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Light scattering and rheological effects in an optical fibre coupled nanoparticle suspension

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ABSTRACT

This study forms the first part of research into enhancing the forward and back scattering of light in an optical fibre using nanoparticles (NPs). This approach has the potential to enhance the sensitivity of optical fibre sensing by increasing the signal-to-noise ratio. The work described in this paper is focused on understanding the scattering of light by a suspension of NPs in refractive index matching liquid. It was noted early in the experimental work that rheological effects related to the viscosity and flow of the liquid affect the scattered light measured and therefore these effects are considered in the analysis. Gold nanoparticles in the tens to hundreds of micrometre size range were selected as the scattering particles based on their optical properties. These are suspended in a refractive index liquid with a similar refractive index to the optical fibre core. Effort was needed to transfer the NPs from their aqueous sodium citrate solution to the paraffin based solution. We investigated two types of interaction with the optical fibre: (i) dropping the NP suspension onto the end of a single-mode optical fibre and (ii) using the NP suspension as an interface between two single-mode optical fibres. It was noted that the surface tension of the liquid, the diameter of the fibre and the spacing between the fibres in case (ii) influence the reflected and transmitted light. In case of excess liquid, droplets flowed down the fibre and interestingly in case (ii) modified the reflected and forward transmitted light as it passed across the fibre interface. Our initial findings are that the influence of the gap between two optical fibres decreased after dropping refractive index liquid into the gap after fibre collimation, which is a beneficial result for understanding the influence of scattered light from a liquid containing NPs. Note, the position between the two fibres can also change due to the weight of the droplet and the fibre ends had to be re-collimated to investigate the influence of the moving droplets. These results will be expanded by additional experiments and modelling of the scattering from the nanoparticales and droplets.

Keywords: Light scattering, rheological effect, optical fibre, nanoparticle

1. INTRODUCTION

Introducing nanoparticles (NPs) into optical fibre has the ability to change the scattered light in optical fibre. One application is to dope NPs into the core of optical fibres to improve the sensing ability of optical backscatter reflectometry to make up for the deficiency of low Rayleigh scattering in conventional optical fibre. For example SMF-28 is generally used in optical backscatter reflectometry, which is a limitation for this sensing technique.[1–3] Recent research has reported experimental results that by doping NPs into optical fibres, the back scattered light increased by 50 dB in an optical fibre doped with MgO NPs compared to SM optical fibre[4] in optical backscatter reflectometry. The application was 3D shape sensing with optical backscatter reflectometry in a spatial multiplexing way.[5] Apart from MgO doping, research has reported that high temperature distributed sensing has a better stability with optical fibre doped with gold-zirconia NPs than SMF-28.[6] These new developments encourage researchers to perform further research about NPs in optical fibres and its applications.

The manufacture of NP-doped optical fibres with a specific NP-size and distribution is not easy to obtain in a low cost way in an optical laboratory. In order to study the scattered light from NPs in optical fibres easily and

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in a controllable way, we expect that by placing a drop of liquid containing NPs between two collimated optical fibres and measuring the transmitted and reflected light, the scattered light collected by the optical fibre can be investigated.Note that the strong light reflection from the ends of the two fibres will have a negative effect on the detection of back scattered light, especially when the size and the concentration of the NPs are low. Therefore, transferring NPs to refractive index match liquid may likely reduce this negative effect.

In our current work, we used gold NPs to scatter light due to their wide availability not only from laboratory synthesis but also from commercial products and their good size and shape controllability.[7,8] These are beneficial for analyzing scattered light theoretically and testing experimentally. In general, it is easier to synthesis gold NPs in a size and shape controllable process in aqueous solution than in organic solvents[9] and the gold NPs we bought from Sigma-Aldrich are also in aqueous solution. Thanks to previous research about transferring gold NPs from an aqueous solution to organic solvent with thiol ligands,[9,10] it may become possible to transfer gold NPs from aqueous solution based on citrate buffer to refractive index matching liquid based on paraffin oil in this paper.

This paper includes five sections. Section 1 is the introduction. In Section 2, the methods and results of transferring gold NPs from aqueous sodium citrate solution to a refractive index matching liquid based on paraffin oil are shown. By dropping liquid on one optical fibre and between two optical fibres, the morphology of the liquid on the optical fibre tip and the intensity of transmitted and reflected light with the change of relative distance between two optical fibres were investigated in Section 3 and Section 4 respectively. Section 5 is the conclusion.

