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Comparative investigations on microextraction and conventional air sampling techniques: challenges and future directions

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REVIEW

Comparative Investigations on Microextraction and Conventional Air Sampling Techniques: Challenges and Future Directions

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Abstract

Microextraction technique (e.g., solid phase microextraction, thin film microextraction, in-tube extraction) brings a revolutionary change in air sampling techniques over the recent few years. This advanced technique exhibits a high pollutant extraction rate, a low retention time, and a lower error margin compared to conventional air sampling techniques. The accuracy range of microextraction technique (MET) was recorded ~90−95% to isolate the volatile organic components, oxygenated and halogenated carbon particles from the air. However, the efficiency of MET increases additional >3−5% when employed by coupled with gas chromatography or gas chromatography-mass spectrometry. The conventional sampling techniques (e.g., bag sampling, grab sampling) on the other hand, displayed the accuracy of ~75−80% which is ~20% lower than MET. The factors that potentially affect the performance of both conventional and MET were thoroughly investigated in this study. For instance, it was observed that the quality of needle coating used in MET significantly affects the pollutant trapping and at least ~5% of total performance cut off due to damaged and corroded coating. In addition, smart sensor-based air sampling techniques are also being investigated as this technique is a recent development in air quality monitoring. This fully automated state-of-the-art technology shows more than 98% accuracy with significantly high sensitivity and pollutant extraction rate. Finally, this investigation distinguishes the potential advantages, disadvantages, and challenges to increase accuracy between advanced and conventional sampling techniques, drawing attention to the urgent need to improve the performance of the air sampling techniques investigated in this study.

Keywords: Microextraction, In-tube extraction, Passive sampling, Adsorption technique, Air pollution

1. Introduction

Air pollution has become a global threat that causes millions of human deaths every year [1]. According to a recent report from the World Health Organization (WHO), approximately 2.4 million people die from air pollution annually [2]. Airborne particulates or air pollutants, floating as a mixture of solid particles, liquid droplets, or gases in the air, are responsible for air pollution. There are plenty of reasons behind pollution, including human activities that often play a critical role in the formation of pollutants and their release into the environment. Due to unplanned urbanization, air pollutants are entering the air; for instance, bituminous mixes with waste concrete aggregates release air pollutants [3]. Wildfire industrial emissions and metal e-waste are also major sources of air pollutants [4−7]. Some of the major air pollutant emission sources are shown in Fig. 1.

There are various forms of air pollutants, broadly classified as inorganic, organic and microbial air pollutants [1−4]. The most common inorganic pollutants are ozone, carbon monoxide (CO), nitrogen oxides (NO_x), sulfur oxides (SO_x), ammonia (NH_3), hydrogen peroxide (H_2O_2), nitric acid (HNO_3),

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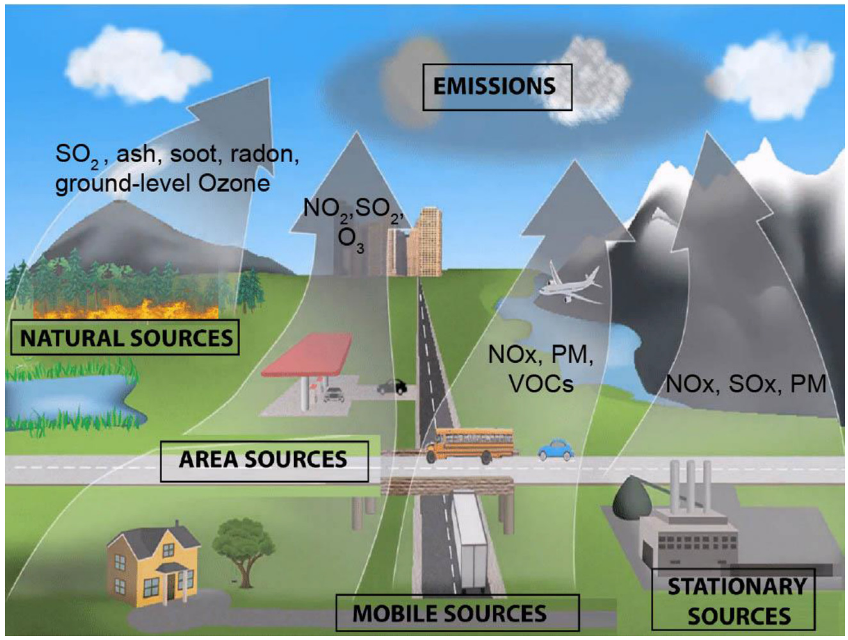

Fig. 1. Different potential sources of air pollutants including natural, area, mobile and stationary sources. Most toxic and carcinogenic air pollutant emissions occur from these sources.

isocyanic acid (HNCO), and hydrogen sulfide (H_2S) [2,5,8,9]. In a recent study, Palliyarayil et al. (2021) introduced a variety of specific sources and percentages of inorganic pollutant emission of corresponding sources to the management of air pollution [10].

The sources of CO, NO_x, SO_2, CO_2 pollutants and their relative contributions to the atmospheric air pollution, are shown in Fig. 2. The adverse effects of these pollutants on human health have become a major concern. The common health issues found are nose and eye irritation, throat itching, vomiting, severe kidney and liver damage, and nervous system damage [2,8,9,11,12]. Organic air pollutants such as dyes, pesticides, organic solvents, detergents, and others are mainly found as industrial byproducts; and other natural sources (e.g., wildfires, and residential areas) (Fig. 1) [13–15]. Similar to the inorganic pollutants, exposure to the organic pollutants has a bad impact on health. The most common studied organic pollutants in the air are volatile organic compounds (VOCs), halogenated volatile organic compounds (HVOCs), persistent organic pollutants (POPs) and related compounds such as polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), monocyclic hydrocarbons (e.g., benzene, toluene, xylene, and aliphatic chemicals), vinylidene chlorides, polychlorinated dibenzodioxins/furans (PCDD/Fs), and organochlorine pesticides (e.g., DDT, chlordane, dieldrin and hexachlorobenzene (HCB)) [1,13,16–18]. The combined effects, from the exposure to these pollutants may cause cancer, severe kidney and liver damage, skin and eye irritation, reproductive losses, immune and nervous system impairments, itching, and hormonal imbalances [17–21]. In addition, industrial breeding of birds (e.g., duck breeding, boiler breeding) and industrial effluents are also responsible for microbial air pollutants (e.g., pollen particles, molds, mildew, dust, viruses, etc., in the atmosphere [20,22,23]. Inhalation and excess exposure to these microbial pollutants may cause eye irritation, liver damage, nervous breakdown, and diarrhea [20,24]. Therefore, it is critical to monitor these pollutants through proper air sampling methods in order to mitigate the pollution.

