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ORIGINAL PAPER

Spark spectrometry of toxic smokes: towards a portable, inexpensive, and high-resolution environment monitoring instrument

Soumendra Singh · Nabarun Polley · Amitabha Mitra · Samir Kumar Pal

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Abstract Inductively coupled plasma mass spectrometry and laser-induced breakdown spectroscopy are the most popular techniques for monitoring toxic gases in the environments. Apart from sensitivity and resolution of the techniques, they suffer from several issues including portability and high cost. For design and realization of a low cost, spark spectrometry based portable instrument for monitoring toxic gases in our environments is the main motive of the present work. We have introduced several toxic smokes into a gas chamber containing our developed instrument. We have also investigated the capability of the instrument for online analysis of suspended particulate matter as well as various gaseous elements in the smokes. We have also developed software for the practical interface. The apparatus has been successfully tested to monitor several toxic fumes including cigarette smoke and NO_x. It has also been demonstrated that the instrument is equally efficient to monitor air quality in the open environment, for example, presence of nitrogen, oxygen, and water vapor in the ambient condition. In the present work, we have demonstrated some important spectroscopic studies including role of water vapor (solvation) in the ionization of potassium, which is an active ingredient of toxic smokes, in the ionization which leading to the generation of atomic emission under the spark spectrometer can also be achieved with our instrument. The sensitivity of the instrument is found to be sub ppm (0.27 ± 0.13 ppm) in the case of cigarette smoke in ambient condition. The simplicity and extremely cost-effective design can provide an alternative method of detection of fumes in air and can serve as a cheap alternative for costly/bulky bench-top instruments.

Keywords High voltage spark · Spark emission spectroscopy · Low-cost design · Environmental pollution detection · RGB analysis · Optical filters · Data acquisition

Introduction

Multiple health conditions including respiratory tract infections, heart disease, and lung cancer have been attributed to breathing polluted air (Fielding 1985). Apart from "smoking", the sources of air pollution causing deadly influx of CO, SO_2, NO_2, and Pb do not need much introduction, and a mammoth of information is already present in the contemporary literature. World wide, cigarette smoking is the leading cause of preventable death and a major public health concern (Boyle 1997). Tobacco use leads most commonly to diseases affecting the heart and lungs. Smoking is a major risk factor for heart attacks, strokes, chronic obstructive pulmonary disease (COPD), emphysema, and cancer (namely lung cancer, cancers of the larynx and mouth, and pancreatic cancer). Some nontrivial sources of air pollution, however, very much intense in some of the countries are also becoming potential threat to the environment (Cao et al. 2011). Particularly, in the developing and under developed countries, there

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S. Singh · N. Polley · S. K. Pal (✉)
Department of Chemical, Biological and Macromolecular Sciences, S. N. Bose National Centre for Basic Sciences, Block JD, Sector III, Salt Lake City, Kolkata 700 098, India
e-mail: skpal@bose.res.in

S. Singh · A. Mitra
Centre for Astroparticle Physics and Space Science, Bose Institute, Salt Lake Campus, Block - EN, Sector – V, Salt Lake City, Kolkata 700091, India

are some nontrivial sources of air pollution including traditional biomass burning (Andreae and Merlet 2001; Galanter et al. 2000). Most dangerous practice of cooking among lower income group people by burning "garbage" containing various kinds of plastics is considered to be sources of lots of poisons in the air (Estrellan and Iino 2010). The smoke out of the garbage-burning commonly contains vapors and solid compounds suspended in the air called particulate matter. The toxic chemicals released during burning include nitrogen oxides, sulfur dioxide, volatile organic chemicals (VOCs), and polycyclic organic matter (POMs). Burning plastic and treated wood also release heavy metals and toxic chemicals such as dioxin (Menad et al. 1998). Smoke from wood and trash contains very small particles that can be breathed deep into the lungs. Once trapped in the lungs, these particles can cause cell damage (Regalado et al. 2006). Associations have been found between day-to-day particulate air pollution and increased risk of various adverse health outcomes, including cardiopulmonary mortality (Pope III et al. 2002; Pope et al. 2004). These alarming figures clearly indicate the need of identification and constant monitoring of suspended particulates with special emphasis on toxic smoke present in ambient air (Duan et al. 2004). There is some evidence that long-term exposure to NO_2 at concentrations above 40–100 $\mu g/m^3$ may decrease lung function and increase the risk of respiratory symptoms (Bonn 2003; Kampa and Castanas 2008). NO_x reacts with ammonia, moisture, and other compounds to form nitric acid vapor and related particles. Small particles can penetrate deeply into sensitive lung tissue and damage it, causing premature death in extreme cases. Inhalation of such particles may cause or worsen respiratory diseases, such as emphysema or bronchitis or may also aggravate existing heart disease (Menzel 1976).

