Rapid detection of Fentanyl using a multifunction nanostructured substrate

Final Report

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1. Introduction

1.1 Acknowledgement

We would like to express our special thanks of gratitude to our advisor and client Dr Meng Lu who helped us to acquire equipment and materials for this project. Also, we would like to send a special appreciation to our professor. Dr. Thomas, Daniel as well as Electronics Technology Group (ETG) who provided us with in class resources to use.

1.2 Problem and Project Statement

The purpose of the project is to design a system that can detect fentanyl in a mixture of chemicals by separating compounds. The system will use "thin layer chromatography" to separate the compounds using a 3D printed optical instrument that detects the existence of fentanyl.

General Solution Approach: The project is split into three smaller design tasks. The first task is to fabricate the chromatography paper and the GLAD(Glancing angle deposition) film. The task will be accomplished as follows:

a) Research the appropriate material and solvent for mobile phase and stationary phase, to separate the fentanyl out from component obviously.

b) Use glancing angle deposition to fabricate the ultra thin layer plate by electron beam.

The second task is to test the chromatography based on paper and film created on the first task.

c) Test the chromatography on GLAD film

d) Test the chromatography on PC-GLAD

The third task is to test the entire instrumentation system

e) Test the system of instrumentation (data analysis)

1.3 Intended Users and Uses

The fentanyl detector is a low cost measurement instrument that can detect and measure fentanyl. It can be used by individuals or corporates involved in detecting and measuring fentanyl. The fentanyl detector can have it applications in the pharmaceutical area to measure the dosage of fentanyl in substances. It can be used by law enforcement at airports and border checkpoints to detect illegal trafficking of fentanyl. It can also be used in laboratories for experiment with fentanyl compounds.

1.4 Assumptions and Limitations

Assumption

Our intention is to make the fentanyl detector affordable, compact and easy to carry, and with high accuracy. Our product would mainly be divided into two sections: the chromatography section and detection section. We expect the chromatography section to be removable, which is easy to connect with other terminal to detect it. Based on our cost estimate for the materials, we assume that our final product would be relatively inexpensive.

Limitations

This project is very research and experimental based and hence various limitations come up at different stages. Our initial goal was to be able to separate and detect liquids and mixtures containing fentanyl.

The fact that a lot of us have very limited knowledge of chromatography and separation of liquids mean we spent a lot of time trying to learn the basic principles as we experimented with whether liquids could flow on the fabricated photonic sensor and then move on to the separation stage. Hence, a big limitation is our limited knowledge and skill on the chemistry separation process involved.

Another limitation is that we are experimenting with different materials such as silicon dioxide and titanium dioxide to see , if any, would work best to give good liquid flow and separation. There is no guarantee that it'll be a success. As such, the research based nature of this project is a limitation to the outcome itself.

1.5 Expected End Product and Deliverables

First semester Deliverables: Our client requirements and expectations for the 1st semester were as follows:

1.) Carry out a simple chromatography experiment
2.) Test dye separation on chromatography paper
3.) Fabrication of GLAD film
4.) Fabrication of Photonic crystal
5.) Test chromatography on GLAD film

Second Semester Deliverables: Our client requirements and expectations changed this semester. Our main tasks were as follows:

1.) Instrumentation: Optical detection setup.

    This setup is explained and shown in Section 2.3 of this document. It consists of the entire setup system for running the proposed experiment with a light source

2.) Nanoporous sensor fabrication: GLAD coating of a PC substrate(360nm period)

    This was a major deliverable for us as is the fabrication of the actual sensor that would be used in the next phase of the project to separate mixtures containing fentanyl

3.) Chromatography sensor test: Have a good capillary flow rate

    We were able to have a good flow rate on the fabricated Silicon dioxide photonic sensor on which the dye was spotted. To ensure this, we also used the BOE etching process to increase the porosity of the sensor and aid liquid flow

4.) Integration of sensor and detection setup

This project is very research and experimental based hence the change in our deliverables from last semester. The initial goal at the start of the project of being able to detect fentanyl is still very realistic and can be continued in the next phase of the project. We are currently on the 4th and final stage of our deliverables for this semester, the sensor integration and detection setup.

2. System Design and Development

2.1 Proposed Design

<u>Chromatography</u>

Chromatography is a technique to separate chemical mixtures into its individual components. Usually, the stationary phase will be fixed in place, and the mobile phase flow through the surface of the stationary phase.Due to the surface structure of the stationary phase, it will slow the movement of the chemical mixture, at least, slower than the mobile phase. The speed of movement is also affected by the interaction between the moving phase ,and the sample. Different components of the chemical mixture have different solubilities with the stationary phase. All of these factors causes the various components in the mixture to move at different speeds and seperate

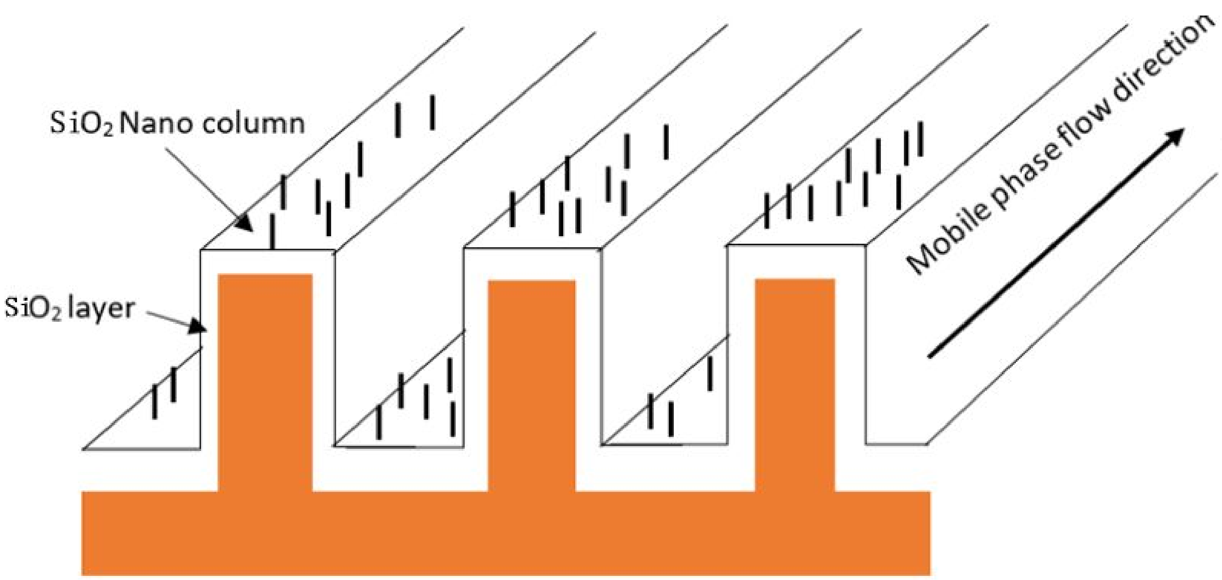

Fig 1:The concave between glass substrate (yellow) and the concave SIO2 nano column (black) forms the channel, and makes the capillary action happen

In this project, we used a glass plate as a fixed phase ,and coated it with a thin layer of silicon dioxide as designed photonic crystal layers could supplement the benefits of time-resolved chromatography methods and increase analyte detection sensitivity which has been shown in Fig1. The photonic crystal layers is work through the capillary force principle, on Fig1.

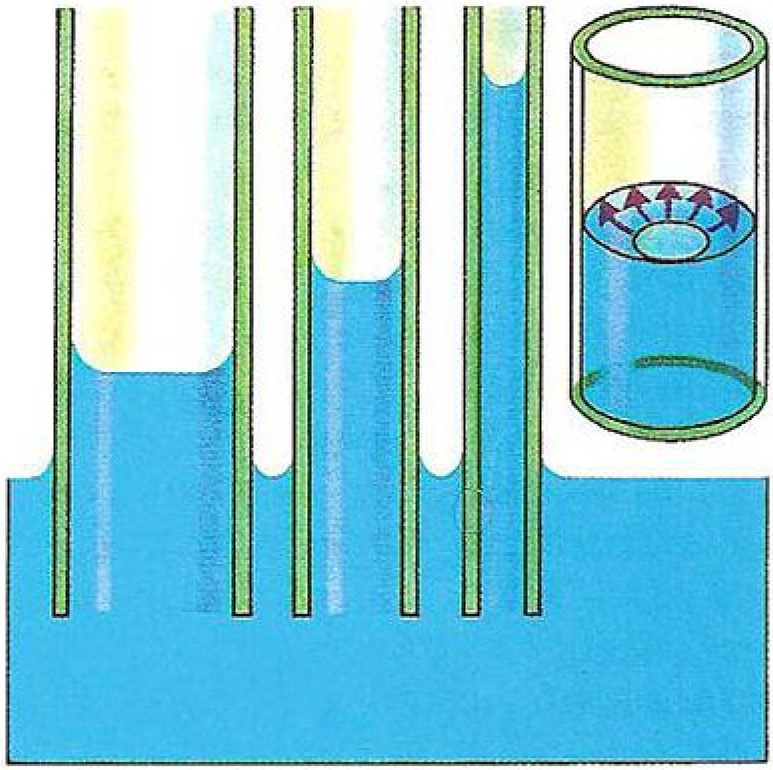

Fig2:As the figure shows above, there are three tubes with different diameters, smaller diameter means smaller superficial area and also gives less gravity. The surface of the infiltrating liquid in the tube is concave. It applies a pulling force to the liquid below to make the liquid rise along the tube wall. When the upward pulling force is equal to the gravity of the liquid column in the tube, the liquid in the tube stops rising.

