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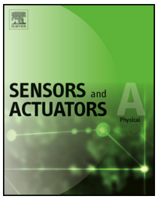

Review

Advanced vapour sensing materials: Existing and latent to acoustic wave sensors for VOCs detection as the potential exhaled breath biomarkers for lung cancer

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ABSTRACT

Lung cancer is the leading cause of death worldwide and has a significant impact on public health across society. Among all types of cancer, lung cancer is typically silent and it is commonly diagnosed at a later stage where treatment is rarely achievable. There is an urgent need for the development of the early diagnosis of lung cancer for an improved survival rate. Preliminary research shows that lung cancer is accompanied by increased oxidative stress which generates volatile organic compounds (VOCs). Hence, breath analysis offers the most promising solution for the early diagnosis of lung cancer as it is non-invasive and radiation free. Potential VOCs biomarkers in exhaled breath associated with oxidative stress and lipid peroxidation have been discussed to provide a quick approach to the diagnosis of lung cancer. Although gas chromatography–mass spectroscopy (GC–MS) able to analyze the VOCs biomarker, it is bulky, high cost, required expertise to handle and consumes a lot of time. Hence, the sensor-based technique provides the solution to overcome the limitation. Recently, acoustic wave sensors such as quartz crystal microbalance (QCM) and surface acoustic wave sensors (SAW) have been used to identify the presence of VOCs in various applications. This is due to its high selectivity, good reproducibility, and fast response sensing materials. The selection of vapour sensing materials plays a crucial role in developing a highly sensitive and selective and fast response acoustic wave sensors. For this purpose, various types of sensing layers from metal oxides, polymers, biopolymers and composites have been studied. We present a critical review of advanced vapour sensing materials that are primarily used in acoustic wave sensors in identifying the presence of various VOCs. Criteria to evaluate the performance of the acoustic wave sensors such as resonance frequency and sensitivity are also discussed.

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1. Introduction

Cancer is the leading cause of death in a society and many issues related with it have a significant impact on public health across society. According to the 2018 report from Global Lung Cancer Coalition, the lung cancer accounted for 13.3 deaths per 100,000 population in Malaysia or 4057 number of deaths from 4686 number of lung cancer diagnosed [1]. This statistical statement is indicating only about 16–17% of the lung cancer patients, survived. This information is tally to the statistical analysis done by the World Health Organisation (WHO) in 2018 where Asia portrayed the highest number of cancer cases by approximately 48 % of the 18.1 million cases worldwide with 70 % of death from lung cancer occurs in the low and middle income countries [2]. Several techniques that are currently in used for lung cancer detection including chest x-ray, computed tomography (CT) scan, fluorodeoxyglucose-positron emission tomography (FDG-PET), bronchoscopy, and lung biopsy [3]. However, these detection techniques are expensive, invasive, time consuming [4], bulky [5], required expertise to handle it [6–8] and relatively large [9]. Besides, positron emission tomography (PET) is a current practice for lung cancer detection. Yet, the screening cost is very high [10]. There is an increasing concern that the early detection of lung cancer can increase the number of lung cancer survival. Exhaled breath analysis has been introduced for early lung cancer diagnosis based on VOCs biomarkers. Detection of disease using exhaled breath is becoming an increasingly extensive non-invasive diagnostic tool to assess information on the health status. By measuring the concentration of endogenous volatile organic compound VOCs, it allows real time alternative for early diagnosis of lung cancer [11]. In recent studies, some common chemicals recognized as VOC biomarkers of lung cancer are styrene, decane, isoprene, benzene, 1- hexane and propyl benzene [11]. Studies showed VOCs from breath samples have high potential for rapid and non-invasive screening and disease detection [12]. Breath analysis has the advantages of being non- invasive, highly sensitive, simple and potentially cheap [13]. This benefit strengthens the need of having an alternative measure of a non-intrusive, portable and effective sensor for lung cancer. Moreover, there are several technology in gas analysis field; chemo-resistive device, gas chromatography- mass spectrometry (GC–MS) [14], as well as the sensor based diagnosis which is currently still developing. Although gas chromatography - mass spectroscopy (GC–MS) able to analyse the VOCs biomarker, it is bulky, high cost, required expertise and consume a lot of time. Hence, sensor-based technique provides the solution to overcome the limitation. Gas sensing devices can detect and identify the presence of VOCs biomarkers for early detection of lung cancer. Current sensing techniques introduced for breath analysis are surface acoustic wave (SAW) sensor [15–18], quartz crystal microbalance (QCM) sensor [19,20], optical sensor, gold nanoparticle (GNP) sensor [21], colorimetric sensor array [22], metal oxide sensor [23] and conducting polymer composited (CPC) sensor [13]. Among them, the acoustic wave sensor such as QCM and SAW sensors are known to have several advantages such as high sensitivity, fast response and good stability [24]. However, despite the versatility of using QCM and SAW sensor in vapour recognition purposes, a critical issue on the sensing materials used still needs to be investigated further.

The issue arises from the current innovated acoustic wave sensors that are at high risk in misinterpreting the frequency change due to the variation in mass density, mechanical stiffness, viscosity, electric and dielectric properties [25]. The reproducibility and the reliability of the sensors are mainly depending on the homogeneity of the sensing layers and detection limits. This is due to the conventional sensing materials present such as metal oxide, and carbon nanotubes on their own are still lacking in providing the finest modification to the acoustic wave sensors for a thorough vapour recognition. Hence, further work is required to cater these challenges. One of the strategies to overcome the drawback in the application of acoustic wave sensors is through the use of alternative sensing materials. There are numerous types of sensing materials with biopolymer that have attracted the attention of recent research work. Therefore, the aim of this paper is to review the vapour sensing materials using acoustic wave sensor for VOCs detection as the potential exhaled breath biomarkers for early detection of lung cancer.

The state of art of existing and various types of sensing materials for acoustic wave sensor such as metal oxides, polymers and composites will be highlighted in this paper. This solution is more effective in way that it is making the sensors to be more sensitive, with faster response time and high mobility compared to the traditional material. Criteria to evaluate the performance of the gas sensors in terms of resonance frequency and sensitivity are also outlined.

This paper is organized into sections where the following section (Section 2) describes and explains the significance of breath analysis for lung cancer detection and the biomarkers involved. Section 3 focuses on the use of acoustic wave sensors, with the basic principle of piezoelectricity along with the related work on biopolymer based QCM sensor whilst Section 4 discusses the findings from the work mentioned throughout the review.

2. Breath analysis

Breath analysis is a method of gaining information on the clinical state of an individual by monitoring the volatile organic compounds (VOCs) present in the exhaled breath. In extensive definition, VOCs are of carbon-based compounds in which is readily evaporated at room temperature. In medical purposes, VOCs are a cocktail of organic compounds present in the content of human breath to be used as the biomarkers [26]. For instance, acetone, a VOC, is highly related to diabetes detection. Patients with diabetes are more likely to have greater percentage of acetone in comparison to a healthy person [26]. The patients with lung cancer undergo VOCs alteration in their body system. Such alteration may cause the occurrence of oxidative stress from the presence of free radicals and reactive oxygen species (ROS), gene mutation or rearrangement, Warburg effect and apoptosis [27].

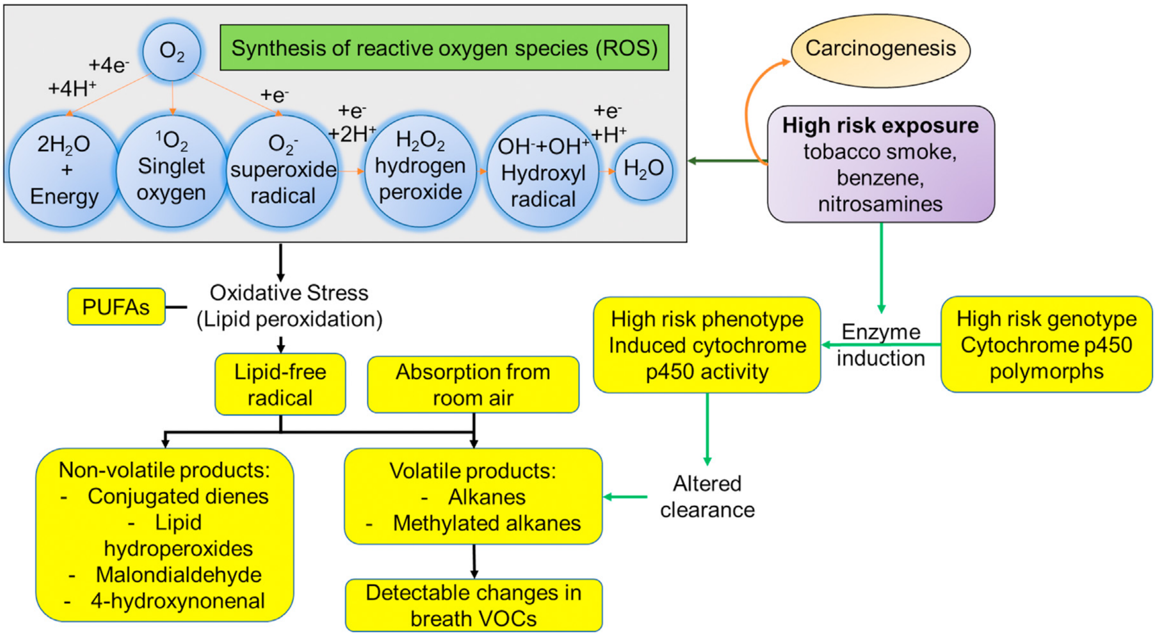

Fig. 1. The hypothetical basis for exhaled breath test and production of VOCs in the exhaled breath associated to oxidative stress, lipid peroxidation and cancer cell alteration due to high exposure to carcinogenic substances [30].

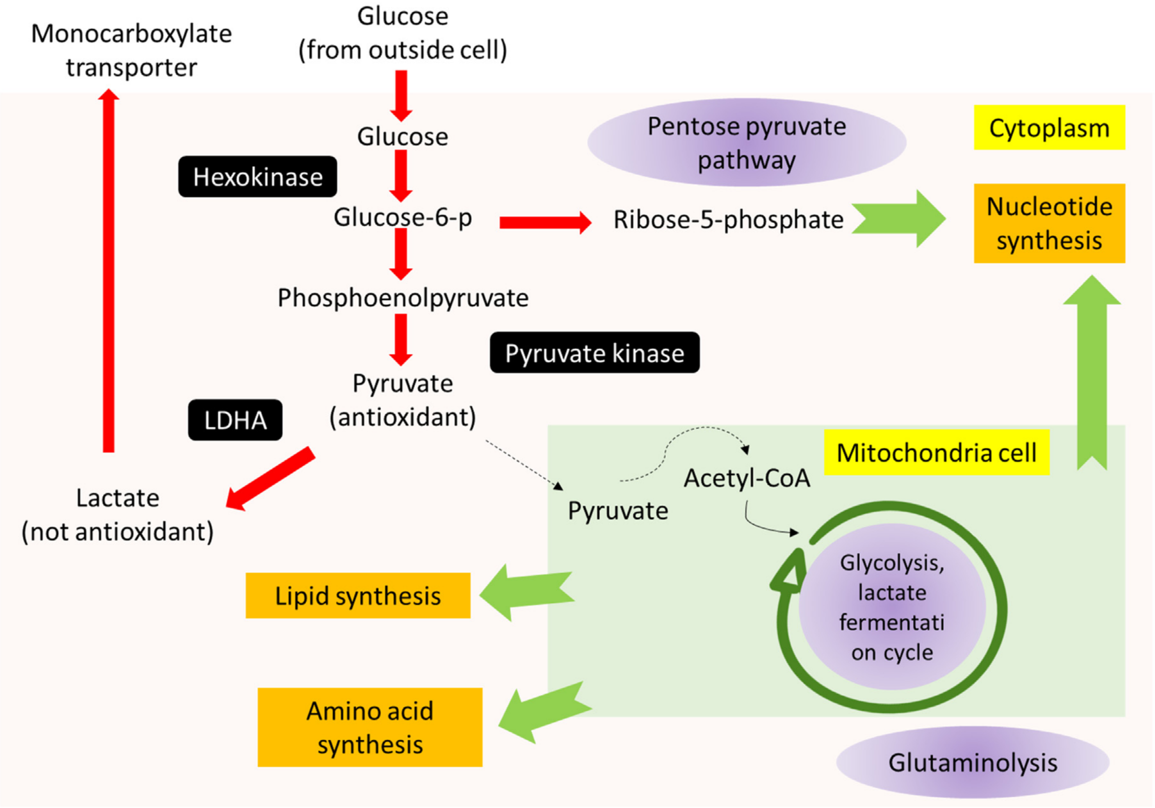

Fig. 2. Illustration of Warburg effect on a typical mammalian cell [37].

