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Trends in chemical sensors for non-invasive breath analysis

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ABSTRACT

The advancements of chemical sensors over the past 50 years have significantly expanded the diversity of available sensors making them more affordable, portable, sensitive, and partially chemically selective. Despite these advancements, the diversity and capability of sensors used in breath analysis has remained limited due to challenges that persist in this field. This encompasses fluctuations in humidity, temperature, and oxygen concentration within breath that all pose challenges that can significantly affect sensor output. This review article aims to introduce and compare the techniques used in current chemical sensor arrays for breath analysis. A brief overview of the discrete chemical sensors and arrays employed in breath research, at both commercial and research levels, is discussed. Furthermore, current trends in data analysis for chemical sensor arrays in breath analysis is described. Finally, a detailed outlook of recent diagnostic results from studies implementing sensor arrays from the last 5 years is outlined.

1. Introduction

The practice of using breath to assess an individual's health dates back over 2000 years [1]. The earliest documented instance dates to the era of Hippocrates (460-370 BCE), a Greek physician who employed the terms *fetor oris* (halitosis) and *fetor hepaticus* (breath of the dead) to describe patients in his medical treatise [2]. Over time, doctors started using a patient's breath odour to provide useful information on their physiological well-being, and, in certain cases, a diagnosis [3]. The first identification of a Volatile Organic Compound (VOC), specifically acetone, in human breath was credited to Wilhelm Petters in 1857 [4]. Shortly after, the elucidation of key chemical pathways, acetone in particular, provided one of the pivotal roles in the emergence of breath research as an independent field of study. Today, the most common approaches to monitor/detect the subtle changes of VOCs in exhaled breath is to use high-end analytical lab instruments [5]. These instruments offer the requisite sensitivity and specificity to discern chemical compounds within an odour sample and, potentially, determine their concentrations. Such devices excel in pinpointing crucial biomarkers linked to a disease state and have been extensively applied in breath analysis, with Gas Chromatography-Mass Spectrometry (GC-MS) considered as the gold standard [5]. Yet, due to its requirement for dedicated infrastructure, size, and need for specialized staff and cost, these instruments are only seen in analytical laboratories where samples are transported to the laboratory once collection is complete. In clinical settings with centralised diagnostic departments, this works well, as many samples can be run relatively quickly with a high degree of confidence. However, this is not appropriate for clinical pathways that require an immediate decision, such as in primary care or rural/developing hospitals. To this end, alternative approaches have been investigated.

One such approach, has been the application of chemical sensors as diagnostic tools. Chemical sensors were defined by the International Union of Pure and Applied Chemistry (IUPAC) as "devices that transforms chemical information, ranging from the concentration of a specific sample component to total composition analysis, into an analytically useful signal" in 1991 [6]. This definition stated that chemical sensors contain two basic functional units: a receptor and a transducer. Whereby, the sensor is classified by the operating principle of the transducer [6]. With this definition, many sensors are incorporated, including optical, magnetic, mass-sensitive, electrical, thermometric, and electrochemical devices [6]. Moreover, chemical sensors can be composed of either a discrete gas sensor designed to detect a single chemical compound or a composite of sensors into an array. These arrays were termed an "electronic nose" (or eNose) and were first proposed in the 1980s by Dodd and Persaud [7]. The gas arrays were capable of utilising ambient air for the carrier gas, in contrast to the MS techniques that require helium, hydrogen or nitrogen. They could also be produced in portable formats and are often simple to use with easily

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Abbreviations:

VOC	Volatile Organic Compound
VSC	Volatile Sulphur Compound
VIOC	Volatile Inorganic Compound
ppb	parts per billion
ppt	parts per trillion
ml	millilitres
ENose	Electronic Nose
COPD	Chronic Obstructive Pulmonary Disease
IUPAC	International Union of Pure and Applied Chemistry
GC-MS	Gas Chromatography-Mass Spectrometry
MOX	Metal-Oxide
MOS	Metal-oxide semiconductor
MEMS	Microelectromechanical Sensors
SAW	Surface Acoustic wave
QCM	Quartz Crystal Microbalance
CNT	Carbon nanotube
SWCNTs	Single-Walled Carbon Nanotube
MWCNTs	Multi-Walled Carbon Nanotube
TMDs	Transition Metal Dichalcogenides
MoS2	Molybdenum Disulfide
WSe2	Tungsten Di-Selenide
GO	Graphene Oxide
rGO	Reduced Graphene Oxide
FVSA	Flexible Virtual Sensor Array
LIG-IDEs	Laser-induced Graphene Interdigital Electrodes
CEN	Chemiresistive ENose
VLNs	Villi-like Metal Oxide Nanostructures
VLNW	Villi-like Nanostructures WO3

interpretable results. These aspects, alongside their capacity to provide an overall odour fingerprint of samples, aligns well with the needs of the medical community.

This review delves into the potential application of arrays of gas sensors in breath analysis, with a particular focus on recent developments within the past five years. The discussed timeframe holds particular significance for breath research, marked by the Covid-19 pandemic, which, while imposing limitations on broader breath research, also introduced new opportunities for the rapid detection and diagnosis of Covid-19 patients. The paper continues with an overview of the breath diagnostic field, providing a brief examination of the current chemical sensors and arrays employed in breath research – at both commercial and research levels. A concise description of the techniques used for analysing the breath data captured by chemical sensors is then provided. Subsequently, this review highlights the results of diagnostic breath studies conducted within the last five years that leveraged chemical sensors in a variety of diseases. Lastly, the paper discusses some of the advantages and challenges associated with the use of sensor arrays/eNoses for breath diagnostics.

2. Chemical sensors in breath analysis

Breath analysis brings an interesting set of challenges when using chemical sensor arrays, due to the atypical characteristics found in breath. To start, it is typically warmer, with a much higher humidity content compared to the surrounding air. It also has a reduced oxygen level and an increase in carbon dioxide concentration. Most discrete VOC gas sensors are not affected by the high carbon dioxide levels, but some, such as metal-oxides, can be affected by this reduction in oxygen. In addition, many sensors are also humidity and temperature dependent, both in terms of their baseline output and their sensitivity, in which water molecules can compete for absorption sites with VOCs. Thus, breath can produce a significant modulation in the sensor output, but this may not be related to changes in VOCs.

The development or integration of reliable chemical gas sensors for breath has become an active area of research. As such, a plethora of chemical sensors have been developed that range from traditional metal-oxide and piezoelectric sensors to more recently advanced nanomaterial and biosensors. The following sections will provide an overview of the working principles associated to sensors recently implemented in breath analysis.

