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Optofluidic gutter oil discrimination based on a hybrid-waveguide coupler in fibre

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Discriminating edible oils from gutter oils has significance in food safety, as illegal gutter oils cannot meet a variety of criteria such as the acid value, peroxide value and quality. To discriminate these illegal cooking oils, we propose an ultrasensitive optofluidic detection method based on a hybrid-waveguide coupler. Prior to the straight waveguide inscription in the cladding of the silica tube using a femtosecond laser, a section of coreless fibre is firstly spliced with the ST to supply a platform for the inscription of an S-band waveguide. Then a pair of microfluidic channels are ablated on the ST using the fs laser to enable liquid analytes to flow in and out of the air channel. In the transmission spectrum, a unique resonant loss dip can be observed, which is produced by coupling the light from the laser inscribed waveguide to the liquid core when the phase-matching condition is met. This hybrid-waveguide coupler with a simplified structure realizes dynamic optofluidic refractive index sensing with an ultrahigh sensitivity of $-112\,743\text{ nm RIU}^{-1}$, a detection limit of $2.08 \times 10^{-5}\text{ RIU}$ and a refractive index detection range from 1.4591 to 1.4622. This novel method can be used for food safety detection, specifically, for the discrimination of gutter oils.

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Introduction

Edible oils are daily essentials for many people and the discrimination between healthy vegetable oil and harmful gutter oil is essential. These illegal gutter oils are second-hand oils refined from cooking waste, gutters, drains, and animal fat. Through a series of simple processes, such as collection, preliminary filtration, boiling and refining, gutter oil is packed and sold to low-end restaurants and small canteens. Intake of this kind of oil is detrimental to people's health, as it cannot meet health criteria such as the acid value, peroxide value and quality. Gutter oil has fewer unsaturated fatty acids than normal vegetable oils and therefore it has a lower refractive index (RI) than normal edible oils (1.457–1.48),¹ which enables optical devices to distinguish illegal oil from edible ones. Recently, many optofluidic methods have been applied to the analysis of liquids and ingredients, such as Fabry–Pérot resonators,² liquid lenses,³ liquid waveguides⁴ and hybrid waveguides.⁵

Hybrid-waveguide couplers consisting of both liquid and solid cores exhibit many unique properties. According to the coupled mode theory, directional light coupling in two adja-

cent parallel waveguides will take place when the phase-matching condition is met. Because of the dramatic difference in material dispersion between solids and liquids, the phase-matching condition can be met only around the intersection of two dispersion curves. Therefore, the directional coupling in hybrid waveguides can only take place in a local wavelength region, resulting in a unique resonant loss dip in the transmission spectrum. The resonant dip is sensitive to a RI change of the liquid core and this phenomenon is useful for sensing applications. For example, benefiting from the high thermo-optics coefficient of liquids ($\sim 10^{-4}\text{ RIU }^{\circ}\text{C}^{-1}$), hybrid-waveguide configurations have already been used for temperature measurements,^{6,7} strain measurements,^{8,9} hydrogen concentration detection¹⁰ and RI measurements.¹¹ All the above mentioned devices based on hybrid waveguides are

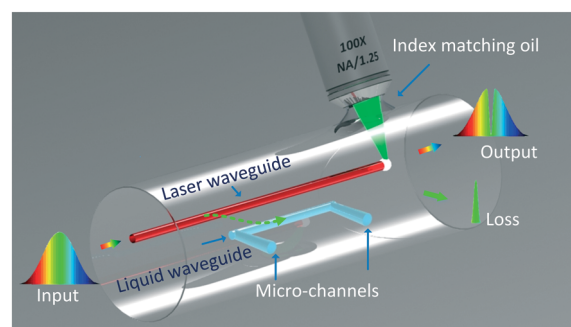


Fig. 1 Schematic diagram of a hybrid-waveguide coupler.