A. Amann et al. highlighted the effect of oxidative stress on the rapid growth of cancerous cells in lung [28,29]. People with cancer, tend to have ROS in excess leading to oxidative stress. Oxidative stress will cause lipid peroxidation resulting in the alteration of VOCs which is then secreted through the exhaled breath. Fig. 1 summarises the hypothetical basis for exhaled breath test and production of VOCs in the exhaled breath associated to oxidative stress and lipid peroxidation [30]. The VOCs that are commonly associated to oxidative stress and lipid peroxidation are VOCs with hydrocarbons; decane, and heptane as well as aldehydes; formaldehyde and hexanal [31]. Aldehydes can be both endogenous and exogenous sources. Exogenous source occurred when humans are exposed to formaldehydes from household products made up from woods, cosmetic, engines and cigarette smoke or second-hand-smokers

Table 1
Summary of the lung cancer biomarkers from previous work.

Author(s)	VOCs	VOCs functional groups
(Jia et al.,2019) [38]	Propanol	Alcohol
	Isoprene, pentane,	Alkanes/ alkenes
	Acetone	Ketone
	Hexanal	Aldehydes
	Toluene, benzene, ethyl benzene	Aromatics
(Lawal & Wallace, 2014) [140]	Acetone	Ketone
	ethanol, 3-methyl-1butanol	Alcohol
(Crucitti, Longo, Rocco, & Rocco, 2018) [141]	2-butanone	Ketone
	1-propanol	Alcohol
	Isoprene	Alkene
	Ethylbenzene, styrene	Aromatics
	Hexanal	Aldehyde
(Masuda, Kato, Kida, & Otsuka, 2019) [78]	Acetone	Ketone
	Butanal	
	Formaldehyde	Aldehydes
	Acetaldehyde	

(SHS) [32]. Meanwhile, formaldehydes and a few other aldehydes for instance, hexanal, heptanal, acetaldehyde and propanal may be obtained endogenously as the by-products of alcohol metabolism by an enzyme known as alcohol dehydrogenase (ADH) [33,34]. Aside from the ROS effect, high exposure to carcinogenic gases may also increase the risk of enzyme induction which will then induces the alteration of cancerous cell. Some common carcinogenic gases are tobacco smoke, benzene, and nitrosamine [30]. Warburg effect related to cancer disease is briefly defined as the overdriven anaerobic glycolysis of the dysfunctional mitochondria [35,36]. It will either cause cancer cells development or rapid growth of the cancerous cells existing in the body. Fig. 2 illustrates the anaerobic glucose metabolism or Warburg effect. Depending on the situation, the biomarkers for a lung cancer patient may differ as VOCs can be obtained from both exogenous and endogenous sources. Lung cancer patients who smoked are most likely to have greater aromatic compounds such as benzene and toluene as compared to a non-smoking patient, in which the endogenous VOCs may be a much more reliable biomarker.

VOCs can be detected in the headspace of lung cancer cells blood vessels and exhaled breath. It has been classified in six different groups; (i) alkanes and alkenes, (ii) alcohols, (iii) aldehydes, (iv) ketones, (v) nitriles and (vi) aromatics [21,31,38,39–41]. There are over 20 biomarkers for lung cancer analysed by Thriumani et al. [14]. Other literature cited that ethane, pentane, aldehydes, isopropane, and isoprene [6] are also enlisted as biomarkers.

From previous work, the identification of biomarkers for lung cancer was challenging and each work claimed to have different number of biomarkers. There is inadequate information to confirm the biomarkers for lung cancer. It is because VOC profiles are influenced by the retention of VOCs in the lungs aside from the error in breath sampling; contaminations from the collection system, expiratory flow rate, humidity and study design; diet, lifestyle, age, medication and disease stage [30]. Haick et al., highlighted that 115 reliable gases were reported as exhaled breath biomarkers [30]. Current technique used to identify the biomarkers is the gas chromatography- mass spectrometry (GCMS) [14]. This technique produces a list of molecules present in the sample. However, this detection technique is expensive, time consuming [42], not portable, required expertise to handle it [6] and relatively bulky [9]. The GCMS technique may inhibit the accessibility for most individuals to get diagnose at the early stage. Recently, QCM and SAW have been used to identify the presence of VOCs in various applications. This is due to its high selectivity, good reproducibility and fast response sensing materials. The identification of lung cancer biomarkers based on previous work are summarised in Table 1 and classified into the chemical functional groups it belongs.

3. Acoustic wave sensor design and principle

There are several methods for VOC sensing such as solid-state sensing method, optical, electrochemical and acoustic wave method based on mass-sensitive sensor as shown in Fig. 3. Mass sensitive sensor-based techniques includes the use of QCM and SAW will be further discussed throughout this paper.

QCM and SAW sensors are both categorised as mass sensitive sensor based on piezoelectricity transduction. The principle of piezoelectricity is applied as the sensing mechanism [43]. Piezoelectricity is defined as the ability of some piezoelectric materials to generate electrical energy when mechanical stress is applied or vice versa. The conversion of mechanical stress to electrical energy is meant to be the direct piezoelectric effect while the conversion of electrical energy to mechanical energy is known as the converse piezoelectric effect [43]. There are two types of piezoelectric crystal sensors: (i) the bulk acoustic wave (BAW) (ii) surface acoustic wave (SAW). QCM sensor is categorised as bulk acoustic wave device. The alternating electric field applied over the electrodes of the QCM sensor leads to the oscillation of the quartz crystal substrate and the transverse acoustic wave propagates through the quartz substrate. The sensor measurement is represented by frequency shifts due to the change in mass and velocity of the wave propagating through the quartz crystal substrates. Fig. 4(a) illustrates the sensing mechanism of QCM towards analyte gas. The working principle of QCM is that it converts the electrical energy to mechanical energy (acoustic wave) and reconverted at to electrical energy as the output measured. When voltage is applied to the top electrode of the QCM, the acoustic wave will be generated and propagated through the quartz piezoelectric substrate. The acoustic wave will be converted back to electrical signal as the output measured in terms of resonance frequency. For sensing purpose, the QCM will be coated with a chosen sensing layer to adsorb the presence of analyte gas. The interaction of the sensing layer with the presence of the analyte gas will cause changes in the wave amplitude and velocity due to the

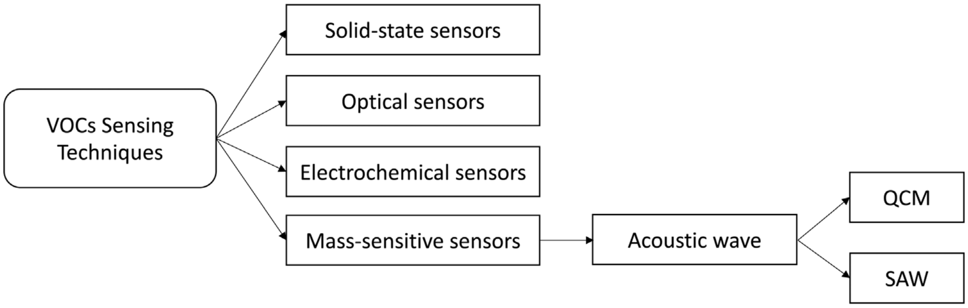

Fig. 3. Several methods in sensing VOCs.

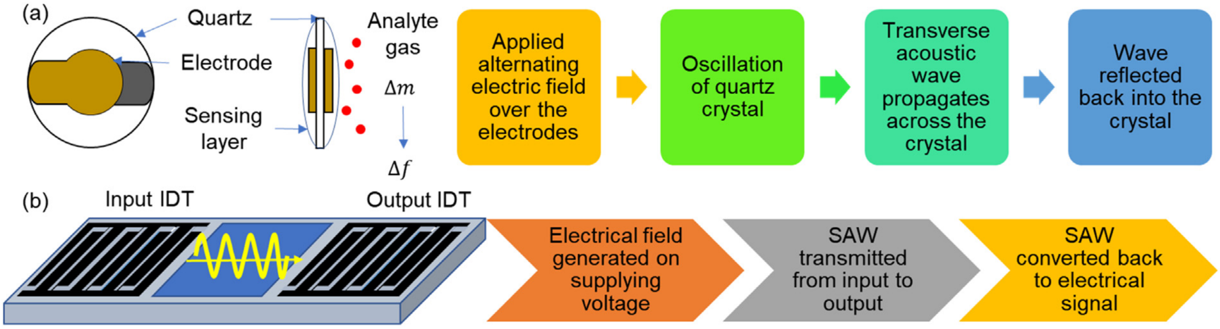

Fig. 4. Sensing mechanism for detection using (a) QCM sensor and (b) SAW sensor.

Table 2
Summary of the key parameters for both QCM and SAW sensors.

Sensor	QCM	SAW
Resonant frequency	$f = \frac{v}{2d}$	$f = \frac{v}{\lambda}$
Frequency shift	$\Delta f = -\frac{2f_0^2 \Delta m}{A\sqrt{\rho\mu}}$	$\Delta f = -C_m f_o^2 h' \Delta m$
Sensitivity	$S = \frac{\Delta f}{\Delta m}$	

mass variations [44,45]. Hence the shift of the resonance frequency will be observed due to the mass variations [43,44].

SAW sensor works slightly different from QCM sensor in terms of the propagation of the acoustic wave. The acoustic wave for SAW sensor propagates on the surface of the substrate instead. When an input radio frequency voltage is applied across the interdigitated transmitter (IDT), it induces deformations in the piezoelectric substrate and give rise to a Rayleigh surface wave confined within one acoustic wavelength on the surface [46]. As the wave transverses the gap between two arrays of the IDTs and it reaches the receiver IDTs, the mechanical energy is converted back into radiofrequency as an output [45]. As there is a change in mass arising from a coating or when vapour is adsorbed on the surface, it yields in a frequency shift and allows the application in monitoring of vapour. Fig. 4(b) shows the sensing mechanism of SAW sensor towards analyte gas.

These parameters play an important role in analysing the frequency shifts; (i) resonant frequency, (ii) frequency shift using the Sauerbery's equation and (iii) sensitivity. QCM and SAW sensor both have different equations involved in providing the three key parameters. Hence, the key parameters are summarised in Table 2 where; f is the resonant frequency, v is the velocity of acoustic wave, λ is the wavelength between IDT, d is the thickness of crystal, Δf is the frequency shift, C_m is the mass sensitivity coefficient, h' is the thickness of sensitive layer, Δm is the change of mass density, f_o is the unperturbed frequency of oscillation, A is the active area of crystal, ρ is the density, μ is the shear modulus of quartz, and Δm is the change of mass density.

