TELKOMNIKA, Vol. 13, No. 4, December 2015, pp. 1133~1144
ISSN: 1693-6930, accredited A by DIKTI, Decree No: 58/DIKTI/Kep/2013
DOI: 10.12928/TELKOMNIKA.v13i4.2047

Fundamental Review to Ozone Gas Sensing Using Optical Fibre Sensors

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Abstract

The manuscript is a review of basic essentials to ozone gas sensing with optical methods. Optical methods are employed to monitor optical absorption, emission, reflectance and scattering of gas samples at specific wavelengths of light spectrum. In the light of their importance in numerous disciplines in analytical sciences, necessary integral information that serves both as a basis and reference material for intending researchers and others in the field is inevitable. This review provides insight into necessary essentials to gas sensing with optical fibre sensors. Ozone gas is chosen as a reference gas. Simulation results for ozone gas absorption cross section in the ultraviolet (UV) region of the spectrum using spectralcalc.com simulation have also been included.

Keywords: *Absorption spectroscopy, Beer- Lambert law, optical fibre, optical method, ozone gas, sensors*

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1. Introduction

In the field of analytical sciences, optical methods have become very relevant to numerous disciplines [1]. Optical methods are employed to monitor optical absorption, emission, reflectance and scattering of gas samples at specific wavelengths of light spectrum [2]. Newton's discovery of the solar spectrum in 1966 is considered to be the beginning of spectroscopy [3]. The entire spectrometric methods solely rely on emission or absorption of electromagnetic radiation [4]. Optical method relevance to science and other disciplines has made it necessary to put together in one piece essential fundamentals which could be a ready guide for all users. The necessity of a review manuscript which is intended to be a reference material is inevitable. This review provides insight into vital fundamentals to gas sensing with optical fibre sensors. It is comprised of optical sensor mechanism [5], advantages of optical sensors [6, 7], optical sensor classification [8], optical gas cells classification [9], Beer-Lambert law [10] and ozone gas and its research challenges [11-13]. Ozone is a trace gas in the atmosphere [14] and is discovered in 1839 [15]. Ozone is a useful gas, but it is a threat to human life [16-19]. Ozone gas relevance has been previously emphasised by the authors [13]. Significant volume of research activities which are not just limited to detection and monitoring are devoted to ozone gas [20-28]. These activities are summarised in Figure 1. Relevant simulation software (spectracalc.com) was used to obtain simulation results for ozone gas absorption cross section.

2. Mechanism of Optical Sensors

"An Optical Sensor (OS) is a photonic system in which an input signal (Ui), modulates certain characteristics (absorption, dispersion, reflection, transmission, etc) of light in an optical system, such that after detection at the receiver, it is also processed and conditioned, the system will deliver an output electrical signal (Uo), which will be an exact reproduction of the object variable. If any of the processes or parts of it use fibre optic technology, a subgroup of the optical sensor known as Optical Fibre Sensors (OFS) or Fibre-Optic Sensors (FOS), is created" [29].

