LABORATORY EQUIPMENT TYPE FIBER OPTIC REFRACTOMETER

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Abstract

Using fiber optics and micro optics technologies we designed innovative fiber optic index of refraction transducer that has unique properties. On the base of this transducer a laboratory equipment type fiber optic refractometer was developed for liquid index of refraction measurements. Such refractometer may be used for medical, pharmaceutical, industrial fluid, petrochemical, plastic, food, and beverage industry applications. For example, it may be used for measuring the concentrations of aqueous solutions: as the concentration or density of a solute increase, the refractive index increases proportionately. The paper describes development work related to design of laboratory type fiber optic refractometer and describes experiments to evaluation of its basic properties.

Keywords

Refractometers, index of refraction measurement, fiber optic index of refraction transducer

1. Introduction

Almost all currently available classical refractometers employ a prismatic element on which the liquid sample is placed [1], [2]. These instruments yield an output that is based on the degree of a light beam at the liquid-prism interface. In the simplest refractometers, the index is determined visually, via a linseed viewing tube, by observing where the output beam intersects a graduated scale. In more modern, digital type instruments, the degree of bending is measured automatically using a linear photo detector array [1], [4].

Using fiber optic technology and micro optics it is possible to design the innovative fiber optic index of refraction transducer that has unique properties [3]. Basically it consists of input - output pair of simple multimode fibers that interrogate a small lens. The cone of light injected into the lens from the input fiber is internally reflected from inside surface of the lens and focused back into the output fiber. When the outer surface of the lens is in contact with a liquid, the attenuation of the light reaching the output fiber depends strongly on refractive index of liquid. Depending on the construction and design of such a transducer, this transducer may have a wide dynamic range. Its relative sensitivity, i.e. the ratio of fractional change of the optical intensity for a given change in index of refraction, is substantial, (of the order of 5 to 10) over a wide range of index, e.g. n = 1.3 to 1.6.

Using one of many commercially available bench type and hand held index of refraction instruments, or refractometers, a few drops of the liquid under test must be withdrawn and placed on a planar measuring surface, while using one of few in-line refractive index monitors that are currently available requires a complex installation, including two or more relatively large ports and/or flow bypass tubes.

The new fiber optic small, probe type structure, transducer elements, can be easily inserted into the top of liquid containers or through a simple fitting in a flow line. It is essential to combine very practical and versatile transducers capable of detecting small changes of index of refraction with "smart" data acquisition and signal processing technology to develop new practical instruments.

The paper describes development work of a flexible fiber optic refractometer and its experimental evaluation. Two types of fiber optic refractometers are considered: a basic fiber optic refractometer (based on the use of one fiber optic transducer) and a most sophisticate differential fiber optic refractometer (based on the use of two fiber optic transducers). Both types of refractometers are developed as laboratory equipments and tested in experiments in laboratory and field conditions.

2. Architecture of Fiber Optic Refractometer

On the base of combination of fiber optics and micro optic technologies a new small, flexible and probe type index of refraction transducer element was fabricated [3] and for practical (industrial) application packaged into a transducer module (Fig. 1) with light source, photo detector and thermistor based temperature sensor element.

Depending on the construction and design this fiber optic transducer module may have high relative sensitivity (of the order of 5 to 10) over a wide range of index of refraction, e.g. n = 1.3 to 1.6 and can be used to measure at measurement points placed at relatively large distance (up to 1.5 km).

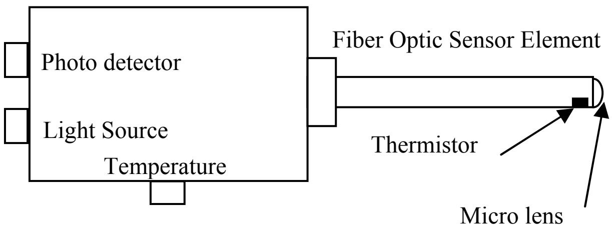

Fig. 1 Fiber optic transducer module

The basic transducer properties are illustrated on Fig. 2 and 3, which show linear and semi-logarithmic plots, respectively, of the optical power output of a typical transducer as a function of index of refraction. For convenience, usually employed micro-lenses have diameter from 5 to 1 mm. In principle, it should be possible to go to even smaller diameters, e.g. 250 to 300 microns - so that relatively small catheter type transducers are feasible. Since the basic design is quite simple it also is possible to produce extremely rugged transducer elements that are suitable for field/industrial applications, even for remote sensing.

