

Time-dependent variation of POF Bragg grating reflectivity and wavelength submerged in different liquids

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Abstract: In this work, we investigate the time-dependent variation of both the reflectivity and resonance wavelength of microstructured polymer optical fiber Bragg grating (mPOFBG) array sensors embedded in silicone rubber and polyurethane resin diaphragms in contact with water and aircraft fuel, respectively. The array sensors were inscribed using two different phase masks with pitches of 557.5 and 580 nm and thermal annealing of the inscribed fiber was used to change the Bragg wavelengths. Both the reflection and the resonance wavelength shift were monitored over 90 days submerged in liquid and two studies were investigated. In the first study, beyond the mPOFBGs coated with the diaphragm, also the rest of the fiber is totally protected between the sensors with the same material used for diaphragms. On the other hand, in the second study, the fiber between sensors is unprotected – in direct contact with liquid. PMMA and TOPAS fibers were used and this study suggests that TOPAS fiber should be a good option for long-term liquid monitoring applications.

Keywords: Polymer optical fiber, Bragg grating sensors; Reflectivity, Fuel, PMMA, TOPAS, Liquid level monitoring systems

1. Introduction

The interest in the development of the technology in polymer optical fibers (POFs) has been growing in the last years. POFs are starting to be considered as a viable alternative to silica fibers in sensing applications due to the different material properties of polymers compared to silica. There are some advantages in certain applications [1–5], such as their high flexibility in bending, low temperature processing, ease of handling, high fracture toughness, and ruggedness, which are properties that silica fibers do not have. In addition, POFs have a high elastic strain limit and low Young's modulus, which makes them advantageous for fiber Bragg grating (FBG) based strain sensing applications [1, 3–5]. As is known, some polymers, such as poly(methyl methacrylate) (PMMA), are humidity sensitive and strongly absorb water [6–8], while other polymers, such as thermoplastic olefin polymer of amorphous structure (TOPAS), were shown to be insensitive to humidity [9,10]. Due to the moisture absorbing capability of PMMA based POFs, which leads to a change in the refractive index and size of the fiber, both of which contribute to a change in Bragg wavelength [6], these fibers are used to develop humidity sensors [8,11], water activity sensor in fuels [7,12], among others. Although some significant research has been achieved in POF Bragg gratings (POFBGs) including these applications, still some answers regarding the time-dependent variation of optical properties in POFBGs remain unclear when they are placed in contact with water and fuel. Therefore, this issue has a lot of importance in different sensor types, such as water activity sensor systems, fuel storage and level systems or biochemical processing [7,12–14].

In recent years, many optical fiber liquid level sensors have been reported to be safe and reliable in different environments and present many advantages for liquid level measurement [13,15–18], including for aircraft fuel level monitoring system utilizing POFBGs [14]. Above all, the moisture or

47 water mixed in the fuel will have little influence on the optical fiber liquid level sensors. However,
48 most of these sensors have not been commercialized, as an alternative to the traditional liquid level
49 sensors based on electro-mechanical techniques, because they exhibit some drawbacks, such as low
50 sensitivity, limited range, long-term instability, limited resolution, high cost, or weakness. In
51 addition, any sensors that involve direct interaction of the optical field with the fuel (either by
52 launching light into the fuel tank or via the evanescent field of a fiber-guided mode) must be able to
53 cope with the potential build up of contamination – often bacterial – on the optical surface. Recently,
54 we have presented new approaches for liquid level sensor systems [13,14], which may overcome
55 these drawbacks but some additional studies need to be investigated and discussed about their time-
56 dependent variation of the POFBGs' optical properties.

