Chapter 5

DUAL MODE PHOTOMETRIC AND NEPHELOMETRIC BASED SENSING FOR ACCURATE ESTIMATION OF SULPHATE AND CHLORIDE LEVEL IN WATER

This chapter discusses the development of a smartphone-based dual mode sensing system that has been used to monitor multiple water quality parameters. The designed tool is based on the detection of transmitted (for photometric based study) and scattered light signal (for turbidimetric based study) intensities from a test sample. The ambient light sensor (ALS) of the phone has been used as a detector for receiving the modulated signals for both the sensing modes of study. The applicability of the designed tool has been realised through monitoring of sulphate and chloride ions concentrations in water. By using the custom developed Android application, the analysis of the considered parameters can be evaluated within the phone itself thus making it a complete standalone system.

5.1 Introduction

Natural water resources contain various dissolved minerals and chemicals such as fluoride, iron, phosphate, nitrite and chlorine etc. Sulphate and chloride are two important elements that are commonly present in all the natural water sources. Both the elements are essential electrolytes which are responsible for many biological activities in human body. Sulphate is important for cell growth and detoxification of the human body [1]. Similarly, chlorides maintain the acidity and hydration level inside the body [2]. Although sulphate and chloride are considered to be important for a healthy diet, still some harmful effects can be observed due to excessive consumption of these chemicals. In general, water resources contain a low amount of sulphate and chloride. However, the growing anthropogenic activities such as improper disposal of industrial waste alter the actual compositions of these naturally occurring minerals. According to the WHO guideline on drinking water safety, the permissible limit of sulphate and chloride in drinking water should not exceed 250 ppm [3, 4]. Usually the excess amount of sulphate and chloride in water enhances the taste and odour to a bitter level. Large amount of sulphate intake can cause diarrhoea. Similarly, hypertension is reported due to the presence of excessive chloride in drinking water [5]. Thus monitoring of these two parameters is critical to prevent from mineral deficiency and excessive consumption.

In recent years numerous smartphone-based analytical platforms have been reported to measure the concentration of these chemical parameters in water [6, 7]. Detection of iron, fluoride, chlorine, nitrite and phosphate has been successfully demonstrated by various research groups [8, 9]. Different colorimetry, photometry, fluorescence and nephelometry based sensing platforms have been built using smartphones that are low-cost, user-friendly and relatively easier to handle [10–12]. To retain the simplicity of the tools, most of these devices are designed by utilising a signal mode of sensing principle. However, very few works related to multiple analytical abilities on smartphone platform have been reported till date [13, 14].

Herein, a dual-mode smartphone-based analytical platform has been designed which has the ability to detect water quality parameter in two modes of sensing schemes- namely the photometric and nephelometric mode. Both the sensing schemes are combined on a single optical setup and then successfully fabricated it by using 3D printing technology. The designed sensor utilises the ambient light sensor (ALS) as a principal detector for detection of modulated signals in both the schemes. The photometric mode records the light that passes through the test sample whereas in nephelometric mode, the sensor records the side scattered light signal. On collecting the light signals, the platform evaluates the optical density (OD) and turbidity of the test sample using the respective sensing mode. The designed platform is the first-ever demonstration of a multi-modal sensing system that uses the ALS of a phone as a signal recorder. The designed tool has been utilised to measure the sulphate and chloride concentration in field-collected water samples. The performance of the tool has been compared with the commercial grade spectrophotometer and nephelometer. A custom designed application has been developed to convert the sensing data into a readable format and making the design system a user-friendly analytical tool. The present study successfully demonstrates the reliable estimation of multiple analyte on a multi-modal smartphone-based analytical platform. The platform is also suitable for monitoring other water quality parameters such as turbidity, salinity, pH, hardness and dissolved oxygen utilising the photometry and nephelometry based sensing assays.

