Chapter 10
Paper Based Sensors for Environmental Monitoring

Pankaj Singh Chauhan, Mohit Pandey and Shantanu Bhattacharya

Abstract Environmental pollution has become the major concern for the environmentalists in the recent times. Continuously increasing population and development of human race demands abundant amount of resources such as electricity and water for domestic as well as industrial work. At the same time, the by-products and remains of industrial processes are being discharged to the environment in form of harmful gases and effluents. Hence, the need of the hour is to monitor and control these contaminants, especially in air and water. The sensors required for this type of monitoring should have stability, cost effectiveness, disposability, and flexibility. Paper based sensing devices have become a promising mean to detect the pollutants and monitor the air and water quality. Paper based sensing devices possess some unique properties such as liquid transport through capillary action, ease in mass production, flexibility of shape/size, and disposability. Paper based sensors also offer high sensitivity, cost effective process, fast response, and flexibility as per the end user requirement. Due to above mentioned characteristics, the paper based sensors have become promising devices for the present environmental monitoring scenario. This chapter explains the fabrication methods, mechanisms, and applications of the paper based sensors for environmental monitoring.

Keywords Sensors · Pollutants · Microfluidics · Adsorption · Chemiresistive

10.1 Introduction

The global development in industrial and transport sector has posed a catastrophic threat to the environment. Rising population demands facilities like clothes, transport vehicles, healthcare, education, and safety in abundance. These demands

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S. Bhattacharya et al. (eds.), *Paper Microfluidics*, Advanced Functional Materials and Sensors, https://doi.org/10.1007/978-981-15-0489-1_10

have revolutionized the respective industries with majorly focusing on fulfilling the demands. These industries discharge a huge quantity of solid, gaseous, and water wastage per day.

These waste products are sometimes discharged directly to the environment such as dumping grounds, open environment, rivers, and ponds (Rai et al. 2018), etc. The phenomenon of 'Global warming' has attracted a lot of attention of world leaders in recent years which relates directly to the air and water pollution. Many countries have now started to consider pollution as a major threat to mankind and are taking various critical steps to reduce it. To maintain the air quality various gas sensors are used to identify and quantify the different harmful gases. These harmful gases are: CO, CO_2, H_2, NO, NO_2, SO_2, H_2S, CH_4, C_2H_2, and O_3, etc. (Chauhan and Bhattacharya 2017; Chauhan et al. 2019a; Kumar et al. 2019). Similarly, various pollutants present in water are identified by using various water sensors. Heavy metals such as: As, Pb, Fe, Cu, Cr, and Al etc. mixes with water during various industrial processes such as steel making, heat treatment, textile colouring, and tanning (Chauhan et al. 2017). At the same time harmful dyes and other chemical compounds are also mixed with water during these operations. These compounds present in the water are the root of various water borne diseases such as cholera, diarrhoea, abdominal pain, headache, and fever, etc. (Chauhan et al. 2019b). The polluted water discharged into rivers and ponds is also harmful for the animals and humans which use this water.

Hence, it becomes a necessity to monitor and control these environmental pollutants. The monitoring system of air and water pollutants demands certain characteristics such as fast detection, accuracy, cost effectiveness, simplicity, and ease of fabrication. At the same time the detection system should be able to detect the presence of contaminants as quickly as possible to take appropriate actions. The traditional methods for the contaminant detection such as: inductively coupled plasma spectroscopy (ICPMS), gas/liquid chromatography, and mass spectroscopy are very accurate and effective but require complex processes to achieve the result (Liana et al. 2012). These methods have the drawbacks of exhaustive sample preparation requirement and complex quantitative analysis. Also, these methods require very expensive machinery, costly reagents, and expert operator to conduct the tests. These drawbacks make conventional detection methods less useful for online/onsite monitoring of the environmental pollutants.

Recent advances in paper based sensing technology have revolutionized the area of environmental monitoring. Now paper is not just a writing, packaging, drawing, and printing material but also serves the purpose of flexible substrate for the sensing material. Paper possesses certain properties such as: liquid flow through capillary action, flexibility, low cost, availability, and lightweight which makes it to be extremely useful for certain type of sensing applications. The cellulose fibre is the main constituent of paper and has hydrophilic nature which allows the liquids to flow through capillary action. Apart from that the properties of the paper can be improved as per requirement by chemical functionalization and treatments. The paper based sensors are widely used for clinical and analytical chemistry due to their portability flexibility, and disposability (Park et al. 2018).