4. Vapour sensing material for acoustic wave sensors

The materials used as the sensing layer play a crucial role in defining the performance of the sensor towards the detection purpose. The importance of a sensing layer is to enhance the sensing mechanism. It is deposited onto the sensor via various methods such as drop casting [47–49], electrospinning [50–52], self-assemble monolayer [53–55], and sputtering [56–58].

Some of the commonly used sensing layers are oxide [24,58–63], carbon nanotubes [48,64–69], polymer [62,70,71] and metal-organic framework [49,72,73]. Metal oxide is known to have high thermal stability and sensitivity while carbon-based material has good mechanical and chemical stability aside from providing a large surface area. Besides, polymers offer high permeability and fast response. Polymer upholds the sorption - desorption phenomena in gas detection whereby these phenomenon are greatly influenced by the thickness and the physicochemical properties of the sensing layer such as the molecular mass and affinity [74]. The molecules of the analyte gas are adsorbed on to the sensor due to the presence of chemical interactions between the gas molecules and the surface of the sensing layer. Meanwhile, the desorption process needs to take into consideration of the binding energy of the analyte gas molecules and the sensing layer [25]. This work highlights the use of polymer in detecting VOCs. Recently, the use of biopolymer has called the attention of many researchers. Biopolymers are known for their biodegradable properties, hydrophilicity and ability to be functionalized in accordance to the analytes to be adsorbed. The following section also emphasises previous work on chitosan and pegylated lipopolymer as sensing materials. Fig. 5 summarises the types of sensing layer on acoustic wave gas sensor.

4.1. Metal oxides

Oxides are commonly co-existing with metals. The presence of at least one oxygen atom in a metal compound is called metal oxide. Metal oxide is known to be a selective sensing material. In brief, metal oxides undergo chemical and electronic sensitization to detect analyte molecules [51].

Zinc oxide (ZnO) is among the metal oxide that most often used for gas sensing purposes [63,75]. Fig. 6 shows the FESEM images of ZnO nanostructure grains [63]. The sensing of an analyte using oxide involves the oxidation reaction between the analyte gas and the sensing material. A study by Horrillo et al., focuses on the use of ZnO film fabricated onto the 99.50 MHz ST-cut SAW device in detecting nitrogen dioxide (NO_2). The experimental work has been done on various gas analytes at concentrations ranging from 400 ppb to 16 ppm. The measurement results depicted NO_2 at concentration 16 ppm gives the highest frequency shift [58]. The selectivity towards $NO_{2,}$ hydrogen, methane, carbon monoxide and acetone were tested separately [58]. The results proved that the ZnO layer was behaving selectively towards NO_2 according to the frequency shift observed for the gases tested. The frequency shift for NO_2 at 16 ppm is approximately 275 kHz as compared to hydrogen, methane, carbon monoxide and acetone were all with below 50 kHz shifts [58]. This work concluded that the use of metal oxide has the potential to be a selective sensing material.

Metal oxide has the advantage in terms of having different shapes in nanoscale. This includes the nanorods and colloid spheres [76]. Fig. 7 shows the illustration of adsorption on ZnO colloid spheres. Aside from ZnO, Tin dioxide (SnO_2), silicon dioxide (SiO_2) and titanium dioxide (TiO_2) are some other frequently used materials for sensing purposes including VOCs sensing [54,77–81]. Xie et al. and Zhu et al., both used QCM to detect humidity using ZnO colloid spheres and mesoporous SnO_2-$SiO_{2,}$ respectively. At 75 % relative humidity (RH), mesoporous SnO_2-SiO_2 attained a shorter response time of 14 s as to 167.7 s for ZnO colloid spheres. This

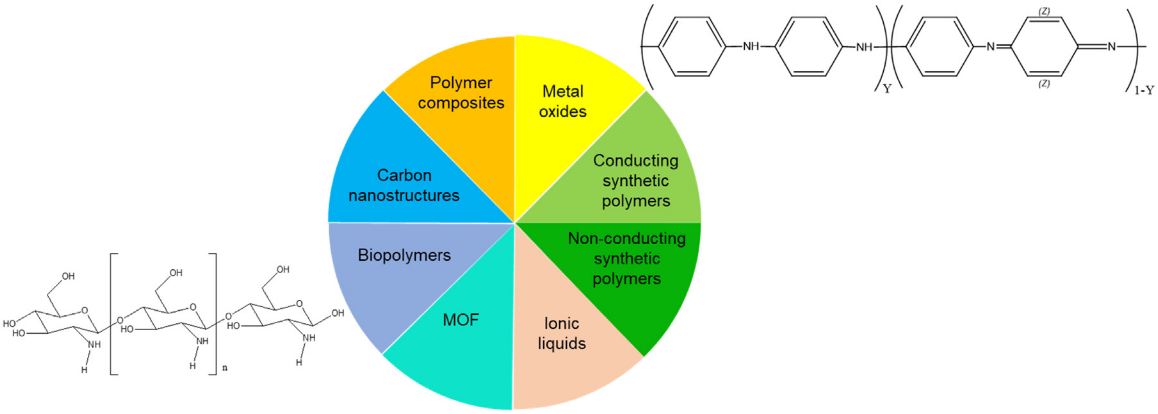

Fig. 5. Different type of sensing layer coated on acoustic wave gas sensors [45].

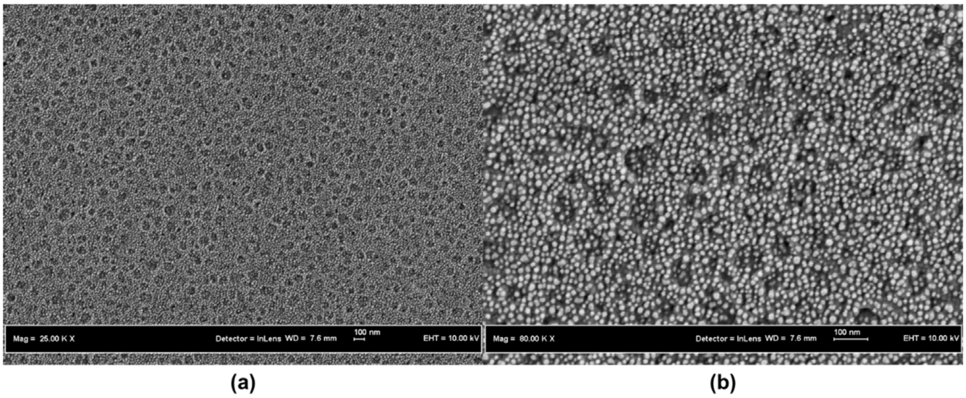

Fig. 6. The FESEM images of the ZnO nanostructure grains under magnification (a) x25k and (b) x80k.

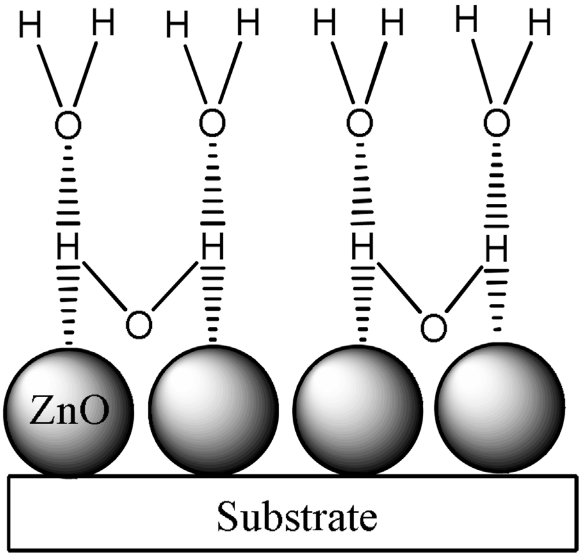

Fig. 7. Representation of adsorption model of water vapour on ZnO colloid spheres [76].

alludes to the existence of an ample amount of mesopores that has allowed the actions of forces of Van Der Waals and weak hydrogen bond between the water vapour molecules to SiO_2 compound. Besides, the addition of a semiconductor SnO_2 promotes the acceleration of response rate [79].

Apart from metals, semi-conducting metal graphene is also known as a quasi-metal that has been experimented as a sensing material. Sayago et al., has conducted a study on modifying graphene into graphene oxide in the application of any sensor. The study highlighted the formation of hydrogen bonding between graphene oxide and analyte gas which implies good sensitivity for sensor application [66]. In [66], graphene oxide was used to detect dichloroethane (DCE), dimethylacetamide (DMA), dimethyl methyl phosphonate (DMMP), and dipropylene glycol ether (DPGME) with a limit of detection 33.3 ppm, 4.34 ppm, 0.0097 ppm, and 0.039 ppm respectively. Thus, graphene oxide promoted the highest sensitivity towards DMMP detection at 3067 Hz/ppm. Despite the ability of oxides in improving sensor selectivity, it requires a relatively high working temperature to achieve a good response. The oxidation reaction puts the use of oxides at the disadvantage of being non-reproducible. Hence, oxide alone on a sensor requires further enhancements. Related works are summarised in Table 3.

4.2. Synthetic polymers

Polymer coatings provide a chemical interface for vapour sensing via sorption of the analytes [74]. The chemical interface is influenced by several factors including thickness of the polymer film and the physicochemical properties of the polymer to the analytes vapour; (i) affinity and (ii) molecular mass [74]. A study by Yadava et al., underlined that the activation energies are associated with the surface site, specifically for the gaseous adsorption and desorption on the solid surfaces [25]. Sorption and desorption phenomena occurring on the surface of the solid eventually create a sensitive area where the diffusion of the vapour molecules in and out of the polymer overlays and causes fluctuation in vapour loading due to the Brownian motion [25]. Fig. 8 illustrates the

Table 3

Summary of the previous work on oxides based coated sensors in detecting VOCs.

Design parameters								
Sensing material	Sensor	Target analytes	Analyte gas concentration	Frequency shift	Response time and sensitivity	Strengths	Limitations	References
ZnO thin film	99.50 MHz ST-cut quartz SAW resonator	NO_2	16 ppm	~275 kHz	–	Sensitivity over wide concentration range	Have were tested with only a few VOCs gas	[58]
		Hydrogen Methane Carbon monoxide Acetone		< 50 kHz < 50 kHz < 50 kHz < 50 kHz		Selective	High limit of detection	
ZnO – SiO_2 - Si	SAW sensor	Toluene	25 ppm to 100 ppm	~100 to 250 Hz (10 % O_2) ~50 to 200 Hz (15 % O_2)	~3 s	Short response and recovery time More sensitive towards polar molecules (toluene – low polarity, octane – non-polar)	Poor response to octane regardless of the concentration	[61]
		Octane		~50 to 75 Hz (10 % O_2) ~50 to 90 Hz (15 % O_2)				
ZnO colloid spheres	QCM	Water vapour	75 % RH	–	167.7 s (Response time at 75 % humidity) ~40 Hz/% (Sensitivity in at 75 % RH)	Increase in sensor sensitivity with increase in size of spheres – steric Short recovery time (8 s)	Less sensitive for analytes at lower concentration	[76]
SnO_2 – SiO_2	10^7 Hz AT-cut QCM resonator	Water vapour	Atomic ratio Sn/Si = 1:1 11 to 96.1 % RH	450 Hz (At 75 % RH)	14 s	Improved the sensitivity in conjunction to the SnO_2-SnO_2	Decrease in surface area decreases the sensitivity of the sensor. Dependency to the concentration of analyte	[79]
SiO_2	9MHz gold electrode QCM	Ammonia	1–200 ppm	–	~200 s	Linearity in concentration of analyte gas detection to the sensor response	Humidity and temperature greatly affect the reproducibility of the sensor and also the selectivity towards analyte gas	[54]
Graphene oxides	ST- cut quartz SH-SAW sensor	DCE DMA	33.3 ppm 4.34 ppm	–	0.9 Hz/ppm 6.9 Hz/ppm	Low detection limit	Slow response time Deposition of the material increases noise	[66]
		DMMP DPGME	0.0097 ppm 0.039 ppm		3067 Hz/ppm 760 Hz/ppm		High working temperature	