2.1. Chemoresistive sensors

Chemical sensors play a key role in detecting and quantifying target compounds through interactions with specialized sensing layers, resulting in measurable signals, such as electrical currents [8]. These interactions change the physical/chemical properties of the sensing layer, which is then detected. In the field of breath analysis, the most common chemoresistive sensors used are metal-oxide semiconductors and conductive polymers. Such sensors are popular with researchers as they are easy to interface with modern electronics, simplifying the integration process [9,10].

2.1.1. Conductive polymers

Conductive polymers have found favour with researchers due to their room temperature operation, ease of deposition and simple resistive measurement regime [11,12]. The sensing layers either consists of a doped organic polymer matrix or an insulating polymer mixed with a conductive filler. The specificity and selectivity of these sensors rely on the interactions between the polymer and targeted analytes, allowing the detection of specific compounds in different environments. In the first form, when the sensor interacts with particular gases or compounds, variations in doping levels within the sensing layer impacts the quantity of charge carriers, inducing changes in the polymer film's conductivity. The change in conductivity, measured via electrodes connected to the sensing layer, is directly related to the presence and concentration of specific analytes [13].

Alternatively, in the second form, an insulating polymer is mixed with a conductive matrix to form a semiconducting material. In operation, the polymer absorbs the chemical and swells. This swelling reduces the conductive paths through the material and results in an increase in resistance of the sensing film. The most common polymers are those used as the stationary phase in columns for gas chromatography, such as polysiloxane and polyethylene glycol [14]. In regard of the conductive filler, carbon black nanoparticles have been the most used, though more recently, other conductive materials, such as gold, have found favour. In general, most conducting polymers have a high resistivity, making measurement more difficult and are affected by the electric field applied to them when being measured. By using a conducting polymer with a conductive frame, the reduction in the overall resistance improves sensor sensitivity [15]. Overall, these sensors have been used in several commercial electronic nose instruments, though only a few manufacturers are currently active. Of these, only the Sensigent Cyrano 320 has been recently used for breath and the sensors are not sold separately [16].

2.1.2. Metal-oxide semiconductors

Metal-oxide semiconductor sensors are composed of a metal oxide layer that responds to target gases by inducing variations in the concentration of charge carriers within the layer, impacting the material's electrical resistance [17,18] (Fig. 1-a). Categorized into two types: P-type and N-type metal oxide variants. P-type sensors use holes as majority carriers; their response to oxidising gases leads to a reduction in

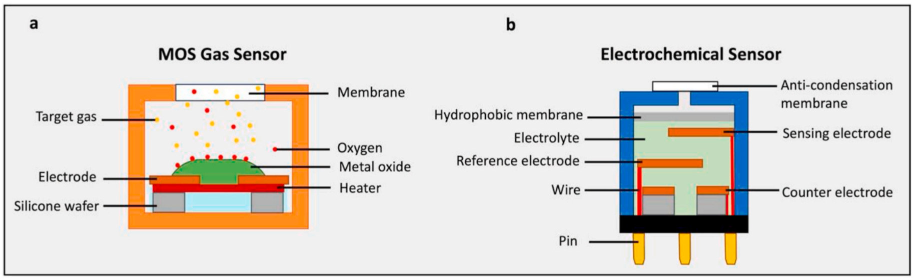

Fig. 1. An illustrated diagram of two sensor types: (a) MOS gas sensor; (b) Electrochemical gas sensor.

sensor resistance, whilst a reducing gas/VOC leads to an increase in sensor resistance [19]. N-type metal-oxide sensors are the opposite in terms of sensor resistance response. The selectivity of metal oxide sensors toward gas types relies on its oxidation or reduction properties, which can be tailored through operational temperature and doping with a catalyst. Almost all current commercial sensors are N-type. This is because the sensitivity is much higher for N- than P-type. However, P-type sensors have much greater humidity tolerance with almost no change in resistance from low to 100%r.h. levels. N-type, in contrast, have significant humidity cross-sensitivity, where high levels of humidity can change the baseline and the sensitivity of the sensors.

MOS sensors are the most utilised gas sensors across the field, particularly in breath analysis [20]. Despite their requirements for high-operating temperatures (in excess of 200 °C) and intolerance of humidity. These sensors are known for their high sensitivity, and ease of purchase. MOS sensors, first developed in the 1960s, have advanced significantly over the last 50 years, moving from the "Taguchi" style sensor to screen-printed ceramic tiles, through to the latest micro-electromechanical sensors (MEMS) based micro-hot plate technology [20]. The latest of these advancements, the MEMS sensors, reduced power consumption by over 95 % compared to the original tube Taguchi design.

2.2. *Electrochemical sensors*

Electrochemical sensors rely on the interaction between an analyte and electrodes submerged in an electrolyte to generate a measurable electrical signal [21]. These sensors form an interface where chemical reactions occur when the target analyte is present [22,23]. The analyte's interaction with the electrode surface induces changes in electrical properties, such as current or potential, which are then measured to determine the analyte concentration [22,24] (Fig. 1-b). Electrochemical sensors hold significant market share through their use in the safety and now environmental monitoring sectors. They beneficially provide a linear output and operate at room temperature but have a relatively large form factor and have some intolerance to temperature and humidity. Historically, they had a lower sensitivity than MOS sensors, however the latest generation of electrochemical sensors now have ppb level sensitivity (developed for air quality applications) making them more applicable to breath analysis.

2.3. *Piezoelectric sensors*

Piezoelectric sensors use the piezoelectric effect in their substrate. This effect is the ability for a material to generate an electric charge when affected by mechanical stress [25–28]. The substrate mounted with electrodes is able to detect slight disturbances on the surface of the sensor leading to an electrical response being generated by piezoelectric crystals in the surface layer [27]. One type of piezoelectric sensor used in breath research is the Quartz Crystal Microbalance (QCM) sensor. These sensors are known to have high sensitivity, by producing measurable frequency changes at low chemical concentrations (low ppb). QCM sensors use a quartz crystal with piezoelectric properties which vibrate at a specific resonant frequency when traversed by an alternative current [29]. When analytes, in gas or liquid form, encounter the surface layer, mass is added to the crystal, altering its resonant frequency [30]. The crystal used and so the device, is extremely sensitive to changes in mass, meaning the QCM sensors have high sensitivity at room temperature operation [31]. The change in resonant frequency in the quartz crystal sensing layer is measured by observing the shifts in the crystal's oscillation frequency, which are then correlated to the presence and specific concentration of analytes in the environment [30,32–34]. These sensors were initially very popular, due to the ease of purchase of the QCM component, ease of coating and the high sensitivity. However, interfacing is much more challenging than chemoresistive sensors, they have a relatively large form factor and are highly temperature dependent. At present, there are currently no commercially available QCM gas/VOC sensors on the market, though there have been previous products and advanced prototype systems that used this technology.