Each column on the surface of the glass plate will stay together to form as channels, micro-scale cracks or nanoparticles. The liquid flow through these channels can accelerated by capillary force as shown in fig2 to make an efficacy separation.

For the capillary force, the attraction force between each molecule, combines them in a liquid called cohesion force. The attraction force between liquid molecules and container molecules is called adhesion force. When the adhesion force between liquid and container is greater than cohesion force within the liquid, the capillary action occurs.

Afterwards, we put the plate into a petri dish filled with organic solvents as a mobile phase. We have to be really careful because we only need enough solvent to dip to the lower edge of the plate, but we can't touch the sample point as it may affect our results.

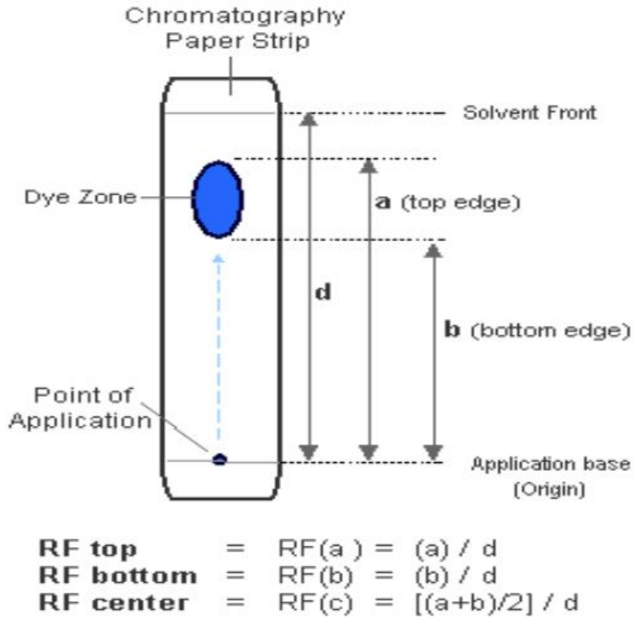

Fig3:The diagram above explains what are the distances to be taken to calculate these RFs

Finally, the components can easily be identified based on the distance it moves through the surface of the plate, count as their RF value (different component has different RF value).

2.1.1 Mobile phase parameters and Stationary phase parameters

Mobile phase :

Acetate:Methanol:Water =65:23:5

Stationary phase:

Thickness of silicon dioxide layers is 1 micrometer

The distance between each column is 360 nanometer

Instrumentation

The instrumentation is the device that takes a series of pictures of the chromatography sensor to allow us to determine if the mixture contains fentanyl. It is made of three principal parts which are the chamber(Fig9: Prototype structure & function), the ESP32 processor/cam(Fig17: ESP32-CAM), and the light source(Fig8:Light transmission and detection setup). There is a cut on top of the chamber that allows the chromatography sensor to slide through and dip about 0.5cm in the solvent at the bottom. The light source comes through the hole on the right and the camera lens goes in the tube on the left. The rest of the circuitry will be left out. There is a picture below (Fig4: sensor before and after separation) that shows the visual description of the design.

Pictures taken by the camera will be stored in a computer and google drive over wifi connected to the ESP32 processor. In the future we will have a code that analyze those pictures and print the result but for this first generation of the project we will be looking at the pictures and make the decision ourselves.

In ideal conditions a picture of the chromatography sensor alone will look like fig4(a), the sensor plus solvent flow fig4(b) , and the sensor plus solvent flow plus separated compound fig4(c). so with fig4(c) we can calculate the retention value of all the compounds present in the mixture. since every compound has a specific retention value we can compare our calculations to the retention value of the fentanyl and make the decision if it present or not. There is a picture below that shows the visual description.

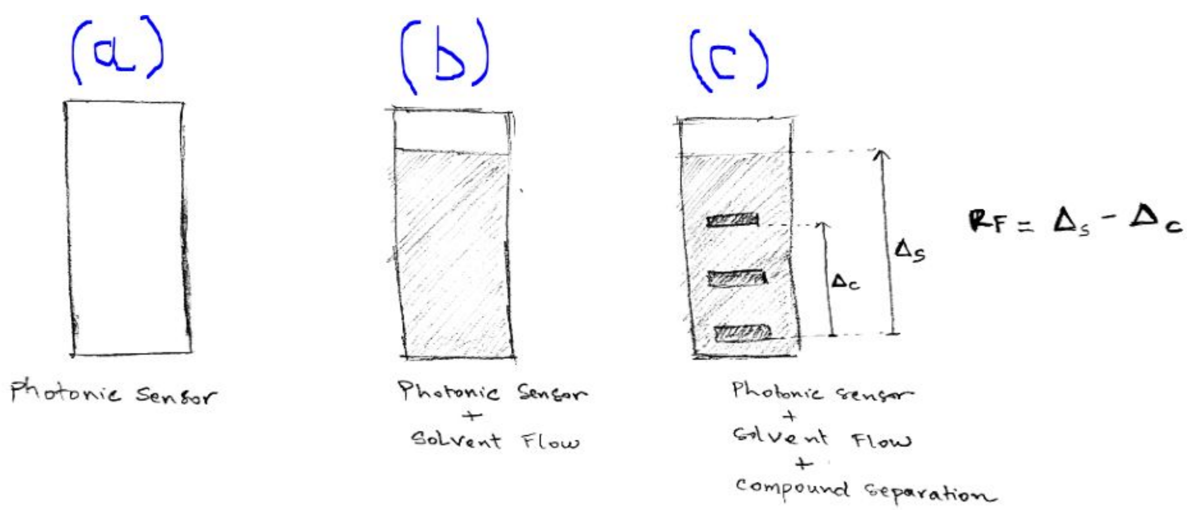

Fig4:sensor before and after separation

<u>3D Body design</u>

The 3D design was done using Solidworks and 3D-printed in the aerospace lab. The overall length is 13.5cm long with a height of 1.5cm and made to pieces, the actual design and a cover. It has a tube to the camera lens which is 5 cm long and 2cm in diameter. The rest is a chamber that is 8.5 cm long and 1.5cm tall that has a cut in the bottom to hold a 2cm diameter petri dish with the solvent in it . It has three holes for the chromatography sensor, the camera lens, and the light source. There is a picture below that shows the visual description.

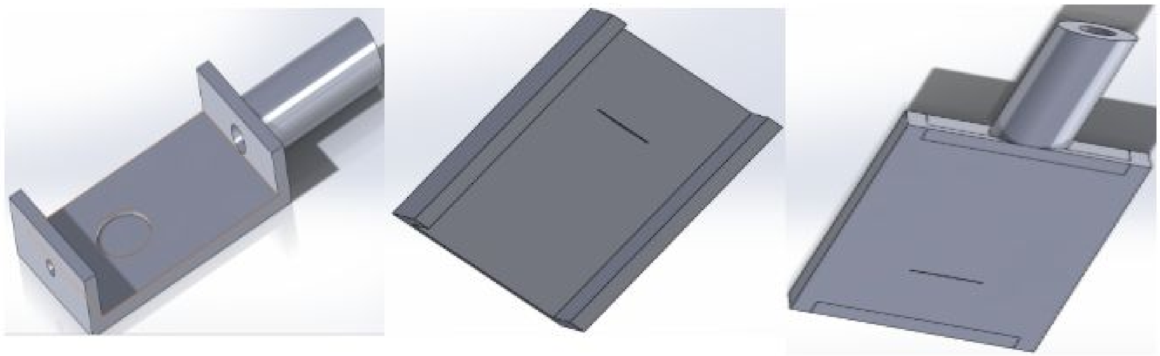

Fig 5:3D prototype

2.2 Design Analysis

By breaking the project into three different parts(test, fabrication and instrumentation), the team able to gain a better understanding of the project. For the test parts, team spend one semester to find out how the chromatography work, and what factors will affect the final separation experiment result, temperature, thickness, material and surface structure etc. After that, the team will try to use the knowledge has been found in the previous semester. After the test team analyze the data has been collected from the experiment, then send it to the fabrication team.

