Chapter 1

INTRODUCTION

This chapter gives an overview of various instrumental methods that are widely used in analytical chemistry and other fields. The chapter includes the journey of bulky scientific instruments towards miniature versions and eventually leads to the development of smartphone-based ultra-compact analytical tools. Applications of smartphone based platforms for water quality assessment have also been discussed thoroughly. The chapter further deals with the thesis problem and possible solutions to them. Finally it concludes with the scope of the research work being carried out at Tezpur University, India and contributions to the scientific community and the society as a whole.

1.1 Analytical methods of material analysis

Analytical methods are used to determine the physical and chemical properties of materials which may consist of a single substance or a mixture of different substances. Numerous analytical techniques are available, that are used to understand the world of material and biological sciences. Presently, the branch of science allocated to study the materials is broadly termed as analytical chemistry that comprises of a wide variety of advanced analytical techniques from simple weighing to highly sophisticated instrument, used for analysis of the natural as well as synthetic substances [1, 2]. These analytical methods can be classified into qualitative or quantitative techniques based on the nature of investigation being performed. In general, the method used to identify an unknown substance is referred as the qualitative method whereas a quantitative technique is related to the evaluation of an analyte concentration present in a sample. These methods are further subdivided into classical and instrumental methods. Classical techniques barely use any instrument. Chemical, flame, gravimetric and volumetric tests are a few examples of classical analytical techniques. These techniques are exceptionally precise and accurate. Although classical methods are highly selective and sensitive, yet the relatively longer analysis time makes them sometimes tiresome and unfavorable. Again, tremendous skill and considerable attention are also required to perform these classical analytical methods [3]. As a result, a more convenient and relatively fast analytical method, wherein specialized instruments are involved, has received more attention as an evolution of post classical methodology [4]. This new analytical method is termed as instrumental method. Instrumental method estimates the physical properties of an analyte to identify or quantify the analyte in a given sample by using a scientific instrument. This method of analysis is usually capable of both qualitative and quantitative study of materials. Colorimetry, photometry, spectrophotometry, microscopy are some noteworthy instrumental analytical methods, details of which are well discussed in the subsequent sections below.

1.1.1 Colorimetric and Photometric methods of material analysis

Colorimetry is one of the fundamental analytical methods where the color of chemically treated samples is visually compared with standard samples or a standard color wheel [5]. The accuracy and precision of this method depend on a person's color perception ability. The results of the colorimetric analysis may vary from person to person. Eventually, the photometric method came into existence and replaces this ocular analytical method [6]. The photometric method uses a photometer which consists of a photo-detector in place of the human eye. A typical photometer consists of a light source, mono-chromator, sample holder, photo-sensitive element and a measuring device. The measuring device measures the responses of the photo-sensitive element upon interacting with the signal coming out of the test sample. This photometric analysis method is somewhat more sensitive and accurate than the colorimetric analysis method.

1.1.2 Spectroscopic methods of material analysis

Spectroscopy is one of the widely used instrumental methods where interactions of electromagnetic radiation (EMR) and a given substance are examined to detect and analyse the constituents of the substance. Over the past few decades various spectroscopic techniques have been developed and subsequently used in various specialized scientific instruments. In analytical chemistry; absorption and fluorescence based spectroscopic techniques are extensively used for sample characterisation [7–9].

Absorption spectrometry

In absorbance spectrometry, the incident radiation absorbed by an analyte has been studied to analyse a sample using a spectrophotometer. A simple spectrophotometer consists of a source, a sample holder and a detector. The guiding principle of such an instrument is based on the measurement of transmitted intensity after it passes through a sample solution. In general, the intensity gradually decreases with the increasing concentration of the analyte. Beer-Lambert law provides an exact theoretical explanation on the working of a spectrophotometer. Again, it can also be used to identify a substance by studying the peak absorbance wavelength from the absorbance spectra as each substance shows a unique EMR absorption pattern [9].

Fluorescence spectrometry

Another important spectrometric technique is the fluorescence spectrometry. Fluorescence is a natural phenomenon where a substance emits radiation on excitation with a suitable energy source. Some substances give rise to the emission of visible light upon interaction with EM radiation, chemical or heat energy [10]. Among these different types of excitation strategy, the most practiced one is found to be the excitation by EM radiation. In this strategy, a light beam of smaller wavelength is incident on a sample and consequently it emits radiation due excitation at a higher wavelength. Fluorescence is an instantaneous effect, where the emission initiates as soon as the excitation takes place and it also vanishes immediately on the withdrawal of the source signal. A fluorometer can be used to measure the fluorescence signal emitted by a fluorophore [11]. A typical fluorometer consists of a light source, mono-chromator, sample holder, optical filter and a photo-detector. The filter stops the incident light and allows only the emitted signal to reach the detector.

1.1.3 Turbidimetric and Nephelometric method of material analysis

It is another important analytical technique that is based on light scattering from a turbid medium. It is a unique analytical technique which represents the correlation between the scattered light intensity and the suspended particles in the medium and it depends on the particle size and concentration, and the incident wavelength [7]. Light scattering based studies can be used for both qualitative and quantitative analysis. Turbidimetry and nephelometry are two important scattering based techniques that provide the measure of suspended solutes present in a medium. The instruments that are used for these two analytical methods are named as a turbidimeter and a nephelometer. A turbidimeter measures the light intensity scattered in a forward direction relative to the source signal. Unlike turbidimeter, a nephelometer records the scattered intensity at an angle normal to direction of the incident signal [8].

1.1.4 Optical microscopic methods of material analysis

Apart from the aforementioned optical-based analytical instrument, optical microscopy is another important instrument that has been widely used for sample analysis in different areas spanning from biology to material research. Optical microscopy is a technique to observe an object in a higher magnification. A convex lens is the simplest optical microscope that produces an enlarged image of the object while looking through it. After that, compound microscopes are developed by arranging multiple optical lenses to obtain well-magnified and well-resolved images of the micro-specimen. A compound microscope can achieve magnification up to $1000\times$ which is restricted by the diffraction barrier [12]. The primary components of a compound microscope are the eyepiece, objective lens, sample stage, illumination source and the condenser lens. Based on the source type, illumination pattern and direction of illumination; different types of imaging schemes like bright-field, dark-field, phase-contrast, point illumination and fluorescence imaging can be realised using a optical microscope [13].