5.2 Experimental design of the dual sensing platform

Figures 5.1 (a) and (b) illustrate the schematic representation of the proposed platform while using it for photometric and turbidimetric based sensing studies respectively. Using the latest 3D designing and printing technology, a robust plastic optical setup has been fabricated and attached with the smartphone shown in Figures 5.1 (c) and (d). The designed attachment is coupled with the ALS of the phone to receive the transmitted as well as the scattered light signals at 90° from the test samples. LEDs are used as primary light source that has been powered by the internal battery of the phone using USB-OTG protocol. LEDs with appropriate peak emission wavelength can be incorporated in the designed tool to carry out any photometric-based sensing. For nephelometric-based sensing, a white LED (*RS Components*, Part No: VLHW4100) has been utilised as an optical source. The designed setup consists of two compact plano-convex lenses, each of focal length 12 mm and diameter 6 mm (*Edmund Optics*, Product Id.: 32-471). One of the lenses is used to collimate the light signal emitted by the LED sources. During the investigation, when light signal is allowed to pass through the test sample, the second lens guides the transmitted signal towards the ALS of the phone for photometric based sensing. In case of nephelometric based sensing, the ALS directly receives the side

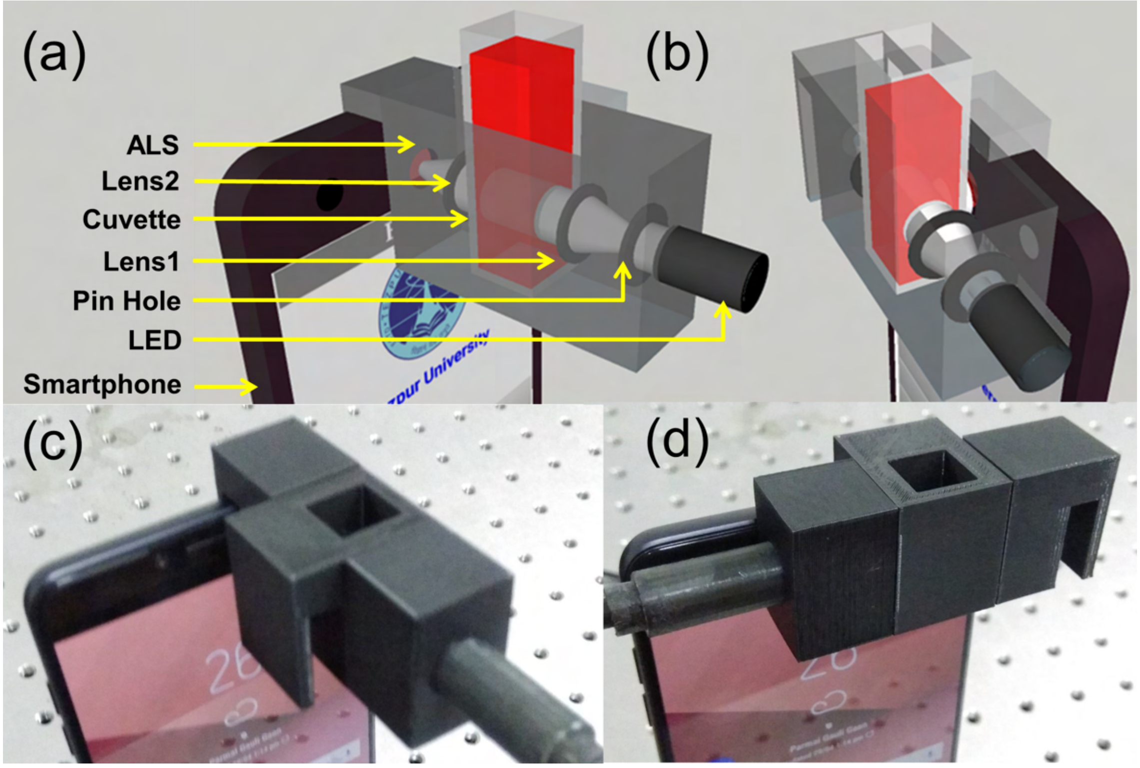

Figure 5.1: Schematic of the designed dual mode smartphone platform (a) photometric mode and (b) turbidimetric mode, (c) and (d) photo-image of the 3D printed dual mode smartphone platform.