Though a lot of experiments, team gets better understanding of its principle. By using the glass plate coated with silicon oxide on its surface as photonic crystal layer to increase the separation efficiency through the capillary force. And also found out that different type components of solvent will also give a huge effect on the result. The solvent will not just work as a carrier, but also a parameter of the whole process. The polarity difference among the components within the solvent will make this phenomenon occurs. Two different types of components which has different polarity will give a different flow rates for each component of the chemical sample to accelerate separation depends on the affinity between the solvent component and the sample component. The polarity difference between the solvent (mobile phase: The mixture of ethyl acetate, methanol and water) and the coat material on the surface of the glass plate (stationary phase: silicon dioxide) will make the same phenomenon occurs, by slowing down the flow rate or vice versa. Before the silicon dioxide, we use the titanium dioxide instead, but experiment result is not very good, so we change it to silicon dioxide. For the mobile phase, what we have done is to try to find them though some academic articles.

One challenge we faced was finding a suitable material ,and structure for the stationary phase the components and its proportion for the mobile phase.

The major challenges faced in the instrumentation are the selection of the microcontroller and some software issues. We started this project with an arduino uno and the arducam but half way through our client wanted something smaller and more wireless so we decided

together to go for the ESP32 board and cam (Fig 17 : ESP32-CAM). The ESP32-CAM is significantly smaller, has 5V power supply and could only be connected over WiFi. We can then directly send images to google drive and have multiple computer access those pictures once uploaded but it was more complicated to use and we had some challenges with the software. One of the main challenges is to implement a connection between the computer and the ESP-32 CAM. While uploading the code on the ESP32-CAM, it was sending error "package header not detected". To fix the problem, we connected the ESP32-CAM to the same network as the computer it is connected to. We did this by changing the ESP32-CAM's code name and the password of the network of the computer it is connected to.
Another issue we faced was finding a way to send data directly from the ESP32-CAM into google cloud. To fix it, we created a file on the computer which is directly connected to google cloud via Google Backup and Sync. As a result , whenever the ESP32-CAM collects any data, it puts this data into a file which is connected to Google cloud.

2.3. Application of the designed photonic sensor

The photonic sensor will eventually be used for separating the fentanyl with other chemical compounds that exist in the mixture. The way that it will be applied on the photonic sensor should be the same as using the chromatography method to separate chemical mixed dye.

- **Prepare the photonic sensor**

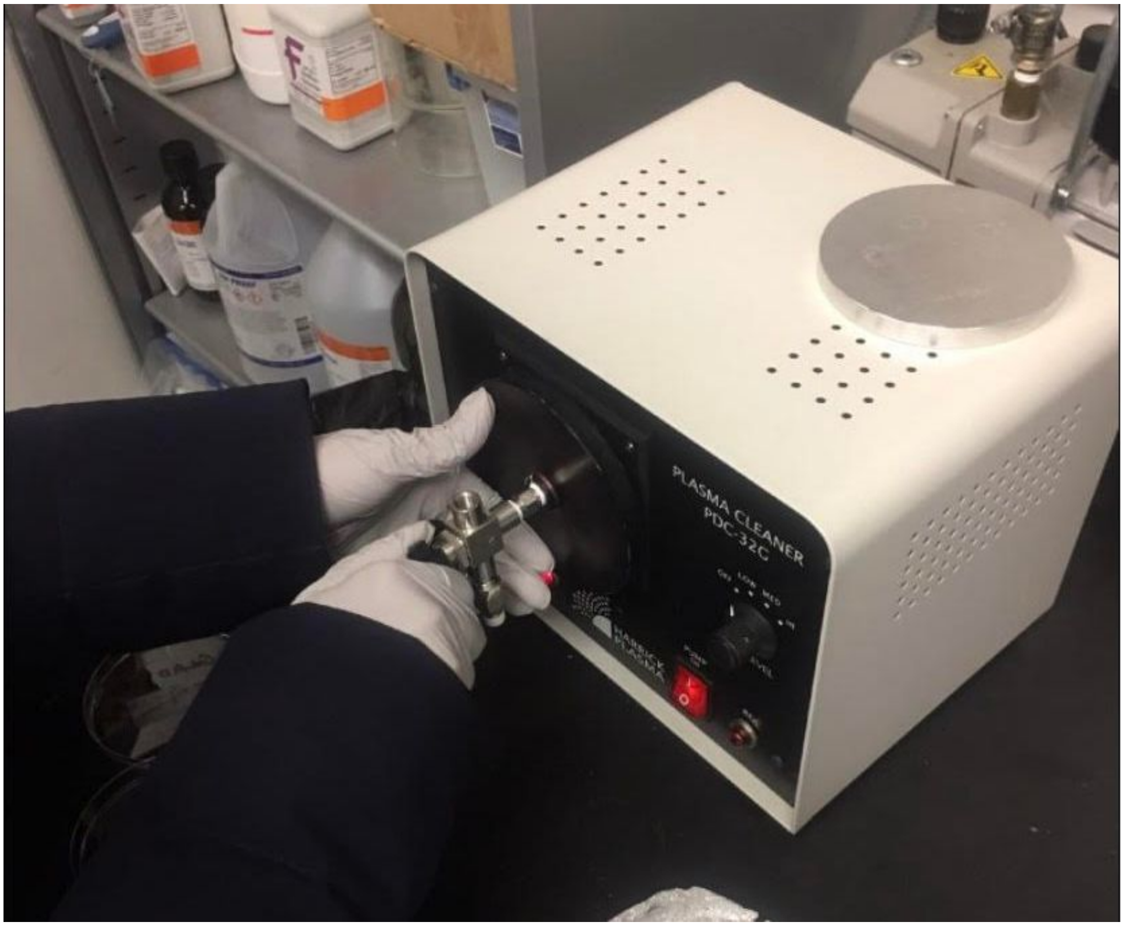

Fig 6: Oxygen plasma machine

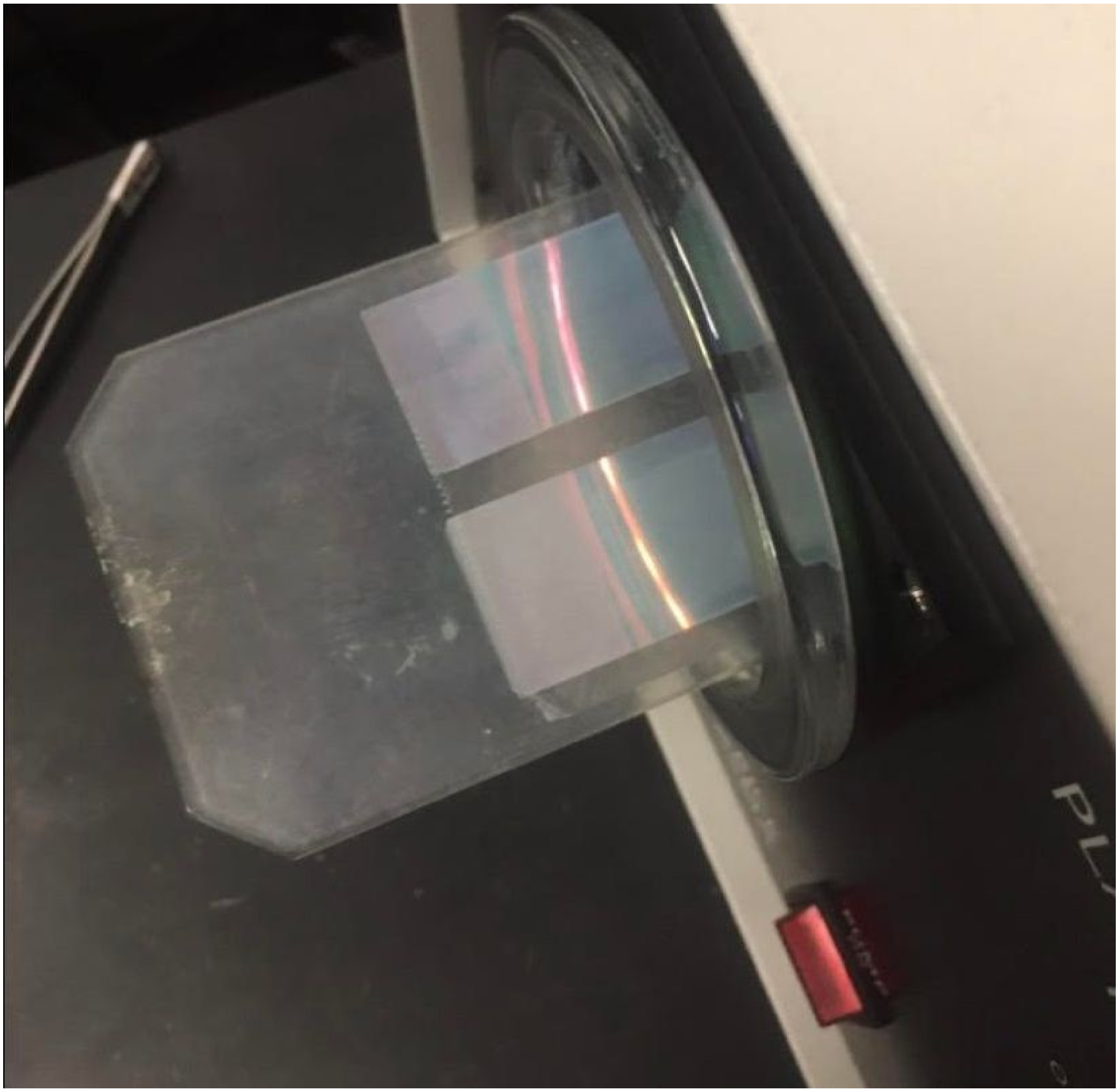

Fig7:Photonic sensor with Oxygen plasma machine

At first, we take out the fabricated photonic sensor. Use the oxygen plasma(Figure 6 & Figure 7) to treat the sensor surface to open up more pores and aid liquid flow on the surface. We then put it on the desk and use a pencil to draw a line on the photonic sensor. The line should be above the surface of the solvent when you put it into the solvent.

