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Development of optical fiber-based refractive index sensor

AIM:
Development of an optical fiber-based sensor to measure refractive index of the given solution.

OBJECTIVE:
To use the U-bent plastic optical fiber probe as a refractive index-based sensor to calibrate and measure the concentration of sucrose in a given sample.

THEORY:

U-bent plastic optical fiber (POF) probes:
The lab developed portable U-bent POF is highly sensitive and can detect small changes in refractive index of the solution. In addition, it is easy to use, and provides a non-destructive method of evaluation.

The sensor works on the principle of intensity modulation by using evanescent wave absorbance and refractive losses. The fiber optic probe senses a change in the refractive index of the medium due to the attenuated total reflection by the evanescent wave interactions of light at the fiber core/cladding interface with the surrounding medium. A relative change in the refractive index property can be correlated with changes in sucrose concentrations in the samples. Thus, this sensor can be readily used to measure the concentration of any given sucrose sample once calibrated.

U-bent fiber geometry is chosen due to improved evanescent wave interactions which results in enhanced sensitivity. The plastic optical fiber consists of polymethylmethacrylate (PMMA) core and fluorinated polymer cladding with refractive indices of 1.49 and 1.41 respectively. The POF of 200um diameter was bent into U-shape by means of glass capillary technique.

The optical set-up consisted of the U-bent POF probe with its distal ends connected to a halogen light source and a fiber optic spectrometer.

U-bent POF probes have several applications and have been used to investigate changes in refractive index as a result of variation in milk fat content and addition of water. Gold sputtered U-bent POF probes have also been used as SPR- and LSPR-based compact plasmonic sensors to measure refractive index with high sensitivity.

Beer-Lambert Law:

The Beer-Lambert Law describes the relationship between the attenuation of light through a substance with its properties. According to the law, there exists a linear relationship between the absorbance and the concentration. The absorbance is directly proportional to the concentration of the sample and the path length used in the experiment.

Absorbance (A) = $\boldsymbol{\varepsilon c l}$ **,**

where ε is the molar extinction coefficient ($mol^{-1}\ cm^{-1}$) or absorptivity, c (mol) is the molar concentration and l is the path length (cm).

Considering monochromatic light transmitted through a solution with an incident intensity of I_0 and a transmitted intensity of I,

Transmittance (T) = $\mathbf{\frac{I}{I_0}}$

Absorbance (A) = $\mathbf{\log_{10}\frac{I_0}{I}} = -\mathbf{\log_{10}} \boldsymbol{T}$

From the above equations,

$\frac{I}{I_0} = e^{-\varepsilon c l}$

The equation holds good at low concentrations and has certain limitations:

- Non-linear behavior at high concentrations due to deviation in absorptivity as a result of electrostatic interactions.
- Large scattering of light due to particulates may result in non-linearity.
- Fluorescence or phosphorescence of the sample may also result in non-linearity.

Refractive index:

The refractive index of a material is an important optical property of a material medium. It is a dimensionless number that describes the speed of light in the medium relative to vacuum.

$$\text{Refractive index (n)} = \mathbf{\frac{c}{v}}$$

where c is the speed of light in vacuum and v is the phase velocity of light in the medium.

The refractive index determines how much the path of light is bent, or refracted and the factor by which the wavelength changes when entering a material. This is described by Snell's law of refraction, $n_1 \sin\theta_1 = n_2 \sin\theta_2$, where θ_1 and θ_2 are the angles of incidence and refraction, respectively, of a ray crossing the interface between two media with refractive indices n_1 and n_2. The refractive indices also determine the amount of light that is reflected when reaching the interface (Fresnel equations), as well as the critical angle for total internal reflection.

The concentration of the sample, wavelength of light used, temperature and pressure are important parameters that affect the refractive index.

APPARATUS & MATERIALS USED:

Weighing machine, sucrose (powdered crystals), 1.5ml Eppendorf PCR tubes, pipette (20 – 200ul), pipette tips, deionized water, Lab developed U-bent sensor with core: 200um optical fiber (Thor Lab), light source, photo detector (Model No: SI50C), RS232 Energy meter (Thor Lab), spectrometer (Ocean Optics Inc., USA) SMA connectors (BFT1 and B10510A Thorlabs Inc., USA).