2.4. *Nanomaterial sensors*

Nanomaterial sensors represent a class of sensing devices employing nanoscale structures to detect and respond to specific analytes [35]. These sensors harness the properties of nanomaterials, utilising their specific physical, chemical, and electronic characteristics for highly sensitive and selective detection [36–38]. By exploiting nanoscale phenomena, such as surface interactions [37,39] and quantum effects [40,41], these sensors have the capability to provide innovative solutions across various fields, including environmental monitoring, healthcare, and industry [42,43].

2.4.1. *Metal-oxide semiconductors*

Metal oxide nanomaterial sensors use the properties of metal oxide nanoparticles or nanostructured films for gas sensing applications [44]. The basic principles are similar to what was described for metal-oxide semiconductors, but with reduced particle size or where ultra-thin layers (either continuous or discontinuous) are used. These sensors typically consist of metal oxide nanoparticles, such as tin oxide (SnO_2), zinc oxide (ZnO), or tungsten oxide (WO_3) [45–47], which exhibit high surface-to-volume ratios, enhancing their sensitivity to gas interactions. Functionalisation of these nanoparticles with catalytic metals can enhance selectivity by tailoring their surface properties to interact selectively with desired analytes [48]. However, they still require high operating temperatures and suffer from some level of humidity intolerance. These materials are often used on the latest generation of MEMS micro-hot plate structures as the thin layer reduces the weight on the heated membrane.

2.4.2. *Carbon nanotubes semiconductors*

Carbon nanotube (CNT) nanomaterial sensors use carbon nanotubes which cylindrical structures with enhanced electrical, mechanical, and chemical characteristics [49,50]. These sensors involve the integration of carbon nanotubes, single-walled (SWCNTs) where a single sheet of graphene is rolled in a cylindrical shape [51–53]; or multi-walled (MWCNTs) where multiple single-walled carbon nanotubes are nested inside each other, either dispersed in a solution or directly deposited onto substrates [54–56]. The working principle relies on changes in electrical conductivity or resistance when exposed to specific analytes [50,52,57]. Interaction between target molecules and the carbon nanotube surface alters the electrical properties due to charge transfer or changes in the nanotubes' band structure [58]. Functionalisation or modification of carbon nanotubes such as electrodeposition of metal nanoparticles can enhance selectivity and sensitivity toward specific gases or molecules [50,58,59]. Commercially, CNT based gas sensors are now available from a small number of suppliers. However, they have yet to achieve mainstream use [60,61].

2.4.3. D-materials

D-material sensors, comprising various 2D materials such as graphene, transition metal dichalcogenides (TMDs), and other emerging 2D materials to use the unique properties of atomically thin layers [62–65]. The characteristics of 2D materials compared to multi-layered materials display better in plane stability as all atoms are arranged in mono-layers and high surface-area-to-volume ratio and high carrier mobility [64–69]. TMDs, comprising materials like molybdenum disulfide (MoS_2) or tungsten di selenide (WSe_2), possess tuneable bandgaps and specific optoelectronic properties [70–73]. D-material sensors detect changes in electrical conductivity, optical properties, or surface interactions when exposed to specific analytes depending on the 2D material composing it [74]. Functionalising these 2D materials or creating [75–78]; which combine various single layers of 2D materials, can enhance selectivity and sensitivity, enabling the precise detection of target molecules or gases.

2.4.4. Graphene oxide sensors

Graphene oxide sensors exploit the properties of graphene oxide, a derivative of graphene comprising oxygen-containing functional groups [79,80]. Graphene oxide is known for its high optical transparency, flexibility and good electrical conductivity at room temperature [81–83]. It is composed of a single 2D sheet of carbon atoms organised in a honeycomb structure [84]. Sensors made of graphene oxide (GO) are made by deposing layers of graphene oxide on substrates or functionalisation onto specific surfaces. Their sensing mechanism primarily relies on changes in electrical conductivity, adsorption, or desorption events when exposed to target analytes [85–87]. Functionalisation strategies enable the selective detection of specific molecules or gases, enhancing sensitivity and enabling tailored detection for diverse applications in gas sensing and biosensing [88].

Reduced Graphene Oxide (rGO) gas sensors rely on the gas adsorption or desorption on the rGO surface, creating changes in charge carrier concentration and material resistance, which overall alters the electrical conductivity of the reduced graphene oxide [89–91]. These sensors offer high sensitivity and selectivity to various gases, capitalising on graphene-based materials' extensive surface area and chemical reactivity of graphene-based materials [92,93]. rGO is issued from the reduction of graphene oxide (GO), where oxygen-containing functional groups from GO sheets are removed or reduced, enhancing their electrical conductivity. Compared to graphene oxide sensors, rGO sensors display higher conductivity and improved sensing performances [94–96]. Their rapid response times, operation at room temperature, and compatibility with flexible substrates are the main key advantages of rGO sensors [89,90,97]. However, limitations include potential issues with baseline drift and long-term stability, impacting reliability over time [98,99]. rGO heightened sensitivity also exposes them to challenges linked to environmental factors like humidity, necessitating calibration and compensation techniques to enhance reliability [100].

2.5. Sensor comparison

The previous section provided an overview on the background of different sensors recently employed in breath analysis. Direct comparisons between these sensors can be difficult due to the variances in sensing materials and operational modes, which can directly influence sensor performance. However, some general characteristics can be compared. Table 1 provides a comparison of the typical capabilities reported for each sensor type. It is important to note that this table does not report on the most optimal sensors available for breath analysis, but standard ranges reported from the literature. As can be seen, there are commonalities observed within each category, except for a few notable exceptions. Power consumption being the most prominent, with all but some MOS and QCM requiring minimal consumption as the operating temperature is low or they employ smart substrates to reduce the power consumption. In the case of the QCM sensor, this was not due to the sensors itself, but because of the technique's requirement for an external device in sensor readings, which can cost over $3000. The requirement for additional hardware for readings also reduced its capability for integration into breath-based analysis. Thus, it is not often utilised in breath analysis outside of research areas. This is similarly observed for conducting polymers and CNT's as they are limited commercial vendors selling sensors. Alongside this, the reported response times for CNT's are considerably higher than the other techniques, which limits their capability for real-time breath analysis. Furthermore, commercial graphene-based gas sensors are relatively new to the field, which means there are few sensors on the market and come with a higher price point. Overall, these factors were the determinants for why CNT's, graphene, and piezoelectric sensors were reported to have the lowest ease-of-integration capabilities. In contrast, MOS has the best integration capability due to its long lifespan, cost, and large commercial range. Electrochemical closely follow MOS sensors in their integration capability but come with a slightly higher price point and longer response time.