2. GOLD NANOPARTICLE TRANSFER

2.1 Experiment Materials

The chemicals used in the experiment for transferring Gold NPs include oleic acid (Sigma-Aldrich), N,N'-dicyclohexylcarbodiimide (Sigma-Aldrich), 2-aminoethanethiol (Sigma-Aldrich) and gold nanoparticles suspension (10 nm diameter Gold NPs stabilized suspension in citrate buffer, Sigma-Aldrich) and methanol. The refractive index of the index matching liquid paraffin oil is 1.48 which is similar to the refractive index of optical fibre of about 1.5. The gold NPs suspension needs to be transferred to an organic liquid for the optical fibre.

2.2 Method and Results

The thiol-termination ligand was generated based on the method of López-Millán et al.[9] The process of generating gold NPs with thiol-termination ligands is shown in Figure 1.

0.3654 g N,N'-dicyclohexylcarbodiimide was added to a test tube and dissolved in 4 mL of methanol. 0.57 mL oleic acid was slowly added to the test tube with a pipette. 0.1364 g 2-aminoethanethiol was added to the test tube and stirred at 750 rpm for 2 h. The tube was then put into a centrifuge for 20 min at 4400 rpm. The liquid supernatant was then poured slowly into another tube (Supernatant 1) and then put into fridge at 2 °C for 16.5 h. After removing the test tube from the fridge and it was put into the same centrifuge for 20 min at a speed of 4400 rpm. The liquid supernatant was then purified with a rotary evaporator and then slowly poured into another test tube (Supernatant 2).

1 mL of Gold NPs suspension was added to a test tube. According to our experience from earlier tests, the quantity of the solution containing thiol-termination ligand and methanol added is vital for gold NPs successfully transferring from citrate buffer to dichloromethane. Too much solution containing thiol-termination ligand or not suitable volume of methanol will lead to the failure of gold NPs transfer from citrate buffer to dichloromethane. In this case, a droplet of liquid from the tube labeled with Supernatant 2 was added to the test tube. Stirring gently the colour changed from transparent red to turbid light red. Then the tube was left at room temperature. After 50 min, about 1.5 mL paraffin oil was added to the tube and then 2 mL methanol(shown in Figure 2a). As shown in Figure 2a, the top layer is methanol (density of methanol is $0.7918\,g/cm^3$, the middle layer is paraffin oil (density of paraffin oil is $0.85\,g/cm^3$) and the bottom layer is gold NPs suspension (density of water is $1\,g/cm^3$). Stirring gently with a stirring needle (shown in Figure 2b), a red colour appeared in the top layer of liquid (shown in Figure 2c). The red colour is caused by light scattered by the gold NPs, so this confirms the gold NPs have been transferred to paraffin oil but their quantity is low according to the colour strength. Some of the top layer liquid was then removed with a syringe and used to drop liquid onto the tip of optical fibres in Sections 3 and 4.

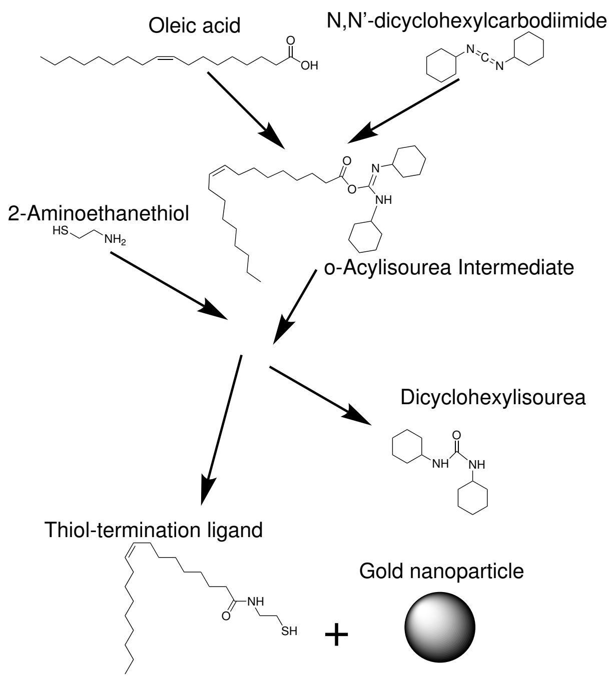


Figure 1: The process of generating gold nanoparticles with thiol-termination ligand based on the method of López-Millán et al.[9]

