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Determination of Refractive Index Dispersion Using Fiber-Optic Low-Coherence Fabry-Perot Interferometer: Implementation and Validation

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Abstract

In this paper, implementation and validation of low-coherence Fabry-Perot interferometer for refractive index dispersion measurements of liquids is presented. Measurement system has been created with the use of four superluminescent diodes with different optical parameters, a fiber-optic coupler and an optical spectrum analyzer. The Fabry-Perot interferometer cavity has been formed by the fiber-optic end and mirror surfaces mounted on a micromechanical stage. The positive result of the validation procedure has been determined through statistical analysis. All obtained results were 99.999 % statically significant and were characterized by a strong positive correlation (r>0.98). The accuracy of the measured result of implemented low coherence Fabry-Perot Interferometer sensor is from 83 % to 94 %, which proves that the sensor can be used in the measurement of refractive index dispersion of liquids.

Keywords: refractive index dispersion, Fabry-Perot interferometer, low-coherence interferometer , fiber optics

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1. Introduction

The refractive index is one of the fundamental optical properties of materials. This parameter depends strongly on the composition of the material, including the dopant materials and impurities contained within. In case of heterogeneous materials, e.g. biological samples such as blood [1] or skin [2], there is a significant variability of refractive indices, caused by its dependence on the various components of the medium under examination, like cell membranes and organelles [1]. Observation thereof enables the detection of small changes of structure and composition of the examined material.

Each application has special requirements, thus it is often necessary to develop new methods or adapt existing ones. In order to measure liquid samples, fiber-optic sensors are suitable because they provide lower invasiveness, as measurements can be performed on a small sample volume, without sample handling procedures, such as chemical pre-treatment.

Several other fiber-optic methods for the measurements of refractive index have been developed. One of such techniques relies solely on the fiber Bragg gratings, which are able to achieve the sensitivity in the range from 10^{-5} to 10^{-7} RIU (refractive index unit), and their subsequent modifications such as long period gratings (LPG) [3,4] and tilted fiber Bragg gratings (T-FBG) [5]. However, the gratings are highly sensitive to temperature and strain, therefore their potential applications are somewhat limited. Ultra-high sensitivities of 10^{-7} RIU are offered by the sensors based on whispering gallery modes [6–8], which require complex micro-resonator manufacturing. Tapered fibers [9] geometry is also used for sensing purposes, especially for the Surface Plasmon Resonance (SPR) [10]. SPR is also a widely used techniques in refractive index measurements, which offers from 10^{-4} up to 10^{-7} RIU sensitivity and selectivity in chemical and biomedical sensing [11–13]. They rely on complex chemical surface functionalization and are highly specialized for narrow and specific applications.

In this paper we present the implementation of the fiber-optic low-coherence Fabry-Perot interferometer (LCFPI) for refractive index dispersion measurements of fluids [14, 15]. The advantages of the sensor over aforementioned methods lie mainly in its high sensitivity and broad dynamic range. In contrast to other sensors, which often require specially produced fibers (e.g. tapered, coated, microstructured, chemically functionalized), our LCFPI sensor does not need any elaborated manufacture procedure, as it uses commercially available components which reduce time and fabrication costs. An open Fabry-Perot cavity configuration is robust, easy to clean and enables modification for various sample holding needs. Ease of construction of this sensor offers the possibility for miniaturization. Measurements may be rapid, which permits further expansion of this method for various applications ranging from on-line process monitoring, bedside intravenous fluid supply control, bulk laboratory analysis and other fast liquid analysis. Moreover, low-coherence interferometry is the only interferometric method of the absolute value measurement of the investigated parameter, thus enabling direct measurements [16].

To confirm the effectiveness of the proposed measurement method we employed statistical validation procedure, described step-by-step, which consists of the statistical tests (Grubb's Test for outliers, T-Student Test, regression analysis) and the determination of metrological parameters. The validation was carried out upon measurements` results of several substances with known refractive indices [17], such as: methanol, acetone, acetic acid, ethanol, buthanol, ethylene glycol, cyclohexane, glycerol, methyl salicylate. In total, over two hundred eighty measurements of these substances were performed. Such a number of measurements and a positive result of the validation allowed us to determine usefulness of the implemented fiber-optic low-coherence Fabry-Perot interferometer for the use in the refractive index dispersion measurements of fluids.

