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REVIEW PAPER

Carbon Dioxide (CO_2) Sensors for the Agri-food Industry—A Review

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Abstract In the food and agricultural industry, sensors are being used for process control, monitoring quality, and assessing safety. There is a growing demand for carbon dioxide (CO_2) sensors in the bulk food storage sector, because CO_2 sensors can be used to detect incipient spoilage and to assess CO_2 levels in modified-atmosphere packages and storage structures. The market potential for reliable and inexpensive CO_2 sensors is huge because of a wide range of applications in the agri-food industry. This review synthesizes information about the types of CO_2 sensors, analyzes their detection processes, provides a broad overview of the innovative research on the development of sensors, sensing mechanisms, and their characteristics, and outlines future possibilities for use of CO_2 sensors.

Keywords CO_2 sensors · Optical sensors · Electrochemical sensors · Metal oxide sensors · Polymer sensors

Introduction

The evolving agriculture and food system has entered in to a consumer driven era with consumers demanding food safety, quality, and convenience. To analyze, design, develop, manage, control, and characterize the biological and environmental processes in agricultural industry, there is a need to collect data. This has necessitated the food and agricultural industry to increasingly rely on sensor technology. For example, sensors are used in the field for monitoring of environmental parameters to help producers to conduct more efficient irrigation or pest control programs, on harvesting machinery for measuring yield per unit area, during storage for measurement of product temperature, for post harvest grading and sorting of fruits and vegetables, and for online monitoring of process parameters during processing. Hence, reliable data collecting sensors are essential for agricultural applications and real-time decision-making.

Sensors for the agricultural and food industry exhibit several differences compared to the traditional industrial sensors in terms of the measurement parameters as well as the environment. For online monitoring of gases in the agricultural and food industry, the sensors should be capable of working under extreme temperatures, pressures, and various other parameters. Environment surrounding raw as well as processed agricultural materials is inherently complex. Presence of multiple microorganisms and other biological agents further makes the sensing of parameters in agri-food industry very challenging. As the agri-food processes are highly variable, the sensors require the capability to handle this variability. Also, the users should be able to interpret easily the computational data from the agricultural sensors. Therefore, intelligent monitoring of CO_2 is becoming important in number of agro-based applications such as plant gas exchange, atmospheric gas monitoring, soil CO_2 flux, biogas composition monitoring (Rego and Mendes 2004), freeze damage detection in

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oranges (Tan et al. 2005), processing of alcohol and beverage industry (Marazuela et al. 1998), and for ethanol and urea production.

Food packaging is often done under a modified atmosphere of nitrogen (N_2), CO_2, and oxygen (O_2), specific to a product and with a purpose to prevent microbial spoilage. Carbon dioxide is widely used in modified-atmosphere packaging, and a decrease in its concentration is a sign of leakage in a package. Freshness and safety of modified-atmosphere food packages can be determined by detection of CO_2 concentrations (Smolander et al. 1997). High CO_2 levels can affect quality of French fries during processing, and therefore, monitoring of CO_2 levels can be used for controlling ventilation in potato storage facilities (Jayas et al. 2001). Instruments capable of sensing CO_2 concentrations of 0.1% will detect spoilage during stored grain in 80% of deteriorating bulks in farm granaries (Singh et al. 1983; Muir et al. 1985). Detection and measurement of extreme levels of CO_2 atmospheres by suitable sensors can protect the safety and health of farmers in agricultural-confined spaces such as manure storage facilities and grain silos (USDA 1996). Hence, there is a significant demand for CO_2 sensors in the bulk food storage sector such as ventilation control, industrial incubator monitoring, food transportation, food processing, food quality monitoring, and horticulture.

Global gas sensor market is US $320 million per year (MNT 2006). The projected market revenue for gas sensors in the year 2009 will be US$3.8 billion (BCC Research 2003; Frost and Sullivan 2000). The Kyoto protocol and the increased awareness on monitoring environmental parameters has fuelled sensors research and encouraged the need for novel smart CO_2 detectors (Coppock 1998). The market potential for CO_2 sensors is huge because of a wide range of applications in the agri-food as well as in other industries. There are limited research articles emphasizing the details of sensors and technology for the detection of CO_2 in the agri-food sector. This review provides details about the CO_2 sensors and their sensing mechanisms. In addition, it provides a broad overview of the innovative research on development of sensors. Both commercial and laboratory research investigations of new and emerging CO_2 sensors and key challenges to current sensor developments are presented.

Carbon Dioxide Sensors

The term sensor is defined as a device or system, including control and processing electronics, software, and interconnection networks that respond to a physical or chemical quantity to produce an output that is measurable and is proportional to the quantity (e.g., gas concentration, ionic strength measurement; Lees 2003).