scatted light signal from the sample. The test sample has been placed in the optical setup in a 10 mm×10 mm quartz cuvette (*Erma*, Product Id.: RE-019). The optical path lengths of the photometric and turbidimetric modes of studies are measured to be 15 mm and 10 mm respectively. In the optical design, a pinhole of diameter 50 μm has been placed in front of the optical source to obtain a well collimated beam of light signal. The dimension of the optical setup is measured to be of 70 mm in length, 40 mm in breadth and 40 mm in height. Technical specifications of the phone used in the present investigation can be found elsewhere [15]. The overall weight of the sensing platform, including the phone, is estimated to be 250 g. In the validation process of the designed tool, concentrations of sulphate and chloride in water samples have been estimated and rigorously tested for the platform's performance through various experiments which are discussed in the later section of this chapter. While assessing the sulphate and chloride concentrations, two different LEDs with peak emission wavelength 420 nm (*RS Components*, Part No: 247-1690) and 571 nm (*RS Components*, Part No: 255-8472) respectively have been plugged-in to the optical setup.

5.3 Development of an Android application for dual mode sensing

A custom designed Android application has been developed to evaluate both the optical density (OD) and turbidity of the test samples for the proposed dual-mode sensing platform. Figure 5.2 shows the screenshot images of the important steps of the developed application. Upon starting the application, a user convenient interface will pop up shown in Figure 5.2 (a). From the available options, a user can select the two modes of sensing option. On clicking the '*OD*' and '*Turbidity*' tab on the home screen, the application switches to photometric and turbidimetric based sensing mode respectively. Figures 5.2 (b) and (c) show the popup window for OD and turbidity measurements respectively. The Android app has also been explicitly customised to estimate sulphate and chloride concentration in water samples. Figures 5.2 (d) and (e) illustrate the steps to be followed for estimation of the considered analyte using the pre-calibrated calibration equations. Besides that, the application can also be re-calibrated in real time which may be required at the time of on-site investigations. Figure 5.2 (f) shows the screen shot image of the application while performing the re-calibration using standard solutions. The recorded data can be saved within the phone's internal memory as well can be shared to anywhere in the world through the available communication network. Figures 5.2 (g) and (h) show the screenshot images of the application by which a user can easily store and share the recoded data.

5.4 Material and methods

As a demonstration to justify the working of the designed dual mode smartphone tool, sulphate (SO_4^{2-}) and chloride (Cl^-) ions concentration have been estimated under various experimental settings. At first, the samples were prepared according to the standard chemical protocols in order to attain a selective evaluation of the considered analyte. Therefore, necessary chemicals were bought from certified suppliers and used as received without further purification. Hexamethylenetetramine ($(CH_2)_6N_4$), barium chloride ($BaCl_2$), magnesium chloride ($MgCl_2.6H_2O$), potassium nitrate (KNO_3), acetic acid (99%) (CH_3COOH), sodium chloride ($NaCl$), nitric acid (69%) (HNO_3) and absolute ethanol (C_2H_5OH) were procured from *Merck Sigma Aldrich*. Sodium sulphate (Na_2SO_4), sodium acetate ($CH_3COONa.3H_2O$), and silver nitrate ($AgNO_3$) were procured from *Renkem*, India. Hydrazine sulphate

(b)

Blank 32.78 LUX

OD 1.49 AU

(c)

Blank 2.91 NTU

Turbidity 92.01 NTU

(d)

Blank 877.03 LUX

OD 1.93 AU

Turbidity 73.43 NTU

Sample contain 143.83 ppm of sulphate

(e)

Blank 681.21 LUX

OD 1.12 AU

Turbidity 36.44 NTU

Sample contain 44.71 ppm of chloride

(f)

0.00 LUX

Conc.	Trans.	Scatt.	Unit
00	5363.00	1.00	LUX
10	4362.00	63.00	LUX
20	3256.00	99.00	LUX
30	2763.00	156.00	LUX
40	2163.00	249.00	LUX
50	1753.00	351.00	LUX

Slope: -71.83 6.75
Interce 5072.38 -15.76
R sqd.: 0.95 0.95

(g)

Snapseed
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Test Sample
Test Sample 1
Test Sample 2

(h)

File Name Test Sample
Enter Data Concentration i

Figure 5.2: Screenshot images of the custom developed Android application.

($(NH_2)_2$-H_2SO_4) was procured from *Sd Fine Chem Limited*, India.