3. Sensor arrays in breath analysis

The expansion of discrete sensors has led to the development of sensor arrays containing a combination of discrete sensors, commonly characterised as an eNose. These arrays normally encompass between 3 and 32 discrete sensors – though much larger arrays have been produced at a research level. In either case, when the eNose is presented with an odour, each sensor responds to key chemical components within the sample. This response is dependent upon the type of sensor utilised and its interaction with the chemical composition. As each sensor is different, the response of each sensor will be distinct, allowing the implementation of pattern recognition or machine learning approaches. Fig. 2 depicts the standard working principles found in a breath analysis sensor array. This process typically includes the collection into an eNose device, response of the sensors to the breath compounds, followed by a reading of the sensor outputs, addition of classifiers, and finally data analysis.

In breath analysis, sensor array devices are typically separated into three groups. The first, is where a commercial instrument is used for analysis. This is the easiest way to access a chemical array approach and is often undertaken by those lacking experience or knowledge in sensors. These units have the advantage of technical support/development, quality control checks and pre-developed methods for breath analysis. The second is the purchase of commercialised gas sensors integrated into a custom array. This again has the advantage of rapid development with easily replaceable sensors. However, the range of sensors is limited to what can be purchased off the shelf. Finally, the third group encompasses the development of customised sensors into a new array. This is the smallest group, primarily due to the challenges faced at every developmental stage, but it does provide the most innovative sensing solutions.

3.1. Commercial (eNose) arrays

The simplest method to undertake breath studies is using a

Table 1

Comparison of the typical sensor properties observed in chemical sensors. Notes: (*) = External Device Required for sensor reading. Sensitivity: Low = ≥ ppm; Average = low ppm-high ppb; High: ≤ ppb. Acronyms: RT = Room Temperature; ppm = parts per million; ppb = parts per billion. N/A = Not applicable.

Property/Material	MOS	Electro-Chemical	Piezoelectric	Conducting Polymer	CNTs	Graphene
Sensitivity	Average	Average	High	Average	Average	High
Specificity	Low	Low	Average	Low	Average	Average
Lifespan	2–10 years	2–3 years	1–3 Year	<1 Year	<1 Year	<1 Year
Humidity Tolerance	Mostly High	Medium	High	Medium	Mostly High	Medium
Temperature Range	−40°C-80 °C	−20°C-60 °C	−10°C-100 °C	25–80 °C	−40°C-150 °C	25–200 °C
Power Consumption	Minor-High	Minor	High*	Minor	Minor	Minor
Response Time	<30s	5–80s	300 Datapoints/sec	5–150s	>30min	30–240s
Drift over Time	High	Medium	High	High	High	Medium
Cost of Sensor	$10-100	$50-500	$40–5000*	N/A	N/A	>$250
Commercially Available	Yes	Yes	Yes	Limited	Limited	Limited
Ease of integration	High	High	Low	High	Low	Low
References	[101–107]	[108–114]	[115–120]	[121–125]	[126–131]	[132–138]

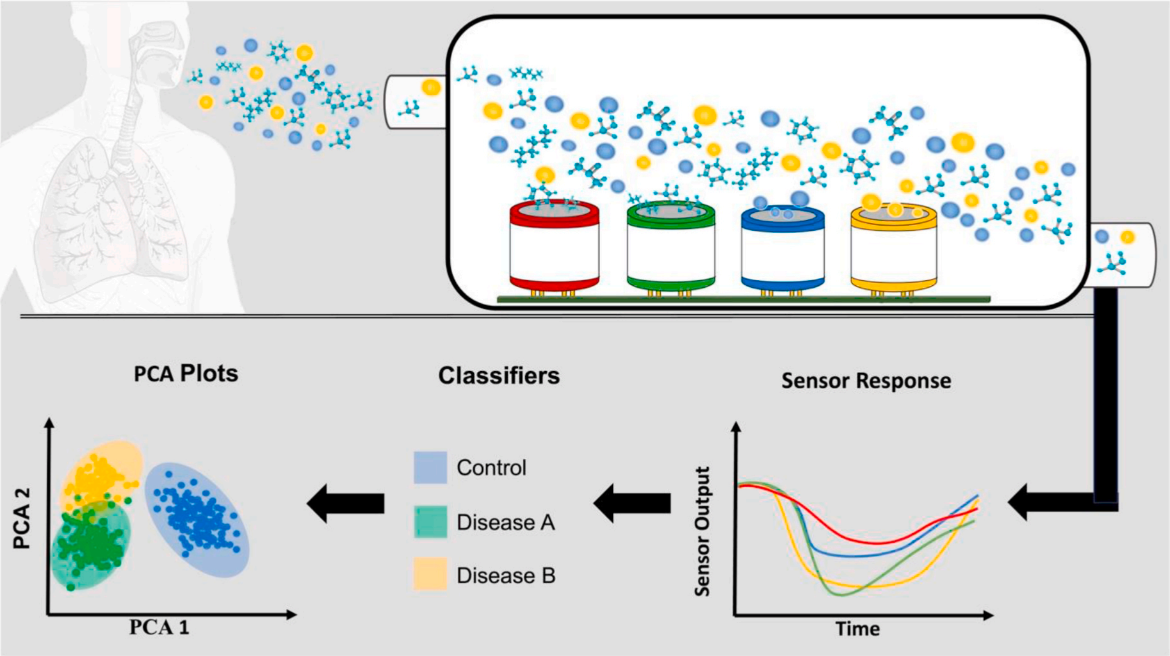

Fig. 2. An overview of a sensor array workflow for breath collection. Image elements modified with permission from Refs. [139,140].

commercially available system. These devices provide a quality-controlled unit with technical support and training. The total cost of these systems can range, but in general these can cost between $10k-$30k, making them relatively accessible for many researchers. These commercial systems can be divided into two groups, those that are designed specifically for breath analysis and those that are more generic instruments adapted for breath research.

Of the generic eNose's, the Sensigent Cyrano A320® is one of the most popular [16]. Released in the early 2000s, this instrument was one of the first portable units [141]. Its use of room temperature conducting polymer arrays meant that its total power consumption was low, allowing for a small form factor with integrated signal processing and machine learning [16]. Moreover, the Cyrano A320® is fully battery powered without the need of a laptop, allowing it to be easily taken into sampling locations [16]. In contrast, the Airsense PEN3® requires an electrical socket due to the ten n-type thick film gas sensors that are high power consuming (sensors are considered "thick" when the sensing layer is greater than 10 μm) [142]. Yet, the form factor is still small, making it easy to fit onto a trolley. The GeNose® was developed to have samples introduced to the device from a separate sampling collection system [143]. This eNose has been targeted towards Covid-19 detection and has similar internal structure and form factor to the PEN3 [143]. Finally, in the last 5 years there has been a single paper using a Bloodhound eNose. This uses an array of 12 conducting polymer sensors. This product is no longer manufactured and has become RoboScientific.

