Chapter 4

GLUCOSE CONCENTRATION MEASUREMENT USING FIBRE OPTIC SENSOR

Abstract

Design and development of an evanescent wave fibre optic sensor for the determination of glucose concentration is described in this chapter. This simple and inexpensive optical fibre based device has been found to be very sensitive at low glucose concentrations with saturation at about 4 gm/litre. This sensor works in a direct fashion and overcomes most of the complexities of the existing techniques for glucose detection. Fabrication details and performance evaluation of a hand held version of the fibre sensor are also given in this chapter.

4.1 Introduction:

The quantitative estimation of glucose is very much essential in different fields of applications like analysis of blood and urine samples, food analysis, industries etc. There are a variety of classified chemical methods for the quantitative determination of glucose in blood and urine samples both in normal and pathological states. We can see the continuous efforts by biochemical workers to evolve methods which are more specific, more rapid and require small amount of sample. The majority of methods for the determination of blood glucose are based on the ability of glucose, in hot alkaline solution, to reduce certain metal ions. The extent of reduction is then estimated by photometric, titrimetric or gasometric methods. Some of the methods are Flin and Wu method, Somogyi-Shaffer-Hartmann method, Nelson-Somogyi method etc. Similarly a variety of chemical methods like Benedict's method, Hankin and Van Slyke method etc. can be seen for the quantitative determination of glucose concentration in urine samples. Apart from the above-mentioned classical chemical methods, we can find a different branch, an instrumental method, which involves enzymatic determination of
glucose. Here, the glucose concentration is determined using an enzyme electrode and this method is completely specific and require only a small amount of sample. The working principle of this method is that, in the presence of enzyme, glucose oxidase, an aqueous solution of glucose undergoes oxidation to gluconic acid with the formation of hydrogen peroxide that can be determined by anodic oxidation at a fixed potential.

Here, in this chapter we are describing a new approach, an optical fibre based glucose sensor, which works on the principle of evanescent wave absorption phenomenon. Evanescent wave spectroscopy is one among the challenging techniques for monitoring chemical processes as well as detection of chemical and biological species. In these types of sensors, the transducing mechanism between a chemical or biological measurand and light intensity can be optical absorption or fluorescence. The intensity of the interrogating light wave coming out of the fibre is directly related to species concentration. By making use of proper calibration, the device can be configured for the detection and measurement of glucose concentration. This simple and inexpensive method has a wide range of applications, where quantitative measurement of glucose concentration is necessary and essential. Apart from the biomedical and chemical applications, this sensor can be used for industrial applications that require on-line monitoring of glucose concentration. Like other optical fibre sensing techniques, this evanescent wave glucose sensor has a lot of merits. This method is very sensitive and the device is immune to electromagnetic interference. Besides these features, it can be used in industries with capability for remote sensing.

4.2 Experimental details:

In order to utilise the evanescent waves in a multimode fibre, a known length (0.09 m) of the cladding portion of a multimode plastic clad silica fibre (200/380 μm) is chemically removed. The procedure adopted for removing the cladding of the fibre is as follows. The plastic sheath of a certain portion of the fibre is removed using a blade and this portion of the fibre is immersed in concentrated hydrofluoric acid for a specific period of time. Then we can easily remove the cladding of the fibre. A sensor cell is
designed for taking the test liquids. The cell consists of a cylindrical glass tube of length 0.11m having a diameter 0.01m and the two sides of the tube are closed using two glass plates. Also, the cell is provided with inlet and outlet provisions. The optical fibre is introduced into the container through the holes provided on the side plates and is permanently fixed such that the uncladded portion of the fibre is straight within the container. This uncladded region acts as the sensing element. Figure 4.1 represents the fibre optic sensor (FOS) arrangement employed in the present investigation.

![Figure 4.1 Experimental set-up to monitor glucose concentration](image)

A 4.25 mW diode laser (LASERMAX INC.) emitting at 670 nm is launched into one end of the fibre and the light emitting through the farther end is fed into a JETRONICS SO 239 photomultiplier tube (PMT). The ends of the fibre are well polished so as to get optimum coupling. Light from the diode laser is launched into one end of the fibre using a short focal length lens and the transmitted power is detected using the PMT. The glucose solution is prepared in distilled water for various concentrations ranging from 0.001 gm/litre to 10 gm/litre. This glucose solution along with Benedict's reagent (quantitative reagent) are mixed in a pre-decided ratio (1:3) and heated on a boiling water bath for five minutes and allowed to cool for some time. This resultant solution acts as the test liquid. When the uncladded region is immersed in the test solution, the evanescent field penetrates into the liquid and interacts with it. As the beam propagates through the fibre it results in the coupling of evanescent wave (EW) to the medium surrounding the core, thereby attenuating the propagating beam.
Usually in fibre optic sensors working on the principle of evanescent wave absorption phenomenon, light of wavelength close to the peak absorption wavelength of the absorbing fluid is launched into the fibre. Then the change in intensity of the transmitted light occurring due to the absorption of the evanescent field penetrating into the medium is measured. The output light is detected using a PMT-volmeter combination and the change in light intensity is measured in terms of change in voltage. Thus for various glucose concentrations, the output voltages have been measured.

4.3 Results and discussion:

Figure 4.2 shows the variation of the output intensity in terms of change in voltage with glucose concentration. The change in the intensity can be attributed to the evanescent wave (EW) absorption taking place at the operating wavelength ($\lambda = 670$ nm) in the sensing region.