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embedded in photonic crystal fibres (PCFs) whose cladding is distributed in periodical air holes, by means of selective infiltration.¹²

In this paper, a hybrid-waveguide coupler comprised of a solid core and a liquid analyte filled core is demonstrated in a pure silica tube (ST), shown in Fig. 1. Because the RI difference between gutter oil and edible oil can lead to different resonant wavelengths, the discrimination of gutter oil can be achieved by detecting the spectral response at the output. Taking advantage of the 3D micromachining capacity of the fs laser, the solid core can be inscribed in the ST consisting of one air channel and pure silica cladding to simplify the structure of directional couplers. Waveguide inscriptions using a femtosecond (fs) laser in glass materials have been widely studied in recent years,¹³ *i.e.* in-fibre Mach-Zehnder interferometers,^{14,15} beam splitters,^{16,17} fibre surface waveguides,¹⁸ and distributed 3D Bragg grating waveguides (BGWs).^{19,20} Two micro-channels are fabricated in the ST to enable the liquid analyte to flow in and out, which enables this chip to be reused. The light propagating in the laser-inscribed waveguide can be efficiently coupled into the liquid core when the phase-matching condition is met. The unique resonant loss dip observed in the transmission spectrum can be used to trace the RI variation of the analyte injected in the ST. The proposed device is capable of detecting the RI difference between oils and discriminating gutter oil from normal vegetable oils, so it is promising for food safety detection applications.

Device fabrication

The flowchart of the device fabrication process is illustrated in Fig. 2. Step 1: a section of lead-in single mode fibre (SMF) is firstly spliced with a section of coreless fibre (CF) and then the CF is cut off ~ 2 mm away from the splicing point, as shown in the schematic diagram in Fig. 2(a). This section of the CF supplies a platform for an S-band waveguide inscription. The optical microscopy image of the related splicing point is shown on the right-hand side of Fig. 2(a). Step 2: a section of the ST (TSP005, Polymicro Technologies) with an air channel diameter of $5\ \mu\text{m}$ and a cladding diameter of $126\ \mu\text{m}$ is spliced with the CF and a ~ 1 cm-long ST is reserved after cleaving, as shown in the schematic diagram in Fig. 2(b). In the process of splicing, both the CF and the ST must be fully fused to eliminate the impurities attached on the fibre end face. Otherwise, the impurities will dramatically decrease the damage threshold of the material, and then significantly degrade the performance of the laser inscribed waveguide at this splicing point.²¹ As shown on the right-hand side of Fig. 2(b), in order to eliminate the impurities, the air channel of the ST is locally collapsed when the silica is totally fused. Step 3: as shown in Fig. 2(c), the component obtained in the last step is spliced to a lead-out SMF with a core-offset of $15\ \mu\text{m}$. The introduced core-offset will make the whole device more compact. Step 4: after completion of the splicing process, the waveguide inscription is then carried out using the