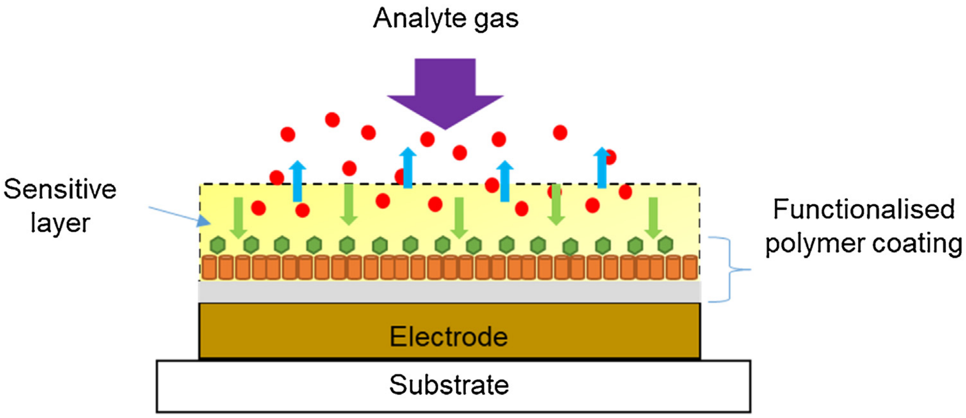

Fig. 8. Sorption-desorption phenomena on polymer coated sensor [25].

sorption-desorption phenomena. The rate of sorption on the surface of the solid is defined as the rate for the vapour molecules to stick or imprinting adhesive behaviour. The rate of desorption depends on the concentration and binding energy of the vapour molecules to the polymer film surface [25]. The study deduced that the polymers selected as the coating for SAW sensor coating must have weak chemical interactions with the analyte molecules for the sensor to operate reversibly [25].

Another study by Afzal et al., investigated the detection of different organic vapours on a coated SAW sensor with different polymers [46]. In addition, the authors examined the effect of the sensing film thickness on the sensing response. The difference in the functional groups of the vapours allowed the sensing response to be more selective. However, further investigation on the properties of the film and the analytes behaviours are required. More studies on VOCs detection have illustrated the capability of synthetic polymers in detecting VOCs [46,56,70,82–87].

Molecularly imprinted polymer (MIP) is also a commonly used method in the deposition of the sensing layer. Some materials such as a mixture of hexanal and (1:1,v/v) mixture of methanol and acetonitrile mixture uphold the ability to form into MIPs according to the properties of the substance [88]. The functionalisation of the MIP layer may provide an innovative path to synthesis a sensing layer with selectivity towards different gases. Experimental work has been done in detecting gas from ketone and ether group using hydrophobic MIP as the sensing layer on a QCM device alongside a few other gases [88]. From the results obtained, the hydrophobic MIP templated with hexanal sensing layer enhances the QCM device sensitivity at 3.11 Hz/ppm towards hexanal at 80 % RH more than other gases tested. Another experimental work by Bhattacharyya Banerjee et al., particularly focused on the detection of benzene and aromatic compounds [89]. The limit of detection attained for benzene, toluene and xylene were 0.98 ppm, 1.25 ppm and 1.42 ppm respectively. The results demonstrated that MIP was more sensitive towards aromatic compounds it has been templated to as benzene achieved the greatest sensitivity at 2.605 Hz/ppm. The aromatic compound was templated with 1,2,3-trimethoxybenzene which explains the selectivity from the use of nonanal gas detection in MIP synthesis previously [88]. The templated MIPs materials make the MIPs more selective and sensitive towards the analyte gas of the same functional group.

Aside from MIP, conducting polymer has a good potential in VOCs sensing application. Several research works have been done on polyaniline (PANI), both its emeraldine salt (ES) and emeraldine base (EB) [44,90]. Both works emphasised the use of 5 MHz QCM in detecting 5 ppm alcohol. Ayad and Torad claimed that thicker film promotes greater chance for hydrogen bonds with EB and thus, increase the sensitivity of the sensor. The study was extended to the use of protonated PANI (ES) thin film [90]. The extended study on PANI (ES) thin film illustrates the improvement in sensing towards the alcohols. The sensitivity of ethanol rises from 2.501 Hz/ppm to 20.7 Hz/ppm. It can be concluded from the study that protonated PANI film gives higher response and sensitivity towards the analyte [90]. The presence of H^+ ions in the protonated film allows greater molecules interaction. Related works on synthetic polymer-based sensor are summarised in Table 4.

4.3. Biopolymers

In the recent development, the use of biopolymer has attracted the attention of many researchers. Biopolymers are known for its biodegradable properties and its ability to be functionalised in accordance to the analytes to be adsorbed. Biopolymers are polymers made of biomolecules that are bonded covalently and obtained from natural sources such as carbohydrates, proteins and fats. Biopolymers are more complex than synthetic polymers in terms of structure and shape which are important for their particular functions [91]. The following section underlines the work on chitosan, cellulose and pegylated lipopolymer as sensing materials.

4.3.1. Chitosan based sensor

Chitosan is a naturally occurring polysaccharides polymer from various sources. There are two reactive paths for chitosan, which are via reaction with free amino groups and the hydroxyl group [57,92–94]. Chitosan is obtained from the deacetylation of chitin [57,95–103]. Fig. 9 represents the structural formula of chitosan. The advantage of using chitosan in the fabrication of thin film includes good mechanical strength, low cost, hydrophilicity and hydrophobicity [47,104,105] and being biodegradable [106–108]. The existence of piezoelectricity behaviour in chitosan marks good potential in using it as a sensing material on a piezoelectric sensor device [109].

Triyana et al., and Ayad et al. both utilised the chitosan as the sensing layer for volatile gases [47,111,112]. Ayad et al. have stepped up their work by incorporating PANI in the chitosan-based sensing layer to detect amines. Both researchers experimented the detection of alcohol using a QCM sensor. Chitosan-based sensing layer used by Triyana et al., showed a lower sensitivity of ethanol 0.4 Hz/mgL^{-1} [112] than the ethanol detection in [47] which is at 19.35 Hz/mgL^{-1}. This may be due to the presence of a self-assembled monolayer L-cysteine and a crosslinking agent that is glutaraldehyde to immobilised chitosan onto the QCM substrate [112] and the difference in measurement setup in which the resonance frequency of the QCM is altered. Chitosan has the tendency to attach and the capacity to be more adhesive with the presence of the self-assembled substance and the crosslinking agent that allows the

Table 4
Summary of the previous work on synthetic polymer coated sensors in detecting VOCs.

Design parameters			Analyte gas concentration	Frequency shift	Response time and sensitivity	Strengths	Limitations	References
Sensing material	Sensor	Target analytes						
Poly-vinylphenol (PVP), polystyrene (PS) and ethyl carbamate (EC)	433 MHz ST-quartz SAW	Propylene, ether, butanone, methylbenzene	–	200 kHz	~150 s	Use of array in detecting flammable vapours	Focuses only on aromatic compounds High resonance frequency	[46]
Polyepichlorhydrine (PECH)	ST-X cut quartz SAW delay line	Alcohol	1.25 mg/mL (PECH)	200 kHz	2 min	High sensitivity in detecting alcohol	The experiment was conducted at high concentration of alcohol only	[56]
Poly etheruretane (PEUT) Polybutadiene (PBD) Poly dimethylsiloxane (PDMS)			2.5 mg/mL (PEUT) 10 mg/mL (PDMS) 5 mg/mL (PBD)	300 kHz 400 kHz 500 kHz				
Poly(butyl methacrylate) PBMA	433 MHz STW	Octane	11.5 ppmv 22.9 ppmv 34.4 ppmv	40 kHz 70 kHz ~100 kHz	3 min	Less coating effort	Specifically built for long term measurement only	[82]
PECH Poly(cholorotrifluoro ethylene-co-vinylidene (PCFV)		Chloroform	2.3 ppmv 6.9 ppmv 11.6 ppmv	3 kHz 11 kHz 17.5 kHz				
L-grease PDMS Polyisobutylene (PIB)		Xylene	0.8 ppmv 1.5 ppmv 4.5 ppmv	8 kHz 19.5 kHz 50 kHz (all shifts were analysed based on PBMA)				
Polymethyl[3-(2-hydroxy)phenyl] siloxane (PMPS)	Single-port SAW resonator	Dimethyl methylphosphonate (DMMP)	5 – 40 ppm	35 – 130 kHz	30 s (response time) 3 kHz/ppm (sensitivity)	Fast response and reproducibility Low limit of detection	DMMP is not listed as one of the lung cancer biomarkers	[70]
PECH	ST-X cut Quartz SAW	Alcohol	–	260 kHz (PECH)	10 min	Present the ability of PEUT for great sensor response with a thickness at 150 nm coating	Not as sensitive as towards octane and toluene when using PDMS	[83]
PEUT PDMS	sensor			500 kHz (PEUT) 200 kHz (PDMS)				
PIB	SAW sensor array	Styrene, isoprene, hexanal, 1,2,4-trimethyl benzene, heptanal, benzene	–	–	–	Simple cleansing process using nitrogen carrier gas Low limit of detection	Spin-coat method made the thickness of film challenging to control	[84]
PEUT, PDMS	118MHz AT- cut quartz love wave device	Water vapour (humidity)	–	–	–	Implementation of automatic spraying system in controlling the deposition of polymeric layer	PEUT is less sensitive towards hydroxyl group as compared to PDMS	[85]
PIB	SAW delay line oscillator	Chloroform, chlorobenzene, o-Dichlorobenzene, n-heptane, toluene, n-Hexane and n-Octane	1 ppm	–	30 s	Multiple thickness of single polymer with potential in enhancing sensor performance	Challenging in classifying the analyte gas detected	[86]

Table 4 (*Continued*)

Design parameters			Analyte gas concentration	Frequency shift	Response time and sensitivity	Strengths	Limitations	References
Sensing material	Sensor	Target analytes						
PS PBD	9MHz AT-cut QCM	Toluene n-octane acetone ethanol	1000 ppm	PS; 60.0 Hz 18.0 Hz 6.2 Hz 3.1 Hz PBD; 69 Hz 72 Hz 2.2 Hz 2.9 Hz	20 s (response time) ~0.095 Hz/ppm (sensitivity - PBD)	Demonstrating the selectivity of analyte gases towards different polymer coatings	Works best at low temperature – lower than ambient	[87]
Hydrophobic MIP	AT-cut 8.98 ± 30k MHz QCM gold electrode	Hexanal Octanal Nonanal TMA NH_3 Ethanol Acetone Acetic acid Diethyl ether	25 ppm (LOD; 2 ppm)	89.89 Hz < 20 Hz < 20 Hz < 20 Hz < 20 Hz < 20 Hz < 20 Hz < 20 Hz < 20 Hz	42 s (80 % RH) 3.11 Hz/ppm	Improve selectivity towards VOCs at high humidity Encountered the sensitivity of QCM towards humidity	Low detection range in concentration	[88]
MIP (Drop coating)	10 MHz AT-cut QCM silver electrode	Benzene Toluene o-xylene m-xylene p-xylene	0.98 ppm 1.25 ppm 1.42 ppm 1.42 ppm 1.36 ppm	–	2.605 Hz/ppm 2.147 Hz/ppm 1.695 Hz/ppm 1.993 Hz/ppm 1.645 Hz/ppm	Low detection limit (0.98 ppm)	Favours to adsorption of benzene Sensor response depicted above 400 ppm	[89]
PANI emeraldine base (EB) thin film	5MHz QCM resonator AT-cut quartz	Ethanol Methanol 1-propanol 2-propanol	5 ppm	~ 60 Hz ~ 40 Hz ~ 100 Hz ~ 80 Hz	2.501 Hz/ppm (ethanol)	Low limit of detection (2 ppm)	Focuses only on alcohol detection	[90]
PANI emeraldine salt (ES) thin	5 MHz QCM AT-cut quartz	Ethanol Methanol 1-propanol 2-propanol	5 mgL^{-1} (120 nm film thickness)	~80 Hz ~120 Hz ~65 Hz ~50 Hz	20.7 Hz/mgL^{-1} 25.344 Hz/mgL^{-1}	Enhances the sensitivity as to the results attained by EB	Focuses only on alcohol detection	[44]