Industrial effluent and waste are one of the biggest factors polluting the atmosphere air. The construction waste [25], ceramic waste [26], bituminous and coal refineries [7], and food processing industries are releasing toxic particles and particulate matter to pollute atmospheric air. Azevedo et al. (2021) claimed that only cement industry contributes to 7% of total CO_2 emissions globally [25]. In addition, oil spill by tanker leakage from the oil refineries also contributes to air pollution by releasing hydrocarbons, aerosol particles and NO_x [27,28]. Urban air contains significantly high particulate matter 2.5 (PM2.5) than rural areas according the latest statics of STATISTA [29].

An advanced air sampling technique is needed when the number of pollutants in the air increases and their harmful effects become more evident. Air

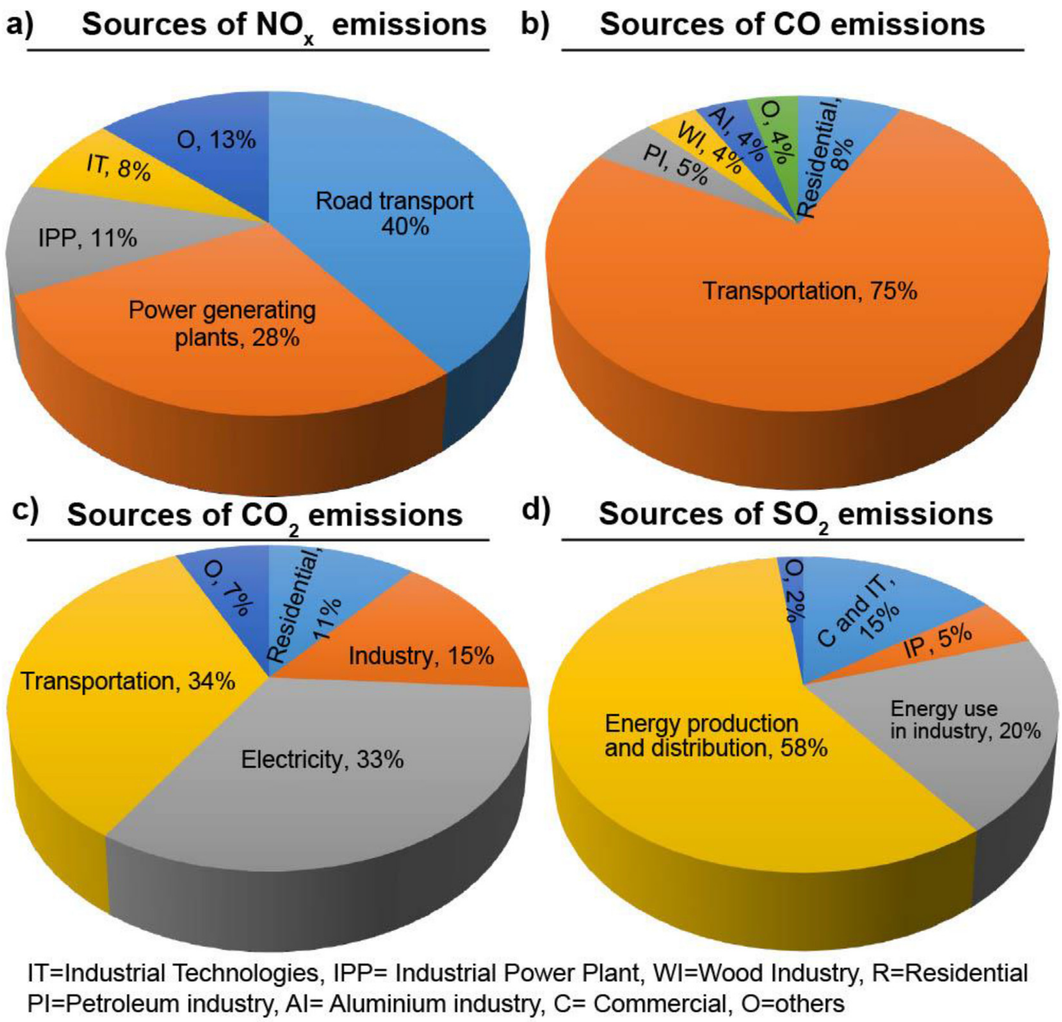

Fig. 2. Sources of CO, NO_x, SO_2, CO_2 pollutants and their contribution (in percentage) to air pollution. The road transport, transportation, electricity, and energy production sources have major contribution to air pollution for NO_x, CO, SO_2, and CO_2 pollutants, respectively. Data adopted and redrawn from [10].

sampling technique is a process used to determine which airborne pollutants are present in the air [9]. The technique used to conduct air sampling for air quality monitoring varies. Though many conventional techniques (e.g., grab sampling, bag sampling, and gravity technique) of air sampling were invented over the past few decades, only a few are considered advanced, for instance, microextraction techniques (MET) are one of them. Advanced sampling techniques such as solid phase microextraction (SPME), and needle trap microextraction (NTME) compensate for the drawbacks of the conventional techniques. It is worth noting that the deployment of a sampling technique could be site specific since all techniques are not equally effective. The settle plate technique, for example, could be used for indoor air sampling, whereas bag sampling and MET are used for outdoor sampling [13–15,30,31]. Also, many of them are coupled with different spectrometry and chromatography which make them unique as a combination of conventional and advanced techniques [16–18].

Many studies have been conducted on air sampling techniques for the further development of the sampling process to deliver information on pollutants present in the air of a specific area [13,15,16,19,20]. For instance, Jaschhof (2019) et al. used gelatin membrane filter for sampling virus aerosol in air and this technique was permitted to air sampling rate within a standard time [21]. However, this approach failed for sampling high volume of sample (i.e., >300 L/min) as reported by Jaschhof [21]. A comprehensive review by Brown et al. (1994) presented a mathematical model to calculate VOCs, Total VOCs in indoor air [22]. However, this investigation lacked experimental trials and factors that could influence the sampling process [22]. In addition, the recent studies such as NTME [32], in-tube extraction (ITEX) [14], sorptive extraction [31] and Stir-bar sorptive extraction (SBSE) [23] used extraction technique to isolate the pollutant from air. However, although some existing review on air sampling techniques specifically discussed about specific air sample technique, none of them overviewed the challenges and advances in all of the conventional and advanced air sample techniques. This comprehensive review, therefore, aims to provide a critical overview on advances and challenges in conventional and advanced air sampling techniques with respective pros and cons, feasibility of each sampling techniques based on the site location, and accuracy of each technique. In addition, impacts of air pollutants on human health are also included. Finally, future outlooks have been suggested for the improvement of air technique which might be useful to the readers, policy makers and stakeholders.

2. Air sampling techniques

Air sampling is the process of trapping pollutants for further analysis to determine the air pollution level of a particular area. Some techniques are not economically feasible, are expensive and sophisticated to maintain, and have a higher error percentage. In contrast, some techniques are cost-effective, easy to handle, and provide higher accuracy. In the current study, we divided all air sampling techniques into two categories: conventional techniques and advanced techniques. Also, information is tabulated and presented in multiple tables to give the readers a quick overview of different sampling techniques.

