambda \quad (7)$$

Thus if we get the interference transmissions wavelength of the FFPR, the concentrations of the multi-solute solution can be calculated.

1.2 Measuring system setup

The measuring system is shown as Fig. 1. The incident light is coupled to the FFPR, The sensor of the FFPR is submerged in to the solution under test, and the interference transmission light from FFPR transmit to the spectrometer.



Fig. 1 Schematic diagram of the measuring system

The spectrometer analyzes the interference transmission light, the peak values in the spectrum are obtained, and using Eq. (7), with a computer installed with Action Script 2.0, the concentrations of the multi-solute solution can be calculated and monitored in real-time.

The schematic diagram of the FFPR sensor is shown as Fig. 2. Two conventional multiple mode fibers with diameter of 125 μ m are cut and coated with high reflective film, then inserted into a capillary with diameter of 250 μ m, and the end facets of the fiber form a F-P resonator (cavity long $d=11.33\pm0.01 \mu$ m). The fiber and capillary are sealed with CO₂ laser, and the liquid solution passes through the capillary via two small holes, of which the diameter about is $8.0\pm0.1 \mu$ m.





The light source is an InGaAs LED with spectrum width of 1 200 \sim 1 700 nm. The resolution of the spectrometer (Advantest Q8383) is 0. 01 nm, and the interference transmissions light of FFPR has is multiple wavelength peak values. Take the light intensity maximum shorter wavelength monitoring.

2 Standardization of the relation between solution concentration and transmission wavelength

Eq. (7) implies the relation between solution concentration and FFPR interference transmission wavelength. But $\partial N/\partial \lambda$, $\partial N/\partial c$, k and d are necessary to be known to calculate the solution concentration, which is impractical in real applications. Thus the standardization of the measuring system is needed to obtain a simple analytical expression for the relation between concentration and wavelength.

A solution including ethanol and glycerin is used for standardization. The mixed solution is prepared with the expression $c = [m_1 / (m_1 + m_2)] \times$ 100%, in which m_1 and m_2 are the mass of the solute and solvent respectively. With the ambient temperature of (20 ± 1) °C, and using electronic balance weighing (Sartorius BS300S-WE1), the instrumental error of the balance is 10^{-4} g, corresponding to the maximum error in the standard solution of $\pm 0.0001\%$, which is far beyond the measurement demand. The preparation method of the solution: such as solution 1, first using solute ethanol (m_1) 10 g, and solvent water 90 g(m_2) mixed. Then using 0.5 g glycerol(m_1) as solutes, and with the ethanol and water mixed solution 99.5 g (m'_2) as solvent mix again. Get mixed solution 1. The rest mixed solution preparation methods similar.

Nine mixed solution of ethanol and glycerin are prepared, the measured transmission wavelengths of the solutions are shown in Table 1.

Table 1 Measuring concentration of mixed standard solution and regression data($T=20^{\circ}C$)

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Solution sequence	1	2	3	4	5	6	7	8	9
Ethanol standard concentration/($\frac{0}{0}$)	10.000 0	15.000 0	20.000 0	25.000 0	30.000 0	35.000 0	40.000 0	45.000 0	50.000 0
Glycerin standard concentration/($\%$)	0.500 0	1.000 0	1.500 0	2.000 0	2.500 0	3.000 0	3.500 0	4.000 0	4.500 0
Transmission wavelength/nm	1 515.19	1 517.63	1 520.00	1 522.31	1 524.55	1 526.72	1 528.84	1 530.91	1 532.91
Regression concentration(E)/($\frac{0}{0}$)	10.034 2	15.012 4	19.999 3	25.012 5	30.017 7	35.001 7	39.999 5	45.001 8	49.995 8
Regression absolute error(E)/($\frac{1}{2}$)	0.034 2	0.012 4	-0.0007	0.012 5	0.017 7	0.0017	-0.0005	0.001 8	-0.0042
Regression concentration(G)/($\frac{0}{0}$)	0.480 5	0.982 0	1.482 1	1.992 0	2.498 4	3.000 1	3.500 9	4.000 1	4.491 9
Regression absolute error(G)/($\%$)	-0.0195	-0.0180	-0.0179	-0.0080	-0.0016	0.000 1	0.000 9	0.000 1	-0.0081

The fitting curve of the solution concentrations and transmission wavelengths are plotted in Fig. 3. We can see that the curve is not



Fig. 3 Regression curve of mixture solution test data linear, thus we use the quadratic as the fitting equation

$c(\lambda) = a\lambda^2 + b\lambda + b\lambda$	C_0	(8)
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where $C(\lambda)$ is the concentration by mass of the solute in the mixed solution, a,b,C_0 are the fitting coefficients, and λ is the interference transmission wavelength. The nine groups of solution are used to fit the coefficients and the resultant coefficients for ethanol are: $a=0.014\ 125\ 45,b=-40.803\ 77$, $C_0=29\ 406.29$. and for glycerinare: a=0.001170473, $b=-3.340\ 211$, $C_0=2\ 374.\ 342$, the fitting equation are shown in Eq. (9) $(c_{\rm E}=0.014\ 125\ 45\lambda^2-40.803\ 77\lambda+29\ 406.29$