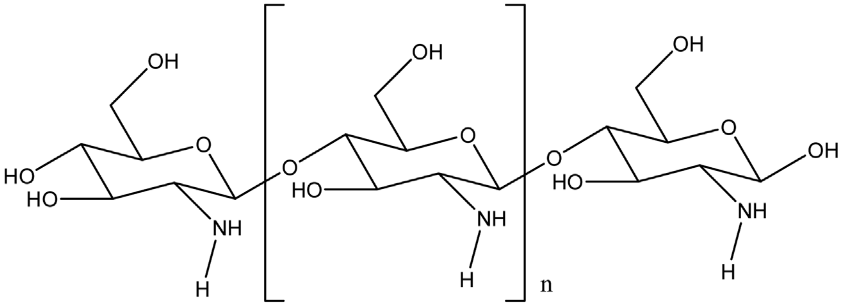

Fig. 9. Chitosan structural formula [110].

adsorption to occur covalently [112]. However, other factors such as the thickness of the coatings, and the preparation of the sensing material may also contribute to the alteration of results obtained. Besides, the fabrication method is one of the essential matters that need to be investigated thoroughly. Further studies on the bonds between the sensing film and the analyte gas are highly essential as the presence of hydroxyl groups and free amino group in chitosan determines the efficiency of analyte gas adsorption. This eventually shows that chitosan has potential in being highly selective to certain gas detection. Further improvements need to be done in functionalisation of chitosan.

4.3.2. Lipopolymer based sensor

Aside from chitosan, lipopolymer is another form of biopolymer that potentially can be used as a sensing layer [113]. Pegylated lipopolymer is derived from lipid derivatives and polyethylene, therefore it is claimed to be able to enhance the stability of lipopolymer [113]. Rahman, Ma'Radzi and Zakaria experimented VOCs of different groups with various lipopolymers such as 1,2-distearoyl-sn-glycero-3-phosphoethanolamine (lipid) lipopolymer, 1,2-distearoyl-sn-glycero-3-phosphoethanolamine-N-[succinyl(polyethylene glycol) 2000] (succinyl) lipopolymer and 1,2-distearoyl-sn-glycero-3-phosphoethanolamine-N-[carboxy (polyethylene glycol) 2000] (carboxyl) lipopolymer [113]. The results obtained showed that the hexanal gas gives the highest frequency shifts to all the pegylated lipopolymers [113] where the frequency shift obtained is the highest on QCM coated with succinyl lipopolymer that is 69 Hz, followed by carboxyl lipopolymer at 35 Hz and lipid lipopolymer at 19 Hz. The uncoated sensors give the least shift in frequency for all analyte gases tested. It can be concluded from the experiment that due to the different physicochemical properties of each gases interacting to the sensing materials, the different groups of analyte gases were selective towards a different type of lipopolymer [113,114]. More detailed research on the interaction of the functional groups in the sensing materials and the analytes should be done to re-evaluate the selectivity of the sensor. The results obtained from Rahman et al., was challenging to be interpreted in concluding which material was selective in detecting different types of analytes [114]. Related works on the biopolymer-based sensor are summarised in Table 5.

4.4. Carbon nanotubes (CNTs)

CNT has contributed to gas sensing application for years. CNT is a promising carbon-based material in providing alteration to the physicochemical properties for sensors. This is due to its unique properties in having a large surface area with high electron mobility and excellent chemical reactivity [115]. These properties made CNTs an ideal alternative sensing layer to enhance vapour adsorption processes.

CNT is commonly found in two chiral structures, (i) single-walled CNT (SWCNT) and (ii) multi-walled CNT (MWCNT) [45,116,117]. SWCNTs is made up of a sheet of carbon atoms or graphene rolled up into the cylindrical structure and crosslinking several SWCNT yields in MWCNT formation. The basic principle of the sorption of analyte gas using CNT is that the molecules of the analyte gas adsorbed onto the wall of CNT will eventually change the properties. This change is signalling that the detection is happening as there is a change in conductivity. CNT has greater surface areas for contact and its ability to detect the change in electrical properties rather than just change in mass improves the sensitivity of a sensor [64]. However, CNT tends to only improve the sensitivity of a sensor but not the selectivity. The improvement CNT brought to the sensing responses can be found in Fig. 10 [116]. Several works on the use of SWCNTs and MWCNTs on a SAW and QCM sensor have been done for VOCs detection [118–122]. Penza et al. have developed SWCNTs and MWCNTs coated 433.92 MHz SAW sensor in the detection of ethanol, ethyl acetate and toluene exposed to the concentration of 85 ppm, 178 ppm, and 93 ppm correspondingly. From both SWCNTs and MWCNTs dispersed into ethanol, ethanol gives the lowest limit of detection LOD at 1.3 ppm and 1.4 ppm. This trend is similarly observed as to when SWCNTs and MWCNTs were dispersed on toluene, resulted in greater absolute sensitivity 37.35 Hz/ppm/kHz followed by ethanol detection 31.70 Hz/ppm/kHz and ethyl acetate 27.25 Hz/ppm/kHz [118,119].

Sayago et al. also investigated the use of CNTs on a SAW sensor to detect octane and toluene exposed at 25 ppm concentration [123]. They claimed that the adsorption mechanism adhered to the physisorption phenomena where Van der Waals interaction takes place. The author tested the selectivity of analytes towards the sensing layer by exposing the sensor to NO_2, NH_3, CH_4, H_2, and CO. Octane retained 35 Hz shift in frequency in 5 s response time. This has highlighted the selectivity of the sensor towards octane was relatively higher towards toluene with 10 Hz frequency change [123]. Consales et al. on the other hand presented work on QCM sensor with SWCNTs coating to toluene [124]. However, a frequency shift of 40 Hz was obtained when the sensor was exposed to a higher gas concentration with a longer response time reached (8 min). The results analysed showed only 0.9 Hz/ppm sensitivity of the QCM sensor towards toluene. Related works are summarised and reviewed in Table 6.

4.5. Polymer with composites

A study by Lal and Tiwari reported on ZnO thin film doped with calcium to coat a langasite SAW sensor in detecting carbon dioxide CO_2 at high temperature [125]. The use of langasite allows the SAW sensor to maintain the piezoelectricity up to a very high temperature. The authors elucidated the chemo-resistive adsorption in addressing the sensing properties at high temperature [125]. Langasite is a promising material for the SAW sensor to work at higher temperature as compared to the temperature of pure ZnO thin film able to withstand [125]. The highest frequency shift 2.469 kHz of CO_2 sensing was observed when operating at 400 °C and exposed

Table 5
Biopolymer coated sensors in detecting VOCs.

Design parameters						Strengths	Limitations	References
Sensing material	Sensor	Target analytes	Analyte gas concentration	Frequency shift	Response time and sensitivity			
Chitosan (Drop casting)	5 MHz AT-cut QCM gold electrode	Methylamine	1.63 mg L^{-1}	∼175 Hz	384.70 Hz/mgL^{-1}	Good mechanical strength Good film forming properties	Less selective towards the alcohol group The reproducibility and reversibility of the sensor is less accurate.	[47]
			2.28 mg L^{-1}	∼500 Hz				
			3.25 mg L^{-1}	∼750 Hz				
			4.88 mg L^{-1}	∼1250 Hz				
			6.5 mg L^{-1}	∼2000 Hz				
		Dimethylamine	0.34 mg L^{-1}	∼50 Hz	160.96 Hz/mgL^{-1}			
			0.84 mg L^{-1}	∼100 Hz				
			1.68 mg L^{-1}	∼200 Hz				
			2.53 mg L^{-1}	∼400 Hz				
			3.37 mg L^{-1}	∼500 Hz				
			4.21 mg L^{-1}	∼800 Hz				
		Diethylamine	12.78 mg L^{-1}	∼10 Hz	7.12 Hz/mgL^{-1}			
			31.95 mg L^{-1}	∼100 Hz				
			44.74 mg L^{-1}	∼225 Hz				
			63.91 mg L^{-1}	∼400 Hz				
			95.86 mg L^{-1}	∼900 Hz				
		Methyl alcohol	7.18 mg L^{-1}	∼80 Hz	7.22 Hz/mgL^{-1}			
			14.4 mg L^{-1}	∼120 Hz				
			28.7 mg L^{-1}	∼250 Hz				
			50.2 mg L^{-1}	∼350 Hz				
			63.9 mg L^{-1}	∼450 Hz				
		Ethyl alcohol	5.6 mg L^{-1}	∼50 Hz	19.35 Hz/mgL^{-1}			
			11.3 mg L^{-1}	∼200 Hz				
			16.9 mg L^{-1}	∼300 Hz				
			22.6 mg L^{-1}	∼450 Hz				
			28.2 mg L^{-1}	∼500 Hz				
			39.5 mg L^{-1}	∼800 Hz				
		Isopropyl alcohol	17.86 mg L^{-1}	∼60 Hz	4.06 Hz/mgL^{-1}			
			35.73 mg L^{-1}	∼150 Hz				
			50 mg L^{-1}	∼200 Hz				
			71.45 mg L^{-1}	∼300 Hz				
			107.18 mg L^{-1}	∼450 Hz				
Chitosan (Dip coating)	10 MHz AT-cut QCM gold electrode	Ethanol	7 mgL^{-1}	3 Hz	0.4 Hz/mgL^{-1}	Good hydrophilicity	This method uses a QCM with higher resonant frequency but giving on 40 Hz of shift in the frequency	[112]

		n-propanol		5 Hz	0.9 Hz/mgL^{-1}	Simple setup and easy to replicate.		
		n-amyl alcohol		39 Hz	4.4 Hz/mgL^{-1} (42 s response time)			
		isoamyl alcohol		24 Hz	3.1 Hz/mgL^{-1}	Promotes a path for chitosan to attached onto the electrodes of the QCM via covalent bond. High sensitivity towards hexanal		
Pegylated polymer (Drop casting)	30 MHz AT-cut QCM gold electrode	Propanol, ethylbenzene, hexanal, decane	Coated with lipid: 6 ppm 2 ppm 1 ppm 1 ppm Non-coated: 20 ppm 2 ppm 2 ppm 1 ppm Coated with carboxyl: 6 ppm 6 ppm 1 ppm 4 ppm Coated with succinyl: 6 ppm 8 ppm 2 ppm 4 ppm	Coated with lipid 6 Hz 15 Hz 19 Hz 33 Hz Non-coated 5 Hz 2 Hz 9 Hz 9 Hz Coated with carboxyl: 9 Hz 19 Hz 35 Hz 21 Hz Coated with succinyl: 25 Hz 39 Hz 69 Hz 50 Hz	Time range 200 s – 480 s	Fast response depending on the functional groups of the pegylated polymer	Shift is not showing a significant improvement as compared to the use of chitosan	[114]

Table 6
Summary of the previous work on CNTs based coated sensors in detecting VOCs.