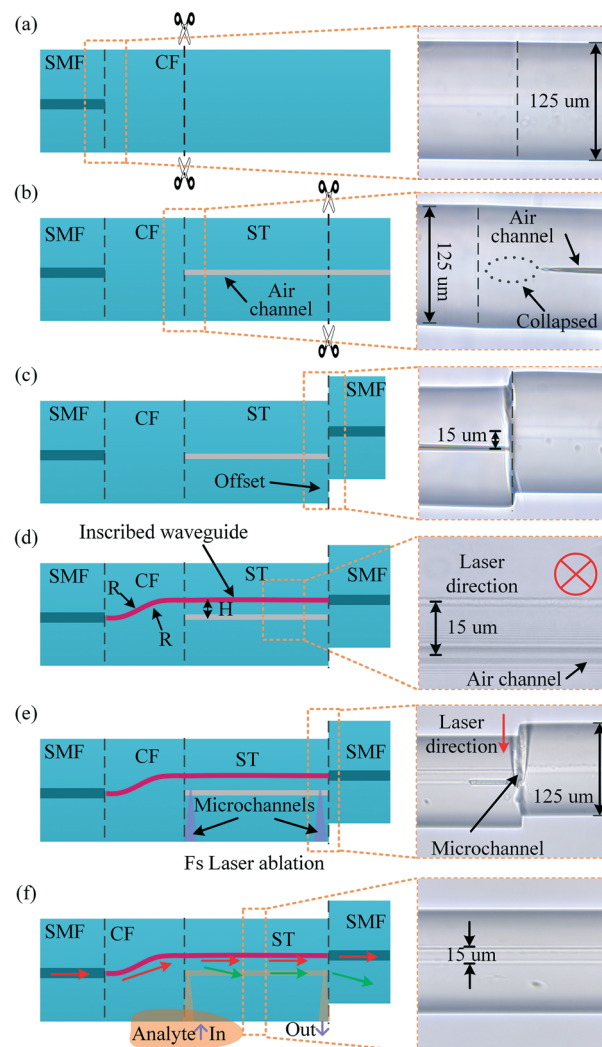


Fig. 2 Flowchart of device fabrication. (a) Fusion splicing between the lead-in SMF and the CF, (b) fusion splicing between the CF and the ST, (c) offset splicing between the ST and the lead-out SMF, (d) fs laser waveguide inscription, (e) microchannel fabrication and (f) analyte injection.

fs laser. In this step, the fs laser (PHAROS, 532 nm, 250 fs and 200 kHz) is employed and an oil-immersed objective lens with an NA value of 1.25 is selected to eliminate the aberration caused by the cylindrical morphology of the optical fibre. The lateral offset of the S-band waveguide inscribed in the CF (as shown in Fig. 2(d)) is adjusted to $15\ \mu\text{m}$ (H) to match the core-offset of the lead-out SMF and the radius (R) of the S-band waveguide is designed to be $50\ \mu\text{m}$. The translation velocity of the fibre is set as $200\ \mu\text{m s}^{-1}$ and the laser pulse energy is set as $250\ \text{nJ}$. The right-hand side of Fig. 2(d) shows the top view of the inscribed waveguide with the laser beam irradiation perpendicular to the figure. Step 5: microchannels connecting the air channel with the surrounding environment are created using fs laser ablation to allow the analytes to flow in and out. These two micro-channels are positioned at both ends of the air channel to ensure that the whole micro-channel can be fully filled with analytes. As shown in

Fig. 2(e), one of the micro-channels has been created, despite the rough inner surface of this channel, which is caused by fs laser pulses. As these channels do not intersect with the laser-inscribed waveguide, the performance of the proposed device will not be affected. Step 6: the final process is to inject analytes into the air channel, and then the fabrication of the hybrid waveguide is accomplished. As shown in the schematic diagram of Fig. 2(f), the light transmitting in the lead-in SMF is coupled into the laser inscribed waveguide (red arrows). Until the light propagates into the ST, some light will be coupled into the liquid core when the phase-matching condition is met (green arrows), and a unique resonant loss dip can be observed in the transmission spectrum because this light propagating in the liquid core cannot be collected by the lead-out SMF and will eventually fade away in the fibre cladding.

The optical microscopy images of the cross-sectional profile of laser inscribed waveguides and the ST are shown in Fig. 3(a) and (b), respectively. There are segments of both negative (black) and positive (white) RI modifications in the laser inscribed waveguide, as shown in the inset of Fig. 3(a). The mode field profile of the laser-inscribed waveguide at a wavelength of 1550 nm is shown in Fig. 3(c), with a width of 10.32 μm in the vertical direction and 9.82 μm in the horizontal direction. The propagation loss of the laser inscribed waveguide is estimated to be 1.2 dB cm^{-1} using a cut back method, which can be improved by optimizing the inscription parameters (translation velocity, laser pulse energy, *etc.*).²² There are many vegetable oils, including coconut oil, butter oil and mustard oil, with different RI values ranging from 1.457–1.48.¹ In this experiment, a similar standard RI liquid (Cargille labs) shown in Fig. 3(d) with a value of 1.460 at 25 $^{\circ}\text{C}$ is used as a kind of oil to test the proposed device,