1.2 Applications of analytical instruments

Analytical instruments play a key role in studying both natural and synthetic substances. The various branches of science viz. chemistry, physics, biology, environmental science utilise these instruments for various scientific purposes. Educational institutes, research laboratories, industries use analytical instruments for different teaching, research and developmental works [14, 15]. Analytical instruments find applications in environmental monitoring which includes the assessment of air, water and soil quality [16]. Such assessment is necessary to contain the pollution level in the environment. Instrumental methods are extensively used to detect and measure any unwanted chemicals or specimens in air, water and soil by evaluating their physical, chemical and biological parameters. Apart from environmental application, analytical methods play a major role in the lifesaving medical applications [17]. In the process of design and development of a new drug molecule, proper characterisations of these synthetic substances by scientific instruments are very crucial. In case of clinical diagnostics, Magnetic Resonance Imaging (MRI), Computed Tomography (CT) scans are example of analytical instruments which are commonly used by all the medical institutes. These techniques allow looking into the inner parts of the human body to identify infections, tumour and many more. Health of the inner organs can also be examined using these instrumental methods.

Use of analytical methods can also be seen in food industry, forensic, archeological and cosmological analysis [18–20]. Food industries use instrumental methods to ensure the quality of their food products as well as to detect any possible adulteration in the raw materials. In forensic analysis, instrumental methods help to identify and quantify the substances found in a crime scene. Archeologist use analytical methods to study the historical objects and also to preserve it for the future. In cosmology, analytical instruments like the telescope and spectroscopes have been extensively used to understand the outer space of the earth. From the discussion it can be concluded that instrumental method is the most convenient analytical technique used in different fields to study, characterise or identify any material of concern.

1.3 Miniaturisation of analytical instruments

Instrumental methods involve the use of sophisticated scientific instruments, the cost of which are generally very high and possess a high level of maintenance. These scientific instruments are bulky and most of them are laboratory confined. Furthermore, almost all instrumental methods require an external computational system to analyse and interpret the recorded data. Somehow these tools require uninterrupted voltage supply for proper functioning. The handling of an analytical instrument is again a complicated task and requires techical knowledge of the whole process to produce accurate and precise results. Trained professionals are required to operate these types of instruments [21]. Thus, these tools are not user-friendly to common people and require extra man-power with proper skill. Conventional analytical instruments thus are not a suitable candidate to be deployed in resource-poor regions. Researchers are consistently working on developing simple and easy to use devices by eliminating the drawbacks of an typical analytical instrument [22, 23]. In due course of time, the development of some cutting edge technologies like the invention of dry cell batteries, light-emitting diode (LED), diode LASERs, liquid crystal displays (LCD), MOLED display, complementary metal-oxide-semiconductors (CMOS) and charge-coupled devices (CCD) help to reduce the size of an instrument without losing its analytical abilities. Moreover, new design and fabrication techniques provide a sufficient support to construct numerous compact optical components such as optical fiber, lenses, filters, mirrors, beam splitters and dispersion gratings as per the requirements [24]. Utilisation of these compact optical components; affordability, portability and sustainability of a scientific instrument has been improved significantly. Miniaturisation somewhat enhances the analytical abilities of instrumental method by achieving relatively shorter analysis time and reducing sample volume. The field investigations and remote sensing are becoming feasible with these modern tools. The incorporation of specially designed computational systems makes such a method more user-friendly with added features such as real-time data sharing and large data storage capacity. In true sense, the miniaturisation of analytical tools brings a new era for material characterisation. Recent trends suggest that instrumentation of analytical devices is directing towards the designing of more compact and sensitive platforms such as lab-on-a-chip and lab-on-a-phone devices [25, 26]. Such devices can record, analyse and share data on a single platform. Recently, smartphone-based analysis has attracted the attention of the scientific community in the fabrication of various smart devices [27–31]. In line with the topic, the following sections discuss about the smartphone-based analytical technique and its importance.

1.4 Smartphone-based instrumental method of analysis

Lately, smartphones are being extensively used for development of compact analytical tools. Rapid up-gradation of smartphone technologies, viz. ever improving hardware and computational performance, battery durability, inbuilt sensors and user-friendly applications (App) make it easy for the researchers to develop different smartphone-based sensing and imaging devices [32–38]. The modern day smartphones are equipped with advanced processors such as graphic processing units (GPU), central processing units (CPU) and subsequently enhances its abilities. The larger random access memory (RAM), read-only memory (ROM) and the various embedded sensors have increased the overall performance of this mobile communication devices [39–43]. With these notable features, researchers around the globe have been actively working to developed various smartphone-based sensing and imaging systems and used in different scientific research and developmental purposes. Inbuilt smartphone sensors such as ambient light sensor (ALS), proximity sensor, USB port, Wi-Fi, headphone jack, camera are often used as signal detector to construct such platforms [44–46]. Usually, the embedded sensors are coupled with custom-developed sensing setups to enhance the analytical abilities of these tools. To analyse the signals recorded by the inbuilt sensors, android or ISO-based applications have been developed and used as signal analysing software. Figure 1.1 depicts the growing number of smartphone-based research articles published in various peer reviewed journals in last one decade. Two distinct keywords, '*smartphone sensing*' and '*smartphone imaging*', have been used to search the research articles in the '*web of science*' database [47]. Presented data indicates that smartphone-related researches are continuously growing over the past one decade.

With the drastic development of smartphone technologies, phones based analytical methods are turning out to be the better analytical platforms for material characterisation compared to the conventional scientific instruments. The compactness of these tools makes them truly field-portable and hence can be utilised in field applications at remote locations. The field portability feature eliminates the difficulties related to the collection, transformation and preservation of samples wherein on site investigation can be carried out using the compact smartphone-based tools. These tools are also ideal for resource-poor regions as they do not require any external electricity, computational devices and dedicated laboratory setup [48, 49]. The cost involvements in smartphone-based analysis, such as fabrication, mainte-