Design parameters						Strengths	Limitations	References
Sensing material	Sensor	Target analytes	Analyte gas concentration	Frequency shift	Response time and sensitivity			
SWCNTs MWCNTs	433.92 MHz ST-X quartz oscillating SAW resonator	Ethanol ethyl acetate toluene	Exposure: 85 ppm ethanol 178 ppm ethyl acetate 93 ppm toluene LOD: SWCNTs in ethanol (ppm) 1.3 2.7 2.8 MWCNTs in ethanol (ppm) 5.0 10.0 9.0 SWCNTs in toluene (ppm) 1.4 1.6 1.2 MWCNTs in toluene (ppm) 10.2 5.2 4.4	–	SWCNTs in ethanol (kHz/ppm) 6.89 3.32 3.17 MWCNTs in ethanol (kHz/ppm) 1.79 0.90 1.01 SWCNTs in toluene (kHz/ppm) 6.43 5.45 7.47 MWCNTs in toluene (kHz/ppm) 0.87 1.73 2.02	Highly sensitive Low limit of detection for alcohols	Selectivity depends on the type of CNTs deposited	[118]
SWCNTs	433.92 MHz ST-X quartz two-port SAW resonator	Ethanol ethyl acetate toluene	1.06×10^{-4}M (11% RH)	100 Hz	–	Low limit of detection	Selectivity depends on the type of CNTs deposited	[119]
MWCNTs			8.06×10^{-4}M (84% RH)	500 Hz		Ambient working temperature		
SWCNTs DWCNTs	158MHz ST-X cut quartz SAW	Octane toluene	25 ppm	~30 Hz (octane) ~10 Hz (toluene)	~5 s ~10 s	Good selectivity towards toluene and octane	Airbrush coating technique failed to give smooth and even dispersion	[123]
CNTs	286 MHz XY-cut $LiNbO_3$ SAW	Carbon dioxide	3.5 %	–	–	Enhanced the sensitivity of the fabrication Simple CNTs growing method	Not reproducible Challenges in repeatability	[142]
SWCNTs	10 MHz AT-cut QCM	Toluene xylene	54 ppm 3 ppm	~40 Hz ~15 Hz	8 min Toluene: -0.9 Hz/ppm Xylene: -1.8 Hz/ppm	Excellent repeatability		[124]

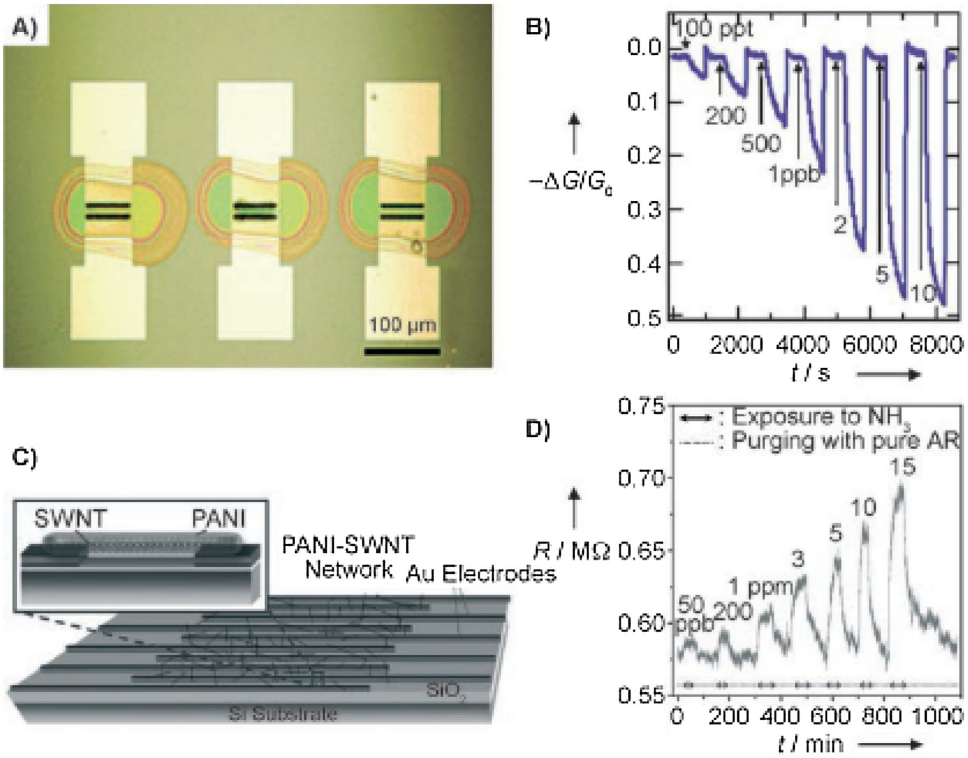

Fig. 10. The images of polymer with SWNT and the sensing response: (A) photo of the device coated with PEI-SWNT, (B) response towards different analyte gas concentration – NO_2 by measuring the conductance Reproduced with permission [143] Copyright 2003 American Chemical Society. (C) Schematic depiction of SWNT-PANI with its response (D) to different concentration of analyte gas NH_3. Reproduced with permission [144] Copyright 2006 Wiley.

to 25, 000 ppm CO_2 concentration. This work also promotes cross-sensitivity of H_2 and CO where 1.605 kHz and 1.322 kHz were obtained respectively from contact with 500 ppm gas concentration. Aside from calcium, nanoclay doped with a synthetic polymer PECH was found to provide good results in detecting analyte gases [126]. It is observed that a reduction in the roughness of the surface of the undoped PECH has improved the sensor performance. Surface roughness often associated with the surface area. A very rough surface will prevent effective adsorption and drops the sensing performance. However, the absence of a good amount of roughness to the surface of the sensing layer reduces the gas adsorption site which eventually results in poor sensing performance. This is based on the attenuation coefficient of the analyte gas to the sensing material. It can be deduced that optimum surface roughness is essential in providing a greater site for effective adsorption. Nanoclay doped PECH gives a roughness value RMS of about 129.280 nm and average roughness of approximately 135.927 nm. It produces highest sensor response at 17.446 Hz followed by doped collodion (RMS ~ 115.860 nm and average roughness ~ 123.112 nm) and doped alkyd resin (RMS ~ 128.101 nm and average roughness ~ 131.431 nm) at 6.174 Hz 12.890 Hz correspondingly [126].

Ma et al., investigated the in functionalization of the organic/inorganic nanocomposites to chitosan film – chitosan/ZnO/CuO in the attempt to improve the sensing performance [127]. The morphological image is shown in Fig. 11. As there is a presence of oxides in the functionalized material, the sensing process requires a high working temperature. Although this sensing material did not work at ambient temperature, chitosan showed a good film-forming property and good biocompatibility that enabled the organic/inorganic nanocomposites to uphold more functionalities [127]. The investigation suggested for more extensive studies needed to be conducted in looking at different composites and/or combination of polymer with chitosan for different groups of VOCs [127]. Besides polymers, metal oxides are also commonly functionalised with CNTs in improving sensor performance [128].

Another study by Sayago et al., examined the characteristics of 69 MHz SAW sensors with embedded iron(II, III) oxide (Fe_3O_4) nanoparticles on the polymer polyethyleneimine (PEI) sensitive layer [129]. The sensors performance was investigated by assessing the effect of different nanoparticles (NPs) concentration and different thickness on the sensitivity of the sensor [129]. The concentration of the NPs was fixed at 0.4 mg/mL with thickness at 13 nm and 7 nm. The thicker sensing layer gives slightly lower sensitivity regardless of the types of VOC exposed to the sensor (ethanol; 0.97 Hz/ppm, methanol; 0.81 Hz/ppm and toluene; 1.43 Hz/ppm) than 7 nm sensing layer (ethanol; 1.63 Hz/ppm, methanol; 1.00 Hz/ppm, and 1.94 Hz/ppm) [129]. The work also presented findings that with the increase in NPs concentration from 0.2 mg/mL, 0.4 mg/mL and 0.8 mg/mL, the sensitivity of the sensor increased persistently as it gives linear relationship to gas molecules adsorption. From the results obtained, the authors concluded that the frequency shift and sensitivity of the SAW sensors increased with increasing NPs concentration embedded in the polymer at a fixed NP dimension [129]. The smallest concentration of NPs used was claimed to give the best results as it led to the largest frequency shift, highest sensitivity, lowest noise, lowest limit of detection and shortest response time for all VOCs detected [129]. Another research on SAW sensor with PEI embedded with SiO_2/Si NPs and MWCNTs for detecting ethanol, methanol and toluene has been conducted by Viespe and Grigoriu [130]. The results showed a better sensing performance when PEI is embedded with MWCNTs as compared to NPs when exposed to 1600 ppm gas. This can be justified by the structural properties of MWCNTs which has provided greater specific surface area as compared to SiO_2/Si NPs [130]. Thus, it improved the adsorption mechanism of the sensing layer. Previous works on having polymers functionalised with CNTs on PIB,

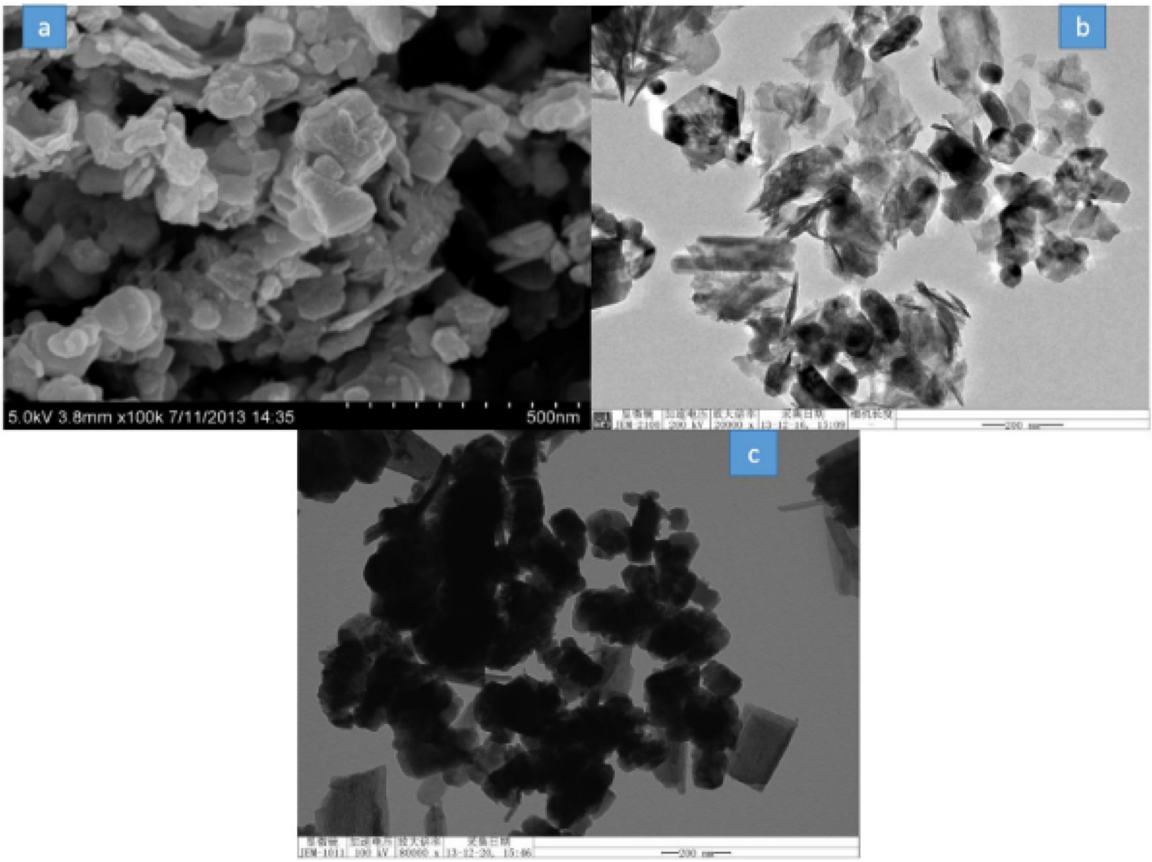

Fig. 11. Micrographs of nanocomposites: (a) SEM image of ZnO/CuO nanocomposite, (c) TEM image of nanocomposites and (c) TEM image of ZnO/CuO/chitosan nanocomposite. Reproduced with permission [127] Copyright 2016 Elsevier Inc.

