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Colloidal Photonic Crystals and its Applications

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1 Introduction

1.1 Background

We all perceive the beautiful aspects of the world around us through color, which can be defined as our perception of light. A color perceived system mainly consists of three parts: a source of illumination, an object to interact with the light and a viewer to perceive the results.

Nature produces color mainly through these three phenomena: pigments, structural color, bioluminescence or a combination of these phenomena. Pigmentary color is a result of selective light absorption by the electrons of pigments. When an object absorbs light of given color, it is the complementary color that is observed. This is the basis of subtractive color mixing theory for pigments. Our project mainly focuses on structural colors, which results from the interaction of light with ordered structural arrangements within the object facilitating selective light reflection. This can be due to interference (thin film and multifilm), diffraction, scattering and photonic crystals.

Structural colors can be further categorised into iridescent or non-iridescent colors. Iridescent colors are defined as colors that change based on the viewing angle of the observer. Such colors are usually the consequence of interference, diffraction, some forms of scattering and photonic crystals, and cannot be reproduced through pigmentary colors. Scattering often produces non-iridescent colors as it results from the irregularity of the structure [1]. Peacock tail feathers is a perfect example of iridescent color formation. The brown-pigmented feathers have structures, which on interaction with light produces blue-green shades.

The principles behind structural colors and their natural presence are discussed in the upcoming sections.

1.2 Physical Mechanism of Structural Colors

1.2.1 Interference

Structural colors are mainly produced through thin film or multi-film interference. In a thin film, light which is reflected from the upper and lower boundaries interfere with each other either constructively or destructively. When light passes from a rarer to denser medium, the reflected light has a phase difference

of π and the transmitted light has no phase difference with the incident ray. The formula for constructive interference of reflected light in anti-reflection coatings having d and n_2 as the thickness and refractive index of the film respectively, is given by:

$$2n_2dcos\theta_1 = m\lambda$$

where θ_1 is the incident angle and λ is the wavelength giving the maximum reflectivity (Figure 1).

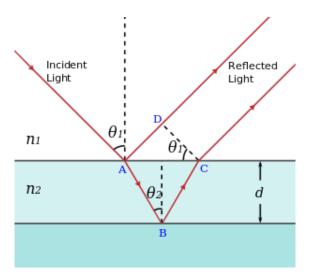


Figure 1: Thin film interference

Multi-film can be considered as a stack of alternate layers of thin-film having different refractive indices. The infinite version of a multi-film forms a 1-D photonic crystal. The condition for constructive interference for a system having alternate layers of refractive indices n_A and n_B with thickness d_A and d_B respectively, is given by (Figure 2):

$$2(n_A d_A cos\theta_A + n_B d_B cos\theta_B) = m\lambda$$

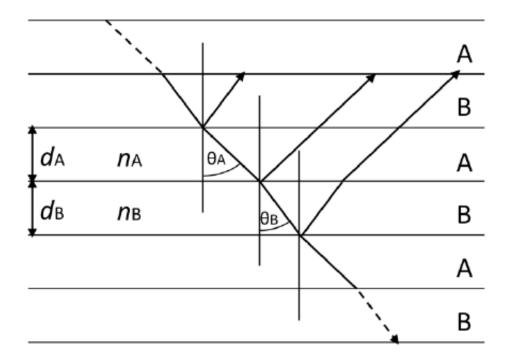


Figure 2: Multi-Film Interference

1.2.2 Diffraction gratings

Diffraction gratings are surfaces periodically corrugated along a particular direction. In Figure 3, α and β are the incident and diffracted angle respectively on a diffraction grating having d as the spacing between the grooves[1]. The grating equation is given as follows:

$$m\lambda = d(\sin\alpha + \sin\beta)$$

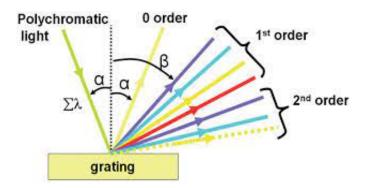


Figure 3: Diffraction

When m=0, the grating behaves like a mirror, resulting in no diffraction and separation of wavelengths. This leads to the law of reflection $\alpha = \beta$ and the

grating equation becomes:

$$m\lambda = 2dsin\alpha$$

1.2.3 Photonic Crystals

Photonic crystals (PCs) are optical periodic nanostructures in one, two or three dimensions having periodicity in terms of refractive index and occurring periods of the order of optical wavelength. In recent years, they have found applications in quantum optics, LED Displays, photonic ICs, photonic crystal fibers and many more.