The first paper based sensor was developed in 1956, to monitor the glucose level in urine (Comer 1956). The later developments resulted into the development of paper based 'dipsticks' and commonly used pregnancy test kit (Von Lode 2005). These type of detection kits are based on immunoassay detection method which commonly consists a sample pad, a reagent pad, and a test line. The sample pad receives the sample to be tested, the reagent pad contains antibodies conjugated to target antigen specific signal indicator, and the test line captures the antibodies which are immobilized on the surface. The signal given in these type of devices is generally a color which is only a qualitative analysis (Yes/No type) of the sample. The example of commonly used paper based detection in chemistry is pH level measuring strips also known as 'Litmus paper'. The color of the litmus paper when dipped in a solution indicates its pH level (acidic or basic) as shown in Fig. 10.1. With further advancements in the paper based sensing technology, the quantitative analysis has also been possible up to some extent. The intensity of the color of the indicator corresponds to the concentration level of a particular gas or analyte (Huang et al. 2014). In the current paper based detection scenario, there are several sensing techniques such as: colorimetry (Li et al. 2018), electrochemistry (Sicard et al. 2015), chemiluminescence (Wang et al. 2018a), electrochemiluminescence (Delaney et al. 2011), and electrical (Mirica et al. 2013) are being used. The application of these techniques is based on the requirement of the particular system such as environmental conditions, type of analyte, and the accuracy level required. These techniques have their advantages and disadvantages which are to be considered before selecting it in a sensing system.

Hence, the paper based detection systems have emerged as an effective sensing lab-on-chip devices due to their fast analysis, portability, disposability, and onsite real time detection for the medical, environmental and industrial applications. Low cost of paper based devices can be easily mass produced and widely used specially in healthcare where a fast analysis is required. This chapter reviews the development/fabrication methods, analysis techniques, application areas, and challenges of the paper based environmental sensing devices.

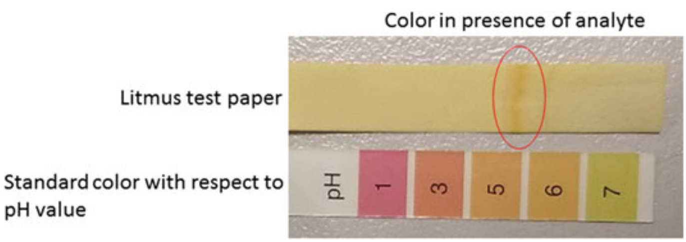

Fig. 10.1 Litmus test paper for the detection of pH level of analyte

10.2 Development of Paper Based Sensor

The choice of paper in paper based sensors becomes essential because it governs the sensing performance of the device. It adheres the sensing material onto its surface which further interacts with the entity to be sensed. Sometimes, the choice of paper depends upon the fabrication steps involved and applications with specific requirements. In recent times, filter paper has become the most favorite choice for the paper based sensing devices (Li et al. 2010). The filter papers are available with different grades which is basically describes the porosity, flow rate, and retention property of the paper (Liana et al. 2012). Few studies suggested the modification of the paper properties by adding the hydrophobic/hydrophilic films to optimize the fluid flow property. The filter paper is made of cellulose fibers which due to capillary action gets swelled by absorption of liquid. Hence the grade number should be clearly chosen so that the porosity can be maintained throughout the detection process.

For detection of biomolecules such as proteins, enzymes, and DNA nitrocellulose membrane is used due to its specific binding properties (Scida et al. 2013). The coating of wax on the paper substrate is used to create hydrophobicity and microchannels. Sometimes, apart from the conventional paper the glossy paper is also used. The glossy paper is fabricated by using inorganic filler material with cellulose fiber. Glossy paper is non-degradable and has smooth surface which is useful in surface based applications.

After selecting the most suitable paper the fabrication and pattering of the paper based sensing device is carried out. In some of the detection methods there is a requirement of fabrication of microchannels, electrodes, or specific layers of some other materials. There are several techniques and methods available for the fabrication of these features by using chemical or physical means. Some of the methods reported earlier by the researchers are photolithography (Shaker et al. 2011), screen printing (Sarfraz et al. 2013), plasma treatment (Li et al. 2010), wax treatment (Sicard et al. 2015), and inkjet printing (Lee et al. 2011) etc. The selection of these methods depends upon the quality of paper, application, cost, and other requirements of the end user. Gosselin et al. proposed a low cost fabrication protocol for a microfluidic device on an embossed paper (Gosselin et al. 2017). The embossing of the paper are used to create hollow microchannels for the fluid flow in colorimetric glucose detection. In this study two layers were coated on the paper substrate. The first layer of Styrene Butadiene Rubber (SBR) provides the waterproofing so that the analyte liquid could not penetrate the paper matrix. While, the second layer of Poly (Vinyl Alcohol)/copolymer Styrene Acrylique (SA) provides the hydrophilicity for allowing the fluid flow on the surface of the paper. The microfluidic channel was created on this coated paper by embossing method. In brief, the paper was pressed against a master (with negative carvings) of the desired shape inside a heated chamber as shown in Fig. 10.2. This device successfully demonstrated the flow of glucose assay through the microchannels (shown in Fig. 10.3).