57 In this paper, a detailed study the time-dependent variation of both the reflectivity and
58 resonance wavelength of microstructured POFBG (mPOFBG) array sensors is reported. The
59 mPOFBGs are embedded in silicone rubber and polyurethane resin diaphragms and placed in contact
60 with water and JET A-1 aircraft fuel, respectively. These array sensors are based on mPOFBGs
61 inscribed in the same fiber spatially separated by 15 cm in the 850 nm spectral region. Both the
62 reflection and the resonance wavelength shift were monitored over 90 days and two studies were
63 made: first study - beyond the mPOFBGs coated with the diaphragm, also the rest of the fiber is
64 totally coated between the sensors with the same material; second study - the fiber between sensors
65 is uncoated (in direct contact with liquid). PMMA and TOPAS mPOFBGs were used and studied in
66 detail, achieving long-term drifts in PMMA, possibly due to the sensitivity of PMMA to water. In
67 contrast, TOPAS mPOFBGs revealed a much improved behavior for long-term liquid monitoring
68 systems suggesting a good option for this type of application.
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70 2. Sensor array fabrication

71 Identical mPOFBGs were inscribed in a doped mPOF fabricated from PMMA – for –fabrication
72 details see [19] – as exhibited in Fig. 1, where the transmission spectrum of one array is shown. The
73 mPOF has a core diameter of 6 μm and an outer diameter of 125 μm . This fiber has been chosen since
74 it –benefits from much less loss in the 850 nm spectral region than at 1550 nm (low loss of 7 dB/m at
75 850 nm spectral region) in order to optimize the reflected peak power of all gratings whilst having a
76 sufficiently long length of polymer fiber. Using a single 75 cm long fiber, the five multiplexed
77 mPOFBGs are inscribed spatially separated by 15 cm, being the largest reported to-date [14]. A CW
78 He–Cd laser with an output power of 30 mW at 325 nm was used to inscribe the gratings array in the
79 doped mPOF. In our inscription system, this fiber has an optimum inscription time of 9 minutes. The
80 inscription process was monitored using a super-luminescent diode from Superlum centered at 835
81 nm (with a power output of 1.25 mW over a spectrum width of 50 nm) and an optical spectrum
82 analyzer (OSA) connected to an 850 nm single-mode silica coupler. To obtain five gratings with
83 different wavelengths, two different phase masks were used with pitches of 557.5 and 580 nm and
84 thermal annealing of the inscribed fiber was used to change the Bragg wavelengths [20]. Following
85 grating inscriptions, the mPOF containing the FBGs was glued into demountable FC/PC connector.

86 For the diaphragm manufacture, the silicone rubber solution was prepared by mixing
87 homogeneously two liquids (SILASTIC® T-4 Base and Catalyst from DowCorning Corporation) in a
88 ratio of 100:10 by volume and curing at room temperature in 24 hours. A different material was used
89 too, polyurethane resin from Liquid Lens is based on a mixing of two liquids - MF633 resin and
90 DK780 catalyst - in a ratio of 100:100 by volume and curing at room temperature in 8 hours (23°C,
91 40% RH). So, we have two types of diaphragms material to test in water and in fuel – silicone rubber
92 and polyurethane resin, respectively. Each type of solution was poured in 50 mm diameter plastic
93 containers with a height of 1.1 mm, in which was also placed the POF piece containing one of the
94 FBGs array. So, we fabricated different FBGs arrays to embed diaphragms to test in water (silicone
95 rubber diaphragms) and in fuel (polyurethane resin diaphragms). With regard to uniformity, all
96 diaphragms obtained had thicknesses around 1.08 ± 0.01 mm, as it was measured using digital