Photons exhibiting wave-like behaviour propagate through the crystal as modes and a group of such allowed modes form optical bands. The forbidden modes constitute the Photonic Band Gap(PBG)[2]. These band gaps result due to destructive interference of light waves originating from the reflections at the interfaces of high and low refractive index materials. For the range of frequencies in the band gap, light cannot propagate through the material in any direction. One must note that the periodicity of the crystal structure must be around half the wavelength of the electromagnetic waves to be diffracted.

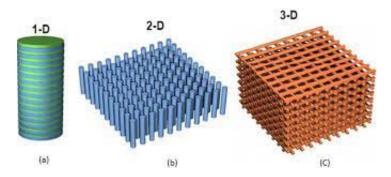


Figure 4: Photonic crystals in 1-D,2-D and 3-D

In 3-D photonic crystals, the color depends on refractive index, tilting angle and the spacing between the close cubic packed (CCP) planes[1]. The equation for the same can be derived by combining Bragg's law and Snell's law:

$$m\lambda = 2d_{111}\sqrt{n_{eff}^2 - \sin^2\theta}$$

1.3 Nature and Structural Colors

There are multiple evidences of structural colors in nature. These examples inspired researchers over the years to fabricate new materials and devices mimick-

ing the structures present in birds, insects and animals for various applications.

1.3.1 Peacock Tail Feathers

The presence of 2D photonic crystals (made up of melanin rods linked by keratin) on the surface of peacock barbules causes the iridescence seen in their tail feathers. The lattice structure of the blue, green, and yellow barbules is almost square, whereas the lattice structure of the brown barbule is rectangular. The lattice constant (rod spacing) and the number of periods (melanin rod layers) in the direction normal to the cortex surface are the only differences, resulting in diverse colors. Furthermore, the number of periods influences the production of additional hues, ultimately resulting in additive, mixed coloration[3].

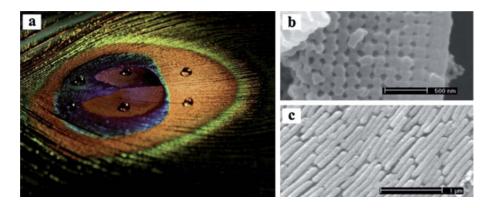


Figure 5: Photonic crystals in peacock feathers [3]

1.3.2 Opal

Another example of naturally occurring photonic crystal is the gemstone Opal. The iridescence observed in opal is due to the close packed, regular FCC arrangement of silica spheres as shown in Figure 6. The mean sphere diameter of the silica spheres in the Scanning Electron Microsope (SEM) image shown below is approximately 280 nanometers(nm)[4], which is half the wavelength of visible light. As light propagates through the opal structure, it gets diffracted and interferes with each other constructively or destructively. Artificial photonic crystals have been synthesised by exploiting the opal structure.

1.4 Objective 7

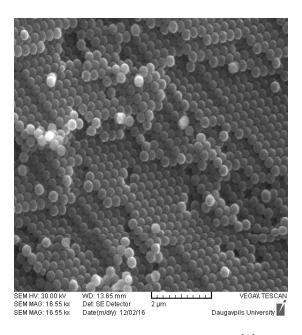


Figure 6: SEM Image of Opal[4]

1.4 Objective

In this project, we are going to synthesize silica colloidal photonic crystals and examine the effect of incorporating carbon based materials such as graphene oxide, reduced graphene oxide (rGO) and graphene quantum dots into the synthesised CPCs.