PECH, and PEUT have been done with the aim to increase the sensitivity of the sensor [130–132].

Work is done by Ayad et al., and Wang et al., were on the functionalisation of the conductive polymers PANI and PEI to aid in the performance of the chitosan-based QCM sensor respectively. The use of both PANI [111] and PEI [50] with chitosan have tremendously improved the performance of the sensor. However, the properties of the polymer used to functionalize with chitosan play a crucial role in determining the selectivity of the sensing layer. Both PANI and PEI showed a strong selectivity towards amines and aldehydes, respectively. The use of chitosan functionalised with PANI and PEI aids in the interconnectivity and provides greater surface area for the adsorption process and rapid response for analyte detection that the measurement can be recorded within seconds [50]. Ayad et al. highlighted the improvement of the QCM sensor by using doped and dedoped Chitosan/PANI sensing layer in detecting amine compounds and alcohols. The sensitivity of the sensor has greatly increased from 26.23 Hz/mgL^{-1} for methylamine and 22.2 Hz/mgL^{-1} to 251.5 Hz/mgL^{-1} and 449 Hz/mgL^{-1} respectively for amines and alcohols [111]. This result is illustrating the selectivity of the sensing layer towards amine compounds as it only improved the sensitivity towards ethyl alcohol by about double the dedoped chitosan/PANI at 3.4 Hz/mgL^{-1}. However, more studies are required in combining the conducting polymers to chitosan for various groups of gases for the purpose of validating the chemical interactions between the analyte gas and the sensing layer. Fig. 12 shows the images of chitosan biopolymer modified with PEI while Fig. 13 shows the sensing mechanism when polymer with composite is used as the sensing layer.

Another composite involves the use of carbon-based composite. Graphene is a carbon-based material which provides a porous mesh structure to the sensing layer, enhancing the sensitivity of the sensor as there is presence of greater surface area for adsorption to occur. A work by Zhang et al. has functionalised and deposited graphene with chitosan onto a QCM device in detecting amine vapours that are methylamine MA, dimethylamine DMA and trimethylamine TMA with molecular mass of the amine vapours increases in the sequence of MA, DMA and TMA [133]. Gas analyte of concentration range from 5 ppm to 150 ppm was exposed to the sensor which then signifies highest sensitivity of the QCM sensor towards TMA. This may reflect the influence of both structural formula and molecular mass of the molecules. The sensitivity of the sensor towards amine vapours rises following the order; 2.3 Hz/ppm for DMA, 2.7 Hz/ppm for MA and 4.8 Hz/ppm for TMA [133]. The molecular mass does not apply to DMA detection. Hence, more study needs to be done regarding the orientation of the molecule in terms of molecular structure and bonding in obtaining effective adsorption. Modifying the graphene oxide with chitosan improved the performance of the sensor as compared to just having graphene oxide as the sensing layer [133]. Carbon-based material in most work showed a better performance in gas detection compared to the oxides [133]. High sensitivity can be obtained as the carbon-based composite typically provides a large surface area for adsorption. Besides, Zhang et al. also reported that graphene oxide/chitosan is selective towards amine vapours when tested with other gases (50 ppm of ethanol, acetone, toluene, n-hexane, ethyl acetate) with all frequency shifts fall below 20 Hz. Amine vapours give shifts of 95 Hz, 83 Hz, and 180 Hz for MA, DMA and TMA consequently [133]. However, carbon-based composite does not provide a good selectivity towards the analytes with low adsorption energy due to the absence of chemical reactions [64]. Based on the analysis, carbon-based material is commonly used as a composite to other

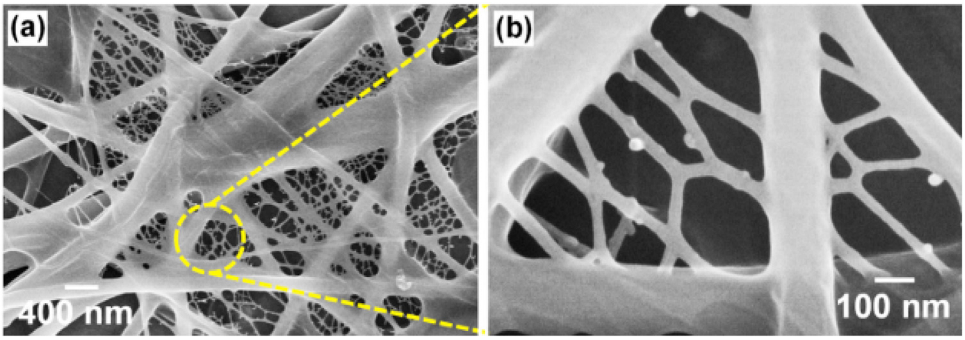

Fig. 12. SEM images of chitosan/PEI membrane Reproduced with permission [50] Copyright 2014 Elsevier Inc.

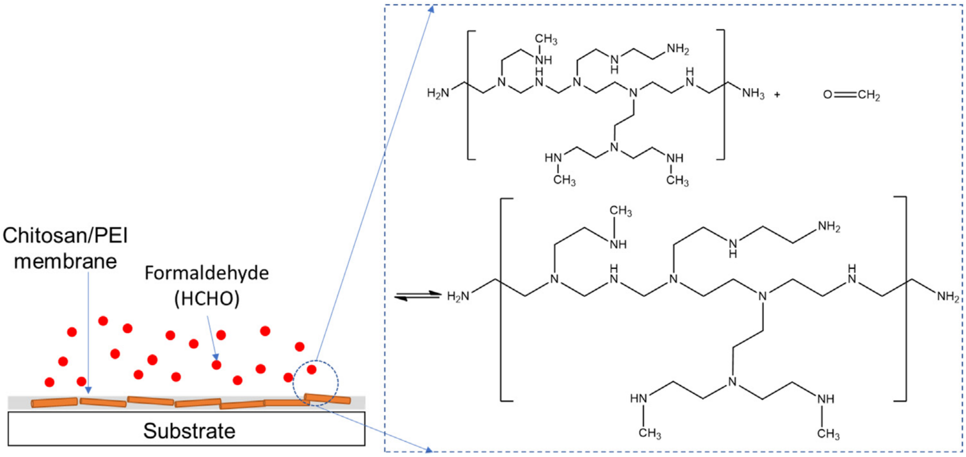

Fig. 13. Schematic illustration of the sensing mechanism of formaldehyde vapour on chitosan/PEI membrane [50].

bases due to the limitation in being selective. Hence, the use of polymer coating attracted the researchers' attention as a polymer is claimed to be more versatile in terms of the ability to be functionalised in accordance to the required purpose. Previous works on carbon-based composites also emphasised the enhancement in sensor performance [134–137].

In another work, chitosan has been used to overlay a cellulose acetate on a QCM device in detecting acetic anhydride vapour at different concentrations [138]. The experimental results show a promising method to enhance the performance of the device with a low limit of detection of acetic anhydride (5 ppm) [138]. This surface modification by overlaying materials on to the sensing device may be implemented in future work on synthesizing a high selectivity and sensitivity device. The related work has been summarised and reviewed in Table 7.

5. Limitation and challenges

Lung cancer cases emerged as one of the diseases with a high mortality rate, an alternative to screening and early detection is crucial in increasing the survival rate. Sensor-based diagnosis, the acoustic wave sensors have a high potential in providing good portability, highly sensitive and fast responding detection method. It also does not require an expert to operate. In improving the sensor performance, the sensing material plays a critical role. The major components of the sensing materials are oxides, CNTs, synthetic polymers, biopolymers and composites.

The high working temperature of oxides and less selectivity of CNTs inhibits the purpose of having a high-performance device. For instance, 10 MHz of AT-cut QCM with SWCNTs as the sensing layer gives only 0.9 Hz/ppm sensitivity towards 54 ppm toluene detection [123]. However, MIP coating has increased the sensitivity of the sensor about 2.147 Hz/ppm towards toluene analyte with a significantly lower concentration of 1.25 ppm [87]. This has drawn the attention of the researcher in developing polymer-based sensors. However, the polymer-based sensor itself requires improvement by having hybrids. Slight change to the composition of the polymer may result in improved sensing performance. This can be justified with PANI (EB) and PANI (ES) on 5 MHz QCM sensor exposed to 5 ppm methanol. The frequency shift of the sensor towards methanol has escalated from 40 Hz [90] to 120 Hz consequently [44]. Functionalisation of the hybrids determined the selectivity and sensitivity of the sensor. Oxides may promote high selectivity, CNTs may provide higher sensitivity while polymers are being versatile and flexible to be functionalised with other materials. Hybrid sensing material enables the mix of any or all the components to form a high-performance sensing device. Materials selection improves selectivity as different mechanisms favour in selective analyte gas. PEI/ Fe_3O_4 provides greater sensitivity (1.94 Hz/ppm) [129] towards toluene rather than PEI-SiO_2/Si (0.87 Hz/ppm) and PEI-MWCNTs (1.23 Hz/ppm) [130]. This stresses that functionalisation of a sensing material is important in determining the selectivity and sensitivity of a sensor.

Biopolymers are discovered to exhibit a good sensing performance aside from preventing any harsh effects on the environment as they are compostable and biodegradable. For example, the use of chitosan/ PANI composite on QCM sensor has improved the sensing performance of the sensor in detecting alcohol (ethanol at 7 mgL^{-1}) compared to monolayer chitosan. The sensitivity of the sensor had risen by about 8 times, from 0.4 Hz/mgL^{-1}

Table 7
Summary of the previous work on polymer with composites sensors in detecting VOCs.