- **Dip the mixed dye on the sensor**

Use the dip tube soak a little bit of the mixed dye and drop the mixed dye on the line that we drew in the first step.

- **Put the sensor into the container with solvent.**

We prepare a clean container with cover, pour some of the solvent into the container, make sure the surface of the solvent is not higher than the dipped dye on the sensor. Then we put the sensor into the container ,and put the cover on the container to make sure the solvent wouldn't evaporate.

After about 15 mins, we observed that the dye is separating as the solvent(mobile phase) went across the surface of the photonic sensor.

- **Observe the result and take the dye out**

We then took out the dye from solvent. The whole process takes about 30mins. As expected to see there are different colors show on the sensor because the mixed dye is separated. This shows that the separation process on the sensor was a success

Experimental Setup (light transmission & detection)

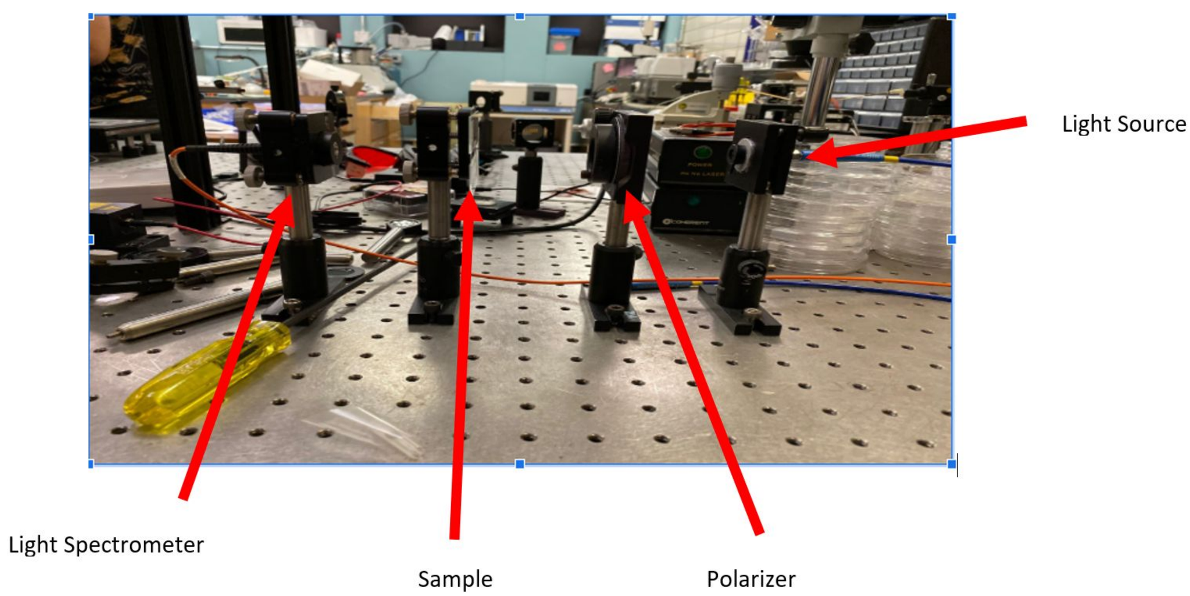

Fig8:Light transmission and detection setup

The experimental Setup is used for detecting if there's fentanyl or not. There are four parts on the setup

- The first part is called light spectrometer which is used to measure properties of light and give the feedback to the computer.
- The second part is called sample holder, we usually put the photonic sensor on the sample holder after done the dye separation.
- The third part is polarizer which is an optical filter that lets light waves of a specific polarization pass through while blocking light waves of other polarization.
- The fourth part is the light source which is the supply of the light.

For our project, We need to use the setup to find out the result. We use the software called OceanView to operate the analysis step and looking for the peakpoint on the graph.

Basically, we would make a comparison before the dye dipped on the photonic sensor and after we dipped the dye on the sensor. Then analyse if there's shift on the graph.

3. Testing and Implementation

3.1 Testing and Evaluation Plan

We will use food dye as drag to test our photonic sensor. The food dye solution contained Brilliant Black BN (~6–7 mg mL−1), LissamineTM Green B (~7–10 mg mL−1), tartrazine (~12–13 mg mL−1), and Acid Red 14 (~12 mg mL−1) dissolved in DI water. The mobile phase we used is (ethyl acetate/methanol/water 65/23/5, v/v/v). We drop around 0.5 microliter on bottom part the photonic sensor and wait several minutes for drying. Afterwards, we dip the photonic sensor in to the solution. We must make sure the dye will slightly higher than the liquid level of the solution, so that the chromatography process will process on the photonic sensor. The solution will then flow upward.

4. Operation manual

The following sections will explain how to run a full experiment using the photonic sensor and also the required steps for setting up and using the ESP 32 camera to take pictures while the experiment is ongoing.

4.1. Prototype with the sensor

The prototype is designed with the structure shows below(Figure 9). It has two parts, the bottom part and the cover, the bottom part is for locating the camera and it has an aluminum petri dish for filling the solvent. There' a hole through the bottom part for locating the camera thus the camera would take pictures or video when the dye is separating. There's another hole on the cover, we can insert the sensor from the hole and make the sensor's bottom edge attach the petri dish. See Figure 9 for details

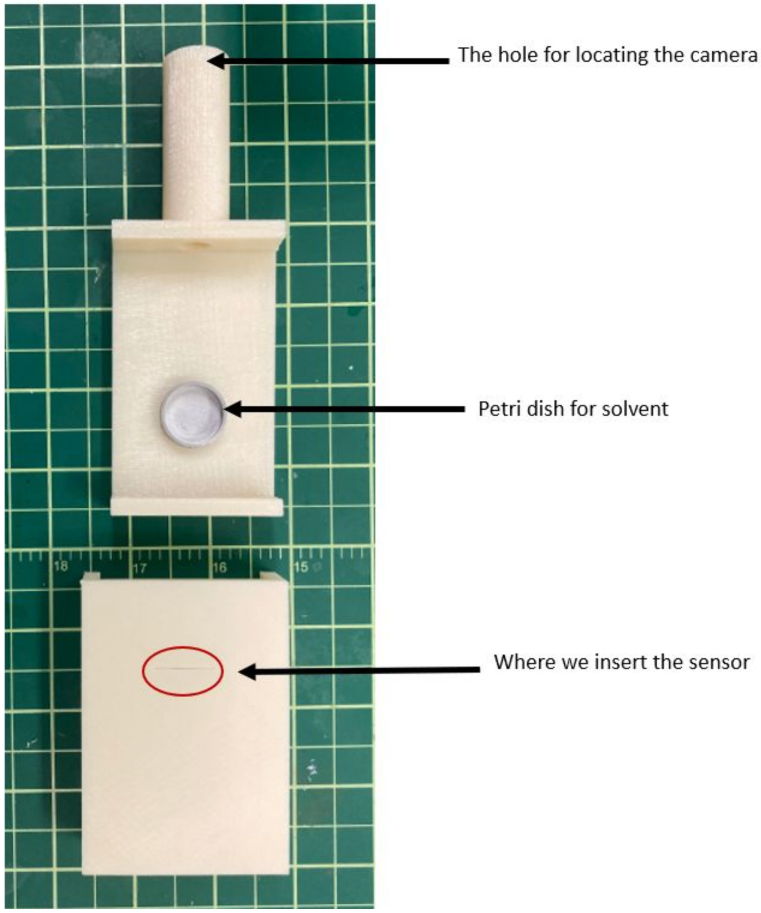

Fig 9:Prototype structure & function

- **Step1: Prepare the photonic sensor**

  Use the tweezers to take out the photonic and put it on the cut machine. Cut the photonic sensor and mike it to be 0.5 inch (wide) * 1 inch (length)