In general, most sensors consist of four major components: (1) a sampling area where the surface chemistry occurs, (2) the transducer, (3) signal processing electronics, and (4) a signal display unit. The two major functions in the gas sensing systems are the recognition of the molecule and transduction of that recognition event into a useful signal. The transducer helps in the conversion of one form of energy to another. The chemical reaction at the sampling area produces a change in parameter such as pH, resistance, conductivity, or capacitance based on the exchange of electrons of ion reactions between the analyte and the surface material (Cattrall 1997).

A number of simplified sensing techniques have been proposed and developed for the routine measurement of CO_2 concentrations. The most common system for CO_2 detection are as follows: Severinghaus type potentiometric sensors (Severinghaus and Bradley 1958), gas chromatography (GC) and mass spectrometers (MS; Sipior et al. 1996), and infrared absorption detectors.

Severinghaus CO_2 sensor consists of a glass electrode filled with an aqueous bicarbonate solution and covered by a thin plastic membrane. This plastic membrane is permeable to CO_2 but impermeable to water and electrolytes. The measuring principle is based on the fact that in an aqueous solution, CO_2 forms carbonic acid, which dissociates into a bicarbonate anion and a proton (Dieckmann and Buchholz 1999). This process results in the pH-value change of the electrolyte solution, which is measured by a pH probe. The main drawback of this type of sensor is that CO_2 is not measured directly, but in its ionic form. Additional disadvantages are that other volatile acid or basic gases could impair the pH-value measurement, and the maintenance costs are very high.

The working principle of GC is selective adsorption and elution of the gas molecules of interest in the GC column. In MS, the sample gas is bombarded with high-energy electron beam causing fragmentation of the molecules in the sample. These fragmented molecules are made to move through a magnetic field because of their charge and are separated based on their charge and mass (Karasek and Clement 1988). Mass spectrometer is capable of producing measurements in near real time, but it is very expensive, while GC is of low cost but requires much longer time to complete an analysis. Hence, both GC and MS cannot be used for real-time measurements. Field portable miniaturized GC-MS are currently under development, focusing on solving the instrumentation, miniaturization, deployment, and sampling issues. A qualitative match purity of 75% is required for positive identification of compounds (CTEH 2007).

Based on the sensing mechanism, CO_2 sensors can be broadly classified as optical and electrochemical sensors.

Optical Sensors

The most commercially available CO_2 sensors are infrared detectors. Infrared detectors produce quick response times and are reliable but can only be used for gaseous CO_2 monitoring. Most of the commercially available CO_2 sensors (Model No. GMT220 Carbocap, Vaisala; Model No. TGS4160, Figaro; Model No. 9510, Alphasense) are nondispersive infrared detectors. The advantages of infrared gas sensors over analytical instruments such as GC-MS include cheaper cost, compact size, easy process control, easy mass production, and continuous measurement (Lee and Lee 2001).

Nondispersive infrared detector (NDIR) is simpler in structure and easy to use. The working principle is based on the energy absorption characteristics of CO_2 in the infrared region. All gases selectively absorb infrared light energy, which corresponds to their own quantized vibrating energy. Carbon dioxide absorbs infrared radiation at wavelengths of 2.7, 4.3, and 15 μm (Skoog 1985). In a typical NDIR sensor, infrared energy passes through two identical tubes (Fig. 1). One is a reference tube, and it is filled with nonabsorbing gas such as nitrogen. The second tube contains the gas sample to be analyzed and is a measurement cell. Filters or monochromators are used in the NDIR to obtain a monochromatic beam from an infrared light source with a wide wavelength range. This light is allowed to pass through both the reference and the measurement cells. The gas in the measurement cell absorbs the infrared light, attenuating the energy that falls on the detector. This attenuated energy is compared to the unattenuated signal from the reference cell. The difference is proportional to the amount and the concentration of gas in the measurement cell.

The disadvantages of NDIR sensors are that they are bulky and expensive. Changes in the level of infrared energy in the system, source illumination variation, light scattering from particulates, and thermal drift are some of the measurement errors in the NDIR sensors (Kinkade 2000). Regular calibration with zero gas such as N_2 becomes essential for accurate measurements. The weatherproof cover or the probe tubing becomes necessary for gas sampling. Detection of CO_2 sensing based on its strong absorption in the infrared region has further disadvantages

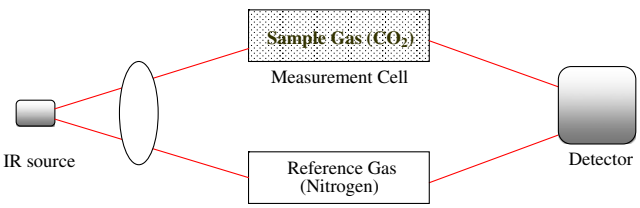

Fig. 1 Schematic showing the working principle of NDIR carbon dioxide sensor

of interference from water vapor and carbon monoxide (Herber et al. 2005).