ICP-MS has by far been proven as the best technique for evaluation of constituents in a mixture (Chang et al. 2003; Jenner et al. 1990; Ionov et al. 1992). In conventional elemental analysis, samples of various types are usually prepared or digested and then introduced as solutions to the plasma by a nebulizer. However, sample digestion is a tedious, labor-intensive, and time-consuming process, and there is also a risk of analyte loss and contamination that may occur during sample preparation (Álvarez-Llamas et al. 2005). Moreover, sample preparation involves risk of human errors, and thus reliability of results with respect to that in the ambient condition is in question. For an ideal case, a sample should be analyzed in its natural form to overcome these drawbacks (Jenner et al. 1990). Recently, injection and testing of direct analyte particularly in gaseous form have been reported (Chang et al. 2003). Other disadvantages of ICP-MS include extremely high cost and non-portability of the instrument to in situ measurement site. The instrument is a bench-top model and requires a relatively clean environment to operate and maintain.

Laser-induced breakdown spectroscopy (LIBS) can be an alternative to ICP-MS as the former technique has distinct advantages over the latter. The principal advantages of LIBS over ICP-MS include the simplicity of the technique, sampling speed, and most importantly less sample preparation. In particular, sample size, analysis time, and less sample manipulation provide distinct advantage in this instrument. Other very important advantage is the suitability for remote and in situ measurements and the instrument's adaptability for field portability (LEE and Sneddon 2002; Song et al. 2002). LIBS like any other technique is not free from limitations. They include material specific laser sample interactions, difficulty in obtaining suitable standards, large interference effects, and possibility of ocular sample damage (Mohamed 2008). Once again, LIBS systems available commercially or indigenously developed in labs are remarkably costly making it non feasible for certain users.

In our present study, we have attempted in situ measurement of cigarette smoke and shown the feasibility of detection of other gasses using our designed set-up. A typical cigarette in burnt condition has lots of elements, but ~82 % (by weight) of the mainstream smoke originates from the gases of the ambient air such as N, O, CO and CO_2 (Dube and Green 1982). In addition to that almost 4,000 compounds have been identified in tobacco mainstream smoke (Löfroth 1989), the majority of which are trace elements. Trace metal analysis using AES has been done previously using costlier techniques like ICP, but tens or hundreds of cigarettes must be smoked on a smoking machine in order to collect sufficient amounts of sample for trace metals analysis, due to the low level found in cigarette smoke (Chang et al. 2006) using ICP. The concentrations of sodium (Na) and potassium (K) present in the mainstream smoke are in microgram per cigarette, which are approximately three orders of magnitude higher than other common elements including cadmium and lead (Chang et al. 2003).

In this work, we attempt to design and construct a low cost and hand held instrument for environmental pollution monitoring. The design utilizes the principles of spark optical emission spectroscopy. The light emitted by high voltage spark through the test sample is collected by collimating optics and is focused at the CMOS detector. The widely available extremely low-cost CMOS detector has been utilized to acquire data for online monitoring of the instrument. The presence of oxygen and nitrogen in the ambient environment has been detected by the instrument as bench mark. We have detected presence of potassium (K) in the toxic fumes including cigarette smoke. Possibilities of efficient detection of other toxic gases including NO_x have also been attempted. The effect of water vapor in the environment on the detection of toxic fumes has been

explored in our studies. By monitoring emission line at 777 nm, very reactive oxygen radical resulting due to the high voltage discharge has been detected. We have also developed software in the MATLAB platform for the online analysis and trigger alarm for the toxic gases in various environments.