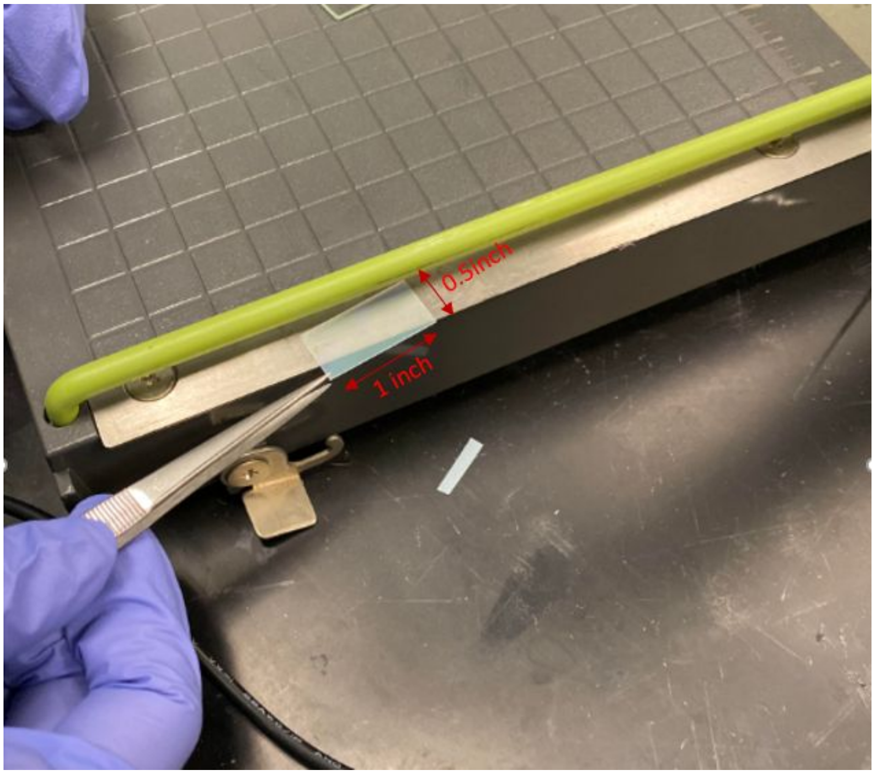

Fig10:cut sensor to size of 1inch*0.5inch

- **Step2: Dip the dye on the photonic sensor**

  We use the packpad with the small tube to soak a little bit of dye and dip it on the photonic sensor (Fig 11). The position of the dye should be located at least 2 mm above the bottom edge of the sensor(Fig 12) so that make sure the dye would not be dissolved by the solvent when we insert it into the prototype.

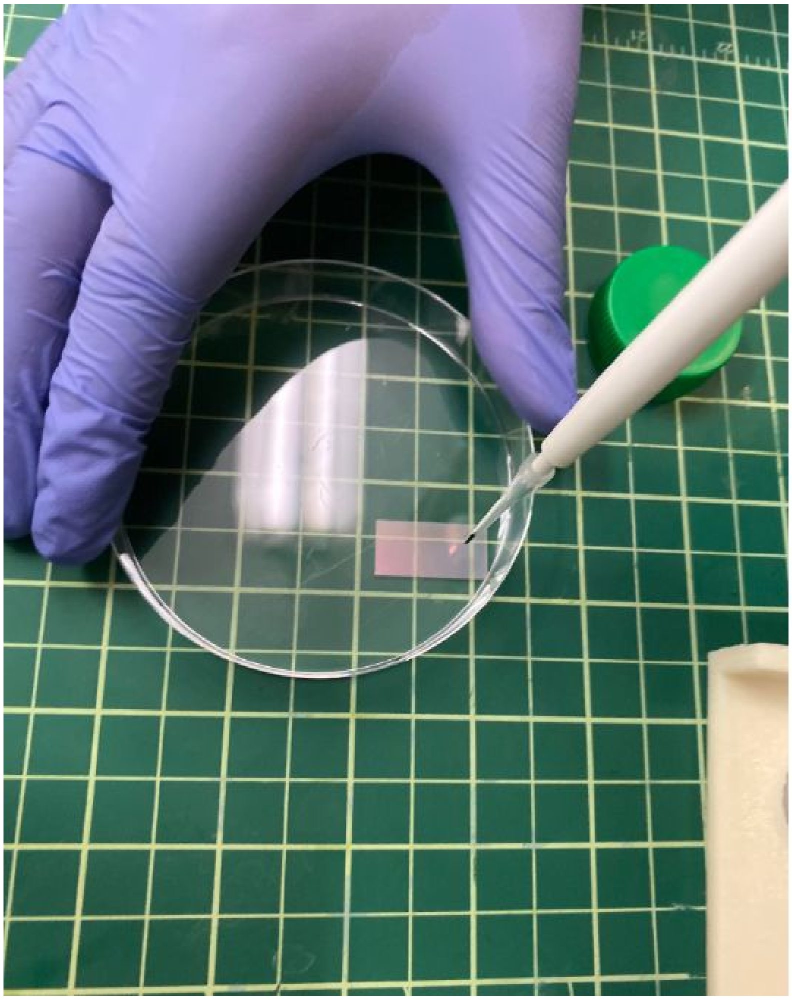

Fig11:Drop the dye on the sensor

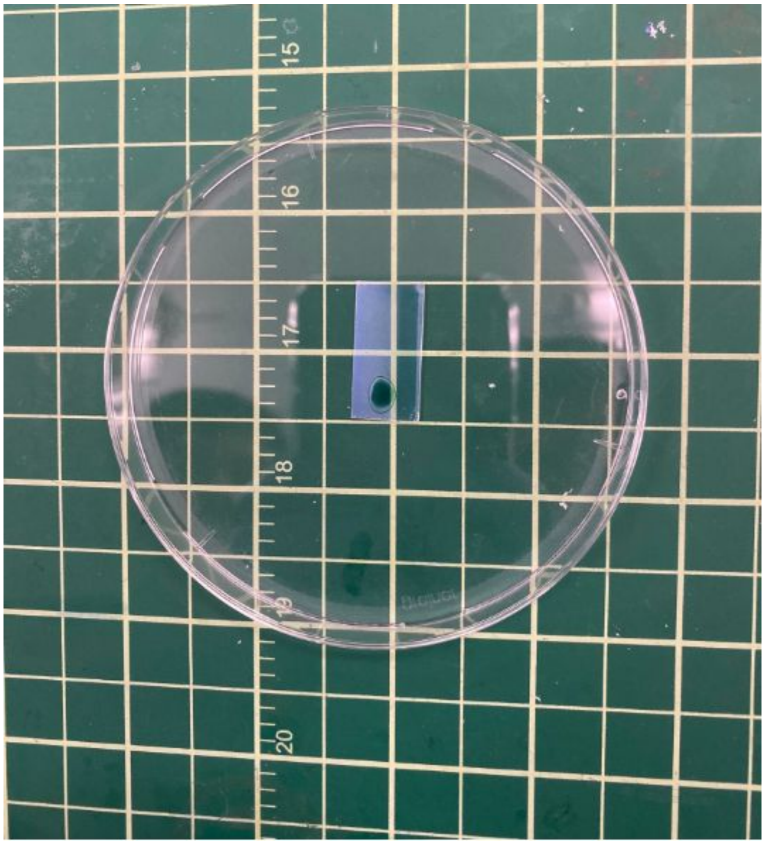

Fig12:Dye on the sample

After dipping the dye on the sample, we have to wait about 10 mins to make sure the dye is totally dry.

- **Step3: Fill the solvent in the Aluminium petri dish**

  Use the packpad to soak some of the solvent and pull it into the Aluminium, you don’t need to make the petri dish to be fully filled.

