

# Birefringent porous glass fiber-optic sensor as a low-cost end-of-service-life indicator (ESLI) for organic vapor respiratory cartridges

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**Abstract:** Birefringent porous glass integrated between two plastic optical fibers terminated by crossed Polaroid™ films was used as a low-cost end-of-service-life indicator (ESLI) for organic vapor cartridges. Birefringence is significantly reduced when volatile compounds condense into the anisotropic porous structure. Such changes could easily be quantified using a low-cost light source and detector and very simple light intensity analysis. We showed that such sensors integrated into an experimental organic vapor cartridge could easily detect the progression of low concentrations of solvents (such as 50 ppm of toluene) into the activated charcoal cartridge and thus truly be used as an ESLI. Since such robust sensors could detect broadband solvents by simple physical condensation similar to the trapping phenomena of activated carbon, they are probably ideal candidates for such application.

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## 1 Introduction

Air-purifying devices, including air-purifying respirator cartridges and canisters, are widely used in the civil and military industries to protect workers against the harmful effects of toxic materials. Such devices usually consist of a filter chamber filled with adsorbent material (generally activated charcoal) that traps (adsorbs or absorbs) vapors or gases on its surface or within its porous structure. As the adsorbent material is completely filled, the air-purifying device loses protective capability for the user against the contaminant. This could have dramatic effects, especially when the contaminant has poor warning properties, *e.g.* if its odor, taste or irritation limit is greater than the permissible exposure limit or if there is insufficient toxicological data to determine an exposure limit.

In establishing new certification standards in 1984, the *U. S.* National Institute for Occupational Safety and Health (NIOSH) encouraged the development of active end-of-service-life indicators (ESLI). Such indicators should detect the presence of contaminants and provide an unambiguous signal warning the user that the filter of the air-purifying device is almost exhausted. End-of-service-life indicators may involve a visual color change that warns the user to replace the filter. Such color changes are sometimes induced by chemical reactions of a usually single-use color indicator which has the great advantage of being simple and economically acceptable. One drawback of such chemical color indicators is, however, that they are usually very specific to the chemical or class of chemicals (such as acids) they should react with. In the case of organic solvents, such chemical reactivity is rarely available to allow the development of a broadband solvent detection based on chemical color indicators.

In order to have wider detection sensitivity, the sensor could rather rely on more physical phenomena such as condensation, adsorption, or absorption into small pores. Such changes in the porous sensing material could advantageously be evaluated using optical methods. Interesting approaches for solvent detection have been proposed using either porous silicon [1,2] or porous glass [3,4]. However, there are still few publications describing the use of optical sensors for solvent detection integrated inside organic vapor cartridges. In some of them porous glass optical fiber sensors have already been proposed as an ESLI for respiratory cartridges [5,6]. In this work, light intensity transmitted through a porous glass fiber was modified in the presence of organic vapors. But for such waveguide type sensors, the length of the fragile porous glass has to be sufficient (at least a few centimeters) in order to provide a good sensitivity to solvents, making them difficult to manipulate and to package into small commercial cartridges.

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We present in this paper a completely different approach still using porous glass but for which the optical anisotropy rather than waveguide properties of the sensitive material are modified in the presence of solvent vapors. It will be seen that with a compact and robust design, a birefringent porous glass sensor can be used with a simple interrogation unit to provide a low-cost broadband ESLI for organic vapor cartridges.

## 2 Materials and Methods

### 2.1 OAT-TS sensor manufacturing

The porous glass was produced from borosilicate glass fibers that were heat treated for several hours in order to induce a spinodal decomposition leading to phase separation. The boron rich phase was then leached out by acid treatment in order to produce a silica rich interconnected porous structure [7]. Fibers were rinsed in water and finally in isopropanol before being stored under vacuum at room temperature.