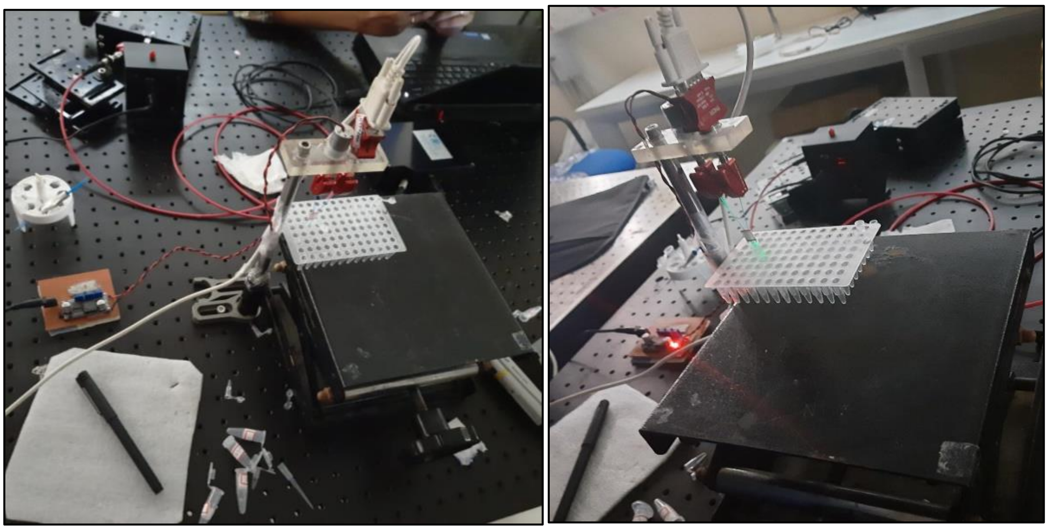

Fig1: Experimental Setup

Fig2: Thor Lab PM Series software

PROCEDURE:

1. Sucrose in powdered form was weighed to generate five different concentrations- 5.2mg, 10.3mg, 15.11mg, 20 mg, 25.6mg and stored separately in five 1.5ml Eppendorf PCR tubes.
2. 100ul deionized water was added to each of the five tubes. The samples were well mixed to completely dissolve the sucrose. Another PCR tube with 100 ul deionized water was used as the blank reference.

3. The absorbance spectra was captured in real time using green light at 530 nm. Reference spectrum was taken while the decladded sensing portion of the U-bent probes was dipped in DI water, before subjecting the probes to various sucrose concentrations. The sensor response was recorded using the Thor Lab PM Series software (Fig2).
4. The absorbance vs sucrose concentration was investigated.
5. The refractive index vs sucrose weight percentage (Brix) was found from calibrated data from International Commission for Uniform Methods of Sugar Analysis (ICUMSA 1974).
6. The refractive index corresponding to the five sucrose concentrations in the PCR tubes was found by extrapolation of the ICUMSA data.
7. Next, the absorbance vs refractive index relationship was investigated for the sucrose concentrations in the PCR tubes.
8. A calibration graph is proposed which allows us to find the refractive index of any known sucrose concentration (% w/w) from the absorbance data.

RESULTS & CALCULATIONS:

Density of water = 1g/ml
100 ul = 0.1 ml. Therefore, 0.1 ml weighs 0.1g or 100 mg

SNo.	Mass of sucrose in 100ul (100 mg) DI water	%(w/w) (Brix)	Output power (uW per unit area)	Absorbance at 530nm $-\log_{10}\frac{I}{I_0}$	Refractive Index (from ICUMSA 1974)
1	0	0	2.111	0	1.33299
2	5.2	5.2	2.012	0.0209	1.3406
3	10.3	10.3	1.883	0.0496	1.3483
4	15.11	15.11	1.763	0.0782	1.3559
5	20.0	20.0	1.618	0.1155	1.3638
6	25.6	25.6	1.491	0.1510	1.3734

Reference with respect to blank: I_0 = 2.111uW (per unit area)