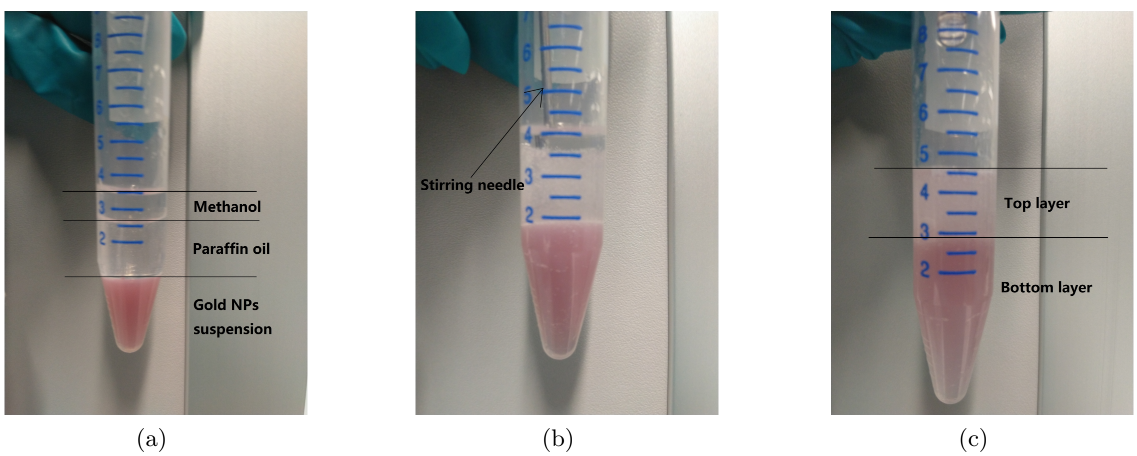


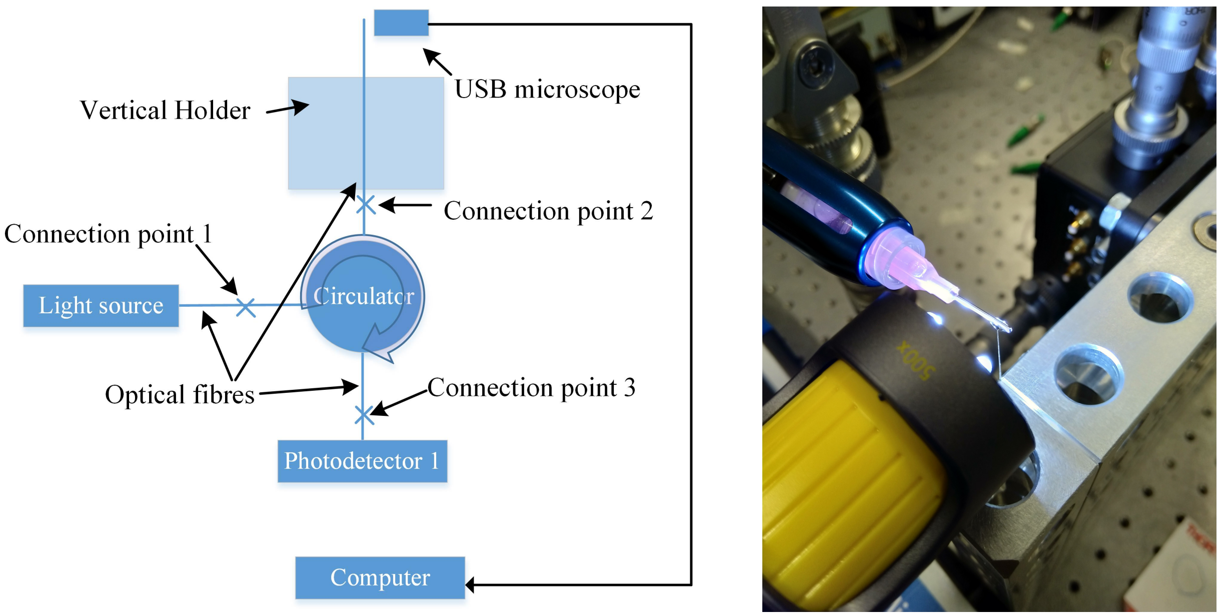
Figure 2: The process of transferring Gold NPs from citrate buffer to paraffin oil (a) Photo taken before stir without stir needle; (b) before stirring with the needle; (c) after stirring

3. DROP REFRACTIVE INDEX LIQUID ON ONE FIBRE

3.1 Experimental Setup

The experimental setup is shown in Figure 3a. The setup includes a superluminescence diode light source (FESL-1550-20-BTF, Frankfurt Laser Company) which is controlled by a laser diode controller (ThorLabs LDC 205) and a temperature controller (ThorLabs TED200C), an optical circulator (single mode, ThorLabs), photodetector 1 (ThorLabs Optical Power Meter PM20), a USB microscope (500x Fixed USB Digital Microscope from Edmund Optics) and a computer.