More recently, there has been a small number of commercial eNoses for targeted breath analysis. These are specifically designed to reduce the need for intermediate collection steps (such as sample bags) by having patients breathe directly into the instrument. All of these commercial eNose devices have integrated MOS-based sensors, which can be associated to the advancements in architecture (in both multi-material use and smart algorithms) and design of these sensors in recent years. Of these types of devices, there are two main units: Aeonose® and SpiroNose®. The former, employs three micro-hot plate based analogue MOS sensors that can be thermally modulated at different rates (from 260 to 320 °C) [144]. As the sensitivity of a MOS sensor is temperature dependent and the rate at which molecules absorb and desorb from the sensors is also temperature dependent, the combination of the two significantly increases the information content from a single sensor [145]. By analysing the output with pattern recognition, frequency components can be extracted from the signal response. This unit is handheld (held by both hands) and has a single mouthpiece to which the user blows directly into the unit [146].

The SpiroNose® by BreathOmix has a similar product design as the Aeonose®[147]. Again, the unit is small (though usually not handheld), with the user directly breathing into the system. It also has a built-in spirometer to control flow going into the unit [147]. Originally, the Spironose® was fitted with five Figaro thick-film metal oxide gas sensors, with the most recent unit fitted with seven [147,148]. However, it is not clear if these are thick film based or MEMS. Finally, one very recent development that has been making headlines is the SniffPhone®, a small, palm sized product that integrates Bluetooth for the direct analysis of breath [149]. The unit is fitted with an array of eight gold nanoparticle gas sensors developed by Technion University in Israel [149]. Though currently not commercially available, it shows the miniaturization potential this technology and possible future outlook of commercial eNoses.

4. Sensor-based data analysis in breath

A critical part of an eNose is the analysis of the data from the sensor array. To identify patterns within this data, careful processing alongside the extraction of meaningful features is needed. Various machine learning methods have been applied in literature according to the task at hand. Most works utilise similar methods, with little variation to what has been applied to the analysis of breath data over the last five years. An overview of the data analysis strategies currently implemented in breath analysis is shown in Fig. 3.

Ensuring the quality of raw sensor data is crucial for extracting meaningful information. Raw data collected from sensors often contains noise, artifacts and variations that can obscure the underlying chemical signatures. Various established techniques exist for mitigating these issues, with the moving average window being the most used method [150–152]. To address challenges such as drift, contrast fluctuations, and scaling issues, baseline manipulation proves to be a valuable strategy [153–155]. This involves subtracting the baseline of each sensor from its corresponding response [156]. Signals normalisation or standardisation is often used to mitigate sensor variability and baseline drift [157] The next critical step is feature extraction of chemical sensor data. This process involves transforming raw sensor signals into informative features that capture the characteristics of breath components. There are various methods employed from statistical measures to advanced signal processing techniques [158,159]. Most commonly, time-domain features are used, representing geometric attributes of the signal response [151,152,154,155,160,161,161–168]. Following feature extraction, it is common to apply a feature selection or dimensionality reduction technique. These methods help identify the most relevant features or components of features that encapsulate the information of interest. Feature selection involves using established statistical methods that assess whether there are significant differences among groups of variables. Meanwhile, dimensionality reduction techniques aim to reduce the number of dimensions in a dataset while retaining crucial components of the data with Principal Component Analysis (PCA) being the most frequently used [152,162,164,165,169].

So far, most studies focus on machine learning classification tasks, where sensor readings are categorized into specific classes [170]. In few cases where there are no known classes, an unsupervised clustering approach was taken to identify patterns or structures within the data [152,160,169,171–180]. There are very few cases of multiclass tasks in the context of breath analysis, and to our knowledge no regression tasks [181]. To evaluate the machine learning tasks, a prevalent approach is cross-validated evaluation, using metrics such as sensitivity, specificity, accuracy and AUC-ROC (Area under the Receiver Operator Characteristic Curve). Finally, the analysis of chemical sensor data for breath analysis does present several challenges, with a primary focus on mitigating overfitting [170,182,183]. To mitigate this, further work should consider controlling model complexities. With the emergence of deep neural network architectures, researchers are increasingly exploring their application to breath data signals [184–186]. The exploration of such advanced frameworks holds promise for enhancing the precision and efficacy of breath data analysis in the future.

4.1. Diagnostic progress of commercial arrays

Alongside the technological progress of breath-based diagnostic tools, there has also been considerable headway in their implementation against a variety of diseases and disorders. Table 2 shows a detailed outline of the diagnostic outputs reported utilising commercial sensor

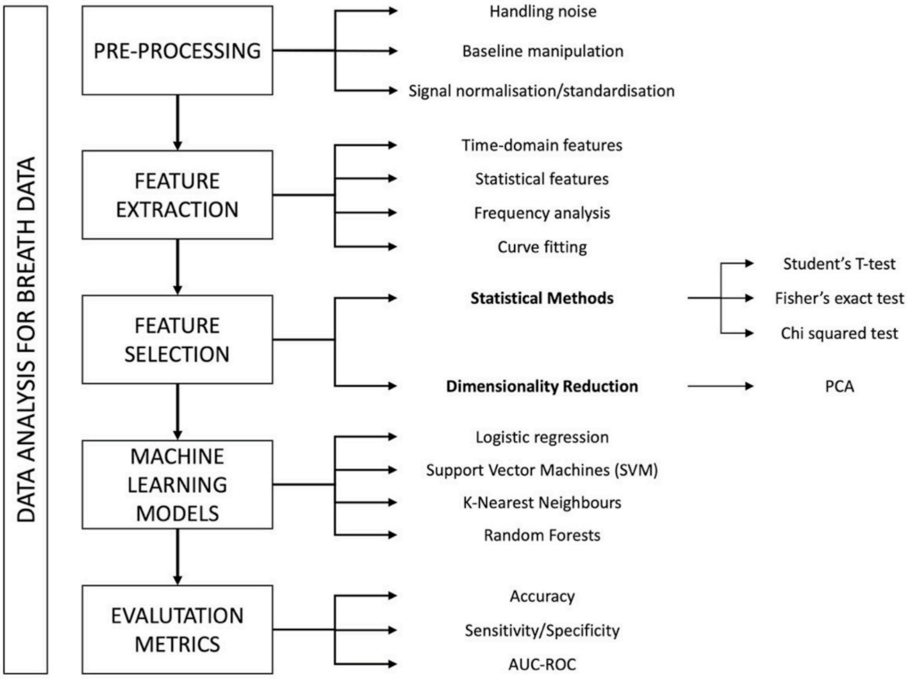

Fig. 3. An outline of the steps taken when analysing breath data captured by chemical sensors and the various common approaches used at each step.

Table 2
An overview of published articles from 2019 to 2023 focusing on diagnostic breath analysis utilising commercial eNose systems.