2 Literature Review

2.1 Colloidal Photonic Crystals

When monodisperse colloidal particles are concentrated or have repulsive interparticle potentials, they tend to form highly ordered arrays known as colloidal crystals. These crystals possess periodic variation in refractive index enabling selective diffraction of light. Colloidal Photonic Crystals (CPCs) exhibit iridescence especially when the colloid particles size is approximately equal to half the visible light wavelength (a minimum condition for diffraction of light)[5]. Different colors can be obtained by varying the periodicity, tilting angle and refractive index of the lattice structure. In a colloidal crystal, diffraction takes place through different planes present in the structure having spacing between

them as d_{hkl} . The wavelength of light for constructive interference is given by:

$$n\lambda = 2d_{hkl}\sqrt{n_{eff}^2 - sin^2\theta}$$

where

$$d_{hkl} = a/(\sqrt{h^2 + k^2 + l^2})$$

and

$$n_{eff}^2 = n_p^2 \phi_p + n_m^2 \phi_m$$

where ϕ_p and ϕ_m are volume fractions of the close packed colloidal particles and matrix, respectively. We must note that colloidal crystals usually appear as close-packed or non-close packed FCC lattice (sometimes BCC lattice is formed for highly repulsive colloids)[5]. CPCs having close packed FCC structure is called opal structure, which owes its name to the FCC lattice of silica particles present in natural opal.

In upcoming sections, we would briefly discuss about opal and inverse opal CPCs.

2.1.1 Opal CPCs

As mentioned before, opal CPCs have close packed FCC lattice of colloidal particles. Their formation is governed by the principle of thermodynamic stabilty by maximising entropy. As the volume fraction of the colloidal particles dispersed in the liquid medium increases, their brownian motion is strongly restricted by the neighbour particles (particularly above 49.4 volume fraction). Under such conditions, ordered arrangement of spheres is favoured as it leads to increased freedom for the particles to move leading to more configurational states and more entropy. Although FCC is known more favored than HCP, entropy discrepancy is very small, leading to coexistence of both structures.

Dip coating is the most common method for synthesising opal CPCs. The hydrophilic substrate is dipped into the aqueous colloidal suspension and slowly taken out and kept until all of the water has evaporated. Water evaporates faster on the meniscus than on other surfaces, causing particles to migrate from the bulk to the meniscus. As a result, particles condense near the meniscus before crystallising on the substrate. Only the close-packed FCC opal structure remains on the substrate after complete drying.[5].

2.1.2 Inverse Opal CPCs

The name being quite intuitive, inverse opals are inverse replicas of opal CPCs, with a close packed FCC lattice of spherical voids surrounded by a solid matrix. The synthesis of inverse opal CPCs involve solidification of sol in the void spaces of opal structure and subsequent removal of the opal structure template. The motivation for making inverse opals includes their use in catalysis, sorption, chromatography, battery materials, and as bioactive materials – uses that benefit from the highly accessible surfaces and relatively large pore sizes of these materials. Moreover, inverse opal CPCs are more preferred for sensor based applications as they have higher void fraction of 0.74.

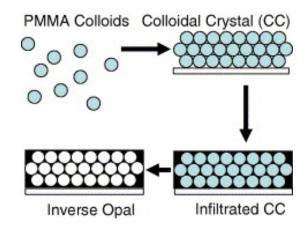


Figure 7: Fabrication of Inverse Opal CPCs [6]

2.2 Carbon Nanomaterials

Carbon based nanomaterials have gained huge research interest worldwide since the discovery of fullerenes in 1985 by H.W. Kroto, R.F. Curl and R.E. Smalley. This was accompanied by the discovery of carbon nanotubes by Japanese researcher S. Iijima in 1991 and that of graphene in 2004. Their extraordinary properties have made carbon nanomaterials a necessity in the field of technology and medicine. in our report, we would be mainly dealing with graphene derivatives such as graphene oxide (GO), reduced graphene oxide (rGO) and graphene quantum dots(GQDs).

2.3 Graphene Oxide 10

2.3 Graphene Oxide

Graphene - the exfoliated form of Graphite has been considered a wonder material for the past few decades due to its extraordinary mechanical and optical properties. Since synthesis of high-quality graphene at an industrial scale at low cost is challenging, other alternative forms of graphene which has similar properties and cam be synthesised easily, are preferred. Graphene oxide (GO) is one of the derivatives of graphene that has garnered attention among researchers in recent years. The structure of graphene oxide is obtained when the contiguous aromatic lattice of graphene is attached with oxygen containing groups such as ketones, carbonyls, alcohols and carboxylic groups.