Received May 21, 2015; Revised September 8, 2015; Accepted September 27, 2015

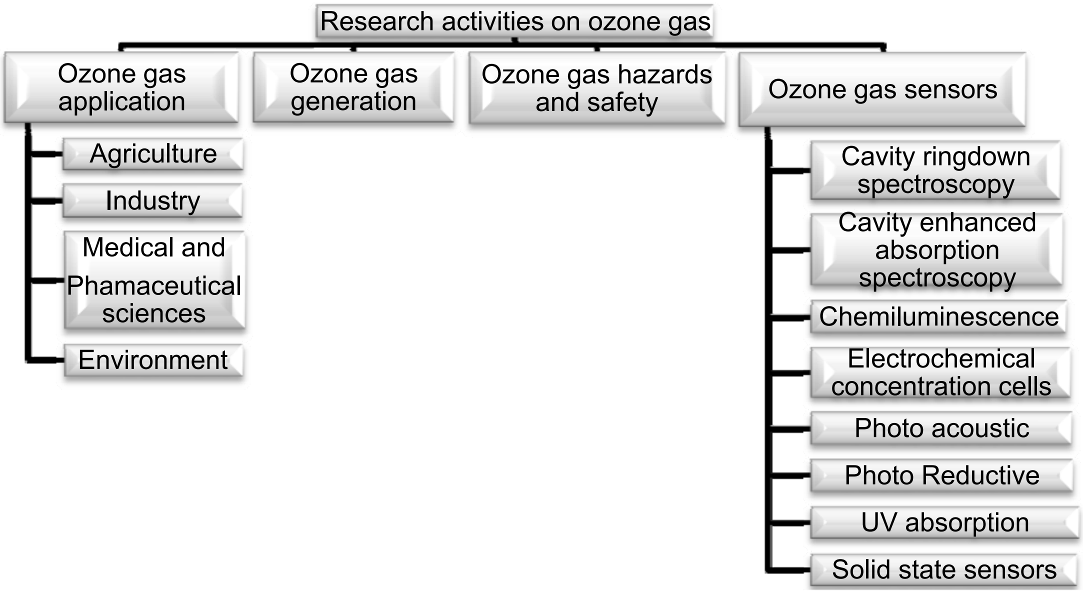

Figure 1. Research Activities on Ozone gas

The interaction of light with matter can be in any one of the following ways: absorption, diffraction, dispersion, reflectance, and interference [1]. Electromagnetic radiations are absorbed by chemical compounds containing covalent bonds. This is as a result of different mechanisms whose effects are seen throughout the electromagnetic spectrum [5]. Absorption of light by a molecule at a given frequency is caused by electron resonance at that given frequency [5, 30]. Light absorption by ozone gas in ultra violet (UV) region (200 to 400 nm, 6.2 to 3.0 electron volt ((eV)) as well as in the visible region (400 to 780 nm, 3.1 to 1.6eV) of the light spectrum [5, 31] is caused by excitation of valence electrons in the atoms of molecules. Light absorption in microwave region (0.3 to 300 cm,) is due to a change in rotation of the bonds in a molecule. Absorption of light in the infrared region (3 to 50 μm, 0.4 to 0.025 eV) and near infrared (0.78 to 3 μm, 1.6 to 0.4 eV) occurs due to the vibration of the bonds of a molecule [32].

While discussion of this paper is focus on absorption spectroscopy, there are other classes of optical sensors such as:

- Reflection spectroscopy [33, 34]
- Luminescence intensity spectroscopy [35]
- Fluorescence lifetime spectroscopy [36]
- Refractive index spectroscopy [37]
- Surface Plasmon resonance or ellipsometric spectroscopy [38]

The classifications are meant to give a clear picture and are not discussed further. Ozone, the gas of interest in this article, absorbs light intensity and hence absorption spectroscopy is dwelled upon in other sections of this article.

Measurement of radiation absorbed by atoms is described as atomic absorption spectroscopy (3). The history of optical sensors can be traced back to when pH indicator strips were developed by immobilizing pH-sensitive indicators on cellulose. The absorption spectrum of each species is unique and can be used to identify and quantify presence of that specie.

3. Merits of Fibre Sensors

The authors have previously [13] highlighted quite a number of different methods for detecting ozone gas such as: cavity enhanced absorption spectroscopy (CEAS) [39, 40], cavity ring down spectroscopy [41], chemiluminescence [42, 43], electrochemical concentration cells [44], photo-acoustic sensors [45, 46], photo reductive [47], solid state sensors [48] and UV absorption [49]. Authors of reference [50] have shown the compatibility of fibre sensors with optical communication systems and their application in electrical noisy systems and explosion prone scenarios. They offer good resistance to corrosion prone environments and high-voltage and high-temperature environments. In Table 1, we compare the performance of optical spectroscopy with other sensing methods.