Fiber Optic CO_2 Sensors

Fiber optics is another optical method for detecting CO_2. These types of sensors are called optodes. Fiber optic sensors contain a chemical sensing layer at the tip of a fiber, which changes optical properties in response to CO_2.

Optical-fiber-based CO_2 sensors have been fabricated by several researchers (Colin et al. 2003; Segawa et al. 2003; Nakamura and Amao 2003) using materials showing absorbance or reflectance changes on exposure to CO_2. The sensing elements such as pH indicating organic dyes or organometallic complexes supported in gas-permeable polymer films have been used. When the sensing element is exposed to CO_2, it creates a change in absorbance or reflectance of the film. After exposure, the sensing spot is examined by an amber light-emitting diode (LED) with peak emission at specified wavelength corresponding to the basic form of the reagent. The LED light shining through the film generates heat at the spot by the nonradiative decay of excited states, resulting in an expansion of the spot and a stress on the polymer film. This creates a charge on the film, and the charge is correlated with the concentration of CO_2 gas.

Mulrooney et al. (2006) developed a fiber optic CO_2 sensor using a pyroelectric detector coupled to the chalcogenide fiber, which converts the changes in incoming infrared light to electric signals. Pyroelectric materials are characterized by having spontaneous electric polarization, which is altered by temperature changes as infrared light illuminates the elements. Pyroelectric CO_2 detectors are suitable for only room-temperature operation.

Mahmoudi et al. (2007) have demonstrated that porous silicon coated with hydrocarbon groups (CH_x) annealed at different temperatures can emit a blue light in response to CO_2 gas. Stabilization of the photoluminescent properties of the porous silicon in an actual sensor device has not yet been established and is undergoing further research.

The advantage of fiber optic gas sensor is that it is chemically inert and not cross-sensitive to the presence of other gases in the background. A fiber optic gas sensor has the drawback of using lenses, which results in interference of transmission of light over time because of dust or soot coating. Hence, a fiber optic sensor needs frequent cleaning of lenses for continuous functioning. Expensive advanced readout equipments are necessary to sense the change at the tip of the fiber.

Sol–Gel Optical Sensors

Bultzingslowen et al. (2002) have fabricated an optical sol–gel-based CO_2 sensor strip using fluorescent pH indicator.

The sensor membrane consists of a ruthenium dye incorporated in polymer nanobeads, and by means of ratiometric method, the analyte-sensitive fluorescence intensity signal is measured. This sensor strip can be located inside a food package and can be sampled by a hand-held scanning device from the outside using wireless technology.

Electrochemical Sensors

To address the need for compact sensors in space research, fire detection, emission monitoring in aerospace applications, medicine, and biology, considerable research is being carried out using nanotechnology and microelectronics to enhance the performance of sensor devices. All the chemical sensors depend on interactions occurring at the atomic and molecular level. Both nano and MEMS (micro-electro-mechanical systems) technologies provide ways to measure variables that until recently were too difficult or expensive to sense. Hence, the potential of nanosensors and nanoenabled sensors is promising in gas sensing applications (Nagel and Smith 2003). Substantially smaller size, lower mass, more modest power requirements, greater sensitivity, and better specificity are the potential benefits of exploiting microelectronics and nanotechnology in sensor fabrication.

Electrochemical or solid state electrolyte sensors are the most predominantly available sensors, which make use of the MEMS and nanotechnology for CO_2 monitoring. These sensors can be classified into ampero-, conducto-, and potentiometric based on the measurement method. In the potentiometric mode, the measured signal is an electromotive force, while in the amperometric mode, an electric current is recorded. In the case of conductometric sensors, the current–voltage plot is analyzed. The materials used to construct sensors should work under extreme conditions such as high temperature and corrosive media.

Based on the variety of principles and materials (metal oxides, polymers, ceramics, or sol–gel), solid state sensors are the best candidates for the development of commercial gas sensors (Capone et al. 2003; Moseley 1997). Solid state electrolyte sensors have numerous advantages such as small sizes, high sensitivity in detecting very low concentrations (at level of parts per million or even parts per billion), possibility of online operation, and due to possible large-scale production, low cost. But these types of sensors also suffer from limited measurement accuracy and problems of long-time stability.