2. Method

In low-coherence interferometry, the resulting interferogram comprises of the signal envelope, which depends on the spectral characteristic of the light source (usually a Gaussian distribution), and its modulation, which forms the spectral pattern [16,18]. The optical path difference between interfering beams is encoded in the spectral modulation of measured spectral signal [18]:

$$I = I_0\left[1 + V\cos(\Delta\varphi)\right] \quad (1)$$

where φ is a phase shift is related to the refractive index changes (Δn):

$$\Delta\varphi = \frac{4\pi}{\lambda}\Delta nL \quad (2)$$

where: I is the measured signal intensity, I_0 is the light source intensity, L is a geometrical path length of the intereferometr, n is the refractive index, λ is the wavelength of interfering beams, V is the visibility of the measured signal. A reflection-mode fiber-optic Fabry-Perot interferometer was implemented, where the interference occurs in the cavity formed between the beams reflected from the fiber-end surface and a parallel mirror. The beam propagates through the sample, placed between these surfaces, and undergoes phase-shift which depends on the refractive index of a sample.

Implemented system (Fig 1.) comprises of: four SLED sources produced by Superlum (Ireland): (1) λ =810 nm, Δλ = 50 nm, P = 3 mW, SLD-381-MP3-DIL-SM-PD; (2) λ =970 nm, Δλ = 50 nm, P = 1,5 mW, SLD-481-MP3-DIL-SM-PD; (3) λ =1310 nm, Δλ = 100 nm, P = 10 mW, SuperlumBroadlighter SLD S-1300-G-I-20; (4) λ =1550 nm, Δλ = 100 nm, P = 20 mW, SuperlumBroadlighter SLD S-1550-G-I-20), a fiber-optic coupler, an adjustable Fabry-Perot cavity formed between the fiber-end and a mirror surfaces mounted on a micromechanical stage, and an optical spectrum analyzer (OSA) (Ando AQ6319, ANDO Japan) with wavelength resolution better than 20 pm and a dynamic range of 70 dBm.

To measure the refractive index of the chemical substance using implemented measurement system, the test sample should be placed between the tip of the optical fiber and a mirror mounted on the adjustable table. Light propagates form the SLED light source through the optical fiber to the Fabry-Perot cavity with sample insterted inside. Interference occurs between incoming beam and the beam reflected from the mirror surface after passing the sample, due to phase-shift induced by the sample, according to the equation (2). The returning intererference pattern signal (1) is guided through the coupler to the spectrum analyzer where it is recorded. To obtain a measurement result, spectral separation should be read from measured signal spectrum (Fig.2b) and compared with reference spectrum (Fig. 2a)

Effectiveness of the implemented Fabry-Perot Low Coherence Interferometer (FP) sensor was confirmed by validation. The validation is a procedure aimed at investigation of measurement methods to obtain objective evidence that the method satisfies posed requirements and that the measurements lead to accurate results. According to ISO/IEC 17025 [19] standard, it is always necessary to carry out the validation procedure before implementation of the new measurement method and after modification of the existing one. While validation gives information about the accuracy of the measurement method (a true value of a parameter is determined), a reliability shows how precise results are [17]. The validation procedure should be carried out according to the following points:

- detection of measurement errors,
- carrying out selected statistical test,
- assessment of the metrological parameters of the process,
- verification of established requirements and assumptions and stating the usefulness of measurement method.

The validation of refractive index measurement method using interferometric low-coherence system was carried out by measurements of nine chemical substances. Separate measurements

were performed for each substance. The measurement procedure for the validation is shown by the block diagram (Fig. 3).

The reference spectrum of an empty FP cavity was recorded by OSA. Then, the sample was placed in the cavity and the measured spectrum was obtained. After thorough cleaning of the cavity the procedure was repeated for other substances. After a sequence of measurements for all substances, the light source was switched and the aforementioned procedure started anew for another light source. This comprises a single measurement series, which was repeated 8 times for the purpose of system validation.

3 Validation

The validation procedure of the low-coherence FP interferometric sensor for refractive index dispersion measurements was performed for nine chemical substances. A detailed, step-by-step procedure is presented for methyl alcohol (methanol). Measured spectral separation between maximums in the spectra of measured signal of methanol is shown in Table 1.

Errors in measurements can be detected by investigating existance of outlying observations in measurement data. This can be done using scatterplot, which illustrates the correlation between dependent variable and determining variable. The scatterplot of presented results is shown on Fig. 4. The graph includes the results of all the measurements, in function of wavelength.