5.4.1 Preparation of standard formazin solution

Initially, 400 NTU stock solution of formazin standard sample has been synthesized in the laboratory. To synthesize the stock sample, 1.00 g of hydrazine sulphate [$(NH_2)_2$ H_2SO_4] and 10.00 g of hexamethylenetetramine [$(CH_2)_6N_4$] were dissolved in 100 ml distilled water in two separate volumetric flasks. On complete dissolution of the chemicals, 5 ml each of the solvent was mixed in a 100 ml beaker and kept it in room temperature for 24 hrs. It is followed by dilution through the addition of 90 ml of distilled water to the freshly prepared solution. The turbidity of the obtained formazin sample is estimated to be 400 NTU which has been used as a stock solution in the present study. To prepare formazin samples of different turbidity levels, a proportionate amount of distilled water was added to the stock solution [16].

5.4.2 Preparation of sulphate sample

For estimation of sulphate (SO_4^2) ions in water samples, the standard APHA protocol has been adopted in the present study [17]. In this method, a suspension of barium sulphate ($BaSO_4$)has been prepared by adding $BaCl_2$ into the sample containing SO_4^2 ions in presence of KNO_3 buffer as shown in the chemical reaction 5.1. The prepared $BaSO_4$ suspension has been investigated under photometric and turbidimetric modes to evaluate the actual concentration of sulphate present in a sample. According to the 4500-SO_4^2E-APHA method, the peak emission wavelength of the optical source should be at 420 nm for an effective photometric analysis of the $BaSO_4$ suspension (OD420). Herein, 100 ppm of standard sulphate solution has been synthesized by adding 1.48 g of Na_2SO_4 in 1000 ml of distilled water. The buffer solution was prepared by mixing $MgCl_2.6H_2O$ (30g), $CH_3COONa.3H_2O$ (5 g), KNO_3 (1g), CH_3COOH (99%, 20 ml) and distilled water (980 ml) in appropriate volume.

$$(5.1) \qquad SO_4^{2-} + BaCl_2 \xrightarrow{\text{Acidic medium}} BaSO_4\downarrow \; + 2\,Cl^-$$

Formation of sulphate suspension

5.4.3 Preparation of chloride sample

The chloride samples have been prepared in a similar way as reported by Luce et al. [18]. The reported method suggests that chloride ions in water can be estimated by forming a silver chloride (AgCl) suspension as shown in the chemical reaction 5.2. The Cl^- ions present in the sample react with $AgNO_3$ to form a turbid solution that can be analysed using a spectrometer or a turbidimeter. To enhance the stability of the prepared turbid solution, a strong acidic medium was mixed with the solution. Following the chemical procedure, 100 ppm of Cl^- sample has been prepared by adding 1.65 g NaCl in 100 ml of distilled water. 0.01 M of $AgNO_3$ solution has been prepared by adding 0.170 g of dehydrated $AgNO_3$ reagent in 100 ml of 0.2 N HNO_3. The peak wavelength absorbance condition of the final solutions was found to be 571 nm (OD571).

$$(5.2) \qquad AgNO_3(aq) + Cl^- \longrightarrow AgCl\,(s)\downarrow + NO_3^-$$

Formation of chloride suspension

5.5 Estimation of sulphate and chloride ions concentration using the photometric mode of the designed platform

Figures 5.3 (a) and (b) represent the characteristic sensor responses while estimating the concentrations of sulphate and chloride ions in photometric sensing mode with the platform. A good degree of linearity has been noticed for both the analyte with coefficient of regression (R^2) of 0.92. For the designed sensing system, the linear ranges are found to be 0–100 ppm and 0–70 ppm for sulphate and chloride samples respectively. The reliability of the designed platform has been evaluated by comparing its responses with a standard spectrophotometer data (*Systronics India*, AU 2603). A strong correlation between the smartphone sensor's and the spectrometer data has been noticed shown in the Figures 5.3 (a) and (b). From the characteristic curves, two calibration equations have been obtained that can be used to analyse unknown water samples.