97 calipers. Simple calculations of volume were made to obtain the same volume of solution in each
 98 diaphragm. More details of the diaphragm fabrication can be found in [13].
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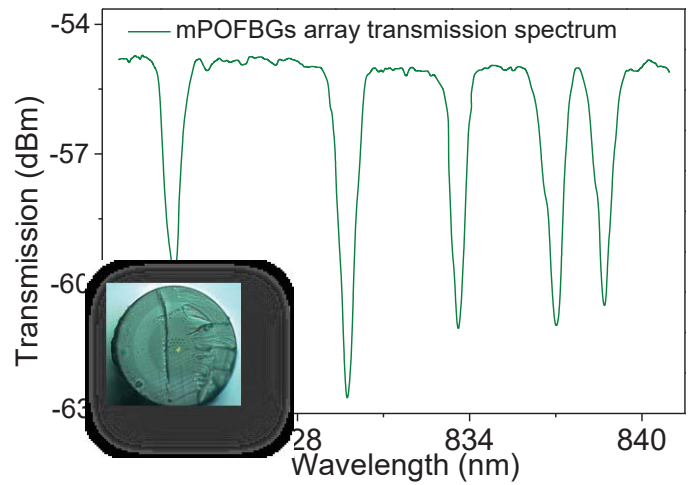
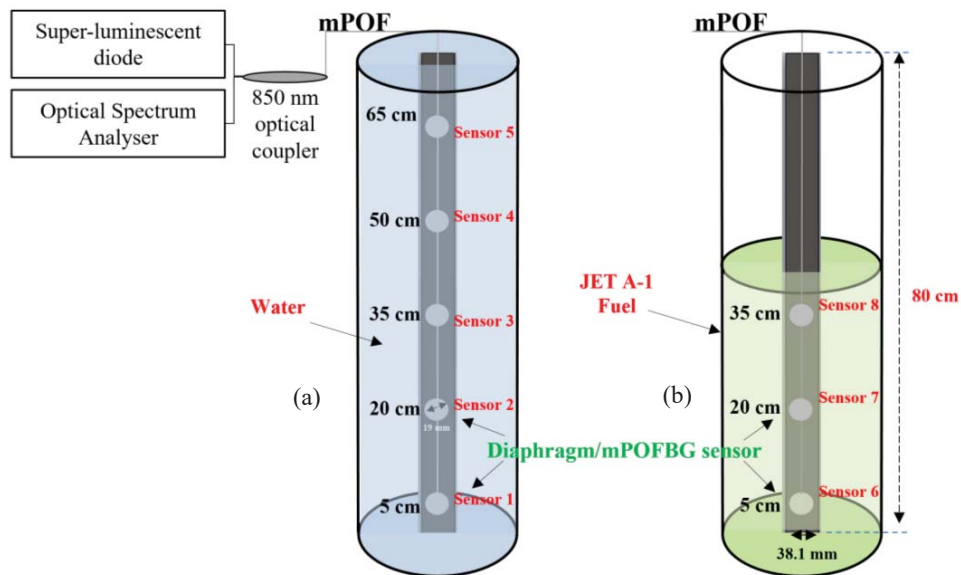


Figure 1. Transmission spectra of an mPOFBGs array with five multiplexed gratings. Inset: Image of the mPOF used with three-ring hexagonal cladding structure [19].

A similar design of the prototype multiple sensor configuration presented in [13,14] was used, which consists of a square acrylic tube (800 mm length), but with no windows drilled at equidistant positions along it as was done in our previous work. The reason is that sensors would suffer deformation if we have windows drilled as was used before. With no holes at the position sensors on tube, we have no influence from sensors in terms of diaphragms deformation. The configuration contains five sensors spatially separated by 150 mm (see Fig. 2 (a)). The sensors were then placed and sealed at positions. To fix the diaphragms in each position, a retaining ring was used, with the diaphragm sandwiched between the tube and retaining ring. Eight screws were used to hold the tube and retaining ring together, producing a strong seal. This square tube containing the sensors is placed inside the cylindrical tube and after that, the cylindrical tube is full of water – all sensors submerged in water (see Fig. 2 (a)). For fuel, the same procedure is done however, only three sensors are submerged and the fuel level is set at 40 cm (Fig. 2 (b)) due to safety reasons when is used fuels.



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Figure 2. Experimental apparatus with sensors submerged in liquid: (a) five sensors in water and (b) three sensors in fuel. All sensors for both cases are spatially separated by 15 cm.