The light whose central wavelength is around 1550 nm was emitted from the light source and went into the optical circulator from connection point 1. Then, the light propagated to one end of an optical fibre (Single mode optical fibre 1550BHP, ThorLabs) through connection point 2. The coating layer of the output end of the optical fibre was removed when the fibre was cleaved. This optical fibre was held by tape in a vertical holder with about 1 to 2 cm fibre optic length extending out of the vertical holder. A syringe containing refractive index liquid with gold NPs was put near the end of the optical fibre. A USB microscope was used to give magnified views of the surface of the optical fibre when refractive index matching liquid with gold NPs was being dropped on the end of the optical fibre. A photo of the end of the optical fibre in the vertical holder is shown in Figure 3b. The size of the bubble is controlled by a Precifluid volumetric dispenser. Light reflected from the end of the optical fibre travelled through the optical circulator to photodetector 1.



(a) (b)

Figure 3: Experimental setup (a) Structual diagram; (b) Photo of the optical fibre end

3.2 Results and Discussion

The top layer liquid was brought from test a test tube to a syringe which had been connected to a fluid dispensing systems (Precifluid volumetric dispenser) to drop liquid onto the optical fibre. The volume of liquid in the bubble was 0.020 52 cc with from the syringe controlled by a pedal on the fluid dispensing system. After several liquid pushed from the syringe and a big droplet was formed at the end of optical fibre. In Figure 3b, the top layer liquid had been taken into the syringe, so the colour of the syringe appeared light red.

Figure 4 is the close-up view of the end of the optical fibre as seen by the USB microscope. This photo has been rotated 180° in order to show the fibre in the vertical position. Because the diameter of the cladding of this optical fibre is about 125 µm, the height of the liquid which was dropped on the end of the fibre can be estimated by comparing to the cladding diameter of 125 µm.

Four photos in Figures 5 show the process of dropping liquid onto one end of an optical fibre. Figure 5a is a photo showing the end of optical fibre immersed in the big droplet (1 to 2 mm). Note that this droplet is not big enough to leave the needle of the syringe. Figure 5b shows the state after one droplet left the syringe part of the liquid flowed along the optical fibre and part of the liquid existed on the end surface of the optical fibre. With the flowing of the liquid, the position of the optical fibre was changing, which can be seen in Figure 5b to Figure 5d. Therefore, the flow of the liquid disturbs the static optical fibre. From Figure 5b, it can be estimated that the height of the liquid on the end of the optical fibre is about 40 µm. Note, the position of the optical fibre was changing due to the weight of the flowing droplet.

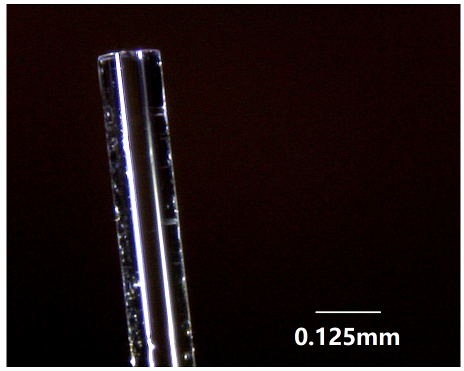
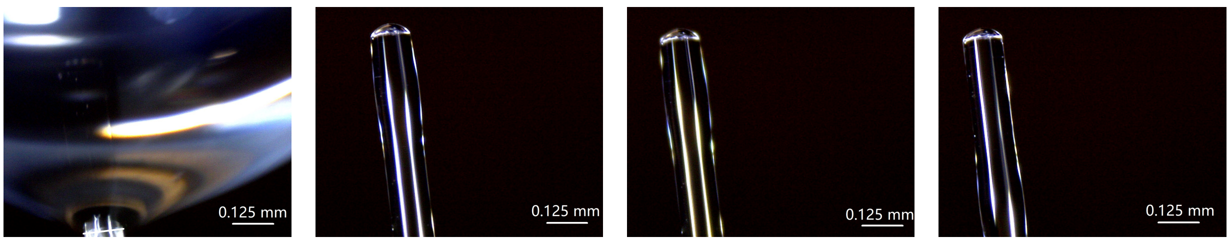


Figure 4: A close look of the end optical fibre from USB microscope



(a) (b) (c) (d)

Figure 5: The process of dropping liquid to the end of optical fibre (a) forming droplet on the end of optical fibre; (b)-(d) three instantaneous status of after dropping liquid on the end of optical fibre

By dropping liquid on the end of one optical fibre, the height of the liquid on the end of the optical fibre was obtained, which gave a direct view of the situation of liquid on an optical fibre. In the next section, the reflected light and transmitted light influenced by liquid between two optical fibres were discussed versus distance.