Location	Disease/ Disorder	Sensor	Sensor Composition	# of patients/ total patients	Sensitivity	Specificity	AUC	Accuracy	Publication	Author(s)	References
Pulmonary											
	Idiopathic Pulmonary Fibrosis	Aeonose®	3 micro hotplate MOS sensors	51/84	88.0 %	85.0 %	95.0 %	–	2019	Krauss et al.	[187]
	Cystic Fibrosis	Aeonose®	3 micro hotplate MOS sensors	20/42	85.0 %	77.0 %	87.0 %	–	2019	Bannier et al.	[188]
	Asthma	Aeonose®	3 micro hotplate MOS sensors	13/35	74.0 %	91.0 %	79.0 %	–	2019	Bannier et al.	[188]
	COPD	Cyranose 320®	32 Carbon nanoparticle composite sensors	116/294	100.0 %	97.8 %	98.6 %	–	2020	Rodriguez-Aguilar et al.	[189]
	Asthma	Cyranose 320®	32 Carbon nanoparticle composite sensors	19/38	79.0 %	84.0 %	80.0 %	–	2020	Tenero et al.	[190]
	SARS-CoV-2	Aeonose®	3 micro hotplate MOS sensors	57/219	86.0 %	54.0 %	74.0 %	62.0 %	2020	Wintjens et al.	[191]
	Ventilator-associated pneumonia	Cyranose 320®	32 Carbon nanoparticle composite sensors	33/59	79.0 %	83.0 %	85.0 %	81.0 %	2020	Chen et al.	[192]
	Idiopathic Pulmonary Fibrosis	Cyranose 320®	32 Carbon nanoparticle composite sensors	32/68	–	–	98.5 %	100.0 %	2020	Dragonieri et al.	[193]
	SARS-CoV-2	PEN3®	10 MOS sensors	27/503	–	–	58.0 %	–	2021	Snitz et al.	[194]
	Small Airway Dysfunction	Cyranose 320®	32 Carbon nanoparticle composite sensors	12/60	92.0 %	95.0 %	98.0 %	–	2021	Tsai et al.	[195]
	SARS-CoV-2	SpiroNose®	7 MOS sensors	298/4593	100.0 %	78.0 %	95.7 %	–	2021	Vries et al.	[196]
	Interstitial lung disease	SpiroNose®	7 MOS sensors	322/370	100.0 %	100.0 %	100.0 %	–	2021	Moor et al.	[197]
	SARS-CoV-2	GeNose C19®	10 MOS sensors	40/83	95.5 %	95.7 %	95.6 %	95.0 %	2022	Nurputra et al.	[198]
	SARS-CoV-2	GeNose C19®	10 MOS sensors	230/460	87.0 %	84.0 %	86.0 %	–	2022	Hidayat et al.	[199]
	Tuberculosis	Aeonose®	3 micro hotplate MOS sensors	91/107	52.3 %	36.4 %	50.0 %	–	2023	Teixeira et al.	[200]
Gastrointestinal											
	Appendicitis	BloodHound®	12 Conducting Polymer sensors	05/50	83.0 %	83.0 %	83.0 %	–	2019	Wong et al.	[201]
	Ventral Hernia	Aeonose®	3 micro hotplate MOS sensors	29/66	79.0 %	65.0 %	74.0 %	–	2020	Mommers et al.	[202]
Neurological											
	Epilepsy	Aeonose®	3 micro hotplate MOS sensors	74/184	76.0 %	67.0 %	77.0 %	71.0 %	2020	Dartel et al.	[203]
	Multiple sclerosis (no meds)	Aeonose®	3 micro hotplate MOS sensors	58/187	93.0 %	74.0 %	80.0 %	–	2021	Ettema et al.	[204]
Lymphatic											
	Sarcoidosis	SpiroNose®	7 MOS sensors	252/300	100.0 %	100.0 %	100.0 %	–	2021	Van der Sar et al.	[205]
Cancer											
	Lung	Cyranose 320®	32 Carbon nanoparticle composite sensors	133/265	96.2 %	90.6 %	–	–	2019	Tirzite et al.	[206]
	Head/Neck	Aeonose®	3 micro hotplate MOS sensors	20/40	85.0 %	80.0 %	85.0 %	–	2019	Van de Goor et al.	[207]

(continued on next page)

Table 2 (*continued*)

Location	Disease/ Disorder	Sensor	Sensor Composition	# of patients/ total patients	Sensitivity	Specificity	AUC	Accuracy	Publication	Author(s)	References
	Lung – PD-1 Therapy	Spironose®	7 micro cross-reactive MOS	65/143	43.0 %	100.0 %	85.0 %	82.0 %	2019	Vries et al.	[208]
	Colorectal	Aeonose®	3 micro hotplate MOS sensors	70/198	95.0 %	64.0 %	84.0 %	–	2019	Keulen et al.	[209]
	Breast	Cyranose 320®	32 Carbon nanoparticle composite sensors	262/443	100.0 %	100.0 %	100.0 %	–	2020	Leon-Martinez et al.	[210]
	Squamous cell carcinoma	Cyranose 320®	32 Carbon nanoparticle composite sensors	22/35	95.0 %	69.0 %	–	–	2020	Fielding et al.	[211]
	Ovarian	PEN3®	10 MOS sensors	28/67	98.0 %	95.0 %	–	96.0 %	2020	Raspagliesi et al.	[212]
	Lung	Aeonose®	3 micro hotplate MOS sensors	91/124	84.0 %	97.0 %	92.0 %	–	2020	Krauss et al.	[213]
	Gastric	SniffPhone®	8 Spherical gold nanoparticle sensors	16/40	100.0 %	87.5 %	93.8 %	–	2021	Leja et al.	[214]
	Breast	Cyranose 320®	32 Carbon nanoparticle composite sensors	351/439	86.0 %	97.0 %	99.0 %	91.0 %	2021	Yang et al.	[215]
	Thyroid Carcinoma	Aeonose®	3 micro hotplate MOS sensors	48/133	73.0 %	69.0 %	76.0 %	–	2022	Scheepers et al.	[216]

arrays from the last 5 years.

Table 3
Examples of commercial sensors used in Breath Analysis systems. Note: not all these sensors are currently available for commercial purchase.

