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U-Shaped Plastic Optic Fiber Sensor for Ethanol/Methanol Determination

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U-Shaped sensor,
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Plastic optical fiber,
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Abstract: In this study, U-shaped fiber optic sensor tips sensitive to refractive index (RI) change were produced, and its performance analyses were carried out. Three sensor tips were used to measure ethanol/methanol-water mixtures in concentrations from 0% to 16.66%. The ethanol sensor tip was able to detect the RI change of 4×10^{-4} RIU. Its maximum sensitivity is 7.71 mV/RIU, and its linearity is 0.9985. The methanol sensor tip was able to detect the RI change of 1×10^{-4} RIU. Its maximum sensitivity is 28.49 mV/RIU, and its linearity is 0.9969. Each sensor tips determine the change of 1.41% concentration in the mixtures. Moreover, the results show that it can provide high precision in the measurement of ethanol/methanol-water mixtures and achieve satisfying stability and repeatability.

Etanol/Metanol Belirlenmesi için U-Şeklinde Plastik Optik Fiber Sensör

Anahtar Kelimeler
U-Şeklinde sensör,
Kırılma indisi,
Plastik fiber optik,
Etanol/Metanol tayini

Öz: Bu çalışmada kırılma indisi (RI) değişimine duyarlı U-Şeklindeki fiber optik sensör uçları üretilmiş ve performans analizleri yapılmıştır. %0 ile %16,66 arasındaki konsantrasyonlarda etanol/metanol-su karışımlarını ölçmek için üç sensör ucu kullanılmıştır. Etanol sensör ucu, 4×10^{-4} RIU'luk kırılma indisindeki değişimi tespit etmiştir. Etanol sensör ucunun maksimum hassasiyeti 7,71 mV/RIU ve doğrusallığı 0,9985'tir. Metanol sensör ucu ise 1×10^{-4} RIU'luk kırılma indisindeki değişimi tespit etmiştir. Bu ucun maksimum hassasiyeti 28,49 mV/RIU ve doğrusallığı 0,9969'dur. Her bir sensör ucu, karışımlardaki %1,41 konsantrasyondaki değişikliği belirlemiştir. Ayrıca sonuçlar, sensör uçlarının sadece etanol/metanol-su karışımlarının ölçümünde yüksek kesinlik sağlamakla kalmayıp aynı zamanda tatmin edici kararlılık ve tekrarlanabilirlik sağladığını da göstermektedir.

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1. Introduction

Alcohol, which is essential both in industrial processes and for humans, consists of two primary forms. These are ethanol (C_2H_5OH) and methanol (CH_3OH). Ethanol is used as an antiseptic in medical cleaning and is widely used in antibacterial hand disinfectants because of its bactericidal and anti-fungal effects [1]. Also, ethanol is the only alcohol type used in alcoholic beverages [2]. Methanol, which is used as a solvent in the industry, is also used to prepare formaldehyde and aniline dyes. Besides, methanol is used in the structure of unleaded gasoline, as an engine fuel component, and cologne production.

However, methanol is highly toxic to humans. Methanol is not metabolized in the body, and after it enters the body, it degrades into two chemicals called formaldehyde and formic acid [3]. Due to these dissociated compounds, a volume of pure methanol as small as 10 ml causes permanent blindness as it damages the optic nerves. A volume of about 30 ml is fatal [4]. It is difficult to distinguish it from ethanol in terms of odor and appearance. It is used illegally instead of ethanol in alcoholic beverages because it is cheap and very readily available in the market. Therefore, it is essential to distinguish these two alcohol types and determine the concentrations in ethanol/methanol-water mixture. While it is possible to detect the difference between these two types of alcohol with advanced measurement methods such as, Raman spectroscopy [5], gas chromatography [6], near-infrared reflectance spectrometry [7], amperometric biosensing [8], and gas-phase biosensing [9]. It is important to detect this in a simple, fast, accurate, and low cost. Detection types based on optical methods that contain these features are the most suitable candidates.

The refractive index (RI) is a fundamental optical parameter frequently used in the identification and concentration of chemical substances in many analytical applications, including the food and processing chemical industry. It can be easily detected with fiber optic sensors (FOSs). RI-based FOSs have superior properties such as being very sensitive, flexible [10], small size, lightweight, highly immune to electromagnetic interference [11], adaptable, or attached to different systems [12]. These features are also widely used in the literature for ethanol and methanol detection [13-16].