2.4 Synthesis

2.4.1 GO Synthesis

The most common method for GO synthesis is by treating graphene with oxidising agents like H_2SO_4 and $KMnO_4$. Hummer's method/ Modified Hummer's method have been widely accepted for GO synthesis since 1958[7].In this method, the oxidation of graphite to graphitic oxide is achieved by treating graphite with essentially a water-free mixture of strong oxidising agents namely concentrated sulfuric acid, potassium permanganate and sodium nitrate. The resultant graphitic oxide is then exfoliated using an ultrasonicator to form graphene oxide.

The use of strong oxidants in these methods introduces many defects in the crystalline network of GO which results in the decrease in the conductive properties of GO as compared to graphene[8].

2.4.2 Stober Process for silica synthesis

Stober process is a commonly used process for synthesis of silica microspheres [9]. In this process, tetraehylorthosilicate (TEOS) is taken as a starting material and is mixed with ethanol. Then, conc. NH_4OH and ethanol are mixed separately in another beaker. The two solutions are mixed with each other after stirring for 5 mins. The resultant solution was allowed to stir for 1 hour. This reaction produces ethanol and a mixture of ethoxysilanols, which can then condense with another silanol or TEOS with the loss of alcohol and water.

Hydrolysis:

$$Si(OEt)_4 + H_2O \longrightarrow Si(OEt)_3OH + EtOH$$

Condensation:

$$2Si(OEt)_3OH \longrightarrow (EtO)_3Si - O - Si(OEt)_3 + H_2O$$

Subsequent hydrolysis and condensation leads to crosslinking. The process results in formation of silica particles with their size ranging from 50-2000 nm.

3 Materials and Methods

3.1 Materials Used

For the synthesis of silica CPCs, ammonia (20-30 %, Merck), TEOS(Sigma-Aldrich) and ethanol (SRLChem) were used. De-ionised (DI) water was used as prepared. For GO synthesis, graphite powder was purchased from Sigma-Aldrich and used as received. Concentrated H_2SO_4 (98%), sodium nitrate ($NaNO_3$) and hydrogen peroxide(H_2O_2) were purchased from Merck. Potassium permanganate(Vetec) was used as received.

3.2 Preparation of silica microspheres

Silica microspheres were synthesised through the Stober process which has been explained in section 2.3.2. 4 ml ammonia, 7.68 ml ethanol and 8.32 ml DI water were taken in a beaker and the solution was stirred for 5 min. Meanwhile, 1 ml TEOS and 18 ml ethanol were taken in another beaker and was mixed thoroughly for 5 min. After 5 min, the two solutions were mixed and the resultant mixture was allowed to stir for 1 hour. The resultant colloidal solution was then centrifuged at 3600 rpm for 15 min. The settled residue was washed and kept for vertical deposition to form silica CPCs.

3.3 Preparation of GO powder

GO was synthesised based on the principles of modified Hummer's method. 0.5 g of graphite powder was taken in a beaker and mixed with concentrated sulphuric acid. The solution was kept for stirring for 15 min in ice cold condition. Subsequently, 2 g $NaNO_3$ was added and the mixyure was allowed to stir for half an hour in ice cold condition. 5 g of pottasium permanganate was added and the resultant mixture was stirred for 4 hours at 35° C. The mixture was

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then diluted with DI water and 10 ml hydrogen peroxide was added resulting in effervescence. It was allowed to stir for 10 min and the final solution was thoroughly filtered and centrifuged until it attained neutral pH to obtain pure GO free from any contamination.

The obtained GO dispersed solution was sonicated using an ultra-sonicator, filtered and dried at 60 ° C to obtain exfoliated GO sheets. The exfoliated GO sheets were then grinded to form GO powder. Figure 8 shows the schematic of the procedure followed for the synthesis and the obtained GO powder.