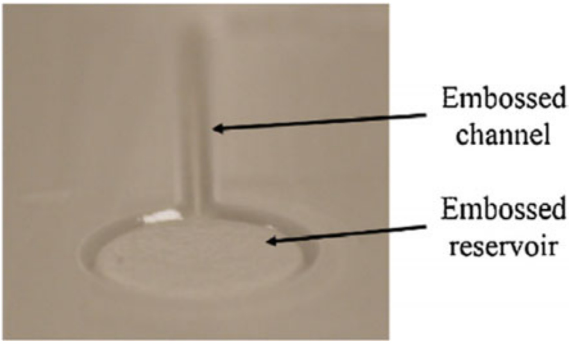

Fig. 10.2 The image of microchannel and reservoir on the embossed paper. Reprinted with permission from Gosselin et al. (2017). Copyright (2017) Elsevier, Sensors and Actuators B: Chemical

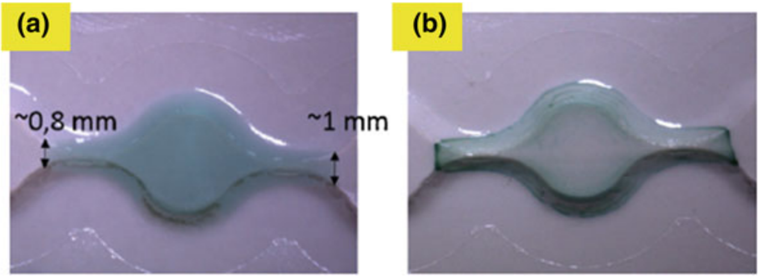

Fig. 10.3 The central well of the microchannels fabricated on an embossed paper with fluid before (**a**) and after evaporation (**b**). Reprinted with permission from Gosselin et al. (2017). Copyright (2017) Elsevier, Sensors and Actuators B: Chemical

Such types of system can be easily used in environmental monitoring such as heavy metals detection from the wastewater etc.

10.3 Detection Techniques

The paper based sensors have different detection techniques for identifying the analyte. These detection techniques are explained in the following section.

10.3.1 Calorimetry Detection

This test is based on the ability to give a semi quantitative detection based on the reaction between target substance and chemical reagents. This reaction involve indicator which results in the color change with the completion of reaction. The detection results can be directly visualized or with the help of some computer software. Generally computer software is used only when the direct visualized is not possible.

This detection techniques is widely used as it provides accurate results at lower cost. Both enzymatic techniques and gold, silver nanoparticles are used in the calorimetry detection. In enzymatic technique, enzyme-substrate complex is formed due to the reaction between enzyme and specific substrate. This complex produces a visible color change. The enzyme is immobilized on the paper with the help of some reagents. Many innovations are made to decrease the limit of detection (LOD) and improve the detection sensitivity (Kumar et al. 2016, 2017, 2018; Almeida et al. 2018). In a study, paper is modified by multi-layering for uniformed color intensity detection of uric acid and glucose (Wang et al. 2018b). Horseradish peroxidase (HRP) combined with specific oxidase (uricase and glucose oxidase), chitosan and different kinds of chromogenic reagents were coated on the microfluidic paper. Produced Hydrogen peroxide reacts with substrates and also oxidizes the co-coated chromogenic reagents which produces color change. Here HRP is used as catalyst and to make the color uniform. The device is then placed under LED lamp which will suppress the external light. Mobile phone and image J software is used for the further analysis of the recorded images. The schematic of the colorimetric detection technique is given in Fig. 10.4.

10.3.2 Surface-Enhanced Raman Spectroscopy (SERS) Based Detection

Another method of detection is using gold (AuNPs) and silver nanoparticles (AgNPs) due to their change in absorption spectrum with change in the size. They are immobilized in the paper and are incorporated to label the secondary antibodies

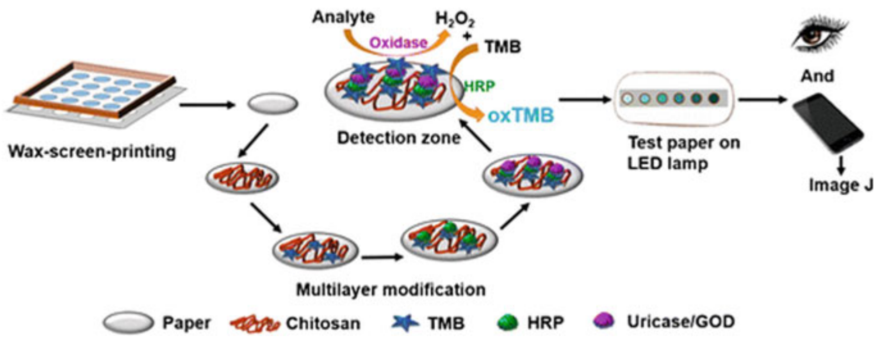

Fig. 10.4 Schematic of screen printed colorimetric assay based detection. Reprinted with permission from Wang et al. (2018b). Copyright (2018) Springer, Nature).