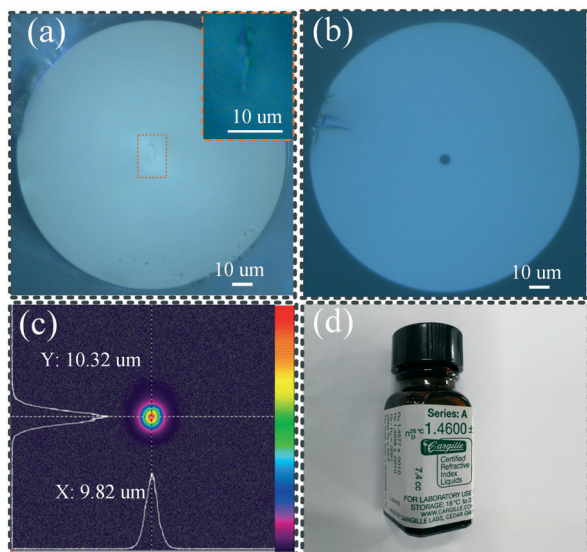


Fig. 3 Detailed cross-sectional profiles of (a) the inscribed waveguide, (b) the ST and (c) the near field mode of the inscribed waveguide at a wavelength of 1550 nm. (d) Standard refractive index liquid with a value of 1.46 at 25 $^{\circ}\text{C}$.

as this purchased oil that is well calibrated by the manufacturer can improve the accuracy of the obtained results. Furthermore, the calibrated RI values can be used to calculate the material's dispersion, which is significant for accurate numerical simulations.

The transmission spectrum of the proposed device was recorded before the injection of the analyte (step 4 in fabrication), as shown in Fig. 4. The total insertion loss is estimated to be 4.98 dB and the observed weak interference may be introduced by the offset of the mode field between the SMF and the laser inscribed waveguide. After the analyte injection, the transmission spectrum was recorded and the unique resonant loss dip was observed around 1550 nm with a resonant intensity of 9.83 dB. It can be easily observed that the full-width-at-half-maximum (FWHM) value of 62.5 nm is wider than that seen in previous reports^{8,11} and it may be explained by the laser inscribed waveguide not being strictly parallel with the liquid core.

Results and discussion

In this experiment, an electric temperature oven with a precision of 0.1 $^{\circ}\text{C}$ is employed to control the ambient temperature. Because the thermo-optics coefficient of the RI liquid (-3.89×10^{-4} RIU $^{\circ}\text{C}^{-1}$) is much higher than that of pure silica (8×10^{-6} RIU $^{\circ}\text{C}^{-1}$), the RI response can be tested by varying the ambient temperature from 25.7 $^{\circ}\text{C}$ to 24.5 $^{\circ}\text{C}$ with a step of -0.2 $^{\circ}\text{C}$. Correspondingly, the RI value of the analyte in the fluidic channel is changed from 1.459727 to 1.460194 with a step of 0.0000778. The resonant dip is shifted towards shorter wavelengths (blue shift) with the analyte RI increasing and the evolution of the transmission spectrum is recorded and shown in Fig. 5(a). The ultrahigh RI sensitivity of $-112\,743$ nm RIU $^{-1}$ is obtained by tracing the resonant wavelength and the linear fitting relationship between the resonant wavelength and the analyte's RI value is shown in Fig. 5(b). The detection range from 1.4591 to 1.4622 can be achieved using a normal optical spectrum analyser (OSA) with a detection wavelength ranging from 1250 nm to 1650 nm. The detection limit (DL) reports the smallest measurable

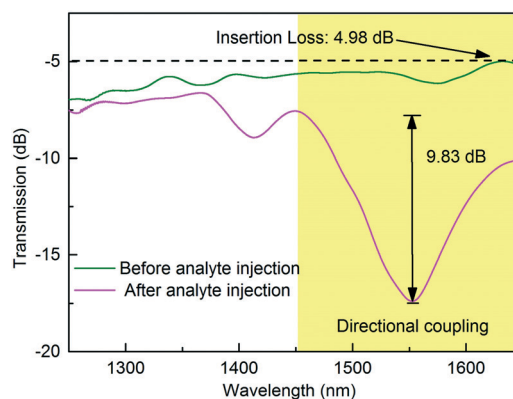


Fig. 4 Transmission spectra of the proposed device before and after analyte injection.

