Suction measurements using fiber optic sensors

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Suction Measurements Using Fiber Optic Sensors

Tay Chia Meng, Benjamin

School of Electrical & Electronic Engineering

A thesis submitted to the Nanyang Technological University
in fulfillment of the requirement for the degree of
Master of Engineering

2005

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research and has not been submitted for a higher degree to any other University or Institution.

05 November 2004

Date

A handwritten signature in black ink, appearing to read 'Ben Tay', is positioned above a horizontal line. The signature is written in a cursive, fluid style.

Tay Chia Meng, Benjamin

Acknowledgements

Life is a sojourn where the journey is the destination. Likewise in this master of engineering project, the journey itself has been exhilarating, with many pitfalls and rewards, an experience ultimately making me a better person. Someone once said: “When there is no hope, there can be no endeavour”. Indeed, sustaining the hope throughout the project is not an easy thing, as I only managed to obtain positive results in the remaining 6 months of my 2 year course. I overheard in the canteen one day an exchange from a lecturer to his student; about how postgraduate students successfully complete their courses. The lecturer said: “Those who successfully complete their postgraduate studies are able to overcome their fear of not being able to find anything of significance. Every research student will encounter a period where they find that they are stuck and going nowhere. Those who marshal their hopes and forge on will find that the light at the end of the tunnel is revealed.”

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“We have a habit in writing articles published in scientific journals to make the work as finished as possible, to cover up all the tracks, to not worry about the blind alleys or describe how you had the wrong idea first, and so on. So there isn’t any place to publish, in a dignified manner, what you actually did in order to get to do the work.”

Richard P. Feynman
Nobel Lecture, 1966

Abstract

Soil suction is important in explaining the behaviour of unsaturated soils. Unsaturated soil refers to soils with solid, liquid and gaseous phases in it (i.e. soil particles, soil water and air), and these kind of soils are the most commonly encountered in the field. Knowing the suction of a particular soil would allow the shear strength, volume change and hydraulic behaviour of the soil to be characterised. Having instrumentation that is simple to use and effective in the field that can measure the various suction components of soil would be ideal, but unfortunately this is not presently the case. Soil is not a man-made material; the difficulties involved in measuring soil parameters are well known as soil does not behave in a linear fashion. Experiments done in the laboratory often do not match the soil conditions on the site where the soil was sampled. It is important to know the suction of soil as the knowledge will, for example, enable safety standards to be improved on construction sites, or improve crop yield by providing perfect soil conditions for agriculture.

Soil suction is commonly referred to as the free energy state of soil water and is measured in kilopascals. There exists a thermodynamic relationship between soil suction and the partial pressure of the water vapor in the soil. Several techniques using fiber optic sensors were investigated to determine which was most suitable for soil suction measurements. For direct measurements of water pressure within the pores of soil (matric suction), the methods explored were: 3 dimensional (3D) shape measurement of meniscus of capillary water, followed by hydrostatic pressure measurements using fiber Bragg grating (FBG) sensors and long-period grating (LPG) sensors. For indirect measurements of matric suction using relative humidity (RH) of soil (total suction), the methods investigated were: the use of Fresnel reflection in cleaved single mode fibers (SMF) and gold tip-coated fibers to determine the refractive index change of air when humidity changes, followed by using absorption spectroscopy to monitor water absorption lines to see how they change as humidity changes, followed by using plastic optical fibers (POF) and LPGs coated with humidity sensitive films to form chemical sensors which can sense the humidity in soil.

From the experimental investigations, some methods were found suitable for measuring soil suction. They include using cleaved SMF for osmotic suction measurements (salt concentration in soil water) and LPG RH sensors for measuring the humidity of soil to find the total suction. The method of using Fresnel reflection in cleaved optical fibers to measure changes of refractive index of air due to humidity changes was not feasible as the change was too small, but the experimental setup can be used to measure refractive index changes of different salt concentrations in solutions, making the fiber optic sensor a suitable osmotic suction sensor for use with soil water. The sensor was calibrated with 12 different salt solutions of different osmotic suction values and it responded well to the wide range of suction values.

For total suction measurements using relative humidity, the LPG humidity sensor shows a good response from 90%RH to 100%RH, making it well suited for soil testing. In the soil tests, soil samples using kaolin were prepared with different matric suctions ranging from 50kPa to 400kPa using the pressure plate technique. The difference in humidity between soils of 50kPa and 400kPa is just 0.24%RH. The LPG sensor was tested with these soil specimens to determine if it can distinguish between the humidities of soils of different matric suction. The resolution and accuracy of the sensor is 0.008%RH and $\pm 0.25\%$ RH respectively. The accuracy of $\pm 0.25\%$ RH, due to system limitations, is equivalent to a suction accuracy of about ± 350 kPa. This is the reason why the LPG RH sensor was not able to distinguish between the humidity values of the soil specimens.

In conclusion, an osmotic suction sensor using cleaved SMF and a total suction sensor using LPGs have been investigated and developed successfully in this project. Further investigation and improvement to the sensor and system design are necessary to improve the accuracy will enable the LPG RH sensor to be an excellent fiber optic soil suction sensor.

For further work, it is also possible to develop further new soil suction sensors that can measure pore-water pressure directly using fiber optic LPG interferometric

sensors, which have excellent refractive index sensitivity compared to single LPG sensors. Small changes in the refractive index of pore-water due to hydrostatic pressure change can then be measured accurately.

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Chapter 1 Introduction

1.1 Motivation

Soils belong to two main categories: saturated and unsaturated. Saturated soils consist of just two phases, solids and water. Unsaturated soils, more commonly encountered in the field, consist of three phases: solids, water and air. They can be found naturally above ground water tables or they can be man-made, by means of soil compaction. The voids between soil particles are called pores and water and air coexist together in these pores in unsaturated soils. The pore-water pressure is negative (relative to atmospheric pressure) due to capillary action, and the liquid creates a surface tension which holds soil particles together.

Soil suction, or total suction, comprises of two components, namely matric suction and osmotic suction. Suction values are measured in kiloPascals (kPa). Matric suction is directly proportional to the negative pore-water pressure within the soil. It is a very important stress variable as it controls the mechanical behaviour of soils such as shear strength and volume change. Osmotic suction is dependent on the salt concentration in soil water. Environmental changes such as evaporation or rainfall cause changes in matric suction that can lead to changes in shear strength or volume of the soil. Changes in matric suction can also be caused by changes of load on the soil. Soil failure is caused by loss of shear strength or ground expansion/subsidence due to volume change of soil and this can cause heavy loss of property and human lives.

It is thus important to measure soil suction but how is it measured? There are direct and indirect measurements of soil suction and of the geotechnical devices that measure matric suction directly, the tensiometer is one of the most commonly used. The tensiometer uses a high air-entry ceramic cup in which resides a water chamber. The high air-entry ceramic material allows water to flow through it, but not air, provided the air-entry value of the ceramic is not exceeded. Water in the

chamber comes into equilibrium with the soil water when the ceramic cup is inserted into soil. The pressure of the water in the chamber is measured by an electrical pressure transducer. Some problems encountered include the cavitation of water in the chamber when the negative pore-water pressure exceeds -90kPa and the diffusion of air into the water chamber slowly with time. Such problems prevent the measurement of a wide range of matric suction and also bring into question the long term reliability of the readings.

Fiber optic sensors have been developed for civil engineering applications, in areas like smart-structure monitoring, which requires physical quantities like strain, bending and temperature to be measured. Fiber optic sensors provide numerous advantages over their electrical counterparts. So far, development of fiber optic sensors for soil suction measurements has been limited in scope compared to fiber optic sensors developed for structural monitoring, hence there is potential for the development of such sensors, where existing problems with electrical or mechanical methods can be solved by using fiber optic methods.

1.2 Objectives

The objectives of this project are as follows:

1. To investigate and develop various fiber optic sensing techniques that can be used to measure physical quantities which in turn can be used to find the suction of soil.
2. To design, construct and characterize the fiber optic sensor(s).
3. To test the sensor(s) in laboratory soil tests to determine its viability.

1.3 Major Contribution of the thesis

The emphasis of this project is to investigate and develop various fiber optical sensing techniques which can be used to measure certain physical quantities like hydrostatic pressure or relative humidity (RH) which then can be related to matric suction of soil. For direct measurements of pore-water pressure in soil, several approaches have been investigated. Firstly, three-dimensional (3D) shape

measurement of the meniscus of water in soil capillaries was considered as the radius of the meniscus is indicative of the negative pore-water pressure in the soil. Negative pore-water pressure is directly related to the matric suction of soil. Next, carbon fiber composite embedded fiber Bragg grating (FBG) sensors were used as a diaphragm and tested to measure negative water pressure change in triaxial cells in an attempt to develop a sensor for negative pore-water pressure measurements. The FBGs were fabricated and embedded in carbon fiber composite material in the laboratory. Thirdly, long period grating (LPG) sensors were tested to investigate the feasibility of using the LPG to measure refractive index changes of water due to hydrostatic pressure change. This was also an attempt to measure negative pore-water pressure. This was followed by investigating various optical techniques for indirect measurements of matric suction using relative humidity of soil. The methods investigated include measuring Fresnel reflection from the end of a single mode fiber (SMF) or gold tip-coated fibers to determine the refractive index change of air due to humidity change. A system was built to measure changes in light intensity from the Fresnel reflection of a fiber due to humidity changes. Absorption spectroscopy using fiber collimators was also explored to find out how the absorption line for water changes as humidity changes. This was followed by using cobalt chloride (CoCl_2) mixed with gelatin (overlay material) as humidity sensitive films for coating onto plastic optical fibers (POF). A simple photodetector circuit was built to measure light intensity changes of the specially fabricated POFs humidity sensors, coated with cobalt chloride and other humidity sensitive coatings, when exposed to changes in humidity. Characterization and calibration of the POF humidity sensors were carried out and the data are presented in Chapter 5. Lastly gelatin coated LPGs sensors were designed and fabricated to form a high humidity sensor suitable for soil tests. Characterization and calibration of the LPG humidity sensor were carried out and the data are presented in Chapter 5. The LPG RH sensor was tested in laboratory based soil experiments using prepared soil specimens with known suction values to determine the effectiveness of the sensor for suction measurements by relative humidity sensing.

1.4 Organization of the thesis

The first part of this thesis begins with a brief discussion on the motivation for this study. The objectives of the project are also outlined in the first chapter. This is followed by some background on soil suction and the various devices and techniques used to measure soil suction in chapter two. Chapter two also contains a literature review on optical methods that have been used by other researchers. Chapter three contains the theory behind the various measurement techniques used in the project. Chapter four covers the research development, including research approaches and the experimental procedure of each approach. Chapter five presents the results obtained and further discusses the results. The chapter ends with some concluding remarks regarding the results. Conclusions for the whole thesis are presented in Chapter six. This is followed by a proposal and some recommendations for future research.

Chapter 2 Literature Review

2.1 Introduction to Soil Suction

Figure 2-1: Dry soil with high suction in Punggol, Singapore (Photo by Eddie Tan)

An interesting phenomenon of soil suction in action is shown in Figure 2-1. The soil, having been baked in the sun for some time, loses most of the moisture within thus leading to highly negative pore-water pressure or high soil suction. As the soil suction becomes very large, cracks form as shown in the picture. However, the moisture in the soil has not reduced below the wilting point, a point where the plant cannot survive.

Soil is made up of different phases, namely the solid phase, comprising soil particles (Figure 2-2) and the liquid phase, which is water in between the pores of the soil particles, and air. In an analogy, soil is very much like concrete, with a matrix of water between pores and soil particles as aggregates that give the soil strength.

Figure 2-2: SEM Image of fine sand particles [1]

There are two states in which soils can exist, namely: saturated and unsaturated. Saturated soils refer to soils near or beneath the water table (Figure 2-3) thus the voids in between soil particles are completely filled with water. Unsaturated soil is of interest in this project because this kind of soil is most commonly encountered in the field.

Figure 2-3: Distribution of soils with respect to the water table

Unsaturated soils refer to soils that are situated above the water table (Figure 2-3) and in the voids between soil particles, air and water exist together. These kinds of soils are difficult to analyze because of the coexistence of solids, liquids and gas. Water in between the pores is under tension, like in a capillary tube. This tension is termed negative pore-water pressure. Atmospheric pressure is taken as the reference pressure of 0kPa. Pressure above atmospheric is termed “positive pressure” and pressure below atmospheric is termed “negative pressure”. The water in tension exerts a compressive force on the soil particles and holds the particles together and thus gives the soil shear strength.

In order to know the stress state of soil or other soil parameters, it is essential to measure the characteristics of fluid pressure between soil particles and this is the basis of soil suction measurements. This is termed “direct measurement” as the instrument will be measuring the negative pore-water pressure or matric suction in a soil directly. Total suction of soil is equal to the sum of the matric suction and the osmotic suction. Suction is measured in kiloPascals (kPa) and matric suction is the difference between pore-air and pore-water pressure in soil. Osmotic suction is dependent on the salt concentration in soil water with respect to pure water. Detailed theoretical explanations are presented in Section 3.1.

Soil moisture content is also another property of soil that can be measured besides pore-water pressure. As soil moisture is related to soil suction of a soil by its soil water characteristic curve (SWCC), once the amount of moisture in the soil (the water content) can be determined, the soil suction can be found via the SWCC. Soil suction found this way is termed “indirect measurement”.

The relative humidity of a particular soil is also indicative of soil suction. In thermodynamic terms, the free energy of soil water (total suction) is related to the partial pressure of the pore-water vapor [2] or relative humidity. Relative humidity measurements directly represent total suction of the soil, which indirectly can be used to find the matric suction. In this case, relative humidity measurement of soils is termed indirect measurement of matric suction.

In summary, most sensing techniques, electrical or optical, are centered on the measurements of:

1. Pore-water pressure in soil
2. Soil moisture content
3. Relative humidity of soil

From these measurable physical quantities, soil suction can either be measured directly or indirectly.

2.2 Traditional Methods for suction measurement

Many kinds of equipment and sensors have been developed to measure the different parameters of soil. As soil is a natural material that does not behave in a predictable way, it is a testament to the difficulties involved that many ingenious ideas have been put forward in developing soil measurement techniques. The standard method of measuring soil moisture content is the thermogravimetric method [3]. This involves oven drying of a known volume of soil and determining the weight loss. This method is time consuming and destructive to the sampled soil. However it is indispensable as a standard method for calibration and evaluation purposes. Among the widely used automated soil moisture measurement techniques are neutron scattering, gamma ray attenuation, soil electrical conductivity (including electrical conductivity probes, electrical resistance blocks and electromagnetic induction), tensiometry, hygrometry (including electrical resistance, capacitance, piezoelectric absorption, infra-red absorption and transmission, dimensionally varying element, dew point, and psychometric), and soil dielectric constant (including capacitance and time domain reflectometry) [3].

Table 2-1 illustrates some commonly used devices or methods that are used in the laboratory and field to measure suction of soils [2].

Table 2-1: Devices for measuring soil suction and its components [2]

Name of Device	Suction Component Measured	Range (kPa)	Working Principle	Advantages/Limitations
Psychrometer	Total	100-~8000	Uses thermocouples and measures temperature difference between dry and wet junctions which is related to relative humidity.	A constant temperature environment of $\pm 0.001^\circ\text{C}$ is needed. Long time needed for equalization.
Chilled Mirror Hygrometer	Total	Entire range	Measures relative humidity by using a chilled mirror to detect condensation of water vapor from the sample. The change in reflectance of the mirror is measured by an LED and a photodetector.	Device has good resolution and accuracy of 0.1%RH and $\pm 0.3\%$ RH respectively. Soil has to be sampled and placed into the device for measurement.
Filter Paper	Total	Entire range	May measure matric suction when in good contact with soil. Moisture content in the filter paper after equilibrium is indicative of the soil moisture content.	Indirect measurement of soil moisture content. Very long equilibration time, in term of weeks.
Tensiometers	Negative pore-water pressure or matric suction	0-90	Extracts water from soil through a high air-entry ceramic disk. Hydrostatic pressure deforms a diaphragm which produces an electrical signal	Difficulties with cavitation and air diffusion through ceramic cup. Working range is small, but very sensitive ($\pm 0.1\text{kPa}$).
Null-Type pressure plate (axis translation)	Matric	0-1500	Soil specimen sits on top of a high air-entry ceramic disk in an air pressure chamber. Air pressure is increased till water pressure is nearly zero. The difference in pressure is the matric suction.	Range of measurement is a function of the air-entry value of ceramic disk. Laboratory based.
Thermal Conductivity Sensor	Matric	0-~400+	Thermal conductivity of a soil increases with an increasing water content. Sensor includes a heater and temperature sensor for measurements.	Indirect measurement of matric suction using a variable pore size ceramic sensor
Mini Suction Probe	Matric	0-1500	A miniaturized tensiometer, where the water chamber is very small to prevent water cavitation and the high air-entry ceramic disk is thin for rapid equalization.	Range of measurement is a function of the air-entry value of ceramic disk. Sensitivity $\pm 2\text{kPa}$.
Time Domain Reflectometry	Matric	0-500	Uses an electro-magnetic (EM) pulse to measure the dielectric constant of soil. Dielectric constant varies with water content of the soil.	Used in the field.
Pore Fluid Squeezer	Osmotic	Entire Range	A heavy-walled cylinder and piston squeezer which removes the pore-water from a soil for electrical conductivity measurements.	Used in conjunction with a psychrometer or electrical conductivity measurement

2.2.1 Psychrometer

Thermocouple psychrometers can be used to measure the total suction of a soil by measuring the relative humidity in the air within the soil or in a region near the soil. Relative humidity is related to the total suction of a soil in a thermodynamic relationship. They operate on the basis of measurements of temperature difference between a non-evaporating surface (i.e. dry bulb) and an evaporating surface (i.e. wet bulb). The difference in the temperatures between these surfaces is related to the relative humidity. One of the most commonly used thermocouple psychrometers is the Peltier type (Figure 2-4), where evaporation is induced by passing a Peltier current through the evaporating junction. Relative humidity is measured after equilibrium is attained between the air near the thermocouple and the pore-air in the soil specimen. The thermocouple junction is cooled by passing a Peltier current through it and condensation forms on the junction. The current is then switched off and the condensation evaporates back into the air. The temperature reduction is a function of the evaporation rate, which is in turn affected by the water vapour (humidity) in the air. The temperature reduction can be detected by measuring the voltage change of the thermocouple wires.

Figure 2-4: Single-junction Peltier psychrometer [2]

The response time of a psychrometer depends on its protective cover and the magnitude of total suction being measured. The response time varies from a few

hours at several thousands kPa suction (above 90%RH) to about 2 weeks at 100 kPa suction (close to 100%RH). It appears that the psychrometer requires a considerably long time for equalization when used to measure low suction values. A controlled temperature environment for the psychrometer ($\pm 0.001^\circ\text{C}$) is absolutely necessary to measure total suctions to an accuracy of $\pm 10\text{kPa}$. A slight lowering of the temperature at such high humidities will immediately produce condensation on the thermocouple, hence producing errant responses. Deployment of the psychrometer in the field is generally avoided as there are significant temperature fluctuations in the field. Corrosion of the metal wires of the thermocouples, caused by the acidic environment of the soil, also causes the psychrometer to respond in an erratic way [2].

Hence, as an electrical sensor for measuring RH of soils, the psychrometer is plagued by several problems, namely long response time, requirement for very stringent temperature control and non-corrosion resistance.

2.2.2 Chilled Mirror Hygrometer

The device uses the dew-point technique and is a chilled mirror hygrometer. The soil sample fills about half the capacity of a small container and is placed in the device in a sealed chamber. The schematic is shown in Figure 2-5 [4]. Relative humidity is measured after equilibrium is established between the air near the mirror and the pore-air in the soil specimen. The chamber contains a mirror whose temperature is precisely controlled by a thermoelectric cooler. There is a different amount of moisture condensing on the mirror at different humidity levels of the air in the chamber. Detection of the exact point at which condensation first appears on the mirror is by a beam of light directed onto the mirror and reflected onto a photodetector. The photodetector detects the change in reflected light when condensation occurs on the mirror, and the temperature at which condensation occurs is recorded by the thermocouple attached to the mirror. The device is equipped with an internal fan that circulates the air within the sample chamber to reduce equilibrium time. The device also has a temperature controller to set the temperature of the specimen at which relative humidity measurement is to be made.

The device has a humidity resolution and accuracy of 0.1%RH and $\pm 0.3\%$ RH, respectively. The humidity accuracy corresponds to a suction accuracy of ± 400 kPa.

Figure 2-5: Schematic of a chilled-mirror dew point device [4]

The device cannot be used in the field as soil has to be sampled and placed into the device before measurements can be taken. The psychrometer has a better accuracy, but the range for the chilled mirror hygrometer covers the whole suction range as its humidity sensing range is from 10%RH to 100%RH.

2.2.3 Filter Paper

It is possible to use the filter paper method to measure the total or matric suction of a soil. The filter paper is used as a sensor and the filter paper method is classified as an “indirect method” of measuring soil suction. The principle of measurement for filter paper is based on the assumption that a filter paper will come to equilibrium (with respect to moisture flow) with a soil having a specific suction. Equilibrium can be reached by either liquid water or water vapor exchange between the soil and filter paper (Figure 2-6). The filter paper is weighed frequently to check if the weight of the paper has become constant (i.e. water content in the filter paper has come into equilibrium with the moisture in the soil sample). When a dry filter paper is placed in direct contact with a soil specimen, it is assumed that water flows from the soil to the paper until equilibrium is achieved. When a dry filter paper is suspended above a soil specimen (non-contact), vapor flow of water will occur from the soil to the filter paper until equilibrium. After equilibrium is established, the water content of the filter paper is measured. When the paper is placed in contact

with soil, liquid flow of water occurs and the equilibrium water content of the paper corresponds to the matric suction of the soil. On the other hand, when the paper is not in contact with soil, the equilibrium water content of the paper corresponds to the total suction of the soil [2].

Figure 2-6: Contact and non-contact filter paper methods for measuring matric and total suction of soil [2]

The filter paper method can be used to measure soil suction with a sensing range in the entire range of suction measurement, from a few kPa to several hundred thousand kPa. The accuracy is not a well established fact, but can range up to several thousand kPa. The measurements are generally performed in the laboratory with soil specimens from the field. The factors affecting suction measurements using filter paper were presented in a paper by Leong et al. [5]. Equilibration time for filter papers typically take up to a few days and great care must be taken when measuring the water content of the filter paper because of the small weights involved. The balance should be accurate to about 0.0001g. It is also difficult to achieve good contact between the soil specimen and the filter paper, hence resulting in the paper measuring the total suction of the soil, instead of matric suction.

2.2.4 Tensiometer

A tensiometer (Figure 2-7) utilizes a high air-entry ceramic cup as an interface between the measuring system and the negative pore-water pressure in the soil. A high air-entry disc acts as a membrane between air and water and it is made of

ceramic. Once the disc is saturated with water, air cannot pass through the disk due to the ability of the surface tension developed by the menisci of water capillaries in the disc. The ceramic cup is connected to a pressure measuring device through a small bore tube. Equilibrium between the pore-water pressure in the soil sample and the water pressure in the ceramic cup is reached when the pressure transducer in the ceramic cup reports a constant reading. The tube is usually made from plastic due to its low heat conductivity and non-corrosive nature. The tube and the cup are filled with de-aired water. The ceramic cup is inserted into the soil until there is good contact. Tensiometers can be used in the laboratory or in the field [2].

