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Research paper

Alcohol Detection in Consumer Products via POF Fiber Sensor and Spectroscopy Techniques

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Abstract

The presence of ethyl alcohol in food and beverages is impermissible in several countries. Therefore, the detection of these chemical substances is essential for preserving halal food industries. The measurement on the concentration of ethyl alcohol in water was conducted based on two techniques; where these are the POF sensing system and the absorption spectroscopic techniques. The detection of low concentrations of ethyl alcohol were performed using plastic optical fiber (POF) bent into U-shape and coil-shape, while absorption spectroscopy was used for the detection of ethyl alcohol at higher concentrations. The measurement of ethyl alcohol concentrations in water were in the range of 0.1 to 100.0 %v/v. Real-time measurements demonstrated that the POF sensor responded well to concentrations from 0.1 to 1.0 %v ethyl alcohol in water with sensitivity of 7.8 per % v/v ethanol concentration. While spectroscopic technique was used to detect higher ethyl alcohol concentrations in the range of 1.0 – 100.0 %v/v. Several selected consumer products have shown to exhibit presence of alcohol in various concentrations below 50 %v/v. Some of these products do not have label on them and some have undeclared content of ethyl alcohol.

Keywords: *Alcohol detection; Ethyl alcohol; Halal food industry; POF fiber sensor; Spectroscopic technique*.

1. Introduction

Ethyl alcohol is the main constituent found in alcoholic beverages and other products that undergo fermentation, where the effect of intoxication from synthetic and fermented ethanol is almost similar [1-2]. Submitting to certain religious demands in establishing the limit of ethanol in food industries is required to facilitate food production [2]. The Holy Quran revealed that alcohol encompasses some good and some evil, but the evil is greater than good: “They ask you about intoxicants and gambling. Say: In them there are great sin and yet some benefit for people, but their sin is greater than their benefit” [3]. The verse expresses the harmful effect of intoxicant (such as wine and drugs) to human, and advice for abstaining from consuming alcohol. Several countries with population of consumers reaching hundreds of millions have limit and control to the use of alcohol in food and drink products [4], as shown in Table 1. Although there are labeling requirements in some countries on products containing alcohol for some health reasons, however some manufacturers failed to follow and abide to the regulations. Ethanol might not be declared in some consumer products or may contain the incorrect amount stated on the packaging products.

Table 1: Percentage of permitted ethanol concentrations in food and drink products in some countries.

Countries	Organizations	Limit of Ethanol (%)
Indonesia	Majelis Ulama Indonesia (MUI)	0.0% synthetic ethanol in final food products) [5], 1.0% natural ethanol [6],
United States of America	Islamic Food and Nutrition Council of America (IFANCA)	0.5% synthetic ethanol in flavourings , 0.1% synthetic ethanol in the final food products [5]
Malaysia	Jabatan Kemajuan Islam Malaysia (JAKIM)	1.0% natural ethanol in non-alcoholic soft drinks., 0.5% synthetic ethanol in the final food products [5]
Thailand	1. Thai Food and Drug Administration 2. The Administration of Organization of the Islamic Act (AOI)	0.5 % natural ethanol or synthetic ethanol [3], 1.0 % natural ethanol or synthetic ethanol [5]
Brunei	Brunei Islamic Religious Council (BIRC)	2 % natural ethanol, 0.0% synthetic ethanol [5][6]
Singapore	Majlis Ugama Islam Singapura (MUIS)	0.5% synthetic ethanol in flavourings, 0.1% synthetic ethanol in food products [5][6]
Europe	The Halal Food Council of Europe (HFCE)	<0.5 % synthetic/natural ethanol [5][6]
Canada	Canadian Halal Food Certifying Agency (CHFCA)	Not allowed [5][6]
United Kingdom	The Muslim Food Board (UK)	Not allowed[5][6]

Canada and United Kingdom are more conservative to prohibit ethanol in all food products while Brunei only banned the usage of synthetic ethanol [5, 6]. A reliable portable device for real-time measurements is required for quantifying ethanol compounds in consumer products, which can detect low percentage of ethanol in mixtures [7]. Plastic optical fiber ethanol sensors have been reported [8 – 11] and all these sensors reports on detecting low concentrations of ethanol. However, for higher concentrations, the POF sensing element tends to swells due to the reaction of polymeric material [poly(methyl methacrylate), PMMA] of POF sensing element with ethanol and causing irregular nonlinear response.