Materials and methods

Materials

All the materials used in the prototype design have been purchased from the local market. To the extent of our knowledge, the capacity of the materials used was of highest purity but also keeping in mind the cost feasibility of the crude materials. A vacuum desiccator (8 L) from Tarson was used as gas chamber for creating desired pressure levels of various analytes. A vacuum pump and pressure gauge was also used to suck air out of the mentioned gas chamber and hence create partial vacuum for pressure dependent studies. Copper sparking electrode of highest purity, plano-convex lenses, optical mounts, High Voltage (HV) source, and cheap CMOS chip is from Rohini Astrophysics, India. To verify the optical density (OD) of the gas chamber, highly coherent laser (He–Ne 632.8 nm) and detector from *Melles Griot* were used to get an estimation of the OD of the smoke in gas chamber. Cigarettes from various brands were used to verify the results hence could be treated as a confirmation of the constituents of various cigarette smokes. Optical filter (*AVJ Optics, India*) of 10 nm FWHM (centered at 768.5 nm) was used in front of the CMOS chip for the specific detection of K^+ line. All chemicals used in this procedure are analytical grade with highest purity, and were purchased from reputed brands. We have calculated the extinction coefficient of the cigarette smoke from various brands in the following way. The cigarette smoke was collected in a transparent bag of volume 28.5 cc, and OD was measured followed by immediate measurement of its weight. The experiment was repeated 5 times, and the extinction coefficient was found to be 11.4 ($\pm$ 1.1) $\times$ 10^{-3}/ppm/cm. In order to estimate the minimum detection limit of our designed instrument, we have measured the intensity of K^+ lines in the gas chamber with different concentrations of cigarette smoke. Simultaneous measurement of OD and minimum detectable intensity due to K^+ lines at 767.67 and 769.90 nm through our CCD device reveals the detection limit to be sub-ppm level (0.27 $\pm$ 0.13 ppm). The measurement strategy for simultaneous detection of OD and K^+ line intensity has been shown in a video in the supporting information. A spectrometer (HR 4000) from Ocean Optics, USA, was used for

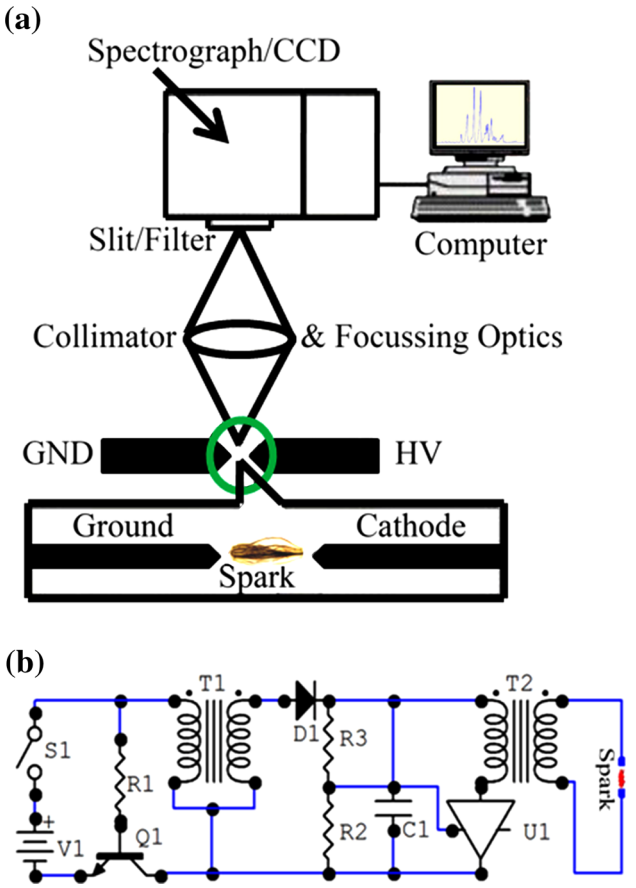

Fig. 1 **a** Schematic diagram of the designed setup. **b** Circuit diagram of the battery operated high voltage power supply

the spectral analysis of the spark emission. Efforts to identify NO_2 present in atmosphere have been tried and successfully achieved. NO_2 was prepared using copper strips and allowing them to react with concentrated HNO_3. The chemical reaction is shown below.

$$4HNO_3 + Cu \rightarrow Cu(NO_3)_2 + 2NO_2 + 2H_2O$$

Design

The design of the opto-mechanical component setup including ray diagram has been explained in Fig. 1a. The setup essentially consists of two copper electrodes connected to a high voltage supply typically at 15 kV. We have used a high voltage current limiting transformer (typically used in powering neon sign) for the spark voltage supply. The electrodes when kept at a distance equals to the spark gap produce an electrical discharge in open air. The electrical discharges across the electrode knock off electrons of the residual gas to higher energy orbital, and when these electrons return to their ground or stable orbital they emit photon which is normally captured by a photosensitive device to provide spectral information. The electrodes