Figure 2-7: Small-tip tensiometer [2]

Once equilibrium is achieved between the soil and the measuring system, the water in the tensiometer will have the same negative pressure as the pore-water in the soil. The pressure of water in the tensiometer is measured by an electrical diaphragm sensor. The pore-water pressure that can be measured in a tensiometer is limited to about negative 90 kPa due to the cavitation of water in the tensiometer. When cavitation occurs, the water in the tube is no longer under tension; the pressure reverts back to atmospheric pressure. Although the working range of the tensiometer is small, it is very sensitive in this range, having a resolution of 0.1 kPa and an accuracy of ± 0.1 kPa.

2.2.5 Null Type Pressure Plate (Axis Translation)

Figure 2-8: Null type pressure plate [2]

Measurements of negative pore-water pressure can be made using the axis-translation technique. The apparatus for doing this is shown in Figure 2-8. The difference between air pressure surrounding the soil and the measured negative water pressure at equilibrium is the matric suction of soil, $(u_a - u_w)$, where u_a is the pore-air pressure and u_w is the pore-water pressure. When the air pressure is atmospheric ($u_a = 0$ as atmospheric pressure is the gauge by which other suction pressures are measured), the matric suction value is numerically equal to the negative pore-water pressure. The axis-translation technique simply translates the origin of reference for the pore-water pressure from standard atmospheric conditions to the final air pressure in the chamber where the soil specimen is situated (i.e. axis-translation). As a result, the water pressure in the measuring system does not become highly negative and thus cavitation of water can be prevented. This is the advantage of axis translation over tensiometry.

Equilibrium is established between the pore-water in the soil sample and the water in a chamber separated from the soil by a ceramic disc. The air pressure in the null type pressure plate is increased to a point where there is no water exchange between the soil sample and the water compartment as measured by a pressure transducer

connected to the water compartment. The water pressure is maintained as close to zero as possible. The difference between the air pressure in the air pressure chamber and the measured negative water pressure at equilibrium is the matric suction of the soil. The sensing range for the null type pressure plate depends on the ceramic disc used, but it is about 0 to 1500kPa, with an accuracy of ± 10 kPa.

The disadvantage of this technique is that the equipment is too bulky to be used in the field, hence it can only be laboratory based [2].

2.2.6 Thermal Conductivity Sensor

Thermal properties of a porous medium have been found to be indicative of the water content of the porous medium. Water is a better thermal conductor than air and the thermal conductivity of a porous medium increases with increasing water contents. A thermal conductivity sensor consists of a porous ceramic block containing a temperature sensing element and a miniature heater. The thermal conductivity of the porous block varies with its water content. The water content of the porous block is dependent upon the matric suctions applied to the block by the surrounding soil at equilibrium. In other words, soil water can enter the porous ceramic block, and equilibrium is achieved when the block water content is in equilibrium with the pore-water pressure in the soil. As water content in the block increases, the thermal conductivity increases as well, hence when the thermal conductivity remains constant, equilibrium has been achieved between the porous block and the surrounding soil. Therefore, the thermal conductivity of the porous block can be calibrated with respect to applied matric suctions [6].

The sensing range of thermal conductivity sensors is from 0 to 400kPa with an accuracy of ± 15 kPa. The response time of the sensor is in the range of hours. Sensor failures have been attributed to moisture coming into contact with the electronics sealed in the porous ceramic. There are also problems with the porous blocks being too fragile. They can crack or crumble during calibration or installation [2].

2.2.7 Mini Suction Probe

The suction probe, modified from a pressure sensor (Druck PDCR81), consists of a pressure transducer with a high air-entry ceramic disk mounted at the tip of the transducer (Figure 2-9). The diaphragm of the pressure transducer responds to the pressure applied. A small gap between the ceramic disk and the diaphragm is filled with water to let the pore-water pressure in soil to come into equilibrium with the water pressure in the gap. Equilibrium between the pore-water pressure in the soil sample and the water pressure in the water compartment of the probe is reached when the pressure transducer in the compartment reports a constant reading.

Figure 2-9: Schematic diagram of a mini suction probe [7]

The mini suction probe is primarily used in the laboratory with soil specimens. Its response time is fast due to the thin high air-entry disk. The sensing range of mini suction probes is from 0 to 1500kPa (using other ceramic discs with higher air-entry values) with an accuracy of ± 2 kPa. When using normal ceramic discs, the small water compartment in the probe prevents cavitation from occurring at very negative pore-water pressures; hence it can measure up to 400 kPa matric suction in soils [7].

2.2.8 Time Domain Reflectometry

A typical time domain reflectometry (TDR) system consists of four main components: a step pulse generator, a coaxial cable, a waveguide and an oscilloscope. During operation, the waveguide is fully inserted into the soil. The step pulse generator produces high frequency electromagnetic (EM) pulses that are periodic square waves. The electric component of the EM pulse is influenced by the dielectric constant of the soil. The EM pulse travels from the pulse generator, through the coaxial cable and the waveguide, and is reflected back from the ends of the guide. The speed of the EM pulses is dependent on the dielectric constant of the soil in which the waveguide is embedded. Hence the time needed from the pulse emission to the detection of back reflection is indicative of the soil moisture condition [8].

Equilibration is not necessary here as the waveguide for the TDR system is inserted directly into the soil when taking measurements. The sensing element comes into direct contact with the soil, instead of having a need for a water compartment for equilibrating pressure with soil water. The sensing range for the time domain reflectometry techniques is from 0 to 500kPa, and with an accuracy of ± 1 kPa.

2.2.9 Pore Fluid Squeezer

The osmotic suction of a soil can be indirectly estimated by measuring the electrical conductivity of the pore-water from the soil. Pure water has a low electrical conductivity in comparison to pore-water that contains dissolved salts. The electrical conductivity of the pore-water from a soil can be used to indicate the total concentration of dissolved salts that is related to the osmotic suction of the soil. The pore-water in the soil can be extracted using a pore fluid squeezer which consists of a heavy walled cylinder and a piston squeezer. The electrical conductivity of the soil water is measured by just applying a few drops of soil water (squeezed from the soil) to an electrical conductivity meter. There's no equilibration needed. A calibration curve can be used to relate the electrical conductivity to the osmotic pressure of the soil [2].

2.3 Fiber Optic Methods

Devices that use light (photons) as information carriers, as opposed to electrons, are quite recent because the field of photonics is quite young by comparison to that of electronics. Using light to measure the same parameters that were measured by electronic devices could open a whole range of capabilities, possibilities and problems.

It was with Charles Kao's landmark paper [9], entitled "Dielectric fibre surface waveguides for optical frequencies", that unlocked the door to modern fiber optics technology. Originally, optical fibers were primarily used in telecommunication applications. It was not until much later that the potential for fiber optic sensing was realized because of the advantages that fiber optic sensors have over conventional electronic sensors [10]:

1. Immunity to electro-magnetic interference (EMI), which induces noise in electrical signals because the fiber sensor and its connecting lead are made of glass, a dielectric material.
2. Robustness - the tensile strength of glass is greater than that of steel, so high mechanical reliability is maintained if the fiber surface is protected.
3. Long-term measurement capability, some fiber sensors using wavelength modulation are very stable and even readings taken after a long time will be valid.
4. Sensors using wavelength modulation could be wavelength multiplexed using a single strand of fiber. Fiber sensors of the same kind or of different types can be joined together.
5. The small size of the sensors caused minimum disruption to the structure in which they are introduced.
6. Sensors have the ability to measure parameters in a hostile or hazardous environment, are highly resistant to corrosion and are waterproof.
7. The sensing point can be situated a long distance from the data collection point as signal degradation in an optical fiber is very small compared to electrical cables.

Most of the equipment used in soil applications today is supplied by Soil Moisture Equipment Corporation [11]. The equipment supplied by Soil Moisture is either electrical or mechanical in nature and no optical based measuring equipment is commercially available.

Most optical or fiber optic based techniques or equipment for soil measurements are newly discovered and prototypes are still under development or product testing. Some examples include: a fiber optic near-infrared (NIR) reflectance sensor for detection of organics in soil [12], a fiber optic sensor that measures soil water content to determine solute transport processes [13],[14],[15] and a fiber optic relative humidity sensor developed by Blue Road Research [16] for soil moisture measurements [17],[18].

Schneider et al. [12] reported the assembly and evaluation of a laboratory NIR reflectance system for remote, real time detection and characterization of organics in soils. This system includes a reflectance sensor, a field-rugged Fourier Transform Infrared (FTIR) spectrometer and low-loss silica fibers, all of which are commercially available components. Reflectance spectroscopy is well suited for such measurements because it can detect and identify both the host soils and organics, and does not require sample handling or preparation. This eliminates the need to draw the pollutants into a remote sensing chamber. The NIR spectral range used, between 1.4 μm and 2.2 μm , is convenient because of the availability of low-loss silica fibers, light sources and detectors. The primary problem involves noisy signals as the spectrometer's detector did not have high sensitivity and the light source in the spectrometer was weak. The probe needs miniaturization before it can be used in devices like cone penetrometers.

Ghodrati et al. [13],[14],[15] developed a fiber optic mini probe (FOMP) that measures soil moisture to determine solute transport processes in soil. The system consists of transmitting a constant beam of light through the input leg of a bifurcated fiber optic miniprobe to a location of interest within the soil matrix. At the probe's tip, the incoming light interacts with the soil matrix where it is partially

absorbed and partially reflected back into the probe. The reflected signal is transmitted through the output leg to a photodetector and quantified. The output signal, which is constant during steady state (i.e., dry soil), decreases as the water content in soil increases. A calibration is necessary to convert the output light intensity to water content values. When measuring solute transport process, the intensity of the output signal, which is constant under steady conditions, changes when a plume of fluorescent water tracer passes through the soil matrix in front of the probe, allowing for in situ measurement of a solute breakthrough curve (BTC) at the "point" of observation.

The FOMP system's major advantages are that it measures water content in very small volumes (15–20 mm³) with a high temporal resolution and is very sensitive to small changes in water content. In addition, the system can be multiplexed to give a spatial water content distribution. Insertion of probes is simple with minimal disturbance to the soil. The FOMP as a soil moisture measurement device has two limitations. The first is that the calibration requires an extremely accurate knowledge of the water content directly in front of the probe. Even in small soil volumes it is difficult to establish a known uniform water content at the measurement scale of the FOMP. The second limitation is the FOMP's apparent decrease in performance in coarse-textured porous media due to poor contact between the probe and the media. Additional studies using probes with larger surface areas may improve these factors.

Blue Road Research has used a polyimide coated relative humidity fiber Bragg grating (FBG) first presented by Kronenberg et al. [19] and developed it into a sensor system for soil moisture measurements. Bare silica fibers are not sensitive to humidity. Polyimide polymers, however, are hygroscopic and swell in aqueous media as the water molecules migrate into them. The swelling of the polyimide coating strains the fiber, which modifies the Bragg condition of the fiber Bragg grating written into the core of the fiber and thus the resultant wavelength shift of the detected optical signal is proportional to the humidity surrounding the fiber.

The key to developing fiber optic sensors for measuring soil suction is to understand the properties of soil as a medium. To link the physics of soil or the physics of soil water to the optical characteristics of the soil or soil water (e.g. refractive index) that change when soil suction changes, the fiber optic sensor can be developed such that it is capable of detecting such optical changes of the soil medium.

There are many papers about fiber optics used in various fields to solve specific problems and some of these papers could be useful in developing new research approaches to soil suction measurements using fiber optic sensors. The breakdown of ideas from relevant literature is presented in the next section.

2.3.1 Direct Measurements of Pore-Water Pressure in Soil

Having geotechnical instrumentation, that is 1) minimally intrusive 2) robust 3) easy to use and 4) accurate, that can measure the conditions of pore-water within the soil is very desirable. The pressure that the water exerts on soil particles, if it is able to be determined, directly relates to soil conditions that are important for a geotechnical engineer to know. On the other hand, direct measurements of pore-water pressure can be very difficult due to the need for water to flow between soil and the water chamber of a suction probe through a high air-entry ceramic disc. Just having a diaphragm without a water chamber in the soil is not practical as soil particles can exert pressure onto the diaphragm, giving erroneous results. Tensiometers, axis-translation apparatus and mini suction probes are the primary instruments to measure pore-water pressure directly (Section 2.2.4, 2.2.5 and 2.2.7).

Some ideas for measuring negative pore-water pressure directly are presented next using various optical techniques from different fields.

2.3.1.1 3D shape measurements

Optical techniques that are used to measure 3-dimensional (3D) shapes using non-contact methods have been reported in the literature [20]-[25]. Zhao et al. [20]-[23] reported a novel fiber optic sensor based on beam reflection with a resolution of $0.1\mu\text{m}$ that can map complex surfaces in 3D. Using straight-line scanning of the probe over the sample, a 3D map of the surface can be reconstructed. Another method using Fourier transform profilometry (FTP) was reported by Zhang et al. [24], who were able to reconstruct a vortex shape from water stirred in a beaker using a magnetic stirrer. Using another technique for non-contact measurement, Pakh et al. [25] used the “focus/defocus” method to map 3D micro patterns in semiconductor wafers to a resolution of $0.2\mu\text{m}$.

How are 3D shape measurements related to soil suction measurements? The spaces between soil particles are called pores and these pores act as capillary tubes that soil water fills. As pore sizes from soil to soil varies due to the size of the soil particles, the meniscus of the columns of soil water will vary as well. The radius of curvature of the meniscus in the soil pores is inversely proportional to the matric suction of the soil. In other words, the smaller the pore radius is, the higher the matric suction of the soil will be.

2.3.1.2 Pore-water pressure measurements using fiber Bragg grating sensors

Fiber Bragg gratings (FBG) are a structure of periodic variations of refractive index in the core of an optical fiber. They are usually used in fiber optics communications applications for applications like add-drop multiplexing or tunable filters [26]. In fiber optic sensing using FBGs, carbon fiber composite embedded FBGs can be used for pressure, strain and temperature measurements [27]-[28]. Since the carbon fiber composite materials can be shaped readily during fabrication, an FBG pressure sensor can be made specifically to measure the pore-water pressure in soil. An existing design for an electrical soil suction probe, the mini suction probe [7], can be converted into an optical counterpart where the electrical diaphragm can be replaced with a carbon fiber composite diaphragm with an embedded FBG.

2.3.1.3 Measurement of refractive index change of water due to hydrostatic pressure using long period gratings

Long period gratings (LPG) are also structures of periodic refractive index variation in the core of optical fibers like FBGs, but the periods of LPGs are in the hundreds of μm range whereas the periods of FBGs are in the sub-micron range. This difference in periods alone enables the characteristics of FBGs and LPGs to be entirely different and hence LPGs can be exploited for their unique characteristics in sensing applications.

LPGs are sensitive to refractive index change of the external medium surrounding the optical fiber. Falciai et al. [29] reported the use of LPGs as solution concentration sensors, where the solution changes refractive index as the concentration of dissolved solute changes and the LPG is able to pick up this change. The refractive index of water changes when hydrostatic pressure is applied. Hence it may be possible to measure the degree of pressure or tension a particular body of water has using an LPG which can measure the change of refractive index as the pressure in water changes. This is applicable to pore-water also. As unsaturated soils have negative pore-water pressures, the LPG could be used to measure the pressures directly and find out the suction of the soil.

2.3.2 Indirect Measurements of Matric Suction using Relative Humidity of Soil

Relative humidity (RH) is a measure of the amount of water vapor in the air compared to how much it can possibly hold at a given temperature. Humidity sensing is important in various areas like industrial process control in semiconductor facilities, in pharmaceutical facilities, in buildings for human comfort, or in geotechnical and agricultural measurements. In industrial process control like semiconductor or pharmaceutical manufacturing, precise control of humidity conditions is necessary for reliable manufactured products. In buildings where people reside or work, humidity levels above 70%RH represent an uncomfortable environment; hence it is important to monitor humidity levels in buildings to keep the occupants comfortable. In geotechnical and agricultural measurements, relative humidity sensing plays a very important role in determining the stress state and moisture content in soil. From a thermodynamic point of view, the humidity of a soil is represented by the partial pressure of the pore-water vapour. In other words, the pore-water pressure of a soil is directly related to its humidity by thermodynamic relationships. The suction in soil (pore-water pressure) determines the shear strength, volume change and hydraulic behaviour of the soil. Typical values of humidity in soils range from 90%RH to 100%RH, but majority of soils have humidity levels of 99%RH and above. It is desirable to have an instrument that can measure small changes of humidity (0.1%RH or less) in the range of 90%RH to 100%RH, and with additional properties of the sensor being simple, reliable, repeatable, easy to install and demodulate.

There are four main issues with measuring RH, namely: The requirement for wide dynamic range, the temperature dependency of RH, the difficulty of making measurements in gaseous environments and limitations of sensors [30]. Firstly, RH is more difficult to measure than most water-related or atmospheric properties, such as temperature, pressure, flow, volume, mass or level. The complexity begins with the broad range of moisture conditions RH sensors must operate in. The difference in moisture content in the air from 10%RH to 100%RH over a temperature scale of 10°C to 40°C can be extremely large, and developing a sensor which can function

over such range is very challenging. Secondly, RH is also a temperature dependent variable. Its value can change significantly with slight changes in temperature. Thirdly, air is a poor thermal conductor and the temperature at any given point can be affected by thermal currents and temperature gradients. Air does not provide a stable temperature environment where RH readings can be taken and this in turn affects the measurement of humidity which can be difficult and time-consuming. Lastly, most RH sensors are non-linear devices with temperature dependencies. Most hygrometric sensors work by changing their electrical properties with variations in humidity and temperature. Calibration of such devices becomes more complex due to the requirement for accurate calibration equipment as well as time-consuming calibration cycles to map the behaviour of the device with RH [30].

Relative humidity sensors include electrical types using physical quantities that vary with humidity like electrical resistance, capacitance, thermal conductivity or wet-dry bulb methods. Electrical humidity sensors are prone to electromagnetic interference (EMI) that disrupts the readings. The long-term stability of the sensors is also in question due to the lifetime of the sensor that is susceptible to corrosion in high humidity environments. Remote sensing using electrical sensors is problematic too, as long distance monitoring requires a large amount of cables, which can be very heavy and expensive. More importantly, most humidity sensors have difficulty measuring humidity near 100%RH due to condensation of water from the atmosphere onto the sensing region. This can cause electrical sensors to short circuit or produce readings that are not repeatable or are erratic.

Researchers are looking actively into the use of optical fibers for RH sensing to overcome the limitations of electrical sensors. The advantages of using fiber optic RH sensors include: immunity to EMI, corrosion free, long lifetime, ability to remote sense due to lightweight and low loss fiber and minimal condensation problems at very high humidity. Relative humidity in soil is easier to measure with regards to instrumentation than pore-water pressure as there is no need for water to flow between soil and the water chamber in the measuring device. With humidity, ideally a simple RH probe can be inserted into soil to perform measurements at any

depth. In this case the inaccuracies inherent in indirect measurements can be offset by the ease of detection (sensor design).

Many approaches for RH sensing using fiber optical techniques have been reported and nearly all of which depend on the measurement of some bulk material property, based on materials whose optical absorption changes on exposure, for example cobalt chloride [31]-[33] or Nafion crystals (dye) [34]-[36]. Other material properties would include refractive index change which causes evanescent wave scattering like sol-gel [37], gelatin [38] or agarose gel [39], or hydroscopic gels (hydrogels) which swell when in the presence of water [40]-[42]. Most fibers used for bulk material coating are usually plastic clad silica fibers (PCS) as the core diameter can range from 200 μ m to 2mm. The plastic cladding is easily removed, thus allowing the high intensity core mode to interact with the surrounding humidity sensitive coating. Other types of fibers are also used for RH sensing, like POFs for their large core size and low cost [43]-[44], SMF with FBGs [19] or LPGs [46].

Humidity sensing using bulk materials depends on the reproducibility of the properties of the sensing material, and requires time for equilibrium to be established between atmospheric water vapour and water in the sensing material. They are also prone to failure in extreme environmental conditions such as high temperatures.

There are other methods developed that do not use bulk material coating on an optical fiber to realize a humidity sensor. Commonly known as tunable diode laser absorption spectroscopy (TDLAS) or absorption spectroscopy in short, this method overcomes existing problems with humidity sensitive coatings [53]-[56]. Though this technique is very sensitive to humidity changes, the equipment used is bulky, complex and costly.

After so much literature on the existing optical methods for measuring RH, some possible approaches to measuring relative humidity in soil for this project are discussed next.

2.3.2.1 Measurement of relative humidity using cleaved single mode fibers

Developing a fiber optic humidity sensor with a simple structure that can measure relative humidity accurately is ideal, as there are other methods like TDLAS which, though very accurate, comprises stringent wavelength modulation requirements and hence the system components are expensive and have a complex structure. Complex systems usually cannot be used in the field as they have more sensitive parts that can break down easily.

A simple layout for a fiber optic humidity sensor would be to make use of the fiber tip to interact with the surrounding environment. It should be small and non-intrusive. Mitschke [47] and Arregui et al. [48] have demonstrated, by fabricating Fabry-Pérot cavities on the tips of optical fibers, that it is possible for the fiber to sense the humidity in the surrounding environment. The Fabry-Pérot cavities, on the other hand, require specialized equipment to fabricate, which limits the simplicity of the fabrication of the sensor.

In another approach, Chang et al. [49] used a simple, cleaved SMF to measure solute concentration and refractive index change in fluids. He proposed that the setup can measure refractive index changes of up to 2×10^{-5} . The sensing technique is based on Fresnel reflection from the tip of the cleaved fiber; hence the sensor is very simple. The system that is needed to demodulate the intensity signal needs to be more stable, to prevent errors from fiber bending or source fluctuations. Since Chang used the sensor to measure different salt concentrations in solutions, it may be possible to modify the setup to produce a simple yet effective humidity sensor, or in another investigation, to develop an osmotic suction sensor which can measure different salt concentrations in soil water.

2.3.2.2 Measurement of relative humidity using gold tip-coated single mode fibers

Electroplating techniques are commonly used in semiconductor manufacturing but when metals are plated onto silica, they can be used for other purposes. Thin films of silver and other metals are known to vary in conductivity with varying relative humidity, while the reflectivity from a metallic surface is strongly dependent upon the conductivity of the metal. Stuart et al. [50] showed that by electroplating the tip of fibers using silver, gold, nickel, copper or other metals, the reflectivity of the thin films changes when the sensor is exposed to different humidities by measuring the back-reflection of light from the tip of the fiber. This method can also be adapted for making a simple and accurate relative humidity sensor.