2.1. Conventional air sampling techniques

2.1.1. Settle plate sampling

Settle plate plays an important role in monitoring the air quality of indoor air for assessing the microbial settlement where maintaining an unidirectional airflow. The settler plate potentially settles out the microorganisms present in the air due to the gravitational effect. In this technique, the plate is placed in a representative location inside a unidirectional cabinet. The infiltration of any microorganism into the cabinet is detected by the plate. A lid is placed on the top of the plate, which is removed during the operation. The pollutant settling rate on the plate depends on the physical characteristics of the particle. Generally, the large particles tend to settle faster than the smaller particles due to gravitational effects. The settling tendency of smaller particles is low due to air currents and air resistance. In most cases, the particles larger than 7.0 m completely settle down, which are also known as 'complete particles' [13]. Settle plates are suitable for places where the larger air pollutant particles are generated, such as industrial areas, hospitals, and residential areas [13,24,33]. Valentina et al. (2019) used the settle plate method for air sampling and quality assessment of the air in the hospital area, which reported in detail the percentage of the microorganisms extracted from the air samples [24]. However, this method is not suitable for small-sized (nano or lower) pollutants [13]. The number of pollutant detections in the settle plate technique is less than some of the more advanced techniques, such as the SAS-Super-180 air sampler [34]. The information on different conventional sampling techniques is summarized in Table 1.

2.1.2. Passive air sampling

Passive air samplers (PAS) have been one of the most widely used sampling techniques over the past few decades. This sampling technique is mostly used for gaseous air pollutants, including volatile organic compounds, nitrogen dioxide, sulfur dioxide, and ozone. Though measuring the amount of persistent organic pollutants (POPs) in the air is limited by this technique [35], A few studies used stationary PAS to measure POPs in the indoor air in residential areas. It is worth mentioning that the PAS device setup is straightforward and easy. The device is placed at a central position in the breathing zone of the inhabitants (typically 1.5 m from the ground) and approximately 20 m apart to ensure the uniformity of air samples. The exposure time frame could be 4–5 weeks, but it depends on the sample collection place and the type of residence investigated [36]. In most cases, PAS is used for personal monitoring rather than occupational air quality monitoring. Nonetheless, it was discovered in several cases that the PAS was used in various industrial areas to measure POPs and indoor air quality [35,36]. PAS has a number of advantages, including being faster, more effective, and less expensive than other sampling techniques that allow direct monitoring of the outcome and concentrations of pollutant particles in the air, particularly indoor air [19,36,37]. This method suffers from several drawbacks, including its limited use for personal safety monitoring, indoor-only air sampling, and relatively higher sampling duration.

2.1.3. Grab sampling

This sampling technique is another easy example of conventional air sampling techniques. The air sample is collected in an evacuated container, e.g., a

Table 1. Notable conventional air sampling techniques accuracy rate, their advantages, disadvantages and challenges.

Sampling technique	Types of pollutants	Advantages	Disadvantages	Sampling Accuracy	Challenges	Ref.
Settle plate	Aerobic bacterial flora, fungi, bacteria,	i Highly efficient hospital, school, and industrial areas. ii Micron size particles can easily extract.	i Less effective for nano or lower size particle. ii Pollutant detection percentage is comparatively low.	≥70%	i Petri damage. ii Exposure time varies with the site locations. iii Nano size particle detection.	[13,24,34]
Grab sampling	Microplastics compositions, microfibers pollutants, CO_x, NO_x	i Using sorbent tubes. ii Plastic bag, canister, glass containers are using as a sample collector.	i Large-exposure times e.g., 4–5 weeks. ii High possibility to damage containers e.g., plastic bags, inflate bags.	≥76%	i Portable pump failure. ii Canister damage iii In-situ sample analysis in sample collection spot.	[19,34,38,40,41]
Bag sampling	Gaseous and aerosol particles.	i Easy to handle and install. ii Cheap, light weight, unbreakable. iii Economical to ship long distances to a laboratory.	i Sample losing by moisture condensation or diffusion. ii Sampling duration varies depending on the sampling area.	≥56–72% (depending on the bag size)	i Loss of pollutant particles due to diffusion. ii Sample recovery rate is low.	[25,30,37,38]
Adsorption	NH_3 cyanogen chloride (CNCl), AsH_3, H_2S, Radon (Rd), PH_3	i Wide range of tubes are available. ii Highly effective to trap air pollutants. iii Coupled with mass spectrometry and chromatography.	i Sophisticated to temperature and pressure. ii Molecular sieves production is expensive. iii Corrosion could degrade efficiency of sorbent tubes.	≥80%	i Maintain the quality of adsorption tube. ii To control the constant temperature and pressure. iii Selection of suitable adsorption agents.	[44–46]
Passive sampling	VOCs, NO_2, SO_2, POPs.	i PAS device set up is easy and straightforward. ii Mostly effective in indoor air sampling. iii Best for personal air quality monitoring.	i Sample exposure time is long. ii Affected by external parameters such as humidity, face velocity, barometric pressure, and temperature.	≥14–20% detection with high precision.	i Passive air samples must be analyzed close in time to collection. ii Influence of particles is unknown. iii Maintain constant sampling rate.	[35,36,47]

flask, plastic bag, or inflatable bag. The condensation of the collected sample inside the container or diffusion through the wall often leads to samples being lost or wasted. However, using a glass or stainless steel container has been a solution to minimize the sample loss [19]. These containers are evacuated before allowing air to fill them up. Alternatively, another container filled with water is used as a collector by draining its water, which is replaced by the filling air sample. These containers are evacuated before allowing air to fill them up. Alternatively, another container filled with water is used as a collector by draining its water, which is replaced by the filling air sample [19]. In a review study, Watson et al. (2011) introduced a more advanced technology of grab sampling to measure the air quality up to a few milliliters [40]. This study revealed that the addition of a sorbent tube and a piston-type pump could make the grab sampling technique more efficient. The reason is that the piston pumps are capable of sampling both large volumes for trace-level pollutants and small volumes for high-concentration chemical pollutants. The most important advantage of this method is the use of a piston pump or constant flow pump, which significantly reduces the sampling duration [40]. In addition, grab sampling is better than many available conventional sampling techniques, as shown in Fig. 3. The major drawbacks of this technique are the difficulty in ensuring the stability of the analytes due to sample transportation in bottles or canisters from the sampling site to the laboratory [48].