The plastic optical fiber (POF) based RI sensors have attracted significant interest from researchers because they contain low-cost solutions for RI determination [17-19]. POF-based RI sensors have been structurally altered by some embodiments such as drilling to increase precision [20], D-Shaped [21], tapering [22], side polishing [23], coiled POF [24], U-bending [25, 26]. Compared to other configurations, the U-bending method is easy to apply to POF, and the light can be more scattering to the environment to improve detection performance.

In this study, U-shaped fiber optic tips were produced, and performance analyzes were made. Proposed sensor tips were used to detect RI changes in ethanol/methanol-water mixture at different concentrations. A sensor with three different fiber diameters was designed. Also, the repeatability of the sensor was tested by standard deviation analysis. As a result of this analysis, the standard deviation of the measurements was below 1. Therefore, it has been determined that the sensor has satisfactory repeatability. As a result of the measures and analyzes, it is evaluated that the proposed sensor performance is high, and it can be used for ethanol/methanol-water mixture detection in alcoholic beverage processing plants.

2. Material and Method

2.1. Theory

Fiber loss mechanisms are used in fiber optic detection. One of the fiber power loss mechanisms in optical fibers is the attenuation of the signal caused by the fiber's bending. As the light passes through the fiber, it travels in the core with a total internal reflection (TIR) phenomenon [27]. Thus, all light beams have a critical angle at the core/cladding interface [28]. This angle is given in Equation (1):

$$\theta_c = \sin^{-1}\left(\frac{n_{clad}}{n_{core}}\right) \tag{1}$$

Where n_{clad}, n_{core} is the RI of the fiber cladding and core, respectively.

Due to curvature and bends along a fiber path, losses occur for the beams traveling through the fiber. Such losses are called fiber bending losses (Figure 1). These losses depend on the core and cladding refractive index differences, the core radius, and the wavelength studied. When the difference between the refractive indices of core and cladding increases, fibers with larger numerical apertures can be formed, and these fibers are less susceptible to bending. These types of losses are also called macro bending losses. Empirically, the macro-bending loss can be expressed in dB [29, 30].

$$\gamma_b = 10 \log \frac{\alpha + 2}{(2\alpha)(a/R\Delta)} \tag{2}$$

Where $\Delta=(n_{core}-n_{clad})/n_{clad}$, relative refractive index difference; R is the bend radius; a is the core radius and α is the profile parameter. Keeping all parameters constant, the change of this loss according to the fiber radius is given in Figure 2.

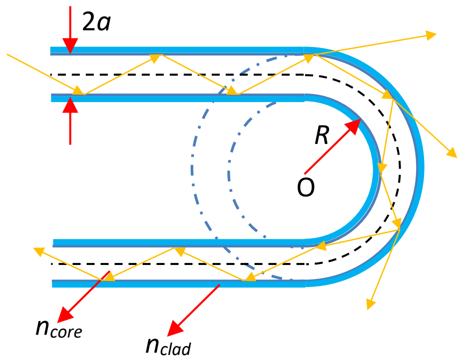

Figure 1. Losses in fiber due to bending effect

As shown in Figure 2, for fibers with constant core and cladding refractive indices, the fiber's loss (leaking into the cladding zone) will increase as the fiber's diameter increases.

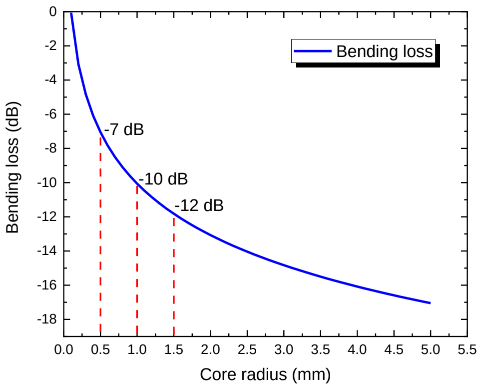

Figure 2. Bending loss versus fiber core radius

However, when the fiber is bent to values less than or equal to a critical radius, a significant part of the light leaks from the coating, and the fiber cannot conduct all light transmission in the core. Therefore, a considerable amount of power loss occurs in the fiber. Although this power loss is regarded as an undesirable situation in fiber optic communication systems, it is crucial in detection applications studies [31, 32].