2.3.2.3 Relative humidity measurements using absorption spectroscopy

Gas spectroscopy is a selective and sensitive technique for evaluating the concentrations of gaseous species. Fiber optics enables gas spectroscopy to be performed remotely utilizing the near infrared and visible parts of the optical spectrum. Spectroscopy is a particularly suitable technique to monitor chemical processes and chemical content. All molecular species have a characteristic absorption and emission spectrum which can give reliable identification of a particular molecular combination and also furnish quantitative values of its concentration [52].

Spectroscopic measurements for solids, liquids and gases all rely on similar approaches, where the measurement of absorption or reflectance characteristics can give a unique signature which can be related to the chemical species under test in a sample.

Direct absorption spectroscopy using fiber optics has certain advantages over existing methods of gas detection, namely [52]:

- Immunity from the loss of sensitivity due to chemical poisoning or saturation which affects all reactive components in the chemical systems which in turn require regular maintenance and/or replacement.
- Intrinsic safety due to the lack of electrical connections between the monitoring point and the remote observation point.
- The ability to operate over wide geographical areas extending many kilometers. Electrical systems are inhibited by the need for power supplies and/or electromagnetic signal pick-up. This is very useful for measuring suction over a large area.
- Precise species identification from the spectroscopic signature. The electrochemically based systems and indicator dye systems all exhibit high levels of cross sensitivity, which means they will react to other chemical species besides the one they are supposed to measure.
- The ability to precisely address multiple gas species by changing the optical source or switching its wavelength to tune to different absorption lines.

Optical gas spectroscopy systems using remote fiber optic links are recently emerging into new applications. The fiber optic systems operate at wavelengths from 500nm to 2000nm, within the transmission window of optical fiber, and this limits the performance somewhat. Whilst many environmentally and chemically interesting gases do have absorption spectra in this wavelength range, for most of the species the absorption lines are relatively weak especially when compared to those in the mid-infra-red (the 3 μ m to 5 μ m band) where silica fibers are opaque. Hence many gases of interest for environmental and safety have much stronger absorption lines in the mid-infrared (3 to 5 μ m wavelength band) than in the near-infrared where optical fibers are transparent (0.5 μ m to 2 μ m wavelength region).

The absorption line strengths are typically 100 times higher in the mid-infrared as compared to the near-infrared and it does seem advantageous to work in the mid-infrared regions. However, there are several difficulties associated with working in mid-infrared regions [52]:

- Mid-IR detectors need cooling systems to improve the signal to noise ratio because of the lower photon energy. Shot noise limited detection is much more difficult to realize in the mid-infrared without detector cooling. For comparable thermal noise performance, a mid-IR detector is maintained at roughly one third the absolute temperature of a near-IR detector.
- Optical source output powers in the near-IR exceed those in the mid-IR by a factor of about 100.
- Even the best mid-infrared optical fibers have very poor transmittance compared to that of silica fiber in the near-infrared. Consequently, the accessible range for fiber systems in the mid-IR is measured in tens of meters rather than kilometers.
- There is an abundance of low cost and high performance near-IR system components as compared to mid-IR system components because of the telecommunications industry.

The many advantages that near-IR system components have over mid-IR components overshadow the fact that absorption line strengths in the mid-IR are much higher than the near-IR.

Edwards et al. [53] investigated different modulation techniques for trace humidity measurements of a few parts per billion (ppb) in the NIR range. Schirmer et al. [54] characterized a fiber-coupled tunable diode laser absorption spectrometer operating in the 1-100ppm humidity range with a fast response time in the NIR range. Zaatari et al. [55] investigated and developed a wavelength tunable diode laser in the NIR region which can be varied over a relatively wide spectral range by changing the current and temperature of the diode. Edwards et al.[56], in a further development, improved the sensitivity, time-response and reproducibility of the tunable diode laser absorption spectrometer for moisture measurements in the NIR region; in tests over the moisture range 15 parts in 10^9 by volume (parts per billion, or ppb_v) to 1ppm_v (parts per million), the spectrometer displayed a short term sensitivity of 1.6ppb_v for an averaging time of 50s and a reproducibility of 12ppb_v over a period of three months.

A fiber-coupled diode laser could be developed with a short cavity length for the absorption cell using fiber collimators but with enough sensitivity to measure small changes of humidity in soil.

2.3.2.4 Relative humidity measurements using plastic optical fiber sensors

Plastic optical fibers, being cheap and having large core diameters, are well suited for chemical sensing as the evanescent field of the fiber penetrates the thin cladding easily to interact with the chemical sensitive coating on the surface of the fiber. This brings about a large modulation of light which can be detected easily. SMFs cannot be used for chemical sensing unless the structure of the fiber is modified to expose the evanescent field, as the core is very small relative to the cladding. Plastic clad silica fibers (PCS) are commonly used for chemical sensing as the core size can reach 1mm in diameter and the plastic cladding can be easily removed such that appropriate coatings can be applied onto the fiber. PCS fibers, however, are not as cheap as POFs and are brittle. POF core sizes can reach 3mm in diameter, larger than commonly used PCS fibers.

Muto et al. [43], has demonstrated that it is indeed possible to fabricate POF humidity sensors. The challenge would be to extend the range of the sensor into the high humidities by changing the humidity sensitive overlay material as well as modifying the sensor design to improve sensitivity.

Russell et al. [31] used cobalt chloride (CoCl_2) as the colorimetric reagent to detect the presence of humidity that was held to the 600 μm diameter plastic clad silica (PCS) fiber by gelatin, the overlay material. Light at 680nm filtered by a monochromator was passed into the fiber and the humidity response of the sensor was reported to be from 60% to 80%RH. Hyszer et al. [32] used a light emitting diode (LED) at 690nm as a light source for a straight 200 μm PCS fiber coated with CoCl_2 and polyvinyl alcohol (PVA). The humidity sensing range was 60% to 100%RH. Bownass et al. [38] used gelatin as the humidity sensing film coated on the cladding of standard telecommunications single mode fibers (SMF). The

whispering gallery (WG) mode interacts with the film at the cladding-overlay interface and causes Fresnel reflection. As humidity changes, the gelatin layers swell and the refractive index decreases. The power of the back reflected light was measured and the humidity response of the sensor starts from 62%RH to the higher humidities. Otsuki et al. [45] found that bending fiber to a small radius improved the response of a sensor drastically, as did Jindal [33]. Jindal et al. used CoCl_2/PVA as sensing film for a curved 200 μm PCS fiber and obtained a sensing range of 3% to 90%RH. A He-Ne laser source at 632.8nm was used. Muto et al [43] used a straight plastic optical fiber (POF) coated with hydroxyethylcellulose (HEC) and polyvinylidenefluoride (PVDF) as the sensing material. The humidity sensing range of the sensor was 20% to 90%RH.

Making use of the advantages of large diameter plastic fiber, higher sensitivity due to fiber bending and low cost humidity sensitive reagents, a simple and low cost plastic optical fiber sensor based on cobalt chloride (CoCl_2) and gelatin coating on the curved sensing point can be achieved.

2.3.2.5 Relative humidity measurements using long period grating sensors

LPGs have very good sensitivity when it comes to external refractive index measurements. This is why LPGs have been used for various applications like measurements of solution concentration [29] and liquid level sensing [57]. Refractive index measurement is the basis behind these applications. Healy et al. [46], in a paper that is the first of its kind, demonstrated that by coating the LPG with a humidity sensitive coating that changes its refractive index when the surrounding humidity changes, the LPG was able to register the refractive index change of the coating by a shift in the shift of resonant wavelength of the cladding mode. Thus, an LPG RH sensor was developed.

To further improve on such a technique would require a different kind of coating to elicit a different response from the LPG. Research needs to be done into how the LPG and the coating can affect the measurement and how to optimize the sensor design.

2.3.3 Fiber Optic Techniques to be developed

The focus of the development of fiber optic sensors for the measurement of soil suction was centered on measurements of pore-water pressure and relative humidity of soil. The techniques that were researched and developed further are:

For direct measurements of pore-water pressure (matric suction) in soil:

1. 3D shape measurements of meniscus of water capillaries.
2. Pore-water pressure measurements using FBGs.
3. Measurement of refractive index change of water due to hydrostatic pressure change using LPGs.

For indirect measurements of matric suction using relative humidity of soil:

1. Measurement of relative humidity using Fresnel reflection from the fiber tip of a cleaved SMF.
2. Measurement of relative humidity using Fresnel reflection from gold-tip coated fibers.
3. Relative humidity measurements using absorption spectroscopy
4. Relative humidity sensing using POFs.
5. Relative humidity sensing using LPGs.

Chapter 3 Theory

3.1 Soil Suction Theory

Soil suction is a stress state variable that controls the behaviour of unsaturated soils. Soil suction is commonly referred to as the free energy state of soil water and it can be expressed in terms of the partial vapor pressure of soil water. The thermodynamic relationship between soil suction (free energy of soil water) and the partial pressure of the pore-water vapor can be written as [2]:

$$\Psi = -\frac{RT}{\nu_{w0}\omega_v} \ln\left(\frac{\overline{u_v}}{\overline{u_{v0}}}\right) \quad (1)$$

Ψ - soil suction or total suction (kPa).

R - Universal gas constant (8.31432J/molK).

T - absolute temperature (K).

ν_{w0} - specific volume of water or inverse of density of water(1/998 kg/m³).

ω_v - molecular mass of water vapour(18.016kg/kmol).

The term $\frac{\overline{u_v}}{\overline{u_{v0}}}$ refers to relative humidity or RH (%) and it is defined as the ratio of the water vapour pressure $\overline{u_v}$ or water vapour content to the saturation vapor pressure $\overline{u_{v0}}$ or the maximum water vapour content at the temperature of the air or gas. Dalton's Law says that the total pressure of a gas is the sum of the pressures of each component. The pressure of a single component is called partial pressure, so the total pressure of air is the sum of the partial pressures of its components.

At 25°C the constants in Equation 1 have a value of 137325 kPa, and this reduces the equation to:

$$\Psi = -137325 \ln\left(\frac{\overline{u_v}}{\overline{u_{v0}}}\right) \quad (2)$$

Table 3-1 shows the relationship between soil suction and relative humidity using Equation 2. Most soils have a suction of 500 kPa and below and their corresponding humidity is above 99.5%RH, making them very wet indeed. In the case of very dry soils with suctions up to 10000 kPa, which are rare, the humidity of the soils still exceeds 92%RH.

Table 3-1: Soil suction values and the corresponding humidity of the soil.

Total Suction (kPa)	Corresponding %RH	Total Suction (kPa)	Corresponding %RH
50	99.96	1000	99.27
100	99.93	2000	98.55
200	99.85	3000	97.84
300	99.78	4000	97.13
400	99.71	5000	96.42
500	99.64	6000	95.72
600	99.56	7000	95.03
700	99.49	8000	94.34
800	99.42	9000	93.66
900	99.35	10000	92.98

Soil suction or total suction consists of two different components, whereby the components can be measured individually.

Components of soil suction [2]:

$$\Psi = (u_a - u_w) + \pi \quad (3)$$

where

Ψ - soil suction or total suction (kPa)

u_a - pore-air pressure (kPa)

u_w - pore-water pressure (kPa)

$(u_a - u_w)$ - matric suction or capillary component (kPa)

π - osmotic or solute component (kPa)

Matric suction component of a soil is measured in kPa and it is usually a negative value because it is defined as the difference between pore-air and pore-water pressure. Pore-air pressure in the field is usually atmospheric pressure and pore-water pressure in unsaturated soils is negative because of the capillary action of the water. The pressures are measured relative to atmospheric pressure hence if the pore-water pressure falls below atmospheric pressure, the pressure is termed negative.

The osmotic suction (due to salt content) of a given soil remains relatively constant even when the water content of the soil changes [2]. Environmental changes and changes in applied loads produce a change in the water content of the soil. The initial water content of compacted soils appears to have a direct relationship with the matric suction component (for example, see Figure 3-1). This means that an approximation can be made using the total suction of the soil. If total suction can be found, the matric suction of the soil can be calculated using the approximation that the osmotic suction remains constant. Therefore, total suction measurements are of importance, particularly in the high suction ranges where the matric suction measurements are hard to obtain [2].

Figure 3-1: Soil water characteristic curve for glacial till [2]

The capillary phenomenon is associated with the matric suction component of total suction. The height of the water column and the radius of curvature of the meniscus have direct relationships with the water content and matric suction in soils. In the case of a soil having a capillary zone, the meniscus results in an increased compression of the soil structure and this increases the shear strength of the soil (Figure 3-2).

Figure 3-2: Meniscus of water in a capillary tube results in a compressive stress on surrounding structures [2]

Figure 3-3: Physical model (a) and phenomenon (b) related to capillarity [2]

Consider a small glass tube that is inserted into water under atmospheric conditions (See Figure 3-3a). The water rises up in the tube as a result of the surface tension in the contractile skin (air-water interface) and the tendency of water to wet the surface of the glass tube (i.e. hygroscopic properties). This capillary behaviour can be analyzed by considering the surface tension, T_s , acting around the circumference of the meniscus. The surface tension, T_s , acts at an angle, α , from the vertical. The angle is known as the contact angle, and its magnitude depends on the adhesion between the molecules in the contractile skin and the material comprising the tube (i.e. glass). The vertical force equilibrium of the capillary water in the tube as shown in Figure 3-3 can be considered as follows: The vertical resultant of the surface tension (i.e. $2\pi r T_s \cos \alpha$) is responsible for holding the weight of the water column, which has a height of h_c (i.e. $\pi r^2 h_c \rho_w g$) [2]:

$$2\pi r T_s \cos \alpha = \pi r^2 h_c \rho_w g \quad (4)$$

Where

r = radius of the capillary tube

T_s = surface tension of water, $T_s=72.75\text{mN/m}$ at 20°C

α = contact angle

h_c = capillary height

g = gravitational acceleration

Equation 4 can be rearranged to give the maximum height of water in the capillary tube, h_c [2]:

$$h_c = \frac{2T_s}{\rho_w g R_s} \quad (5)$$

Where R_s = radius of curvature of the meniscus (i.e. $r/\cos \alpha$)

The contact angle between the contractile skin for pure water and clean glass is zero (i.e. $\alpha=0$). When the α angle is zero, the radius of curvature, R_s , is equal to the radius of the tube (Figure 3-3). Therefore the capillary height of pure water in a clean glass is [2]:

$$h_c = \frac{2T_s}{\rho_w g r} \quad (6)$$

The radius of the tube is analogous to the pore radius in soil. Equation 6 shows that the smaller the pore radius in the soil, the higher the capillary height will be. As a result of higher capillary height, the water pressure becomes more negative (Figure 3-3b). In other words, the smaller the pore radius in the soil, the higher the pore-water pressure in the soil. The capillary height can be plotted against the pore radius using Equation 6, where the contact angle is assumed to be zero, as shown in Figure 3-4 [2].

Figure 3-4: Relationship among pore radius, matric suction and capillary height of different soils [2]

Figure 3-5: Relationship between size of soil particles and the capillary height [2]

As the soil particles increase in size, the pore radius in between soil particles will increase, limiting the capillary height in the pores and thus reducing the matric suction (Figure 3-4). Strictly speaking, as soil particles become coarser, the matric suction value becomes smaller due to the limited capillary effect (Figure 3-5). In unsaturated soils, the pores of the soil particles give rise to a capillary effect complete with a meniscus that reflects the magnitude of matric suction as defined by the equation shown below [2].

$$(u_a - u_w) = \frac{2T_s}{R_s} \quad (7)$$

3.2 FBG Theory

An optical fiber is a thin, transparent, flexible strand of silica that consists of a core surrounded by cladding. Figure 3-6 shows this structure. The typical dimension for the core of a single mode fiber (SMF) is 9μm (in diameter) and the cladding is 125μm in diameter. The core and cladding of the fiber is made of the same glass (SiO₂) with the core being of slightly higher refractive index ($n_1 \approx 1.48$) than the cladding ($n_2 \approx 1.46$). The difference in refractive indices can be achieved by doping silica with different dopants like boron (to reduce index) or germanium (to increase index). A third layer, called the buffer coating, is applied over the cladding to protect the core-cladding structure (Figure 3-6). It is made from a different coating from the core and the cladding. It not only protects the glass fiber but also make it easier to handle by making its diameter larger [58].

Light is guided within the core of the fiber by total internal reflection (Figure 3-7). For total internal reflection to occur at the core-cladding boundary, the core refractive index (n_1) must be greater than that of the cladding (n_2). Under this condition, light can travel inside the core not only along the longitudinal axis but also at various angles to the axis without leaving the core, even if the fiber is bent [58].

Figure 3-6: Architecture of an optical fiber consisting of a high index core, lower index cladding and a protective buffer

Figure 3-7: How light propagates by total internal reflection (TIR) in an optical fiber

(Figures 3-6 and 3-7 source: www.howstuffworks.com)

A fiber Bragg grating (FBG) is a periodic perturbation of the refractive index along the fiber length that is formed by exposure of the fiber core to an intense optical interference pattern [59]. Usually the grating is written onto a section of photosensitive or hydrogen loaded SMF fiber using ultra-violet (UV) lasers that produces an interference pattern when the laser beam travels through a quartz phase mask [60].

The FBG reflects a wavelength called the Bragg wavelength that corresponds to the pitch of the grating. This relationship is shown in the equation below [59]:

$$\lambda_b = 2\Lambda n_{eff} \quad (8)$$

where λ_b is the Bragg wavelength, Λ is the pitch of the grating and n_{eff} is the effective refractive index of the fiber. An illustration of the transmitted and reflected spectra in an optical fiber with a FBG is shown in Figure 3-8.

Figure 3-8: Reflection and Transmission Spectrum of an FBG. The reflected wavelength is termed the Bragg wavelength [61]

The pitch of the FBG, and thus the back-reflected Bragg wavelength, is very sensitive to external perturbations like strain or pressure. This makes the FBG an excellent sensor in which parameters like strain can be calibrated against wavelength shift. Further developments of the FBG sensor from writing the FBG to embed it into suitable packaging materials are discussed in Section 4.3.

3.3 Fresnel Reflection at Fiber End

Fresnel reflections occurs when a beam of light transits from one medium to another medium of a different refractive index. At the interface of the two media, there will be some light reflected due to the difference of refractive indices of the two media. Fresnel's equations govern this relationship of light reflection or transmittance at the interface of two mediums.

When the electric field component of the EM wave, $\vec{E}$ is parallel to the plane of incidence at the interface between two dielectric media, the amplitude reflection coefficient is [63]:

$$r_{//} = \frac{n_t \cos \theta_i - n_i \cos \theta_t}{n_i \cos \theta_t + n_t \cos \theta_i} \quad (9)$$

where n_t is the refractive index of the medium for the transmitted wave, n_i is the refractive index of the medium for the incident wave, θ_i is the angle of incidence and θ_t is the angle of transmittance.

When $\theta_i = \theta_t = 0^\circ$, the fraction of the total power which is reflected at the interface is represented by the reflectance:

$$R = r_{//}^2 = \left(\frac{n_t - n_i}{n_t + n_i} \right)^2 \quad (10)$$

Assuming that light within the optical fiber encounters the air-glass interface at 0° incidence angle, Equation 10 can be used to find out n_t (the refractive index of the medium surrounding the fiber tip) with n_i fixed and the reflected power measured to form a ratio with the incident power to calculate the reflectance, R . In other words, the reflectance is a number that indicates what percentage of light is reflected from an interface, in this case, an air-glass interface.

Fresnel reflection provides a good mechanism whereby a change in the external refractive index (of air) caused by different relative humidity values results in different intensities of propagating light to be reflected back into the fiber at the air/glass interface of the fiber tip. Figure 3-9 illustrates Fresnel reflection in different external mediums.

Figure 3-9: Effect of Fresnel reflection on the intensity of light within the fiber with different external mediums and refractive indices

Fresnel reflection from the fiber tip varies with different refractive indices of the external environment; hence different intensities of light are coupled out of the fiber. Generally, the higher the refractive index of the surroundings, more light will be coupled out of the fiber and less light will be reflected back. This is valid for a range of $1 < n < 1.448$ for external media. The refractive index of the core of the fiber is constant at 1.448.

The relationship between the refractive index of air and its relative humidity is known, and it is described by a set of equations proposed by Edlén [64] and updated later by Birch and Downs [65]. Edlén's equations are a series of empirical equations giving the refractive index (n) as a function of the temperature, pressure, humidity level and wavelength based on the definition of standard air. The current definition of standard air uses the composition from [65] with the exception that CO_2 is 450ppm (0.05%) at 100kPa, 20°C and 50%RH (to match laboratory conditions).

The equations are [66]:

$$(n-1)_s \times 10^8 = 8343.05 + 2406294(130 - \sigma^2)^{-1} + 15999(38.9 - \sigma^2)^{-1} \quad (11)$$

where σ is the vacuum wavenumber of light (μm^{-1}) and the subscript s indicates that the values refer to standard air as defined above.

$$(n-1)_p = p \left(\frac{(n-1)_s}{96095.43} \right) \left(\frac{1 + 10^{-8}(0.601 - 0.00972t)p}{1 + 0.003661t} \right) \quad (12)$$

where t is temperature ($^{\circ}\text{C}$) and p the pressure (Pa).

Lastly,

$$n_{pf} - n_p = -f(3.7345 - 0.0401\sigma^2) \times 10^{-10} \quad (13)$$

where f is the partial pressure of water vapour (Pa).

Stone et al. [67] provides a detailed explanation on how to perform calculations with Edlén's equations.

3.4 Absorption Spectroscopy Theory

The various discrete energy levels associated with atoms and molecules means that, in general, absorption of electromagnetic radiation by a chemical species takes place at well defined wavelengths (λ) or frequencies (f) defined by:

$$E_2 - E_1 = \frac{hc}{\lambda} = hf \quad (14)$$

where $E_2 - E_1$ is the difference in energy levels of an electronic or molecular transition, hf is the photon energy required to induce the transition, h is Planck's constant and c is the free space velocity of light [68].

Since the energy levels associated with an atom or molecular are unique, the absorption spectrum serves as a "fingerprint" identification of the chemical. The actual wavelengths at which absorption takes place depend on the transitions involved. Electron transitions within atoms and molecules occur at short

wavelengths (high photon energy) corresponding to the UV-visible part of the spectrum. Transitions in vibrational or rotational states of molecules occur at energies corresponding to the near-IR and mid-IR band of the spectrum. Typically, a vibrational absorption line has a degree of fine structure superimposed on it corresponding to quantized rotational energy levels, so rather than being a single line, the absorption spectra consist of a series of closely spaced lines. Also, absorption can occur at frequencies that are harmonics or a combination of harmonics of the fundamental [68]. Some examples of near-IR lines for common gases are shown in Table 3-2.

Table 3-2: Near-IR absorption lines for some common gases [68]

Gas	Chemical Symbol	Wavelength(μm)
Oxygen	O ₂	0.761
Carbon dioxide	CO ₂	1.573
Carbon monoxide	CO	1.567
Water vapour	H ₂ O	1.365
Nitrogen dioxide	NO ₂	0.8
Methane	CH ₄	1.665
Acetylene	C ₂ H ₂	1.53
Ammonia	NH ₃	1.544
Hydrogen sulfide	H ₂ S	1.578

The chemical concentration may be related to the measured optical absorption through the Beer-Lambert law, which is given by:

$$I = I_0 \exp[-\alpha_m l C] \quad (15)$$

Where α_m is the molar absorption coefficient for light of intensity I_0 falling on a cell of length l in cm, and C is the gas pressure in Pascals or atmospheres.