For sulphate,

$$C = \frac{(OD + 0.008)}{0.011} \tag{5.1}$$

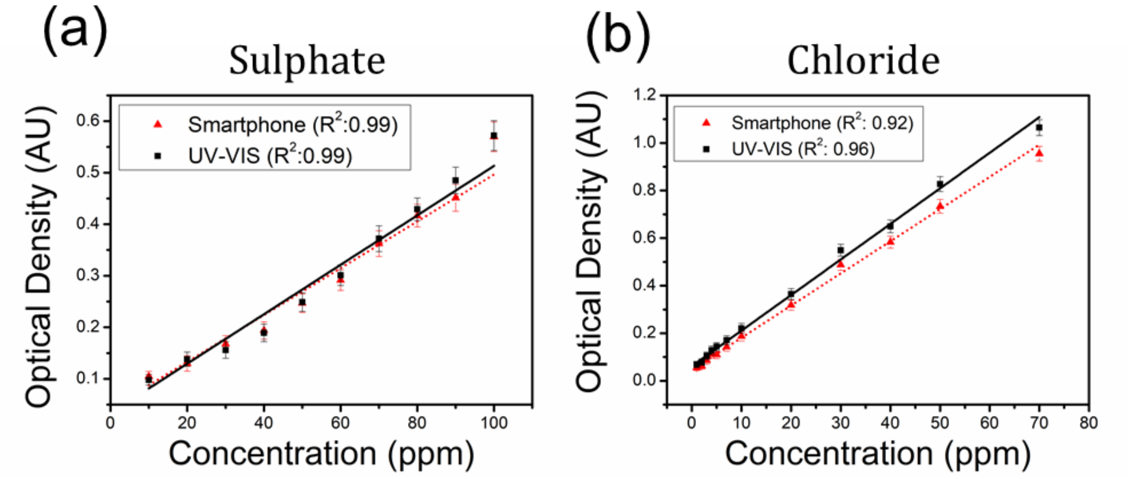

Figure 5.3: Calibration curves and performance comparison of the designed tool with the laboratory instrument while measuring (a) sulphate and (b) chloride ions using the photometric mode.

For chloride,

$$C = \frac{(OD + 0.05)}{0.013} \tag{5.2}$$

where, C is the analyte concentration in the sample and OD is the optical density measured by the designed platform.

5.6 Estimation of sulphate and chloride ions concentration using the nephelometric mode of the designed platform

The performance of the nephelometric mode of sensing has been evaluated by measuring sulphate and chloride ions. Prior to that, the nephelometric mode is calibrated using the standard formazin protocol. Figure 5.4 shows the characteristic sensor response curve while recording the light intensities scattered by formazin solutions with varying turbidity level. A linear variation in scattered intensities and turbidity levels has been observed within the range of 0 to 400 NTU. The R^2 value is estimated to be 0.99. Finally, a calibration equation 5.3 has been evaluated to measure the turbidity of an unknown sample from the recorded responses.

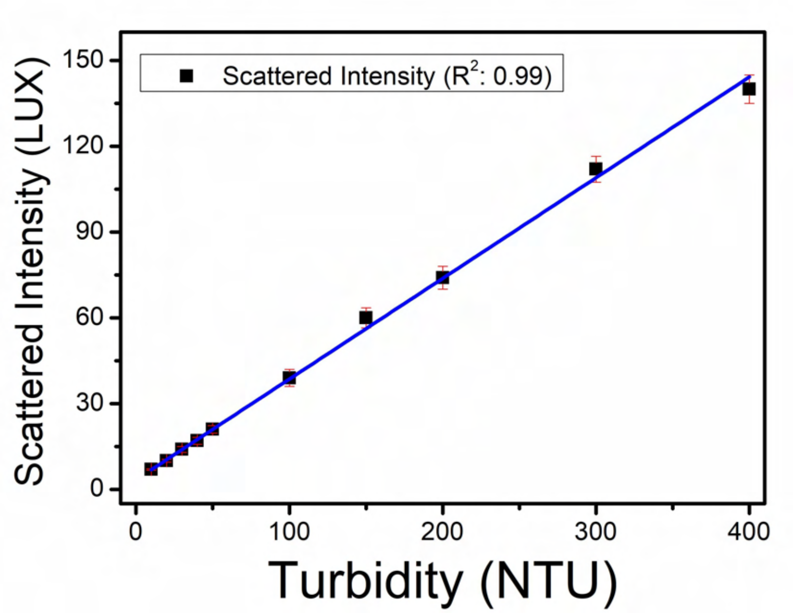

Figure 5.4: Calibration curve obtained for the standard formazin samples using the designed smartphone platform.

$$T = \frac{(I - 0.38)}{0.35} \tag{5.3}$$

where, T is the turbidity level of the standard formazin solution and I is the scattered light intensity measured by the designed platform.

