# **Optical fiber sensor for gasoline blend quality control**

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# ABSTRACT

This work shows the use of a long period grating to analyze the quality of a commercial gasoline blend. Samples with different single and double solvent proportions are characterized. It is shown that the addition of controlled amounts of a standard solvent allows differentiating legal blends from tampered blends. With the proper choice of that solvent, it is possible to shift the LPG operation towards a higher sensitivity range, and even samples that deviate little from the legal gasoline blend can be identified.

Keywords: Long period grating, optical fiber sensor, fuel quality, refractive index sensor.

# **1. INTRODUCTION**

The fiber grating technology has imparted a great development not only to the optical telecommunications, but to the optical sensing field as well. Gratings in optical fibers are a periodic perturbation of the wave-guide properties along its longitudinal axis, generally of the core refractive index. The grating can be classified into two general groups, depending upon the periodicity of the refractive index modulation. Fiber Bragg gratings (FBG) are short-period devices (typically a sub-micrometer period), while long period gratings (LPG) present periodicities from 50 - 700 micrometers. Whether it is a FBG or a LPG, the device will promote a coupling between the modes that fulfill a specific phase matching condition, changing the spectrum of the light propagating through the fiber. As this match condition is dependent on several external parameters (e.g. temperature, strain or surrounding media refractive index), these fiber-transducers are very suitable for sensing purposes.

Typically, FBG deployed as sensors are of the order of 1 mm length, with sensitivities to temperature and strain of 13 pm/°C and 1 pm/ $\mu\epsilon$  respectively, for gratings operating at 1300 nm<sup>1</sup>. LPG are typically devices with lengths of the order of 30 mm, and the sensitivities can be much more strongly affected by the local environment parameters when compared with the FBG sensitivities. However, as the LPG sensitivity is also dependent upon the cladding modes involved in the coupling mechanism<sup>2</sup>, the combination of influences allows to produce devices that have a range of responses to a particular parameter from positive to negative values, or they even can be insensitive to the physical parameter. Temperature sensitivities as high as 100 pm/°C or as low as 1.8 pm/°C have been reported<sup>2,3</sup>, and strains sensitivities of -2.2 pm/ $\mu\epsilon$  or 0.04 pm/ $\mu\epsilon$  can be obtained<sup>3</sup>. From refractive index measurements, concentrations of solutions of sodium chloride, calcium chloride and ethylene glycol have been measured, with sensitivities equal to, or better than, that of conventional Abbe refractometry<sup>4</sup>. Some characteristics of such devices (as passive operation and the easiness for insertion into an optical fiber link) make them of special interest to monitor dangerous or inflammable environments.

For the LPG, the grating pitch results in coupling between the fundamental core mode to several cladding modes, leaving attenuation dips in the transmission spectrum of the fiber. The wavelength of the resonance dip that corresponds to the coupling between the fundamental core and the *m*-th cladding modes,  $\lambda_m$ , obeys the phase matching condition<sup>5</sup>:

$$\lambda_m = \left( n_{co} - n_{cl}^m \right) \cdot \Lambda \tag{1}$$

where  $\Lambda$  is the grating period, and  $n_{co}$  and  $n_{cl}^{m}$  are the effective refractive indexes of the core mode and of the *m*-th cladding mode, respectively. The effective refractive index of the cladding is dependent on the refractive index of the surrounding medium, which enables LPG to be used as refractive index sensors<sup>6,7</sup>.

In Brazil, anhydrous alcohol is added to commercial gasoline blends to increase the octane number and to save costs in a legal proportion between 24 and 26 %. However, as anhydrous alcohol and some solvents have lower prices (about

60 %) than gasoline, a common malpractice is to increase the solvent or alcohol concentration beyond the allowed one in the commercial gasoline that is sold to car owners.

This work uses an LPG to analyze the quality of Brazilian commercial gasoline blends. The method relies on the controlled addition of a standard solvent to the commercial gasoline blend, followed by the analysis of the LPG response.

## **2. EXPERIMENTAL**

The long period gratings are produced in a standard telecommunication fiber applying a point-to-point electrical arc discharge from a fusion splicer using a similar technique used by Rego *et al*<sup>8</sup>. The advantage of using electrical arcs to write the gratings is that no special fiber (hydrogenated or pre sensitized) is required. An optical set-up is used during the writing process to monitor the transmission spectrum through the fiber, allowing the process to be interrupted when the measured spectrum shows suitable characteristics for an intended application.