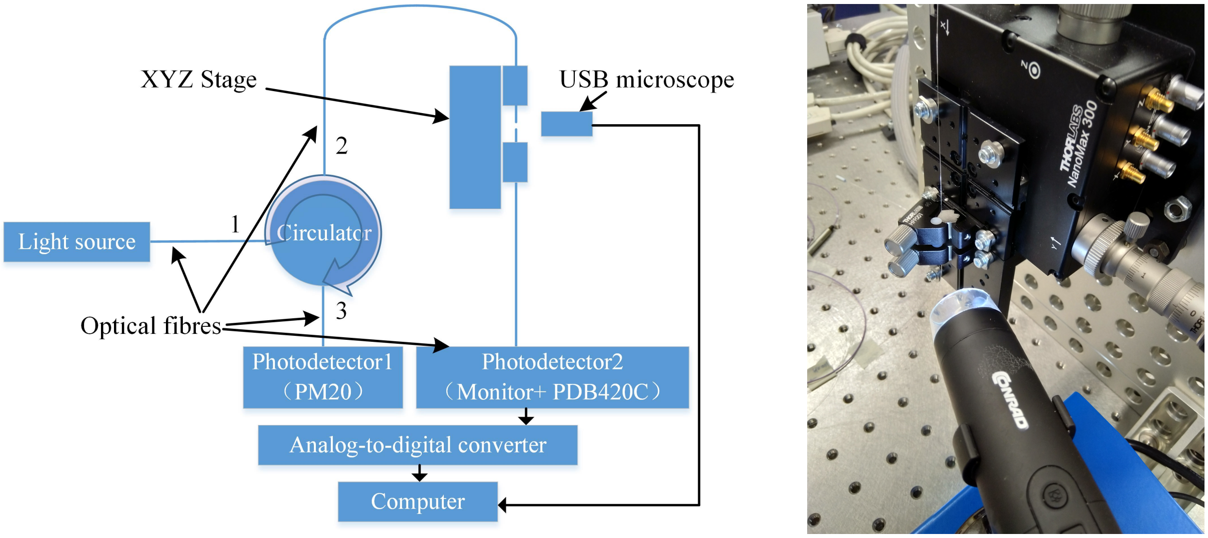
4. DROP REFRACTIVE INDEX LIQUID BETWEEN TWO OPTICAL FIBRES

4.1 Experimental Setup

The second experimental setup is shown in Figure 6a. The setup includes the same light source (FESL-1550-20-BTF) controlled by a laser diode controller (LDC 205) and a temperature controller (TED200C), an optical circulator from Thorlabs, photodetector 1 (PM20), photodetector 2 (Single channel of balanced detector PDB420C), an XYZ Stage (3-axis stage from ThorLabs), analog-to-digital converter (Picoscope), a USB microscope (Conrad) and a computer.

In Figure 6a, light was emitted from the light source and went into optical circulator. Then, the light propagated into a single mode optical fibre, whose end was clamped at an upper position of the XYZ stage. Then the light was coupled into another optical fibre (1550BHP), whose end was also clamped on the XYZ stage but at the lower position. The coating layer of both of the ends of optical fibres were removed and the end faces were cleaved. A syringe containing refractive index liquid with gold nanoparticles was put near the end of the optical fibre at the upper position. The USB microscope was used to give an enlarged view of the gap between two optical fibres. A photo of the XYZ stage is shown in Figure 6b. Reflected Light was collected by photodetector 1. The transmitted light was collected by photodetector 2 and then the signal was converted by the analog-to-digital converter and then collected by the computer.

The signals obtained on computer from photodetector 2 were voltage signals. In order to make the signals from photodetector 1 and photodetector 2 comparable, the value named equivalent PM20 power is used. Using the Laser diode controller as a light source the relationship between the equivalent optical power from PM20 and voltage from photodetector 2 was obtained.