Equation 5.3 has been employed to measure the turbidity of sulphate and chloride samples in the present investigation. Utilising the nephelometric mode of the designed platform, turbidity level of standard sulphate and chloride samples have been recorded as illustrated in Figures 5.5 (a) and (b). The figure shows the calibration curves that have been obtained for both the analyte with R^2 values more than 0.92. Nephelometric responses of the platform were then compared with a standard nephelometer (*Systronics India*, µC-turbidity meter). The calibration equations for sulphate and chloride ions are estimated as follows.

For sulphate,

$$C = \frac{(T + 2.37)}{0.68} \tag{5.4}$$

For chloride,

$$C = \frac{(T - 13.18)}{3.27} \tag{5.5}$$

where, C is the analyte concentration in the sample and T is the turbidity measured

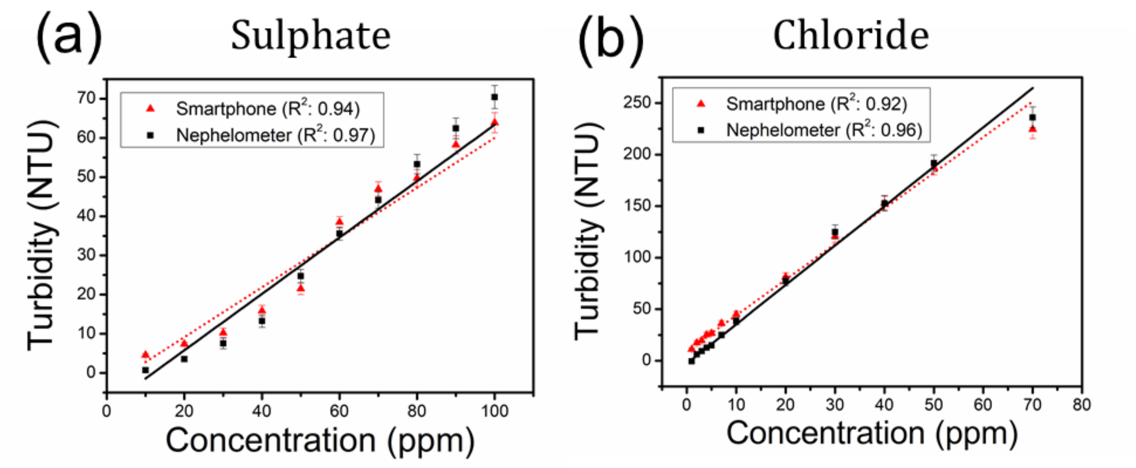

Figure 5.5: Calibration curves and performance comparison of the designed tool with the laboratory instrument while measuring (a) sulphate and (b) chloride ions using the nephelometric mode.

by the designed platform.

The sensitivity, limit of detection (LoD), relative standard deviation (%RSD) and percentage bias (%Bias) of the designed sensing system are shown in Table 5.1. The high sensitivity, low LoD limits and acceptably low %RSD and %Bias indicate that the designed platform is reliable for monitoring of sulphate and chloride ions with high degree of precision.

Table 5.1: Figure of merits of the designed platform while measuring sulphate and chloride ions concentration in water.

SN	Figure of merits	Sulphate		Chloride	
		photometry	turbidimetry	photometry	turbidimetry
1	Sensitivity	0.011 AU/ppm	0.68 NTU/ppm	0.013 AU/ppm	3.45 NTU/ppm
2	LoD	0.45 ppm	0.5 ppm	0.43 ppm	0.4 ppm
3	%RSD	8.99%	7.25%	8.94%	9.72%
4	%Bias	8.89%	8.69%	9.40 %	9.28%