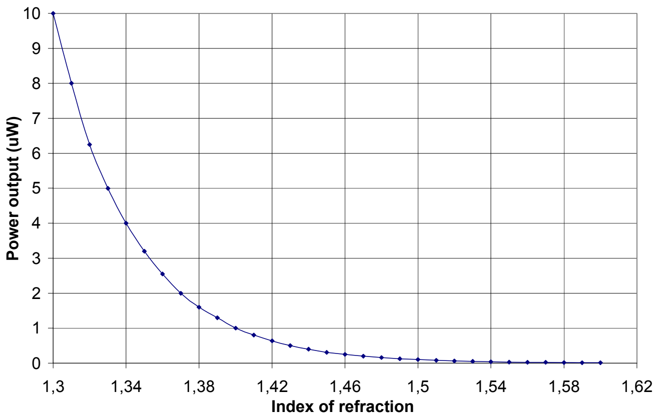

Fig. 2 Linear plots of the transducer output vs. refractive index

Since the index of refraction is strongly dependent on temperature and also, to a lesser degree, on wavelength, these effects must be corrected for in designing and/or using such instruments. They are of major importance when one attempts to determine an index of refraction to 1 part in 10.000 or better, which is the desired sensitivity for high quality laboratory type instrument. Their importance can be seen in the case of water, for example, which has temperature coefficient $\Delta n/\Delta T$ of 1.5 parts in 10.000 per °C.

As with any other refractometer, any instrument that employs this fiber optic based transducer module must be capable of correcting for the intrinsic temperature dependence of a liquid's index of refraction [2], [3], [4]. In addition, however, for an intensity type fiber optic sensor, other corrections and precautions must be taken especially if, as already mentioned, a precision of 1 part in 10.000 is to be attained. It will be necessary to employ low noise electronic circuitry and/or correct for photo detector dark current, especially at higher indices, where the output light intensity is strongly attenuated. It also will be necessary to correct for light source and photo detector temperature sensitivities and for any stray light that might affect the photo detector. In one sense, in terms of capabilities of today's microprocessor controlled "smart" sensor technology, it should be straightforward to design instruments that automatically "massage" the raw transducer data to correct them for each of these effects [2], [3], [4].

Several architectures for designing optical refractometer equipment are possible:

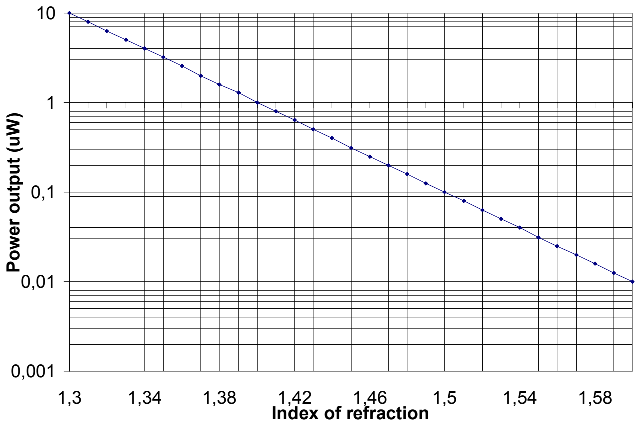

Fig. 3 Semi-logarithmic plots of the transducer output vs. refractive index.

One type of the basic equipment architecture proposed is to produce computer (PC) controlled laboratory instrument capable of determining the index of refraction of various liquids to 1 part 10.000 or better. Another type is to produce microprocessor controlled, handheld and other similar types of small, portable refractometers, probably of somewhat reduced accuracy that can easily be used for field type measurements. A third type is to produce in-line refractive index monitors for use in chemical, industrial, food processing, and other similar facilities. Refractometers are frequently used for medical, pharmaceutical, industrial fluid, chemical, petrochemical, plastic, food, and beverage industry applications. For example, they are used for measuring the concentrations of aqueous solutions: as the concentration or density of a solute increase, the refractive index increases proportionately. Included in such measurements is the percentage of sugar in fruits, soft drinks, canned syrups and other solutions. They also are used to determine salinity of aquariums and of solutions used in food processing, freezing point of coolants and deicing fluids, the charge status of acid batteries, the serum protein and urine specific gravity, etc.