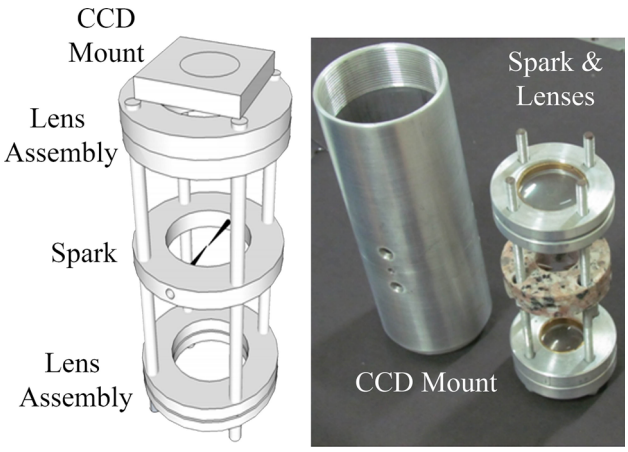

Fig. 2 Mechanical design of the setup is shown in the *left panel*. *Right panel* shows photograph of the setup. Simplicity and portability can easily be evident from the picture

are attached with a granite housing, which is mounted over four long screws. The light emitted by the spark setup is collected by a pair of plano-convex lens on each side of the electrodes. The electrodes and the optical detector are kept at the respective focus of the lenses. In another low-cost design, the high voltage current limiting transformer has been replaced by 1.5 V DC (pencil battery) operated sparker. The modification leads the setup to be ultra portable (hand held). The detailed circuit diagram of the sparker circuit is shown in Fig. 1b. The bench mark spectra obtained from both the setup(s) show difference in the emission lines (in the UV–Vis region) of nitrogen and oxygen due to difference in the spark voltage and gap, however, consistent with the reported values of the emission lines(Kuffel et al. 2000; Kasuya et al. 1999). We have also observed that emission lines for water vapor (Hα) and K^+ are independent of the setup (Vodacek et al. 2002). The entire setup except the computer can be placed inside gas chamber for performing controlled pressure dependent experiments. The mechanical design and the real photograph of the setup is shown in Fig. 2.

Charge coupled device (CCD) detector

The setup is connected to a computer via a USB wire for real time data monitoring and analysis. Commercially available CMOS array offers reasonably good signal to noise ratio (SNR), reduce the cost as well as radio frequency interference (RFI) free output signal. A computer program has been developed for online monitoring of the signal from the detector. Alarm sounding system has been designed to set off as soon as the signal value crosses a certain threshold value. The data are also stored in a data file on the computer hard drive for future offline analysis.

Software

The programming code has been developed on the MATLAB platform. As shown in Fig. 3, the flow chart clearly shows that the input signal is disintegrated into RGB component values. Each matrix is separately plotted, and the red components (characteristic of K^+ ion) are connected to a virtual scope for display of current values. The matrix of values was converted to unique numbers considering the weight of each bit of the matrix. A threshold value has been provided in the software for alarm considering the toxicity of the gas and the sensitivity of the site under monitoring. For example, hospitals and schools are more likely to have very low threshold levels, whereas other public places may have higher tolerance.

Results and discussion

Test of the instrument with standard samples for bench marking

Figure 4 shows typical emission spectra of the spark in our instrument with various discharge voltages. As shown in Fig. 4a, the emission spectrum of air contains several characteristic emission lines of nitrogen (500.23, 516.95, 567.21, 592.98 nm) and oxygen (462.69). A relative faint characteristic of Hα line (656.56 nm) (Leahy-Hoppa et al. 2010) indicating that the presence of water vapor in the ambient atmosphere is distinct from the spectrum. Upon insertion of water vapor to the experimental gas chamber (desiccator), enhancement of the Hα line is clearly evident from Fig. 4b. It has to be noted that the emission spectrum of the spark discharge in air very much depends on the DC voltage (–HV, Fig. 1), the spark gap, and the material used in the electrode (Frey et al. 2009). The spectrum of the spark discharge in air at DC voltage of 15 kV shows emission in the UV region. The characteristic emission lines of air are indicated in the spectrum as shown in Fig. 4c. The spectra of air as indicated in Fig. 4a, c are consistent with that of the reported literature in the similar experimental condition; thus, it can be considered as benchmark of our experimental setup (Ralchenko 2005).