Table 1. Gas sensors comparison

Sensor Type	Merits	Demerits
Photo acoustic Spectroscopy	▪ High sensitivity ▪ Response time is fast ▪ Measurement is free from background noise ▪ Requires no reference as a result of noise(51)	▪ Selectivity is poor for photo acoustic system that utilises infrared light sources (52)
Photo reductive gas sensor	▪ Good sensitivity ▪ Short response time ▪ Inexpensive	▪ Temperature requirement is high ▪ Energy dissipation is high (53, 54)
Electro-chemical Sensors	▪ They are portable ▪ Exhibits high sensitivity. ▪ They are inexpensive (55)	▪ There is the depletion of electrolyte when used for sensing high ozone concentrations. ▪ It requires frequent maintenance (56, 57)
Metal oxide ozone sensors	▪ Broad range of application (58)	▪ High temperature requirements of detectors which translate into: ▪ High energy consumption. ▪ High cost ▪ Fabrication and size limitations ▪ (55, 56, 59)
Solid State	▪ Consumes less energy ▪ Good sensitivity ▪ Fast response time ▪ Inexpensive ▪ Light weight	▪ Characteristic activity is high ▪ Film sensor thickness requirement is large when applied for ozone sensing (56, 60, 61)
Chemilumines-cence.	▪ Fast response time (43)	▪ Requires to be calibrated within every one hour (every 1 to 60 minutes) (43). It is not absolute.
Optical spectroscopy	• It is a rapid and direct means of sensing gases with good cross sensitivity (57) • Require no consumables either for calibration or operation • Anti-electric magnetic interference, • Excellent electrical insulativity, and • Suitability of long-distance online measurement	▪ Gas sample must be able in a distinct manner to either absorb, emit, or scatter transmitted light rays at specific region of the light spectrum (7, 57, 62); ▪ Expensive ▪ Large in size (6)

4. Sensor Classification

Fibre optic sensors can be classified based on method of fibre application in sensor system and modulation mechanism [8]. The classification is illustrated in Figure 2.

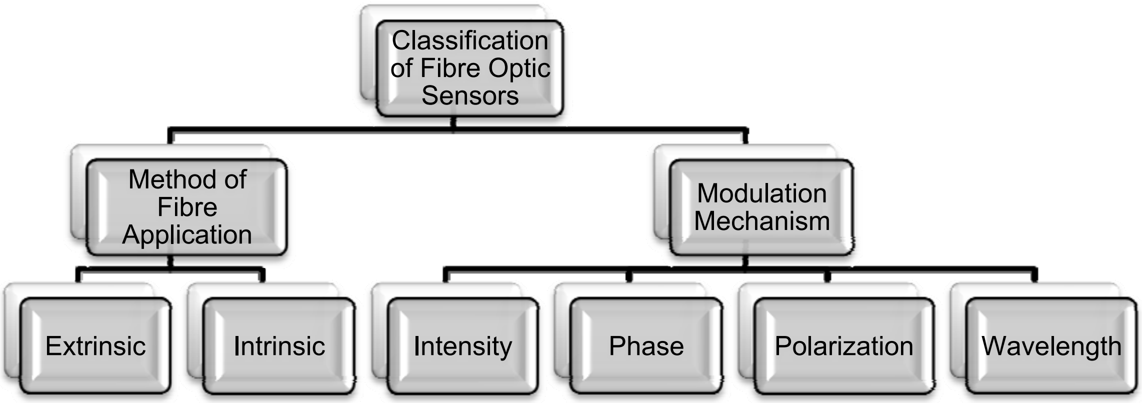

Figure 2. Optical sensor classification

4.1. Classification Based on Fibre Application

Fibre optic sensors are categorised into intrinsic and extrinsic types.