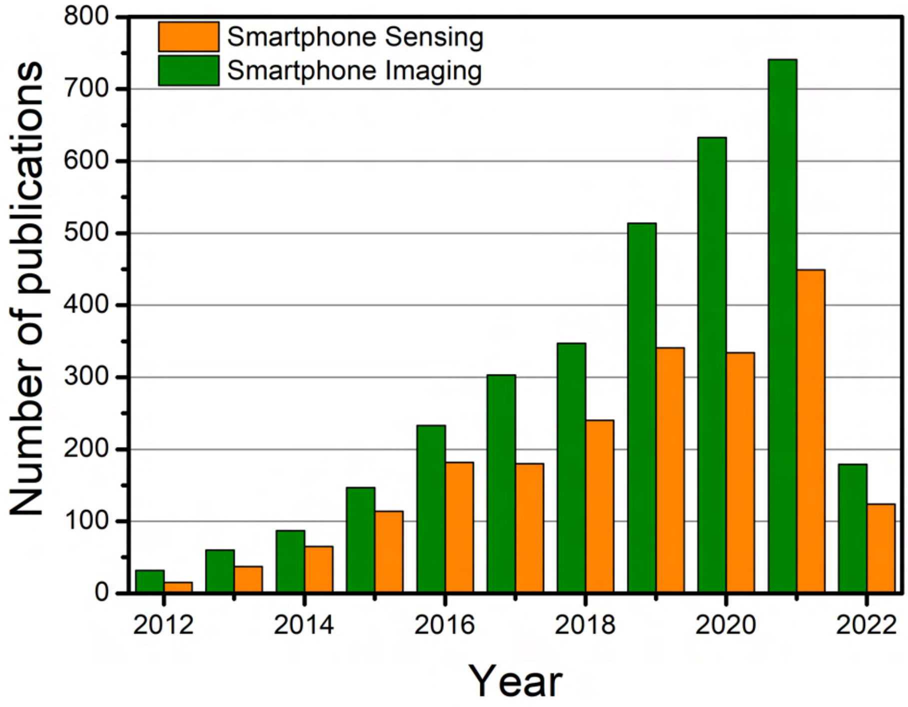

Figure 1.1: Schematic representation of increasing number of research articles in the field of designing the smartphone-based sensing and imaging platforms. Data are taken from the '*Web of Science*' database on 10th May 2022 [47].

nance and sampling cost are found to be remarkably low. However, the size and cost of smartphone-based platforms are reduced significantly, nevertheless the performances of these tools are still comparable to the conventional ones [50, 51]. It is also observed that the standard operating protocols (SOP) of smartphone-based analysis are quite simple and yield an amicable user experience with the operators.

Recently, the working of smartphone spectrophotometers, fluorometer, turbidity readers, microscopes, colorimeters and pH meters have been demonstrated by various groups [52–56]. Figure 1.2 shows some smartphone analytical platforms. The performance of these instruments has been assessed by promoting them in different sensing and imaging applications which include chemical sensing, bio-sensing, heavy metal detection, clinical and various diagnostic purposes. Erickson et al., 2014 proposed a system that can measure and monitor cholesterol levels on a smartphone [57]. They have measured cholesterol levels with 1.8% accuracy by analysing the

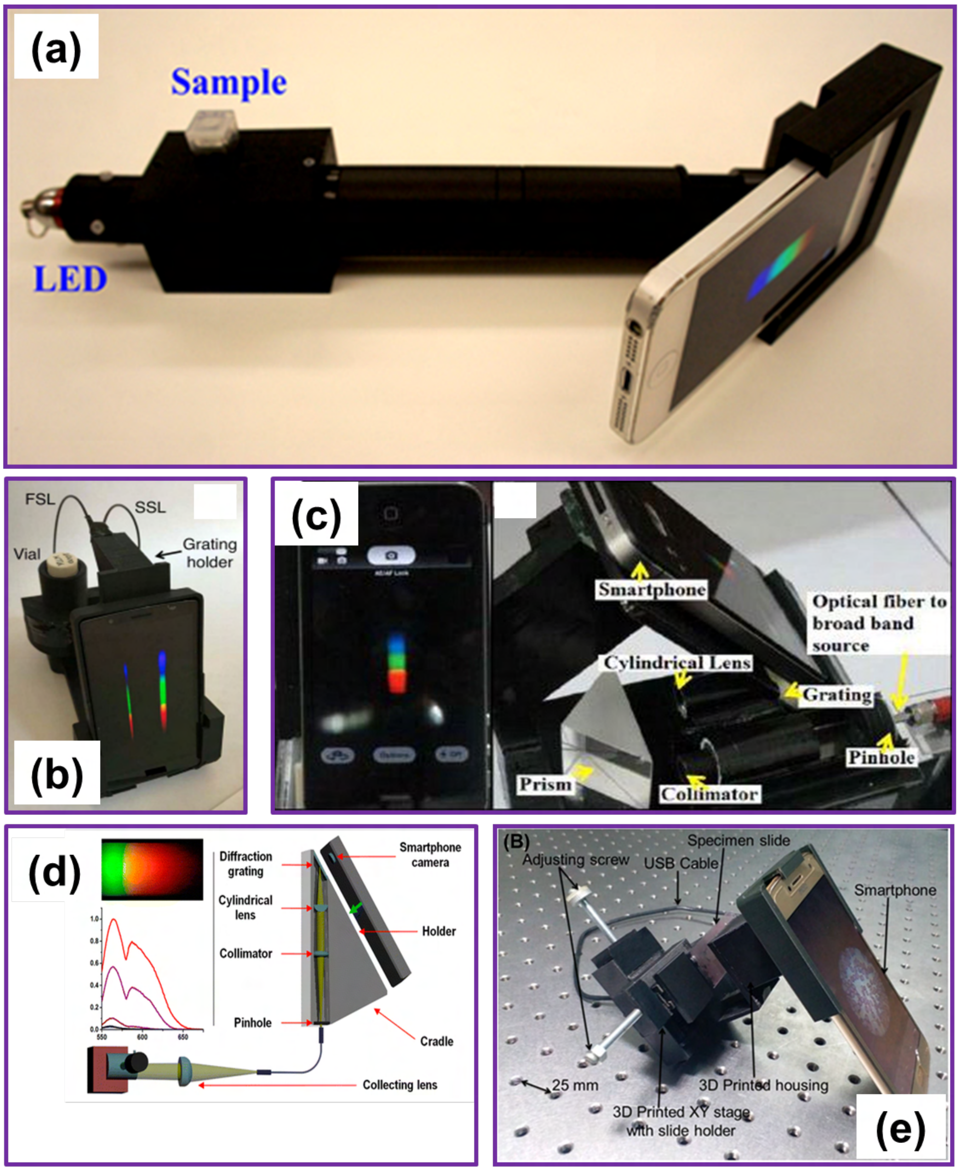

Figure 1.2: The development of different smartphone-based analytical platforms; (a) a smartphone based opto-sensing platform, figure reproduced from [52] with permission from *American Chemical Society*, (b) a smartphone turbidimeter, figure reproduced from [53] with permission from *Optica Publishing Group*, (c) an evanescent wave coupled smartphone spectrometer, figure reproduced from [54] with permission from *IEEE Publishing Group*, (d) a smartphone based fluorescence spectrometer, figure reproduced from [55] with permission form *American Chemical Society* and (e) a smartphone-based microscope, figure reproduced from [56] with permission from *Royal Microscopical Society*.

sample images acquired by the device. Dutta et al., 2015 have demonstrated the working of a smartphone-based colorimetric sensor to estimate the pH level in different water resources [58]. The spectral resolution of the proposed tool has been evaluated to be 0.3 nm per pixel. The system is sensitive within the pH range of 6 to 8. Yoon et al., 2014 have demonstrated a smartphone-based platform to detect *E. coli* from the field-collected water samples [59]. The system was based on paper-based microfluidics and the limit they have achieved was found to be 1 CFU/100ml. Han et al., 2016 built a soil classification sensor using a smartphone [60]. Guner et al., 2017 have presented the usability of a smartphone by designing a hand-held Surface Plasmon Resonance (SPR) based biosensor [61]. Bayram et al., 2018 have developed a smartphone-based turbidimeter to estimate the turbidity of natural water samples [53]. The system can estimate both forward and side scattered light intensities on a single platform and analyse both the signals to estimate the turbidity of the test solutions. The limit of detection of the developed setup was 6 NTU.