The setup is shown to identify the presence of toxic gasses and aerosols in the high voltage spark. The experiment has been repeated with introduction of cigarette smoke from various brands in the gas chamber, and the results are found to be absolutely repetitive. We have found that an emission doublet (767.67 and 769.90 nm) is unanimously present in all the man made toxic gasses including fumes from firecrackers, mosquito repellant, and cigarette. It is well known that potassium Nitrate is one of

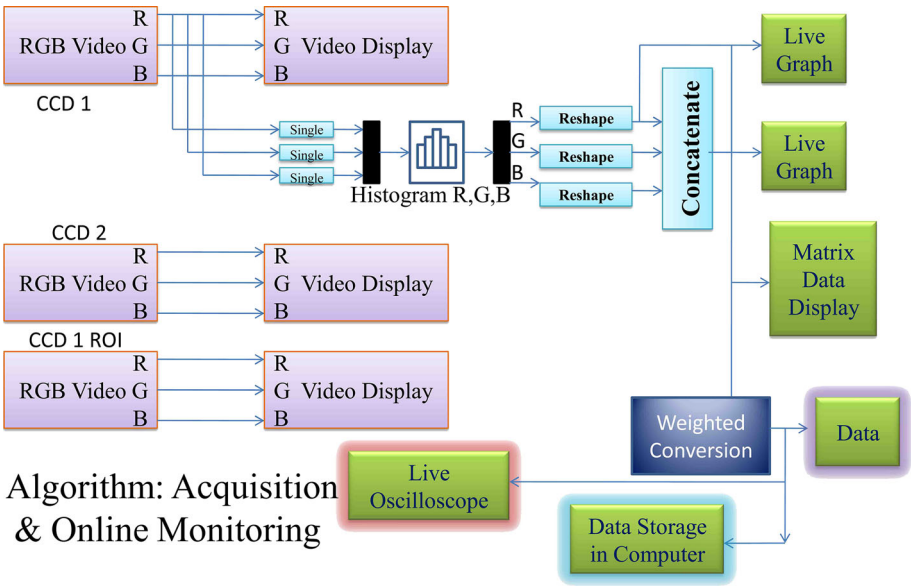

Fig. 3 Flow chart of the developed software in the MATLAB platform for the data acquisition and online monitoring/alarm sounding

the active ingredients of those products for their continuous burning (Wannamethee et al. 1997). Thus, our finding of K^+ emission is consistent with the fact and could be used as marker of the toxic fumes. Figure 5 shows scanning and zoomed spectra in presence and absence of cigarette smoke. The experiments were repeated with smoke from other non polluting sources like burning paper, cow manure, etc., and the results were compared with emission lines of indoor air. Cow manure is commonly used as burning material for cooking and other purposes particularly in developing countries. It was evident from our experiment that K^+ line is the characteristic of the cigarette smoke compared to others.

In order to make the setup to be cost effective, we have replaced the costly spectrometer with a combination of the interference filter and CCD detector specifically sensitive at K^+ doublet line. The center wavelength of the filter is chosen to be around 769 nm to accommodate the potassium doublet and reject all other light signals which may be the signature of different other constituents of the atmosphere. Figure 6a shows the pass band of the filter along with the transmission efficiency in percentage. It is quite evident that filter has transmission efficiency of around 60 % at peak wavelength of 769 nm. The signal characteristics in presence of the filter are shown in Fig. 6b. Optical signal received from the setup clearly suppresses all other wavelength and allows signal of K^+ ions' emission spectra to the spectrometer. The spectrometer was then removed, and CMOS chip was introduced to capture live signal (red) from the spark in presence of cigarette smoke. As expected, the CMOS array recorded no signal for spark under ambient conditions in the absence of toxic smoke. However, with the introduction of cigarette smoke and signal was recorded on the computer screen. Figure 6c shows a screen shot of the live signals recorded during the acquisition. The region of interest has been marked with a circle. The developed software was programmed to set a threshold level as soon as the signal crosses a certain prefixed level (e.g. 2.0 here) as shown in Fig. 6d. The threshold level can be fixed according to the sensitivity of the place under test. Figure 7 shows a relationship of the OD of the cigarette smoke in our gas chamber with the intensity of K^+ line. In the experiment, we have measured the OD by using He–Ne laser as described earlier. We have observed that the dependence of normalized K^+ line intensity slightly deviates from the linearity in the experimental window of OD up to three. The sensitivity of the instrument for cigarette smoke was measured and found to be at sub-ppm level.