as detecting components. The color change is observed because of the aggregation of nanoparticles with the antibody-analyte (antigen) interaction. This change in the property of the nanoparticles is quantified with the surface plasmon resonance (SPR) effect which utilizes the plasmons. The disturbance of electrons from their mean position is the reason for the generation of these quantum oscillations known as plasmons. This also increases the electromagnetic field near the surface of the nanoparticles which can become the basis for the surface-enhanced Raman spectroscopy (SERS) (Wei et al. 2015). The Raman spectrum is different for different molecules depending upon the vibrational modes present. The SERS effect enhances the Raman cross section after the interaction of a molecule with AuNPs (Li et al. 2018) and AgNPs (Wei et al. 2015). The electromagnetic field of near the nanoparticle gets enhanced due to this type of interaction which can be detected from the spectroscopy analysis.

However, these nanoparticles leaks from the paper upon washing due to which leaching indicator and binders are incorporated in the device. Apart from the binder, many other reagents are also used to increase the specificity and enhance the detection. One of the successful attempt were made to detect the contamination of mercury ion in water with the help of gold nanoparticles (Chen et al. 2014). Citrate capped gold nanoparticles with the average size of 13 nm were synthesized and is mixed with ssDNA in the colloidal form to make the ssDNA-attached gold nanoparticle colloid. This colloid is printed on the paper to make the working device. As Hg^{2+} ion comes in contact with the printed colloid, it reacts with the present thymine to form the thymine- Hg^{2+}-thymine coordination bond and this aggregated the attached gold nanoparticles to show the color change. Such type of studies confirm that SERS based sensors can be used for the detection of biomolecules and metallic ions from different type of test samples.

10.3.3 Electrochemical Detection

By the virtue of its maturity and well-understood working principle, electrochemical detection is widely used in sensing applications. Integrating it with paper based devices gives us low cost, portability, high selectivity, sensitivity, low power consumption as its benefits to name a few (Määttänen et al. 2013). When an electric current is passed through the electrolyte, electrodes functions in a specific manner which leads to the detection of some entity on further analysis. A typical electrochemical sensor consists of three different electrodes: working electrode, counter electrode and reference electrode as shown in Fig. 10.5. Current is passed between the working and counter electrode which can be easily fabricated on the paper using conducting inks. In the electrolytic cell arrangement, the analyte is detected at the working electrode and the counter electrode is used to maintain the electrical circuit. It collects the current flowing in the circuit and also limits to the current flowing in the system and reference electrode. The reference electrode is generally made of

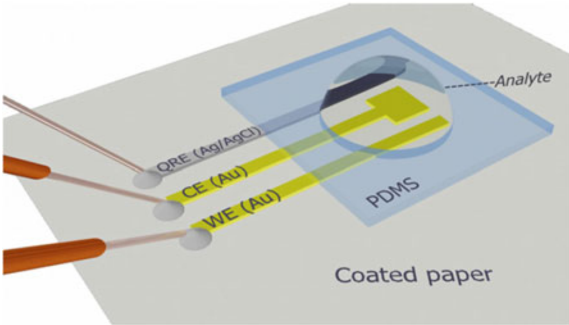

Fig. 10.5 Schematic of paper based electrochemical sensor with illustration of electrodes. Reprinted with permission from Määttänen et al. (2013). Copyright (2013) Elsevier, Sensors and Actuators B: Chemical

Ag/AgCl and is placed away to avoid any contact with the reaction to maintain the known constant potential (Määttänen et al. 2013).

Many experiment directly uses this setup while constant modifications are made frequently to enhance the detection on paper based device. For detection of trace amount of heavy metals (Cd^{2+} and Pb^{2+}) in the water using electrochemical detection (square-wave anodic stripping voltammetry), miniaturized carbon based sensor was fabricated (Shen et al. 2017). The contaminated water being the analyte here is flown along the microfluidic channel and both the electrodes are made facing each other at the ends of the channel. This device detects the trace amount of heavy metal, and is very robust with results highly reproducible.