(a) (b)

Figure 6: Experimental setup (a) Structual diagram; (b) Photo of near 3-axis stage region

4.2 Results and Discussion

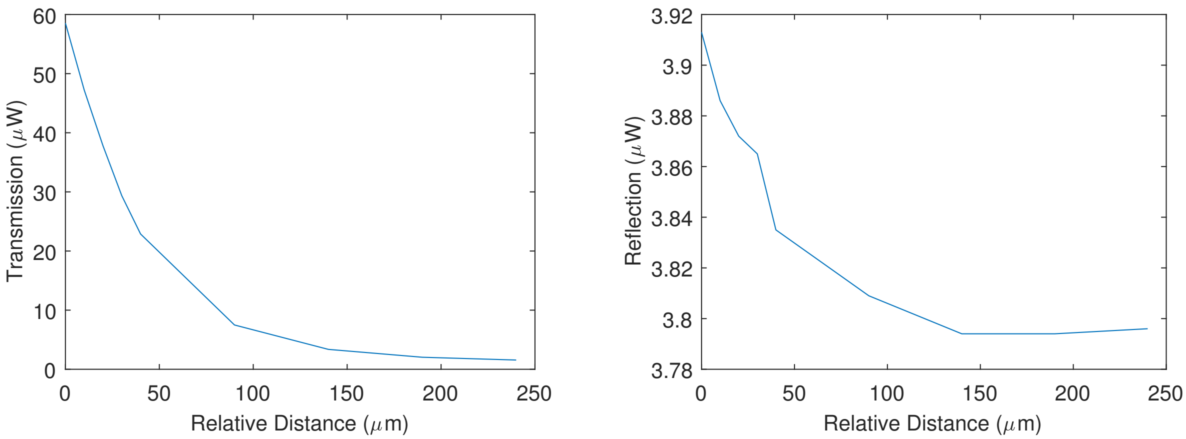
The position of the optical fibre at the lower position on the 3-axis stage was not movable. Only the optical fibre clamped on the upper position of the 3-axis stage was adjustable in X, Y, and Z directions by three tuning knobs.

The first step of the test was the collimation of the two optical fibres. By adjusting the knobs, the two optical fibres became closer to each other and the transmitted light signal obtained by photodetector 2 became bigger. When the two optical fibres were close enough but not touching and the optical power displayed by the optical power meter reached its maximal value, the position of the end of the upper optical fibre was at the original point (O). Figure 7a is the photo which shows the situation that the upper optical fibre was at the original point after collimation. Figure 7b is the photo with coordinates added.

(a) (b)

Figure 7: Collimation of two optical fibres (a) without coordinates; (b) with coordinates

First the two optical fibres were moved 240 µm in the Z direction. Then, the fine tuning knob was adjusted to make the two optical fibres closer in the Z direction and the reflected light power from photodetector 1 (PM20) and voltage from photodetector 2 (Monitor+ of PDB420C) were recorded.Then according to the calibration, the transmitted light optical power was obtained. The transmitted light optical power and reflected light optical power for different distances between two fibres being close are shown in Figure 8a and 8b respectively.

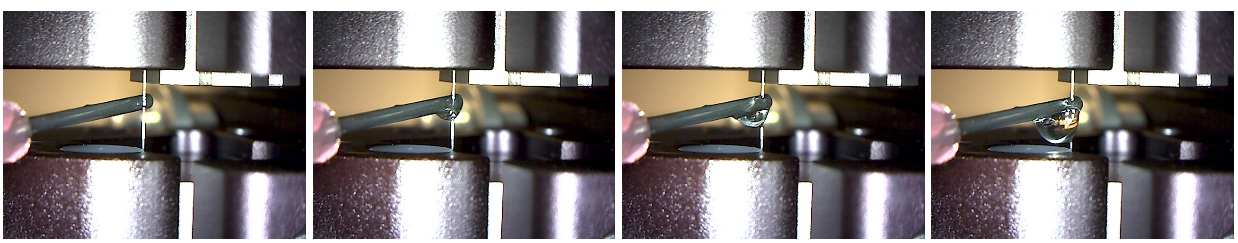


(a) (b)

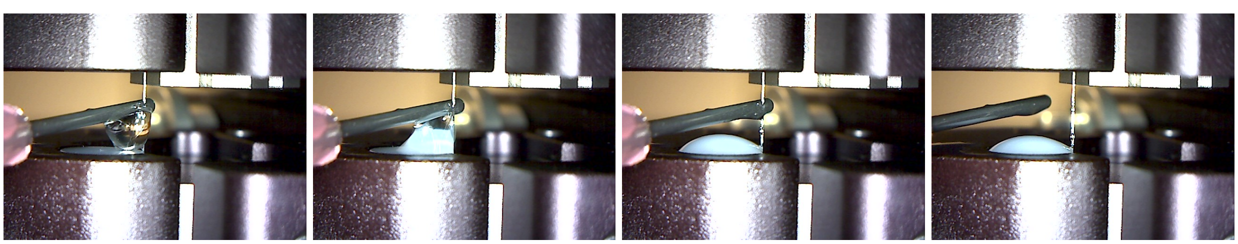
Figure 8: Optical fibre position without refractive index liquid (a) Transmitted light; (b) Reflected light

As shown in Figure 8a, the optical power of transmitted light increases from about 1.5 µW to about 58.5 µW. In Figure 8b, the reflected light increases from about 3.80 µW to about 3.91 µW, both following a similar exponential curve.