As shown in Fig. 8a, the designed setup is capable of monitoring NO_2 in the gas chamber. Emission peaks at the wavelength band from 400 to 600 nm reflecting that the unambiguous presence of NO_2 is evident from the spectrum. Inset of Fig. 8a shows the enhancement of N(II) line compared to that in ambient air. It has to be noted that the presence of NO when excited by the spark will result in emission lines of N(I), which are characterized mostly by peaks in the band of 700–900 nm. Thus, the recorded spectrum is evident to be due to the presence of NO_2 only (Ralchenko 2005). We have also tested our setup for the detection of extremely reactive atomic oxygen radical. In a recent study, using atomic spectroscopy in the visible region shows that emission at 777 nm is a clear indication of the presence of the very reactive oxygen radical (Krstulović et al. 2006). The origin of the emission line at 777 nm is found to correspond to the oxygen atom transition O ($3p^5p \rightarrow 3s^5s$) as shown in Fig. 8b. The details of the species and their interaction

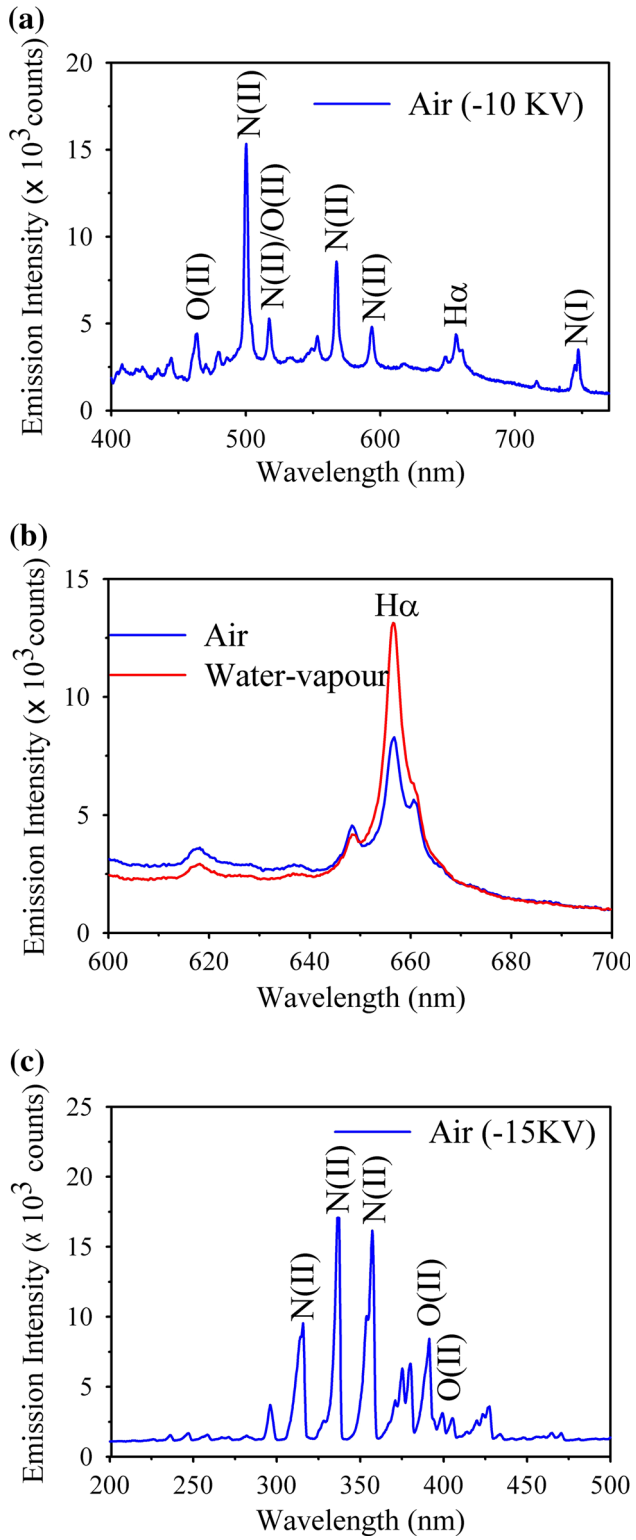

Fig. 4 Benchmark spectra of air and water vapor from the designed setup

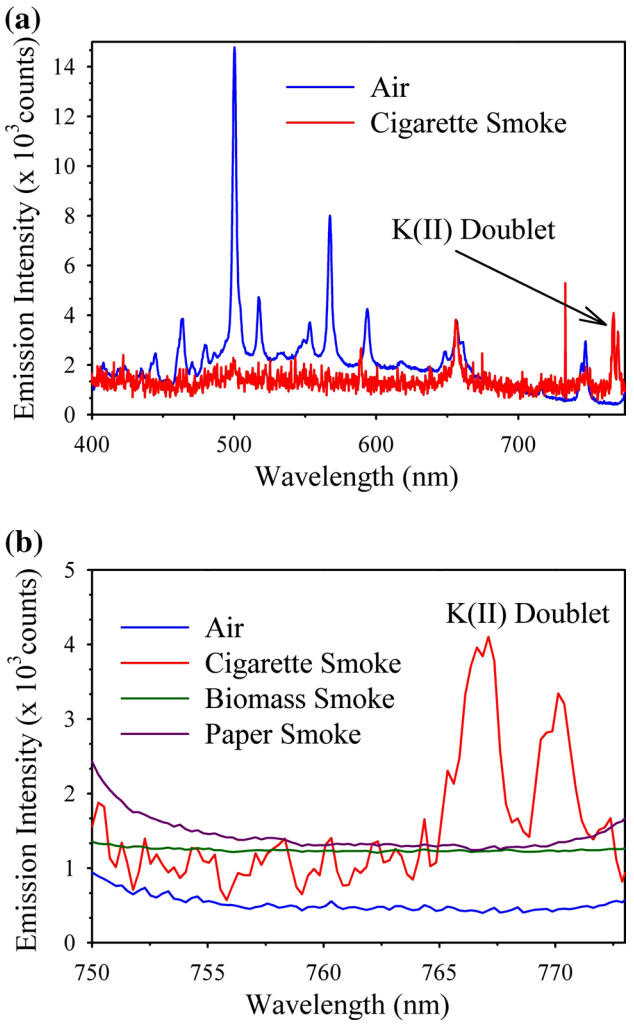

Fig. 5 Spectroscopic detection of K^+ ion emission as a distinct marker of cigarette smoke cow manure was used as a source of the smoke from biomass

with other organic molecule are described in the earlier literature. It has been shown that the excited energy state of O-777 triplet is close to the excited upper energy levels of Carbon responsible for emission peaks at 601 nm which can result in non-radiative energy transfer and successive generation of O-777 reactive element (Milosavljević et al. 2011). As described earlier for the low-cost detection of cigarette smoke, a simple modification would be able to replace the costly spectrometer by the filter-CCD (centered at 777 nm) for the reactive atomic oxygen detector.

In a practical application of the designed setup, we intend to use the system in open environment (not in gas chamber). However, the presence of water vapor (humidity) with various proportions in ambient open environment is unavoidable. Thus, in order to investigate the role of humidity in the detection sensitivity of the toxic fumes, we have allowed controlled water vapor in the gas chamber during the spectroscopic detection of various emission lines of the fumes. As shown in Fig. 9a, we have observed