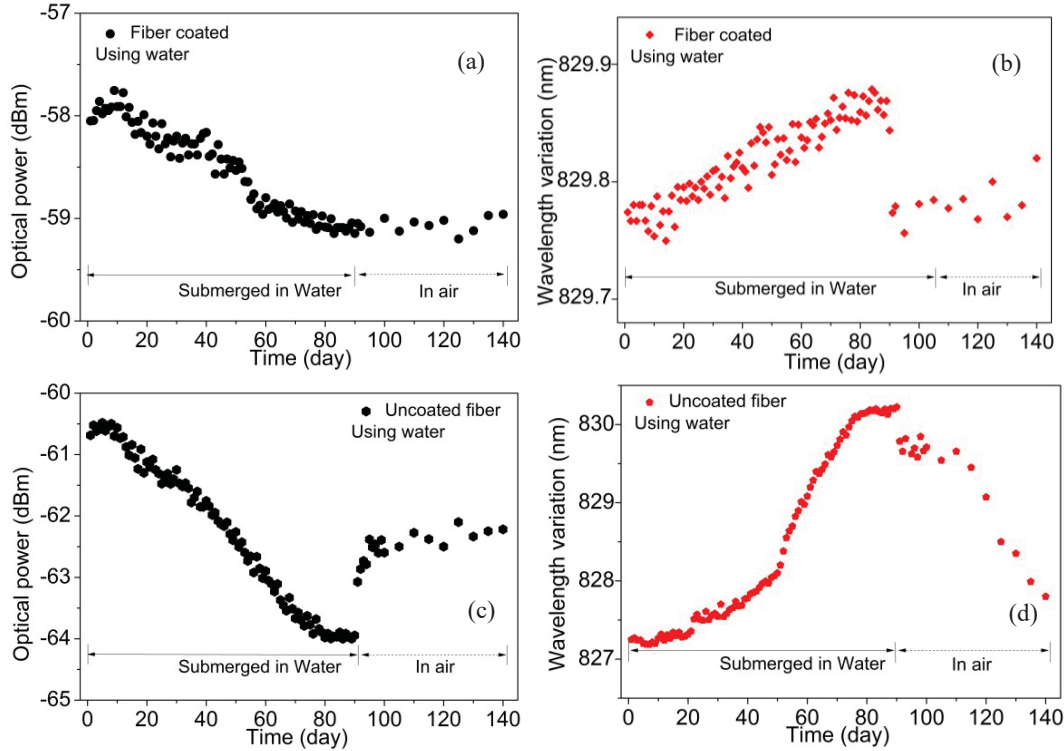
133 3. Experimental results and discussion

134 Experiments were carried out to evaluate the mPOFBG variation over time, both the reflection
135 peak and the resonance wavelength shift were recorded over 90 days with liquid tank full and two
136 studies were made: a) beyond the mPOFBGs coated with the diaphragm, also the rest of the fiber is
137 totally coated between the sensors with the same material used for diaphragms (no contact between
138 liquid and fiber and for that we protected/coated these fiber parts with the same diaphragm material);
139 b) the fiber between mPOFBG/diaphragms is uncoated or unprotected (it means that the fiber is in
140 contact with the liquid). For that, we used our mPOFBGs embedded in silicone rubber diaphragms
141 to test in water and polyurethane resin diaphragms to use in fuel, since previous tests revealed the
142 silicone rubber material to be unsuitable for prolonged use in fuel [14]. Furthermore, polyurethane
143 material has been successfully employed in our previous work [14] which shows good promise for
144 long term use in fuel. So, we also coated the entire fiber since PMMA material has affinity to water
145 [12,21].