Sections of plastic optical fiber (POF) 1 mm in diameter were cut, polished, and terminated on one side by a small disc of Polaroid™ thin film. Two such POFs were aligned into a slotted metal tube with Polaroid™ films facing each other and oriented in order to obtain complete extinction of transmitted light. Then birefringent porous fiber was inserted into the slot and oriented at a 45° relative to the polarizer axis in order to maximize the sensor contrast. Once assembly was optimized all elements were fixed with epoxy adhesives. The diameter of the center part of the sensor prototype was 1.8 mm. A more compact version of this optical anisotropy technology transmission sensor (further referred to as an OAT-TS sensor) could easily be manufactured since transmitted light levels were not an issue.

Transmission profile of each OAT-TS sensor was obtained using a halogen light source and a photo spectrometer from Ocean Optics (SD-2000). Such profile was used in order to adjust the three different light intensities of each red-green-blue (RGB) color in order to cover an optimal dynamical range without saturating the light detector of the signal conditioner.



**Figure 1 : Left: OAT-TS sensor connected to RGB light source and to light detector. Right: OAT-TS sensor connected to OAT-DL signal conditioner (2 channels).**

### 2.2 OAT-DL signal conditioner

We designed a two channel signal conditioner (further referred to as OAT-DL data logger) suitable for the birefringent OAT-TS sensor. Basically the unit cyclically controls the intensity of two RGB light emitting diodes (LEDs) and records for each channel the light intensity (separated in 1024 levels) collected by a phototransistor. The self-powered unit can be programmed or interrogated by a computer via a RS-323 communication port. The unit was set to record light intensity at the three RGB wavelengths every 30 s. Convenient light source and light detection interfaces have been developed in order to facilitate connection directly to bare  $\varnothing$  1 mm FOPs as seen in Figure 1.

The two extremities of each OAT-TS sensor were simply connected to the RGB light source and the light detector (see Figure 1 left). Initial light intensity of each RGB color was set to a predetermined value (selected according to transmission pattern) and was constant during the whole experiment. Light intensity variation was thus only due to shifts in the transmitted spectra of the OAT-TS sensor in response to solvent vapor detection [3,8].

### 2.3 Test equipment for solvent cartridge challenge

In order to simulate a normal use of an organic vapor cartridge, two OAT-TS sensors were embedded at different locations (located at 20 mm and 80 mm from the gas inlet) inside an  $8 \times 18 \times 163$  mm aluminum chamber filled with activated carbon (extracted from new organic vapor cartridges). They were connected to the two channels of an OAT-DL signal conditioner. The dimension of the experimental cartridge chamber was reduced in order to minimize experimental breakthrough time.

Dry nitrogen gas flowing at 1 L/min was used for the cartridge challenge test. The addition of solvent (toluene) was done by continuously injecting (using a Harvard Apparatus syringe pump, model #PHD 2000) a controlled amount of liquid into an expansion chamber heated at 90°C to ease solvent vaporization. The slow injection flow of the liquid solvent was adjusted in order to have a 50 ppm concentration during the entire challenge test.