Two clearly distinct points are noticeable on the scatter plot (Fig. 4.). This may indicate the existance of outliers. To support this hypotesis it is necessary to conduct a statistical test. For our purpose a Grubb's test for outliers was performed and the results are shown in Table 2. Grubb's test statistic G is calculated for extreme values, according to the formula[17]:

$$G = \frac{\max\left|X_i - \overline{X}\right|}{\sigma}, \tag{3}$$

where: X_i is the most extreme observation from the mean, $\overline{X}$ - is the mean of all observations including the possible outlier, σ - is the standard deviation of all observations including the possible outlier. If G is higher than a Grubbs test critical value (shown in Table 2), then X_i is considered an outlier.

Calculated Grubb's test statistics (Table 2.) for the wavelengths 0.81, 0.97 and 1.55 μm are higher than the Grubb's Test statistics critical value taken from the statistical tables, thus there is no basis to reject the hypothesis that outliers exist. Probability that outliers do not occur in the results is expressed as p-value. For the purpose of validation procedure p-value level shall be greater than 0.05[18]. All outlying observations were excluded from the results, which were then again processed with Grubb's test for outliers, as presented in Table 3. Variation in p-values in presented Grubb's test results is directly corresponding to the number of valid N. Grubbs test is typically used for groups with more than 25 elements (N) but due to high power of the test it can be used for smaller number of elements. However, this may result in a significant variation in calculated p-values, as can be seen in Table 3.

Calculated Grubbs test statistics values are lower than Grubb's Test statistics critical value – there are no outlying observations among the experimental results (with p-value higher than 0.365), which once agains proves considering scatter plot of cleansed data. (Fig. 5).

Obtained results follow the normal distribution, therefore further analysis consists of the parametric statistisc. The parametric statistics are used to verify hypothesis about parameters of normal distributions. One of them is T-Student Test, which allows to determine whether there is a statistically significant relationship between the sample and the mean value of sample population. Obtained results were tested by calculating T-Student test statistic t_n described as[18]:

$$t_n = \frac{X - \overline{X}}{s}\sqrt{n}\,, \tag{4}$$

where: X is the value of a sample, $\overline{X}$ is a mean of sample population, n is a number of samples in a population, s is a standard deviation. Then, the calculated t_n is compared with test statistics t, taken from the T-Student distribution tables for a set significance level. When $t_n > t$, then the correlation between the sample and the mean value of sample population is statistically significant (at set significance level). For experimental results, calculated t_n were in range from 15.42 to 538.46. The comparison of obtained test statistics with the T-Student distribution tables yielded highly statistically significant correlation of experimental results with confidence level 0.001. This indicates that the measurements are 99.999 % statically significant.

The next step is to calculate r-Pearson correlation, which is a measure of linear relationship between two variables. Correlation value r ranges from -1 to 1, where -1 is a strong negative correlation, 1 is a strong positive correlation and zero means random relationship between variables. r-Pearson correlation analysis is conducted by determination of r parameter according to equation[19]:

$$r = \frac{N\sum XY - \sum X \sum Y}{\sqrt{[N\sum X^2 - (\sum X)^2][N\sum Y^2 - (\sum Y)^2]}}, \tag{5}$$

where: X, Y are paired variables. It is necessary for results to have strong negative or positive correlation - only then, the method is valid. Calculated values of r-Pearson correlation coefficient (7) for presented method equal 0.9943 (strong positive correlation). This indicates very significant relation between experimental data and wavelength.

Determination coefficient R^2, describes the degree to which the regression model explains the experimental data, and is defined as[20]:

$$R^2 = \frac{\sum_{i=1}^{N}(x_p - \mu)^2}{\sum_{i=1}^{N}(\bar{x} - \mu)^2}, \tag{6}$$

where: x_p is the value expected by the regression model, μ is the mean, $\bar{x}$ is the value of a sample. The determination coefficient takes values from 0 to 1, while R^2 is closer unity, then the regression model is better suited. Calculated determination coefficient equals 0.9886, which means that the linear regression model explains more than 98% of the variability of the obtained results.

After calculation of r and R^2, regression analysis was performed. The regression analysis is based on predicting the value of one variable based on the other variable. If the independent variable is known, it is possible to estimate the value of the dependent variable based on the regression equation. The regression analysis of the obtained measurements yielded the equation which relates the spectral separation to the refractive index:

$$n = 1.3268 - 0.0163\delta + 0.0025\delta^2 \tag{7}$$

where: n is refractive index, δ is a spectral separation. Estimated equation is a second degree polynomial which sufficiently fits measured results (Fig. 6).

After calculating n from equation (7) for several wavelengths it is possible to determine refractive index dispersion curve (8):

$$n(\lambda) = 1.3569 - 0.0719\lambda + 0.0228\lambda^2 \tag{8}$$

where: n - refractive index, λ - wavelength [nm]. A determined refractive index dispersion curve of methanol from equation (8)is compared with literature data [21] and is shown on Fig. 7. The measured data almost completely agrees with the litarature data.