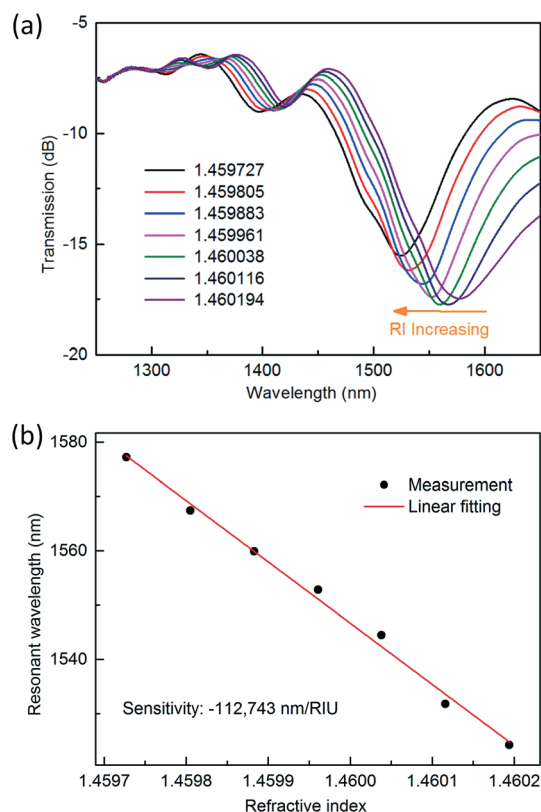


Fig. 5 RI response of the proposed device. (a) Evolution of the transmission spectrum with the increase of the analyte's RI and (b) linear fitting relationship between the resonant wavelength and the analyte's RI.

RI change, affected by the sensitivity (S) and the sensor resolution (R) of the devices: $DL = R/S$. The resolution of this optofluidic sensor is dominated by the FWHM (62.5 nm).²³ At the same time, with a signal to noise ratio (SNR) of 50 dB, a DL of 2.08×10^{-5} RIU can be obtained, which enables this device to measure tiny variations of an analyte's RI.

In the numerical simulation, a finite element method is employed to analyse the transmitting modes of the hybrid waveguides. The material dispersion of pure silica and the standard liquid's RI has also been considered.⁸ The dispersion curve of the liquid is calculated using Cauchy's dispersion formula:

$$n(\lambda) = a + \frac{b}{\lambda^2} + \frac{c}{\lambda^4}$$

with a RI of $n_D = 1.4600$, $n_C = 1.4577$ and $n_F = 1.4658$, which are calibrated by Cargille labs, and the dispersion curve of the liquid can be approximately calculated. The thermo-optics coefficient (-3.89×10^{-4} RIU $^{\circ}\text{C}^{-1}$) can be calculated by measuring the RI of standard refractive index liquid at each temperature using a high precision Abbe type refractometer. The model is constructed according to the geometrical parameters shown in Fig. 3, including the inner diameter, the

outer diameters and the mode field profile of the laser inscribed waveguides.

The mode profiles of the liquid and solid cores at the operation wavelength of 1550 nm are shown in Fig. 6(a) and (b), respectively. Different to that of the liquid core, the modelled mode profile of the laser inscribed waveguide is asymmetric for a better match with the real mode profile observed in this experiment. The directional coupling can be observed at the resonant wavelength and the mode profile is plotted in Fig. 6(c). In order to investigate the RI response of the hybrid-waveguide coupler, mode dispersion curves of the liquid and solid cores have been calculated at the RI values of 1.460194 and 1.459727, respectively, as shown in Fig. 6(d). As the effects induced by the RI vibrations of the liquid core can be neglected for the laser inscribed waveguide, the second mode dispersion curve of the laser inscribed waveguide is therefore not plotted in Fig. 6(c). The intersection between the mode dispersion curves of the liquid and solid cores, where the phase-matching condition is met, has a blue-shift of 51.2 nm when the liquid RI value varies from 1.459727 to 1.460194. Accordingly, the RI sensitivity of $-108\,016$ nm RIU⁻¹ is calculated, which agrees well with the one obtained in the experiment. The insets of Fig. 6(d) show the mode profile of the liquid core (at a RI value of 1.460194) at operation wavelengths of 1490 nm and 1590 nm, respectively. The weak coupling shown in the insets demonstrates that the solid core can couple with the liquid core in a 100 nm-wide wavelength region, which is one of the reasons why the FWHM of the resonant loss dip (in Fig. 4) is wider than that of the PCF based hybrid waveguide devices. The spectrum performance of the proposed device may be improved by increasing the distance between the liquid and