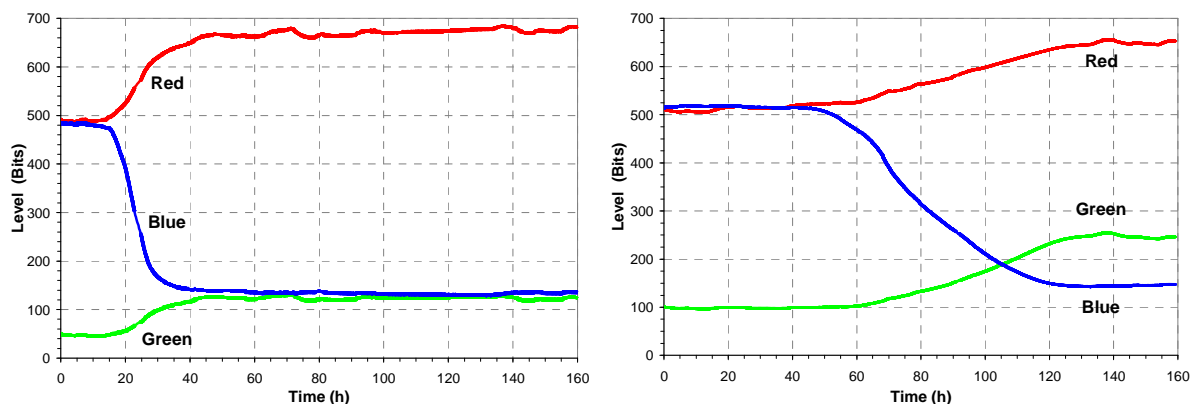
### 3 Results and Discussion

In the past we presented work showing that birefringent porous glass could be used for volatile organic compound detection [3,8]. The porous structure of the glass (with pore diameters ranging from 2 nm up to >60 nm) was proven to have the ability to condense solvent vapors that in turn changed the optical anisotropic properties of the material. Such variations could quite easily be observed when the birefringent glass was placed at a 45° between two polarizers. If a white light source was used, solvent detection could be identified by color change, or by light intensity change if a single or narrow wavelength light source was used [8]. Light intensity measurement is probably the best choice for precise quantification of the changes related to solvent concentration, even though color changes make such technology very attractive for more passive sensor development. So in order to first provide a proof of concept, we selected to measure light intensity for better quantification of the measurement.

The use of solvent detection within the cartridge is of particular importance since depending on the mixture or the sequence of the solvent vapors the cartridge is exposed to, the cartridge breakthrough is modified by solvents being displaced by others which are more strongly bonded to the activated charcoal [9]. Since the adsorption capacity of the carbon and the breakthrough time for weakly adsorbed compounds are decreased significantly by exposure to mixtures, it is important in real life to be able to rely on real measurements rather than on complex calculations to predict actual cartridge solvent breakthrough.

We miniaturized the sensor design in order to provide a sensor that could be integrated into respiratory cartridges without disturbing much the flow distribution. The reduction in overall cost of the sensor was achieved by selecting Ø 1 mm plastic optical fibers and commercial Polaroid™ film. Only two millimeters of birefringent porous glass were used as a sensing part in our prototypes. This is one of the great advantages over the other approach involving waveguide property changes of long porous glass fiber, since in the OAT-TS sensor the most fragile part of the sensor is protected by metal tubing and can easily survive the activated carbon packing process and ultrasonic welding of the respiratory cartridge lead.

In order to validate the concept of the use of an OAT-TS sensor as an ESLI for a cartridge filled with activated carbon, we placed two OAT-TS sensors at two different positions (at 20 mm and at 80 mm from the gas inlet). The two OAT-TS sensors were connected to a dual channel OAT-DL signal conditioner as shown in Figure 1 (right). The aluminum experimental cartridge was then challenged with 50 ppm of toluene. Such a low concentration of organic vapor was selected since it corresponds in the USA to the threshold limit value (TLV) for toluene which for this solvent is lower than its permissible exposure value (PEV). It was also representative of a realistic average solvent concentration that could be present in selected industrial environments.



**Figure 2: Light intensity signal changes observed at 3 different wavelengths (corresponding to RGB colors of commercial LEDs) for 2 OAT-TS sensors positioned at 20 mm (left) and at 80 mm (right) from a gas inlet inside an experimental activated carbon cartridge during a 50 ppm toluene challenge (nitrogen @ 1 L/min)**