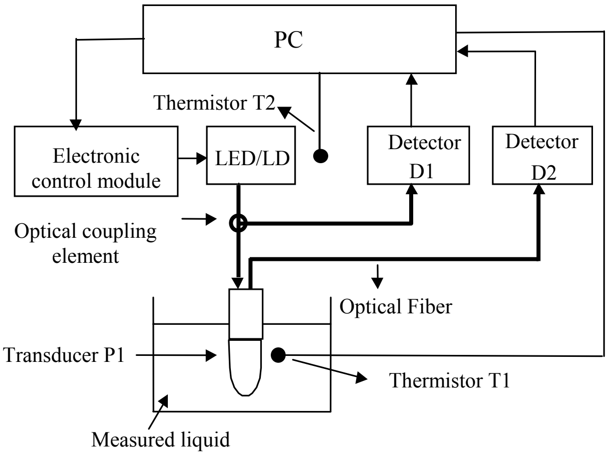

Fig. 4 Block diagram outlining the design of a basic fiber optic refractometer.

In the following part of this paper we consider the design of laboratory type equipment. Referring to the block diagram on Fig. 4, the basic architecture, as presently conceived, consists of the following elements: a light emitting diode (LED) or semiconductor laser diode (LD) light source and its electronic driver/pulser circuit; a monitor photo detector to determine the output light level of the used light source (D1), a second photo detector (D2) to record the return light from the fiber optic transducer module (P1); and a PC to automatically control the system and process the data from the various elements. PC will compute the ratio of the intensity of the transducer output for an unknown liquid and that recorded earlier for a standard liquid, e.g., water.

The PC will then determine the index of refraction, either by a comparison and interpolation process between this ratio and those in a calibration data lookup table, or by computation using a transducer response equation. In addition, the temperature of the liquid sample and of the source/detector module will be measured simultaneously with the index of refraction, to correct for their temperature dependence. The liquid sample temperature will be determined using a thermistor, as indicated in Fig. 4, or using a fiber optic temperature sensor in applications requiring an all dielectric transducer, e.g., for use in explosive or high voltage environments [5].

A second possible architecture of the equipment system, as outlined in Fig. 5, was also developed. Basically, it employs a differential technique and would allow measurements/comparisons of index of refraction to a very high precision. Instead of taking comparative readings of index of refraction of a known and an unknown liquid at separate time, as would be done with the architecture outlined in Fig. 4, both readings would be taken simultaneously, using two index of refraction transducers (P1 and P2), one in the unknown and the other in a standard liquid sample.

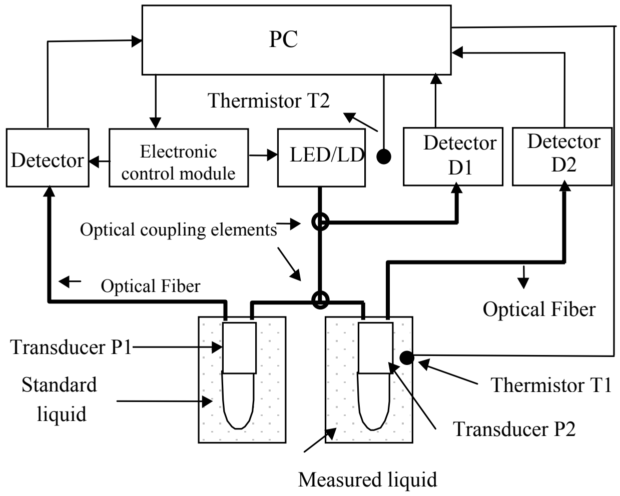

Fig. 5 Block diagram outlining the design of a differential type fiber optic refractometer

This technique would be especially useful when extreme relative accuracy/precision is required. In analyzing the various computer (PC) controlled operations required to carry out each of the above described procedures individually, they all appear quite straightforward in terms of available sensing and signal processing technology. However, effects associated with their use in multiple combinations, and the ultimate precision achievable under real world conditions, must be examined experimentally. The objectives of the proposed laboratory fiber optic sensor equipment will be to design and assemble working models of several different computer (PC) controlled index of refraction instruments. These will then be thoroughly tested under both laboratory and typical field conditions to determine the ultimate precision and accuracy achievable with various designs.