2.1.4. *Bag sampling technique*

Bag sampling is particularly suitable for short-term samples, including aerosols and airborne pollutants [49]. This technique provides detailed information on air pollutants, including particle size distributions [30]. A variety of sampling bags (e.g., plastic bags, Tedlar bag and electrically conductive (Velostat™) bags) are used based on their availability in different regions across the world, as reported in literature [30,43,49]. Schulz et al. (2004) developed a new method for measuring the vertical profiles of CO_2 by using a special type of bag named the Tedlar bag [43]. An air pump is connected to the sample collector bag in bag sampling techniques. The pump is connected to two solenoid valves that control the air flow rate inside the bag. The bag is filled with air using a Teflon-lined Tygon tube. The sampled air passes through a glass fiber filter for pollutant removal and ease of analysis [30,43]. A typical arrangement of a bag sampling technique is depicted in Fig. 4. The accuracy of bag sampling techniques is not as high as the advanced techniques we will be discussing in the latter part of this study, however, the error percentage of this technique is less than 0.5 ppm found elsewhere [43]. This technique is very useful for sampling gaseous and aerosol particles. Moreover, this techniques is handy, cost-effective and provides high precision which makes it popular [30,42,43]. A major drawback of this technique is the potential of losing samples due to condensation or diffusion, as discussed previously [19]. As a result, if a sample must be stored for an extended period of time due to an instrument problem or for any other reason, this technique is inapplicable [30].

2.1.5. *Adsorption technique*

Adsorption air sampling method is one of the oldest and prominent techniques to separate toxic

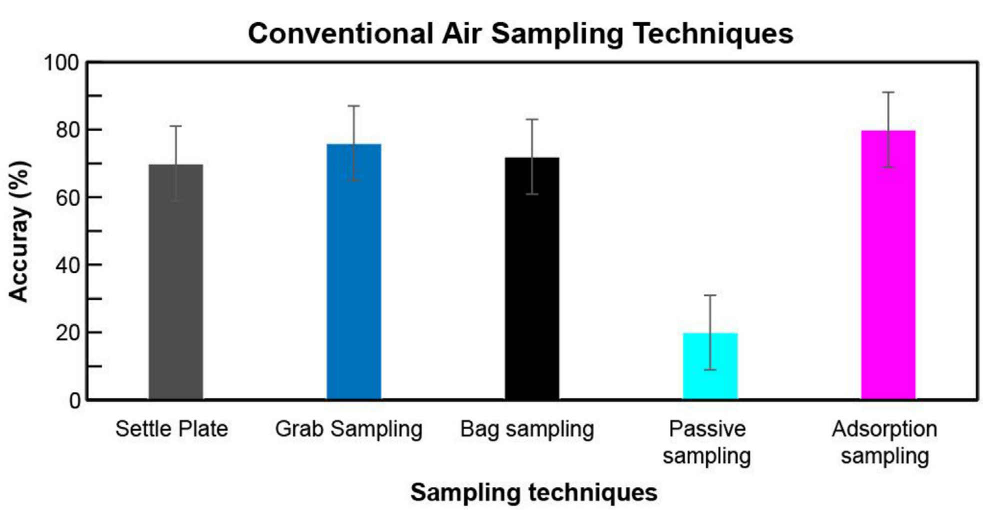

Fig. 3. The comparison of sampling accuracy (in percentage) among different conventional air sampling techniques. The comparison pronounced that the adoption sampling techniques provides the maximum accuracy provides the.

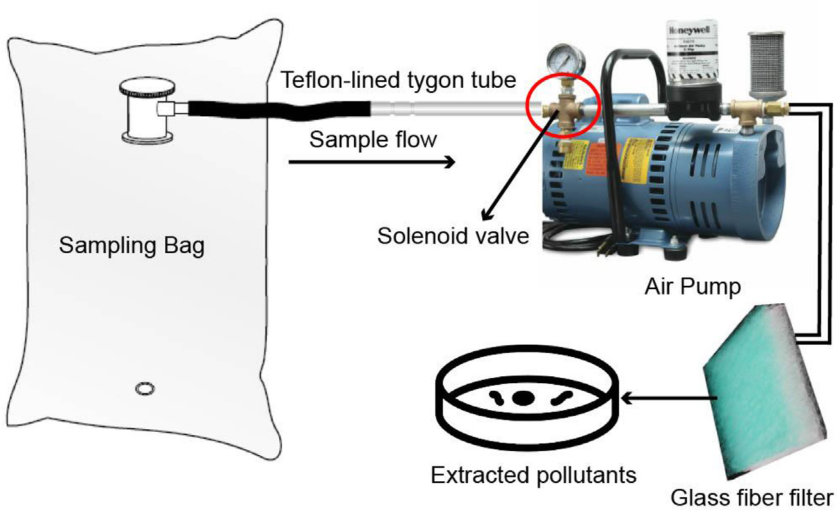

Fig. 4. Schematic diagram of a typical bag sampling technique.

pollutants from the air. Different materials like activated charcoal, char (e.g., biochar), silica gel, activated alumina, activated carbons, and porous zeolites are commonly used in air sampling technique through adsorption of air borne particles. However, adsorption process is temperature and pressure sensitive [44]. The mechanism of pollutant adsorption when charcoal is utilized as an adsorbent is shown in Fig. 5. In this method, charcoal leaching is performed using a suitable solvent, such as carbon disulphide (CS_2). The solution leached with charcoal is analyzed using a gas chromatographer [44]. Harrison et al. (2013) introduced a stainless steel made adsorption tube with gas chromatography (GC) was used for the collection of air pollutants from the air [50]. A special type of Millipore filter (type GSWP 04700) was set up at the entrance of the tube to separate the air pollutants. This filter runs a pump and a gas meter. After collecting the required amount of sample, the tube was removed and the sample was stored for further analyses [50]. In an extensive review study, Woellner et al. (2018) revealed that the adsorption technique using metal−organic framework was highly effective to detect the hazardous trace gases (ammonia, cyanogen chloride, arsine, hydrogen sulfide, radon, phosphine and others) in the air [45]. The accuracy (up to 0.1 ppb) and the efficiency of adsorption technique are much higher than the other conventional techniques discussed above [44,45,50]. However, this method has some disadvantages including corrosion to the adsorption tube, maintaining the constant temperature and pressure, and selection of suitable adsorbents [44−46].