It is further observed that the smartphone-based sensing systems have found increasing applications in variuos fields that include environmental monitoring, point-of-care diagnostics, bio-sensing, food quality assessment etc. [62–65]. The smartphone-based sensing has evolved the material characterisation and quantification process to an excellent height. The applicability of smartphone-based analytical method is very broad; however, present thesis work emphasized on the design of truly field-portable and user-friendly analytical platforms that can be used for water quality assessment.

1.5 Importance of water quality assessment

Water quality assessment is directly related to the detection of water contaminants that may present in drinking water or natural water bodies. Pollution in water is generally caused by various artificial acts, although some natural phenomena are also involved in this. Poor disposal of industrial and domestic waste is one of the major man-made concerns of water pollution. These would carry a lot of unwanted chemical and pathological microorganisms to the natural water resources. Again, commercial extractions of natural resources such as fossil fuels, coal and minerals negatively impact the water bodies [66]. One of the prominent natural phenomenon related to water pollution is the natural breakdown of rocks. While disintegrating, the composite minerals from the rock are released into the natural water bodies and eventually make them contaminated [67]. The contamination level in water is estimated based on its different quality parameters. Depending on the various water pollutants, the water quality parameters can be classified into four major categories: physical, chemical, heavy metal and biological pollutants. Each pollutant has its unique toxicity profile for human beings [68]. Pollutants residing in the heavy metal category are considered the most acute water pollutants. However, the excess amounts of the other pollutants in water are also harmful to human beings. Hence, various international and national agencies have set threshold concentrations of each pollutant to identify and eliminate waterborne threats [68, 69]. Several diseases such as cholera, dengue, diarrhea, Japanese encephalitis, fluorosis, hepatitis, lead poisoning, malaria, typhoid are originated from poor management of water resources and increasing contamination level in it [70].

It is a global concern to provide clean and safe water to each individual on earth for daily their consumption and other usage. In this aspect, comprehensive efforts have been made by various non-profit organizations and governmental bodies worldwide. However, statistical data shows that a large portion of the world population still lives without proper water connectivity [71]. Organizations such as the WHO and the United States–Environmental Protection Agency (US-EPA) have implemented various policies to overcome the problems stated above [68, 69]. The US-EPA's drinking water regulation is comprised of a number of standard rules. These regulations set guidelines for the tools and processes to be used for assessing the water quality. These can be used by different regional water quality monitoring bodies on a local level for water quality assessment. Some examples of standard operating procedures (SoP) published by the US-EPA include the chemical contaminants rule, the arsenic rule, the ground water rule, and the total coliform rule [72–74]. More than 90 distinct forms of water contaminants are being regulated by these rules. These strict guidelines are primarily aimed at safeguarding the general public's health by preventing from various water-borne diseases. Research suggests that developing and underdeveloped countries are more vulnerable to water-related problems. According to the report published by WHO in 2021, at least 2 billion people worldwide depend on natural water resources such as tube well, rivers and ponds for their daily usage [75]. As a developing country; India is still facing various water-related issues [76]. Therefore, India needs to implement strict water safety policies and a coordinated effort from both the governmental and the non-governmental organizations to eradicate water borne diseases. In this regard, a radical change in terms of technique and technology can be considered an important solution to water-related complications. Conventional water laboratories are not suitable for regular monitoring of water qualities. To overcome that, compact and accurate sensing platforms are extremely desirable. Over the past one decade, smartphone-based sensing is evolving as a decent yet a promising alternative in the field of water quality assessment [77–79]. A detailed literature review on smartphone-based platforms employed in water quality assessment has been discussed below for a clear understanding of the topic.

1.6 Water quality assessment on smartphone-based platforms

Smartphone-based platforms are extensively used to examine different water quality parameters. Many research articles on water quality assessment have been published in various scientific journals. Herein an exclusive literature review on optical sensing of water quality assessment has been discussed. Other sensing methods such as electrochemical, impedance-based measurement are beyond the scope of this thesis [80, 81]. The literature review has been split into four distinct subsections based on the four primary quality classifications of water.

1.6.1 Heavy metal detection on smartphone-based platform

The presence of heavy metals like arsenic, mercury, lead, zinc, cadmium in natural water bodies has always been an alarming concern. Level of heavy metal contamination is not the same and varies differently for different type of water resources. It is seen that the rate of metal contamination in groundwater resources is higher compared to the other resources. However, the presence of heavy metals in any water sources is considered as toxic and can cause severe threat to the living organisms. A small quantity of heavy metal in drinking water can cause severe damage to the nervous system and the kidneys of human beings as they gradually accumulate within the body. Long term exposure to heavy metals can be carcinogenic[82]. A comprehensive literature review on heavy metal detection in water samples using smartphone-based platforms has been discussed here.

As shown in Figure 1.3 (a), Wei et al., 2014 have developed a smartphone platform to quantify the concentration of mercury (II) ions in water samples [83]. De-