The next step is dropping liquid with the syringe. The process of dropping liquid is shown in Figure 9a-9h. After dropping liquid, the transmitted light decreased to about 1.8 µW and the reflected light decreased to about 3.36 nW.



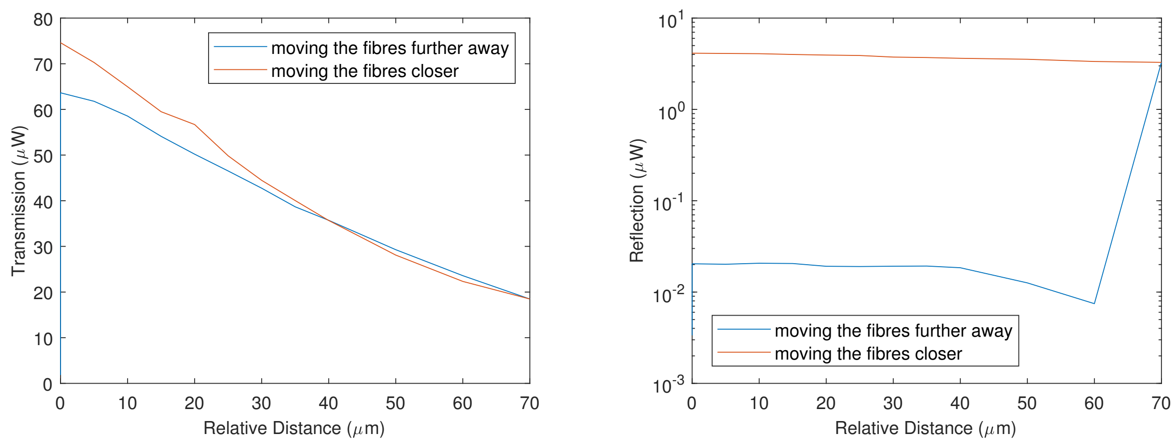
(a) (b) (c) (d)



(e) (f) (g) (h)

Figure 9: The process of dropping liquid on optical fibres (a)-(h) are the photos of dropping liquid process in a sequence

The transmitted light decreased dramatically because in the process of dropping liquid from one side of optical fibres the liquid pushed the optical fibres away from their original position. After adjusting the fine tuning knobs of the Y and Z directions, the transmitted light increased to about 63.6 µW and the reflected light increased to about 20.4 nW. Note that due to the influence of the liquid to bend the optical fibres, it may not be the maximum transmitted light value. From this new position, by tuning the Z direction fine tuning knob, the optical power of transmitted light and reflected light were obtained when the distance between two optical fibre were initially far away and then close to each other, which are shown in Figure 10a and Figure 10b respectively. Blue lines in Figure 10 represent the process of two optical fibres moving far away from each other, and the orange lines represent the process of two optical fibres moving close to each other.



(a) (b)

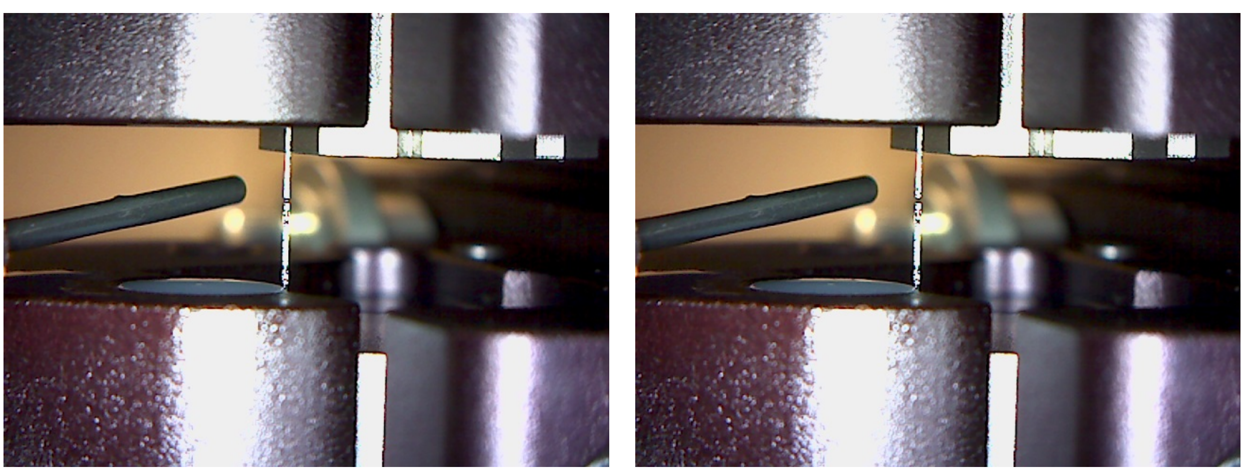
Figure 10: Moving optical fibre with refractive index liquid (a) Transmitted light; (b) Reflected light