The swelling of POF sensing element lowers the refractive index of POF and more evanescent waves escaping out from the solution, thus lowers the intensity of light being detected. Here, we present a simple demonstration of POF ethanol sensor that can detect low concentrations of ethanol in water mixture with the range of 0.1–1.0%v/v; which is of appropriate sensing level to be deployed in countries suggested in Table 1.

Spectroscopic technique has been used to identify, thus verify the presence of ethyl alcohol in mixtures based on the absorption frequency thumbprints over a broad range of spectrum in the ultraviolet, visible and infrared regions [12, 13]. Hence, it serves both purposes, identifying traces of ethyl alcohol in solution mixtures and quantifying ethyl alcohol at higher concentrations. Here, we present the absorption characteristics of alcohol with concentration in the range of 1.0–100.0%v/v within near-infrared (NIR) wavelength regions. Several consumer products in the market were selected and tested in identifying and quantifying the percentage of alcohol compound in the products.

2. Experimental

The experimental work was to determine the compositions of ethanol at low and high concentrations. Two separate experiments were setup and these are the plastic optical fiber sensing system and spectroscopic technique. Short length of POF with the fiber jacket removed were bent into U-shape and coil-shape and these will act as the elements for sensing ethanol concentrations in water mixtures. Evanescent waves escaping out around the corners of the bend resulted in lowering the intensity of light detected at the output end. Tighter bending of POF caused more evanescent waves to leak out into the solution. Furthermore, changes in refractive index surrounding the bent POF affected the amount of light leaking out to the surroundings. Sensitivity of the POF sensory system depends very much on the ability to detect the difference on the change of refractive index surrounding the fiber bend. The outside bending radii of U-shape (r_1) and coil-shape (r_2) are 4 mm and 6 mm, respectively, as shown in Fig. 1.

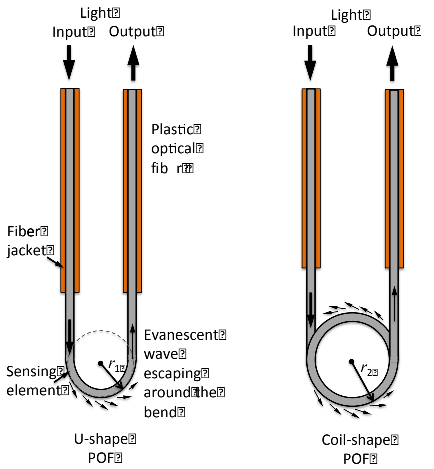

Fig. 1: U-shape and coil-shape POF with outside bending radius of 4 mm and 6 mm, respectively.

The light injected into U-shape and coil-shape POF sensing elements was from a 1 mW red LED light source with about 25 nm spectral width at FHWM and the output was detected using Ocean Optics USB4000 spectrometer as illustrated in Fig. 2. Data was recorded via computer-based Ocean Optics SpectraSuite software. The two POF sensors were tested for the detection of ethanol-water mixtures from 0.1 to 1.0 % v/v ethanol concentrations placed inside black box.

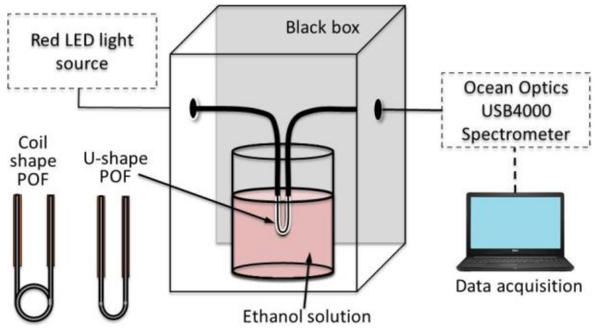

Fig. 2: U-shape and coil-shape POFs sensing system utilizing red LED and spectrometer.

Near infrared absorption spectroscopy for qualifying and quantifying ethanol-water mixture was setup as demonstrated in Fig. 3. Light emission from Avantes AvaLight-Hal tungsten halogen lamp of wavelength range 0.25 to 2.5 µm was used as broadband light source, spectrometer from Avantes model AvaSpec-2048 and computer for spectral acquisition.