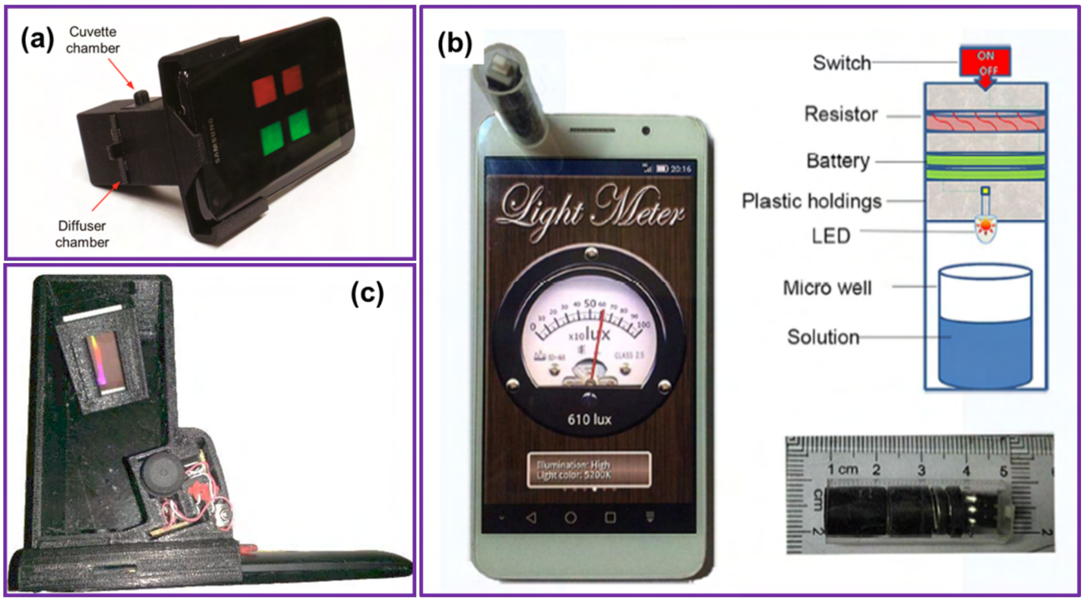

Figure 1.3: The development of smartphone-based analytical platforms for heavy metal detection in water; (a) a smartphone based opto-mechanical platform to detect mercury ions, figure reproduced from [83] with permission from *American Chemical Society*, (b) a smartphone readout device for chromium ion estimation, figure reproduced from [84] with permission from *Royal Society of Chemistry*, and (c) a dual-mode smartphone spectrometer used in zinc ions estimation, figure reproduced from [85] with permission from *SPIE Publishing Group*.

signing of the platform includes fabrication of a battery-powered opto-mechanical attachment that contains a two-wavelength illumination source and an assembly of two cuvette sample holders. Two light sources with peak emission wavelengths at 523 nm and 625 nm were used to illuminate the samples. The platform has been designed to record the plasmonic resonance wavelength shift of a colorimetric assay that was prepared by mixing gold nanoparticles (AuNPs) and mercury (II) ions. An Android application has been developed to evaluate the green and red channel intensities of the captured images and correlate it for mercury (II) ion estimation. Yu et al., 2016 have developed a smartphone readout device to detect chromium (III) ions in water samples with the help of an inbuilt optical sensor of a smartphone shown in Figure 1.3 (b) [84]. This portable readout device is based on Enzyme-Linked Immune-sorbent Assay (ELISA). A freely available Android application has been used to record and analyse the transmitted light signal in the demonstration. A 450 nm peak emission LED has been used as a light source powered by an external 3 V battery. Hossain et al., 2015 have demonstrated the estimation of zinc (II) ion detection on a dual-mode smartphone spectrometer shown in Figure 1.3 (c) [85]. The platform has been designed to record both the absorption and fluorescence spectra of a test sample within the same optical setup. For absorbance and fluorescence measurement, a white and a ultra-violet (370 nm) source have been utilised respectively. The group further used a nanoimprinted gold-coated diffraction grating to split the colors of the light signal, which was eventually captured by the camera of the smartphone. The dispersive image has been processed with the help of an Android application to obtain the required absorbance and fluorescence spectrum. The platform's performance has been tested by measuring the zinc (II) ion concentration in water samples using a sensitive UV-absorbing and blue-emitting fluoro-ionophore. Detection of heavy metals using smartphone by other groups can also be found elsewhere [86, 87].

1.6.2 Minerals and Chemicals detection on smartphone-based platform

The most commonly encountered chemical species in water samples are the essential minerals. These minerals and along with some dissolved ions are collectively considered as the chemical parameters of water. These dissolved solutes are further categorized as organic and inorganic chemicals. Inorganic chemicals in water include calcium, magnesium, iron, fluoride, chloride, sulphate etc. Oxygen, carbon and nitrite are examples of organic chemicals present in water. Excessive abundance of these chemicals in water can cause severe heath issues. For example too much iron in water can cause stomach problems and nausea. Similarly the excess content of fluoride in water can cause severe damage to the teeth and bones [92, 93]. Recently, different compact smartphone-based platforms have been developed to assess these chemical contaminations in water.

Peng et al., 2019 have demonstrated a smartphone colorimeter to determine the trace concentration of calcium shown in Figure 1.4 (a) [88]. The working of the designed platform is based on the dispersive liquid-liquid micro-extraction (DLLME) principle. The designed platform evaluated the calcium ion concentration by extracting the blue channel intensity of the captured images using a smartphone application. Sumriddetchkajorn et al., 2013 & 2014 have designed two mobile platforms