4.1.1. Intrinsic Optical Sensors

In an intrinsic fibre optic sensor, light is restricted within the optical fibre and modulation of the light signal is within the fibre [8, 63]; it is illustrated in Figure 3.

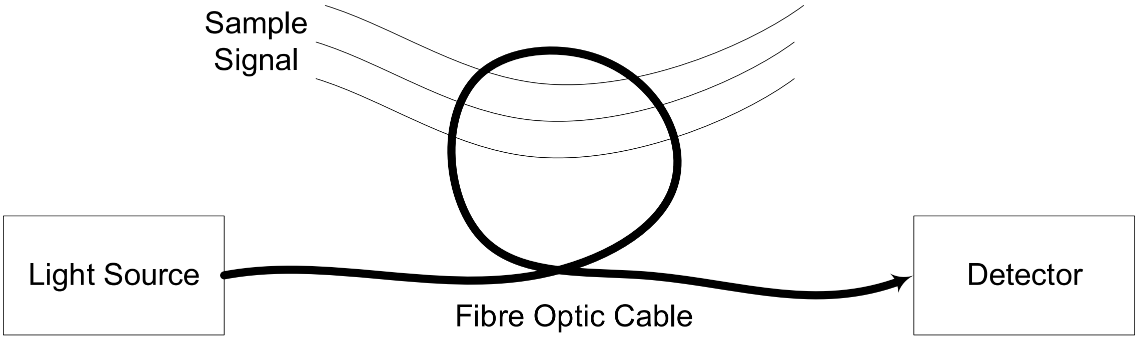

Figure 3. An Intrinsic Fibre Optic Sensor

4.1.2. Extrinsic Optical Sensors

In an extrinsic sensor, interaction between light signal (i.e. light signal modulation) and the sample to be measured takes place outside the optical fibre cable in a gas cell generally referred to a cuvette [8, 64]. It is illustrated in Figure 4.

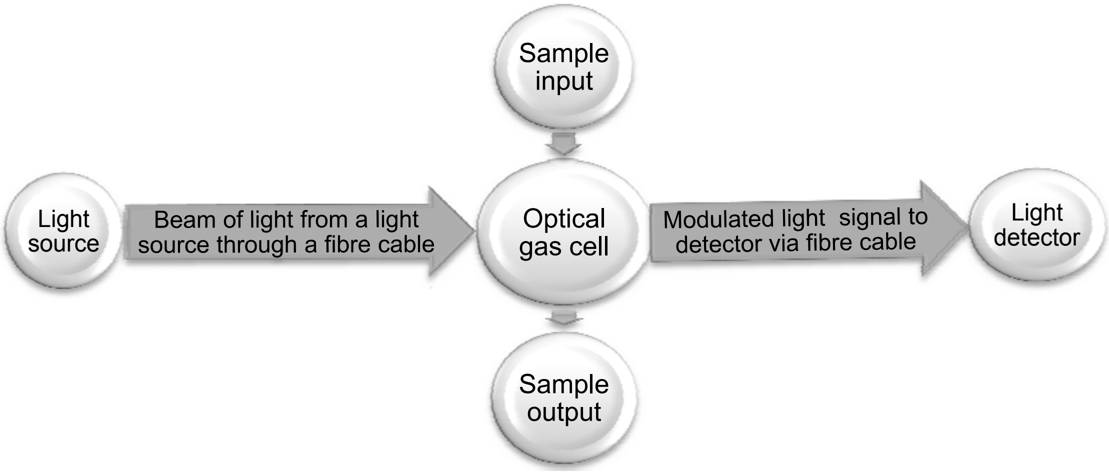

Figure 4. An Intrinsic Fibre Optic Sensor

4.2. Classification Based on Modulation Mechanism

In the application of light for sensing in fibre optic sensors, different characteristics of light are modulated to achieve sensing. These characteristics include: intensity, phase, polarization and wavelength [8]. Ozone gas measurement with optical absorption spectroscopy is detected by light intensity modulation.