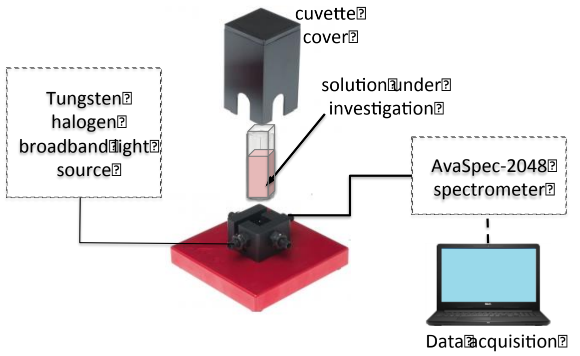

Fig. 3: NIR absorption spectroscopic measurement for 1.0 – 100.0 % v/v concentration of ethanol-water mixtures and some consumer products.

Several consumer products were selected for detection of ethanol composition, where some of the products have undeclared content of ethanol in the products. The experimental setup was used for identify the content of ethanol in the products and simultaneously determines the quantity of ethanol concentrations. Concentrations of ethanol in water mixtures from 1.0 – 100.0 % v/v were used as the control samples and act as a reference to ethanol concentrations in the measured samples of consumer products. These commercial products are listed in Fig. 4.


(a) Gin and tonics (Ethanol content from label: 6.3%)


(b) Vodka (Ethanol content from label: 35%)


(c) Listerine Original Ethanol content: 26.9% - 31% [15][16][17]


(d) Listerine Bright and Clean Ethanol content: 25-26% [15][16][17]


(e) Listerine Cool Mint Ethanol content: 18-22% [15][16][7]


(f) Listerine Green Tea Ethanol content label: 0%


(g) Colgate Plax Ice


(h) Colgate Plax Fruity Fresh


(i) Plandula Herbal Base Oralcare

Fig. 4: Selected consumer products used for detection of ethanol content and their concentrations.

3. Results and Discussion

3.1. Low ethanol concentration detection from U-shape and coil-shape POF sensor

Concentration of ethanol-water mixture was tested for spectral response from POF sensor in the range of 0.1 to 1.0 % v/v. The percentage of ethanol-water concentration that was tested was made to acceptably low, so as not cause the swelling of POF sensing element, which might lead to nonlinear responses to the change of mixture concentrations. Material for plastic optical fiber is generally made from poly(methyl methacrylate) (PMMA). Mixtures of ethanol-water are quite often being used as solvent for PMMA and for 80% ethanol concentration, it has shown to have maximum solubility. However, low ethanol concentration did not have significant reaction towards POF and did not cause swelling, thus it is acceptable to be used as ethanol sensor to within this limit. Consistent results were obtained over repeatable measurements from POF sensors for the range of ethanol concentrations.

The fiber jacket (cladding layer) bending portions of POF for U-shape and coil-shape were removed for light interaction with surrounding medium in determining the properties of the medium. Assuming the POF sensing element did not swell when immersed in the mixture, the POF refractive index would remain the same before and after immersion into the mixture. Interaction at the interface of POF and medium modulates the light intensity due to the change in refractive index of the medium. Some amount of light energy is lost from the bent POF as evanescent waves into the surrounding medium. The refractive index difference between the POF and the ethanol-water mixture surrounding the U-bend POF would reduce, thus light will be loosely guided and eventually more evanescent wave escape out into the surrounding medium. The change in refractive index of the medium is due to the change in chemical concentration of the medium. The evanescent wave attenuates exponentially with radial distance from the POF-medium interface. The depth of penetration of evanescent waves decreases with increasing refractive index contrast at the POF core and medium (cladding) interface [11, 14]:

$$d_p = \frac{\lambda}{2\pi\sqrt{n_{POF}^2 \sin\theta_i - n_{medium}^2}} \quad (1)$$

where λ is the wavelength of the incident light, θ_i is the angle of incidence of light, n_{POF} and n_{medium} denote the refractive indices of core of POF and mixture medium (cladding), respectively.