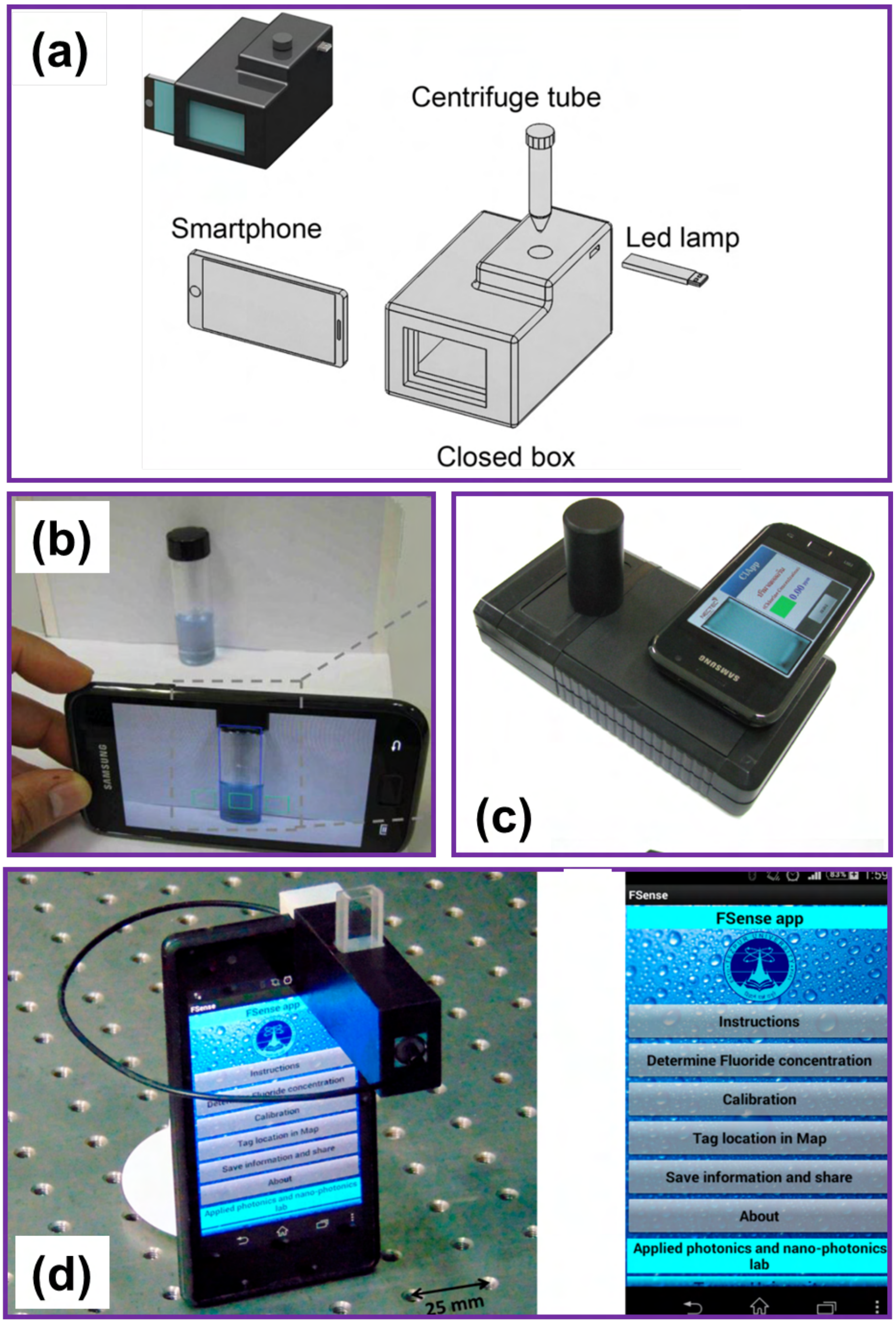

Figure 1.4: The development of smartphone-based analytical platforms for estimation of chemicals and minerals in water; (a) a smartphone based colorimeter to detect calcium ions, figure reproduced from [88] with permission from *Elsevier publishing group*. (b) a smartphone self-referencing colorimeter for chlorine estimation, figure reproduced from [89] with permission from *Elsevier publishing group*, (c) a smartphone colorimeter to detect chlorine in water, figure reproduced from [90] with permission from *IEEE Publishing Group* and (d) a smartphone photometric fluoride level monitor, figure reproduced from [91] with permission from *American Chemical Society*.

based on the colorimetric analytical assay to monitor chlorine in the water. The first mobile platform was based on the self-referencing principle, where the colour of the analyte sample was compared with a standard colour in the analytical procedure. A custom-designed optical chamber with white background has been used to capture the images of the analyte samples by the camera on the phone. The RGB value extracted from the captured images has been used to determine the chlorine concentration in water shown in Figure 1.4 (b) [89]. The group has improved the previous platform by constructing a more reliable optical chamber and improving the image processing algorithm. The redesigned platform showed improvised performance while measuring residual chlorine in water shown in Figure 1.4 (c) [90]. In 2016, Hussain et al. designed a fluoride sensor utilising the ALS of the phone as a photo-detector shown in Figure 1.4 (d) [91]. The designed tool is based on photometric sensing of fluoride ions in water samples that estimate the test samples' absorbance using Beer Lambert's principle. The designed tool records the intensity of the transmitted light signal after passing through the sample and correlates its value to the concentration of fluoride ions present there. A custom-developed smartphone application has been utilised to analyse the recorded data. A number of research articles are found in this context where different chemical parameters of water have been monitored successfully on smartphone-based analytical platforms [94–96].

1.6.3 Estimation of physical parameters on smartphone-based platform

Smartphone-based analytical techniques are also being implemented to estimate different physical properties of water. Physical property includes the colour, taste, odor and turbidity of a water sample. Toivanen et al., 2013 designed a smartphone-based tool to measure the transparency of water samples using the Secchi disk method [100]. It consists of a container with two measuring tags encrypted in it. The phone camera captures the image of the water sample filled inside the container from above. The captured images were then sent to the central processing unit to estimate the transparency using pattern recognition method. Hussain et al., 2016 have designed a turbidimeter to record the scattered intensity by a suspension shown in Figure 1.5 (a) [97]. The infrared sensor of the phone has been used to design the