The LPG used in our experiments is 3.6 cm long, with a period of 595  $\mu$ m and 60 interaction points, fabricated in a fiber with a cladding radius of 62.5  $\mu$ m and a core radius of 4.5  $\mu$ m. The arc parameters are 12 mA for the current and 0.5 s for the fusing time. For gasoline blend analysis the LPG is inserted into a specially designed glass container with four openings, two of them used to insert the optical fiber and the other two ones to add and to drain the gasoline samples. After the LPG insertion into the c ontainer, the fiber ends are held to a void fiber-bending interference on the s ensor response, and the glass container is fastened to avoid its movement. Another parameter controlled during the measurements is the temperature, which is constant within  $\pm$  0.5 °C. Figure 1 shows the set-up used to measure the LPG wavelength shifts when it is immersed in the samples. A LED source (central wavelength at 1550 nm and half bandwidth of 52.0 nm) and an Optical Spectrum Analyzer (0.1 nm resolution,  $\pm$ 5 pm of wavelength in air, obtained using LabView software, is 1520.7 nm at 22 °C. The refractive indexes of the samples are measured with an Abbe refractometer.



Figure 1- Experimental set-up used to analyze the gasoline blend quality.

This set-up allows controlling the high sensitivity of LPG to mechanical strain and the high evaporating rate of gasoline samples. Two needles are fastened in the both opposit glass openings with the help of rubbers, and the fiber is inserted through them. Moreover, this set-up allows changing the fiber sensor without removing the container glass or other parts of the experimental apparatus.

The gasoline blend samples used in the experiments are prepared according to Table 1. In that table pure gasoline stands for a sample of regular or legal gasoline without anhydrous alcohol, and sample 1 represents a legal commercial gasoline. The other samples are prepared with a legal alcohol proportion, but contain an illegal solvent addition in their compositions. In samples 2,3, 4, 5, and 6 only one type of solvent is added to the blend, whereas in the samples 7 and 8 two types of solvents are simultaneously employed in different proportions. Table 1 also shows the measured refractive indexes of the blends (samples 1, 2, 3, 4, 7 and 8), and the calculated values (with the superscript \*) based on the solvent proportion samples and the refractive index of both pure solvents and pure gasoline.

Usually, the fuel quality control is performed in the gas stations by measuring only the alcohol proportion in the commercial gasoline, by means of density measurements. That control technique is not sensitive when other solvents plus alcohol are added to the blend in a fraudulent way that keeps the legal alcohol proportion.

| Sample | Pure gasoline | Anhydrous    | Solvent    | Solvent | Alcohol        | Solvent        | Refractive |
|--------|---------------|--------------|------------|---------|----------------|----------------|------------|
|        | (ml)          | alcohol (ml) |            | volume  | proportion (%) | proportion (%) | index      |
|        |               |              |            | (ml)    |                |                |            |
| 1      | 300           | 100          | -          | -       | 25.0           | -              | 1.4105     |
| 3      | 240           | 95           | Kerosene   | 45      | 25.0           | 11.8           | 1.4142     |
| 5      | 240           | 95           | Turpentine | 45      | 25.0           | 11.8           | 1.4124     |
| 6      | 300           | 110          | Turpentine | 30      | 25.0           | 6.8            | 1.4121     |
| 7      | 300           | 110          | Kerosene   | 30      | 25.0           | 6.8            | 1.4125*    |
| 8      | 300           | 110          | Naphtha    | 30      | 25.0           | 6.8            | 1.4090*    |
| 9      | 270           | 120          | Kerosene   | 32      | 25.0           | 6.7            | 1.4144     |
|        |               |              | Turpentine | 58      |                | 12.1           |            |
| 10     | 270           | 120          | Kerosene   | 58      | 25.0           | 12.1           | 1.4153     |
|        | _             |              | Turpentine | 32      |                | 6.7            |            |

Table 1- Composition of gasoline blends used in the experiments. Refractive indexes are measured for temperatures between 20.1 °C and 20.4 °C. The values with (\*) are caculated ones.