With the increase of the Z distance between two optical fibres, the transmitted light has decreased. However, it is different for reflected light. For about 40 µm of the Z distance range, the reflected light kept the low values of about 20 nW. When the Z distance increased slightly to over 40 µm, the reflected signal started to fluctuate and to increase to about 3.9 µW. In this case, the function of using refractive index liquid to decrease the surface reflection of two optical fibres lost efficiency. Even when moving the two optical fibres closer to each other again, as shown in colour orange in Figure 10b, the reflected signal did not decrease. The detail of this process is shown in Table 1.

In Table 1, the fluctuation details are shown. When the Z distance reached 70 µm, the detected reflected light signal fluctuated a lot. From the USB microscope, it can be seen in Figure 11b that there was no liquid connecting to both of the two optical fibres at position Z=70 µm. It is different from the photo taken before moving optical fibre (at Z=0 µm) which is shown in Figure 11a, the liquid between the two optical fibres optically connected the two fibres together. With the two optical fibre's separation decreases again, the reflected signal appeared as a complex fluctuation behavior from a large fluctuation to a slight fluctuation to a small fluctuation and finally to a slight fluctuation again. Although this process cannot be fully recorded by the microscope, it may be deduced that some liquid existed on two ends of the optical fibres flowed and did not have the ability to recover its original situation even if the Z distances changed to the same positions.

Table 1: Transmitted light and reflected light vs distance after dropping liquid

Z /µm	Y /µm	X /µm	Transmission /µW	Reflection /nW	Reflected light fluctuation
0	0	0	1.8	3.36	N/A
0	7.5	1.5	63.6	20.39	N/A
5	7.5	1.5	61.7	20.16	N/A
10	7.5	1.5	58.5	20.68	N/A
15	7.5	1.5	54.1	20.53	N/A
20	7.5	1.5	50.1	19.15	N/A
25	7.5	1.5	46.5	19.03	N/A
30	7.5	1.5	42.7	19.18	N/A
35	7.5	1.5	38.6	19.26	N/A
40	7.5	1.5	35.7	18.46	N/A
50	7.5	1.5	29.2	12.6	N/A
60	7.5	1.5	23.5	7.46	N/A
70	7.5	1.5	18.4	3276	N/A
60	7.5	1.5	22.3	3348	large
50	7.5	1.5	28.0	3541	slight
40	7.5	1.5	35.6	3698	slight
35	7.5	1.5	40.0	3698	small
30	7.5	1.5	44.4	3742	small
25	7.5	1.5	49.8	3895	small
20	7.5	1.5	56.6	3937	small
15	7.5	1.5	59.4	3997	slight
10	7.5	1.5	64.9	4076	slight
5	7.5	1.5	70.2	4100	slight
0	7.5	1.5	74.6	4127	slight



(a) (b)

Figure 11: With refractive index liquid (a) Z distance 0 µm; (b) Z distance 70 µm

5. CONCLUSION

Our initial findings are that the influence of the gap between the two optical fibres decreased after dropping refractive index match liquid into the gap after fibre collimation, which is a beneficial result for understanding the influence of scattered light from a liquid containing NPs. Note, the position between the two fibres can change due to the weight of the droplet and the fibre ends had to be re-collimating to investigate the influence of the moving droplets. These results will be expanded by additional experiments and modelling of the scattering from the nanoparticales and droplets. Based on these initial findings, we plan to eliminate the influence of rheological effects which lead to the light signal changes with fiber optic ferrules on the ends of optical fibres and investigate the characteristic of back and forward scattered light from gold NPs in the refractive index matching liquid between two optical fibres' ends in the next step.

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