SYNTHESIS OF GO BY HUMMER'S METHOD

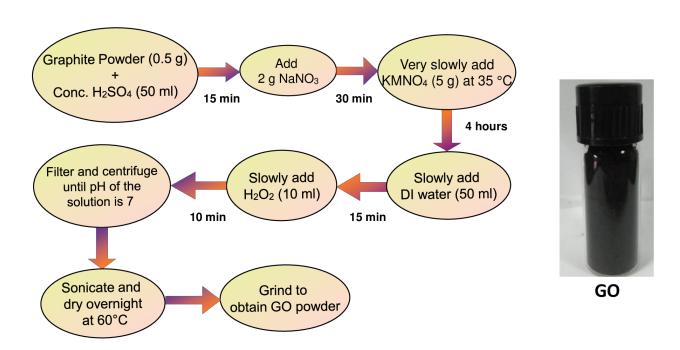


Figure 8: Synthesis of GO using Hummer's Method

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An Application Demonstration based on CPCs Developing an Android Application Based on Tear Fluid Glucose Sensing

1 Abstract

Blood glucose sensing is an important parameter for monitoring diabetic symptoms among patients. Recent technologies available in the consumer market involves the use of glucometer, which works by analysing a small amount of blood from your fingertip. However, this method is quite expensive and painful too. Hence, recent research on glucose monitoring explores non-invasive ways to detect monitor glucose level among diabetic patients. One such way is to monitor tear glucose level and correlate that with blood glucose level.

In our report, we have developed an Android application (app) based on the work of Alexeev et al.[1] with the aim of making glucose monitoring more accessible and affordable. The app has been developed in Android Studio and the code was written in Kotlin language. The purpose of the app is to provide the glucose concentration corresponding to the change in color of the contact lens incorporated with a tear glucose sensing photonic crystal.

2 Background

2.1 Tear Glucose Monitoring using PCs

In our previous report on colloidal photonic crystals, we have discussed in detail about photonic crystals (PCs). PC based sensors change color according to the change in concentration of the analyte used in the experiment.

In the sensor developed by Alexeev et al., the photonic crystal sensing materials would be contained in a contact lens or ocular insert. The sensing material consists of a face-centered cubic array of colloidal particles embedded in a hydrogel. The array spacing is 250 nm, designed such that the array diffracts visible light. The color diffracted changes with the tear glucose concentration. A simple mirrored compact-like device would illuminate the sensor material with white light. The color of the sensor would be determined by viewing the reflected (diffracted) light and comparing it with an exterior color wheel calibrated in terms of the blood glucose concentration [1].

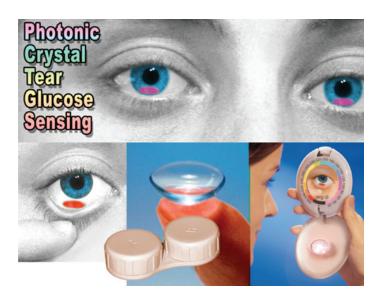


Figure 1: Tear based Glucose sensing PCs [1]

2.2 RGB Color Model

Any color model is designed to produce more colors from a set of few primary colors. The RGB color model is based on the principle of additive color mixing theory where the three primary colors- red, green and blue are mixed in different proportions to produce a myriad of other colors. We can have 256 different shades for each primary color, resulting in over 16 million unique combination of colors.

When one of the components has the highest intensity, the color is a hue close to the primary color (reddish, greenish, or blueish), and when two components have the same highest intensity, the color is a secondary color hue (a shade of cyan, magenta or yellow). The sum of two primary colors of equal intensity creates a secondary color: cyan is green+blue, magenta is blue+red, and yellow is red+green. The result of mixing all of the primary colors in equal intensities is white [2].

So why is RGB color model significant in this project? The captured image by the camera of your Android phone is nothing but a collection of large number of pixels. Each pixel is composed of tiny but orderly arranged and closely packed RGB light sources. The corresponding RGB value in every pixel adds up to determine the color of the entire image. Hence, knowing the RGB value of a pixel provide us direct information about the color in that area.