Design parameters								
Sensing material	Sensor	Target analytes	Analyte gas concentration	Frequency shift	Response time and sensitivity	Strengths	Limitations	References
Calcium doped ZnO thin film	Langasite 9.1 MHz SAW Platinum IDTs	CO_2 CO H_2	25,000 ppm 5000 ppm 500 ppm 500 ppm	2.469 kHz 1.031 kHz 1.605 kHz 1.322 kHz	87 s 53 s 87 s 68 s	Promotes cross-sensitivity of H_2 and CO at high temperature (400 °C)	High optimum temperature lead to high power usage	[125]
Nanoclay doped PECH	10 MHz AT-cut gold plated QCM	Sulphur mustard (toxic gas)	0.17 ppm (lowest concentration detected)	Undoped: 6.823 Hz Doped by nanoclay wt%): 1.173 kHz (5%) 3.234 kHz (10 %) 17.446 kHz(12.5 %) 4.673 kHz (15 %) 4.304 kHz (20 %)	60 s response time at 15 % wt% doped	Good surface adhesion as compared to undoped polymer Film uniformity Able to detect analyte at low concentration Increased in sensitivity	Good surface adhesion as compared to undoped polymer Film uniformity Able to detect analyte at low concentration Increased in sensitivity	[126]
ZnO/CuO/ Chitosan (Drop casting)	6 MHz AT-cut QCM	Toluene Formaldehyde Dichloromethane Cyclohexanone	–	–	–	Improves greatly the adsorption properties as compared to the organic/inorganic nanocomposites on its own Good performance for different types of analytes	Challenges in desorbing the analyte gas because of the strong both chemical interaction and/or reaction	[127]
SWCNTs/CdA	315 and 433 MHz two port ST-X quartz SAW resonator	SAW 315 MHz Ethanol Ethylacetate Toluene SAW 433 MHz Ethanol Ethylacetate Toluene	1.1 1.0 0.6 0.7 0.9 0.7	–	20.05 Hz/ppm 39.0 Hz/ppm 43.7 Hz/ppm	High sensitivity towards organic vapours	Improve in sensitivity via increase in resonant frequency of the sensor instead of improvement in the functionalisation of the coating	[134]
MWCNTs/CeO_2	434 MHz AT quartz SAW sensor	Acetone Ethanol	45 %	200 kHz		Higher sensitivity towards acetone Promotes the use of semi-conducting sensing material	Sample preparation requires high temperature, up to 500 and 800 °C	[136]
SWCNTs in Cadmium	315, 433, 915 MHz SAW resonator and 10 MHz AT-cut QCM	SAW Ethanol Ethylacetate Toluene QCM	0.23 0.69 0.20	– – –	131.84 Hz/ppm ~40.0 Hz 144.7 Hz/ppm	Very low limit of detection		[115]

		Ethanol Ethylacetate Toluene	1–180 ppm 1–500 ppm 1–150 ppm	– – –	0.32 Hz/ppm 0.15 Hz/ppm 0.29 Hz/ppm			
PEI/ Fe_3O_4	69 MHz ST-X cut quartz SAW	Ethanol Methanol Toluene	PEI only (0 nmNP diameter, 0 mg/mL): 320 ppm 360 ppm 262 ppm (NP diameter; 13 nm, 0.4 mg/mL): 139 ppm 166 ppm 95 ppm (NP diameter; 7 nm, 0.4 mg/mL): 65 ppm 105 ppm 54 ppm (NP diameter; 50 nm, 0.2 mg/mL): 203 ppm 311 ppm 165 ppm (NP diameter; 50 nm, 0.4 mg/mL): 212 ppm 343 ppm 200 ppm (NP diameter; 50 nm, 0.8 mg/mL): 240 ppm 309 ppm 206 ppm	– – –	0.56 Hz/ppm 0.50 Hz/ppm 0.69 Hz/ppm 0.97 Hz/ppm 0.81 Hz/ppm 1.43 Hz/ppm 1.63 Hz/ppm 1.00 Hz/ppm 1.94 Hz/ppm 0.81 Hz/ppm 0.53 Hz/ppm 1.00 Hz/ppm 1.06 Hz/ppm 0.66 Hz/ppm 1.13 Hz/ppm 1.13 Hz/ppm 0.88 Hz/ppm 1.31 Hz/ppm	Fast response time (9 s response time)	High limit of detection Not considered selective towards alcohol as compared to toluene	[129]
PECH and PEUT with MWCNTs	ST-X cut SAW	Octane Toluene	25 to 100 ppm	~10 to 100 Hz ~50 to 450 Hz	30–40 s response time	Sensor sensitivity favours to toluene. Fast response, fast recovery time and good repeatability	Least sensitive towards octane	[131]
PEI – SiO_2/Si NPs and MWCNTs	69.4 MHz SAW delay line	Ethanol	176.5 ppm	–	1.19 Hz/ppm, 10.5 s response time (MWCNTs-PEI)	Low noise level for SiO_2/Si NPs	The experiment was conducted a high analyte concentration 1600 ppm	[130]

Table 7 (*Continued*)

Design parameters						Strengths	Limitations	References
Sensing material	Sensor	Target analytes	Analyte gas concentration	Frequency shift	Response time and sensitivity			
			15.2 ppm					
			823.5 ppm		0.79 Hz/ppm, 12 s response time (SiO_2/Si-PEI)			
					0.51 Hz/ppm, 60 s response time (PEI)	Improve overall sensor performance		
		Methanol	184.2 ppm		1.14 Hz/ppm			
			16.7 ppm		0.72 Hz/ppm			
			1050 ppm		0.40 Hz/ppm			
		Toluene	170.7 ppm		1.23 Hz/ppm			
			13.8 ppm		0.87 Hz/ppm			
			750 ppm		0.56 Hz/ppm			
PIB - CNTs	ST-X cut SAW sensor	Octane	200 ppm	~1400 Hz (PIB)	7.036 Hz/ppm	Reversible Favours in detecting octane	Excess and undersupply of CNTs dropped the sensing performance	[132]
				~1600 Hz (PIB-MWNTs (2%))	8.082 Hz/ppm			
		Toluene		~800 Hz	4.596 Hz/ppm			
				~550 Hz	3.002 Hz/ppm			
				~600 Hz	3.282 Hz/ppm			
				~400 HZ	2.016 Hz/ppm			
Superhydrophobic n-octadecylsiloxane (PODS) – PEI	QCM	Hexanal	Gas tested as concentration 50 ppb	0 Hz (PODS)	–	Low limit of detection Good water resistance properties	Selectivity at lower concentration is not subtle	[71]
				5.9 Hz (PODS-PDA-PEI)	0.118 Hz/ppb, 23 s response time			
				6.1 Hz (PDA-PEI)	0.122 Hz/ppm, 24 s response time			
Chitosan/ PANI (Drop casting)	5 MHz AT-cut QCM gold electrode	Methylamine	(Dedoped)			Greater surface for adsorption to occur	Did not solve the sensitivity towards alcohol.	[111]
			$0.65\ mgL^{-1}$	~10 Hz				
			$1.6\ mgL^{-1}$	~100 Hz				
			$3.25\ mgL^{-1}$	~220 Hz	$26.23\ Hz/mgL^{-1}$	Enhanced the sensitivity of the detecting analyte gases		
			$9.76\ mgL^{-1}$	~500 Hz				
			$16.3\ mgL^{-1}$	~700 Hz				
			(Doped)					
			$0.981\ mg\ L^{-1}$	~140 Hz				
			$1.962\ mgL^{-1}$	~300 Hz				
			$2.94\ mgL^{-1}$	~600 Hz	$251.5\ Hz/mgL^{-1}$			
			$4.9\ mgL^{-1}$	~1150 Hz				
			$6.89\ mgL^{-1}$	~1550 Hz				
		Dimethylamine	(Dedoped)					
			$1.6\ mgL^{-1}$	~30 Hz				
			$2.25\ mgL^{-1}$	~50 Hz				
			$3.2\ mgL^{-1}$	~100 Hz	$22.2\ Hz/mgL^{-1}$			
			$6.4\ mgL^{-1}$	~300 Hz				
			$9.64\ mgL^{-1}$	~460 Hz				
			$16\ mgL^{-1}$	~600 Hz				
			(Doped)					
			$0.49\ mgL^{-1}$	~150 Hz				
			$1.94\ mgL^{-1}$	~700 Hz				
			$2.94\ mgL^{-1}$	~1500 Hz	$449\ Hz/mgL^{-1}$	Provides a fast response towards the adsorption process		
			$4.85\ mgL^{-1}$	~2500 Hz				
			$6.5\ mgL^{-1}$	~2800 Hz				

Chitosan/ PEI (Drop casting)	5 MHz AT-cut QCM gold electrode	Ethyl alcohol	(Dedoped) 7.1 mg L^{-1} 14.2 mgL^{-1} 21.3 mgL^{-1} 35.5 mgL^{-1} 49.7 mgL^{-1} 71 mg L^{-1}	∼20 Hz ∼40 Hz ∼60 Hz ∼90 Hz ∼120 Hz ∼180 Hz	3.4 Hz/mgL^{-1}			
			(Doped) 10.8 mgL^{-1} 21.6 mgL^{-1} 32.4 mgL^{-1} 43.2 mgL^{-1} 64.8 mgL^{-1}	∼30 Hz ∼100 Hz ∼160 Hz ∼260 Hz ∼470 Hz	8.6 Hz/mgL^{-1}			
		Formaldehyde	5 ppm 15 ppm 35 ppm 85 ppm 185 ppm	Chitosan, Chitosan + PEI (1500 Hz), Chitosan + PEI (8000 Hz): ∼0 Hz ∼2 Hz 2 Hz, 4 Hz, 7.5 Hz 2.5 Hz, 7.5 Hz, 15 Hz 4.3 Hz, 14.58 Hz, 31.92 Hz		Aids in the interconnectivity and provides greater surface area (Nanofibers-net-binary NNB structure for chitosan) for adsorption process Rapid response for analyte detection	Limit of detection at 5 ppm	[50]
Graphene oxide/ chitosan nanocomposite	6 MHz AT-cut Ag-coated QCM electrode	MA DMA TMA DEA TEA Ethanol Acetone Toluene n-hexane ethyl acetate	5 to 150 ppm amine vapour for vapour determination (LOD) 2.2 ppm (MA) 2.6 ppm (DMA) 1.3 ppm (TMA) All exposed to 50 ppm gas analytes for selectivity test	Selectivity test: 95 Hz (MA) 83 Hz (DMA) 180 Hz (TMA) Other gases: < 20 Hz	Sensitivity for amine vapour: 2.7 Hz/ppm (MA) 2.3 Hz/ppm (DMA) 4.8 Hz/ppm (TMA)	Highly influenced by the structural formula and molecular mass Selective towards amine vapour Shortened response time with presence of GO as it gives greater adsorption site	Does not report the effect of gas molecules interaction with the sensing layer in terms of the chemical groups present involved	[133]
Cellulose acetate nanofibers -chitosan	10 MHz AT-cut QCM (OpenQCM) gold electrode	Acetic anhydride	200 ppm	62 Hz	44 s response time	Low limit of detection	Not sensitive towards other VOCs	[138]
		Acetone Toluene Ethanol Methanol Xylene	5 ppm (LOD)	<5 Hz <5 Hz <5 Hz <5 Hz <5 Hz	0.234 Hz/ppm			

[112] to 3.4 Hz/mgL^{-1} [111]. Nonetheless, functionalization of the polymer determines the target gas. Chitosan/PANI may give a frequency shift of about 20 Hz [111] for 7 mgL^{-1} ethanol exposure while chitosan/PEI dropped below 5 Hz although exposed to a higher ethanol concentration (50 ppm) [133]. More initiative in developing biopolymer-based sensor should focus on the functionalisation of the hybrid sensing materials in accordance to the selective analyte gases. The properties of the complex biopolymer may require deeper knowledge in the physicochemical properties.

6. Concluding remarks

Acoustic wave sensor is surging rapidly in developing towards gas sensing application. This is due to the demanding health diagnosis issues especially in detecting cancerous diseases as most diagnosis methods are invasive, takes a long time to produce results and requires expertise to handle. Recent studies highlighted the use of breath analysis using acoustic wave sensors. Tonnes of work on acoustic wave sensors are currently progressing into producing a highly sensitive measures to cater to the limitation of current methods. The use of a sensing layer on the acoustic wave sensors determines the sensitivity and selectivity of the sensors towards the analyte gases. In our insights, metal oxide is highly sensitive towards analyte gases involving oxidation reaction. However, the concern is that most studies showed the need for high temperature to achieve the optimum sensing response. Polymers are now intensively used for sensing application. This includes the use of biopolymers. Biopolymers are showing great potential to be developed into a high selectivity and sensitivity towards the analyte gases.

However, each material that stands on its own will require modification to increase the selectivity and sensitivity of the sensor. Presently, the use of biopolymer attracts much research for this scope of the study. Fabricating biopolymer with composites allows both physicochemical and morphological modification of the sensing layer. This is a good method to increase the surface area for a greater surface for adsorption when using, for example, CNTs, and another polymer such as PANI. Modification of the sensing layer has been used using several types of materials, but there is still room for improvements. Further investigation on properties of each material and analytes gases are required for further enhance the use of acoustic wave sensor for gas sensing purposes.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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