5. Basic Experimental Setup for Ozone Detection via Optical Absorption Spectroscopy

A typical absorption spectroscopic experimental setup is made up of the following components: source of light radiation, a monochromator (except when light source is a laser). Light sources can either be broadband or chromatic [65]. Light emanating from a broadband light source must be propagated through a collimating lens to eliminate scattering effects. Light coupler, waveguide (fibre, fibre bundle, planar wave guide), variable attenuator, lenses (optical), cuvette (absorption cell or gas cell), light detection unit (spectrometer, photo detector), amplifier, secondary filter, transducer, data acquisition unit, data processing unit, and display unit [1, 66]. Figure 5 is a typical experimental setup for ozone measurements using optical absorption spectroscopy. It is the typical extrinsic setup.

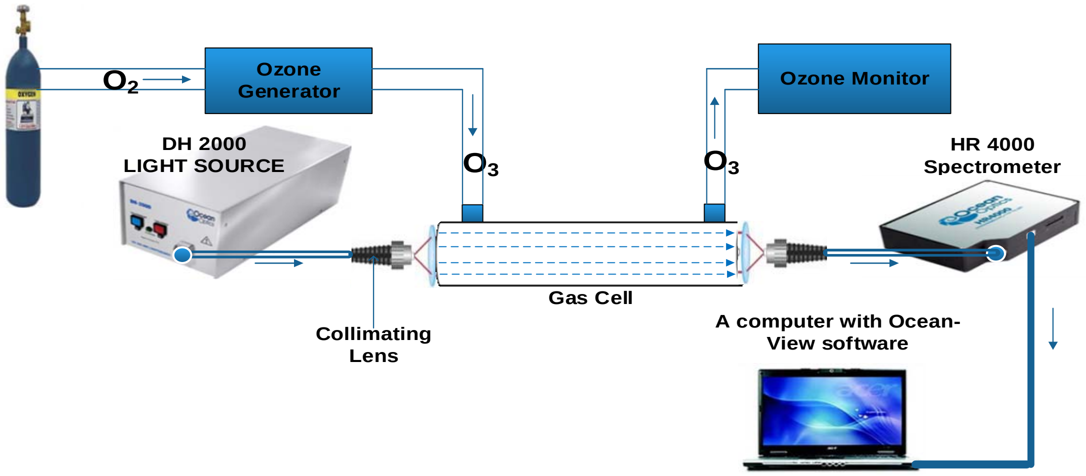

Figure 5. A basic layout of an optical absorption spectroscopy for ozone measurements

5.2. Classification of Gas Cells

The design of an optical gas cell in absorption spectroscopy is a major factor that affects the overall system performance in the form of sensitivity and speed of response. Authors of reference [9] have classified gas cells based on the principles of light transmission. The classification includes transmission type, reflective type, slow light and refractive index periodic change. More information on this can be obtained from reference [9].

6. Beer-Lambert Law

Absorption spectroscopy is the quantification of the energy that molecules absorb and and is translated to the bending and stretching of the bonds between the atoms in the molecules [67]. The working principle of gas cells in optical gas sensor is based on the Beer- Lambert law. According to Beer and Lambert, the concentration of a sample can be determined by detecting the intensity of the output light. Beer-Lambert law describes the relation of the input light and the output light that are affected by the measuring gas.

Beer's law: it states that the fraction of the incident light absorbed is proportional to the number of the absorbing molecules in the light-path and will increase with increasing concentration or sample thickness [10].

Lambert's law: it states that the fraction of monochromatic light absorbed by a homogeneous medium (sample) is independent of the intensity of the incident light and each successive unit layer absorbs an equal fraction of the light incident on it [10].