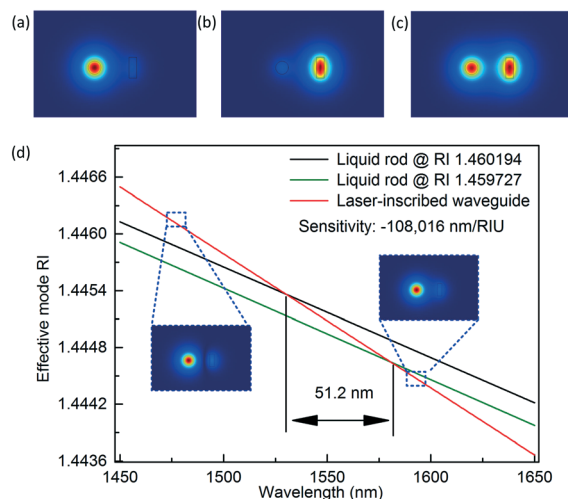


Fig. 6 Mode profiles of (a) the analyte channel and (b) the laser inscribed waveguide outside the resonant wavelength region; (c) mode profile inside the resonant wavelength region; (d) phase-matching relationship of the coupling between the laser inscribed waveguide and the analyte channel with RI values of 1.460194 and 1.459727, respectively. The insets show mode profiles of the analyte channel at operation wavelengths of 1490 nm and 1590 nm, respectively.

solid cores to achieve a narrower FWHM. However, it may lead to a longer coupling length and, consequently, result in a greater insertion loss.

The proposed scheme works for analytes having a RI slightly higher than that of the background, and therefore, it can also be applied to aqueous solutions if the fibre is made of polymer materials such as Teflon with RI values ranging from 1.27 to 1.31.²⁴

Conclusions

In conclusion, an in-fibre hybrid-waveguide coupler comprised of two solid and liquid cores has been demonstrated both with experiments and simulations, with an experimental ultrahigh RI sensitivity of $-112\,743\text{ nm RIU}^{-1}$ and a detection limit of 2.08×10^{-5} RIU. This device is able to discriminate gutter oil from vegetable oils due to its ability to detect tiny vibrations in the RI value of oil. Some of the light propagating in the laser inscribed waveguide is coupled into the liquid core when the phase-matching condition is met and then the resonant loss dip can be produced. To achieve a reusable chip, microfluidic channels are created in the ST using fs laser ablation to enable liquid analytes to flow in and out of the air channel. It is worth noting that this device has achieved one of the highest RI sensitivities ($\sim 10^5\text{ nm RIU}^{-1}$), compared with other RI sensors based on micro/nanofibre Bragg gratings ($\sim 10^2\text{ nm RIU}^{-1}$),²⁵ plasmonic interferometric sensors ($\sim 10^2\text{ nm RIU}^{-1}$),²⁶ whisper gallery mode resonators ($\sim 10^2\text{ nm RIU}^{-1}$),²⁷ long period fibre gratings ($\sim 10^3\text{ nm RIU}^{-1}$),²⁸ FP cavities ($\sim 10^3\text{ nm RIU}^{-1}$)²⁹ and surface plasmon resonance ($\sim 10^3\text{ nm RIU}^{-1}$).³⁰ The DL of this device (2.08×10^{-5} RIU) is comparable with that of an SPR-based sensor ($\sim 10^{-5}$ RIU)³¹ and a Mach-Zehnder interferometer ($\sim 10^{-5}$ RIU),³² and is higher than that of sensors based on localized surface plasmon resonance ($\sim 10^{-4}$ RIU)³³ and partial refraction ($\sim 10^{-2}$ RIU).³⁴ The proposed device can have applications in food safety detection, specifically in discriminating the quality, category and purity of oils. Furthermore, this hybrid-waveguide coupler with low cost and a simplified structure is used for dynamic ultrasensitive RI measurements and is expected to have potential applications in the detection of aqueous solutions.

Conflicts of interest

There are no conflicts of interest to declare.

Acknowledgements

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