3. Advanced air sampling techniques

3.1. Needle trap microextraction

Needle trap microextraction (NTME) is a promising new technique for air sampling for volatile organic compound (VOC) analysis, as it provides the combined advantages of SPE and SPME [15,20,32]. A needle trap device (NTD) is used that is

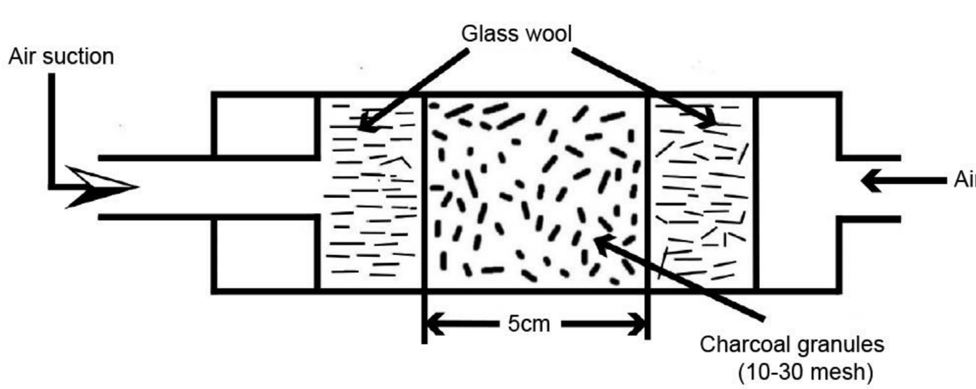

Fig. 5. A typical adsorption tube filled Charcoal, a GC packing material, for collecting air samples. The mesh number of the charcoal granules should be in the range of 10−30. An air pump and a filter are set up inside this tube to suck the pollutant particles.

comprised of a syringe needle with a particular gauge (e.g., 23 gauge) and a quartz wool bed. This bed is enabled by a glucose injection. The NTD should be connected to a syringe that collects the sample, facilitated by a plunger moving in an upward and downward direction. The NTD is then thermally desorbed into the GC inlet, and the amount of VOC particles bound in the GC system is determined by the quartz-wool bed [15,24]. In the past year, Lan et al. (2020) used the NTME method to trap the air using epoxy glue as a polymer coating on the needle and performed further laboratory analyses. To extract VOCs from the sample, the sampling flow rate was 30 mL/min with significant high accuracy (5 error percentage).

The advantages of the NTME are fairly extensive. For instance, this technique requires a small quantity of sample and sorbent materials, which makes it economical and easy to handle. Also, there is no effect of humidity on the sorbent material, which provides higher accuracy, and the error percentage is compensated by the adjusted standard deviation [15]. Trefz et al. (2012) introduced a polymer NTD which is very efficient with good reproducibility and sensitivity for most VOCs [32]. Moreover, an NTD with a side hole needle has been reported to be more effective due to the absence of epoxy glue to hold the sorbent materials [51]. In contrast, the NTME shows low sensitivity when a small quantity of sample is used. Increasing the sample volume would be a solution, but it would be time consuming. The overall experimental set-up of NTME is sophisticated, and inappropriate handling could generate a high error percentage [15,18,52]. Also, NTME is a leading edge technique that can only isolate the VOCs or Mold volatile organic compounds (MVOCs) in the air of a particular sampling location [20,53].

3.2. Solid phase microextraction

The air sampling technique based on the adsorption has been used widely over the past few decades for the analysis of trace elements and pollutants present in the sample matrix [54]. A new advanced air sampling technique called solid phase microextraction (SPME) was invented by Professor Janusz Pawliszyn of Waterloo University, Canada in early 1990s [55]. The essential part of an SPME device is the silica fiber (approximately 10 mm in length) coated with a suitable polymeric adsorbent (e.g., dimethyl siloxane) [54,55]. The coated silica fiber is first placed inside a needle and then the needle itself is set with a syringe like arrangement, as shown in Fig. 6. The pollutant particles are adsorbed in the coated fiber and then a GC injection port is used to complete the thermal desorption of the adsorbed compounds [54–56]. The SPME plays a tremendous role to prepare samples quickly in both indoor and outdoor sampling locations. Koziel et al. (2006) introduced an advanced use of SPME for the assessment of the analytes present in the odorous livestock gases [57]. The researchers used GC-Olfactory-based assessment of swine and beef cattle odors. Though the air sampling and characterization of odorous livestock gases is a difficult task, the SPME technique proved to be very efficient to extract the odorants and other chemical pollutants from ambient air [57].

The SPME provides a wide range of advantages. For instance, it is very difficult to work with small volume sample with the conventional techniques whereas SPME technique successfully deals with small volumes. Also, SPME allows rapid sample preparation, extraction [55] and transfer to the analytical instrument [58,59]. Likewise, this technique can be used to understand the pollutant

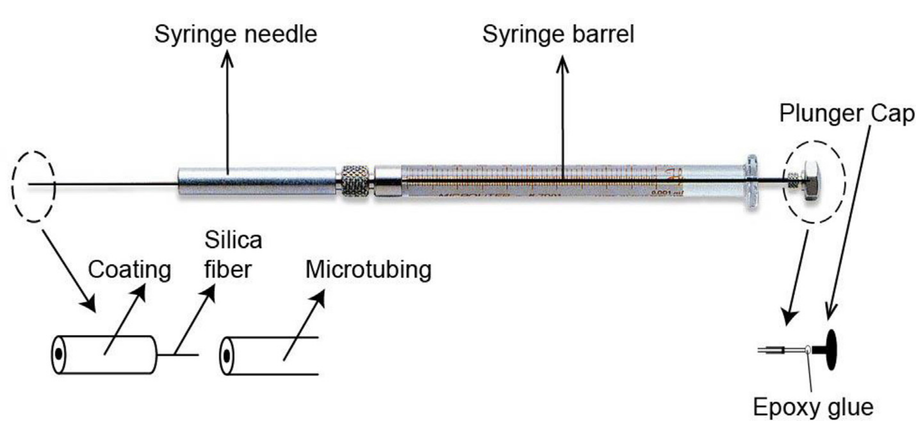

Fig. 6. A typical syringe is used in several microextraction sampling techniques especially in SPME whereas the coating types and thickness inside the syringe may vary depending on the sample.

particles distribution in multiphase systems and speciate different forms of the analytes within a sample [31,55,56]. However, the SPME has some disadvantages over its many advantages. The notable drawbacks of SPME are the coating damage due to scraping and needle bending during the sample agitation. Also, limited flexibility and coating thickness in fiber could result in low amount of coating on the SPME fiber that affects its efficiency [54–56,60,61]. A brief summary of the advanced sampling techniques is reported in Table 2.

3.3. Thin film microextraction

Thin film microextraction (TFME), first introduced in the beginning of 2001, has already become one of the most popular and widely used air sampling techniques [71,72]. The TFME is an advanced mode of the SPME which offers a significant improvement of sensitivity by increasing the surface area to volume ratio along with the extraction phase volume [16,17]. Olcer et al. (2019) described a mathematical equation, as given below in Equation (1), which denotes that the sensitivity increased with an increase in the volume of the extractive phase.

$$n_e^{eq} = \frac{K_{es} V s V_e}{K_{es} V_e + V_s} C_s^0 \quad (1)$$

Here, n_e^{eq} is the extractive phase at equilibrium and C_s^0 is the initial concentration, K_{es} represents the distribution coefficient for analyte in the both extractive phase and sample matrix, V_s and V_e denote the volume of sample matrix and extractive phase, respectively [65,72].