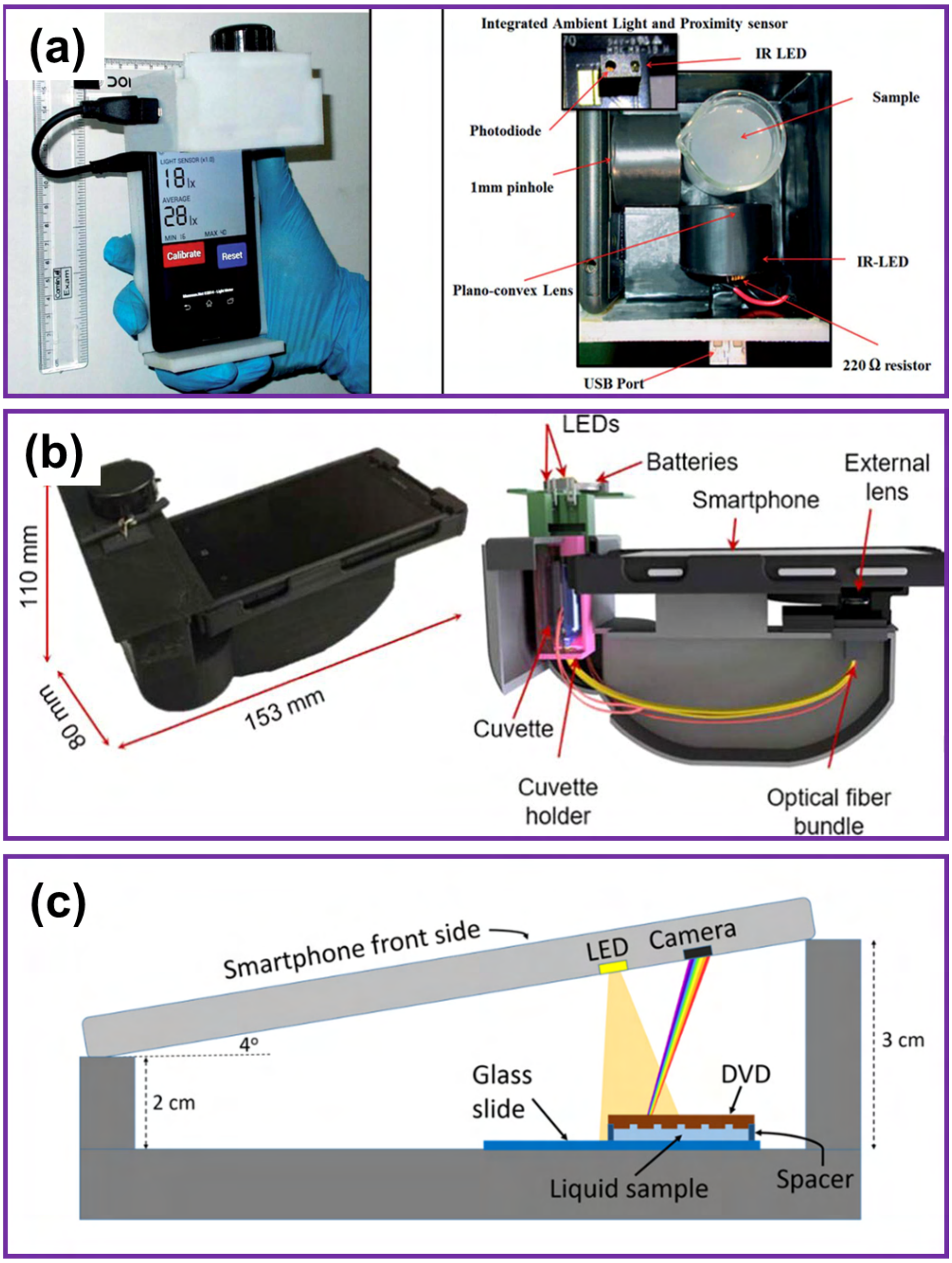

Figure 1.5: The development of smartphone-based analytical platforms for estimation of physical properties of water; (a) a smartphone-based turbidimeter to measure turbidity of water, figure reproduced from [97] with permission from *Royal Society of Chemistry*, (b) a smartphone turbidity reader, figure reproduced from [98] with permission from *Nature Publishing Group*, and (c) schematic of a smartphone based refractive index reader, figure reproduced from [99] with permission from *MDPI Publishing Group*.

turbidimeter. An LED with a peak emission wavelength of 870 nm was used as an incident source. The tool showed linear responses within the turbidity range from 0 NTU to 400 NTU. Koydemir et al., 2019 designed an optical fiber-based turbidimeter to simultaneously measure the forward and side scattered light intensities shown in Figure 1.5 (b) [98]. The dynamic range of the turbidimeter was estimated in the range from 0.3 NTU to 2000 NTU. Barrios et al., 2021 have developed a smartphone-based opto-sensing platform to determine the refractive index of water sample using a low-cost DVD grating shown in Figure 1.5 (c) [99].

1.6.4 Detection of biological contaminants on smartphone-based platform

Pathogenic bacteria, fungi and algae in water are classified as biological pollutants. The detection of *Escherichia coli* (*E. coli*) and total *coliform* are used to measure the bio-contamination in water. Park et al., 2015 demonstrated a smartphone assist platform for *E. coli* detection in water samples shown in Figure 1.6 (a) [59]. A paper substrate with three microfluidic channels was fabricated and used to collect the light intensity scattered by the bacteria present in the substrate. Patil et al., 2019 designed a compact incubator to detect the presence of *coliform* in the sample shown in Figure 1.6 (b) [101]. The incubator consists of a heating element controlled by a microcontroller and powered by a 9 V external battery. Inside the incubator, the test sample was inserted using a cylindrical vial. The designed incubator was attached to a smartphone to capture the image of a black and white pattern encrypted inside the vial's bottom lid. As the bacteria grew inside the incubator, the sample's turbidity increased and gradually blurred out the black and white pattern. Gunda et al., 2014 have demonstrated a mobile kit for simultaneous detection of *E. coli* and *coliform* by using a smartphone application shown in Figure 1.6 (c) [102].

1.7 Statement of the research problem and research motivation

There is no doubt that instrumental methods are reliable analytical technique in studying the underlying properties of all kinds of material. However, the major concerns associated with the instrumental method are the involvement of bigger and costly scientific instruments and complex analytical procedures. Recent progresses in developing novel instrumental methods are to some extent successful in eliminat-