The combination of the two laws together yields the Beer-Lambert law. If radiation of intensity I_0 (zero sample concentration) is directed at a sample in a path length L, radiation of intensity I_t leaves the sample [68]. Beer-Lambert law shows the mathematical expression of the relation between the absorbing samples concentration (c) and absorbance (A). It written as follow:

$$A = \varepsilon \times c \times L \tag{1}$$

Where:
ε = molar absorption coefficient (m^2 mol^{-1})
c = sample concentration (mol m^{-3}) and
L = optical path length in (m)

In an experimental scenario, measurements are obtained in the form of transmittance *T* defined as:

$$T = e^{-\varepsilon cl} = \frac{I_t}{I_0} \tag{2}$$

The ratio $\frac{I_t}{I_0}$ is defined as the transmittance *T*:
From equation 2, absorbance *A* can also be defined as:

$$\mathrm{A} = \ln\frac{I_0}{I_t} = \varepsilon cL = \text{Optical density } (D)\text{, optical depth (69) or optical thickness (70)} \tag{3}$$

7. Absorption of Light by Ozone

Ozone gas detection via optical absorption spectroscopy is generally accepted [71]. This method has an inherent advantage to measure ozone absolutely without the requirement for consumables to operate or calibrate [7]. Whereas, ozone measurement with the method of chemiluminescence is not absolute, it has to be frequently calibrated. Chemiluminescence technique requires to be calibrated every 1 to 60 minutes [43]. Ozone absorbs light in the Hartley band (200–310 nm) [72], the Huggins band (310–375 nm), the Chappius band (375–603 nm), and the Wulf band (beyond 700 nm). It has peak absorption at 253.65nm ($\sigma_{253.65} = 1.147 \times 10^{-17} cm^2/\text{molecule}$) (73) and 603nm ($\sigma_{603} = 5.18 \times 10^{-21} cm^2/\text{molecule}$) (64).

7.1. The Absorption Cross Section of Ozone

Error free measurement of ozone gas is dependent upon ozone gas absorption cross section [74]. Hence, lots of research efforts are devoted to investigate the accurate value of ozone absorption cross section [64, 75-78]. Spectralcalc.com simulator has been used in this review to show the effect of temperature on absorption cross section in the Hartley band. Figure 6 shows absorption cross section of ozone gas obtained by simulation with spectralcalc.com at temperatures of 200 K and 300 K respectively. Ozone gas absorption cross section at 253.65 (actual spectral line is 253.6526 nm) is $1.16 \times 10^{-17} cm^2/\text{molecule}$ and $1.14 \times 10^{-17} cm^2/\text{molecule}$ at temperatures of 200 K and 300 K respectively. Absorption cross section decreases with increase in temperature from 200 K to 300 K. The percentage decrease is 0.95 % at a measurement wavelength of 253.6 nm. Malicet *et al* reported a decrease of 1 % in absorption cross section for a temperature rise from 218 K to 295 K [79]. Similarly, Serdyuchenko *et al* reported a slight decrease in absorption cross section with temperature increase in the Hartley band [80]. The result thus obtained is in good agreement with previous works.

8. Materials Compatibility with Ozone

Not all materials are compatible with ozone gas. Ozone gas compatibility with common materials used for ozone sensing in literature is compared in Table 2.

The rating in the table depicts chemical effect of ozone on the listed materials. A material rated "A" (excellent) implies ozone has no effect; "B" (good) ozone has minor effect. Other categories not included in the table are "C" (fair), which implies ozone effect is moderate and "D" means ozone has a severe effect on the material. The rating as defined by Ozone solutions is for ozone gas concentrations greater than 1000 ppm [81].