A fiber with a large core radius is more affected by bends. This situation is compatible with the loss values in the equal radius of bending (R=3 mm) of three fibers with radii of 0.5 mm, 1 mm, and 1.5 mm in Figure 2. This loss is also related to the refractive index of the medium surrounding the fiber. For example, as the sensing medium's refractive index increases, the amount of light scattered from the clad/medium interface increases. Therefore, a decrease in the light intensity coming to the photo-detector will be observed.

2.2. Preparation of sensor tips and Experimental setup

The fiber sensor tip is an essential part of the detection system. Three sensor tips were produced using plastic optic fibers with diameters of 1, 2, and 3 mm in this work. To produced U-shaped POF sensor tips, fibers were heated up to softening temperature and then wrapped on a mandrel with 3 mm diameter. In the study, U-shaped POF sensor tips formed with 1 mm, 2 mm, and 3 mm diameter fibers will be called U1, U2, and U3, respectively.

This study's primary purpose is to design a sensor system that can detect possible differences between ethanol and methanol concentration values for each alcohol processing stage. Therefore, an optical sensor system was designed and implemented to see the ethanol/methanol-water mixture. The image of the sensor tips is given in Figure 3a. The block diagram of the experimental setup is shown in Figure 3b. The light source and photo-detector are 660 nm LED (Avago Technologies, HFBR-1524Z) and a photodiode-IC receiver (HFBR-2524Z). Also, the signal processing and amplifier are designed at the photo-detector output. The amplifier's signals were sent to both the designed microprocessor board and the PC oscilloscope (Picoscope 3206MSO). According to the theory described above, it is expected that as the refractive index of the medium increases, the light scattered out of the fiber (sensing medium) increases.

Consequently, a lower light intensity comes to the photo-detector. However, in this study, with the design made in the signal processing circuit, the increasing response decreased light intensity. Therefore, the sensor response is increased depending on the increasing refractive index of the medium.

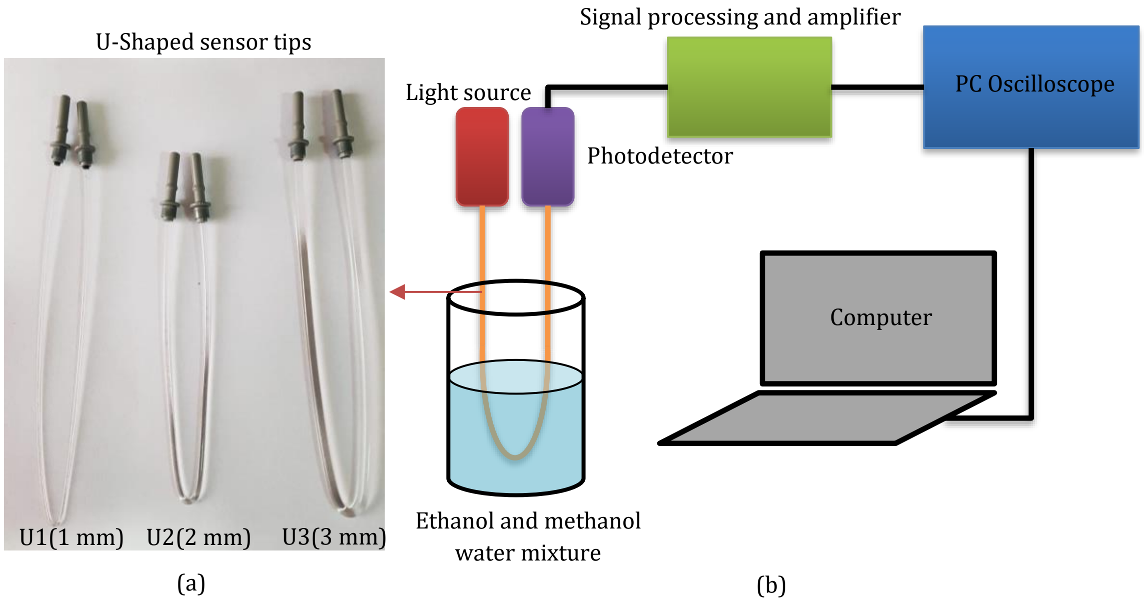

Figure 3. (a) U-Shaped sensor tips, (b) The block diagram of the experimental setup

3. Results

The first measurement values were obtained by immersion the tips of U1, U2, and U3 into 5000 µL distilled water, respectively. Then, by adding 100 µL of ethanol, the first mixture with a concentration of 1.9608% was formed, and sensor responses were obtained. Similarly, 100 µL of ethanol was added to the previous mixture until the concentration was 16.6667%. In addition, the refractive index values of each mixture formed were measured with an IR280D (Insmark Co.) refractometer. All experiments were carried out by controlling the temperature in a climatic laboratory to the RI of the solutions that do not fluctuate with a temperature change. The experiments were carried out by keeping the room temperature at 25^{0}C (±1^{0}C). The sensor tip responses (mV) obtained from the measurements, the refractive indices, and concentrations of each mixture are given in Table 1.