3. Laboratory Equipment Design

The block scheme of the laboratory fiber optic refractometer equipment is shown in Fig. 6. Considering both hardware and software architecture (Fig. 4 and Fig. 5) is conceived such as to enable realization of basic and differential type of fiber optic refractometers. The front panel of the equipment is connected to fiber optic transducer modules (P1 and P2) through BNC type connectors.

There are two types of transducers available: a LED sensor module and a LD sensor module, which can be used with described laboratory refractometer. The LED diode sensor module is based on a pair of LED (HFE4020) as photo emitter and photodiode (CLD42) as photodetector. LD sensor module consists of LD (HFE4080-321) as photo emitter and phototransistor (CD1440) as photo detector.

The analog element contains a driving circuit for the LED and/or LD based transducer, which produces appropriate current for specific type of transducer, respectively. There is a circuit for back measurement of driving current for LED and/or LD based transducer, for more accurate driving/detection rate in the analog element. A detection circuit of photodiode for LED sensor module and/or circuit of phototransistor for LD sensor module, respectively, is responsible for amplifying and adjusting of measured currents within range of ADC (Analog Digital Converter). A detection circuit of thermistor adjusts a voltage from thermistor to ADC range (0-10V).

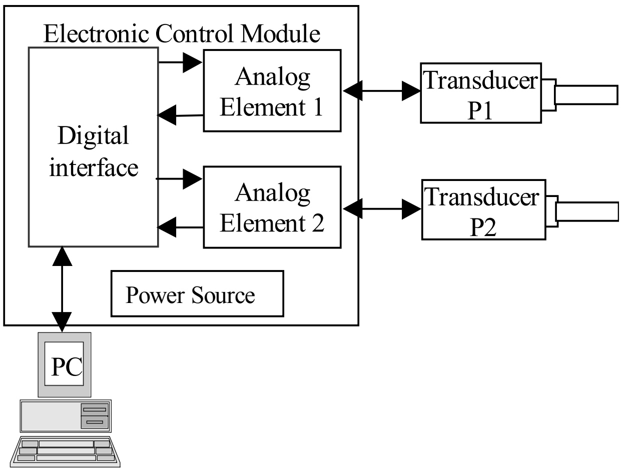

Fig. 6 Block scheme of the laboratory fiber optic refractometer

The described instrument is designed as a laboratory instrument controlled by computer (PC) which is powered from the standard supply network ~230V/50Hz. There are transformers and stabilization circuits for stabilization of necessary voltages (+5V, ±15V) inside the control module.

The interface sensor-computer is developed with the help of the I/O interface chip (MHB8255), Fig. 7. The interface chip is responsible for communication of control module with computer via parallel PC interface. A part of I/O chip controls a 16-bit ADC, starting of conversion and reading out of data to computer. A synchronization register REG1 is used for matching of the different widths of ADC (16-bit) and I/O chip (8-bit). Analog multiplexer 8:1 is addressed and controlled by I/O chip. There are 4 analog channels for each transducer (driving current, LED diode sensor detection, LD diode sensor detection and thermistor data). The I/O interface chip generates a data word for 12-bit DAC (Digital Analog Converter), for driving current of LED and/or LD diode transducer, respectively. The second synchronization register REG2 is required since the data line of I/O chip (8-bit) is different to width of DAC data line (12-bit).

The program package for operation with laboratory refractometer is written in design environment of the LabWindows/CVI version 4.0.1. The program allows performing of refractive index measurements with basic or differential architecture. In the main control window (Fig. 8) we can see the value of the measured refractive index, temperature of the measured liquid and the value of the actual driving current for each measurement. Within a window menu it is possible to change the method of measurement, type of sensor and communication port number. Besides mentioned features it is also feasible to modify driving current (0-100 mA), measure characteristic curve of optical power for connected sensors, get relation of optical output from LED and/or LD sensor module to temperature, respectively. These features can be useful when they are used for experiments with new or unknown sensors.