5.7 Performance evaluation of the sensor in presence of other interfering elements

In the next step, the performance of the designed sensing platform has been evaluated in presence of other interfering elements. Four ions namely potassium (K^+), barium (Ba^{2+}), nitrite (NO^{2-}) and phosphate (PO_4^{3-}) have been considered to evaluate the variation in the sensor responses. Standard sample solutions of sulphate and chloride ions each of concentration 50 ppm have been prepared in the laboratory. Similarly, solutions of K^+ (10 ppm), Ba^{2+} (2 ppm), NO^{2-} (1 ppm) and PO_4^{3-} (0.1 ppm) are also prepared for the investigation. 5 ml solution of each of the interfering element was added separately to an equal volume of sulphate and chloride samples. Following this, the prepared solutions have been investigated under the designed tool. Figure 5.6 shows the characteristic histogram representation of the sensor responses recorded in the present study. The figure clearly shows that the sensor response has not been affected due to the presence of the interfering elements.

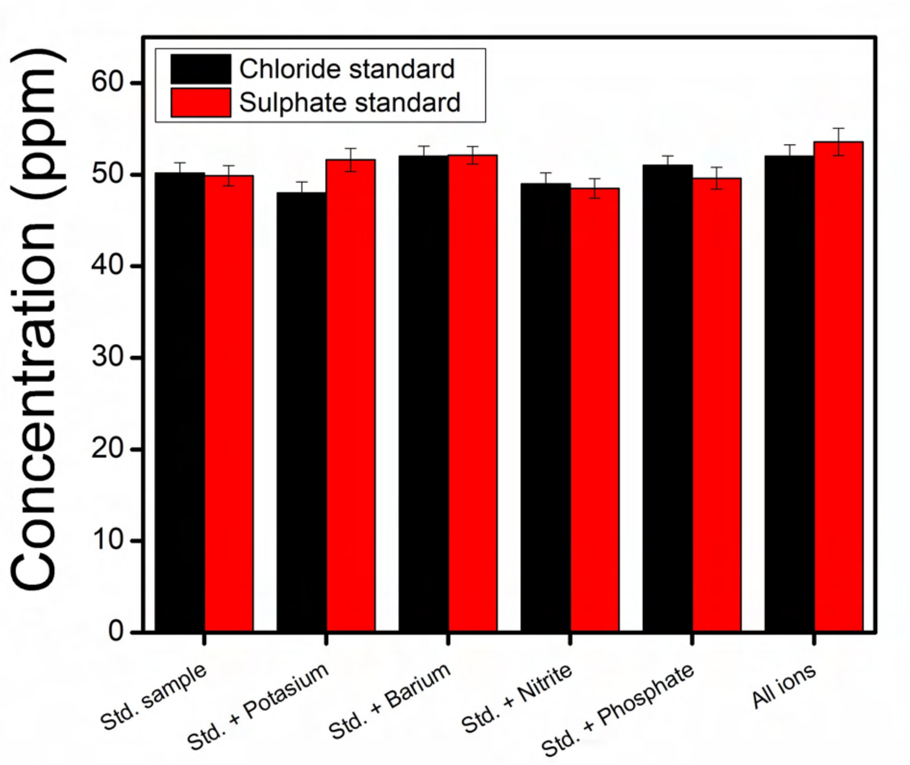

Figure 5.6: Histogram representation of designed smartphone platform responses in presence of K^+, Ba^{2+}, NO^{2-} and PO_4^{3-} ions with concentration 10 ppm, 2 ppm, 1 ppm and 0.1 ppm respectively in the standard sulphate and chloride samples.

