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AN ON-LINE PROCESS FIBER OPTIC REFRACTOMETER FOR MEASURING EDIBLE OIL HYDROGENATION

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ABSTRACT

The process of edible oil partial hydrogenation has improved steadily over the past decades, but few on-line process instruments exist capable of measuring the extent of hydrogenation. This work describes the design of a prototype, on-line fiber optic refractometer for controlling and monitoring of oils. It uses an established correlation between the degree of hydrogenation of an edible oil and its refractive index (RI).

The refractometer cell uses a bare optical fiber in direct contact with processing oil. Equations are given describing the power transmission characteristics of an optical fiber as a function of its cladding RI. Comparisons between calculated and experimental data are shown using test liquids flowing through the refractometer.

INTRODUCTION

Soybean oil consists of triglyceride molecules. Typically, each molecule's three fatty esters occur in 1 of 3 unconjugated carbon-carbon double bond forms: linolenic, linoleic and oleic. Linolenic, with all 3 double bonds, is the most common. It is also the most easily oxidized. As the oil undergoes catalytic partial hydrogenation, selective double bonds are altered and the oil becomes more stable. Its melting temperature also increases (Table 1). Measuring and controlling these characteristics accurately and consistently is important to the oil processor (Hastert, 1988).

Table 1. Characteristics of fatty acids.

Fatty Ester	Relative Oxidation Rate	Relative Hydrogenation Reactivity	Melting Point
Stearic	1	--	70°C
Oleic	10	1	16°C
Linoleic	100	20	-7°C
Linolenic	150	40	-13°C

Hydrogenation is accomplished by mixing the oil, a small amount of catalyst (nickel powder) and hydrogen gas in a pressurized, heated reactor. Time, pressure and temperature determine how many hydrogen atoms become attached to the esters, altering their characteristics (Hastert, 1981).

The relationship between extent of oil hydrogenation and its refractive index (RI) is well-known. Increasing hydrogenation reduces RI. Measuring RI is the accepted standard method for assessing hydrogenation levels during edible oil processing (Bailey, 1982). For soybean oil, RI ranges from 1.4635 to 1.4564 at 50°C (Riceland Foods, 1989). The method is accurate, using standard refractometers (AOCS, 1973). But it is performed off-line, which interrupts the hydrogenation process for significant periods of time. The process is stopped; a sample extracted, and its RI determined using the refractometer, generally with a heated sample cell. The process is repeatedly nudged foward until the desired hydrogenation is reached. If overshoot occurs, the batch must be used for a recipe requiring more hydrogenation. Increased hydrogenation (saturation) results in a more expensive and less saleable oil.

METHODS AND MATERIALS

The fiber optic refractometer cell, shown in Figure 1, incorporates a single fiber in which a portion of the jacket and cladding have been chemically removed, exposing the silica core. The processing oil circulates around the exposed core, and light passing through the fiber is attenuated according the oil's refractive index.

The numerical aperture (NA) of an optical fiber is defined as NA = sinα (Figure 2). Light entering the fiber within the NA propagates through its length; all other light is lost. Using Snell's law at the fiber end and at the core/cladding interface, NA may be derived as (Snyder & Love, 1983):

$$NA = \sin\alpha = (n_{co}^2 - n_{cL}^2)^{1/2}$$

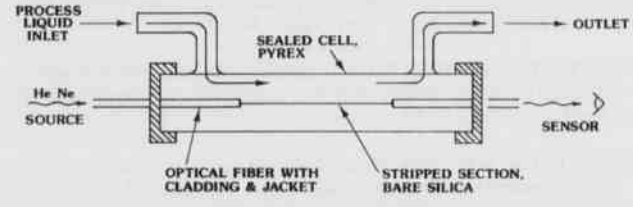

Figure 1. Prototype fiber optic refractometer cell.

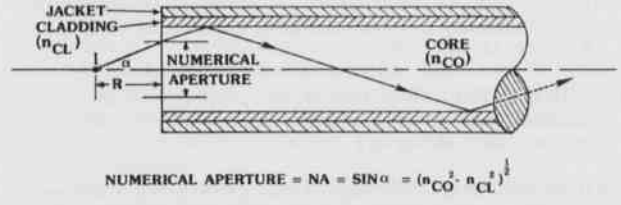

Figure 2. Light transmission in an optical fiber.

where n_{co} and n_{cL} are the core and cladding RI's, respectively ($n_{co} > n_{cL}$). Considering a point source, with intensity, I, at a distance, R, from the fiber end, the area, A, of the NA is approximately:

$$A = \pi(R\sin\alpha)^2 = \pi R^2(n_{co}^2 - n_{cL}^2)$$

The power, P_t, transmitted through the NA is:

$$P_t = I\Omega = \frac{IA}{R^2} = \pi I(n_{co}^2 - n_{cL}^2)$$

where Ω is the solid angle subtended by the NA.

In the fiber optic refractometer, the processing oil becomes the fiber cladding, affecting the fiber's light transmission characteristics (Paul & Kychakoff, 1987; DeGrandpre & Burgess, 1988; Harmer, 1983). Figure 3 shows calculated values of normalized P_t vs n_{cL} for n_{co} = 1.4590. The relationship is approximately linear over the range of interest.