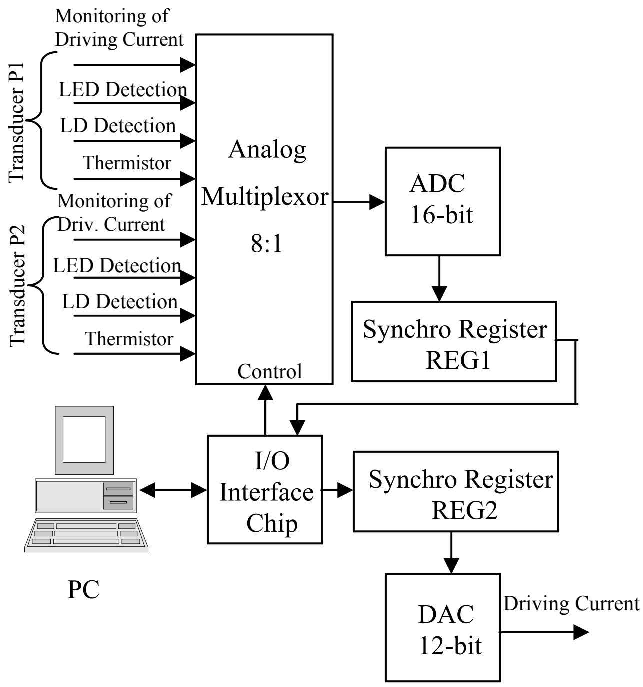

Fig. 7 Block scheme of the digital interface to PC.

4. Experiments and Results

The performance of the developed laboratory fiber optic refractometer equipment was evaluated by various testing measurements; this is demonstrated here by results of two basic laboratory experiments and measurements of index of refraction of various petrochemical products.

A) Basic laboratory experiments:

- Dependency of the refractive index of propylene glycol on temperature (Fig. 9).
- Dependency of the refractive index of water propylene glycol solution on propylene glycol concentration (Fig. 10).

The data on the index of refraction of propylene glycol/water mixture, are good to use since it is simple to clean sensor after dipping in the various solutions, since it is necessary that sensor face be clean and dry to get a good reading in air and also before dipping in water to get

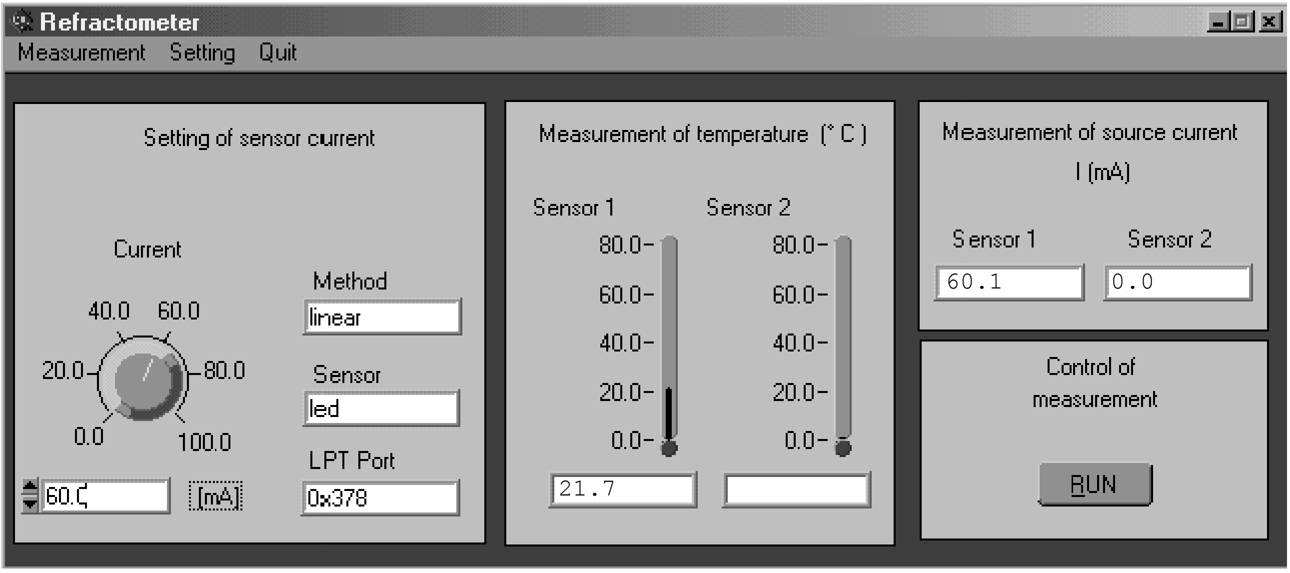

Fig. 8 Main control window of the laboratory refractometer.