The basic configuration of a tunable laser absorption setup is a tunable laser, connected to an absorption cell (where the gas whose concentration is to be

measured flows through the laser beam) and finally a detector to measure the change in intensity of the laser beam. In normal applications, the simplest way to determine the absorption line of a particular gas species is to use the direct absorption technique. The gas sample of interest is drawn into an absorption cell at a reduced pressure. Usually a multi-pass absorption cell based on the design of White [69] or Herriott [70] is used as the sampling cell. Using base paths of 0.3 to 1.5 meters, such cells result in total absorption pathlengths ranging between 10 and 200 meters. An absorption line scanned using the direct absorption technique produces a lineshape shown in Figure 3-10(a). This is achieved by either ramping the injection current of the laser or by ramping the temperature of the laser at frequencies of 10 to 100Hz. A photodiode detects the change in intensity of the laser source during scanning of the absorption line, thus a lineshape like in Figure 3-10(a) is obtained. The stronger the absorption line (a higher concentration of gas molecules), the deeper the lineshape will be [71].

For the “First Harmonic Lineshape” and “Second Harmonic Lineshape” shown in Figure 3-10(b) and (c) respectively, these lineshapes are obtained by detection of harmonics, formed by the vibrations of gas molecules, by wavelength modulating the output of the laser. In addition to direct absorption, tunable diode laser measurements are frequently carried out using the technique of harmonic detection. Most frequently, second harmonic detection is employed. In this mode, an external modulation waveform in the kilohertz frequency domain is simultaneously superimposed on the diode laser scanning current (a ramp). A lock-in amplifier is used to detect the second harmonic frequency $2f$, (twice the modulation frequency) with the modulation frequency f set as the reference. In most instances, the second harmonic frequency (detection at twice the modulation frequency) is chosen. First harmonic frequency is not commonly used [71]. Figure 3-10 illustrates the direct, first, and second harmonic absorption lineshapes.

Figure 3-10: Illustration of the (a) direct, (b) first, and (c) second harmonic absorption lineshapes [71]

As shown, second harmonic detection produces a zero baseline signal, thus eliminating the necessity of measuring small differences between two large intensities, I and I_0 , as is the case for direct absorption. Further advantages of second harmonic detection over direct absorption are [71]:

- (1) the elimination of a strongly sloping background often present in direct absorption;
- (2) reduced susceptibility to low frequency noise due to the kHz detection regime;
- (3) enhanced discrimination against signals that do not have a strong wavelength dependence such as the broad absorption tails of ambient H_2O vapor.

Employing second harmonic detection, minimum detectable absorbances ($\ln I_0/I$) of 10^{-5} to 10^{-6} are frequently obtained in TDLAS systems using total pathlengths around 100 m. This corresponds to minimum detectable concentrations ranging between a few parts-per-trillion (pptv) to parts-per-billion (ppbv), depending upon the absorption cross section [71].

Figure 3-11: A typical tunable diode laser system using wavelength modulation [55]

A typical tunable diode laser system using wavelength modulation is shown in Figure 3-11. The temperature of the diode laser is stabilized and its wavelength is tuned by a current ramp which can be generated by an AD/DA card from a computer or from a function generator. An additional frequency modulation current of f is injected. The laser beam interacts with the absorption line of the gas to be measured and is detected using a photodiode. The signal of the photodiode is fed to a lock-in amplifier, which can detect the $2f$ derivative embedded in the signal with f as the reference signal. The strength of the signal is determined by the amount of gas present.

3.5 Optical Fiber Evanescent Wave

When a light beam travels through the core of an optical fiber, even with total internal reflection, some portion of the incident field will penetrate the cladding. This portion is called the evanescent wave. The evanescent wave exists only under the condition of total internal reflection and the wave propagates in the same direction as the core wave.

The electric field of the evanescent wave decays exponentially in the cladding at a distance x from the core/cladding interface: [72]

$$E(x) = E_0 \exp\left(-\frac{x}{d_p}\right) \quad (16)$$

where E_0 is the electric field amplitude of light at the core-cladding interface and

$$d_p = \frac{\lambda}{2\pi\sqrt{n_1^2 \sin^2 \theta - n_2^2}} \quad (17)$$

Here λ is the operating wavelength, n_1 and n_2 are the refractive indices of the core and cladding respectively and θ is the angle of incidence at the core-cladding interface. The power flow in an optical fiber having an absorbing cladding of length L can be expressed as: [72]

$$P(C) = A[\exp(-\gamma_1 CL) + \exp(-\gamma_2 CL) + \exp(-\gamma_3 CL)] \quad (18)$$

Where A is a constant and γ_1 , γ_2 and γ_3 are three effective evanescent wave absorption coefficients which correspond to different modal groups having different penetration depths and C is the concentration of the absorbing species surrounding the core region.

When a chemical reagent is coated around the core of the fiber (assuming the cladding is removed), the evanescent wave of the fiber interacts with the film and change the intensity of the light propagating in the core through scattering, absorption etc. The change of the light intensity at the output of the sensing probe may be approximated using the following formula [32]:

$$I = I_0 \exp \frac{-\alpha r L C}{D} \quad (19)$$

where

I_0 – Input light intensity

α – absorption coefficient of coating

r – thickness of reagent layer

L – Length of sensing segment

C – reagent concentration

D – diameter of the uncladded optical fiber

In this project, CoCl_2 was used as the chemical reagent which interacts with water vapor in the air in a well-known colorimetric interaction to change its colour from deep blue (anhydrous state) to pink (hydrated state). The chemical formula of hydrated cobalt chloride is $\text{CoCl}_2 \cdot 6(\text{H}_2\text{O})$. The anhydrous form has high optical absorption between 550nm and 750nm with the peak at 690nm [31]. The hydrated form has little or no absorption. CoCl_2 forms salt crystals that adhere poorly to the polymethyl methacrylate (PMMA) ($n=1.489$ at $\lambda=680\text{nm}$ [43], where n is the refractive index of PMMA and λ represents the wavelength of light) core of the plastic optical fiber that was used. An overlay material that is optically clear and hydrophilic is needed, so that the fiber evanescent wave can interact with the sensing film and water vapor can diffuse into the film and interact with the CoCl_2 to change its absorbing properties. Gelatin is a suitable material and CoCl_2 dissolves readily in it to form a transparent film on the fiber. Dry gelatin has a refractive index of 1.5, found by ellipsometry [31], but when gelatin absorbs moisture it swells and its density decreases, leading to a reduction in refractive index of the film [38]. The refractive of the gelatin film ranges from 1.474 (dry gelatin) to 1.341 (liquid gelatin), the reason for this range is explained in the next section.

3.6 LPG Theory

Fiber gratings consist of a periodic perturbation in the optical fiber, generally of the refractive index of the core, and fall into two general classifications based upon the period of the grating. Short-period fiber gratings, or FBGs, have a sub-micron period and act to couple light from the forward propagating mode of the optical fiber to a backward, counterpropagating mode. Long Period Gratings (LPGs) have periods typically in the range of $100\mu\text{m}$ to 1mm . The LPG promotes coupling between the propagating core mode and co-propagating cladding modes. The high attenuation of the cladding modes results in the transmission spectrum of the fiber containing a series of attenuation bands centered at discrete wavelengths, each attenuation band corresponding to the coupling to a different cladding mode. Examples of the transmission spectra of LPGs are shown in Figure 3-12.

Figure 3-12: A typical transmission spectrum of a LPG [76]

The exact form of the spectrum and the center wavelengths of the attenuation bands are sensitive to the period of the LPG and the length of the LPG. Changes to the LPG, in terms of the environment, of temperature, strain, bend radius and the refractive index of the medium surrounding the fiber, causes changes to the period of the LPG together with the differential refractive index of the core and cladding modes. This then modifies the phase matching conditions for coupling to the cladding modes thus resulting in a change in the central wavelengths of the attenuation bands.

The fiber itself has two waveguide structures, one being the high-index core surrounded by the lower index cladding. The core of the optical fiber supports the fundamental forward propagating core mode. The other structure is the cladding surrounded by air, or other optically transparent materials used as sensor coatings. The cladding of the optical fiber supports the forward propagating discrete cladding modes. Phase matching between the mode propagating in the core of the fiber and a forward propagating cladding mode is achieved at the wavelength, λ , expressed by [73]:

$$\lambda = [n_{eff}^{co}(\lambda) - n_{eff}^{clad,i}(\lambda)]\Lambda \quad (20)$$

Where $n_{eff}^{co}(\lambda)$ is the effective refractive index of the propagating core mode at wavelength λ , $n_{eff}^{clad,i}(\lambda)$ is the effective refractive index of the i th cladding mode and Λ is the period of the LPG. Equation 20 leads to wavelength selective couplings to cladding modes. The transmission spectrum will then exhibit losses at discrete wavelengths that match the coupling from core to cladding mode.

Since the LPG spectrum is sensitive to change of index of refraction of the external medium, it is widely used as a sensor for chemical and protein concentration and refractive index measurement. Measurement is based on the degree of shift of the resonance wavelength with respect to change in external refractive index. However, the measurements are valid only if the index of refraction of the external medium (n_3) is less than that of silica or cladding (n_2), $n_3 < n_2$. When $n_3 > n_2$, the phase matching equation will not be satisfied. The absence of a guided region in the cladding will render total internal refraction (TIR) not possible for the cladding modes. Instead, the cladding modes will be rendered as leaky cladding modes.

There are two cases when the external medium exceeds the refractive index of the fiber cladding, that of an infinite and finite layer of external medium surrounding the cladding of the LPG. The effect of an infinite layer of external medium is discussed first. The absence of a guided regime in the cladding entails that the phase matching relation is no longer valid. Some energy will be reflected at the cladding-external interface and the ratio is determined by Fresnel coefficients. Hence the cladding modes are attenuated because of the refraction losses and are not guided cladding modes anymore. The only difference from the guided cladding modes is that the attenuated cladding modes are not sensitive to the external medium. In other words, the resonance wavelengths do not shift with the value of the external refractive index owing to the absence of modal confinement by the external medium [74]. The only change that occurs to the spectra of the cladding modes is that the coupling intensity of the resonant dip changes due to the reflection of the attenuated cladding modes at the cladding/external medium interface. Stegall et al. [75] have also experimentally shown this phenomenon (Figure 3-13). It can be observed as

the external medium refractive index increases beyond that of cladding, the resonance cladding mode dip changes with different external refractive indices without significant shift of the resonance wavelength.

Figure 3-13: Experimentally measured core mode transmission losses for several external refractive indices [75]

Finite layers of external medium surrounding the LPG have another effect, as shown by experimental and theoretical studies on thin-film coatings on LPGs which details that the resonance wavelength of the LPG is dependent on the thickness of the coating [77]. Rees et al. [77] demonstrated that as coating thickness was increased, but not exceeding 250nm, the resonance wavelength was displaced towards shorter wavelengths. Even though the refractive index of their coating material was 1.5, the average refractive index of the thin-film coating is less than that of the cladding. Hence there is an increase in $n_{eff}^{clad,i}$ (Equation 20) which results in a blue shift for the resonant wavelength λ . The cladding region loses its guiding ability when the thin-film thickness exceeds 250nm. No cladding modes were observed and this corresponds to a match between the average refractive index of the thin-film coating and that of the cladding.

In this project, gelatin was used to coat the LPG, to form a film around the cladding of the fiber as it is an optically clear medium which can change its refractive index when humidity in the air changes. Gelatin, with a dry refractive index 1.5, will swell and reduce its density upon absorbing water vapor. This will consequently lead to a drop in its index of refraction.

A brief background on gelatin will explain its various optical properties. Gelatin, primarily collagen, is a long fibrous protein that makes up the mechanically strong but elastic tissue of the body. The structure of the protein consists of a triple helix of repeating amino acid sequence and a single globular head portion that makes up a small part of the protein itself. The long repeating sequence is not soluble in water and tends to self-associate. However, once it is boiled, it dissociates into separate strands at the molecular level. When the gelatin, in powder form, is dissolved in hot water, it returns to its molecular strands that move about randomly in the solution. On cooling, if the concentration of the chains is high enough, they may entangle with each other. The most hydrophobic portions of these chains may stick to each other semi-permanently. This entanglement provides the molecular strength (elasticity) of the hydrogel. Hydrogels can be considered as a three-dimensional web of polymer chains with a highly irregular structure, which has the capability to fluctuate. The open and fluctuating structure leaves room for things to diffuse through the hydrogel within the water matrix. However once the water matrix is removed via evaporation after cooling, the whole structure is a collapsed 3D web. This is the case for the gelatin coating used on the LPG. Gelatin, like most protein, consists of atoms with higher atomic number than H_2O , so the index of refraction is higher than that of water. It ranges from 1.474 (dry gelatin) to 1.341 (liquid gelatin) [86].

Thus in conclusion, the change of index of refraction for gelatin is dependent on the amount of water molecules which has diffused through the 3D polymer chains. The refractive index decreases with an increase in relative humidity due to swelling and an increased water content in the gelatin.

Chapter 4 Research Development

4.1 Flowchart of Research Approaches

The flowchart describes the research approaches attempted and the order that they are described in this chapter. First, investigations and developments for direct measurements of pore-water pressure in soil are described, followed next by investigations into techniques for indirect measurements of matric suction using relative humidity of soil. The investigations include frequency modulated and intensity modulated sensing schemes. In-fiber gratings were also utilized for greater sensitivity and accuracy for the various sensors developed.

4.2 3D Shape Measurement of Meniscus

Capillary phenomenon is associated with the matric suction component of total suction. The height of the capillaries and the radii of curvature of the menisci throughout a particular soil have direct implications on the water content versus matric suction relationship as characterized by the soil-water characteristic curve. From Equation 7, matric suction can be calculated if the radius of the meniscus of water is known as the surface tension T_s is a fixed quantity. In other words, in order to calculate the matric suction of a given soil, one way is to know the curvature of the meniscus within the capillary channels of the soil particles.

Soil has to be imaged from top downwards. Hence a simple two dimensional (2D) image is not sufficient because the image will just indicate the shape of the soil particles and the meniscus, but not the depth. The depth is important because the meniscus can then be recreated and then its radius calculated. Three dimensional (3D) image capturing techniques have been explored and Figure 4-1 shows the different methods used for shape acquisition. Non-contact measurement techniques will be required, because the act of contact measurement itself will change the properties of the soil that is being measured.

Figure 4-1: Breakdown of shape acquisition methods [79]

Figure 4-2: Breakdown of Optical shape acquisition methods. [79]

Optical methods are the focus and Figure 4-2 shows the different methods used for optical shape measurement. Here the choice is to be made for resolution. A typical size of clay particles is about $1\mu\text{m}$ and the meniscus between clay particles will be slightly less than this. Clay particles have the finest size for soil particles, so the resolution needed to measure them is high. Interferometric methods are the most accurate, offering resolutions of up to minute fractions of a wavelength, depending on the resolution of the detector used, but approximately in the order of $10^{-6}\lambda$.

However, moiré or holographic interferometric setups are very bulky and they are expensive to setup, thus limiting their use in the field. The image processing algorithm needed to process the images is complicated; it needs to be able to convert a 2D fringe image like those captured by moiré interferometry into a 3D picture.

The limitations with regards to the soil involve the non-uniform scattering of menisci throughout the surface of the soil. Soil is a non-uniform, natural material and there will be menisci of different shapes and sizes scattered non-uniformly throughout the sample. A method to tabulate and average all the readings to give a mean suction value may not be accurate.

It is not practical to use meniscus shape measurement to determine the matric suction of soil because:

- Interferometric methods are needed to obtain the resolution to measure the shape of meniscus of water in fine soil, but they are expensive and bulky to set up. Furthermore, the setup cannot be used in the field.
- Image processing algorithms are complicated; they need to convert a 2D image to a 3D one.
- Soil is non-uniform and the distribution of menisci on the soil surface will be non-uniform. Finding and averaging them to find the matric suction will not be accurate.

Therefore, measuring the radius of the menisci of capillaries throughout the soil surface is not practical or reliable. Next, the sensor design and experimental setup is presented for direct measurement of pore-water pressure using FBG sensors.

4.3 Direct Measurement of Pore-Water Pressure Using FBG Sensors

The objective of this section is to develop a fiber optic sensor that is capable to measure pore-water pressure directly. The sensor consists of a diaphragm of carbon fiber composite material with an embedded FBG that measures the degree of bending with respect to the external pressure, and a sensor mount with an air chamber upon which the diaphragm can be seated and bent due to pressure differentials. The sensor is modeled after a similar electrical one developed for matric suction measurements by Meilani et al.[7]. See Section 2.2.6, Figure 2-9 for the schematic of the mini suction probe.

The development of the FBG sensor can be divided into 3 parts:

1. The diaphragm
2. The diaphragm mount
3. Testing and calibration setups

4.3.1 Design of the Sensor Diaphragm

FBGs are sensitive to strain and temperature changes; hence they are widely used as strain and temperature sensors. When the grating pitch or the effective refractive index of the FBG changes due to external parameters like strain or temperature, the Bragg wavelength, of the reflected FBG spectra, shifts (Equation 8).

Figure 4-3: The reflection spectrum of an FBG.

Figure 4-3 shows the reflection spectrum of an FBG and depending on the change in pitch of the grating, the Bragg wavelength can shift left (to shorter wavelengths) or to the right (to longer wavelengths). This makes the FBG an excellent sensor where the sensing information is encoded into wavelength shift and not intensity change. Once the physical quantities that are to be measured are calibrated with wavelength shift, the FBG becomes a sensor that is not affected by source intensity fluctuations as the measured quantity is not intensity. The readings obtained from measurement of wavelength shift of the reflected FBG spectra are very stable, free from any perturbations except the quantity to be measured. On the other hand, demodulating wavelength signals is more complex than the demodulation of intensity changes of the optical signal. This results in a complex demodulation system as compared to intensity demodulation systems.

The FBGs used in the experiments were fabricated using the commonly used phase mask technique. The first step in the fabrication of FBGs is to hydrogen load standard telecommunications single-mode fiber (SMF), Corning SMF-28, in a hydrogen loading chamber for 7 days at 1500psi and at a temperature of 70°C to photosensitize the fiber.

A tunable laser source (TLS), Ando AQ4321D, synchronized with an optical spectrum analyzer (OSA), Ando AQ6317, is connected to the hydrogen loaded fiber. The optical setup is shown in Figure 4-4. The fiber is exposed to UV light by scanning a laser beam from a frequency doubled Argon-Ion laser emitting UV light of 244nm wavelength through a phase mask. The quartz phase mask, from Ibsen, has a fixed grating period ($\approx 1074\text{nm}$) which creates an interference pattern on the fiber core by interfering the +1 and -1 order of the diffraction pattern. The fundamental order, or zeroth order, is suppressed by the mask.

The quartz phase mask is a vital element to create a periodic grating in the core of the photosensitive fiber, called a fiber Bragg grating. The phase mask is a diffractive planar fabricated optical element which splits the incident UV beam into diffraction orders according to [52]:

$$\sin\left(\frac{\theta_{mPM}}{2}\right) = \frac{m\lambda_{UV}}{\Lambda_{PM}} + \sin(\theta_i) \quad (21)$$

Where θ_i , θ_{mPM} and Λ_{PM} are the beam incident and diffracted angles with respect to the normal to the phase mask and the period of the phase mask pattern respectively. $m(0, \pm 1, \pm 2, \pm 3, \dots)$ is the diffracted order. $m(0)$ is the zero order or fundamental order or mode. For a given UV wavelength a phase mask may be designed to concentrate the split power into ± 1 orders, while minimizing the zero-order [52].

The interference pattern created by the phase mask inscribes a periodic structure of high and low refractive indices in the fiber core; this is the fiber Bragg grating. The resultant reflection spectra of an FBG fabricated using a phase mask of grating period of 1074nm has a center wavelength of about 1550nm. To prevent any further

changes in refractive index in the core of the fiber after the grating has been formed (i.e. to stabilize the FBG spectra) the optical fiber was annealed at 85°C for 6 hours.

Figure 4-4: FBG fabrication setup

Hao [61] used carbon fiber composite material (Hexcel Composites, Fibredux 913C-XAS) to sandwich the FBG to form a sensor package after curing. The advantages of embedding the FBG include:

1. Improved linearity of readings
2. Improved stability of readings
3. Very good repeatability
4. Wide force sensing range
5. Lower temperature conductivity

Carbon fiber composites are made of a series of carbon fibers all aligned in one direction with an epoxy matrix holding them together. Composite materials (laminates) are usually sold in a roll (see Figure 4-5) that consists of one long sheet of laminate. The sheet of laminate can be cut up into any shape and identical pieces of laminate can be stacked together to form a sensor package with a FBG sandwiched between the laminate layers. One single layer of laminate is termed single-ply. Many laminate layers can be sandwiched together to form multiple plies

with different carbon fiber orientations (cross-ply) or uni-directional orientation (parallel-ply), as shown in Figure 4-6.

Figure 4-5: Unidirectional reinforcement laminates [62]

Figure 4-6: Orientation of fibers in a laminate: The fiber directions can be arranged to meet specific mechanical performance requirements of the composite by varying the orientation [62]

Being lightweight and extremely strong are among the unique properties of carbon fiber composites. An optical fiber is embedded into carbon fiber composites to protect the FBG (the sensing region on the optical fiber), and also for lateral forces to be transferred to the FBG through the composite. The number of layers used and the position of the fiber in the layers determine the range and sensitivity of the FBG sensor. The naming convention for the FBG sensor with regards to the number of layers and orientation of the fibers are described subsequently.

Figure 4-7: FBG sandwiched between carbon fiber composite layers.

The layering configuration in Figure 4-7 is 0/0/FBG/0, each 0 indicating a layer and the degree of orientation of the carbon fiber. In this case, the carbon fiber orientation is parallel to the fiber; hence the degree of orientation is 0° . For example, configurations like 4-3, // -ply (parallel ply), 0/0/0/0/FBG/0/0/0, means that there are four layers of carbon composite above the FBG and 3 layers of carbon composite below it. The orientation of the carbon fibers are parallel to the fiber, hence // -ply. For configurations like 4-3, X-ply (cross ply), 90/0/90/0/FBG/0/90/0, every alternate layer is oriented 90° to the axis of the fiber. The immediate layers next to the fiber are always parallel to the fiber.

Figure 4-8: Effect of lateral force on different sides of the same sensor.

When a lateral force is applied on the double layer carbon composite side (Figure 4-8), the bottom layer experiences tension, hence the fiber is stretched and the grating pitch be elongates. As a result, the wavelength reflected will shift to the longer wavelengths. Conversely, if a lateral force is applied from the single layer

side, the FBG experiences a compression, hence the grating pitch is compressed and the wavelength reflected will shift to the shorter wavelengths. Embedding the FBG in different number of layers and at different positions within these layers will produce different effects for the overall sensor package.