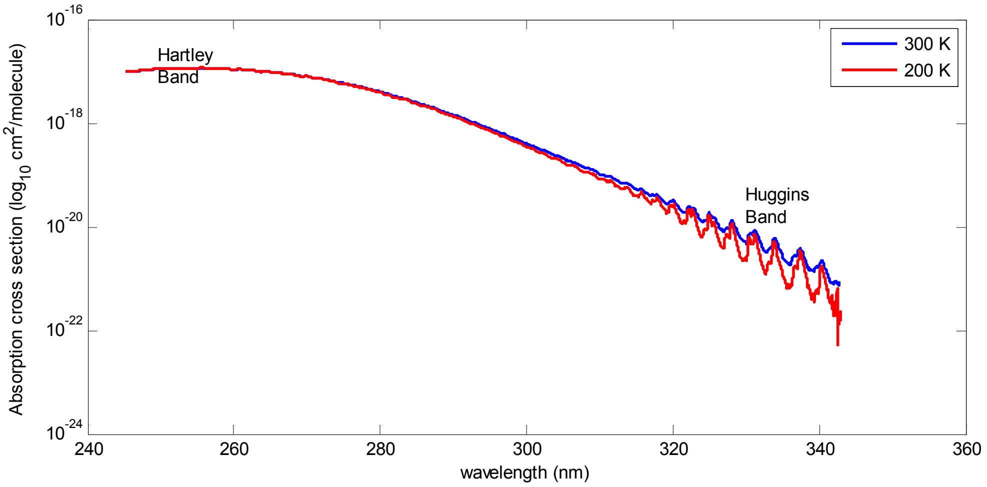

Figure 6. Spectralcalc.com simulation of ozone absorption cross section at 200K and 300K

Table 2. Materials compatibility with ozone (81)

Material	Rating	Example of applications
Aluminium	B - Good	(64)
Brass	B - Good	(82)
Glass	A - Excellent	(53, 83)
PTFE (Teflon®)	A - Excellent	(30)
Silicone	A - Excellent	(84, 85)
Stainless steel - 304	B - Good/Excellent	(86)
Stainless steel - 316	A - Excellent	(86)
Viton®	A - Excellent	(87)

9. Research Challenges

Recent research activities on ozone gas sensing with optical absorption spectroscopy include sensitivity enhancement through optical path length and ozone gas absorption cross section optimization [88] and effect of noise reduction on absorption cross section of ozone gas in the visible spectrum [89]. Redefinition of the value of ozone gas absorption cross section in the UV for accurate measurements of ozone gas [90] and preservation of linearity of Bear-Lamberts law by measuring ozone gas concentration at an alternate sampling wavelength of 279.95 nm [85, 91]. Ozone gas measurement in the visible spectrum using LED as a light source at 605 nm [92] and sensitivity enhancement through light propagation at incident angle [93]. Temperature and pressure dependence of ozone gas absorption cross section in the UV and visible spectrums [80, 94]. Performance indicators/metrics of ozone sensors and sensors in general include selectivity, sensitivity, accuracy, resolution, response time, fabrication cost, dynamic range, precision and linearity [58, 95-99]. Sensor requirements either in performance, physical, or cost, are application dependent [100]. Research activities on sensors in general and ozone sensors in particular, are aimed towards meeting recent sensing requirements, strengthening and upgrading some or all of the aforementioned parameters [11, 12, 49].

10. Conclusions

The review paper summarises necessary information. It is a ready reference material for new researchers in the field of absorption spectroscopy for ozone sensor application. Issues discussed include basic operating principles of optical sensors and its mechanism. Optical sensors as well as optical gas cells were classified. Specific properties of ozone gas were also highlighted. Recent research activities have been enumerated. Spetralcalc.com simulation software was used to demonstrate possibility of obtaining preliminary results before experiments are conducted.

Acknowledgements

The authors would like to thank Universiti Teknologi Malaysia (UTM) for sponsoring this publication under Research University Grant (RUG) Scheme, grant no: 05J60 and 04H35. The Ministry of Higher Education (MOE) Malaysia is acknowledged for provision of Fundamental Research Grant Scheme (FRGS) grant no: 4F317 and 4F565. The Nigerian Education Trust Fund (ETF) is also acknowledged for the financial support giving inform of Tertiary Education Trust Fund (TET-Fund).

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