Table 1. Responses of the sensor tips for Ethanol.

Ethanol Concentration	RI	U1	U2	U3
%	**RIU**	**mV**	**mV**	**mV**
0.0000	1.3333	1.002	1.052	1.303
1.9608	1.3338	1.002	1.056	1.339
3.8462	1.3343	1.002	1.060	1.381
5.6604	1.3348	1.002	1.063	-
7.4074	1.3353	1.003	1.066	-
9.0909	1.3357	1.003	1.071	-
10.7143	1.3361	1.003	1.074	-
12.2807	1.3366	1.004	1.077	-
13.7931	1.3370	1.004	1.080	-
15.2542	1.3373	1.004	1.083	-
16.6667	1.3377	1.005	1.086	-

When the performance values of U2 are examined from Table 1, it was determined that U2 detects the smallest 1.41% ethanol change in the solution. In comparison, this circumstance is an average of 1.66% in all measurements. The slightest difference between the RIs is 1.3373-1.3370=3×10^{-4} (in Table 1, lines 10 and 9), the enormous difference is 1.3353-1.3348=5×10^{-4} (Table 1, line 5 and 4). In Figure 4a, U2 output versus percentage ethanol concentration is given. Figure 4b shows the linear fitting of the refractive index (RIU) versus U2 Output (mV). As can be seen from the graph, remarkable linearity with 0.9985 and sensitivity of 7.71 mV/RIU was obtained between the refractive index and the U2 output.

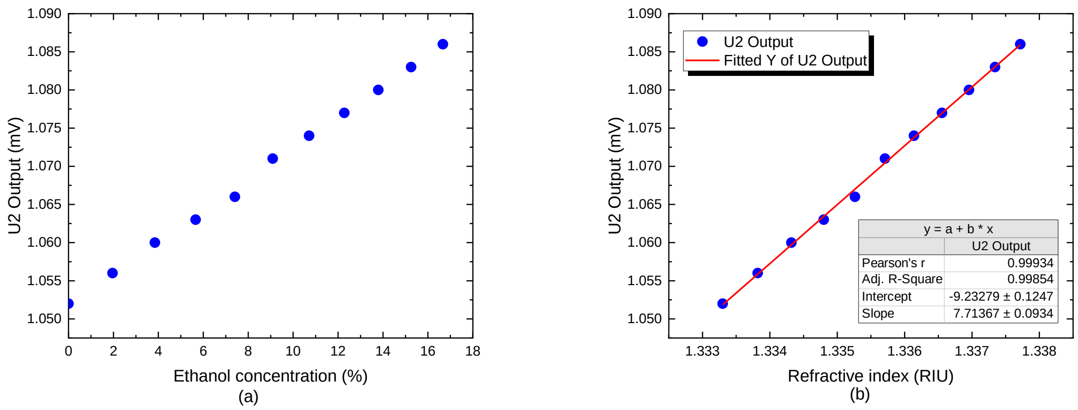

Figure 4. (a) U2 output vs. percentage ethanol concentration, (b) U2 output vs. refractive index change of ethanol

The experimental procedure for ethanol in the first paragraph of the Result section was applied exactly in methanol, and the data obtained are similarly given in Table 2. Unlike selecting the most suitable tip used in ethanol detection, tip number U3 performance is the best in methanol detection. Since methanol has a lower refractive index than water, different from Table 1, refractive index values in Table 2 decreased depending on the increasing concentration.

U1 remained unresponsive (even more unresponsive) as with ethanol measurements. On the other hand, U2 responded in a narrow band like U1, unlike ethanol measurements. Therefore, it was determined that the best tip for methanol is U3.

Table 2. Responses of the sensor tips for Methanol.