Fig. 5 shows that with increasing concentration of the ethanol-water mixture, the penetration depth increases and the absorbance of the light into the medium also increases, thus lowers light intensity detected at the POF output. Concentration of ethanol-water mixture is proportional to the refractive index of the mixture. The U-shape POF sensor responded linearly towards variation of ethanol-water concentrations as shown from the spectral intensities. The bending radius of POF is one of the factors that determines the sensitivity of detecting the change in refractive index surrounding the POF along the bend. With a bend radius of 4 mm of U-shape POF, it is sufficient to detect 0.1 % v/v ethanol concentration. The linear regression coefficient R^2 is 95.04%, while the sensitivity of the sensor in terms of optical intensity and mixture concentration is 1.339 per % v/v where it was derived from the slope of the graph in Fig. 6.

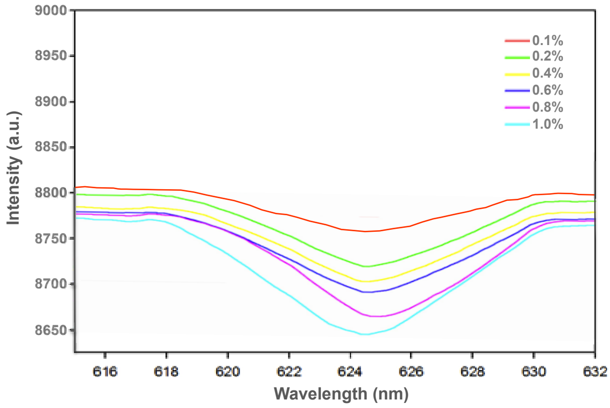

Fig. 5: Spectral response of U-shape POF from 0.1 – 1.0 % v/v of ethanol concentrations in water.

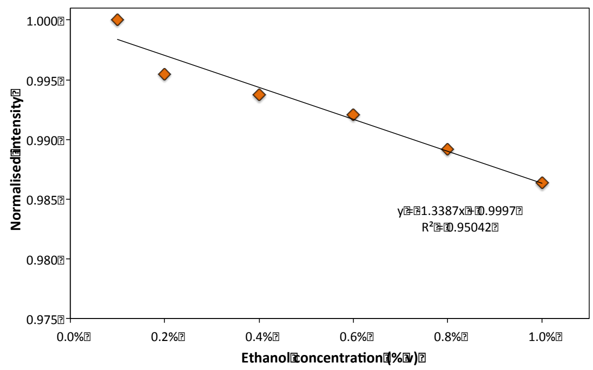

Fig. 6: Response of U-shape POF sensor to ethanol concentration at 1.0% v/v and below, with sensitivity of 1.339 per %v/v and 95.04% linearity.

One and half turn bend of coil-shape POF was to gain higher sensitivity from the sensing element through longer interaction length along the bend with anticipation of larger number of evanescent waves escaping into the medium and it has shown to be more effective, where the sensitivity has improved to 7.8 per %v/v with 97.3% linearity, as shown in Fig. 7.

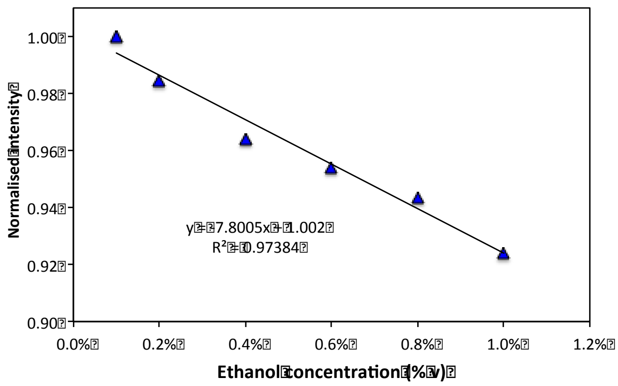

Fig. 7: Coil-shape POF sensor responded to ethanol-water mixture with higher sensitivity.

Smaller bending radii tends to allow more evanescent wave to escape out into the mixture, where deeper depth of penetration at smaller incident angles, thus higher absorption into the medium resulting higher sensitivity. Micro-cracks might developed around outer part of the fiber bend and the fiber shape along the bend can also deformed while bending the fiber into shape. These defects in the POF sensing element would eventually create undesired response. These micro-cracks and fiber deformation can be avoided using appropriate technique in bending the fiber with appropriate thermal treatment during the process. The plot in Fig. 7 was derived from the optical spectra in Fig. 8, where the dips in light intensity increased with higher ethanol-water concentrations.