Small differences between plotted dispersion curves are due to standard uncertainty of measurements and the wavelength accuracy of OSA, equal to ± 50 pm. The standard uncertainty of results was determined based on the standard deviation of the results. Calculated values of standard deviation have been also used to determine reproducibility of presented method. Tested chemical substances were measured in eight series. Each series required measurements of nine independently prepared samples. All measurements were made with

uniform conditions and by the same lab assistant. Low values of standard deviation between results for each substance collected from eight series, indicate sufficient reproducibility of the proposed approach. Calculated values of standard deviation are shown in Table 4.

The maximum calculated value of standard deviation is approximately 0.0087. This is indicated by an acceptable degree of standard uncertainty of results below 0.1, thus confirming the reliability of the measurement method.

Prior to the statistical analysis of measurement results, certain assumptions, regarding the qualititative requirements of the test method, were stated. They concerned the statistical parameters and metrology. The measurement result are discarded when the value of the Grubbs test statistic was higher than the critical value read from the critical value statistical table at a level of probability $p > 0.05$. This is a widely accepted criterion for statistical analyses[22,23]. Since this measurement method will be further developed for biophotonics research, it has been decided to increase the acceptance criteria for subsequent tests. For T-Student's test, a confidence level of less than 0.01 was adopted. The lower value of the coefficient of determination is set at 0.90 and the r-Pearson correlation coefficient can not be lower than 0.95. Acceptable standard deviation should not be greater than 0.17. The validation was carried out taking into account all qualitative requirements of the measurement method. The results are presented in Table 5.

It can be concluded that tests of all substances meet set requirements. Very good T-Student's tests are of particular note as their results are characterized by a statistical dependency hundred times greater than specified in the requirements. For all models, the determination coefficient is much higher than 0.98, and the r-Pearson correlation coefficient exceeds 0.99. The resulting accuracy is from 83 % to 94 % depending on the tested chemical substance. Due to the fulfillment of all qualitative requirements, the validation process was completed with positive result, thus confirming the suitability of the Low-Coherence Fabry-Perot

interferometer method applied to measure the refractive index of liquids. Basing on the analysis of the obtained measurement results for each of the measured substances, the refractive index dispersion curves were determined (Table 6).

Determined refractive index dispersion curves are plotted together with theoretical curves on Fig. 8. It can be seen that the measurement results coincide with the reference theoretical values. Discrepancies could be due to the fact that the theoretical values of the refractive indices of the test substances were determined at different temperatures. The value of the refractive index is dependent on the temperature of the sample, thus the discrepancies may appear due to the fact that the experiment was performed at room temperature and temperature conditions were contolled with a non-contact pyrometer designed for small-sample measurements [24].

The results of validation of the designed test method suggests that it can be used to determine the refractive index of the liquids.

3. Conclusion

In this paper, we have presented implementation and validation of low-coherence Fabry-Perot interferometer for refractive index dispersion measurements of liquids. Measurement system has been created with the use of four SLED sources, a fiber-optic coupler and an optical spectrum analyzer. An adjustable Fabry-Perot cavity was formed between the fiber-end and a mirror surfaces mounted on a micromechanical stage. The greatest advantage of presented sensor is that it relies on commercially available components and requires no special fabrication of the fiber. The positive result of the validation procedure was determined through statistical analysis. All obtained results were 99.999 % statically significant and were characterized by strong positive correlation ($r>0.98$). The resulting accuracy of implemented LCFPI sensor is from 83 % to 94 %, which proves that the sensor can be used in the measurement of refractive index dispersion of liquids.

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7. Tables

Table 1. Spectral separation for methanol [nm].

Measurement procedure no.	Central wavelength			
	$\lambda = 0.81$ µm	$\lambda = 0.97$ µm	$\lambda = 1.3$ µm	$\lambda = 1.55$ µm
1	0.9750	1.4249	2.4498	3.7300
2	0.9750	0.7000	2.3998	3.5002
3	0.9750	1.3875	2.5202	3.5002
4	0.9625	1.3848	2.3998	3.4795
5	0.9250	1.4499	2.4498	3.4795

6	0.9250	1.3749	2.3798	3.5804
7	0.9250	1.3501	2.4301	3.5002
8	0.9596	1.3749	2.5497	3.5997

Table 2 Results of Grubb's Test for all data points.