The procedure for embedding an FBG in carbon fiber composite is as follows. The carbon fiber sheet has to be cut into pre-determined sizes which are then used to sandwich the FBG. Before curing the composite material is soft as the matrix binding the carbon fibers together has not hardened yet. The mold needed to cure the carbon fiber is made of metal plates, wrapped with Teflon sheets. One plate is wrapped with a porous Teflon sheet with a cotton sheet underneath it. The other plate is wrapped with just a non-porous Teflon sheet. The Teflon sheets allow the carbon fiber composite to be removed without much difficulty due to its non-stick nature.

The carbon fiber composites were then layered onto one metal plate akin to the way a sandwich is prepared. For example, if a 3-2 configuration is to be fabricated, first 3 layers of composite material are laid out on the metal plate, followed by the FBG, placed in the center and aligned parallel to the carbon fibers, and finally the last 2 layers are placed on top of the FBG to seal it in the package. The mold is secured tightly with screws such that the carbon fiber composite remains flat and does not change shape during curing. The entire mold is then placed into a vacuum oven, set at 120°C for 1.2 hours at 1.5°C/min ramp, for curing. After curing the matrix hardens and binds the carbon fibers very tightly together to form a very strong composite material.

4.3.2 Design of the Diaphragm Mount

A simple mount need to be fabricated to hold the diaphragm and provide an air tight chamber so that the diaphragm can bend according to the external pressure. As the length of the FBG was 5mm, the carbon fiber composite used to embed it was 2cm long; sufficient to cover the FBG and provide enough room for the diaphragm to

bend. The FBG sensor (diaphragm) covers the metal mount like a lid and is secured into place using epoxy.

Figure 4-9: Modeling of metal casing (for embedded sensor to mount upon) using Solidedge.
Blue: Carbon fiber composite. Black: Optical fiber. Yellow: Metal casing.

Figure 4-10: Embedded Sensor mounted onto a metal case.

The case was modeled using Solidedge (Ver. 8.0) software (Figure 4-9). The embedded sensor is then fixed to a metal case (Figure 4-10) using epoxy (Araldite Slow Setting epoxy). The purpose of the metal case is to provide a compartment filled with air at atmospheric pressure, a constant at lab conditions. If the external pressure is different from the pressure of air in the compartment, the carbon composite diaphragm will bend, leading to an applied strain on the FBG thus shifting the detected Bragg wavelength.

4.3.3 Testing and Calibration Setup

A series of carbon fiber diaphragms were made with different layering configurations to find out the sensitivity and range of the sensor. After fabrication, the diaphragms were first tested using a loading setup (Figure 4-11) which applies fixed loads to the diaphragm (Chatillon Force Gauge model TCD200), causing it to bend and the Bragg wavelength of the FBG to shift. The diaphragms were tested on both sides to simulate a positive bending force and a negative bending force. The testing results are shown in Section 5.1.

Figure 4-11: Chatillon Force Gauge setup for testing the FBG sensors

The diaphragms were then fixed onto the mounts and the package was then placed into a triaxial cell shown in Figure 4-12, whose interior pressure can be increased by applying fluid pressure. Positive pressure was applied using air pressure and negative pressure was applied using vacuum pressure. Negative pressure response is of interest because the sensor will be used to measure negative pore-water pressures, which are below atmospheric pressure.

Figure 4-12: Experimental setup with a fiber optic sensor in a triaxial cell (pressure cell)

Each test was performed in a triaxial cell and tested in the positive pressure region up to 600kPa and in the negative pressure region up to -90kPa . The reason for the limited negative pressure was because the vacuum pressure line could provide a maximum pressure of only -90kPa . After the pressure in the cell was varied, the subsequent wavelength shift of the FBG was recorded using an OSA (Anritsu MS9710B) using the experimental setup shown in Figure 4-13. The light source used was an SLD source. Calibration results are presented in Section 5.1.

Figure 4-13: Experimental Setup for triaxial cell testing

4.4 Measurement Of The Refractive Index Change Of Water With Hydrostatic Pressure Change Using LPGs

Due to the sensitivity of LPGs to external refractive index change, using them to measure refractive change of water when pressure is applied may be possible. In soil, pore-water is under different degrees of negative pressure, and if the LPG is able to pick up changes in hydrostatic pressure by measuring the refractive index change of the fluid, this will make the LPG a very good sensor as a fiber optic soil suction sensor.

The LPG used was a 450 μ m period grating and the 4th cladding mode was observed for a wavelength shift when hydrostatic pressure is applied on the fluid surrounding the LPG which will change the refractive index of the fluid. The LPG may be able to detect this refractive index change. A bare LPG with the buffer layer stripped away was placed into a triaxial cell (Figure 4-14). The light source used is a broad band source and the optical measurement instrument used is an Anritsu OSA (MS9710B). The triaxial cell was filled with water and hydrostatic pressure was applied via a fluid pressure line. Only positive pressure (pressure above atmospheric pressure which is 0kPa) is applied. The wavelength shift of the LPG was recorded.

Figure 4-14: Experimental Setup for detecting the refractive index change of water with hydrostatic pressure change using LPGs.

4.5 Measurement of Relative Humidity Using Fresnel Reflection From The Tip Of A Cleaved Single-Mode Fiber

By measuring the intensity of light reflected (Fresnel reflection) from the cleaved surface of an optical fiber, the refractive index of the external medium can be calculated using a simple formula. If a humidity sensor can be realized using this concept, the sensor can be very simple in design, which is an added bonus in soil measurements as complex sensors tend to break down easily thus limiting their use in the field.

Chang et al. [49] have developed a Fresnel ratio meter to measure solute concentrations and refractive index change in fluids. This project uses the same concept to attempt to measure relative humidity. In order to investigate the feasibility of using Fresnel's reflection in optical fiber to measure humidity, Edlén's Equations (Section 3.3) is used to calculate the refractive index change of air with respect to change in relative humidity.

Figure 4-15: Calculated Refractive Index of Air versus Relative humidity using Edlén's equations.

Using Edlén's equations (Section 3.3), the change in refractive index of air against relative humidity is shown in Figure 4-15. The gradient of the graph shows that the refractive index of air changes only about 1.18×10^{-8} for every 1%RH change, an extremely small change. The temperature of air was fixed at 25°C and the air pressure was fixed at 101325Pa. The laser wavelength used was 1.55μm. As the change in refractive index is small, a comparison is made by integrating Fresnel's formula, Equation 10, with the refractive indices obtained in Figure 4-15. A graph of reflected power in the fiber (Figure 4-16) was plotted against relative humidity, with optical power incident into the fiber being fixed at 0.6mW.

Figure 4-16: Simulated reflected power in fiber at air-glass interface (fiber tip) against relative humidity

Figure 4-16 shows that for every 1% change of RH, the reflected power will change by 10^{-12} W and for the full range of humidity from 0 to 100%, the total change in power will be 10^{-10} W. This change in optical power is too minute to be detected by conventional photo detectors. Therefore, it is not suitable to use the technique of Fresnel reflection in optical fibers to measure relative humidity in air as the resolution of standard photodetectors is not enough to pick up minute changes in the measured signal.

It may not be feasible to use Fresnel's reflection in an optical fiber to measure humidity changes in air, but it certainly can be used to measure salt concentration in a liquid, as the refractive index of the liquid changes when the salt concentration changes or when different salts are mixed together. Since Chang et al. [49] has shown that it is feasible to use Fresnel's reflection in fiber to detect minute changes in salt concentration; a pilot study was conducted for this project to show that a customized setup can be used to prove that:

- i. The fiber optic sensor cannot respond to changes of relative humidity in air.
- ii. More importantly, the fiber optic sensor can detect changes in salt concentrations using specially prepared salt solutions, thus showing that it can be used as an osmotic suction sensor for soil.

4.5.1 Development of the experimental setup for salt solution concentration measurements using Fresnel Reflection in a single-mode fiber

Figure 4-17: Experimental setup for measuring changes of reflected intensity from the tip of an optical fiber due to Fresnel reflection

The customized experimental setup is shown in Figure 4-17. The basic idea is to split the light signal and use one portion of it as a reference so that even if the signal fluctuates due to the source, taking a ratio of the signal and the reference will eliminate the fluctuation thus leaving the measurement signal from the sensor behind.

Light from the tunable laser source (Ando TLS AQ4321D) passes through a polarization controller (Thorlabs Fiber Polarization Controller FPC560) which polarizes the light in the fiber in one direction. This helps to reduce the birefringence effect in the fiber, which causes sudden fluctuations in the output signal. The light is transported by the fiber to the fiber coupler (Wideband coupler, 1550nm single window, 50:50 coupling ratio) where it is split into 2 paths, each with half power from the light before the split. One path leads to a photo detector (1550nm single mode) which converts the light to an electrical current. This serves as the reference signal. Light from the other path passes through a circulator (Oplink Comms Inc. single mode 3 port circulator), to a fiber patch cord (single mode fiber) with a polished face at the connector end which serves as the sensing point. Light is reflected back from the fiber tip, back to the circulator, and diverted to another photo detector which converts the light signal to an electrical one. The electrical signals are amplified and filtered to remove high frequency noise and 50Hz noise from the power lines. Next, the reference and measurement signals are input into a data acquisition card (National Instruments NI6035E DAQ) in a computer and the data recording is controlled by a Labview program (Labview version 6.1).

One of the methods used to calibrate humidity sensors is to use salt solutions that produce known relative humidity values when they are saturated. Table 4-1 shows the different kinds of salt solutions and their associated humidity. Different values of relative humidity can be obtained for different salt concentrations in the solution. After using the salt solutions for humidity testing of the fiber optic sensor to show the absence of response to humidity change, the solutions can then be used for salt concentration testing by dipping the sensor into the solutions instead of suspending

the sensor in air above the solution in a sealed chamber for humidity testing. The osmotic suction of the solutions, due to their known relative humidity value, can be calculated. Together with the response of the fiber optic sensor, a calibration curve of osmotic suction can be tabulated to show that the sensor can be used as an osmotic suction sensor.

Table 4-1: Salts and their associated relative humidity values in saturated solutions [4]

Solutions	Relative humidity from ASTM E 104 (%)	Measured relative humidity (%)
Magnesium Chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$)	32.8 ± 0.2	32.7
Magnesium Nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$)	52.9 ± 0.2	53.0
Sodium Chloride (NaCl)	75.3 ± 0.1	75.3
Potassium Chloride (KCl)	84.3 ± 0.3	84.3
Potassium Nitrate (KNO_3)	93.6 ± 0.6	93.9
Potassium Sulphate (K_2SO_4)	97.3 ± 0.5	97.7
0.5 molal KCl	98.4*	98.3
Pure distilled water	100	99.9

Preparation of salt solutions:

Table 4-2 shows the relationship between sodium chloride molality and the osmotic suction. The molality of a chemical species in solution is the number of moles of that solute per kilogram of solvent. Hence the concentration of sodium chloride can be varied in solution to give humidity values from 75.3%RH to 99.9%RH. The assumption made here is that in a sealed chamber with a salt solution, the osmotic suction of the solution is equal to the total suction. Therefore it is possible to use Equation 2 to find the equivalent relative humidity of a NaCl solution once the molality of the solution is known. Figure 4-18 shows the sealed chamber with the fiber probe, made up of a fiber patch cord connector with a polished face, and a container of salt solution encased in it.

Table 4-2: NaCl molality and Osmotic Suction values at different temperatures [2]

NaCl Molality	Temperature				
	0°C	7.5°C	15°C	25°C	35°C
Osmotic Suction (kPa)					
0	0.0	0.0	0.0	0.0	0.0
0.2	836	860	884	915	946
0.5	2070	2136	2200	2281	2362
0.7	2901	2998	3091	3210	3328
1.0	4169	4318	4459	4640	4815
1.5	6359	6606	6837	7134	7411
1.7	7260	7550	7820	8170	8490
1.8	7730	8035	8330	8700	9040
1.9	8190	8530	8840	9240	9600
2.0	8670	9025	9360	9780	10 160

Figure 4-18: Fiber patch cord connector with polished end in a sealed chamber with salt solution for humidity testing.

The calculations can also be performed in reverse, in other words, for a fixed relative humidity; it is possible to calculate how much solute is needed to be mixed into a certain amount of solution. The calculations are shown as follows:

Step One:

Using Equation (2), the total suction has to be calculated from the desired humidity of the NaCl solution.

Example:

If the desired %RH is 90%RH,

$$\Psi = -137325 \ln \left(\frac{u_v}{u_{v0}} \right)$$

$$\Psi = -137325 \ln(0.9)$$

$$= 14468.63 \text{ kPa}$$

Step Two:

Since osmotic suction is equal to total suction in a salt solution, in order to calculate the NaCl molality, the linear fit equation from Figure 4-19 can be used. Figure 4-19 was plotted using values of osmotic suction and NaCl molality at 25°C from Table 4-2.

Figure 4-19: Osmotic suction relationship with NaCl molality at 25°C

Example:

$$\Psi = 4894.1011X - 123.78624 \quad (\text{From Figure 4-19})$$

$$14468.63 = 4894.1011X - 123.78624 \quad (\text{Total suction value from Step One})$$

$$X = 2.9816 \text{ mol/Kg}$$

Step Three:

If 400g of distilled water is going to be used for mixing the NaCl,

$$X1 = 2.9816 \times 0.4$$

$$= 1.19264 \text{ mol}$$

Since the atomic weight of NaCl is 58.44g/mol, thus the amount of NaCl that must be added to the distilled water to achieve 90%RH is:

$$\begin{aligned}\text{Amount of NaCl} &= 1.19264 \times 58.44 \\ &= 69.70\text{g}\end{aligned}$$

Twelve solutions were prepared in order to calibrate the fiber sensor (Table 4-3) using the chamber. Four of the solutions (at humidity of 32.7%RH, 53.0%RH, 75.3%RH and 97.7%RH) were used for the humidity test for the sensor. The twelve solutions were used for the salt concentration test as they provide a good range of salt concentration for testing. The osmotic suction values were calculated using Equation 2 by substituting the humidity values of the solutions into the equation.

Table 4-3: Prepared Salt Solutions, their calculated humidity and osmotic suction values.

S/No	Salt Name	Relative Humidity (%RH)	Osmotic Suction (kPa)
01	Magnesium Chloride	32.7	153,501
02	Magnesium Nitrate	53.0	87,185
03	Sodium Chloride	75.3	38,958
04	Sodium Chloride	78	34,120
05	Sodium Chloride	81	28,937
06	Sodium Chloride	84	23,943
07	Sodium Chloride	87	19,124
08	Sodium Chloride	90	14,469
09	Sodium Chloride	93	9,966
10	Sodium Chloride	96	5,605
11	Potassium Sulphate	97.7	3,195
12	Pure Distilled Water	100	0

4.6 Measurement of Relative Humidity Using Fresnel Reflection From The Tip Of A Gold Tip-Coated Fiber

Following up on the cleaved SMF experiments, gold was coated onto the tips of cleaved SMF and MMF. This is an attempt to achieve greater intensity modulation of the light within the fiber core. According to Stuart et al. [50], electroplating the tip of fibers using silver, gold, nickel, copper or other metals causes the reflectivity of the thin films to change when the sensor is exposed to different humidities. This is observed when intensity of the back-reflection of light from the tip of the fiber changes with humidity.

With the availability of a gold sputtering machine in the laboratory (Jeol Fine Coater JFC-1200), sputter coating the tip of optical fibers becomes easily achievable. The thickness of the gold film coated onto the tip of the fiber can be calculated using the following [51]:

$$d(A)=kIVt \quad (22)$$

where

d – thickness in angstroms

k – 0.07 if using gold as a target

I – coating current in mA

V – voltage used in kV, 1 in this case

t – time in seconds

The current used was fixed at 30mA and the voltage used was 1kV. For example, for a desired thickness of 20nm, t should be:

$$\begin{aligned} t &= \frac{d}{kIV} \\ &= \frac{200}{(0.07)(30)(1)} \\ &= 95.24 \text{ seconds} \end{aligned}$$

For 20nm thickness, $t=95.24$ seconds

For 50nm thickness, $t=238.1$ seconds

For 100nm thickness, $t=476.2$ seconds

The accuracy of the thickness, as stated by the manufacturer, depends on the distance of the surface to be coated to the gold target as well as the quality of the gold foil target and the coating current used. Since the instrument was meant for sample preparation by coating a layer of gold over a sample before imaging using a scanning electron microscope, the accuracy is not good, with an error of $\pm 10\%$ of total thickness coated [51].

In this experiment, 3 different coating thicknesses were used: 100nm, 50nm and 20nm. For the 50nm and 20nm coating thicknesses, both MMF and SMF were used. The SMF used was standard 9 μm core fibers cleaved at the tip to achieve a flat surface. The MMF used was 50 μm core fibers cleaved at the tip also. For the 100nm coating thickness, only MMF was used.

After coating the fibers, the gold tip-coated fibers were put into a climate control chamber (Feutron KPK35). The climate control chamber has the ability to control both the temperature and the humidity within. The humidity range of the chamber is 10%RH to 98%RH and the sensitivity is 0.1%RH with an accuracy of 3.0%RH. The temperature range of the chamber is 10°C to 95°C and a sensitivity of 0.1°C with an accuracy of 0.5°C. The experimental setup is shown in Figure 4-20. Hence it is a much faster way to calibrate the response of the humidity sensors than to use salt solutions, though it is less accurate. The measurement system used to collect the results is the same as the one used in Figure 4-17.

Figure 4-20: Experimental Setup for Gold-Tip coated Fiber Experiments

4.7 Relative Humidity Measurements Using Absorption Spectroscopy

Previous methods utilized the idea of refractive index change of a medium (water or air) to modulate the intensity of light within the fiber. The biggest problem with this is that the change in refractive index of the medium due to hydrostatic pressure change or relative humidity is very small. Hence the modulation of light in the optical fiber due to this small change in refractive index is minimal too, resulting in the difficulty of trying to detect very small changes in intensity of light within the fiber against a very high intensity background.

Another way to look at the problem is to identify the unique optical characteristics (intrinsic) of humidity which change as humidity changes, instead of the changes brought upon the medium that absorbs humidity (extrinsic). Since humidity is simply water in vapour form, as a chemical compound, H_2O has very well documented optical properties such as refractive index and more importantly, its absorption spectra; the wavelengths of light which it absorbs.

Figure 4-21: The absorption spectra of water for all wavelengths [80]

The absorption spectrum of water is shown in Figure 4-21. This information is obtained from the HITRAN database [80]-[81]. As can be observed, the absorption spectrum of water begins with some relatively weak absorption lines in the 1300nm

region followed by another group of slightly stronger lines near 2000nm. This is followed by a 3rd group of much stronger lines at 2700nm. The last group of absorption lines, the strongest of all the groups, is situated around 6000nm.

Figure 4-22: Absorption spectra of water in the NIR region [80]

The focus is on the first group of absorption lines near the 1300nm region as that is within the telecommunications window of the optical fibers being used. Figure 4-22 shows a more detailed graph of the absorption lines in that region.

Figure 4-23: Absorption spectra of water in a focused region [80]

The absorption line of interest is the one at 1392.530nm (Figure 4-23). This line was used by Edwards et al. [53] to determine relative humidity of air. The advantage of this line is that it is relatively far away from the closest line, which

might interfere with the readings. In the absorption experiments for this project, this absorption line was used.

The simplest approach is based on a single absorption cell architecture as shown in Figure 4-24. The basic sensitivity levels are such that with even relatively weak absorption lines in the near-IR and compact absorption cells, detection thresholds in the tens to hundreds of parts per million are readily achieved.

Figure 4-24: Single pass absorption cell architectures using fiber collimators [68]

A tunable diode laser system was setup with fiber collimators as the single pass absorption cell. The intention was to use wavelength modulation to extract a $2f$ derivative signal from the photodetector, which can then be analyzed and calibrated with the level of humidity (concentration of water vapor in the ambient air). One problem encountered, however, was the lack of temperature control of the distributed feedback (DFB) diode laser used. Each DFB laser is fabricated to lase at a particular wavelength and in order to change the lasing wavelength to a particular absorption line of water, temperature control is required for coarse tuning and current injection can be used for fine tuning. The DFB diode laser that was used in the setup was from PD-LD Inc, with a lasing wavelength at 1390nm and 1mW power. Due to the low cost, there was no temperature control; there was no way to tune the wavelength of the laser to the required wavelength followed by stabilizing

the temperature so that the wavelength of the laser can be fixed at one value (e.g. aligned to an absorption line of water).

Figure 4-25: Simplified absorption spectroscopy setup. l is the absorption cell length.

Therefore, a simpler setup was used, shown in Figure 4-25, for measuring the absorption spectra of water vapor in air. The light source used in this setup is no longer a DFB diode laser, but an ultra broadband source termed as a “white light source” from Ando (AQ4303B) with a light output from 400nm to 1800nm. The light travels through the single-mode fiber to a fiber collimator (FOCI, 1550nm SMF collimator) which collimates the light from the fiber core to a beam whose diameter is 3mm wide. The single-mode fiber used here is Corning SMF-28 and the specifications can be found in Table 4-4.

Table 4-4: Fiber specifications for Corning SMF-28 [82]

Operating Wavelength	1550nm
Fiber loss at 1550nm	$\leq 0.30\text{dB/km}$
Cutoff Wavelength	$\lambda_{\text{cct}} < 1260\text{nm}$
Mode Field Diameter	9.50 to 11.50 μm at 1550nm
Core diameter	8.3 μm
Clad Diameter	125 μm
Numerical aperture	0.13
Effective group index of refraction (N_{eff})	1.4681 at 1550nm

The beam travels through air, where some wavelengths are absorbed by the moisture in the air, and is subsequently coupled back into fiber via another fiber

collimator which was aligned to the first one. The spacing between the two fiber collimators is the length of the absorption cell and in this experiment the length was set at 20mm. The OSA (Ando AQ6317) is used to analyze the spectrum received and to record data.

Due to the low optical output intensity of the white light source and the high sensitivity settings of the OSA to filter noise, the acquired waveform at the water absorption peak of 1392.53nm was very noisy. Using a signal processing program to filter the data to get a clean signal was explored. The program used was Matlab and the toolbox used was the Wavelet toolbox, which works by using unique wavelets to deconstruct the noisy waveform to details and approximations at different levels. The details contain the unwanted noise and the approximations represent the filtered signal.

Wavelets are mathematical functions that cut up signals into different frequency components and then examine each component with a resolution matched to its scale. They have proven to be very efficient and effective in analyzing a very wide class of signals and phenomena, and have advantages over the classical Fourier methods in analyzing physical situations where the signal contains non-stationary or transitory characteristics like discontinuities and sharp spikes [83],[84].

In traditional Fourier analysis, sinusoids (sines and cosines) are chosen as the basis to represent signals. The utility of Fourier transform lies in its ability to analyze a signal in the time domain for its frequency content. The transform works by first translating a signal in the time domain into a signal in the frequency domain. The signal can then be analyzed for its frequency content because the Fourier coefficients of the transformed function represent the contribution of each sine and cosine function at each frequency. However, Fourier analysis has one serious drawback. In transforming to the frequency domain, time information is lost. It is not possible to tell when a particular event occurs by looking at the Fourier transform of a signal. This deficiency can be corrected by using the short-time Fourier transform, which analyzes a windowed section of a signal at a time, to

provide some information about the time-frequency content of a signal event. Nevertheless, this information has limited precision that is constrained by the size of the window. Furthermore, once the time window size is decided, it remains the same for all frequencies (Figure 4-26a).