10.3.4 Luminescence Based Detection

This detection technique includes fluorescence, chemiluminescence and electro-chemiluminescence, and is widely used with the microfluidic devices. It is more sensitive compared to the photometric measurement because of the signal strength dependence on intensity of incident radiation and analyte concentration (Almeida et al. 2018). In this method, direct emission of the light as well as fluorescence quenching of analyte is used for the detection. Xin-Ran et al. fabricated a polymer grafted paper based fluorescent sensor by using CdTe quantum dots (Wang et al. 2015). Paper based device was fabricated to detect Cu^{2+} ions using fluorescence inhibition. The schematic of sensing mechanism and fluorescence spectra is shown in Fig. 10.6. Chemiluminescence and electrochemiluminescence are generally more sensitive than fluorescence measurement owing to the low background light signal. This low signal is due to the absence of the excitation light source. Here also, detection can be done by emission or inhibition. However, electrochemiluminescence measurements are considered to be better due to the electrochemical control of timing and location of excitation on the paper based device which is not possible in chemiluminescence.

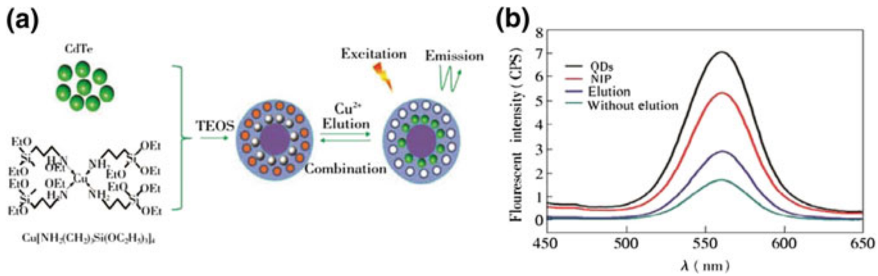

Fig. 10.6 Schematic diagram representing the sensing mechanism involved in the detection of Cu +2 (**a**), and the result of fluorescence intensity variation during detection (**b**). Reprinted with permission from Wang et al. (2015). Copyright (2015) Elsevier, Chinese Journal of Analytical Chemistry

These techniques are widely used and integrated with the microfluidic device for the low cost and robust detection of the analyte. In similar approach. a microfluidic detection chip was fabricated for detection of formaldehyde in various food sample, which consist of micro paper analytical device (Liu et al. 2018). This µ-PAD is fabricated by the wax printing and implanted with acetoacetanilide reagent. Basic Hantzsch reaction process is used for the detection in which, formaldehyde-acetoacetanilide complex (dihydropyridine) is produced. The concentration of formaldehyde is detected from the UV light – induced fluorescence intensity of the formed complex.

10.4 Paper Based Sensors for Environmental Monitoring

Paper based sensors are widely used in environmental pollution monitoring. The application of these devices for water and air quality monitoring is described in following sections:

10.4.1 Paper Based Sensor for Water Quality Monitoring

Paper based sensors are widely used for water quality monitoring. Water based contaminants such as harmful heavy metals, chemicals, dyes, and bacteria are very injurious to the health of flora and fauna. These contaminants should be detected and removed from the usable water as early as possible to eliminate the spreading of the various waterborne deceases. The onsite and fast detection system is required for these contaminants to improve the water quality. The fast detection system cannot be based on the conventional methods such as liquid chromatography and mass spectroscopy. The advantage of fast detection by using paper based sensors provides a feasible solution to the water health quality monitoring system. Hossain et al. developed a paper based sensor for the detection of organophosphate pesticides in food and beverages (Hossain et al. 2009). In this method, the paper substrate is used to detect pesticides by means of change of colour from yellow to blue due to enzyme catalysed hydrolysis of the substrate in presence of the pesticide acetylcholinesterase and indophenyl acetate (IPA). The paper strips were successfully tested for milk and apple juice samples. As shown in Fig. 10.7, the reagentless lateral flow paper based sensor detects pesticides effectively. Polyvinyl Amine (PVAm) is used as a cationic capture agent on the paper which captures anionic products of the reaction and preserves the response. This type of sensor can be used for the detection of pesticides from water samples.

Sicard et al. followed the similar approach and developed a paper based analytical device (μPAD) to detect organophosphate pesticides (Sicard et al. 2015). The mechanism for the detection was based on the inhibition of coated