The basic principle of this technique is straightforward, and a schematic diagram of TFME method is displayed in the Fig. 7a [65]. The sample is coated with a thin film during the extraction phase using a metal alloy or fused silica with a typical diameter of 150 μm.The thickness of the coating should be in the range of 7–100 μm. The primary mechanism of this technique depends on the diffusion of the analytes from the sample matrix to the extraction phase to fulfill the boundary conditions and ultimately reach the equilibrium state between the two phases [65]. Kermani et al. (2012) and Jochmann et al. (2006) claimed that high coating volumes should be considered for low concentration samples, whereas low coating volumes for high concentration samples could increase the sampling efficiency [14,73]. Moreover, another study used polydymethyl siloxane (PDMS) coated glass wool fabric to increase the efficiency and sensitivity of TFME [73]. This study reported a 5% error rate in pollutant detection, compared to 8% in their previous work [73].

The TFME technique has become very appealing for analytical and bioanalytical applications due to its numerous advantages. This technique provides a completely new geometry, which significantly decreases the sampling preparation and analysis time. In a critical review of TFME coupled with mass spectrometry and liquid chromatography, Mirnaghi et al. (2013) mentioned that it is possible to measure the free and total concentrations of the pollutants or analytes present in a single sample matrix by applying proper calibration strategies [16]. The accuracy of the TFME has been reported to be way better than the other available conventional sampling techniques [16,17,72]. The TFME provides enhanced sensitivity, rapid sample preparation in both on-site and off-site applications, a thinner coating than the SPME or NTME, a high extraction rate, a plethora of geometry options, and direct extraction of analytes from complex matrices without the need for pretreatment of the sample (since it offers an open-bed system) [16,71].

Over the significant number of advantages, TFME has some weaknesses [16,65]. In a comprehensive review study, Jiang et al. (2012) reported that the reusability of the coating used in the TFME is quite difficult, which is a major drawback. In addition, TFME is not fully automated so that it requires a thermal desorption-cooling interface for the execution of efficient thermal desorption and reconcentration of pollutants/analytes [73].

3.4. Solid-phase dynamic extraction

Solid-phase dynamic extraction (SPDE) is a relatively new sampling technique used in conjunction with GC-mass spectrometry to analyze VOCs, MVOCs, and other analytes in the sample matrix. This novel sampling technique is equally useable for the analysis of pollutant particles in air, water, and food matrices [31,61]. Moreover, SPDE is an advanced extension of SPME which has been developed to overcome some of the drawbacks of SPME. The primary principles of the SPDE are almost identical to SPME except the sample injection tool. A polymer coated (e.g., 50 μm PDMS) steel needle is used in SPDE rather than just a fiber [65,66] as shown in Fig. 7b [74]. Jochmann et al. (2006) reported that four different kinds of SPDE needles are commercially available on the market [31]. The sample matrix is collected in an ampule vial, and a coated SPDE needle is placed inside the ampule. The analytes are concentrated on the needle surface coated with PDMS of a gas-tight syringe. It is worth noting that the

Table 2. Summary information of advanced air sampling techniques including pros and cons, accuracy and challenges of described techniques.

Technique	Pollutant type	Advantages	Disadvantages	Accuracy	Challenges	Ref.
NTME	VOCs, MVOCs	i Requires small quantities of sample. ii User friendly, cost-effective and no humidity effect.	i Low sensitivity towards small sample size.	i High accuracy (>90%) with less error percentage.	i Sophisticated and inappropriate handling could generate high error.	[15,32,51]
SPME	Odorous and livestock gases,	i short sampling preparation duration. ii Works finely with small sample volume.	i Needle could be break during sample agitation. ii Less flexibility	i High precision with less standard deviation.	i Coating thickness degrading. ii Maintenance of needle quality.	[31,55,56,58,62]
TFME	Naphthalene ($C_{10}H_8$), fluorene, $C_{14}H_{10}$, $C_{16}H_{10}$, $C_{12}H_{10}$, $C_{16}H_{10}$	i Solvent-free & inexpensive. ii High extraction efficiency & sensitivity. iii Coupled with mass spectrometry & chromatography.	i . non-reusability of needle coating. ii Not fully automated. iii Cooling down process requires thermal desorption.	Detection limits up to 19 pg/mL depending on the types of analytes.	i . Optimization of surface area-to- volume ratio. ii Membrane materials improvement. iii Uniform coating thickness.	[53,59,63,64]
SPDE	VOCs, MVOCs, Polar volatile organic compounds (PVOCs), alcohols.	i . Stainless steel needle provides more accuracy. ii Thermal desorption technique using.	i Chemical desorption is not available. ii Bar code reader facilities are absent.	i High analytical efficiency (>90%) for sorption and solvent-free extraction	i Maintaining internal coating of needle is tough enough. ii Maintain constant desorption rate.	[31,16,65,66]
SBSE	Organic solid, liquid, gaseous particles- C_7H_8O, C_6H_6O, $C_8H_{14}ClN_5$, $C_{10}H_8$, $C_{16}H_{23}N_3OS$	i Magnetic stirring rod use ii Low sample loss and high accuracy. iii Rapid sample preparation	i Limited range of air pollutants to extract. ii Twisters are inadequate.	≥90% pollutants could be extracted over SPME	i Using polymeric stir bar. ii Maintaining stir bar quality and coating thickness.	[23,67,68,51,69]
ITEX	VOCs, halogenated hydrocarbons, BTEX, gasoline, oxygenates.	i Offers wide range of autosamplers. ii High extraction, sensitivity & recovery rate.	i Designed for chemical desorption. ii Multiple sampling session is unavailable.	≥90–98% Provides highest accuracy.	i Maintain needle diameter. ii Tough to control.	[15,34,70]

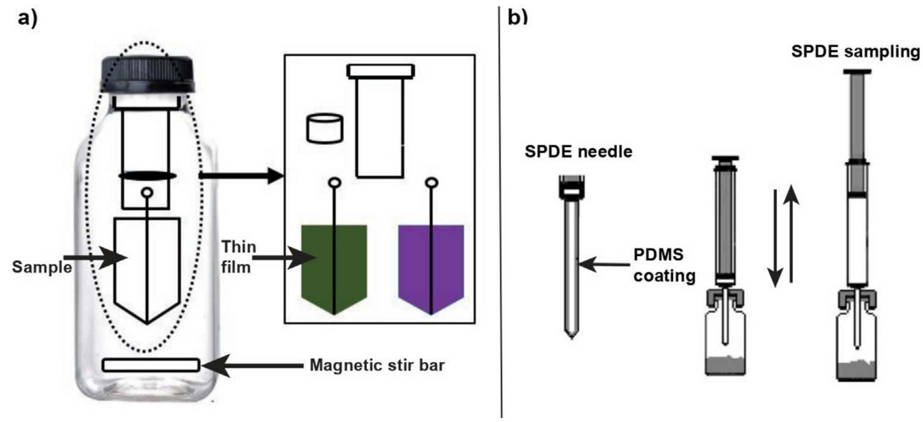

Fig. 7. The typical experimental setup of — a) TFME [65], and b) SPDE [74]. In the case of SPDE, Poly(dimethyl siloxane) which is widely known as PDMS is used as polymer coating on needle to adsorb analytes from air sample.