Figure 4-26: Coverage of Fourier basis functions and wavelet basis functions in the time-frequency plane [83]

Figure 4-27: Examples of some wavelets. The acronym of the wavelet family and the wavelet number in that family is used to describe wavelets, e.g. Db2 – Daubechies 2, sym2 – symlets 2, coif1 – coiflet 1 and coif5 – coiflet 5 [85]

A wavelet is a waveform with limited duration and has a mean value of zero (Figure 4-27). The fundamental idea behind wavelets is to analyze the signals according to scale. Wavelet analysis involves breaking up a signal into shifted and scaled versions of the original (or mother) wavelet [85]. Scaling of the mother wavelet allows the windowing size to vary (as opposed to a fixed window size for the short-time Fourier transform, see Figure 4-26b), thus enabling analysis with different

resolutions to be carried out. Localized information of signal events are also generated from the shifting of the analyzing wavelet. In comparison to Fourier analysis, local features of a signal can be described better with wavelets. By using the wavelet coefficients, original signal or function can be represented in terms of a linear combination of the wavelet functions. This implies that data analysis can be performed using just the corresponding wavelet coefficients. It is important to note that wavelet transforms do not have a single set of basis functions like the Fourier transform, which uses just the sine and cosine functions. Instead, wavelet transforms have an infinite set of possible basis functions. Thus, wavelet analysis can provide an immediate access to information that is obscure to other time-frequency methods.

a) Filter Banks splits the signal (s) into approximation coefficients (A) and detail coefficients (D)

b) After one iteration, the resultant approximation signal has lost some of the noisy “details” due to discrete wavelet transform (DWT). cD-Detail coefficients, cA- Approximation coefficients.

Figure 4-28: Discrete wavelet transform using complementary filters of wavelet [85]

An efficient way to implement the discrete wavelet transform is to use filter banks [85] (Figure 4-28a). The original signal is passed through two complementary filters (consisting of a highpass filter and a lowpass filter) related to the analyzing wavelet to produce two signals. Each of the two signals is then downsampled by a factor of two. The resulting output gives the discrete wavelet coefficients of the signal, which are made up detail and approximation coefficients (Figure 4-28b). The detail coefficients made up the low-scale, high frequency components of the signal while the high-scale, low frequency components of the signal are characterized by the approximation coefficients. The decomposition process can be iterated to obtain wavelet coefficients at different scales by passing successive approximation coefficients through the complementary filters. This multiple-level decomposition yields a hierarchical set of approximation and detail coefficients of the original signal.

Hence to de-noise a signal, the more times the signal is decomposed using the DWT, the more details it loses, hence an optimal number of iterations has to be found for any signal, otherwise the resultant de-noised signal will be different from what the actual signal should be.

For the experiment, the absorption cell was placed in the Feutron climate control chamber. The temperature was fixed at 25°C and the humidity was varied from 40%RH to 95%RH in steps of 5%RH. At each humidity setpoint, 3 scans were recorded with the OSA and these 3 scans were filtered using Wavelet Transform and analyzed. Finally a graph of sensor response (in terms of intensity) against humidity was tabulated.

4.8 Relative Humidity Sensing Using POFs

Another approach to measure relative humidity is not to measure the tiny change in refractive index of the medium (air), but to measure the optical change of certain humidity sensitive materials using optical fibers when humidity changes. The optical changes brought about by such materials are usually significant and easily measured. This is chemical sensing using fiber optic sensors and the chemical in question here is H_2O . Making use of the evanescent wave that penetrates into the surroundings from the core of a fiber, the light can be scattered or absorbed by a humidity sensitive film. And the resultant intensity change can be easily measured using a photo detector. In this experiment, cobalt (II) chloride was used as the humidity sensitive coating with gelatin as the overlay material that is coated onto the surface of plastic optical fibers.

Plastic fibers were chosen because such fibers have a large core, which allows a greater intensity of light to travel within the fiber. Since the sensing technique here is intensity modulation, the change in intensity of light is more significant because a higher intensity is being carried by the fiber. The plastic fiber can also be bent more easily than SMF by applying heat and it does not break as easily as standard single-mode fiber which is made of SiO_2 .

Figure 4-29: Experimental setup for POF RH sensor

The experimental setup used is shown in Figure 4-29. An aluminium gallium arsenide (AlGaAs) light emitting diode (LED) from Kingbright was used as the light source. It has a peak wavelength of 660nm, a spectral line halfwidth of 20nm and a viewing angle of 20°. The plastic optical fiber used has a core of PMMA and a very thin cladding of fluorinated polymer. Due to the large 2mm fiber diameter, no connectors were used. The plastic fiber was bent to a radius of 1cm at the sensing end using a flame. A momentary application of heat to the POF can sufficiently soften and bend it around a mandrel. Powdered cobalt hexahydrate (3% by weight) and gelatin (5% by weight) was mixed with distilled water to form an aqueous solution that was heated in an oven to 65°C for 10 minutes. The curved fiber end was then dipped into the solution and then left out in the open to dry. Figure 4-30 illustrates the appearance of the sensor. The dipping was done three times to obtain three coats. The measured thickness of the film was 30µm, using a vernier caliper that is accurate to 20µm to measure the diameter of the coated fiber. A large area (1cm²) silicon photodetector was used to detect the optical intensity variations from the POF and a digital multimeter (HP34401A) displays the corresponding voltage.

Figure 4-30: The POF sensor after a film of CoCl₂ and gelatin has been applied. The fiber diameter is 2mm.

The sensing end of the POF was placed into a climate control chamber (Feutron KPK35). The humidity range of the chamber is 10%RH to 98%RH and the

sensitivity is 0.1%RH with an accuracy of 3%RH. The temperature range of the chamber is 10°C to 95°C and a sensitivity of 0.1°C with an accuracy of 0.5°C. The temperature within the chamber was fixed at 25°C for all the experiments and the humidity was varied from 47%RH to 95%RH to test the response of the sensor.

The sensing range, resolution, sensitivity, accuracy and hysteresis response of the POF RH sensor were investigated. Furthermore, the effects of bending radius of the fiber at the sensing point as well as fiber core diameter on sensitivity of the humidity sensor were studied in order to find the best way to improve sensing performance.

4.9 Relative Humidity Sensing Using LPGs

As there is a limit to the sensing range of POF RH sensor, another kind of fiber optic sensor, an LPG coated with gelatin, was tested and found to have good response in the high humidity region (>90%RH), thus making it suitable to be used as a soil suction sensor.

The LPG used for the experiment was fabricated using the amplitude mask technique, see Figure 4-31. The type of photosensitive optical fiber used was standard telecommunications optical fiber, Corning SMF-28, hydrogen-loaded for 7 days at 1500psi and at a temperature of 70°C.

Figure 4-31: System setup for fabrication of LPG [86]

A broadband source was launched on one end of the photosensitive fiber while the other end of it was connected to an optical spectrum analyzer (OSA), ANDO AQ6317. A section 10mm long on the optical fiber was scanned repeatedly through a 300 μ m amplitude mask using a frequency doubled Argon-Ion laser emitting UV light at the wavelength of 244nm. In this experiment, the LPG was fabricated with its highest order cladding mode within the optical communication window, 1520nm to 1620nm. Reasons for doing so are to enable the use of current light source as well as to offer flexibility in deploying the sensor in both industrial environment and optical communication systems. To stabilize the LPG spectra, all the optical fibers were annealed at 85°C for 18hrs [86].

In the final part of the fabrication, the LPG was coated with gelatin. The gelatin solution was prepared by mixing dry gelatin powder with distilled water. The ratio was 1g of gelatin with 20g of distilled water, or 5% by weight of the solution. The solution was then heated up to 65°C for the gelatin to be completely dissolved. Dip coating was initially used for the coating of LPG; however this method did not yield an even layer along the grating region as many uneven lumps of gelatin were formed on the fiber. Applying an even coat of gelatin over the grating region was accomplished using a fine brush [86].

Once the coating of gelatin is dry, a brass tube with the center portion filed away to form an opening is positioned such that the LPG is in the center of the opening (Figure 4-32). Protective tubing is inserted over the fiber into the brass tubing. The joints on both sides of the tubing were epoxied so that the protective tubing will not come loose and to keep the LPG permanently straight. Finally heat-shrink tubing is placed over the joints to prevent unnecessary contact or bending of the fragile parts.

Figure 4-32: Packaging layout for LPG RH sensor

Figure 4-33: LPG high humidity sensor prototype packaged together with a brass tube to prevent bending

Figure 4-33 shows the actual prototype of the LPG RH sensor with some dimensions. Besides using gelatin as the main coating agent, experiments with LPGs coated with poly-vinyl alcohol (PVA) were also conducted. It was observed that gelatin was less sensitive in the lower humidity range between 40%RH and 85%RH, but was highly sensitive in the high humidity region between 90%RH and 100%RH. Although PVA offers better sensitivity in the lower humidity range, its high humidity sensitivity was inferior to that of gelatin. Thus to develop a LPG for use as a high humidity sensor, gelatin was chosen as the main overlay material.

Figure 4-34: Experimental setup for LPG RH

sensing. [86]

To conduct the humidity tests, the LPG sensor was placed in a climate control chamber (Feutron KPK35) where both humidity and temperature can be controlled. Due to the limitation on the lowest possible RH that could be achieved by the chamber, the test started from approximately 43% RH. The humidity was slowly ramped to 99%, with 5% step intervals from 43% to 90% and 1% step intervals from 90% to 99%. All readings were taken using the OSA with a synchronized tunable laser source (TLS) (ANDO AQ4321D) as the light source. The chamber temperature was maintained at 25°C throughout the experiments. The OSA has a level resolution of 0.01dB and the TLS has a level stability of 0.05dB [86].

The tests done include: sensitivity, resolution, accuracy and hysteresis of the sensor. Further investigations include finding how different grating pitch affects the sensitivity of the sensor and the calibration of the sensor with respect to humidity for soil testing.

4.10 Soil Test Procedures

The LPG RH sensor, being suitable for testing in soil due to its good response in humidity ranges greater than 90%, is going to be used in actual soil in laboratory conditions to test its response and resolution. Before actual testing can take place, the soil has to be compacted and equilibrated to fixed suctions values. In general the process is as follows. Powdered kaolin soil is compacted into standard shapes with fixed moisture content initially such that all soil samples have the same structure after fabrication. After that, the soil specimens are put into a pressure plate to fix a pair of soil specimens at different soil matric suctions. From each pair, one soil specimen was tested with the null-type to ascertain the matric suction and the other specimen was tested with the LPG RH sensor. All soil samples were tested using the Aqualab chilled mirror hygrometer to find out their RH. Finally the soil specimens are placed into an oven to remove all moisture and the difference in weight of the specimens before and after baking represents the gravimetric water content.

Fabricating the soil specimens:

90g of distilled water was added to 400g of dry kaolin soil (fine white powder) and mixed thoroughly for 15 minutes. A sieve was used to separate the bigger lumps so that the loose soil can be of a uniform particle size. The bigger lumps were just broken apart and put through the sieve again till all of the soil particles are of the same size. The amount of water used here corresponds to 22% water content throughout the soil. Next, a mold was used to compact the soil into cylindrical soil specimens. The size used here is 5 cm diameter by 3 cm height for the soil samples to be prepared. A certain weight of soil powder was measured and poured into the mold. A loading machine compressed the mold such that the soil within gets compressed from powder form into a cylindrical shape.

After the soil specimens have been crafted, they were wrapped first with cling wrap followed by aluminium foil to protect the specimens and to keep the moisture content of the specimens constant. Lastly the specimens were placed into a controlled environment chamber for storage.

Preparation of the soil samples using the pressure plate:

Figure 4-35: Saturating soil specimens at fixed matric suctions using the pressure plate apparatus

One out of every two specimens had a 5mm diameter hole drilled through the center of the specimen before changing the specimen's suction in a pressure plate (Figure 4-35). A hand drill was used for this purpose. Two soil specimens, one with a hole and the other without, were loaded into the pressure plate at a time. The air pressure was then adjusted to a value that corresponds to the required matric suction of the soil specimen (e.g. 100kPa).

Testing procedure of the soil specimens:

Once the soil specimens were ready for testing, the specimen without the hole was tested for matric suction using the null-type equipment. This is to check the actual equilibrated matric suction of the specimens. Some soil chipped off from both specimens were tested in the Aqualab (Decagon, Water Activity Meter model series 3) chilled mirror hygrometer to check the relative humidity of the soil, which can then be compared with humidity values obtained from the LPG RH sensor. The Aqualab hygrometer has a sensing range from 10%RH to 100%RH with a resolution of 0.1%RH and an accuracy of $\pm 0.3\%$ RH. The accuracy corresponds to a

suction accuracy of $\pm 400\text{kPa}$. After these tests were completed, the soil specimen with a hole was tested by inserting the LPG RH sensor through the hole and modeling clay was used to plug the hole on both sides of the specimen so that the volume of space within the soil that the sensor has to measure soil humidity is small. This was followed by wrapping the whole package tightly with cling wrap and aluminium foil to ensure that the humidity of the specimen does not change. The setup is shown in

Figure 4-36. The tests were done in the climate control chamber as the temperature was kept constant at 25°C .

After the testing with the LPG RH sensor, the soil specimen was finally weighed and put into an oven for a gravimetric test. Once all the moisture was driven out from the sample, it was weighed again. The difference in weights represents the gravimetric water content of the soil specimen.

Figure 4-36: Experimental setup for soil test using LPG RH sensor in the climate control chamber

Chapter 5 Experimental Results and Discussions

5.1 FBG Sensors for Measurement of Pore-Water Pressure

Table 5-1 below shows the results of testing various FBG embedding configurations with the Chatillon force gauge. The surface area of contact with the carbon fiber composite was calculated to be $6\text{E-}06\text{m}^2$ (6mm x 1mm). As the force gauge values are in Newtons, they are converted to kilopascals (kPa) by the means of the simple formula: $\text{Pressure} = \text{Force} / \text{Area}$.

Table 5-1: Chatillon Force Gauge Test Results

FBG ID	FBG Config	Positive Force Gradient pm/N	Negative Force Gradient pm/N	Calculated positive pressure pm/kPa	Calculated negative pressure pm/kPa	Comments
A1	1-1, //-ply					Values fluctuates, no stable reading.
A2	2-1, X-ply					No linear relationship.
A3	2-2, //-ply	2.6	2.6	0.016	0.016	
A4	3-1, //-ply	22.24	14.91	0.133	0.089	
A5	3-1, X-ply	31.82	29.71	0.191	0.178	
A6	3-2, //-ply	7.92	8.85	0.047	0.053	
A7	3-2, X-ply	15	8.69	0.090	0.052	
		17.22	14.15	0.103	0.085	
B1	0-1					Waveform disappears. Erratic.
B2	2-1, //-ply	5.43	6.33	0.033	0.038	
C1	2-1, X-ply					No linear relationship.
C2	3-2, X-ply					No linear relationship.
C5	4-2, //-ply	8.7	11.02	0.052	0.066	
C6	4-2, X-ply	2.77	2.4	0.017	0.014	
C7	4-3, //-ply	2.62	2.3	0.016	0.014	
C8	4-3, X-ply	11.2	15.4	0.067	0.092	

From the table, the sensors with erratic responses are excluded from the triaxial cell test. In general, the more embedding layers used (i.e. thicker diaphragm) the stiffer was the package. A stiff package would not be sensitive to small changes in pressure but will be able to sustain very large forces.

Table 5-2: Triaxial Cell Pressure Testing Results

FBG ID	FBG Config	Positive Pressure Gradient pm/kPa	Negative Pressure Gradient pm/kPa	Comments
Pre "A" No. 4	2-1, //-ply	-0.991	-1.22	Good response of sensor in negative pressure region.
A4	3-1, //-ply	-0.602	-0.671	
A5	3-1, X-ply	-1.08	-1.19	Good response of sensor in negative pressure region.
A6	3-2, //-ply			Sensor with broken fiber. Could not take readings.
A7	3-2, X-ply	-0.403	-0.41	
B2	2-1, //-ply	-0.273	-0.0101	Too much epoxy used. Response of sensor was not good.
C5	4-2, //-ply	-0.349	-0.35	Large residual stress due to thick layers. Response of sensor is slow.
C6	4-2, X-ply	-0.0574	-0.134	Large residual stress due to thick layers. Response of sensor is slow.
C7	4-3, //-ply	-0.02	-0.104	Large residual stress due to thick layers. Response of sensor is slow.
C8	4-3, X-ply	-0.0607	-0.0706	Large residual stress due to thick layers. Response of sensor is slow.

In the triaxial cell test, the sensors were subjected to a maximum positive pressure of 600kPa and a maximum negative pressure of -90kPa. From the data in Table 5-2, the general trend is that the thicker the layers, the poorer the sensitivity of the sensor response. The best sensitivity was obtained for 2-1, //-ply, configuration. The positive pressure sensitivity is at -0.991 picometers of wavelength shift per kilopascal (-0.991pm/kPa). The negative pressure sensitivity is at -1.22 picometers of wavelength shift per kilopascal (-1.22pm/kPa). The greater the wavelength shift per kPa, the more sensitive the sensor is. The accuracy of the OSA is ± 70 pm; hence it is not able to detect small changes in wavelength shift. Other equipment with greater resolution like FBG interrogation systems, e.g. the picowave interrogation system from Micron Optics Inc., has resolutions up to 1pm. Using OSAs or

interrogation systems with greater resolutions will be able to register small changes in pressure. Another alternative will be to redesign the sensor for it to become more sensitive to small changes in pressure.

Figure 5-1: Temperature Response of different layering configurations of prepreg material.

An experiment to test the temperature response of the sensors was conducted. As the sensors respond to temperature changes, which in turn affects the pressure readings, the temperature response of the sensors need to be characterized. The results are shown in Figure 5-1. Even though the sensors had different numbers of prepreg layers, their response to temperature is very similar as the gradients of the lines varies no more than 3pm/°C from each other. The maximum gradient recorded is 11.83pm/°C, which is below the measurement accuracy of the Anritsu OSA, thus the temperature effect on the readings is not significant.

The range of the sensors is limited by the birefringent effect in optical fibers. When a large force is applied onto the sensor, the birefringent nature of fiber comes into play, causing the grating to deform [61]. The waveform shape of the reflected signal changes to a point where it is difficult to find the peak wavelength of the waveform (Figure 5-2).

Figure 5-2: Waveform of reflected signal from FBG sensor as applied pressure increases.

Although the test was performed with positive pressure (high negative pressure cannot be achieved due to the limitation of the vacuum feed line), the sensor was meant to be used for negative pressure measurements. But due to the reason that the diaphragm is secured on four sides of the mount with epoxy to keep water out, the diaphragm cannot bend easily either inwards or outwards, thus uneven forces are applied on the FBG in which a flattening of waveform will occur like that shown in Figure 5-2.

Advantages of using FBGs for hydrostatic pressure sensing:

- ✓ Wavelength modulated, hence intensity fluctuations due to source or bending of fiber does not affect measurements.

Disadvantages of this method include:

- ✗ The sensing portion is large and the carbon composite diaphragm cannot be further reduced in area due to the limit in FBG length. This makes the sensor difficult to implement in experiments.
- ✗ Cavitation occurs very easily with the large area diaphragm; hence the range of negative pressure is limited.
- ✗ The range of sensor is limited due to birefringent effect of the FBG.

To improve the sensitivity and range of the FBG sensor for measurements of negative pore-water pressure it is necessary to overcome a few problems, namely:

- ➡ Metal casing may have to be redesigned to hold the diaphragm in a more secure way such that it can bend properly to improve sensitivity and reduce birefringent effect.
- ➡ Diaphragm has to be redesigned to provide more sensitivity, a configuration of 2-1, 45°/0/FBG/0, where the top layer is angled 45° to the axis of the fiber is possible.

The conclusion is that the sensor is not very suitable for direct measurements of negative pore-water pressure due to the large size of the diaphragm (carbon fiber composite) which is limited to the length of the FBG, the ease at which cavitation can occur on the large diaphragm at negative pressures and the lack of sensitivity of the sensor to small changes in pressure. Hence the next investigation focused on the possibility of using LPGs to measure the refractive index change in liquid water when hydrostatic pressure is applied. The use of LPGs as the sensor reduces the number of parts needed for the sensor thus making it simpler.

5.2 LPG Response for Hydrostatic Pressure Test

Figure 5-3 shows the results of the experiment using an LPG in an attempt to measure the refractive index change of water when pressure is applied to the fluid. There is no observable trend or an indication that the sensor does respond to refractive index changes in the water. The reason for this can be found in a release from the International Association for the Properties of Water and Steam [88].

Figure 5-3: Wavelength shift of resonant wavelength of the LPG with respect to hydrostatic pressure change

Table 5-3: Variation of the refractive index of water with respect to hydrostatic pressure at a wavelength of 1.01398 μm and 0°C [88]:

Hydrostatic Pressure/ kPa	Refractive index of water
100	1.326135
1000	1.326279
10000	1.327710
100000	1.340435

For the pressure range from 100 kPa to 1000 kPa, the change in refractive index of water per kPa is 1.6×10^{-7} . The theoretical sensitivity of an LPG is about 10^{-5} which is much less than required resolution of 10^{-7} , thus the LPG was unable to respond to the refractive index change of water when hydrostatic pressure was applied. A further development to improve the refractive index sensitivity of the sensor is necessary for the sensor to work as a pore-water pressure sensor.

Advantages of using LPGs for hydrostatic pressure sensing:

- ✓ Wavelength modulated, hence intensity fluctuations due to source or bending of fiber does not affect measurements.
- ✓ Simple sensing setup, just the bare fiber with an LPG without any other parts reduces possibility for sensor failure.
- ✓ Only standard single mode fibers needed, thus keeping the size of the sensor as small as possible.

Disadvantages of using LPGs for hydrostatic pressure sensing:

- ✗ Excellent sensitivity to external refractive index change required.
- ✗ A single-ended LPG sensor is required for ease of handling.

Possible improvements:

- ➡ Use LPG interferometric sensors which have excellent refractive index sensitivity and it can be made single-ended (See Section 5.7).

The LPG sensor can be improved by using more sensitive sensing techniques like fiber optic interferometry. Details are discussed in Section 5.7. The next investigation focused on using Fresnel's reflection from the tip of an optical fiber to determine the osmotic suction of different salt solutions. A pilot study to determine if the sensor can be used for humidity sensing was also conducted.

5.3 Measurement of Salt Concentration in a Solution Using Fresnel Reflection from the Tip of a Single-Mode Fiber

5.3.1 Humidity test results

This section is focused on a pilot study to ascertain if the sensor is capable of measuring relative humidity changes of air. A single humidity calibration using four salt solutions was conducted. Figure 5-4 illustrates the response of the laser source over time. It fluctuates to a great degree but it was found that the laser fluctuates around a mean value. In the case of Figure 5-4, the mean value is 3.58V, assuming that the gradient is negligible for the block of time when the reading was taken.