Manufacturer	Technology	Example Sensors Used	Reference
Figaro	Taguchi Style	TGS822 (Organic Solvents) TGS826 (Ammonia)	[219,220]
Figaro	Ceramic Tile	TGS2600 (Air contaminants) TGS2610 (Hydrocarbons) TGS2620 (Organic Solvents)	[221]
Figaro	MEMS	TGS8100 (Air contaminants)	[222]
Hanwei	Taguchi Style	MQ-2 (Hydrocarbons) MQ-3 (alcohols) MQ-9 (Carbon monoxide) MQ-135 (Aromatics) MQ-137 (Ammonia) MQ-138 (Organic Solvents)	[222]
SGX Sensortech	MEMS	MiCS-6814 (Tripple sensor oxidising/reducing gases and Ammonia) MiCS-4514 (Dual sensor for oxidising/reducing gases)	[223]
ScoiSense (previously AMS)	MEMS	AS-MLV-P2 (Reducing gases) CCS801 (Air contaminants) CCS811 (Air contaminants) iAQ-Core C (Organic Solvents)	[223–226]
Bosch	MEMS	BME680 (Air contaminants)	[220,227]
Sensirion	MEMS	SGP30 (Air contaminants) SGP40 (Air contaminants)	[223]

4.2. Custom arrays with commercial sensors

After commercial arrays, the most common strategy is for researchers to develop their own instrumentation using commercial sensors. The reason for this can be three-fold: (1) there were no commercial systems available for their specific need, (2) the cost of the commercial unit was prohibitively expensive, (3) they wanted to deploy a custom operational mode or data processing strategy that would not be possible on a commercial system. Hence, the quickest way to develop such a system is to purchase commercial sensors and fit them into a custom unit. This reduces the development time and relative quality control measures associated with the sensors. The most popular approach is to use commercial MOS sensors. As this is one of the oldest sensor technologies, it is consistently available, has good sensitivity, reasonable stability, and long lifespan [217]. Most MOS sensors are produced by Figaro and FIS in Japan, both of which were founded in the 1960s, and are still used extensively today [218]. Since then, the field has expanded to include companies such as Winsen Sensors making thick film sensors or MEMS based micro-hot plate sensors made by companies such as ScoiSense, SGX, Microtech, and Bosch (Table 3). These later developed sensors offer the opportunity to make smaller and lower powered arrays. Some of the disadvantages of commercial sensors in breath analysis include their limited sensitivity and selectivity, which can result in false positives or false negatives. Commercial sensors may also have limited dynamic range, which can make it difficult to detect low concentrations of VOCs in breath samples, as they were developed for non-breath applications. Finally, commercial sensors may not be able to detect all relevant VOCs in breath samples, again due to what the sensor was originally developed to detect, which can limit their utility in certain breath applications.

4.3. Custom arrays and sensors

The most challenging approach in the development of an eNose for breath research is the design of custom sensors and their integrated systems. Yet, the advantage of developing a custom product that is built specifically for breath can often out-weigh these challenges. These products are also difficult to be copied, thereby ensuring product protection. However, due to the considerable effort, the number of researchers who have created full custom solutions is small. To expand upon this, the following passages will illustrate the complexity necessary to designing custom sensors for breath analysis in two recent studies involving distinctive techniques.

Danjun Wang and colleagues designed custom sensors into a Flexible Virtual Sensor Array (FVSA) [228]. This array incorporated laser-induced graphene interdigital electrodes (LIG-IDEs) and a sensing layer of MXene for the detection of VOCs in human breath. The LIG-IDEs, constructed from highly conductive sp2-hybridized carbon, were fabricated through a cost-effective laser direct writing process on a polyimide (PI) substrate, enabling the conversion of sp3 carbon in PI to LIG. This method ensured easy fabrication, but at an expense. Simultaneously, the MXene sensing layer, composed of a Ti3C2Tx film on the LIG-IDEs' surface, demonstrated high surface area, tuneable surface chemistry, and superior electrical conductivity. As an FVSA, this single sensor generated a multidimensional response close to those obtained using an eNose, demonstrating its diverse sensing capabilities without employing a combination of different sensors. The selection of LIG-IDEs was based on their highly conductive sp2-hybridized carbon structure, while MXene offered a high surface area and tuneable surface chemistry, leading to precise VOC recognition and accurate concentration prediction in varying backgrounds. The FVSA successfully identified alcohol content in human breath samples with an accuracy of 88.9 % through blind analysis.

Furthermore, Hi Gyu Moon and colleagues designed chemiresistive eNose (CEN) sensors based on villi-like metal oxide nanostructures (VLNs) and villi-like nanostructures WO_3 (VLNW) organised in a 2 × 2 array configuration [229]. The array incorporated VLNs, VLNW, and Au-functionalised versions of both sensors, with each type being used twice within the array. These sensor types were chosen for their unique nanostructures, offering enhanced sensitivity to Nitric Oxide (NO) vapor at high humidity levels stemming from their large surface-to-volume ratio and high porosity. Additionally, Au-functionalised VLNs and VLNW were included to assess the impact of gold catalysts on sensor performance. The purpose of these sensor arrays was to detect and identify specific biomarkers like NO and NH_3, which have been previously associated with asthma and kidney disorders. However, the study did not integrate patient data and only demonstrated the CEN's potential through the analysis of the NO and NH_3 through *in vitro* experiments. The results showed good sensitivity towards the VOCs, but additional testing utilising breath samples will be required before its potential for clinical applications can be inferred.

4.3.1. Diagnostic progress of custom arrays

In addition to commercial sensors, as stated above numerous studies have been conducted on the development of novel chemical sensor arrays utilising either custom or commercial sensors. Table 4 shows a detailed outline of the diagnostic outputs reported utilising custom sensor arrays from the last 5 years.

5. Concluding remarks

This review has focused on illustrating the working principles of the chemical sensors that have been utilised in breath analysis. Furthermore, commercial sensor arrays and the development of custom arrays, both with commercial and custom sensors, have been discussed. Finally, an extensive examination of the diagnostic outputs in studies implementing sensor arrays for breath analysis were reported. As can be seen in the diagnostic tables (Tables 2 and 4), there have been numerous studies conducted implementing sensor arrays, both commercial and custom, in the last 5 years. Most of the sensor array studies were focused on pulmonary diseases and cancer. Starting in 2020, many of these studies became focused onto SARS-CoV-2. However, this was not observed in custom arrays where only two studies encompassed custom sensors; none were found utilising commercial sensors. The diagnostic outputs of the studies reported sensitivities, specificities, and AUC values above 80 %, but a few of the commercial sensors were able to achieve 100 % diagnostic capabilities in several diseases. However, the custom sensor arrays had more variable diagnostic outputs than the commercial sensors, particularly associated to specificity.

Overall, the combination of these results showed considerable promise, while also revealing that there are still several major hurdles that must be overcome. The first of these, is that breath is a very different environment to normal air - the changes in humidity, temperature, and oxygen concentration can produce large variances in sensor outputs greater than those observed in chemical concentration of a disease [251]. Although it is possible to mitigate these issues through sampling control, the additional hardware reduces the portability of the unit and increases the overall unit cost. Issues surrounding humidity tolerance are well documented, and presently, most of the sensor elements used in breath analysis have a relative intolerance to humidity, resulting in changes to the baseline and sensitivity [251].