146 The experimental data was collected from sensors – containing the sensors in water and fuel.
147 The room temperature was maintained at 23°C throughout the experiments. Figs. 3 show the case
148 when the mPOFBGs are embedded in silicone rubber, which is water permeable [13] and thereafter
149 placed in the water tank. It shall be noted that similar behaviors were achieved for all sensors (see
150 Table 1) and Figs. 3 show the behavior from sensor 1. From Figs. 3 (a) and (b), one can be observed,
151 for the first case (coated fiber), that the reflected peak power and resonant wavelength show a very
152 slight change of their values after 90 days. The reflected peak power throughout the whole
153 investigation decreased 1.38 dB and the total wavelength shift was 140 pm. In the second case
154 (uncoated fiber), the reflected peak power and resonant wavelength keep slightly stable during the
155 first 15 days with no significant variation however, after that time, both reflected peak power and
156 resonant wavelength do not reach a steady state, excepting the last 10 days, as shown in Figs. 3 (c)
157 and (d), respectively. The decrease of reflectivity throughout the whole investigation was 3.58 dB and
158 the total wavelength shift was 3.05 nm. After that, the sensors were out of water to check if they
159 recover for their initial reflected level and Bragg wavelength. Their behaviors can be also seen in Figs.
160 3, showing that, in the coated case (see Figs. 3 (a) and (b)), the differences are not so significant after
161 50 days since the sensors and the fiber between sensors are well protected with the same diaphragm
162 material – just a small wavelength change is observed however, the equilibrium is not completely
163 reached. On the other hand, in the uncoated case (the case where the fiber is not protected with the
164 same diaphragm material), its recovery level is more evidenced (see Figs. 3 (c) and (d)), where from
165 Fig. 3 (d) we can observe that after 50 days in air the behavior tends to approximate the initial
166 conditions. The reason of this behavior is due to liquid absorption into the FBG from the unprotected
167 fiber outside the diaphragm. According to [21], for constant temperature, the Bragg wavelength
168 change is directly related to the water content inside the fiber. The water content in POF depends on
169 the relative humidity (RH) of the surrounding. Higher humidity means more water content in the
170 fiber. It consequently gives rise to a larger permeability coefficient for PMMA due to absorbed water
171 induced plasticizing effect on polymer materials [22], which leads to a larger water transportation
172 rate between the fiber and the surrounding. According to a recent work [11], the authors claim ~2.75
173 nm of wavelength shift when a PMMA mPOFBG is placed in an environment chamber setting the
174 RH from 10% to 90% at 25°C. In our case, we achieved a wavelength change of 3.05 nm when whole
175 fiber and sensors (mPOFBG embedded in diaphragms) are in contact with water. The reflected peak
176 power and wavelength variation values for each sensor submerged in water were extracted and are
177 summarized in Table 1. If we compare both cases – coated and uncoated fiber – the difference in terms
178 of peak power and wavelength shift is around 1.7 dB and 2.8 nm, respectively (in the best case – Table
179 1). This difference is significant for liquid level systems, so the best option from both cases will be to
180 use the fiber coated, where we have less changes of optical properties from sensors, however, long-
181 term drifts are measured as observed in Fig. 3. We noticed a considerable positive wavelength shift
182 in our embedded POFBG based sensors. Some potential reasons for that behavior come up with is
183 the following. As well shown in [23], the wavelength change of the POFBG sensor at large humidity
184 rates consists of the contributions from the fiber swelling and refractive index change induced by