Figure 2 shows the light intensity changes at three different wavelengths (corresponding to the three RGB colors of a commercial LED) recorded for the two sensors. It could be seen that the first OAT-TS sensor detects the presence of toluene trapped into activated carbon after about 15 h (increase of red and green and decrease of blue light intensities). A plateau is obtained after about 40 h indicating that at this position the toluene concentration trapped into the carbon remains constant. The second OAT-TS sensor detects the presence of toluene after about 50 h (same behavior as the first sensor) and reaches a plateau after about 140 h. Before 50 h, this sensor has a constant and very stable intensity baseline indicating that no toluene is detected at this position since it is trapped efficiently by activated carbon packed in front of this position. Interestingly the second sensor, situated 4 times further than the first one, detects the toluene solvent front inside saturated carbon about less than 4 times later and it takes about 4 times longer to reach the plateau (~90 h instead of 25 h) which is well in agreement with the progression of toluene saturation inside the porous activated carbon (note that inlet limit conditions probably affect the first sensor's detection response). In our example, the first OAT-TS sensor is fully saturated before the second sensor starts detecting the toluene diffusive progression into the carbon bed. Thus, depending on the position where the OAT-TS sensor is placed into the activated carbon bed, it could indicate to the user how the cartridge is saturated and thus serve as a true ESLI. It is also worth mentioning that intensity at different wavelengths could change in opposite directions (e.g. such as observed for blue versus red light intensities) when detecting solvent inside the cartridge. Such behavior allows a more reliable design where light source intensity variations could be corrected by calculating the intensity ratio at two different wavelengths.

The presence of activated carbon surrounding the OAT-TS sensor is an ideal medium since it protects porous glass from possible contaminations. Since birefringent porous glass that we developed could detect indifferently all tested organic solvents [3,8] and due to the fact that its physical structure is similar to that of activated carbon, the use of the optical anisotropy technology described in this paper is probably an ideal situation for a commercially successful broadband solvent sensor that could be used as an ESLI for respiratory cartridges.

#### 4 Conclusions

We demonstrated that a birefringent porous glass sensor could be used as a low-cost end-of-service life indicator for organic vapor cartridges. Thanks to the fact that with this approach only a small sensitive porous glass section is required, we show how a birefringent porous glass fiber-optic sensor could easily be integrated inside an organic vapor cartridge and be potentially used as an end-of-service life indicator (ESLI) for commercial cartridges. We demonstrate that such robust sensors could be sensitive to very low solvent concentrations. The simple designs of the sensor and interrogation unit allow further miniaturization of the described prototypes. This promising new technology is currently under development at FISO Technologies adding new opportunities to our great variety of existing fiber-optic sensors (for details see [www.fiso.com](http://www.fiso.com)).

#### 5 References

- [1] Liu R., Schmedake T.A., Li Y.Y., Sailor M.J. & Fainman Y., "Novel porous silicon vapor sensor based on polarization interferometry", *Sensors and Actuators - B*, **87**, 58-62 (2002)
- [2] Dorvee J. & Sailor M.J., "A low-power sensor for volatile organic compounds based on porous silicon photonic crystals", *Physica Status Solidi - A - Applied Research*, **202** (8), 1619-1623 (2005)
- [3] Pinet É., Vachon-Savary M. & Dubé S., "New optical sensor for volatile organic compounds (VOC) using birefringent porous glass", *Sensors 2004 Proc. of 3<sup>rd</sup> IEEE Int. Conf. on sensors*. (Vienna), 1293-1296 (2004)
- [4] Pinet É. & Duplain G. "Method and sensor for detecting a chemical substance using an optically anisotropic material", *PCT Patent #2004057314 (A3)* (2004)
- [5] Caron S., Bernard P., Vernon M. & Lara J., "Porous glass optical fiber sensor as an end-of-service life indicator for respiratory cartridges", *Sensors and Actuators - B*, **102** (2), 198-206 (2005)
- [6] Bernard P., Caron S., St-Pierre M. & Lara J., "End-of-service indicator including porous waveguide for respiratory cartridge", *US Patent #6,375,725 B1* (2002)
- [7] Antropova T.V. & Drozdova I.A., "The influence of synthesis conditions of porous glasses on their structure", *Glass Phys. Chem.* **21** (2), 131-140 (1995)
- [8] Pinet É., Dubé S., Vachon-Savary M., Côté J.-S. & Poliquin M., "Sensitive chemical optic sensor using birefringent porous glass for the detection of volatile organic compounds", *IEEE Sensors J.*, **6** (5), in press (2006)
- [9] Yoon Y.H., Nelson J.H. & Lara J., "Respirator cartridge service-life: exposure to mixtures", *Am. Ind. Hygiene Assoc. J.*, **57** (9), 809-819 (1996)