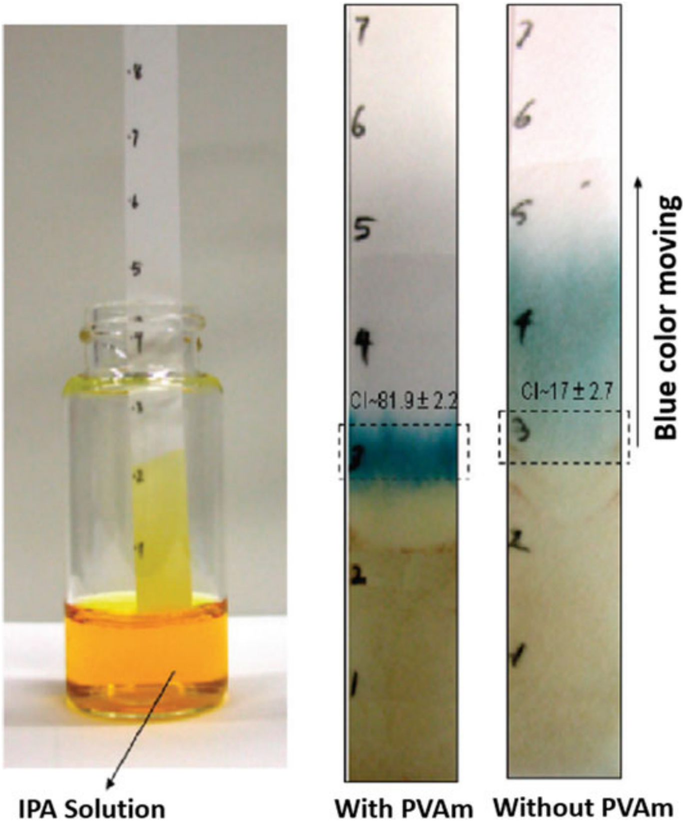

Fig. 10.7 Paper strip for the detection of pesticides from the food products in a lateral flow detection system (The dashed boxes show the coated regions). Reprinted with permission from Hossain et al. (2009). Copyright (2009) American Chemical Society, Analytical Chemistry

acetylcholinesterase by the contaminants present in the water sample. Also, the monitoring system was linked to the smartphone and centralized web portal to collect the data on large scale as shown in Fig. 10.8.

The steps involved in the detection are explained schematically in Fig. 10.9. The detection mechanism is based on the change of color of indoxyl acetate from

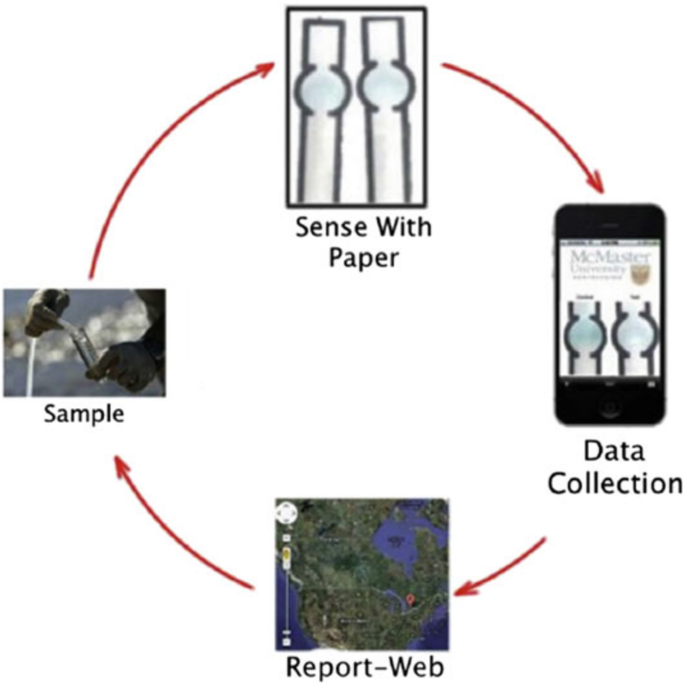

Fig. 10.8 Schematic of detection system by using cell phone and web portal. Reprinted with permission from Sicard et al. (2015). Copyright (2015) Elsevier, Water Research

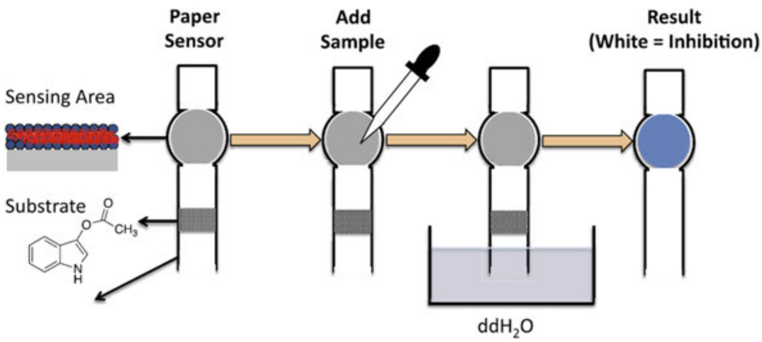

Fig. 10.9 Steps for pesticides sensing from water samples. Reprinted with permission from Sicard et al. (2015). Copyright (2015) Elsevier, Water Research

colorless to blue in absence of pesticides and white in presence of pesticides. The pesticides present in the water sample inhibit the acetylcholinesterase (AChE). The AChE was sandwiched between the silica gel layers over the treated paper substrate.