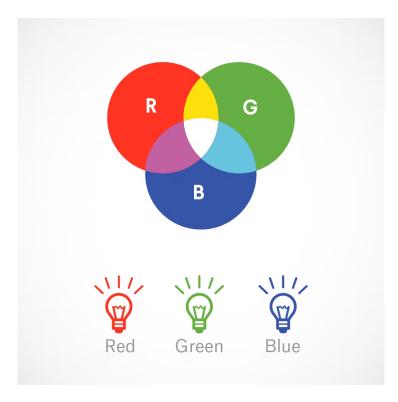


Figure 2: RGB Color model

2.3 HSV Color Model

The RGB color model can be regarded as a computer-friendly language. However, we humans do not see every color as a combination of red,green and blue lights. We may be able to designate magenta as a mixture of red and blue easily, but we would find it difficult to define dark green using the RGB model. To eliminate this issue, the HSV color model was developed in the mid 1970s. It is a cylindrical color model which remaps the RGB model into a 3-D space incorporating saturation and lightness.

'H' stands for hue of a particular color, which can be defined as the angle at which that particular color is present on the RGB color wheel. Hence, 0° is red, 120° is green and 240° is blue.

'S' stands for saturation which determines the amount of color used. It can be thought of as how much gray is added to the system. A color with 100 % saturation gives the pure primary color and the same color with 0 % saturation yields gray.

'V' stands for value, which determines the brightness. A color with 0 % brightness is completely black while a color with 100 % brightness yields the purest color. Since value is equivalent to brightness, the HSV color model is also referred to as HSB color model.

2.4 Android Studio

Android Studio is the official Integrated Development Environment (IDE) launched by Google in co-operation with JetBrains, developed specifically for Android development. It's based on the IntelliJ IDEA editor, which supports features such as smart editing, advanced code refactoring, and deep static code analysis[3]. It also allows you to run and debug your applications in an Android Virtual Device (Emulator). The latest versions of Android studio supports many programming languages such as Java, C++, Kotlin, etc.

2.5 Kotlin

Kotlin is a multi-paradigm, object oriented programming language developed and designed by JetBrains in 2011, specifically to fulfill the need of extra features for Android developers. The latest version of Kotlin: Kotlin 1.7 has been released on June 9, 2022. Kotlin was announced as an official Android development language when it was launched at Google I/O in May 2017.Kotlin is still the most extensively used programming language on Android as of 2020, with Google predicting that 70 percent of the top 1000 apps on the Play Store are built in this language. It has been found that Google uses Kotlin across 60 of its programmes, including Maps and Drive. Many Android apps, such as Google's Home, are being converted to Kotlin and so require both Kotlin and Java. On Android, Kotlin is praised for its null-pointer safety as well as features that make code shorter and more readable [4].

3 Design

Figure 3 shows the User Interface (UI) of the application. There are two buttons on the top: one to capture image using the camera integrated in the Android device and the other to pick image from the photo gallery of your device. Below the two buttons is an ImageView, which will load the image captured with camera or taken from gallery into the app. A color view below the ImageView will display the color of the pixel where your finger is placed on the image. The RGB value, HSV value and the glucose concentration for that particular color will be simultaneously recorded.

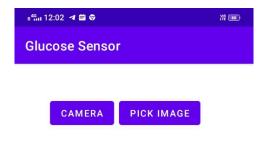


Figure 3: Application User Interface

4 Explanation of Program Listings

4.1 Capturing Image using Camera

The given program is written in $Main_Activity.kt$ file and inside the onCreate() function. In line 2, the app is instructed to recognize the camera button through its ID, which was defined while creating the $activity_main.xml$ file. Lines 5 and 6 are written to clear the cache after every new image is loaded by the user. In the next line, an intent is defined which acts as a messenger and requests another app component (here, camera) to launch the requested activity. If camera returns the request, then the function startActivityForResult() launches the camera of the android phone.

```
camera=findViewById(R.id.capImage)

camera.setOnClickListener {
    imageView.setImageBitmap(null)
    imageView.destroyDrawingCache()
    val takePictureIntent = Intent(MediaStore.ACTION_IMAGE_CAPTURE)

if (takePictureIntent.resolveActivity(this.packageManager)!=null) {
        startActivityForResult(takePictureIntent, RequestCode)
    }else{
        Toast.makeText(this,"Unable To Open Camera", Toast.LENGTH_SHORT).show()
    }
}
```