Metal Oxide Sensors

A metal oxide gas sensor consists of a sensitive layer (sensing material) deposited over a substrate provided with electrodes for the measurement of the electrical characteristics. The sensor element is heated by its own heater, which is separated from the sensing layer and the electrodes by an insulating layer (Fig. 2). This heating element can be a platinum or platinum alloy wire, a resistive metal oxide, or a thin layer of deposited platinum. The electrodes connect to the sensing material to form a closed-loop circuit.

The mechanism of gas detection of metal oxide sensors is based on the chemical reactions that occur on the sensor surface. In the presence of gas, the metal oxide causes the gas to dissociate into charged ions or complexes, which results in the transfer of electrons. This causes a conductance or resistance change of the surface layers when exposed to the analyte. This change in conductivity is directly related to the amount of CO_2 present in the environment, resulting in identification and quantitative determination of the gas concentration. The built-in heater, which heats the metal oxide material to an operational temperature range that is optimal for the gas to be detected, is regulated and controlled by a specific circuit (Datta 1992; Barsan and Weimar 2001). A pair of biased electrodes is imbedded into the metal oxide to measure its conductivity or resistivity change. Typically, the sensor will produce a very strong signal at high gas concentrations.

Resistive metal oxide gas sensors are expected to generate revenues of US $2.5 billion by 2010 (Hooker 2002). In the MEMS-based microelectronics sensor design, selection of sensing material is an essential criterion. Several materials have been reported to be used as sensing material for detecting CO_2 (Table 1).

NASICON Sensors

NASICON powder ($Na_{2,8}Zr_2Si_{1,8}P_{1,2}O_{1,2}$), also a metal oxide, has been proposed to be used as a promising solid electrolyte in electrochemical sensing of CO_2. High ionic conductivity, high chemical stability, and the less influence of humidity on NASICON makes it a preferred material for

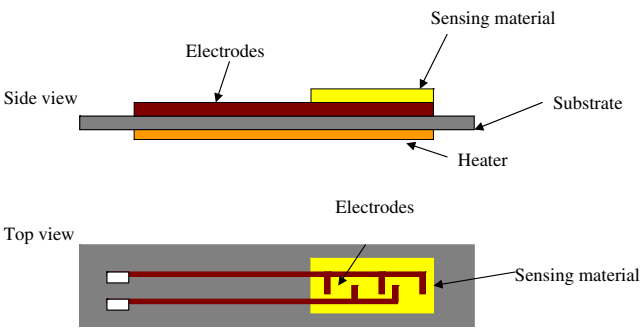

Fig. 2 Schematic showing components used in a typical metal oxide sensor

Table 1 Characteristics of metal oxide sensor materials for detecting CO_2

Types of sensing materials	Detection range	Operating temperature (°C)	Reference
$BaTiO_3$–CuO	0–2,000 ppm	400	Mandayo et al. (2006)
LaOCl and SnO_2	400–2,000 ppm	350–550	Diagne and Lumbreras (2001)
$LaCl_37H_2O$–SnO_2	0 to 2,500 ppm	400	Kim et al. (2000)
CrO–BaTiO3–La203–CaCO3	0% to 7%	700	Haeusler and Meyer (1996)
CuO–$BaTiO_3$ and NiO–$BaTiO_3$	100 to 6,000 ppm	800	Ishihara et al. (1991)

CO_2 sensing (Jasinski et al. 2006). Several researchers (Zhu et al. 2005; Pasierb et al. 2004) around the world, including National Aeronautics and Space Administration (NASA), United States, have proposed that NASICON is the best sensing material for CO_2 sensors (Table 2).

The advantages of semiconducting metal oxide gas sensors over competing technologies such as conducting polymers, electrochemical, and quartz crystal are robustness and long-term stability (Williams and Pratt 2000).

Most commercially available microfabricated sensors (e.g., Cole-Palmer; Figaro) are made of a substrate heated by wire and coated with a metal oxide, semiconducting film. The sensors rely on the changes of conductivity induced by the adsorption of gases and subsequent surface reactions. These micromachined sensors operate at a relatively high temperature of 200–450 °C, which results in significant power consumption. The sensors cannot function without small heaters (usually platinum or gold) on the back side to keep the sensors at operating temperatures. Because of the high operating temperatures, metal oxide sensors are inappropriate in potentially flammable environments (Dickinson et al. 1998) or explosive environment such as grain stores. Metal-oxide-based gas sensors are not recommended to monitor quality of food, because irreversible binding of sulfur compounds may cause poisoning (Schaller et al. 1998). The output of the metal oxide gas sensors varies logarithmically with the gas concentration. This limits the accuracy of the sensor and the overall measurement range of the sensor. Metal oxide sensor performance can also be affected by device cross-sensitivity to environmental factors such as temperature and humidity. Though the metal oxide sensors are relatively low in cost, the stability and repeatability of the sensor are poor.