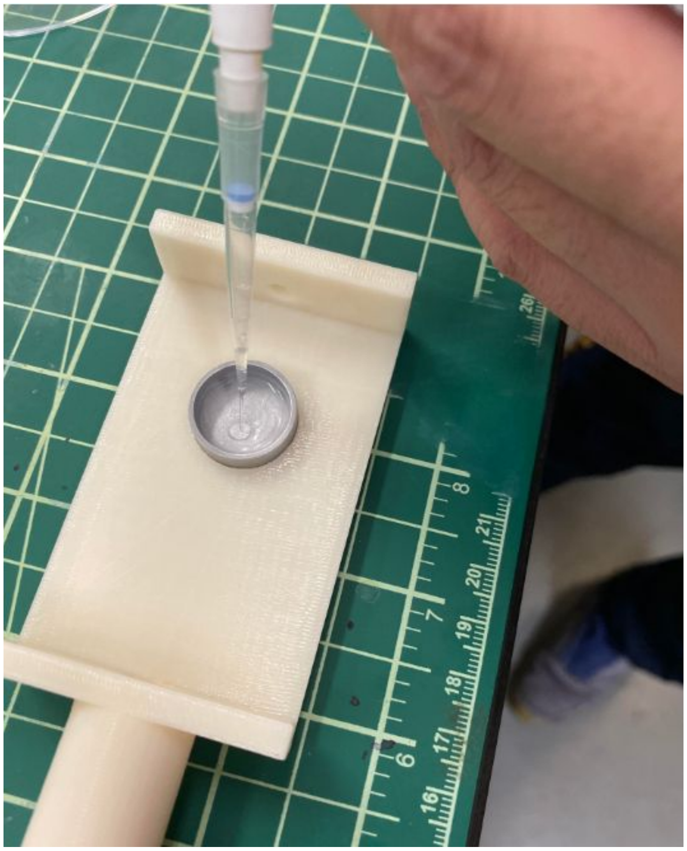

Fig13:Filling the petri dish with solution

- ❖ **Step4: Insert the photonic sensor**

  Use the tweezers to take the photonic sensor, carefully insert it through the cover of the prototype

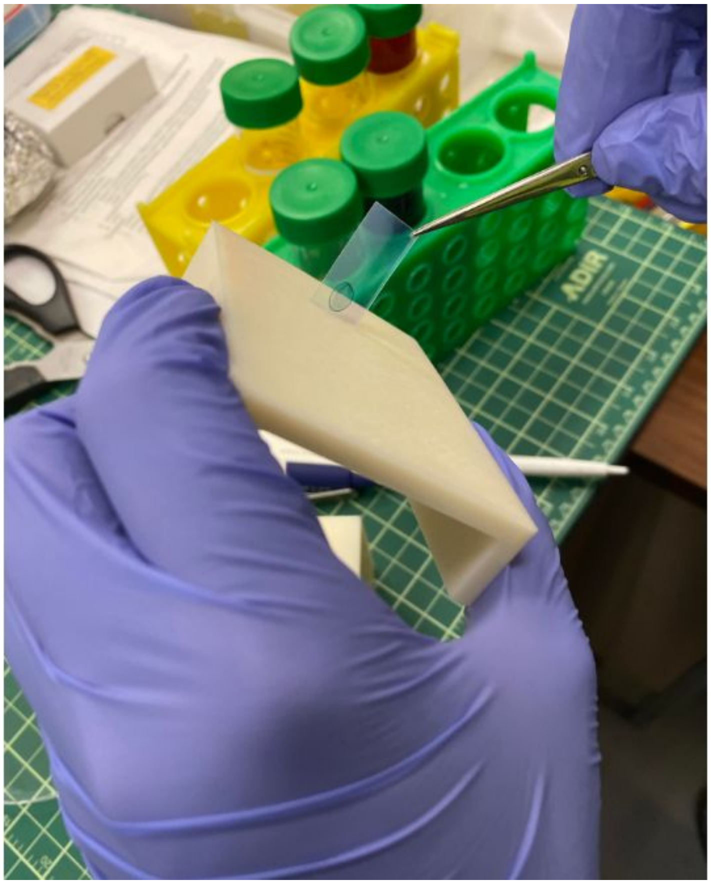

Fig14:Insert the sensor into the cover of the prototype through the hole

- **Step5. Connect the camera with the prototype.**

  After the sensor is inserted, take the cover back to the prototype,the sensor's bottom edge would attach to the surface of the solvent. Then connect the camera to the prototype(Fig 15). **See detailed camera instruction & camera operating steps in 4.2**

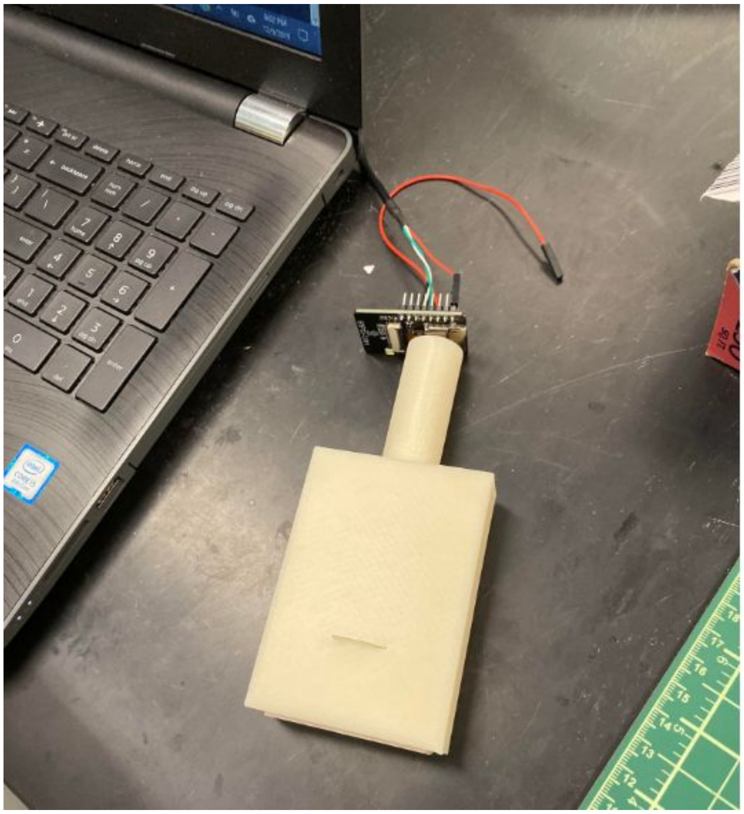

Fig15:Camera with the prototype

- **Step 6. Observe the result.**

  Wait until the dye is separated, then we can see the separation on the laptop screen.

Fig16:Separation of the dye

4.2. Camera instruction & operating steps

The camera used in this project is ESP32- CAM. The ESP32 Camera is a very small module based on ESP32 chip and 0V2640 camera sensor. Besides several GPIOs to connect to peripherals, it also comes with a microSD slot, wifi and bluetooth capability. All these features are useful for storing files directly on the module or into a cloud based system.

Getting started with ESP32-CAM

Some useful steps are needed to make the ESP32-CAM works properly.

❖ **Step 1: Component required**

- The ESP32-CAM board
- FTDI programmer
- Arduino IDE
- ESP32 library and webserver

Fig 17 : ESP32-CAM

Fig18 : FTDI programer

❖ **Step 2: Connecting the ESP32_CAM to the FTDI programer**

Connect the FTDI programmer as in fig 8

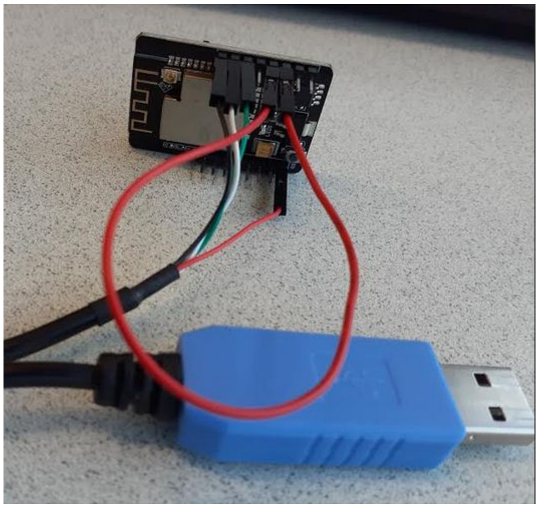

Fig 19: ESP 32 cam & FTDI

- **Step 3: Installing ESP 32 Board in Arduino IDE**

  Code and libraries are needed to make the ESP 32 work properly. Most code and libraries are provided from the manufacturer that we could find in the link below.

https://electronicguru.in/wp-content/uploads/2019/05/esp32-CameraWebServer.zip

- **Step 4: Setting up with the code**

  After successfully installing the necessary libraries and the code, go to the arduino IDE, select file > Example > ESP 32 > Camera and open the CameraWebServer example.

CameraWebServer | app_httpd.cpp | camera_index.h | camera_pins.h

```
1  #include "esp_camera.h"
2  #include <WiFi.h>
3  //#include "soc/soc.h"
4  //#include "soc/rtc_cntl_reg.h"
5  //
6  // WARNING!!! Make sure that you have either selected ESP32 Wrover Module,
7  //            or another board which has PSRAM enabled
8  //
9
10 // Select camera model
11 //#define CAMERA_MODEL_WROVER_KIT
12 //#define CAMERA_MODEL_ESP_EYE
13 //#define CAMERA_MODEL_M5STACK_PSRAM
14 //#define CAMERA_MODEL_M5STACK_WIDE
15 #define CAMERA_MODEL_AI_THINKER
```

Fig 20: The code

- **Step 5: Uploading the code**

Some set up are needed before uploading the code.

- Type in your wifi credential to make sure the ESP32-CAM and your computer are connected on the same network.

  example: const char* ssid = ”put_your_wifi’s_name”

  const char* password = “your_wifi_password”

- Make sure you select the right camera in the code as shown in line 15 in Fig 20.