185 relative humidity. Also, from [23] the authors conclude that the fiber volume change induced by
 186 water swelling takes a longer time to reach equilibrium than the refractive index change. On the other
 187 hand, the diaphragms embedding POFBG may play a key role, where the PMMA absorbs water,
 188 consequently the PMMA material swells after a long period in liquid and thereafter a significant
 189 change in its elastic modulus is achieved.
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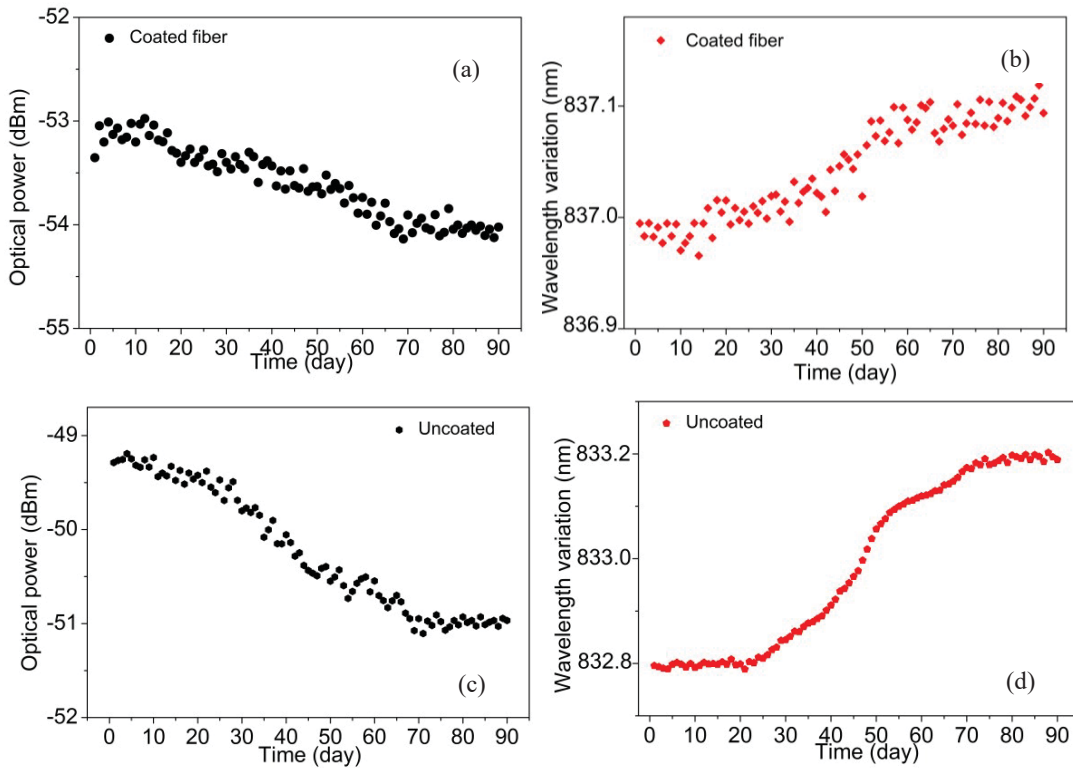
Figure 3. PMMA mPOFBGs reflection behavior and resonance wavelength variation over the time when the fiber is (a,b) coated and (c,d) uncoated, respectively. Sensors were submerged in water 90 days and afterwards 5 days in air.

Table 1. Reflected peak power and wavelength variation values for each sensor submerged in water during 90 days.

Sensor number	Reflected peak power variation (dB)		Wavelength shift	
	Coated	Uncoated	Coated	Uncoated
Sensor 1	1.38	3.58	140 pm	3.05 nm
Sensor 2	1.25	3.78	198 pm	2.99 nm
Sensor 3	1.78	3.48	185 pm	2.94 nm
Sensor 4	1.46	3.11	210 pm	3.18 nm
Sensor 5	1.57	3.35	159 pm	3.09 nm

Similarly, pressure sensors were fabricated to carry out some tests using JET A-1 aviation fuel. This fuel presents a density around 0.810 kg/L at 15 °C (less than water density, which is around 0.999 kg/L at 15°C [24]). Fig. 4 shows the case when the sensors are embedded in polyurethane resin diaphragms and thereafter placed in the fuel tank. The behavior from sensor 7 is shown in Fig. 4. From Figs. 4 (a) and (b), for the first case (all fiber coated), it can be observed that both reflected peak power and resonant wavelength show a slight change of their values after 90 days of experiment –