Polymer-Based CO_2 Sensors

Polymer-based CO_2 sensors are mostly miniaturized version of the Severinghaus CO_2 probe. The advantage of polymer-based gas sensor over metal oxide sensor is that polymer films are capable of broadly detecting and identifying various constituents in the air in addition to target analyte. Hence, polymer sensors are multifunctional, and in general, polymer-based sensors are used in an array.

Tongola et al. (2003) fabricated a pH indicating polypyrrole-based CO_2 sensor. Carbon dioxide diffusing through the polypyrrole membrane forms carbonic acid, which dissociates and alters the pH of the internal electrolyte. A potentiometric probe made of bicarbonate-doped polypyrrole membrane as the pH-sensing device detects the changes in concentration of the gas. An X-ray photoelectron spectroscopy analysis showed that the material displayed poor repeatability and reproducibility of potentiometric responses.

Oho et al. (2002) developed a CO_2 sensor operating under high humidity using polymer composite such as poly vinyl alcohol and poly aniline anthranalic acid. The developed sensor was able to detect CO_2 in the range between 300 and 150,000 ppm under a relative humidity of 80%. A polyaniline thin film deposited on gold electrodes

Table 2 Characteristics of NASICON sensing materials for detecting CO_2

NASICON sensing material	Detection range	Operating temperature (°C)	Reference
NASICON/Li_2CO_3–$BaCO_3$	100–1,000 ppm	345	Zhu et al. (2005)
NASICON/Li_2CO_3–$BaCO_3$	500–12,600 ppm	475	Pasierb et al. (2004)
NASICON/Na_2CO_3–Ba_2CO_3	0–4%	425	Ward et al. (2003)
NASICON/Na_2CO_3–Ba_2CO_3	6 ppm—100%	600	Wang and Kumar (2003)
NASICON/Li_2CO_3	350–1,000 ppm	450	Kaneyasu et al. (2000)
NASICON/Perovskite oxides	100–2,000 ppm	300	Shimizu and Yamashita (2000)
NASICON	0–5%	450	Yang and Liu (2000)
NASICON/Li_2CO_3–$BaCO_3$	300–5,000 ppm	400	Lee et al. (1995)

showed an increase in electrical conductivity when exposed to CO_2 gas (Irimia-Vladu and Fergus 2006; Takeda 1999).

Cui et al. (1998) developed a CO_2 gas sensor system using polyaniline-deposited platinum electrode surface. This sensor can be used for dissolved CO_2 in aqueous phase environments. Modifications using gas-permeable membrane and reference electrodes can make this sensor usable for gas phase applications.

Conclusions

Development of suitable and application-specific sensors for use in the food and agricultural industry is still a challenge. Researchers at the Canadian Wheat Board Centre for Grain Storage Research, University of Manitoba, Winnipeg, Canada are currently developing CO_2 and odor sensors using conducting polymers for grain and food quality monitoring.

Characteristics of a good sensor includes sensor reversibility, accuracy, repeatability, ease of use for nontechnical users, reliability, data gathering, power sources, long-life time energy source, and zero cross sensitivity. Interface requirements, heat dissipation, and the need to deal with electrical and mechanical interference and the noise (Nagel and Smith 2003) are the issues to be considered for efficient sensor design.

Flow control is very critical in the sensors into which gaseous analytes flow through. The surfaces of the sensors are prone to degradation from the effects of foreign substances, heat, humidity, and dust.

Material engineers and chemists are working on developing and characterizing new gas-permeable membranes to solve the water vapor and humidity interference on the sensing layer. The sensor selectivity can be fine-tuned over a wide range by varying the material structure (crystalline or amorphous) and morphology, dopants, contact geometries, operation temperature, or mode of operation. Leaching of the sensor chemistry into the food product can be avoided by proper packaging of the sensor components.

Nanoenabled and microelectronics-based CO_2 gas sensors market is in its early stage and expected to grow rapidly. A number of challenges need to be overcome before the microelectronic gas sensors can be deployed for real-time use. The semiconductor technologies for designing the chips have been advanced, but the integration of electronic designs are underway. Sensor fusion will be the next stage of development.

In the future, sensors will be wireless and will provide real-time process data monitoring for stored food quality and environment with unprecedented accuracy. In all cases, cost is an overriding parameter in the deployment.

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