  Example: #define CAMERA_MODEL_AI_THINKER

- Follow these steps to upload the code: Go to tools > Board and select ESP32 Wrover Module > Go to Tools > Port and select the COM port the ESP32 is connected to >Partition Scheme, select “Huge APP (3MB No OTA)” Then, click the upload button to upload the code to the board

- **Step 6: Getting IP from the Serial Monitor**

Before getting the IP address from the serial monitor, we need to disconnect GND and Io0 from the ESP32 CAM board and press the reset button

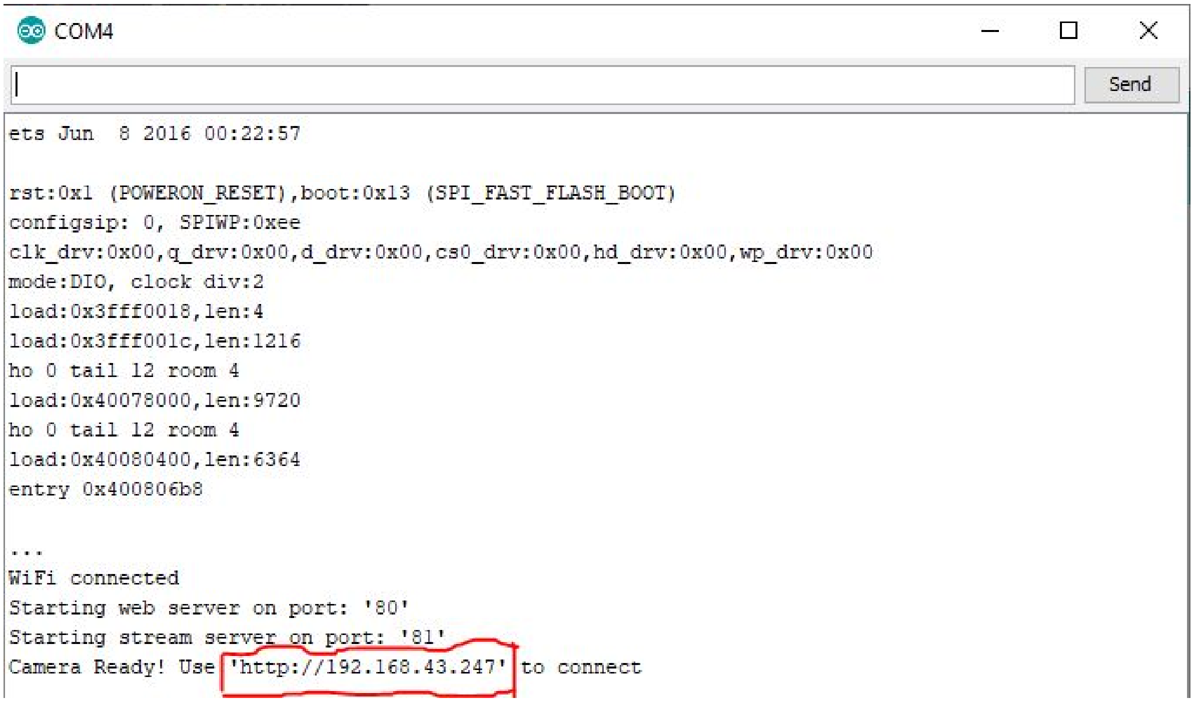

Fig 21 : IP address

After the board is successfully connected, copy the IP address shown in Fig 21 and past in any browser on your computer on the same network as the ESP-32 CAM.

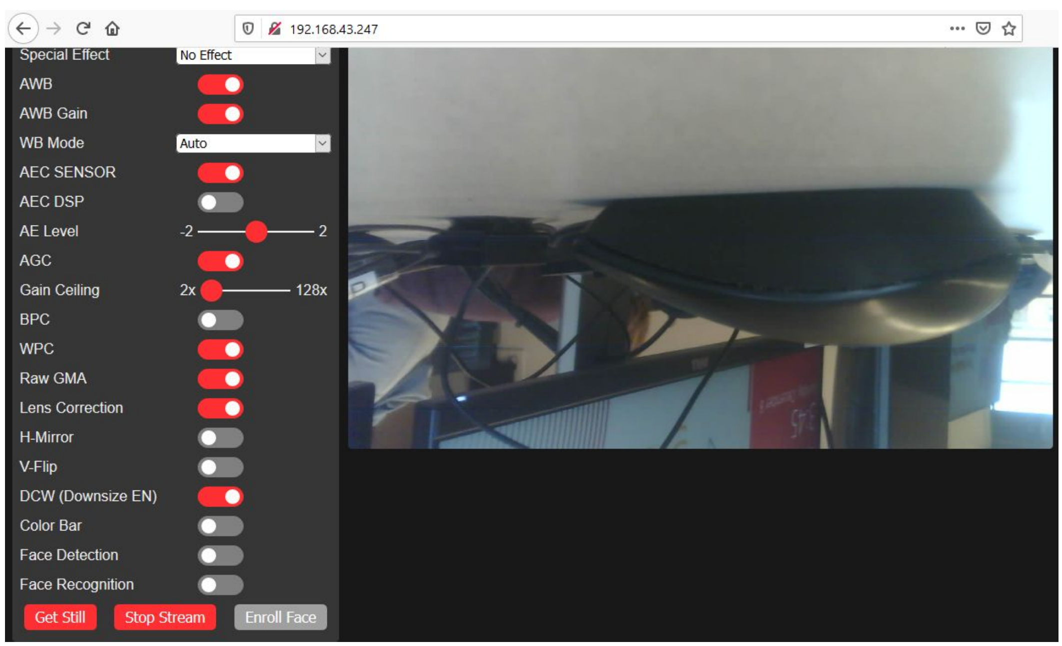

Fig 22: data streaming from a browser

In the browser, we could click on “get still” to take a pic and “start stream” to start recording.

- ❖ **Step 7: sending data to the cloud**

After taking a picture or record videos from the ESP32-CAM, we could store all the data on google drive or any cloud system. In our project we are using the google cloud to store our data. To successfully store data from the ESP-32 CAM, we need to have Google Backup and Sync install on the computer the ESP32-CAM is connected to. We also need to create a file on the computer that is directly connected to the google cloud via Google Backup and Sync.

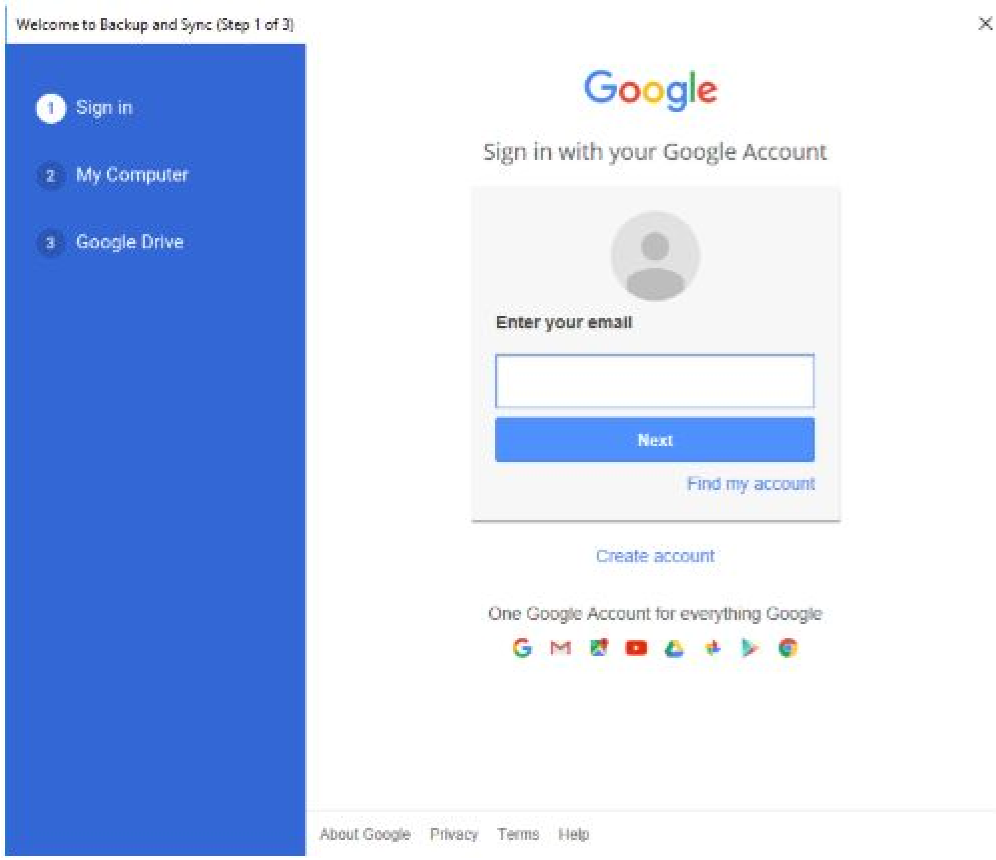

Fig 23: Sign in in google cloud

After installing Google Backup and Sync, we will be prompt with the screen in Fig 23 to sign in our existing google account.