 $\begin{pmatrix} c_{\rm E} = 0.014 \ 125 \ 45\lambda^2 - 40.803 \ 77\lambda + 29 \ 406.29 \\ c_{\rm G} = 0.001 \ 170 \ 473\lambda^2 - 3.340 \ 211\lambda + 2 \ 374.342 \end{cases}$ (9)

Eq. (9) implies the relationship between solute concentrations and transmission wavelength of the FFPR $c = C(\lambda)$. The measured transmission wavelength is substituted into equation Eq. (9) for the regressive calculation, and the results are shown in Table 1. The average absolute error of the ethanol solution is S = 0.0095%, while of the glycerin solution is S=0.0082%. We can get the regressive coefficient of R=0.99998, which certify the stability of the standardization of Eq. (9)

3 Results and discussions

3.1 Measurement test

The real measurement was carried out for 7 groups of ethanol and glycerin mixed solutions, the ambient temperature was also 20° C, and the results are shown in Table 2.

Solution sequence	1	2	3	4	5	6	7		
Ethanol solute/(%)	6.970 0	12.100 0	21.500 0	26.450 0	33.200 0	40.600 0	50.200 0		
Glycerol solute/(%)	0.150 0	0.700 0	1.650 0	2.120 0	2.820 0	3.600 0	4.500 0		
Transmission wavelength/nm	1 513.64	1 516.21	1 520.66	1 522.94	1 525.99	1 529.12	1 533.01		
Ethanol concentration measurements/($\frac{9}{0}$)	6.975 5	12.100 7	21.416 2	26.405 9	33.310 3	40.669 0	50.200 3		
Glycerol concentration measurements/($\%$)	0.142 8	0.672 6	1.626 5	2.133 2	2.830 1	3.567 9	4.516 7		

Table 2 Measurement test data(T = 20°C)

The test results were monitored by the computer, and the monitored concentration variations of the ethanol and glycerin are shown in Fig. 4. Fig. 5 shows the average absolute error of the system, with the ethanol of S = 0.045% (or 0.45 g/L) and glycerin of S=0.019% (or 0.19 g/L), and the measured results are stable and reliable under the consideration of the random error.





(b) Glycerol concentration monitoring variations





Fig. 5 Monitoring system application error distribution

3.2 Error analysis of the system

The intensity fluctuation of the light source has no impact on the measured results since the measured wavelengths are the parameter for determining the results, while the light source we used are quite stable.

Mainly, the system measurement error are caused by the resolution limit of the spectrometer, and the approximation made for the coefficients among the equations. The resolution of the spectrometer is 0.01 nm, which would cause the maximum error and minimum error to 0. 022 3%and 0. 001 2% respectively according to the fit equation of concentration and interference transmission wavelength. Such errors are randomly distributed and can be minimized with multiple measurement results. The error caused by the coefficient approximation can be minimized by increasing the coefficient digits.

Other methods can be implemented to further improve the stability and accuracy of the system: 1) Using high accurate tunable power source to stabilize the working voltage; 2) Improve the algorithm to minimized the random error; 3) Improve the fitting model, such as subsection fitting, or increase the point number during fitting.

4 Conclusion

Measurement of the multi-solute concentrations of in mixed solution using FFPR employs the advantages of conventional fiber sensor system, while also have the merit of insensitive to the LED output power, thus vast kinds of light source can be used in this system. As the concentration of ethanol varies between 10% to 50% and glycerin varies between 0.5% to 4. 5%, the test shows that the measured concentrations have the absolute deviations less than 0. 009 5% and 0. 008 2%. The fitting equations only involved arithmetic, thus suitable for processing via computers, and the merit of realtime monitoring. The system only have to standardize the specific environment when the operation temperature changes, no other radical changes are needed.

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利用光纤法布里-珀罗谐振器干涉透射波长同时测量 混合溶液中多溶质浓度

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摘 要:为了同时探测混合溶液中多种溶质的浓度,提出一种用光纤法布里-珀罗谐振器作为传感器测量混 合溶液中多溶质浓度的测量系统.从理论上分析了光纤法布里-珀罗谐振器干涉透射波长与混合溶液浓度之 间的关系,采用光纤法布里-珀罗谐振器干涉透射波长实现了高准确度同时测量混合溶液中多溶质浓度的原 理和可行性.构建了由 InGaAs 发光二极管光源、光纤耦合器、FFPR 传感器、光电信号转换和放大器、光谱 分析仪等组成的测量系统.对乙醇和甘油的9组标准混合溶液进行测量实验,并用测量结果标定了混合溶液 中各溶质浓度与光纤法布里-珀罗谐振器传感器干涉透射波长之间的数学解析关系式.根据数学关系式用 Action Script 2.0 脚本语言编写程序,计算机实时监控了混合溶液中各溶质浓度的变化过程.

关键词:混合溶液;多溶质溶液的浓度;光纤法布里-珀罗谐振器传感器;干涉透射波长;光谱分析仪



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