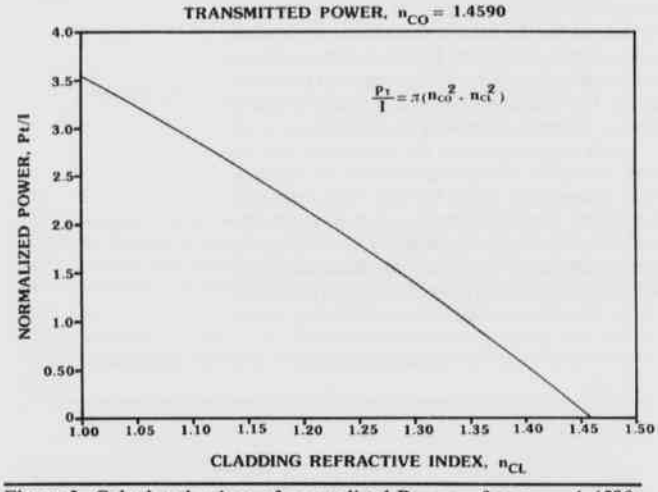

Figure 3. Calculated values of normalized P_t vs n_{cL} for n_{co} = 1.4590.

P_t vs RI was measured using a helium-neon laser as the source and an EG&G radiometer as the sensor. The refractometer test liquid, held at 30 °C, was a solution of corn syrup (RI = 1.4756) and distilled water (RI = 1.3319). The solution RI was varied by dilution. The test liquid was maintained at 30 °C to remove temperature effects. The test liquid RI was determined with an Abbe' refractometer. A least squares fit on the data predicted a core RI of 1.4581. Figure 4 shows the correlation of measured and calculated curves.

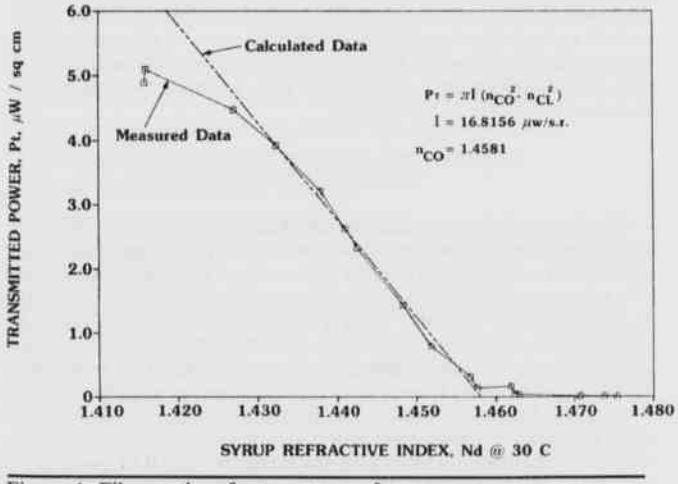

Figure 4. Fiber optic refractometer performance.

DISCUSSION

The response of the fiber optic refractometer cell agrees closely with calculations. The extrapolated least squares fit curve intersected the abscissa at RI = 1.4581. This was unexpected because references had stated the RI of silica at 1.4590 (Corning Glass, 1965). However, data from the specific optical fiber manufacturer shows the RI to be 1.45847 (Fiberguide Ind., 1990). The helium-neon light (wavelength = 632.8 nm) may account for error since the standard light for measuring RI is the D-line of sodium (wavelength = 589.3 nm). The Abbe' refractometer is calibrated to this standard. This difference in wavelengths and unequal dispersions of the core and liquid would introduce error. Deviation from the calculated curve at low RI is due to the fiber's own NA. Its cladding RI is approximately 1.4100 (Fiberguide Ind., 1990). As the test liquid RI decreases below that of the cladding, the fiber's own NA limits P_t.

The effect of temperature on oil RI is significant, and a refractometer cell design must take into account this temperature dependence. Soybean oil, for example, has an RI temperature coefficient of −.000385/°C (AOCS, 1973). Thus to achieve the desired sensitivity, the uncertainty in temperature must not exceed 0.1 °C. Accuracy will be achieved either by closely controlling the cell/liquid temperature or by measuring the cell/liquid temperature and compensating the reading.

The present refractometer cell design exhibits optical noise as the solution RI approaches coincidence to the core RI. Ideally, power throughput is zero at coincidence: the data does not indicate this null value. Observing the refractometer cell under test conditions shows that the light being lost in the optical fiber becomes trapped inside the cell, possibly reflecting light back into the fiber. An improved cell with light absorbing walls and fiber shielding, except on the exposed core section, is being constructed for further testing.

The final design must also take into account the presence of nickel catalyst during hydrogenation processing. The catalyst concentration is approximately one part in 5,600. Its absorbance characteristics, though small, may have an effect on the fiber's power transmission.

CONCLUSIONS

The concept of optical power transmission vs refractive index for the fiber optic refractometer holds true as calculated. The expected relationship shows that such a device could be used to perform real time measurements on edible oils and generate a signal proportional to refractive index. This signal could be used in a process control loop to automatically control the partial hydrogenation of such oils.

Moreover, this instrument holds promise of performing refractive index measurements on liquids compatible with optical fibers and whose RI's are close to that of a fiber.

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