5.8 Analysis of sensor response with field-collected water samples

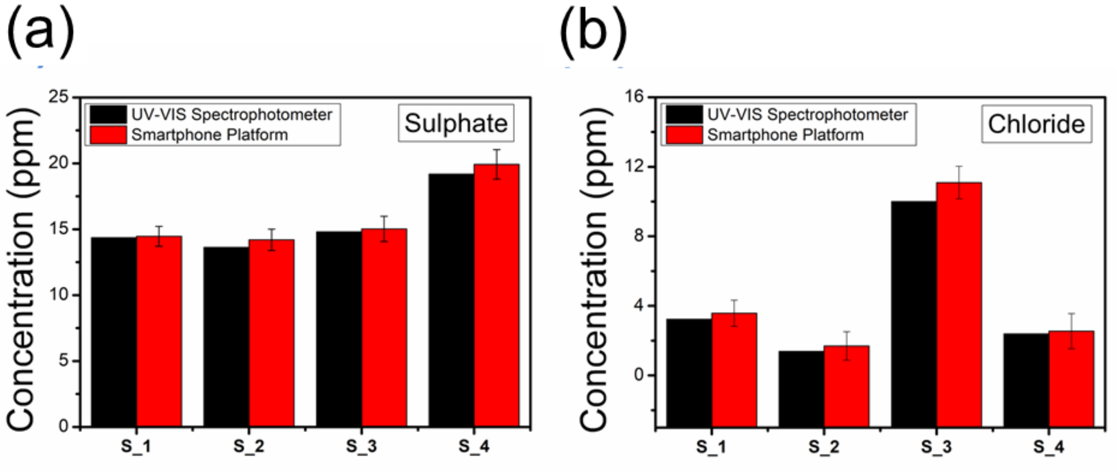

Figure 5.7: Histogram representation of (a) sulphate ion and (b) chloride ion concentration in field-collected water samples that have been estimated using the UV-VIS spectrophotometer and the smartphone based sensing platform.

In the final step, the field applicability of the proposed platform has been realised by monitoring the sulphate and chloride ions concentration of field-collected water samples. Samples from nearby water resources of Tezpur University, namely; well water of Napaam Village, Sonitpur (S_1), public water supply of Napaam Village, Sonitpur (S_2), swimming pool water of Tezpur University (S_3) and tap water of Tezpur University (S_4) were collected for the study. All the field-collected water samples were initially filtered with grade-1 *Whatman* paper thrice to remove the suspended particles. Following the procedures described in section 5.4, the water samples were treated with the standard reagents for selective determination of sulphate and chloride in water. Figures 5.7 (a) and (b) show the characteristic responses of the designed platform upon measuring the sulphate and chloride ions concentration of the considered water samples. The figures also include the corresponding spectrophotometer data of the same samples recorded by the standard UV-VIS spectrophotometer. A good degree of accuracy has been noticed while comparing the experimental data with the standard tool. The maximum variation between the data recorded by the designed tool and the standard tool was found to be 8.9% and 9.2% for sulphate and chloride ions respectively. The maximum %Recovery was calculated to be 104% for sulphate and 105% for chloride ions for the field-collected water samples.

Table 5.2: Performance comparison of the designed sensor with the works reported in the line of the present research

SN	Specification	Fluoride detection	Chromium Detection	Reported work
1	Mode of operation	Photometric	Readout device	Combined photometric and nephelometric
2	Operational range	0 – 20,000 lux	Not calculated	Turbidimetric mode: 0 to 4000 NTU Photometric mode: 0 – 50,000 lux
3	Sensitivity	Not calculated	Not calculated	Turbidity: 0.05 NTU/ppm OD: 0.001 AU/ppm
4	Analyte	Fluoride	Chromium (III) Ion	Sulphate, Chloride
5	LoD	0.025 ppm	0.81 ppb	Sulphate: 0.5 ppm Chloride: 0.4 ppm
	Reference	[19]	[20]	Present work

The designed system is a first ever demonstration of a multi-modal sensing platform that utilises the ALS of the phone to record the incoming signals. Prior to this work, some multi-model sensing platforms have been designed using the rear camera of the phone [19, 20]. Table 5.2 illustrates the performance comparison of the present work with the works reported in the similar line of research in the recent past.

5.9 Summary

In summary, the working of a dual mode smartphone sensing system that estimates sulphate and chloride ions concentration reliably has been described in this chapter. Two sensing schemes namely the photometric and nephelometric based sensing have been realised on a single platform to estimate the aforementioned parameters. Both the sensing modes can be implemented interchangeably through plug-and-play transition. The performance of the tool has been evaluated through comparing the experimental data with the standard UV-VIS spectrophotometer and a nephelometer. The LoDs estimated for sulphate and chloride ions in water are found to be well below the permissible limit recommended by the WHO. The custom designed application developed for the proposed work converts the designed sensor into a truly user-friendly sensing system. It is envisioned that the proposed platform can be used for monitoring of other chemicals (both organic and inorganic) and microbial particles in water and could emerge as an alternative platform in those regions where access to laboratory grade tools is very limited.

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