Figure 5-4: Tunable Laser Source Response with Time

The laser source fluctuation is in the unit of voltage because a photodetector was used to measure the intensity change of the light source. The photodetector circuit converts the current signal (from the photodetector where incident photons create electron hole pairs in the semiconductor) to voltage which is then sampled by the data acquisition card in a computer. The purpose of Figure 5-4 is to show that the light source does fluctuate and it affects the reading taken at the photodetector to some degree. If the source intensity fluctuation is not corrected to some degree, the actual sensing signal will be affected by the fluctuation as well.

When taking readings, it is important not to look at individual points but an adequate sample, or a number of points, is required so that the gradient of the best fit line is negligible thus rendering the mean to be accurate. A series of readings were taken for each salt solution in a chamber.

Figure 5-5: Calibration of sensor with 32.7%RH

Figure 5-5 illustrates the response of the sensor due to a relative humidity of 32.7% using MgCl_2 solution. The top waveform in the figure represents the reference waveform. The fluctuations are due to the source fluctuation. The reference waveform's mean value will only change when the source output light intensity changes. The measurement signal on the other hand will change according to any external change in the refractive index of the external environment. To compensate the effects of source fluctuations, a ratio of the measured signal to the reference signal can be taken. The ratio (response of the sensor) is calculated by:

$$\text{Ratio} = \frac{V_{\text{measured}} \times 10}{V_{\text{Reference}}} \quad (23)$$

In the case of Figure 5-5, the ratio is calculated in the following way:

$$\text{Ratio} = \frac{8.857}{9.584} \times 10 = 9.241$$

Figure 5-6 shows the consolidated graph where the sensor was calibrated against 4 different saturated solutions producing 32.7%RH, 53.0%RH, 75.3%RH, and 97.7%

RH. The sensor was calibrated against a wide range of humidity readings initially so that a general trend could be obtained to see if the sensor responded to changes in relative humidity. It did not respond, as demonstrated by the graph in Figure 5-6, where the ratios from the sensor remained nearly constant even though the humidity was varied over a wide range. The reason for this is because the change in refractive index of air as a result of a change in humidity is extremely small, in the order of 10^{-8} , as shown in Figure 4-15. This pilot study shows that this experimental setup cannot be used for humidity sensing.

Figure 5-6: Calibration curve for fiber optic sensor response (voltage ratio) versus relative humidity

5.3.2 Salt concentration measurements in an aqueous solution

In the salt solution concentration experiment, the fiber sensor was put directly into the salt solutions to measure the response of the sensor to different salt concentrations. There were 12 solutions with each corresponding to a different osmotic suction value (Table 4-3). Figure 5-7 shows the results of the ratio (response of the sensor) versus the osmotic suction values of the salt solution.

Figure 5-7: Sensor response with respect to calculated osmotic suction values of different saturated salt solutions

The fiber sensor was able to detect small changes in the concentration of the salt solutions by measuring the refractive index change of the solution. The osmotic suction of a solution is related to the concentration and types of salt in the solution and it can be calculated from the relative humidity that a saturated salt solution is associated with by using thermodynamic relationships like Equation 2.

Figure 5-7 shows that the cleaved SMF sensor is capable of measuring the osmotic suction of the different salt solutions, being very sensitive in the suction range lower than 20MPa and being less sensitive when the osmotic suction increases. Fiber optic osmotic suction sensors have not been reported in literature before; hence this is a simple and effective experimental setup for measuring osmotic suction. Actual soil tests have to be performed and the results should be compared with measurements from conventional instruments in order to characterize the fiber optic sensor for future work.

Such a fiber sensor for osmotic suction measurements has advantages over conventional electrical sensors like being less susceptible to corrosion, being small and unobtrusive, being capable of real time monitoring as well as remote monitoring over large distances.

Advantages of using Fresnel reflection in cleaved SMF for humidity sensing:

- ✓ Sensor is simple, just a cleaved SMF is sufficient.
- ✓ The sensor is able to measure the osmotic suction of a solution based on the different refractive indices of the solutions that have different kinds and concentration of salt.

Disadvantages of using Fresnel reflection in cleaved SMF for humidity sensing:

- ✗ The refractive index change of air due to humidity change is too small for the sensor to register.

Possible improvements:

- ➡ Perform soil tests and make a comparison of results with conventional instruments to characterize the performance of the fiber optic sensor.

Since the cleaved SMF was unable to register the refractive index change in air due to humidity change, a metal film coated onto the tip of the fiber could help to modulate the reflected light in the fiber to a higher degree. The next investigation focused on coating the tip of the fiber with a layer of gold so as to improve the humidity sensitivity of the sensor.

5.4 Relative Humidity Measurements Using Fresnel Reflection from the Tip of a Gold Tip-Coated Fiber

Table 5-4: Results for gold tip-coated fiber

Fiber Type=> Sensor ID=>	100nm Coated		20nm Coated				50nm coated			
	MM	MM	MM	MM	SM	SM	MM	MM	SM	SM
	G1a	G3a	1	2	1	2	1	2	1	2
%RH	Voltage Ratio	Voltage Ratio	Voltage Ratio	Voltage Ratio	Voltage Ratio	Voltage Ratio	Voltage Ratio	Voltage Ratio	Voltage Ratio	Voltage Ratio
40	3.21979	3.63588	1.82025	3.2961	6.4396	6.22631	3.40291	4.66314	6.24231	3.89768
50	3.55118	3.23875	2.09841	3.69951	6.3505	6.22086	4.30479	3.85205	5.56294	3.87909
60	2.94626	3.17042	2.06257	3.43324	6.36351	6.21254	3.15883	3.98401	6.18639	3.85693
70	2.52455	3.28013	1.86328	4.24743	6.36775	6.24971	3.72362	4.00892	6.18943	3.86569
80	3.09544	2.8152	1.85094	3.6684	6.44511	6.27589	3.90802	3.97724	6.2558	3.88147

A plot of the results shown in

Table 5-4 showed there was no discernable trend in the response of the sensors with respect to humidity regardless of the thickness of gold coating or the type of fiber used. Apparently the core size of the MMF at 50 μ m was not enough for the modulation of the light intensity in the fiber core to be good enough to be detected, hence the erratic response of the various sensors. Stuart et al. [50] used PCS fibers with a core diameter of 600 μ m, which allows a much greater intensity of light to interact with the metal film at the tip of the fiber.

As the gold tip-coated fiber showed that the modulation of light is still too small to be detected, another unique method was investigated to find out if it is suitable for humidity sensing. The next investigation focused on using the unique optical absorption characteristics of water itself to measure relative humidity in the air using absorption spectroscopic techniques.

5.5 Relative Humidity Measurements Using Absorption Spectroscopy

The absorption line of water at 1392.530nm, using the experimental setup shown in Figure 4-25 to record, is shown in Figure 5-8. The reason why it is so noisy is due to the white light source having a low output intensity which is further attenuated when the light has to pass through the air via the fiber collimators. Furthermore, the OSA settings at a resolution of 0.05nm and a sensitivity of “High 2” cuts down the light incident to the OSA. Hence some signal processing is necessary in order to filter out the noise and retrieve the waveform of the original absorption line. This is necessary as the dip of the absorption line varies with the amount of moisture in the air and this will give an indication of the humidity of the surroundings.

Figure 5-8: Absorption line of water at 1392.530nm obtained using the absorption spectroscopy setup

Figure 5-9: Matlab Wavelet Toolbox showing the 1D decomposition of the signal (s) into approximation (a5) and details (d1- d5)

The Matlab Wavelet toolbox was used to decompose the signal and reconstruct the filtered signal, in this case, the approximation (Figure 5-9). The wavelet used was coiflet 5 and the level of decomposition is 5. The filtered signal is shown in Figure 5-10.

Figure 5-10: Reconstructed signal after wavelet analysis

The waveform of the absorption line is much cleaner than the original waveform captured by the OSA after filtering. For the humidity test, at each humidity set-point, 3 scans were made and filtered using wavelet transform. The scans for each individual humidity set-point for the humidity test are shown in Figure 5-11. The value of the dip of the deepest waveform was used for the 3 scans of each humidity set-point. This is because the variation in dip intensity between 3 scans of the same humidity set-point can be quite large. A plot of the consolidated humidity test is shown in Figure 5-12.

Figure 5-11: Humidity calibration results (after wavelet transform) for each humidity set-point, (a) to (l), using the absorption setup. Each humidity set-point has 3 scans that are filtered through wavelet transform.

Figure 5-12: The compiled humidity calibration using the absorption spectroscopy

The intensity variations up to 30dB for the graph in Figure 5-12 are due to the use of wavelet transform to de-noise the original signal. An explanation of how wavelet transform can be used to de-noise signals is found in Section 4.7. Judging from the results in Figure 5-11, using the wavelet transform technique to de-noise the original signal (e.g. Figure 5-8) is not feasible due to the reason that the original signals were too noisy, so much so that a filter of the signals resulted in great intensity variations. The data obtained from the original signals generally have a smallest value of -100dBm (at the dip) and when the intensity was too low to be recorded, the OSA records the data point as -210dBm, observed from the data file. From Figure 5-8, it can be observed that the OSA fails to capture many intensity points, and hence records them as -210dBm. When each data set is filtered using wavelet transform, the difference between -100dBm and -210dBm leads to large approximation errors, which can be observed from Figure 5-11 (f) and (i). Even at a fixed humidity set-point, 3 similar scans at the same settings vary wildly from each other. Hence the original signal's intensity should be increased by using a stronger light source.

Another important point to consider is the length of the absorption cell. According to the Beer-Lambert law, the path length of the cell must be long in order to obtain good absorption. This simply means that the further the collimated light beam travels through the air, the greater the absorption; hence the modulation of the light

will be more significant as well. The absorption cell length used in this experiment was only 20mm, a very short absorption length.

Advantages of using absorption spectroscopy for relative humidity measurements:

- ✓ Simple sensing setup.
- ✓ Only standard single mode fibers needed.

Disadvantages of using absorption spectroscopy for relative humidity measurements:

- ✗ Specialised and expensive light source needed.
- ✗ Absorption cell path cannot be too short or else modulation won't be sufficient.
- ✗ A folded absorption cell is needed in order to make a probe that is single-ended (connected only on one end with fibers).

Some improvements that could be made:

- ➡ Use a brighter light source.
- ➡ Use an absorption cell with a longer path length.

As the absorption spectroscopic setup did not work successfully, the next investigation focused on developing a chemical sensor out of optical fiber. The chemical to be detected is H_2O , and a humidity sensitive film was coated onto an optical fiber. The modulation of light within the fiber will be significant due to the properties of the humidity sensitive film when humidity in air changes.

5.6 Relative Humidity Sensing Using POFs

Figure 5-13 shows the hysteresis response of the POF sensor. There is a response from the sensor beginning from 60%RH and terminating at 95%RH. The linear region is from 65%RH to 85%RH and the gradient of the increasing humidity curve from 65%RH to 85%RH is $0.07425\text{V}/\%\text{RH}$ and the corresponding gradient for the decreasing humidity curve for the same range is $0.07485\text{V}/\%\text{RH}$. The hysteresis error is only 0.0006V , a negligible amount in terms of humidity. A change of $0.01\%\text{RH}$ is detectable as the digital multimeter is capable of displaying voltages up to the fourth decimal place. The sensor responds quickly to changes in humidity, in terms of seconds, and the voltage readout stabilizes to a constant value within a minute. The equilibrium time is humidity dependent. The higher the humidity is, the longer the readings take to stabilize.

Figure 5-13: Hysteresis response of the POF RH sensor

In order to test the repeatability of the response of the sensor, the humidity within the chamber was ramped up and back down three times. Figure 5-14 shows the comparison of the results of the three tests for increasing humidity and Figure 5-15 shows the comparison of the results of the three tests for decreasing humidity.

Figure 5-14: Humidity ramp up for POF RH sensor

Figure 5-15: Humidity ramp down for POF RH sensor

The experimental data were fitted to a curve and the standard deviation for the increasing humidity curve was found to be about 0.05V, an uncertainty in measured relative humidity in the linear region of about $\pm 0.67\% \text{RH}$. The repeatability error is about 2.7% for the whole range of voltage response due to humidity change.

Likewise, the standard deviation for the decreasing humidity curve was found to be 0.02V, an uncertainty in measured relative humidity in the linear region of about $\pm 0.27\%RH$. The repeatability error is about 1.1% for the whole range of voltage response due to humidity change.

The POF is a lossy fiber, hence even without bending the fiber, some light can be observed to be leaking from the side of a straight fiber. Bending the fiber causes an increase in bending loss at that point and also shifts the peak of the mode field distribution away from the fiber axis towards the outer edge of the bend (Figure 5-16). This causes a stronger evanescent wave to propagate through the thin film. The evanescent wave penetrates the thin cladding of the POF easily to reach the film. Therefore, a higher intensity modulation can occur due to the increased interaction between the evanescent wave and the absorbing thin film that acts as a cladding for the POF. As demonstrated later, a different bending radius will affect the sensitivity of the sensor. The humidity sensing range is fixed by the properties of the $CoCl_2$ /gelatin film, as observed by Russell et al.[31], where there is little or no water absorbed from 30%RH to 50%RH.

Figure 5-16: Mode field distribution and light paths at the bent region of the POF sensing portion

Apart from the higher optical intensity modulation expected from bending the fiber, an increase in the intensity of the evanescent wave will improve the sensitivity of the sensor as well. For fibers with smaller core diameters, the evanescent tail of the core mode cannot easily penetrate the cladding as the intensity of the core mode is smaller and the cladding of the fiber is thicker with respect to the smaller core size. A higher intensity of evanescent wave will also allow a greater modulation with the absorption cladding film because more light can be absorbed. One way to improve the optical intensity of the evanescent wave in a POF is to use larger diameter fibers that can carry higher optical power and also have larger core to cladding ratios.

Figure 5-17: Effect of fiber diameter on sensitivity and range of POF RH sensor

An experimental investigation was conducted to find out the effect of different fiber parameters, like fiber core diameter and bending radius, on the sensitivity of the POF humidity sensor. Figure 5-17 shows the effect of different fiber diameters and Figure 5-18 shows the effect of different bending radius on the response of the POF sensor. It can be observed from Figure 5-17 that the larger the fiber diameter, the better the sensitivity of the sensor with respect to humidity. All sensors used in the fiber diameter experiment have constant bending radius of 2mm, a constant coating thickness of 10 μ m and a fixed composition of 5% gelatin and 3% CoCl₂ by weight. Figure 5-18 shows that the smaller the bending radii of the sensing portion of the

fiber, the better the sensitivity with respect to humidity. In this experiment, the fibers used have a fixed diameter of 2mm, a constant coating thickness of 10 μ m and a fixed composition of 5% gelatin and 3% CoCl₂ by weight.

Figure 5-18: POF RH sensor response for different bending radii

Figure 5-19: POF RH sensor response to different coating thickness

Figure 5-19 illustrates the effect of different coating thicknesses on the sensor response. In general, the thinner coatings enable the sensor to have a better response

with respect to humidity because the moisture can diffuse faster through the film as compared to the thicker films. Thin films not only promote fast sensing response, but also save material.

Figure 5-20: POF RH sensor to different types of humidity sensitive coatings

Poly vinyl alcohol (PVA) and gelatin can be used as the overlay material for CoCl_2 . In order to find out which is the best overlay material, or a combination of both is better, an experiment has been carried out to verify this. One coat on a 2mm POF with a 2mm bending radius was used for all materials. Figure 5-20 shows the results. Using gelatin as the overlay material achieves a better intensity modulation of the light in the fiber at the higher humidities. Using PVA, on the other hand, allows the sensor have a greater range of sensing for humidity than if gelatin was used. The other hybrid combinations of gelatin and PVA as the overlay material for CoCl_2 just give a sensor response that is in between that of the two. Using just pure gelatin or pure PVA without the CoCl_2 as overlay material produces interesting results from the sensor.

Figure 5-21: POF RH sensor response with pure gelatin or PVA coating only.

Figure 5-21 shows the sensor response when either gelatin or PVA without CoCl_2 is used as the overlay material. Assuming that the refractive index of gelatin decreases when humidity increases, the critical angle for TIR within the fiber increases too, therefore providing less loss at the bent sensing portion of the fiber (i.e. better light confinement). The photodetector voltage will then increase as humidity increases.

Conversely, assuming that the refractive index of PVA increases as humidity increases, the critical angle for TIR within the fiber becomes very shallow, leading to poorer light confinement at the bent portion of the fiber. The photodetector voltage will then decrease as humidity increases.

There are some issues to be resolved before the sensor can be used effectively as a relative humidity sensor for measuring soil humidity. The main problem is that the humidity sensing capability of the sensor tapers off at 90%RH. This is not suitable for measuring soil humidities as the most common humidity range in which soils fall in is from 90%RH to 100%RH. There were some difficulties in coupling light from the LED to the fiber without connectors. In order to get the best coupling, the fiber should be mounted as close to the LED and photodetector as possible,

otherwise there will be discrepancies in the voltage response of the sensor. There is also the problem of bending loss as the sensor's method of sensing is intensity modulation by the humidity sensing film. Hence any bending of the fiber will change the intensity of the light detected. In the experiment, the fiber lengths are kept short so that unnecessary bending is minimized, but if the sensor is to be used in the field, the probe will consist of the sensing portion of the fiber and the rest of the fiber will have to be long in order to reach the data collection unit. Bending the fiber will lead to a change in the recorded intensity that is not due to humidity change. A solution to this problem might be to use multi-wavelength referencing for the sensor, where the sensing film absorbs one wavelength and the second wavelength is not in the absorption range of the film. Hence intensity changes due to fiber bending and not due to humidity changes can be eliminated. If the sensing range of the sensor can be improved by using other humidity sensitive materials as coating for the sensor, the POF RH sensor could prove to be very valuable for soil humidity measurements.

Advantages of using CoCl_2 coated POF sensor for humidity sensing:

- ✓ POFs are cheap and the humidity sensitive coating is readily available and easily applied.
- ✓ Sensor is very durable and the coating does not come off easily.

Disadvantages:

- ✗ The humidity sensing range and the accuracy of the sensor is not sufficient for soil humidity sensing.
- ✗ Sensor is susceptible to bending losses and source fluctuations as it is intensity modulated.

Possible improvements:

- ➡ Find new coating materials that can increase the humidity sensing range.
- ➡ Improve sensor and system design to reduce bending loss and source fluctuation errors.

The humidity sensing range and the accuracy of the POF sensor can be improved by using in-fiber based gratings. As the humidity sensing range of the POF sensor does not extend beyond 90%RH, in the next investigation, an LPG was used as a refractive index sensor to sense the change of index in a film of gelatin, coated around the cladding of the LPG, when humidity of the surroundings changes. This sensor has good sensitivity and range in the humidity range from 90%RH to 100%RH, which makes it very suitable to be used as a fiber optic soil suction sensor.

5.7 Relative Humidity Sensing Using LPGs

The humidity response of the bare LPG, after it was coated with gelatin, was measured by Tan et al. [86] by placing the sensor into the Feutron climate control chamber. The highest order cladding mode of the 300 μ m LPG, the 7th mode, was recorded on the OSA and the intensity of the resonant dip (dBm) was measured as humidity was changed in the chamber.

Figure 5-22: Spectra change of LPG RH sensor with respect to humidity change (Bare LPG)
[86]

The humidity response of the LPG sensor is due to the properties of the gelatin coating (detailed descriptions of gelatin structure can be found in Section 3.6). At humidities lower than 90%, insufficient amounts of water molecules accumulate within the 3D lattice of polymer chains which constitute gelatin to effect a refractive index change that is significant enough to show a large change in the LPG spectra. Above humidity levels of 90%, however, due to the higher concentration of water vapour in the air, enough water diffuses into the gelatin coating and changes the refractive index significantly, which the LPG detects and thus the output spectra changes accordingly.

Two observations can be made from Figure 5-22; firstly, the graph shows that as the RH increases, the coupling strength of the highest wavelength attenuation band decreases, resulting in a decrease of LPG resonance dip. However, the most significant response is when the relative humidity is 90%RH and above, making the sensor very useful for high RH sensing. The advantage of measuring the intensity change of the transmission of the LPG instead of the resonant wavelength shift lies in the simplicity of the demodulation system. Wavelength demodulation systems are significantly more complex than intensity demodulation systems which just consist of a light source, the sensor and a photodetector to detect intensity changes.

In the second observation, a small shift in the resonance wavelength towards the shorter wavelengths or blue shift was noticed. However, the rate of change of wavelength with RH is lower than the rate of change of resonance dip with RH. The changes in the coupling strength and the blue shift of the spectrum can be attributed to the change in the refractive index of the thin gelatin coating as the RH changes. Initial refractive index measurement on liquid gelatin, using a Kyoto Electronics RA-130 refractometer, yields a value of 1.3408. Gelatin was later spin-coated on a glass plate and cured under room temperature for 24 h. A measurement of the new refractive index of the coating (using Metricon 2010 Prism Coupler) yielded a refractive index of 1.474, with a coating thickness of 6.836 μm . As the refractive index of pure solid gelatin is close to 1.5, it is obvious that liquid gelatin, having high water content, will have a much lower refractive index. A thickness of 6.836

μm presents an infinite layer on the LPG and despite the ambient surrounding (air) having a refractive index of 1, the average refractive index would be close to 1.474. From photos taken using a scanning electron microscope (SEM), it was estimated that the thickness of the gelatin coating on the LPG is approximately 297.5 nm [86].

Measurements of the resonance wavelength of the gelatin coated LPG RH sensor taken, before the coating was applied and after the coating was applied and cured, yield a blue shift of 6.486 nm. Having a 297 nm thick coating of gelatin on the LPG and a blue shift of 6.486 nm in the resonance wavelength, shows that the average refractive index of the gelatin coating is less than the measured 1.474 and is also less than that of cladding. In addition, it was suspected that the average refractive index of the gelatin coating is close to the refractive index of the cladding region. This is due to the big change in the resonance dip of the highest wavelength attenuation band as the average refractive index of the gelatin coating changes with RH. For any cases of infinite and finite layer of external medium covering the LPG, when the refractive index of the external medium (higher than that of cladding) approaches that of cladding, the cladding modes disappear as the cladding losses its guiding characteristics [74],[75],[77],[78].

An increase in RH increases the water content in the gelatin coating resulting in a reduction in density, and thus a reduction in refractive index. From Equation 20, with the decrease in average refractive index of the gelatin coating and hence $n_{eff}^{clad,i}$, the resonance wavelength would shift towards the longer wavelength side. In addition, if the initial average refractive index is close to that of cladding, the reduction in average refractive index should not cause any major changes in the resonance dip [78]. In Figure 5-22, the response is in opposite trend to what is expected. The average refractive index of the gelatin-coating seems to increase with RH as shown by a blue shift of the resonance wavelength and a reduction in the resonance dip. It is suspected that due to swelling of the gelatin coating, the LPG may have experienced tension, in which is attributed from both material and waveguide contributions. Thus, this results in changes in its grating period, UV-induced index modulation in the fiber core and the differential effective index [76].