![Figure 4.2](image)

**Figure 4.2** Variation of output light intensity with glucose concentration.
The absorption spectra for the test liquids at various glucose concentrations are taken using a UV-Vis-NIR spectrophotometer (SHIMADZU Model No. UV - 160 A). A typical absorption spectrum of the experimental liquid at a glucose concentration of 4 gm/litre is given in figure 4.3.

It is observed that there is a shift (807 nm to 730 nm) towards the propagating wavelength (670 nm) for the maximum absorption wavelength of the test solution with glucose concentration. This enhances the EW absorption resulting in a change in output intensity. As the concentration increases, shift is found to increase. At higher concentrations, the graph shows a saturation. However, the device can be used for reliable measurements up to a glucose concentration of about 4 gm/litre. The evanescent absorbance $A$ of the uncladded fibre is given as
where 'r' is the fraction of the power outside the core, $\alpha$ is the bulk absorption coefficient of the surrounding medium and $L$ is the length of the uncladded region of the fibre. This shows that for a fluid medium obeying Lambert-Beer law of absorption, evanescent absorbance depends linearly on the exposed fibre length, fluid concentration and the $V$ parameter. From our investigations we have found that variation of the output intensity at lower concentrations is linear to a fair approximation and at about 4 gm/litre, it shows saturation. Thus the plot can be divided into two portions, a linear region and a saturation region. This nonlinearity is due to the adsorption of the white precipitate on the fibre core, which is formed as a result of the reaction between Benedict's quantitative reagent with glucose solution on heating. Benedict's quantitative reagent contains potassium thiocyanate as well as copper sulfate and in the presence of the former a white precipitate of cuprous thiocyanate is formed on reduction by glucose. The small amount of potassium ferrocyanide in the quantitative reagent also aids in keeping cuprous oxide in solution. As the concentration of glucose in the test solution increases, more and more quantity of Benedict's reagent is consumed in the chemical reaction with glucose, which in turn increases the amount of white precipitate produced.

Different groups have observed this nonlinear behaviour of the output light intensity with concentration of the fluid in the uncladded region. Followed by our investigations on evanescent wave fibre optic sensor for the glucose concentration measurement, L M Bali et al developed an optical sensor based on monitoring the light scattered by the red particles of cuprous oxide produced as a result of the reaction between glucose and Benedict's qualitative reagent. These investigations have also shown that there is a saturation at about 4 gm/litre for a test solution taken in the ratio 1:3. One of the limitations of using Benedict's qualitative reagent is that it can also be

$$A = \frac{r\alpha L}{2.303}$$
reduced by many other reducing substances that are not carbohydrates, such as glucuronic acid, salicylic acid, uric acid, creatinine and homogentisic acid etc.62

From our investigations, it is observed that the optimum ratio of glucose solution with Benedict’s reagent is 1:3. Core of the fibre used for our sensor is made of silica and its surface is neutral having no net surface charge. When this uncladded fibre is immersed in aqueous solution of glucose, H+ and OH− ions as well as H2O react with the surface to form an amphoteric hydroxylated layer on the surface of the silica core of the optical fibre.72 The white precipitate formed due to the reaction between glucose solution and Benedict’s reagent will experience considerable electrostatic attraction towards the silica core which leads to surface loading of the silica core and subsequent saturation of the output intensity.

Our fibre optic sensor system can be employed to monitor glucose concentration at low levels with good sensitivity. The dynamic range of the sensor can be varied by increasing the length of the uncladded region of the fibre. Even though a wide range of conventional and cheap methods are available for monitoring glucose in urine, their detection range is well above 0.2 gm/litre, which is the physiological limit.62 The fibre optic sensor presented here with very high sensitivity at very low concentrations is ideally suited to monitor glucose even below this limit of 0.2 gm/litre. Also, the urine required for analysis can be kept small.

By making use of the advantages of the optical fibre sensing technology and the results obtained in our investigations, we have fabricated a hand-held device for the determination of glucose concentration. This FOS is made cost effective by using an LED instead of a laser diode and by replacing the photomultiplier tube by a photodiode. The block diagram of the hand-held fibre optic glucose sensor is presented in figure 4.4.
Protective coating from a small portion of the fibre is removed using a blade and this portion of the fibre is immersed in tetrahydrofuran for about 10 minutes. Then this cladding region can be easily removed which acts as the sensor head. The light propagating through this multimode fibre is detected using a PIN photodiode (Motorola MFOD 71). The signal is amplified in two steps. First, the signal is amplified using a current amplifier which involves OP 07 and the second stage is assembled using two stages of TL072 (amplifier). The circuit diagram for the detector-amplifier system is as shown in figure 4.6.

![Figure 4.6 The circuit diagram of the detector-amplifier system](image)

The amplified analog output is then converted to digital signals using an A/D converter and displayed on an LCD. Investigations carried out using this sensor have shown a linear response for low glucose concentrations. Measurements have also been carried out by replacing the LED by a laser diode emitting at 670nm (4.25 mW). The response obtained for different glucose concentrations is given in figure 4.7.
Figure 4.7 Response of the fibre optic sensor device (a) concentration vs. output voltage (b) log (concentration) vs. output voltage
4.4 Conclusion:

In this chapter we have discussed the design and development of an optical fibre based glucose sensor. It offers very good sensitivity at low glucose levels and its linear response region is very much within the physiological limit and hence can be used for the quantitative determination of glucose concentration in urine samples.

Even though it is inexpensive, there are a few drawbacks for this sensor. It needs heating of the test solution. Another disadvantage is the adsorption of the white precipitate on the fibre core formed as a result of the reaction of glucose solution with Benedict's reagent. This limits its application to disposable devices to measure glucose concentrations.