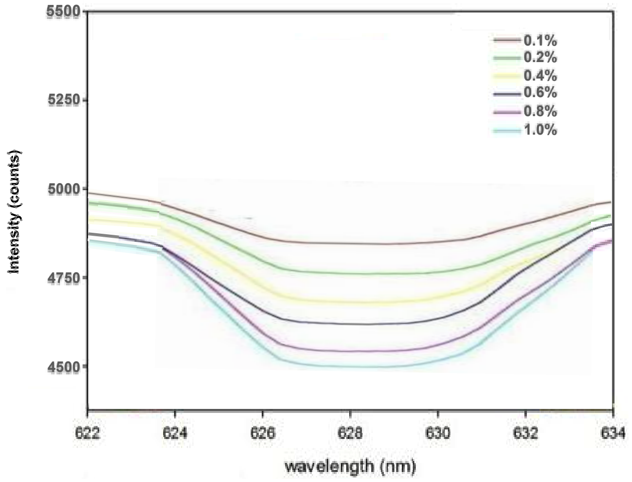

Fig. 8: Spectral response from coil-shape POF sensor.

3.2. Quantifying and qualifying ethanol in consumer products via spectroscopic technique

Fig. 9 shows the spectral absorbance of light from 1.0 - 100% v/v ethanol-water mixture and the absorbance is directly proportional to the concentration of the mixture as shown in Fig. 10. The two peaks of the spectra at around 1.695 µm and 1.735 µm are the distinct characteristics of ethanol in the near-infrared (NIR) wavelength region. These peaks are the fingerprints of ethanol in this region, and these significant peaks are shown in the typical spectrum (red line) in inset of Fig. 9 [13]. The blue line is absorbance spectrum of water.

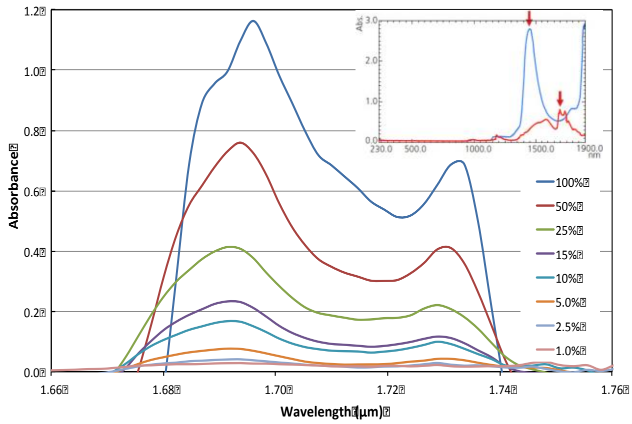

Fig. 9: Absorbance spectrum of ethanol-water mixtures at 1.66 µm – 1.74 µm wavelength range. Inset: Absorbance spectra of water and ethanol (blue line: water; red line: ethanol) – cited from Shimadzu Scientific Instruments [13]

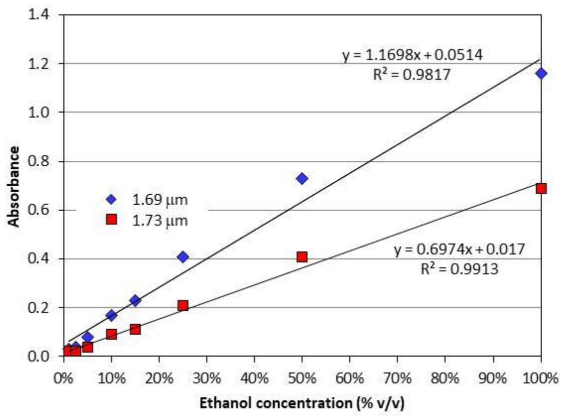

Fig. 10: Absorbance of light for ethanol concentrations ranging from 1% - 100% for 1.69 µm and 1.73 µm peak wavelengths.

The modulated light intensity received at the output detector was due to the absorption of light with the change of the ethanol concentration. The peak absorption wavelengths are at 1.69 µm and 1.73 µm, where both of these wavelengths indicate linear response to ethanol concentrations, with the absorption at 1.69 µm having slightly higher response. The presence of ethanol in a mixture can be identified from the absorbance spectrum in the NIR wavelength region. Concurrently, the ethanol concentration can also be quantified, thus these results can be used as control samples and reference guide for detecting and measuring the percentage of ethanol concentrations in all other compounds, such as shown in Fig. 11.