10.4.2 Paper Based Sensor for Air Quality Monitoring

Paper based sensors are also very useful in detection of harmful gases present in environment. Various toxic gases generated from industries, automobiles, and chemical industries etc. are released in the atmosphere (Chauhan and Bhattacharya 2018). These gases are harmful to human and animal life because of their toxic and explosive nature. Paper based sensors are now widely used for the rapid detection of these harmful gases which is helpful in monitoring, regulating, and alarming the nearby controlling and safeguarding authorities.

Wang et al. developed a metal-organic framework (MOF) with amino functionalization on a paper substrate and utilized it for the colorimetric and luminescence detection of toxic sulfur dioxide SO_2 gas (Wang et al. 2018b). SO_2 gas is widely used in making of pesticides, dyes, and pulp making. The long-term exposure to the SO_2 gas causes respiratory and nervous system damage for human beings. The MOF's are crystalline porous materials with π-conjugated structure which facilitates the luminescence property. Wang et al. used Zinc and benzenedicarboxylate (BDC) as metal and organic counterparts, respectively (Wang et al. 2018b).

In this work, amino group acts as an electron donating group and transfers electrons through the metal ions during the formation of MOF. The interaction with SO_2 gas hinders this electron transfer phenomenon and results into luminescence turn on effect. From Fig. 10.10, it is evident that the luminescence intensity of the MOF-NH_2 is maximum for SO_3^{2-} ions with respect to the other interfering ions present in the solution. Also, the luminescence response of MOF- NH_2 coated paper for different anions is shown in Fig. 10.10c. The similar paper based sensor coated with MOF- NH_2 was exposed to the different harmful gases and the maximum luminescence can be observed for SO_2 gas in Fig. 10.11.

Following a similar approach, Maity and Ghosh went one step ahead and developed a visual color changing paper based sensor for ammonia gas detection (Maity and Ghosh 2018). This method doesn't require any special instrument but visual analysis is sufficient to detect the presence of ammonia gas. In this study, the disposable paper was coated with perovskite halide $CH_3NH_3PbI_3$ (MAPI) which has black color. In presence of toxic ammonia gas the color change from black to yellow due to the conversion of MAPI into PbI_2. Figure 10.12 shows the change of color of the test paper from black to yellow after exposure of toxic NH_3 gas (30 ppm). The X-ray diffraction analysis (Fig. 10.12b) confirms the formation of PbI_2 from MAPI in presence of NH_3 gas. These studies indicate that paper based gas sensing devices are being explored and widely used for environmentally toxic and polluting gases.

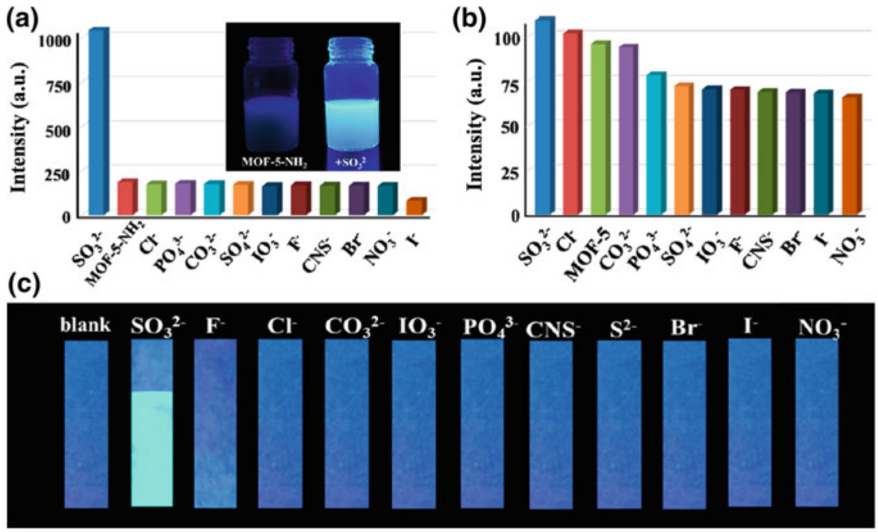

Fig. 10.10 **a** Variation of luminescent intensities of MOF-5-NH_2 and **b** MOF-5 with other anions. **c** Image of test strips in presence of different anions under UV light (365 nm). Reprinted with permission from Wang et al. (2018b). Copyright (2018) American Chemical Society, Analytical Chemistry