Methanol Concentration	RI	U1	U2	U3
%	RIU	mV	mV	mV
0.0000	1.3333	1.002	1.052	1.303
1.9608	1.3331	1.002	1.052	1.295
3.8462	1.3329	1.002	1.051	1.290
5.6604	1.3328	1.002	1.051	1.286
7.4074	1.3326	1.002	1.051	1.281
9.0909	1.3324	1.001	1.050	1.277
10.7143	1.3323	1.001	1.049	1.273
12.2807	1.3321	1.001	1.049	1.269
13.7931	1.3320	1.001	1.047	1.264
15.2542	1.3319	1.001	1.047	1.260
16.6667	1.3317	1.001	1.046	1.256

When the performance values of U3 are examined from Table 2, the smallest difference between the RIs is 1.3324-1.3323=1×10^{-4} (in Table 2, lines 6 and 7), the biggest difference is 1.3328-1.3326=2×10^{-4} (Table 2, line 4 and 5). In Figure 5a, U3 output versus the percentage of methanol concentration is given. Figure 5b shows the linear fitting of the refractive index (RIU) versus U3 Output (mV). As can be seen from the graph, remarkable linearity with 0.9969 and sensitivity of 28.49 mV/RIU was obtained between the refractive index and the U3 output. The methanol detection sensitivity of U3 was determined to be almost four times higher than the ethanol sensitivity of U2. Therefore, it is evaluated that the concentration changes of methanol, which is present in low rates in alcoholic beverages, can be detected by U3.

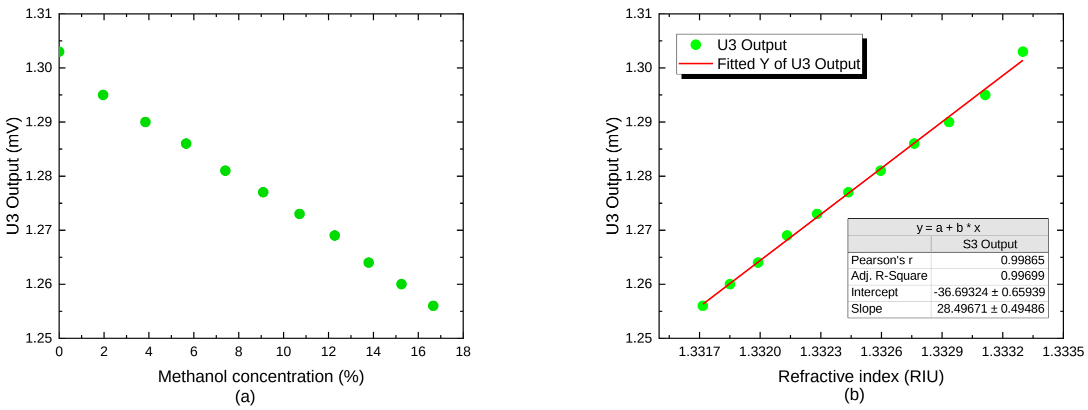

Figure 5. (a) U3 output vs. percentage methanol concentration, (b) U3 output vs. refractive index change of methanol

The sensors' repeatability was investigated by making three measurements at each concentration with the U2 and U3 tips. For this purpose, the standard deviations in the measurements were examined. The minimum standard deviation for U2 was 0.47, while the mean of the standard deviation was 0.89. Similarly, the minimum standard deviation for U3 was 0.65, while the mean of the standard deviation was 0.91. Therefore, it was determined that the results in the measurements were very close to each other.

4. Discussion and Conclusion

In this study, U-shaped POF optical sensor tips with three different diameters are produced to determine the refractive index changes of ethanol/methanol-water mixture concentrations. Sensor tip measurements are made by preparing distilled water-ethanol and water-methanol mixtures in different concentrations (range 0-16.66%). The response of each sensor tip is obtained by performing repeated measurements of mixtures. At the same time, the refractive indices of each mixture are measured. Depending on these values, the sensor responses are determined according to the refractive index changes and determined that the sensor tip with a diameter of 2 mm showed the best performance in ethanol measurements. The sensor tip detected the refractive index change of 4×10^{-4} RIU in the mixtures, and its sensitivity is determined as 7.71 mV/RIU. In methanol measurements, it is determined that the 3 mm diameter sensor tip exhibited the best performance. The sensor tip detected the refractive index change of 1×10^{-4} RIU in the mixtures, and its sensitivity is determined as 28.49 mV/RIU. Based on the data obtained, it has been shown that the proposed sensor tips can detect low amounts of ethanol and methanol changes with acceptable sensitivity in the production of alcoholic beverages.

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