Moreover, most commercial units and custom systems typically rely on existing sensors that can be bought off the shelf. This reduces the development time of the unit, however as these sensors were not developed for this purpose, they often do not have the required sensitivity/specificity needed for breath diagnostics (typically down to ppb to ppt). In addition, it often requires expertise in these sensor technologies to maximise sensitivity and selectivity. Therefore, to create a simple sensor array is relatively easy, but to make a reliable and repeatable system is much harder. Though the potential for breath analysis is huge, it is still small compared to other market sectors, such as safety, reducing the drive for sensor manufacturers to produce target sensors. To increase sensitivity, it is possible to add some form of pre-concentration to the unit, but again, this adds complexity and cost to the final product. Also, the pre-concentrator needs to be designed to deal with the changes in environmental conditions produced by breath. Some groups have attempted to get around this by developing their own sensors and sensor system. In this case, the sensors can be tailored to have a high sensitivity for target breath molecules. The downside is that it is difficult for other researchers to replicate these sensors and even harder to move these sensors into commercial manufacturing. The time for initial development to market often takes many years. This is simply a result of the challenges in upscaling and the need for quality control changes, repeatability, etc.

In conclusion, the use of sensors arrays used in breath analysis shows considerable diagnostic promise. Sensors can be made cheap (sub $20 per sensor), small, portable, battery powered, and can use air as the carrier gas. This makes the widespread use of these devices achievable without the considerable high cost of analytical techniques such as GC-MS that are currently the gold standard in breath analysis. Continued multi-disciplinary work will be necessary to develop stable sensors for direct breath analysis that can be readily manufactured. However, with the current rapid advancements in sensor technology, its miniaturization, improvements in sensor operational modes, and the use of machine learning to extract data from the sensors, it is likely that the tools will be more available to allow researchers to achieve the potential for breath analysis.

CRediT authorship contribution statement

Trenton K. Stewart: Writing – review & editing, Writing – original draft, Methodology, Investigation, Data curation. **Ines E. Carotti:** Writing – review & editing, Writing – original draft, Investigation, Data curation. **Yasser M. Qureshi:** Writing – review & editing, Writing – original draft, Investigation, Data curation. **James A. Covington:** Writing – review & editing, Writing – original draft, Supervision, Resources, Project administration, Data curation.

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.

Table 4
An overview of published articles from 2019 to 2023 focusing on diagnostic breath analysis utilising either custom or commercial sensors in novel arrays.

Location	Disease/ Disorder	Sensor	Sensor Composition	# of patients/ total patients	Sensitivity	Specificity	AUC	Accuracy	Publication	Author(s)	References
Custom Sensors											
Pulmonary											
	Alveolar echinococcosis	Metal nanoparticle array	8 ligand-metal nanoparticle sensors	14/22	92.9 %	88.9 %	–	92.9 %	2019	Welearegay et al.	[230]
	SARS-CoV-2	Nanomaterial Sensor Array	8 gold nanoparticle Sensors	33/91	100.0 %	61.0 %	81.0 %	76.0 %	2020	Shan et al.	[231]
	Respiratory Failure by Sars-CoV-2	MOS Sensor Array	4 custom MOS sensors	25/37	92.0 %	68.2 %	81.0 %	–	2022	Bax et al.	[232]
	SARS-CoV-2	Nanomaterial Sensor Array	64 nanomaterial sensors	54/64	66.0 %	68.0 %	–	–	2022	Duanmu et al.	[233]
Metabolic											
	Diabetes	Graphene Composite Array	GO/SnO2/TiO2 Sensor	17/30	70.1 %	69.2 %	–	60.5 %	2019	Kalidoss et al.	[234]
	Diabetes	Polymer QTF Array	3 Modified QTF Sensors	5/70	–	–	–	95.3 %	2022	Parmar et al.	[235]
Cardiovascular											
	Coronary artery disease	Nanomaterial Sensor Array	12 gold nanoparticle Sensors	50/85	69.0 %	67.0 %	68.0 %	–	2022	Agmon et al.	[236]
Hepatic											
	Liver Cirrhosis	Semiconductor/ nano array	5 commercial semiconductor/ 6 nanomaterial sensors	22/54	–	–	96.5 %	–	2021	Zaim et al.	[237]
Cancer											
	Lung	MOS Sensor Array	6 MOS sensors	65/118	95.0 %	100.0 %	97.5 %	97.2 %	2019	Kononov et al.	[238]
	Gastric	Nanomaterial Sensor Array	5 gold nanoparticle sensors	99/441	82.0 %	78.0 %	88.0 %	79.0 %	2019	Broza et al.	[239]
	Lung	Nanomaterial Sensor Array	WO_3 nanowire sensors	32/44	–	–	–	98.6 %	2020	Saidi et al.	[240]
	Lung	rGO-M Array	Reduced Graphene Oxide Sensors	48/108	95.8 %	96.0 %	99.6 %	–	2020	Chen et al.	[241]
Commercial Sensors											
Pulmonary											
	Tuberculosis	MOS Sensor Array	16 MOS sensors	24/51	95.0 %	82.0 %	92.0 %	–	2021	Iswari Saktiawati et al.	[242]
	COPD	MOS Sensor Array	5 MOS sensors	38/110	81.6 %	95.8 %	–	90.0 %	2021	Binson et al.	[243]
Metabolic											
	Diabetes	Composite Gas Array	6 Composite gas sensors	20/30	–	–	–	96.0 %	2020	Sarno et al.	[244]
	Diabetes	Custom Composite Array	6 MOS + 2 SnO2 + DHT22 Sensors	62/100	–	–	–	86.6 %	2023	Kapur et al.	[245]
Gastrointestinal											
	Inflammatory Bowel Disease	WOLF eNose	10 electrochemical sensors + 2 NDIR + photo-ionisation	30/39	87.0 %	89.0 %	93.0 %	–	2019	Tiele et al.	[246]
Cardiovascular											
	Coronary artery disease	Electrochemical array	19 electrochemical sensors	22/48	82.6	80.2 %	–	81.5 %	2021	Tozlu et al.	[247]
Urinary											
	Chronic Kidney Disease	Single Sensor	MQ-135 semiconductor sensor	21/38	85.7 %	64.7 %	86.0 %	76.3 %	2019	Umapathy et al.	[69]
Cancer											
	Lung	Chemirestive	4 MOS sensors	6/16	85.7 %	100.0 %	–	93.8 %	2019	Marzorati et al.	[248]
	Lung	MOS Sensor Array	5 S_nO_2 MOS sensors	40/80	–	–	–	66.0 %	2021	Marzorati et al.	[217]
	Lung	Custom Composite Array	7 MOS + 2 electrochemical + 2 additional sensors	98/214	92.1 %	95.1 %	–	93.5 %	2021	Liu et al.	[249]
	Lung	MOS Sensor Array	5 MOS sensors	32/104	84.4 %	93.1 %	–	90.4 %	2021	Binson et al.	[250]

Data availability

No data was used for the research described in the article.

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