This will have an effect on the resonance wavelength and the coupling strength, which may be greater than the effect of pure refractive index decrease in the gelatin coating. Further experimental investigations of this phenomenon are ongoing.

Figure 5-23: Detailed response of the LPG RH sensor in the high humidity region (Bare LPG)
[86]

Some investigations were made by Tan et al. [86] on the various characteristics of the LPG RH sensor like sensitivity, resolution, hysteresis, repeatability and stability. The sensor behaves linearly with humidity in the range tested (Figure 5-23) and from the data obtained; the sensitivity of the sensor was found to be 0.833%RH/dBm. During the test, the sensor's response was observed to change within seconds when humidity was changed. The resolution of the sensor is limited by the measurement system, in this case the OSA. The OSA can measure optical intensity to a minimum step of 0.01dB with an error of ± 0.3 dB. With this, the LPG RH sensor resolution and accuracy was calculated to be $\pm 0.008\%$ RH and $\pm 0.25\%$ RH respectively [86].

Figure 5-24: The gelatin-coated LPG RH sensor was subject to three cycles of variation of humidity to determine the repeatability, repeatability error and hysteresis error [86].

During the repeatability test (Figure 5-24), the LPG RH sensor was subjected to three cycles of humidity up ramp and three cycles of down ramp. The hysteresis error was found to be $\pm 0.203\%RH$. In comparison, the typical hysteresis error of commercial relative humidity sensors is rated at $\pm 1\%RH$, which makes the LPG five times more accurate [86]. The LPG RH sensor exhibits a repeatability of 99.71%, which corresponds to a repeatability error of $\pm 0.877\%RH$. More accurate measurements of relative humidity are not possible as the size of the humidity chamber presents some problems like the humidity fluctuations and non-homogenous humidity distribution. Hence the LPG RH sensor could achieve a repeatability error of less than $\pm 0.877\%RH$ if a more accurate humidity calibration chamber is used [86]. The stability error (long term measurements) is less than $0.04\%RH$, which was limited by the stability of the light source [86].

Figure 5-25: Effect of different periods on the response of the LPG RH sensor (Bare LPGs)
[87]

Figure 5-25 shows the response of three LPGs with different periodicities of 300µm, 450µm and 550µm. The LPGs with longer periods have fewer cladding modes and these guided cladding modes are confined closer to the core when propagating in the cladding. This limits the interaction of the highest order cladding mode with the external medium. For LPGs with shorter periods, the number of guided cladding modes increase and the highest order mode propagates near the cladding/external medium interface; hence the interaction with the surroundings is much higher. As a result, the 300µm LPG is much more sensitive to changes in refractive index of the external medium than the other LPGs [87].

From the results obtained, the gelatin coated LPG RH sensor provides a promising high RH sensor with good resolution, low hysteresis, good repeatability and stability. Combined with the advantages of optical fiber, the gelatin coated LPG RH sensor is well suited to perform accurately in extremely humid environments like soil.

Calibrations for soil tests:

Using the Feutron climate control chamber, an accurate calibration of the packaged LPG RH sensor was attempted using the average of 3 cycles of humidity ramping. The calibration is to be used to analyze the intensity values obtained from the soil tests and convert them to humidity values. The calibration curve is shown in Figure 5-26.

Figure 5-26: Linear Fit of data obtained for humidity calibration using Feutron climate control chamber (Packaged LPG)

Another calibration of the LPG RH sensor was attempted using salt solutions. It was found later that the calibration results conducted in the climate control chamber were not accurate due to the reason that humidities above 98%RH are not reliable, according to the specifications of the oven. The soil tests were conducted after the first calibration was completed and the results obtained from the soil tests were found to be out of the range of the calibration curve. Thus the calibration using salt solutions was initiated but could not be completed before the sensor failed. This method is better than using the humidity chamber as salt solutions enable fixed and stable humidity levels to be achieved in a small container. The humidity chamber is very large; hence maintaining an accurate and stable humidity level is not possible. Using salt solutions, on the other hand, is a tedious process of calibration as only

one prepared solution produces one humidity value. And the solution has to be left overnight with the sensor in a container for the humidity to achieve equilibrium.

While conducting the calibration of the sensor using salt solutions, it was noticed that the response of the sensor was different from results obtained previously. After 4 readings were taken, the calibration was stopped. Table 5-5 shows the planned and actual readings taken from the calibration using salt solutions.

Table 5-5: High humidity calibration of LPG RH sensor using salt solutions

Calculated %RH	Equilibrium LPG Intensity (Dip) /dBm	%RH Measured by Aqualab
100%	-14.71	99.9%RH @ 25.4°C
99.50%	-15	99.3%RH @ 25.4°C
99%	NA	NA
98.50%	NA	NA
98%	NA	NA
97%	NA	NA
96%	NA	NA
95%	-25.16	95.4%RH @ 25.4°C
94%	NA	NA
93%	NA	NA
92%	NA	NA
91%	NA	NA
90%	-39.34	89.9%RH @ 25.2°C

The result of the calibration is shown in Figure 5-27. Only the calibration conducted using salt solutions can be used, as it is more accurate but it is an approximation of the real response due to a lack of more data points.

Figure 5-27: High humidity calibration of LPG RH sensor using salt solutions

A comparison was made between the responses of the bare LPG

1. When it was first fabricated,
2. After the LPG was packaged in a bronze tube, and
3. After some time when sensor began to fail.

The tests were all conducted in the climate control chamber. The result is shown in Figure 5-28. The LPG sensor underwent many tests at high humidity for sustained periods of time, including characterization, calibration and soil testing. The gelatin film could be saturated with water that remains in the structure, hence the changing response of the sensor.

Figure 5-28: Comparison of LPG sensor response under functioning and failing conditions

Advantages of the LPG RH sensor:

- ✓ Condensation at humidities near 100%RH does not affect it.
- ✓ Relatively temperature insensitive.
- ✓ Gives results that are repeatable, stable.
- ✓ The LPG itself has a very long life span, but the film is the limiting factor.
- ✓ After packaging, the sensor is very durable and resistant to rough handling.

Disadvantages of the LPG RH sensor:

- ✗ Gelatin is an edible protein; hence it can get moldy.
- ✗ Gelatin can get saturated as well, hence after heavy usage in very humid environments, the sensor will start to fail.
- ✗ The fiber is sensitive to bending loss.
- ✗ The LPG is double-ended; there is a need to develop a single-ended probe.

Possible improvements:

- ➡ Develop other humidity sensitive coatings which improve upon the properties of gelatin.
- ➡ Improve the sensor and system to achieve better humidity sensitivity and accuracy.
- ➡ Achieve single-ended capability for the LPG, so that making a soil suction probe is more feasible.

The soil tests are conducted next, using prepared soil specimens of fixed matric suction values. The LPG RH sensor was used with the soil specimens to find out their relative humidity and the results are compared with the values taken using other equipment.

5.8 Soil Test Results

Figure 5-29: Soil water characteristic curve for kaolin clay [89]

Kaolin clay is mainly made up of the mineral kaolinite. The theoretical soil water characteristic curve of kaolin is shown in Figure 5-29. As the matric suction of the soil decreases, the water content of the soil increases quickly, until a maximum

limit. This limit is the maximum of water that the soil can hold at a saturated condition.

Table 5-6: Soil Test Results

Soil ID	Matric Suction /kPa	Gravi-metric Water Content /%	Calculated %RH based on matric suction	Measured %RH using Aqualab	LPG Dip Intensity /dBm	Calculated %RH from LPG intensity
SP1-15 SP1-16	58.6	21.55	99.96%RH @ 25°C	99.8%RH @ 25.4°C	-15.02	99.52
SP1-03 SP1-06	84.07	17.33	99.94%RH @ 25°C	99.8%RH @ 25.4°C	-15.26	99.42
SP1-13 SP1-14	122.85	17.82	99.91%RH @ 25°C	99.8%RH @ 25.4°C	-14.84	99.59
SP1-17 SP1-18	207.14	14.45	99.84%RH @ 25°C	99.4%RH @ 25.5°C	-15.06	99.50
SP1-9 SP1-12	386.22	9.32	99.72%RH @ 25°C	99.4%RH @ 25.4°C	-15.02	99.52

Table 5-6 shows the tabulated results from the tests conducted on the soil specimens. The theoretical values of %RH based on values of matric suction were calculated from Equation 2. After equilibration at a fixed matric suction value using a pressure plate, each specimen is tested for:

1. Matric suction value using axis-translation technique, to check the actual equilibrated matric suction value of the specimens.
2. Gravimetric water content using oven drying to measure the weight loss of the soil specimens.
3. Relative humidity using the Aqualab chilled mirror hygrometer, so that the humidity values can be compared with those taken by the LPG RH sensor.
4. Relative humidity calculated from intensity values using LPG RH sensor.

The difference between theoretical relative humidity of soil specimens with matric suctions of 58.6kPa and 386.22kPa is just 0.24%RH. Soils with matric suctions of 500kPa and below are the most commonly found in the field. Ideally, a humidity sensor that has a resolution of approximately 0.01%RH would be able to measure ± 50 kPa of suction in the field. However, some of the most accurate commercial electrical RH sensors have a resolution and accuracy of $\pm 0.03\%$ RH and $\pm 2\%$ RH

respectively [90]. The Aqualab, which uses the chilled mirror hygrometer technique for measurement of humidity, has a resolution of 0.1%RH but an accuracy of $\pm 0.3\%RH$, which corresponds to a suction accuracy of $\pm 400kPa$. The LPG RH sensor has a resolution and accuracy of $\pm 0.008\%RH$ and $\pm 0.25\%RH$ respectively. The LPG RH sensor has a suction accuracy of about $\pm 350kPa$, an extremely large value. Though the LPG sensor has a better resolution and accuracy than the Aqualab hygrometer, the LPG RH sensor is still not able to distinguish between the humidity values of the soil specimens. There could be other factors that is limiting the accuracy of the sensor and has to be looked into for further investigations.

Figure 5-30: Comparison between relative humidity measurements of different devices for soils of different matric suction values

Figure 5-30 illustrates the responses of the LPG RH sensor and the Aqualab with respect to matric suction. A theoretical line is included in the graph for comparison (Data from Table 5-6). The closer the device response comes to this line, the better the device is for measuring soil suction.

Advantages of the LPG RH sensor as a soil suction sensor:

- ✓ For an intensity demodulated fiber optic sensor, it is very sensitive and yet easy to demodulate by just using a photodetector.

Disadvantages of the LPG RH sensor as a soil suction sensor:

- ✗ As the RH change in soil is very small (0.36%RH) as the suction in soil varies from 0kPa to 500kPa, the resolution of the sensor must be very good ($\approx 0.01\%RH$) and the error must be small ($\approx \pm 0.03\%RH$). This is very stringent specifications for a RH sensor as there are none developed today that meets such specifications.
- ✗ Sensitivity to system fluctuations like source fluctuations or bending loss increases the error of the readings of the sensor drastically. This is an inherent problem in intensity-modulated systems. Some compensation techniques can be employed to reduce source fluctuation and bending loss errors.
- ✗ The LPG sensor is a double-ended probe, where one end of the LPG is connected to the light source and the end is connected to the measurement device. To be used effectively in soil, a single-ended probe where only one end of the fiber is connected is required.
- ✗ Short lifetime of the gelatin coating limits the reusability of the sensor.

5.9 Possible Improvements

In the investigations of this project, both frequency modulated (FBG sensors and LPG sensor for hydrostatic pressure measurements) and intensity modulated (the rest) sensing techniques were employed. Intensity sensing techniques are simple in concept and the challenge was to make a very sensitive sensor while avoiding the problems inherent in this technique. There were many techniques that were tested but did not work due to some inherent problems. However, the groundwork was laid for other methods that worked. The LPG RH sensor developed last has the best resolution and sensitivity when compared with the POF RH sensor. However, intensity fluctuations not due to the measurand is still a problem.

In order to achieve high sensitivity, it is necessary to utilize the technique of fiber optic interferometry. Interferometric methods, like the 3D shape measurement method described previously, are very sensitive and have high resolution because the sensing technique is based on the interference of light between the sensing and reference arm of the device. The difficulty is to minimize the bulky equipment that is needed and also the difficulty in demodulating of the signal. An in-fiber Mach-Zehnder interferometer has been demonstrated by Duhem et al. [91] using 2 LPGs and a single-ended in-fiber LPG Michelson interferometer has been developed by Swart [92] (Figure 5-31).

Figure 5-31: The LPG Michelson sensor. (a)System configuration. (b)Details of the optical paths in the fiber [92]

The implication of the LPG Michelson interferometer is that only one end of the LPG needs to be connected. The other end is silvered and acts as a mirror. This is perfect for a single-ended probe. Also, the in-fiber interferometer formed by the LPG enables it to have an excellent sensitivity to external refractive index changes. With an LPG interferometric sensor that is very sensitive to external refractive index changes, there are two possibilities. The first possibility is to use such a sensor to create an even more sensitive LPG RH sensor as the interferometric sensor is much more sensitive to external refractive index than a single LPG. The second possibility is more interesting, where it may be possible to use the sensor to measure refractive index change of water due to hydrostatic pressure change where a single LPG was not feasible (Section 5.2). It is better to use the interferometric LPG sensor to measure directly the pore-water pressure in soils, which requires very sensitive response to extremely small refractive index changes in the medium ($\approx 10^{-7}$), than for humidity sensing as the sensor will need to have more parts like the gelatin coating thus making it more susceptible to failure.

Chapter 6 Conclusions and Recommendations

6.1 Conclusions

Several fiber optic sensing techniques have been explored for soil suction measurements. The research approach was split into two main categories: direct measurements and indirect measurements of soil suction. Firstly, for the direct measurements of pore-water pressure in soil, three different techniques were considered. First of all was the 3D shape measurement of menisci of water capillaries in soil to calculate the matric suction. This method is not feasible as 3D shape measurement techniques are complex and cannot be used in the field to measure meniscus of water that can be as small as $1\mu\text{m}$ across. Also, averaging the matric suction values obtained across the whole soil is not accurate. The second method that was investigated was the use of embedded FBG sensors to measure hydrostatic pressure. The minimum length achievable for the FBG was 5mm and this limits the size of the diaphragm as the carbon fiber composite has to be longer than this length to allow enough space for the diaphragm to bend. Since the carbon fiber composite diaphragm is large, cavitation of water when under negative pressures becomes certain. The diaphragm mount needs to be secured on four sides with epoxy for the diaphragm to be attached for maintaining an air-tight compartment. This causes birefringent effect in the fiber when the pressure is high, and this limits the sensing range. The third method investigated was the use of an LPG to measure the refractive index change of water when hydrostatic pressure was applied. The sensor showed no response to the change of refractive index of water mainly because the change of index is so small that it is very much below the sensing capability of a single LPG.

The investigations moved on to indirect measurements of matric suction using relative humidity of soil. Five methods were investigated in this category. Firstly, measurement of relative humidity using Fresnel reflection from the tip of a cleaved SMF was attempted. The modulation of light in the fiber due to refractive index change of air (when humidity changes) is too small to be detected. The photodetector simply is not able to detect optical power changes of 10^{-12}W per

1%RH change. But when the fiber tip was placed into the salt solutions that were used for humidity calibration, it was found that the sensor was able to distinguish the osmotic suction of the solutions as the refractive index changes in liquids is larger as concentration/composition of the solutions changes. This sensor can be further tested in soil as an osmotic suction sensor. The second method was an attempt to increase the modulation of light at the fiber tip using gold tip-coatings. As the moisture diffuses into the gold film at the fiber tip, the conductivity hence the reflectivity of the film changes with humidity. The modulation of light in the fiber, as found in the experiments, was still not enough to produce a repeatable response to humidity change. The third method uses the absorption spectrum of water in an attempt to determine the relative humidity of air. Using absorption spectroscopy, one absorption line of water at 1392.530nm was chosen for monitoring. Using a white light source, a pair of fiber collimators to form an absorption cell of short length and an OSA, the waveform of the absorption line was captured. Unfortunately the waveform was noisy due to the low source power, but Wavelet Transform was used to filter the noise away. Several humidity tests were conducted but the waveforms were too noisy and the filtered approximations were not consistent, thus leading to no discernable trend for the technique after humidity testing. The fourth method investigated was a POF humidity sensor, coated with CoCl_2 and gelatin as the overlay material. The sensor worked well and had a linear humidity response range of 65%RH to 85%RH. The resolution of the sensor is about 0.01%RH and it has an accuracy of about $\pm 0.67\%$ RH. The hysteresis error is negligible. Both bending loss and light coupling is an issue for this sensor due to intensity modulation and large fiber diameter. The sensing range of the POF RH sensor is not good enough for soil suction measurements as most soils have humidity of 90%RH to 100%RH. Hence the fifth and last method investigated was the use of a gelatin coated LPG sensor. The LPG sensor has a humidity response from 90%RH to 100%RH with a resolution and accuracy of $\pm 0.008\%$ RH and $\pm 0.25\%$ RH respectively. Some calibrations were done on the sensor to prepare for soil testing.

In the soil tests, soil specimens were made from kaolin clay and compacted into cylindrical shapes. The specimens were saturated at different matric suctions (50kPa to 400kPa) using the pressure plate technique. During testing, the soil specimens were tested using the null-type equipment, the Aqualab hygrometer, the LPG RH sensor and finally the gravimetric water content test. The LPG sensor did not respond to the soil specimens as the difference in humidity between soils of 50kPa and 400kPa is just 0.24%RH. The accuracy of $\pm 0.25\%$ RH for the sensor, due to bending loss and system limitations, is equivalent to a suction accuracy of about ± 350 kPa, which is the reason why the LPG RH sensor was not able to distinguish between the humidity values of the soil specimens.

In conclusion, an osmotic suction sensor using cleaved SMF (measuring the changes of refractive index in salt solutions) and a total suction sensor using LPGs (measuring the RH of the soil) has been developed and investigated successfully in this project.

For future work, a potential area to investigate is using LPG interferometric sensors for pore-water pressure measurements. The external refractive index sensitivity can be increased beyond that of a single LPG and that would increase their effectiveness in measuring small changes of refractive index due to hydrostatic pressure changes in soil water.

6.2 Recommendations

There are certain limitations that need to be addressed before the various techniques can be used for soil suction measurements. Several suggestions and recommendations for future study are discussed below.

➤ **For the measurement of the refractive index change of water due to hydrostatic pressure change using LPGs:**

1. Direct measurement of pore-water pressure is the most desired measurement but unfortunately it is very difficult to achieve. In this project, a single LPG was used to measure the refractive index change of water, but the change was below the LPG's sensing threshold. Using a LPG based interferometer might solve the problem of poor sensitivity. The LPG interferometer has been shown to have superior refractive index sensitivity as compared with single LPGs due to its structure. Having a fiber sensor, without any coatings, which can measure hydrostatic pressure change by the refractive index change that results in the water, is very desirable. Therefore, a single-ended fiber optic soil suction probe, using the LPG Michelson interferometric technique, that can be used in soil to measure pore-water pressure directly can be developed.

➤ **For the measurement of osmotic suction using Fresnel reflection using cleaved SMF:**

1. Though this sensor is not capable of measuring relative humidity in air due to extremely small variations of refractive index, it measures the difference in concentration of salt solutions well because the refractive index change in liquids is larger than gases. The sensor was shown to respond when to different osmotic suctions of salt solutions. Soil testing can be conducted with pore-water squeezed from soils using the fiber optic sensor to find out if the measured osmotic suction corresponds to the actual value.

➤ **For relative humidity measurements using absorption spectroscopy:**

1. The absorption spectroscopic setup used in this project was simple but had two limitations. The light source used is very broadband (400nm to 1800nm), but the output power is very weak. This leads to noisy recorded spectra. Since only one absorption line need to be monitored, a broadband source is not needed. But the NIR absorption lines of water are situated in a part of the spectrum where light sources are not common, hence broadband sources may need to be used, but with high optical power.
2. The absorption cell length needs to be longer. A special design is needed for the absorption path to be longer and minimize the optical loss. A special fiber probe may need to be built for this purpose.

➤ **For relative humidity sensing using POF RH sensor:**

1. Chemical sensors using bulk material are always dependent on the material for the response of the sensor. Likewise in the POF RH sensor, the CoCl_2 film limits the sensing range of the sensor as it has its own characteristics under different humidity conditions. If a better humidity sensing film is found, the response of the sensor will naturally improve.
2. The POF RH sensor is susceptible to bending loss (being intensity modulated) and light coupling into fiber problems due to its large fiber diameter. System improvement can help to minimize such issues.

➤ **For relative humidity sensing using LPG RH Sensor:**

1. As the LPG operates in transmission mode, the two ends of the fiber must be connected. This poses a problem for probe design as inserting a probe with two ends connected into the ground is not feasible. To enable single-ended probe functionality, it is possible to have an LPG with just one end connected. It was shown by Swart [92], by cleaving one end of the LPG and silvering that end to promote reflectivity.

2. Gelatin's good points are that it is reversibly affected by humidity and is easy to apply. However, it can be adversely affected by bulk water and mould. The degradation of sensor response after several months of intense use. Chemical sensors using bulk material is usually limited by the material. A better coating material that can improve the sensing range and the resolution of the sensor. Porous Sol-gel is a possible coating material that can be developed further.
3. Improved packaging for the sensor is necessary if the sensor is to be inserted into soil to take readings. Only moisture should be allowed to pass and neither soil particles nor liquids should come into contact with the sensor.
4. Bending loss in the fiber is a major issue as the sensor is intensity modulated. Any change in the light intensity within the fiber that is not due to the measurand affects the readings and this causes errors. A system improvement can reduce such problems.
5. The temperature sensitivity of the sensor should be reduced if the sensor is to work under field conditions. As temperature in the field fluctuates quite a bit, the sensor itself should either be insensitive to temperature changes or the system should have temperature compensation in-built.

Author's Publications

Journal Papers:

- (1) C.M. Tay, K.M. Tan, S.C. Tjin, C.C. Chan, N.Q. Ngo and X.Y. Dong, "A High-Resolution Tunable Fiber Bragg Grating Filter", *Microwave and Optical Technology Letters*, vol. 42(2), (2004), pg. 89-92.
- (2) C.M. Tay, K.M. Tan, S.C. Tjin, C.C. Chan, H. Rahardjo, "Humidity sensing using plastic optical fibers", *Microwave and Optical Technology Letters*, vol.43(5), (2004), pg. 387-390.
- (3) K.M. Tan, C.M. Tay, S.C. Tjin, C.C. Chan, H. Rahardjo, "High relative humidity measurements using gelatin coated long-period grating sensors", *Sensors and Actuators B*, available online 2 March 2005.

Conference Papers:

- (1) C.M. Tay, K.M. Tan, S.C. Tjin, C.C. Chan, N.Q. Ngo and X.Y. Dong, "High-Resolution Tunable Fiber Bragg Grating Filter", *Proceedings of SPIE*, vol. 5590, (2004), pg. 128-134.
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