sample volume should be a specific amount for GC- or GC-MS-controlled SPDE. The sample matrix injects into the GC or GC−MS by pulling in and pushing out the plunger of a gas-tight syringe where the needle should be connected to the syringe. Thermal desorption is used to recover the analytes, which are then transferred to an injector body for further analysis [16,75]. Castro et al. (2015) performed a study to optimize the volatile pollutants in a gaseous liquid sample using the headspace-SPDE (HS-SPDE) method, which could isolate a wide range of pollutants (i.e., aliphatic esters, alicyclic compounds, sulfur compounds, etc.). The pollutant detection rate of this study was 95%. Another comparative study reported that the SPDE has high sensitivity to isolate pollutants with a low error percentage (3−4%) [74].

SPDE has a wide range of advantages, including high sorption capacity due to a sufficient amount of stationary phase, a faster extraction rate due to continuous pumping, mechanical stability along with a high diffusion rate, high accuracy in an analytical lab, and a high recovery rate due to its sampling condition [16,61,76]. In addition, SPDE could be applied for the detection of wide range of analytes including halogenated pesticides, chlorinated hydrocarbons, drugs, and different food matrices reported elsewhere [61]. Despite all the advantages mentioned above, it is worth mentioning that SPDE has several minor drawbacks. For instance, chemical desorption, two axes automation and bar code reader facilities are absent in SPDE sampling technique [59].

3.5. Stir bar sorptive extraction

Accuracy and efficiency are nonnegotiable terms for a sampling technique to minimize cost and effort related to analyses. The stir-bar sorptive extraction (SBSE) is one of the best sampling techniques to analyze gaseous, liquid, and solid sample matrices. In an effort to develop a new type of air sampling technique this technique was invented back in 1999. The SBSE is another modification of SPME and their basic working principles are identical [23]. The principle of this technique relies on sorption extraction, whereby the analytes or pollutants are extracted into a magnetic stirring rod [39,61,66]. The extraction of analytes is performed by maintaining a phase ratio between the polymer coating and the sample matrix. The widely used polymer coating is polydimethylsiloxane (PDMS). Baltussen et al. (1999) found a 10−40 mm stir bar with a polymer coating of 55−219 μm thickness to be a good selection [67]. The stir rod is directly placed inside the sample and the analytes are trapped on the polymer coating. In the next step, the analytes are recovered by a thermal desorption mechanism (e.g., Gerstel TDS-2) as shown in Fig. 8a [68,67].

The recovery rate in SBSE has been reported higher than the typical SPME technique [15,68,75]. Another study found that when PDMS was used, SPDE had a higher analyte recovery and extraction rate than SPME, ranging from 46% to 70% [67]. SPDE was used to trace element analysis by [23] and they also reported higher detection and recovery rate of pollutants in the case of SBSE in compare with SPME. In addition, the SBSE offers a rapid and simple sample preparation, solvent free and compatible with modern extraction techniques such as GC, HPLC, and CE [16,40]. Over its many advantages, this technique has a few drawbacks including limited range of analyte detection and less availability of sorbent materials [51].

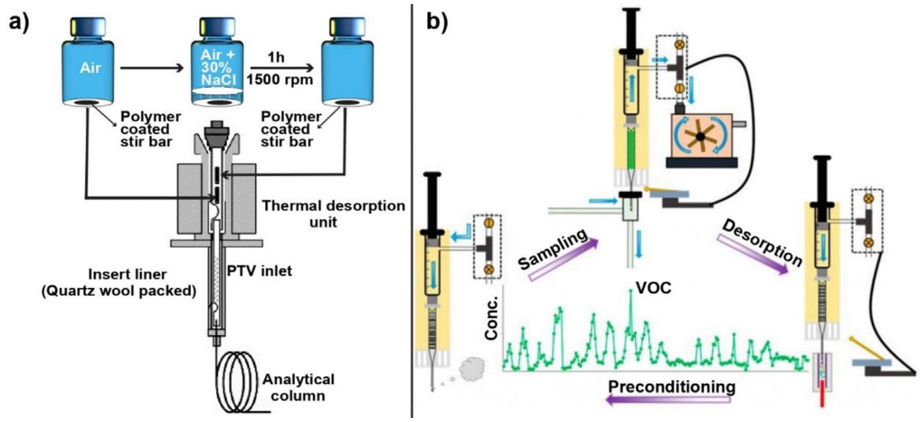

Fig. 8. The schematic diagram of two advanced air sampling techniques — a) SBSE (redrawn from the reference [68]), and b) ITEX (redrawn from the reference [77]) for trapping air and further steps for the laboratory analyses.

3.6. In-tube extraction

In-tube extraction (ITEX) as shown in Fig. 8b [77] is the most recent sampling technique among all other microextraction methods [15]. This micro-extraction technique is completely automated and offers wide variety of auto sampling [15,70]. A gas tight syringe with a special type of stainless steel needle coated with sorbent materials is used in ITEX technique [15]. Jochmann et al. (2007) described that the stainless-steel needle is divided into two parts. The lower part of the needle contains an ordinary needle cannula with a side hole for septum penetration into the GC or GC–MS inlet, whereas the upper part of the ITEX needle is a tube with a large diameter packed with sorbent materials [14,15]. The gas-tight syringe as well as the sorbent tube are surrounded by an electric heater and fan to resist the sample condensation and assist thermal desorption at the inlet of the GC or GC–MS system to collect analytes [40,41]. Lan et al. (2020) reported that the ITEX system itself enables an independent desorption temperature from injector temperature to keep the GC inlet unoccupied during the movement of syringe plunger and maintains a fixed desorption flow rate into GC inlet. In another study the former author reported a highest extraction rate (>98%) with high precision using fully automated ITEX-GC/MS system [77]. In another study, Jochmann et al. (2007) reported six folds efficiency to isolate volatile organic compounds from sample using ITEX [78].

The ITEX offers a wide range of advantages over the other sampling techniques including less sample preparation time, circumvention of unfavorable extraction and injection conditions, high extraction rate and a wide variety of sorbent materials [34,40,41]. Among some drawbacks, this newest technique is only designed for the thermal desorption rather than chemical desorption, is unable to perform multiple sampling session at the same time [59], and is not a good choice for breath air sampling [15].