217 the reflected peak power decreased 1.21 dB and the total wavelength shift was 154 pm. In the second
 218 case (uncoated fiber), the reflected peak power variation shows no significant change for the first 10
 219 days and the resonant wavelength reaches a steady state except between 20th and 70th day, as shown
 220 in Figs. 4 (c) and (d), respectively. However, these changes were small when compared with the water
 221 case. The decrease of reflected peak power after 90 days of experiment was 1.85 dB and the total
 222 wavelength shift was 0.46 nm, of which 0.41 nm was the change resulting from the 20th day to 90th
 223 day. We noticed that PMMA material from POF does not absorb fuel and the reason for that small
 224 variation after 90 days is probably due to the amount of water in fuel, which is absorbed by PMMA
 225 [7]. The reflected peak power and wavelength variation values for the three sensors submerged in
 226 fuel were extracted and are listed in Table 2. From these results we can conclude that the PMMA
 227 POFBGs reflectivity and wavelength changes in fuel is much smaller than in water. Also, from both
 228 cases, coated and uncoated fiber, the difference in terms of reflected peak power and wavelength
 229 shift is less than 0.80 dB and 380 pm (see Table 2), respectively.
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233 **Figure 4.** PMMA mPOFBGs reflection behavior and resonance wavelength variation over the time when
 234 the fiber is (a,b) coated and (c,d) uncoated, respectively. Sensors are submerged in JET A-1 fuel.
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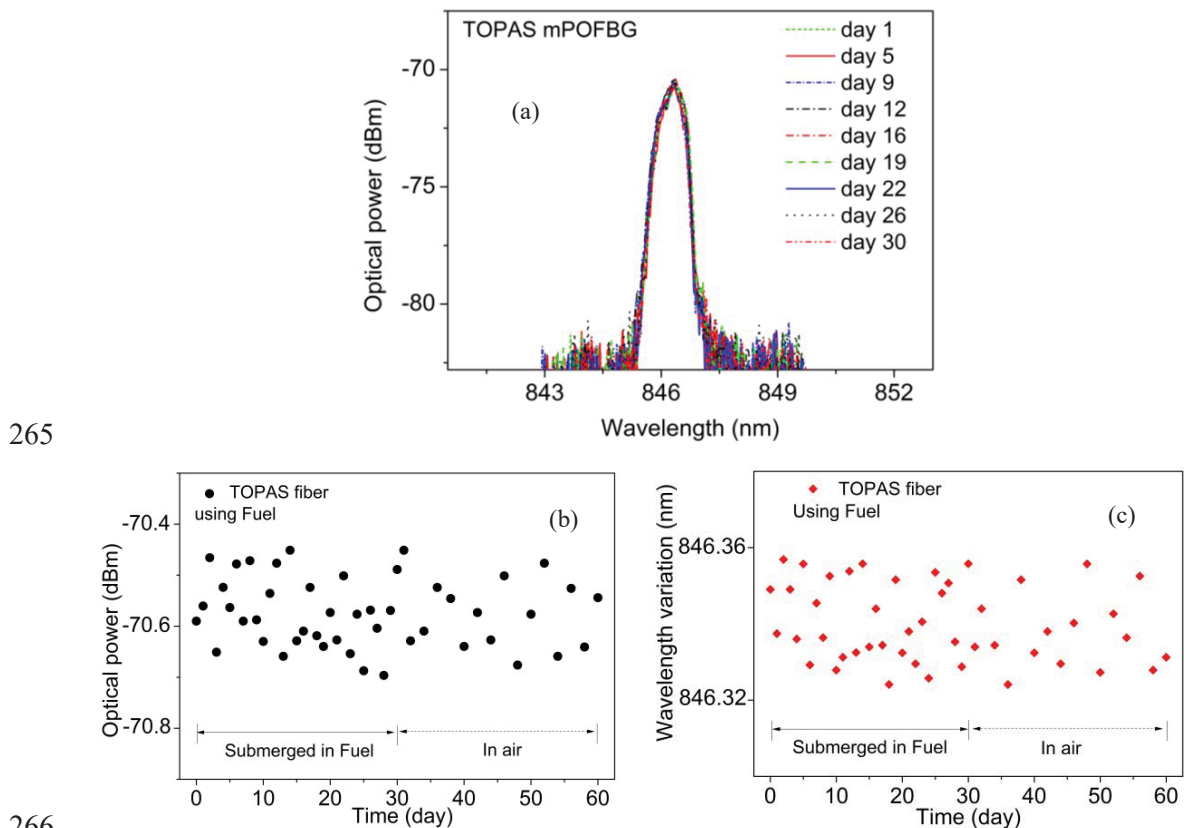
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Table 2. Reflected peak power and wavelength variation values for each sensor submerged in fuel.