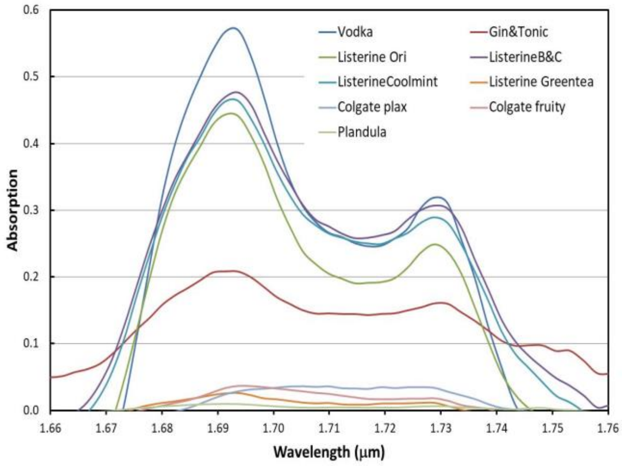

Fig. 11: Absorption spectra of selected commercial samples measured in the NIR wavelength region.

Selected commercial products of common essentials and beverages were tested for ethanol content and their contents were compared to the reference samples for the amount of ethanol presence in the samples. The contents of alcohol specified on labels of vodka, gin and tonic are 35 % and 6.3 %, respectively. Comparing their amount to the reference samples, vodka shows slightly higher in alcohol contents to about 37.5%, while gin and tonic exceed 6.3% to about almost 13%.

The gin and tonic with the brand name of Storm Twistonic has higher than usual alcohol content of 6.3%. In some countries such as in UK, quantifies the actual alcoholic content in drinks, and gin itself can have at least 37.5% alcohol by volume. Combination of gin and tonic would be double the amount of alcohol content. Storm Twistonic might have specified 6.3% volume of alcohol for gin and the volume could be double (close to 13%, as in Fig.12) for gin with tonic combined, however this assumption could not be verified as the product specifications varies from manufacturers. Mouthwash Listerine “cool mint”, and “bright and clean” are found to be exceeding the amount of alcohol specified by their labels. While the content of alcohol of Listerine “original” was measured to be within the limit stated. Traces of alcohol found in Listerine “green tea”, Colgate “plax ice” and “plax fruity fresh”, and Plandula “herbal based oral-care” could be resulted from the formation of natural alcohol from the reaction of herbs and fruits with the content of the products.

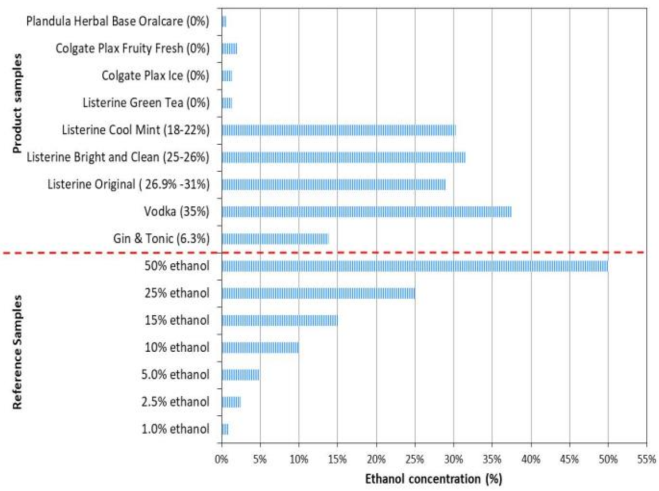

Fig. 12: Percentage of ethanol content in commercial product samples compared to reference concentrations measured at 1.69µm wavelength.

4. Conclusion

An in-situ measurement of alcohol content can be devised to detect alcohol mixture concentration below 1% using simple construction of plastic optical fiber sensor. The spectrometer in the sensing system can easily be replaced with a more robust, compact, cheaper and highly sensitive photodiode without compromising its sensitivity. The portable cost-effective sensing device can be assessable to users in detecting alcohol content conveniently at any commercial premises. Identifying and differentiating presence of ethanol from any other family of alcohols via ethanol spectral fingerprint has been verified through its absorbance properties in the NIR wavelength region. Furthermore, the alcohol content can also be quantified through the whole range of concentration from 1.0 to 100% (±0.5%) using the spectroscopic technique.

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