3.7. Sensor technology in air sampling

Due to the excessive spread of electronics industry globally, sensor-based air sampling technology becomes popular over the past few years. The reason of its simplicity, handy, user friendly, very low error margin and cost-effectiveness. There are wide range of sensors are using for air sampling including gas sensors and liquid-phase sensors [79]. The gas sensors uses for trapping all types of gaseous and aerosol pollutant whereas liquid-phase sensors could trap the air pollutant bigger than the gaseous particulates, for instance no-volatile material [80,81]. A receptor collects the air sample, and this receptor should be connected to a transducer. This combination of receptor and transducer is connected to a sensor and another measurement electronic device is automatically read the sensor data according to the user defined program. The collected data from the electronic device is analyses in the later part to determine the actual percentage of pollutants in a specific air sample. The sampling accuracy is more than 98% using the sensor-based technology with suffering from less drawbacks.

Sensor based air sampling is very useful for the area under out of electricity coverage or low coverage. Direct current (DC) or solar energy could be the alternative source of electricity to run the device contains the sensor for air sampling. A typical setup for air sampling and monitoring using smart sensor technology is displayed in Fig. 9 [82]. The major drawback of this system is expensive and sophisticated experimental design.

4. Future directions

Air pollution is increasing with an alarming upward slope and severe effect on the environment. Thus, a highly efficient air sampling technique is a must to analyze air sample to keep our environment clean and healthy. With the conventional techniques, many of them are not fully automated hence the error percentage is typically high. In addition, the sampling parameters should be a potential area for future research. Some sampling techniques are sophisticated in terms of sampling duration, temperature, and sample flow rate whereas some are prone to instrumental problem, sample loss, and less accuracy. However, proper combinations of sensitivity, sampling volume, sampling accuracy and retention time, and pollutant extraction rate could lead to the development of a hybrid technique such as MET-GC, and MET-GCMS.

Also, there are plenty of techniques that are on the track toward modification, as discussed in earlier sections. In terms of advanced sampling techniques, plenty of parameters (e.g., high accuracy, sensitivity and extraction rate) have been improved. It is worth noting that the techniques are sensitive to the types of pollutant particles, extraction rates, and types of polymer coating. The use of polymer coating in the microextraction technique could be a potential area of research to develop a new air sampling technique. Since the crossover technologies between conventional and advanced techniques have been widely accepted, the future of sampling techniques is claimed to be quite promising. For instance, there is the development of a brand-new sampling technique with an on-site detection kit to reduce the duration of laboratory analyses. A recent development has introduced the simplest air sampling technique to trap airborne particulate by passive deposition of pollutants on a petroleum jelly-coated microscope slide. In addition, it is now well known that the TD-GC or GC–MS techniques have several potential drawbacks such as the shifting of retention times, variable detector response, and masking of high-volatility compounds. A new system with the combination of small sample volumes and a high sensitivity Time-of-Flight MS could resolve these issues [40]. Recently, new sorbent materials are being made available that use microextraction techniques to enhance the sample analysis process for a wide range of applications. The thermal desorption technique in association with GC, GC–MS, and GC-olfactory-MS needs further research to enhance the maximum accuracy of sample analysis. A high-volume air sampler offers a wide range of advantages, including a high air flow rate at a low pressure drop, high particle storage capacity, and low moisture recovery. However, further research should be conducted to discover why this technique cannot detect all chemical pollutants other than sulfates and nitrates [19]. A smart sensor-based technology resolves a wide range of issues facing the earlier mentioned sampling techniques. Though smart sensors are still expensive and having limited availability in many countries. Further research needs to be done to minimize the cost of this state-of-the-art technology to sustainable improvement in the air quality monitoring.

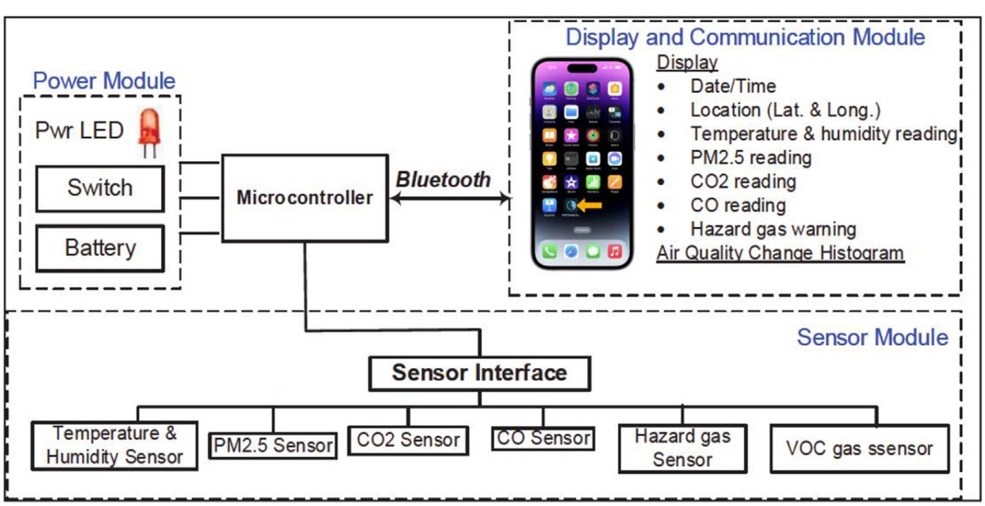

Fig. 9. An advanced system of sensor-based air quality monitoring setup; A Power module is connected to a digital microcontroller which could be controlled by any Wi-Fi/Bluetooth active digital device which could uses for sensor module to collect air sample and further analyses to determine the pollutant percentage in the air sample. The figure is slightly edited from the reference [82].

5. Conclusions

This study discussed an extensive comparison between conventional and advanced techniques of air sampling with pointing out the potential improvement needed to maximize the performance of each technique. The conventional air sampling could be useful for the rural areas and the area far from locality due to relatively less concentration of pollutants particles than urban area. In addition, these techniques are comparatively less expensive, handy, and user friendly. However, this air sampling techniques suffering from several drawbacks including low sensitivity, accuracy and low pollutant extraction rate. The MET shows a several fold higher pollutant extraction percentages than the conventional air sampling techniques. These techniques also owing to a few numbers of disadvantages including the coating quality of the syringe, metal part of experimental setup suffering from corrosion and not fully automated. However, MET coupled with GC, GC−MS or HPLC based hybrid techniques could significantly increase the sampling accuracy and fast analyses. Furthermore, hybrid techniques have shown excellent sensitivity, linearity, detection capacity, rapid sample preparation, and high extraction rate which maximize the laboratory accuracy over the conventional techniques. These new technologies offer smaller volume of air samples in combination with powerful detection techniques, such as time-of-flight mass spectrometry, ITEX, SBSE or SPME. Finally, we also investigate the sensor-based air sampling techniques which increased the sampling accuracy, time and sensitive significantly. This technique offers a digitally controlled automated system to continuously monitor the air quality in both indoor and outdoor air. However, more effort is required to overcome the drawbacks of the currently available sampling techniques and therefore, need more investigation to accomplish further improvement.

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