The function onActivityResult() is declared outside the onCreate() function. after the intent is launched and the camera captures the image, the image is stored in form of a bitmap 'takenImage'. The ImageView is then set to the recorded bitmap to display the loaded image.

```
override fun onActivityResult(requestCode: Int, resultCode:Int, data: Intent?) {
    if(requestCode==RequestCode && resultCode== Activity.RESULT_OK){
        val takenImage = data?.extras?.get("data") as Bitmap
        imageView.setImageBitmap(takenImage)
    }else {
        super.onActivityResult(requestCode, resultCode, data)
    }
}
```

4.2 Picking Image from Gallery

The registerForActivityResult() function registers the user's callback and sets the image URI to the ImageView. The setOnClickListener() button has the same function as in the case of camera button that is it redirects the user to the gallery once it is clicked. The chosen image is then displayed on the ImageView.

4.3 Recording Glucose Concentration from the color

The setOnTouchListener() function on the imageview records the user's touch on the image loaded. The red,green and blue colors of the corresponding pixel are

assigned to the variables r,g and b respectively. The recorded color is then displayed in the colorview.

From line 14, we are converting the obtained RGB value to HSV value. We are then declaring two new variables of double type: gc for glucose concentration and lambda for wavelength. We are only considering colors with saturation and value from 50 % to 100 % because this range yields the pure and bright color with minor variations. The fact that RGB values cannot be converted to wavelengths necessitated the conversion of RGB to HSV values. However, hue values between 270 and 360 yield pink or magenta which do not have a corresponding HSV value in the spectrum. For hue values between 0 to 270, there is an approximate linear relationship between wavelength and hue[5].

The parameters q0,a0 and b0 are obtained by doing a hyperbolic decline curve fit on the plot between diffracted wavelength and glucose concentration given in Alexeev et al.'s paper[1]. The obtained equation is directly inserted into the code.

For any other color, apart from those described above, the glucose concentration is set to 0 and the result displayed is 'Unknown'.

```
imageView.setOnTouchListener { v, event ->
               if(event.action == MotionEvent.ACTION_DOWN || event.action ==
     MotionEvent.ACTION_MOVE){
                   bitmap=imageView.drawingCache
                   val pixel=bitmap.getPixel(event.x.toInt(),event.y.toInt())
                   val r =Color.red(pixel)
                   val g=Color.green(pixel)
                   val b= Color.blue(pixel)
                   colorView.setBackgroundColor(Color.rgb(r,g,b))
12
                   val hsv = FloatArray(3)
                   Color.RGBToHSV(r,g,b,hsv)
14
                   val hue = hsv[0].toInt()
                   val sat= (hsv[1]*100).toInt()
                   val value=(hsv[2]*100).toInt()
18
19
                   var gc:Double=0.0
20
                   var lambda: Double=0.0
                   val q0 = 627.5736999
23
                   val a0=3.522073
24
                   val b0= 15.01114
26
                   if (hue in 0..270 && sat in 50..100 && value in 50..100) {
27
                       lambda = 650.0 - 0.925*hue
                       gc = (a0/b0)*((lambda/q0).pow(-b0)-1)
                   }else{
30
                       gc = 0.000
31
```

```
32
                   val dec = DecimalFormat("##.000")
                   gc = dec.format(gc).toDouble()
34
                   if(gc!=0.0) {
36
                       displayText.text = "RGB: $r,$g,$b \nHSV: $hue,$sat,$value \
37
     nGlucose Concentration(mM): $gc "
                   }else{
38
                       displayText.text = "RGB: $r,$g,$b \nHSV: $hue,$sat,$value \
39
     nGlucose Concentration(mM):Unknown "
41
             }
42
43
               true
```

5 Results and Discussions

Figure 4 below shows the display of the glucose concentration corresponding to the colour change in the sample. The picture has been taken from Alexeev et al.'s paper on tear glucose sensing[1]. The same results will be obtained if the user takes the picture of the contact lens used and analyses the glucose concentration.



Figure 4: Color and Glucose concentration displayed for 4th sample

References

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