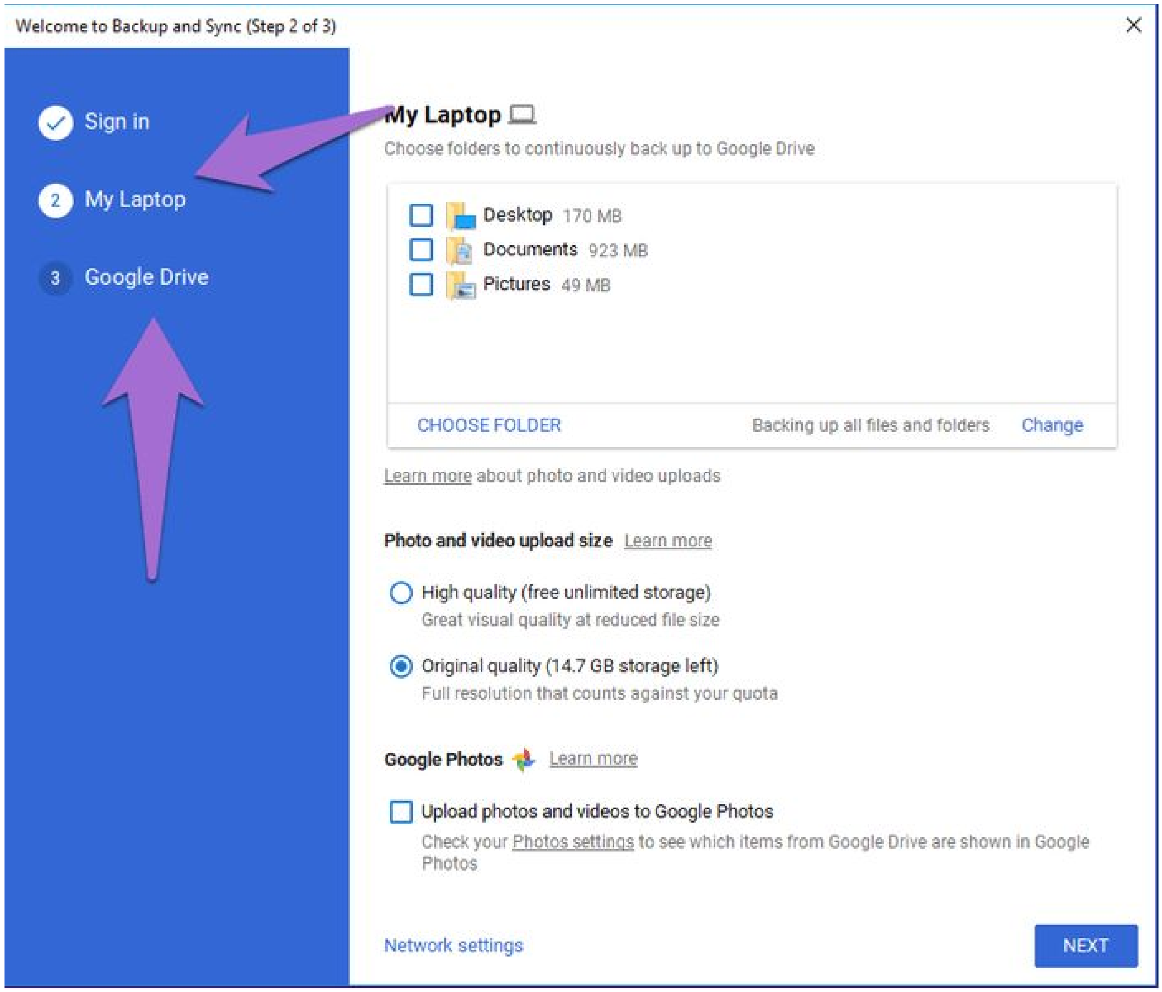

Fig 24: Creating a file on the computer that will directly connected to google drive

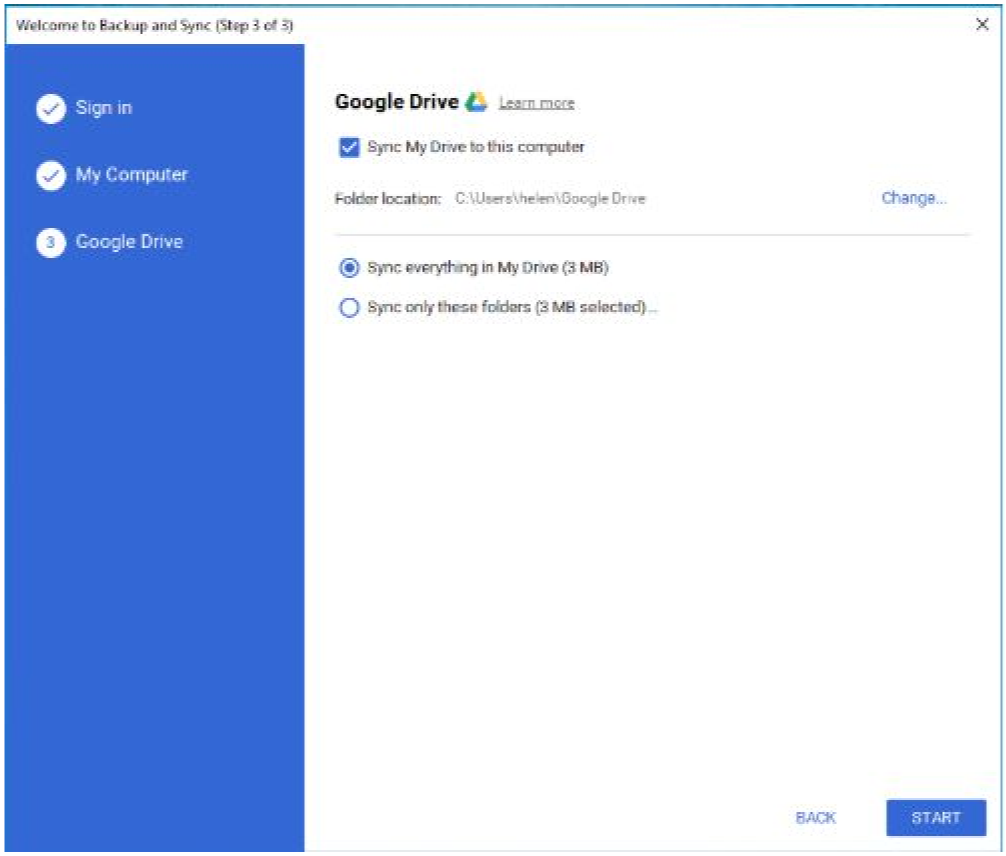

Fig 25: Choose a particular folder where all the data of the ESP32-CAM will be stored and click on "Sync only these folders" from Fig 25 to automatically send all the data to google cloud.

5. Closure Materials

5.1 Conclusion

This project gave a good research background into the viability and use of photonic sensors as separators and detectors of liquid. We were able to determine that a fabricated silicon dioxide substrate photonic crystal has a good surface to allow liquid flow and separation of experimental dye. We also were able to design a 3D prototype to act as a housing for the entire experiment setup and program an ESP 32 camera to take pictures of the photonic sensor as the separation process is ongoing. This project is a continuing research experiment and we will talk about some more work that needs to be done in the next section.

5.2 Future Work

The next phase of this project will involve the main testing stage of experimenting with fentanyl mixtures on the photonic sensor to see if there is good liquid flow and separation can me made. In this phase, we were able to use a dye as a substitute to separate on a fabricated silicon dioxide substrate photonic sensor and see liquid flow.

TEAM MEMBER INFORMATION

Ugerah Abalu:

I am a senior in Electrical Engineering with a focus in Power Systems

Yifu Zhang:

I am an Electrical Engineering student with focus on integrated circuit. I recently already applied to the concurrent program at Iowa State University.

Hao Wang:

I am a senior Electrical engineering student with the interest area in VLSI. I used my knowledge in logic circuits to help my team. My major contribution in the group was to test the photonic sensor and give feedback to the fabrication group.

Oluwole Eteka:

I am an Electrical Engineering student with focus on integrated circuit. I am a senior in my last semester for a bachelor degree. I am more interested to work in electronic and automation systems but I have some past experience in power from my last internship.

Kossi Egla:

I am a senior in Electrical Engineering and my focus is analog and digital VLSI design. After graduation, I will be working as electronic manufacturing engineer. Hope to use my engineering experience and solve problems where needed.

Zhengyang Tang:

I am an Electrical Engineering student with the interest area in integral circuit and microelectronic. After graduation, I hope to use my knowledge to solve the problem and learn new technique during work

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