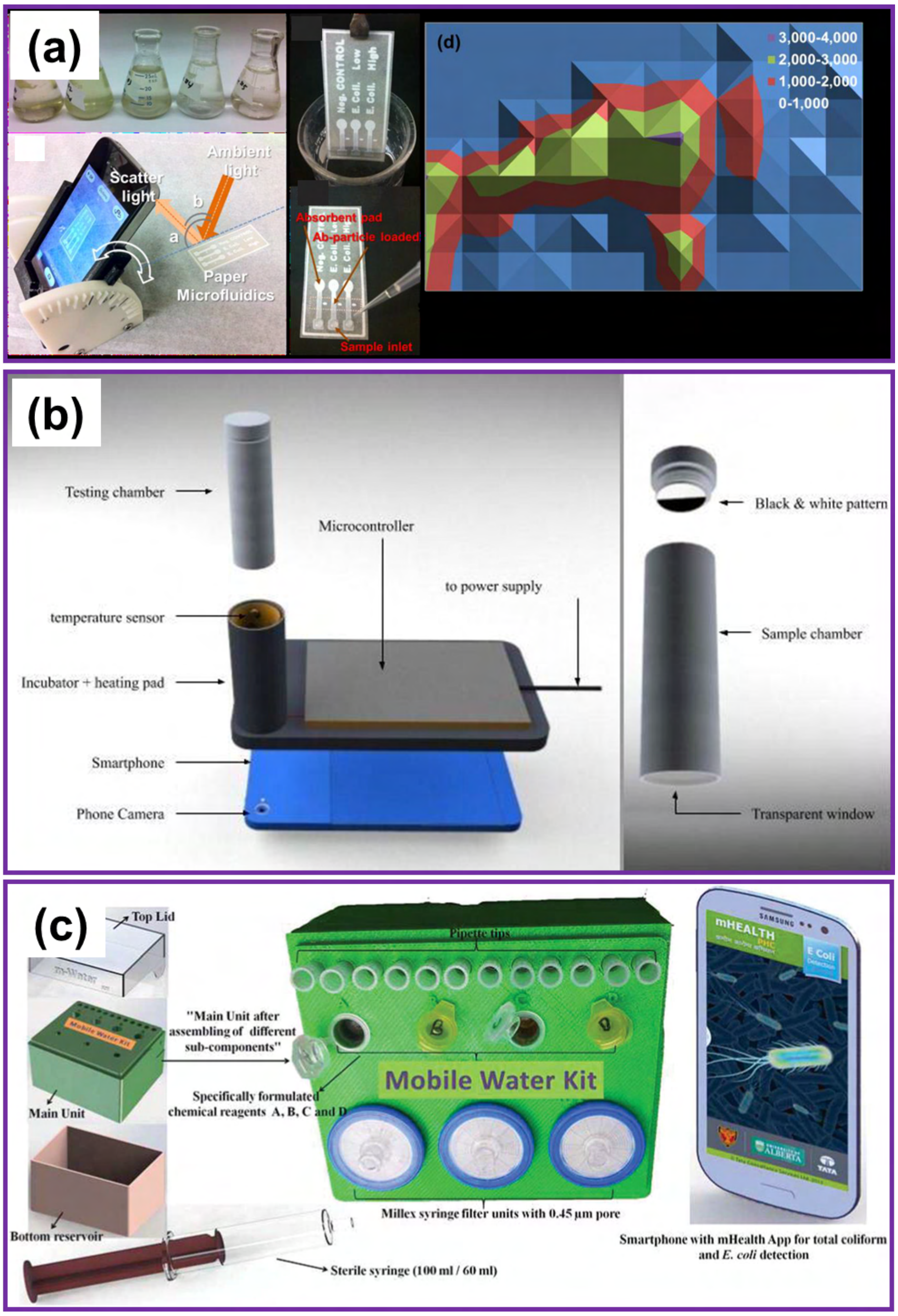

Figure 1.6: The development of smartphone-based analytical platforms for detection of bio-contamination in water; (a) a smartphone based bacteria detector, figure reproduced from [59] with permission from *IEEE Publishing group*, (b) a smartphone assisted bacterial incubator, figure reproduced from [101] with permission from *Faculty of Biotechnology and Food Sciences*, and (c) a dual-mode smartphone tool for detection of total *coliform* and *E. coli*, figure reproduced from [102] with permission from *Royal Society of Chemistry*.

ing these concerns. Continuous researches on reducing the physical dimension and the cost of an analytical instrument have unlocked immense scope for the researchers across the globe. Miniaturisation of scientific instruments unleashes the instrumental method from sophisticated laboratories into the field applications. With such alteration, analytical tools are becoming easily accessible for investigating in remote and resource-poor regions. No specific laboratory environment is needed to conduct the in-field and real-time investigations. Such achievement can be very helpful for countries with limited resources like India to combat various environmental and agricultural-related issues. Recent development in smartphone-based sensing and imaging techniques also contributes to these great causes. Understanding the state of the problem, present thesis work has been inclined towards the development of compact and cost-effective smartphone-based analytical platforms that can be deployed for water quality assessment and extending the same to various other applications.

1.8 Scope and Contribution of the Thesis

The most important objective frequently found in analytical chemistry involve the advancement of the analytical techniques and its application to relevant environmental, medical, food problems. In line to this, the present research works are directed towards developing advanced smartphone-based analytical platforms for estimating heavy metals, chemicals and biological contaminants in water samples. The designed platforms are based on optical sensing of analyte concentration in water. Colorimetry, fluorescence, nephelometry, microscopy and photometry based analytical approaches have been adopted to develop various sensing and imaging platforms. The inbuilt optical sensors of the smartphone, namely, the ALS and the rear camera have been utilised to devise some original analytical platforms. Custom designed Android applications for each system have been developed to analyse the recorded data. During the research work, estimations of mercury, zinc, sulphate and chloride ions in water have been carried out successfully. The thesis work further demonstrates the investigations on different biological specimens wherein growth kinetics of bacterial samples and imaging of blood cells have also been carried out as a part of the research work. The designed smartphone-based analytical platforms are compact, cost-effective and easy to use. The thesis's contributions to smartphone-based sensing and imaging for water qualities and micro-biological assessments are summarized below.

1. At first, a fluorescence-based sensing system has been developed to estimate mercury ion concentration in field-collected water samples. The sensing principle of the system is based on the measurement of quenching in fluorescence intensities emitted by R6G solutions in the presence of mercury ions. Here, the fluorescence signal has been captured by the rear camera of the phone and further analysed on a custom-developed Android application. The application extracts the V channel values of the images and correlates them with the mercury concentrations. The designed platform has a detection limit of 32 ppb. While comparing with Atomic Absorption spectrometer (AAS) data, a good degree of correlation between the sensor responses has been observed.

2. In the second work, a smartphone nephelometer has been designed to monitor bacterial growth kinetics in the laboratory environment. The designed platform uses the ambient light sensor (ALS) to record the light scattered by the bacterial sample followed by the estimation of the samples' turbidity. It records turbidity of the bacterial sample at different time points while growing in an incubator for 24 hrs. Finally, the designed system evaluates the growth curves of *Escherichia coli (E. coli)* and *Bacillus subtilis* (*B. Subtilis*) bacterial samples. Subsequently, the system's performance has been evaluated with two gold-standard techniques such as OD600 and colony-forming (CFU) measurement in the present study.

3. A dual-mode sensing platform has been developed for multi-analyte sensing studies in the next step. The sensing system can measure a given sample's absorbance as well as its turbidity. ALS of the phone has been used in the present study as an optical detector. Two sensing schemes, photometry and turbidimetry, can be carried out in the designed platform through plug-and-play mode. As proof-of-concept, sulphate and chloride concentrations of field-collected water samples have been examined using the designed sensor. The system can estimate the sulphate and chloride concentrations as low as 0.5 ppm and 0.4 ppm. A custom-designed application has also been developed to record and analyse the incoming light signal and estimate the analyte concentrations.

4. In the fourth work, the development of a smartphone-based analytical platform using two different embedded optical sensors has been executed. The system can measure the absorbance and fluorescence emission of a fluorophore simultaneously. In the designed system, the ALS of the phone records the transmitted light signal to measure the absorbance of the test sample. Fur-

ther, the phone's rear camera has been utilised to measure the fluorescence emission. Finally, the platform has been used to estimate zinc concentration in water. The limit of detection of the system is found to be 0.1 ppm.

5. Finally, the working of a universal phone holder has been demonstrated by performing multiple analytical studies. The holder has been designed to couple with all variants of smartphones. Three different company-make smartphones, namely Samsung, Xiaomi and Motorola, have been selected for the present study to realise the universality of the phone holder. All the three phones have unique physical dimensions along with differently positioned rear camera. Multiple analyses such as colorimetric, fluorescence and microscopic studies have been performed by utilising the proposed holder. The present study involves the estimation of transmitted and emission intensities of R6G solutions. Again, high-quality images of micro-particles such as blood cells are also captured by a custom-developed microscopic setup in the final work. The platform has been developed as a universal analytical platform where a smartphone can be used to conduct different analytical investigations.

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