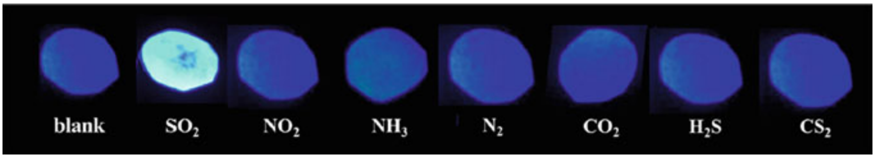

Fig. 10.11 Luminescent response of test strips in presence of different gases under UV light (365 nm). Reprinted with permission from Wang et al. (2018b). Copyright (2018) American Chemical Society, Analytical Chemistry

10.5 Challenges

Although paper based environmental sensors possess large number of advantages over the conventional detection methods but there are certain challenges which are required to be addressed for the future of paper based sensing devices. One such issue is the admission of the sample to the test paper. The sample to be tested is sometimes at a considerable distance from the testing region of the paper which alters the concentration to be analyzed. Also there are chances of evaporation of the sample. There is also the requirement of improving the immobilization efficiency of biomolecules, enzymes, and other nanomaterials over the porous paper substrate. The nanoparticles can be attached to the fibres during fabrication of paper. Also the treatment of the paper can be done with functionalization of different groups

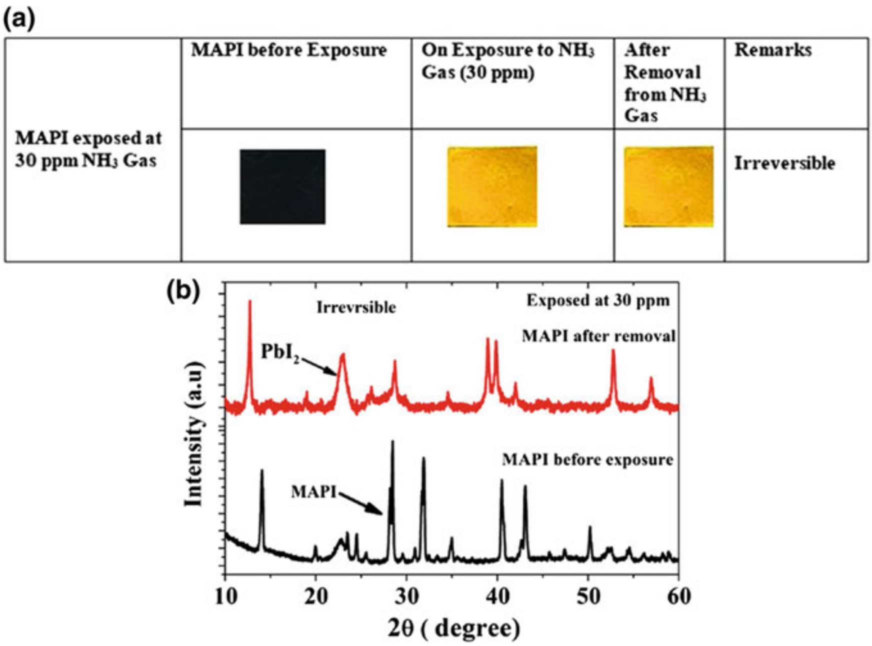

Fig. 10.12 **a** The colour response of test paper in presence of NH_3 gas (30 ppm) and **b** XRD analysis of the test paper before and after exposure of NH_3 gas. Reprinted with permission from Maity et al. (2018). Copyright (2018) Nature, Scientific reports

(carboxylic, hydroxyl, and amino etc.) to facilitate the attachment of nanoparticles and create hydrophobicity/hydrophilicity. There is also the need of different type of papers depending upon the application. The electrode printing for electronics require smooth hydrophobic type paper. The analysis of colorimetric detection method sometimes becomes subjective to the user. Now a day's smartphones are being used to minimize the issue of relative color change for the small variation of analyte properties.

10.6 Conclusion

In this book chapter paper based environmental sensing devices have been discussed. Paper based sensors have advantage of improved microfluidic activity due to high porosity, capillary action of cellulose fibres, flexibility of the paper, and easy functionalization/treatment of the paper. The paper based substrate provides easy, fast, and cheap point of care detection of environmental as well as bio-entities. The paper based sensors are also efficient in immobilization of enzymes, bio-molecules, and chemical compounds due to their high porosity and adsorption capability. In this chapter, the environmental aspect of the paper based sensors have been studied. Paper based devices are widely used in water quality monitoring by the rapid and simple detection of pesticides, heavy metals, and other chemical pollutants present in the water samples. At the same time, paper based gas sensors are also trending for the effective and simple detection of polluting gases such as NH_3, SO_2 etc. The main advantage of paper based sensors working on colorimetric detection is the visual analysis without involving any sophisticated instruments. The paper based sensors are now being used in environmental as well as other field of detection technology.

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