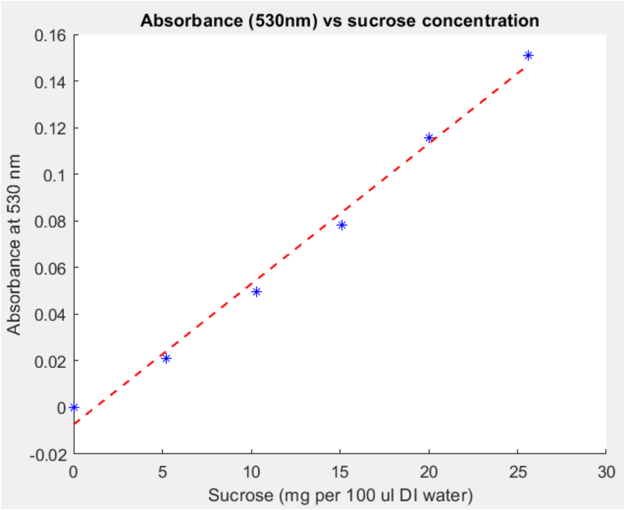

Fig3

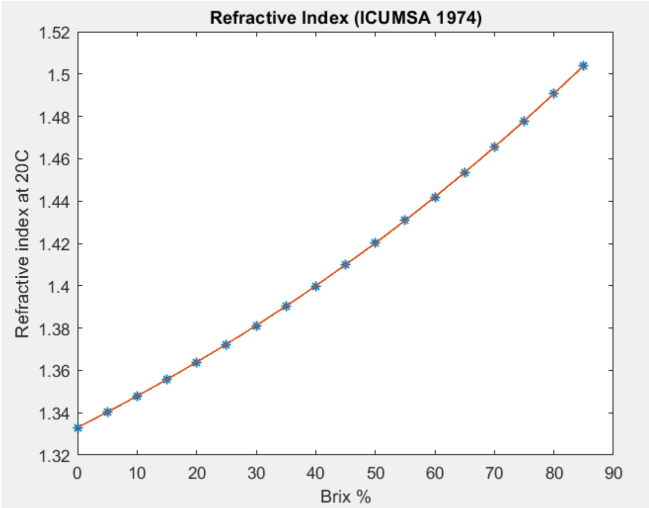

Fig4

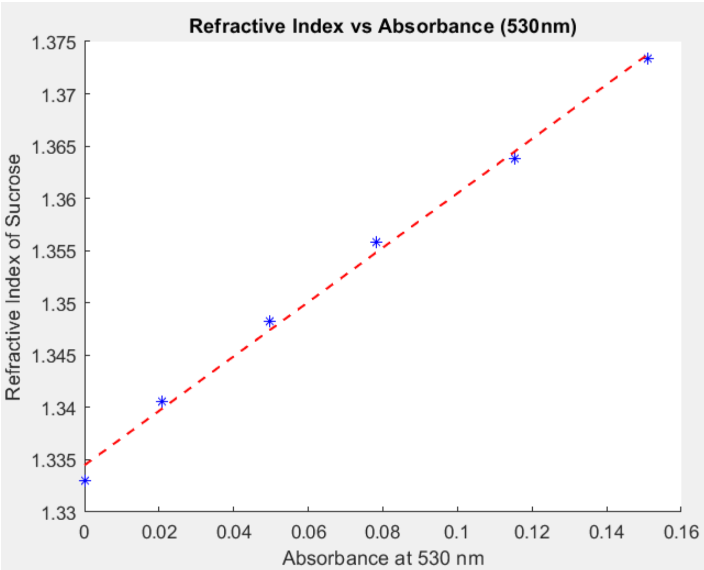

Fig 5

The absorbance varies linearly as the concentration of sucrose increases (Fig3) as supported by the Beer-Lambert Law.

The refractive index for various Brix was obtained from ICUMSA 1974 [Ref 1]. One degree Brix is **1 gram of sucrose in 100 grams of solution** and represents the strength of the solution as percentage by mass.

The refractive index corresponding to the sucrose concentrations in the Eppendorf tube was found by extrapolation from Fig 4.

The calibration graph for the Refractive Index vs Absorbance (530nm) shows a linear behavior (Fig 5).

CONCLUSIONS:

A refractive index sensor has been developed using the U-bend probe. Thus, given any unknown sucrose sample of known concentration, the corresponding absorbance and refractive index can be calculated from the calibrated data or vice versa.

CRITICAL REMARKS:

1. Accuracy in experiment procedure would ensure better calibration of the data to obtain a more sensitive sensor.
2. The Brix calibration was obtained at 20 ^oC, while the room temperature while performing experiments was 27-28 ^oC. Refractive index data at room temperature would provide more accurate results for the calibration.
3. Green light has been used for this calibration. The refractive index is sensitive to the wavelength of light used.

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