Sensor number	Reflected peak power variation (dB)		Wavelength shift	
	Coated	Uncoated	Coated	Uncoated
Sensor 6	1.32	1.92	140 pm	0.50 nm
Sensor 7	1.21	1.85	154 pm	0.46 nm
Sensor 8	1.32	2.08	161 pm	0.54 nm

Although the coated case showed very low changes, indeed in both cases - using water and fuel - the equilibrium is not completely reached as observed in Figs. 3 and 4. Therefore, some experiments

249 were conducted using TOPAS mPOFBGs, which is an insensitive material to humidity as already
 250 reported in some works [9,10,25]. An array of two sensors (sensor 9 and sensor 10) based on TOPAS
 251 mPOFBGs embedded in polyurethane resin were produced and placed in contact with fuel to collect
 252 some measurements (reflected peak power and wavelength changes) over 30 days. Note that we use
 253 the TOPAS fiber unprotected between sensors (in direct contact with liquid). Fig. 5 (a) shows the
 254 sensor 9 and there are no significant changes in the TOPAS mPOFBG optical spectrum since the
 255 TOPAS fiber is insensitive to humidity. The wavelength shifts throughout the whole investigation
 256 for sensor 9 and sensor 10 were 39 pm and 36 pm, respectively, which is smaller than one third than
 257 the wavelength variation observed by coated sensors (compare Table 2 and Table 3). The reflected
 258 level for both sensors (sensor 9 and 10) presents no significant change – less than 0.35 dB in both cases
 259 (see Table 3). Fig. 5 (b) and (c) show the TOPAS mPOFBG reflection behavior and resonance
 260 wavelength variation over the time submerged in JET A-1 fuel, respectively. Indeed, the same
 261 behavior is kept when the sensors are out of fuel after 30 days. To conclude, TOPAS fiber is a
 262 promising fiber type to use in this kind of applications where a significant quantity of water may be
 263 present.
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 267 **Figure 5.** (a) Reflected spectra of the TOPAS mPOFBG over 30 days when the sensor 9 is submerged in fuel.
 268 TOPAS mPOFBGs (b) reflection behavior and (c) resonance wavelength variation over the time for sensor
 269 9 (when submerged in fuel during 30 days and afterwards in air during 30 days).
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Table 3. Reflected peak power and wavelength variation values for each sensor submerged in fuel using TOPAS mPOFBGs.

Sensor number	Reflected peak power variation (dB)	Wavelength shift (pm)
Sensor 9	0.26	39
Sensor 10	0.34	36

5. Conclusions

For the first time, tests in water and JET A-1 fuel to investigate the time-dependent variation of both the reflectivity and resonance wavelength of mPOFBG array sensors embedded in diaphragms were reported. Both the reflection and the resonance wavelength shift were monitored over 90 days and two studies were made: the first case when all fiber is protected with the same diaphragm material and the second study when the fiber between sensors is uncoated – in direct contact with liquid. We concluded that the sensors in fuel can experience less change in terms of reflectivity and wavelength when compared in water because the aviation fuel has the ability to hold a certain amount of dissolved water. Also, when we used sensors in water, significant differences can be observed when we coat all fiber comparing with the uncoated case, achieving long-term drifts, possibly due to the sensitivity of PMMA to water. In the fuel case, these differences are significantly reduced as were shown. Although the coated case showed very low changes, indeed in both cases - using water and fuel - the equilibrium is not completely reached. Tests with an alternative POF material – TOPAS – revealed a much improved behavior, suggesting that TOPAS fiber should be a good option for long-term fuel monitoring applications. The study may be a very useful property for liquid level monitoring systems and even in corrosive environments such as fuel or oil tanks.

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