a calibration/comparison reading. Even though the index varies with wavelength we take the water reading at 20°C to correspond to an index of 1.3330, and refer all other readings to this value. The dn/dT for water in the range 15 to 30°C is 0.0001 per degree °C, while that for 100% propylene glycol is 0.0003. Thus for glycol/water solutions one could assume a linear dependence of dn/dT, that is, e.g. assume $dn/dT = 0.0002$ for a 50% solution. In all instruments with automatic temperature compensation, it is assumed that they are to be used with water solutions, and it is assumed that $dn/dT = 0.0001$. We may use also propylene glycol, either 100%, taking its index to be 1.4312, or 100% plus a few mixtures.

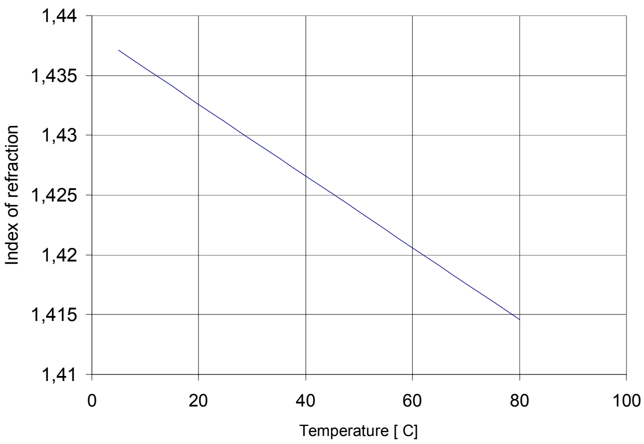

Fig. 9 Refractive index of propylene glycol vs. temperature (°C)

B) Measurements of petrochemical products of the developed equipment

As field tests we use some petrochemical products of known index of refraction. They make it difficult to clean the sensor face however. We recommend keeping the containers of mixtures sealed when not in use since they tend to drift in index, though this is not much of a problem. We frequently make up relatively large sample, e.g., 100 to 200 cc, and then use one half for our measurements for while and after a week or two, take a reading in the other half that has been kept well sealed. That way we can determine if there has been any shift in the index of our measurement sample. The results of measured index of refraction are depicted on Tab. 1, and are in very good success as compared with results obtained by classical methods.

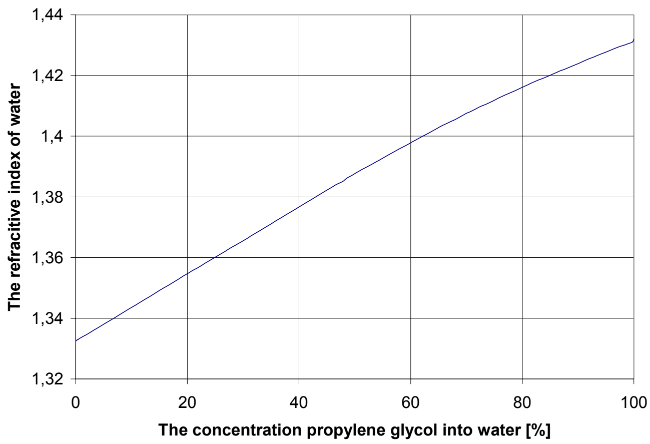

Fig. 10 The refractive index of water vs. propylene glycol concentration.

5. Conclusion

During the development work a PC controlled laboratory type fiber optic refractometer for liquid index of refraction measurements was constructed. Both hardware and software were conceived to allow for realization of basic and differential type of proposed fiber optic refractometer architectures. Basic properties of the equipment were evaluated in laboratory and field type experiments with measurements of standard and petrochemical product liquids.

Petrochemical products	Refractive index	Temperature (°C)
Water	1.3333	21
Synthetic alcohol	1.362	21
Propylen glycol	1.4268	21
Mobil VS-200	1.4399	21
Mobil motor 5W-50	1.4678	21
Oil drive	1.4757	21
Madit drive	1.4828	21

Tab. 1 Result of petrochemical products

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