λ [μm]	Valid N	Mean	Grubb's Test statistics	Grubb's Test statistics critical value	p-value
0.81	8	0.952767	1.17037625	2.03	1.000000
0.97	8	1.305895	2.45493008	2.03	0.000010
1.3	8	2.447376	1.70673673	2.03	0.467423
1.55	8	3.546189	2.11074608	2.03	0.056877

Table 3 Results of Grubb's Test for data points after excluding detected outliers.

λ [μm]	Valid N	Mean	Grubb's Test statistics	Grubb's Test statistics critical value	p-value
0.81	8	0.952767	1.170376	2.03	1.000000
0.97	7	1.392453	1.700991	1.94	0.365041
1.3	8	2.447376	1.706737	2.03	0.467423
1.55	7	3.519937	1.624798	1.94	0.490555

Table 4 Calculated values of standard deviation for all wavelengths.

λ [μm]	Valid N	Minimum	Maximum	Std.dev.
0.81	8	0.924984	0.975039	0.023738
0.97	7	1.350074	1.449906	0.033776
1.3	8	2.379819	2.549720	0.059964
1.55	7	3.479471	3.599712	0.049098

Table 5 The results of all performed test for measured substances.

Substance	Grubb's Test result	T-Student Test result	Determination coefficient R^2	r-Pearson correlation	Standard deviation
Methanol	p=0.37	0.0001	0.9886	0.9943	0.0600
Acetone	p=0.62	0.0001	0.9855	0.9927	0.0997
Aceticacid	p=0.32	0.0001	0.9883	0.9941	0.1177
Ethanol	p=0.6	0.0001	0.9824	0.9911	0.0906
Buthanol	p=0.28	0.0001	0.9893	0.9946	0.0653
Ethyleneglicol	p=0.37	0.0001	0.9832	0.9916	0.1068
Cyclohexane	p=0.21	0.0001	0.9817	0.9908	0.1609
Glycerol	p=0.1	0.0001	0.9829	0.9914	0.0685
Methylsalicylate	p=0.27	0.0001	0.9914	0.9957	0.0651

Table 6 Determined dispersion curves for all measured substances.

Substance	Determined dispersion curve	Theoretical dispersion curve
Methanol	$n = 1.3569 - 0.0719\lambda + 0.0228\lambda^2$	$n = 1.3582 - 0.0739\lambda + 0.0234\lambda^2$

Acetone	$n = 1.3656 - 0.0185\lambda + 0.0061\lambda^2$	$n = 1.3661 - 0.0193\lambda + 0.0062\lambda^2$
Aceticacid	$n = 1.3926 - 0.0589\lambda + 0.0185\lambda^2$	$n = 1.3943 - 0.0617\lambda + 0.0195\lambda^2$
Ethanol	$n = 1.3667 - 0.0149\lambda + 0.0042\lambda^2$	$n = 1.368 - 0.0177\lambda + 0.0056\lambda^2$
Buthanol	$n = 1.4078 - 0.0254\lambda + 0.0074\lambda^2$	$n = 1.4093 - 0.0284\lambda + 0.009\lambda^2$
Ethylene glicol	$n = 1.4719 - 0.096\lambda + 0.0295\lambda^2$	$n = 1.4763 - 0.1042\lambda + 0.0331\lambda^2$
Cyclohexane	$n = 1.4338 - 0.0209\lambda + 0.0065\lambda^2$	$n = 1.435 - 0.0233\lambda + 0.0075\lambda^2$
Glycerol	$n = 1.4817 - 0.0257\lambda + 0.0078\lambda^2$	$n = 1.4833 - 0.0284\lambda + 0.0088\lambda^2$
Methyl salicylate	$n = 1.5537 - 0.0554\lambda + 0.0171\lambda^2$	$n = 1.5555 - 0.059\lambda + 0.0188\lambda^2$

8. List of figure captions

Fig. 1 The implemented low-coherence fiber-optic Fabry-Perot interferometer measurement setup: (1) low-coherence source, (2) Sensing Interferometer, (3) coupler, (4) optical spectrum analyzer, (5) Signal Processing.

Fig. 2 The measured spectrum of a reference signal (a) and the methanol (b).

Fig. 3 The block diagram of measurement procedure for validation.

Fig. 4 The scatterplot of results. Two noticable datapoints are suspected to be outliers.

Fig. 5 The scatterplot of data cleansed after Grubb's Test

Fig. 6 The estimated relation of spectral separation to refractive index for methanol. Fitting was achieved with a second degree polynomial function.

Fig.7 The results of measured refractive index dispersion curve of methanol. Measured data are shown as circles, while reference data are squares.

Fig.8 The refractive index dispersion curve of measured substances. Measured results are shown in circles, while theoretical data is in squares.