For all the gasoline samples analysis, the wavelength shifts are measured relatively to the LPG immersed in a reference fluid. That procedure is employed to avoid influence in the LPG dip wavelength due to temperature changes in measurements taken in different conditions. Also, as the reference fluid chosen is the anhydrous alcohol, the described procedure provides a cleanness of the LPG surface, removing films of heavy hydrocarbons that could be deposited on the fiber. As the solvent addition to the gasoline samples results in blends with different refractive indexes, the LPG sensor immersed in such samples shows different wavelengths for the ressonance dip. However, depending on the relative sensitivity of the LPG to a particular solvent, and also on the magnitude of change in the refractive index, the wavelength shifts become difficult to be detected. A way to overcome this drawback is by injecting a controlated amount of a suitable standard solvent to the sample under analysis, so that the refractive index is shifted to the higher sensitivity range of the LPG.

#### **3. RESULTS AND DISCUSSION**

When the LPG is immersed in a fluid the wavelength of the attenuation dip shifts relatively to its position in air. This shift occurs due to the external medium refractive index change. In figure 2 is shown the typical behavior of an LPG attenuation dip when the external medium refractive index is changed<sup>9</sup>. From that figure, one can observe that the attenuation dip shifts towards lower wavelengths when the external medium refractive index to the cladding one, the higher the wavelength shift and consequently the grating sensitivity.

In order to o btain the L PG response when it works in different sensitivities ranges, two standard solvents are used: anhydrous alcohol (n = 1.3628) and soy oil (n = 1.4742). For the later, the maximum amount added to the gasoline blend produces a sample whose refractive index lies below of the cladding refractive index. When the anhydrous alcohol is added to the samples, the LPG wavelength shifts for the gasoline blends are shown in figure 3.



Figure 2- LPG wavelength dip position for different external medium refractive indexes (see ref. 9).



Figure 3- LPG resonance wavelength shifts for different gasoline samples when anhydrous alcohol is used as the standard solvent.

From this figure one can observe that, for 25 % of alcohol concentration, all the analyzed samples can be distinguished from the sample 1 (the commercial legal gasoline blend), as illegal presence of solvents in the blends results in wavelength shifts that can be measured. However, due to the small difference between the wavelength shifts obtained for samples 3 and 4 (0.09 nm), it becomes difficult to distinguish between these two tampered samples. The LPG sensitivity curves for all the gasoline blends are shifted towards lower wavelengths when turpentine and/or kerosene are added to the blends (samples 2, 3, 4, 5, 7, and 8). In other hand, naphtha addition (sample 6) shifts the sensitivity curve towards higher wavelengths. Such behavior is expected, as turpentine and kerosene addition increases the refractive indexes of the samples (in comparison with sample 1), whereas naphtha addition decreases the refractive index of the sample (see table 1 and figure 2).

Figure 4 shows the wavelength shift for samples 3 and 4 when soy oil (instead of anhydrous alcohol) is used as the standard solvent. As it can be seen from that figure, becomes easier to distinguish between these two tampered samples

for soil oil concentrations in both 30 - 40 % and 55 - 60 % ranges. For these two ranges, the average difference between the wavelength shifts is 0.23 nm and 0.36 nm, respectively. For solvent concentrations from 40 % to 55 % the two curves experiences an approximation, what difficult the individual identification of the samples. The shape of the curves is a combined effect of the change in the grating sensitivity and the increase of the solvent concentration itself.



Figure 4- LPG resonance wavelength shifts for samples 1, 3 and 4 when soy oil is used as the standard solvent.

#### **5. CONCLUSION**

We described the use of a long period grating sensor for gasoline blend quality control. For all the used samples, the device showed to be able to identify the tampered samples when compared with the legal commercial gasoline sample. It is also possible to distinguish if the solvent used to produce an illegal blend belongs to a family of higher or lower refractive index, comparatively to the refractive index of the legal gasoline blend. The obtained results show that even a tampered blend with a little proportion of solvents can be distinguished from the legal gasoline with the addition of a standard solvent to the sample under analysis. A further improvement in the sensor performance can be obtained by writing long period gratings with lower bandwidths and/or a more pronounced attenuation dip.

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