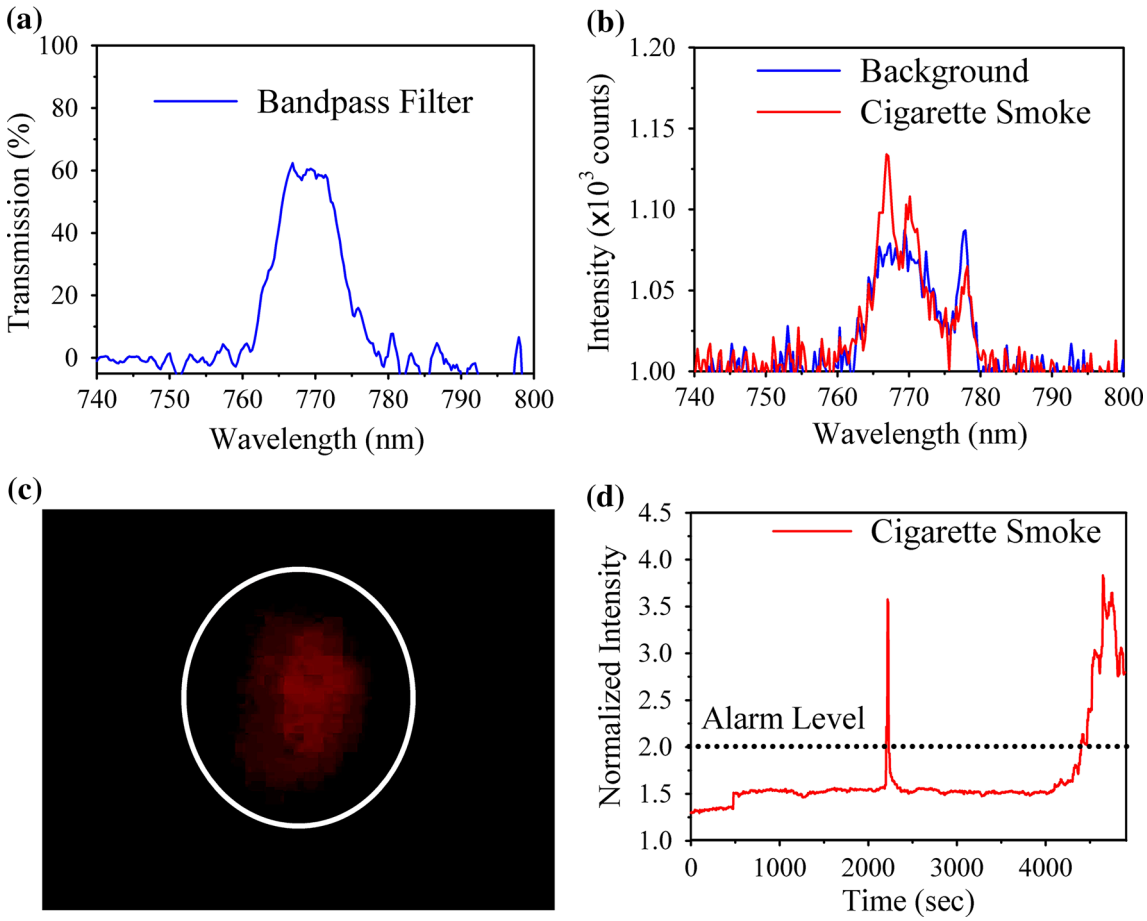

Fig. 6 **a** Transmission characteristics of the optical filter used as a replacement of the costly spectrograph. **b** Spectrum of the cigarette smoke through the filter. **c** Signal acquired by computer through the filter-CCD combination in the place of spectrograph. **d** Test results of the low-cost version of the setup. The data acquisition and monitoring/alarm sounding are done through our developed software

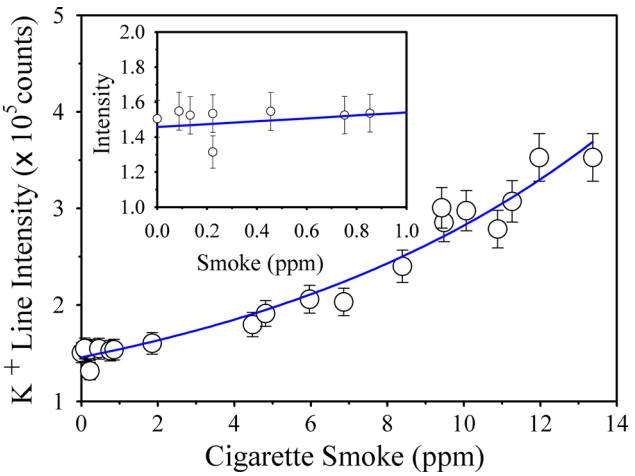

Fig. 7 Intensity of the K^+ emission line in the cigarette smoke as a function of the concentration of the smoke in the gas chamber (*open circles*). The *solid line* is the guide to the eyes. *Inset* highlights low smoke concentration region, and the corresponding K^+ emission intensity revealing the sensitivity of our designed instrument

significant enhancement of K^+ emission line, reflecting higher smoke detection sensitivity in presence of water vapor. Thus, in order to calibrate the setup, one has to take care of the humidity in the environment under investigation. We have also observed the insignificant effect of the NO_x detection sensitivity in presence of water vapor as shown in Fig. 9b. Ingestion of both the gases in water may play a role in the detection sensitivity. Although, detail conclusion on enhanced sensitivity of K^+ ion and NO_x lines invites further experimental and theoretical investigations. Some qualitative argument may be made for the explanation of the observation. From the spectra (Fig. 9b), the position of Hα line (due to water vapor) in comparison to the emission line under investigation is concluded to play a significant role. K^+ (at 767.67 nm) indicating that the cigarette smoke is lower in energy compared to that of the Hα line (656.56 nm). Thus, K^+ ion duly solubilized in the water cluster can be excited without much interference from the host matrix (water cluster). On the other hand, the characteristic peak of N(II) (500.32 nm) is higher in energy

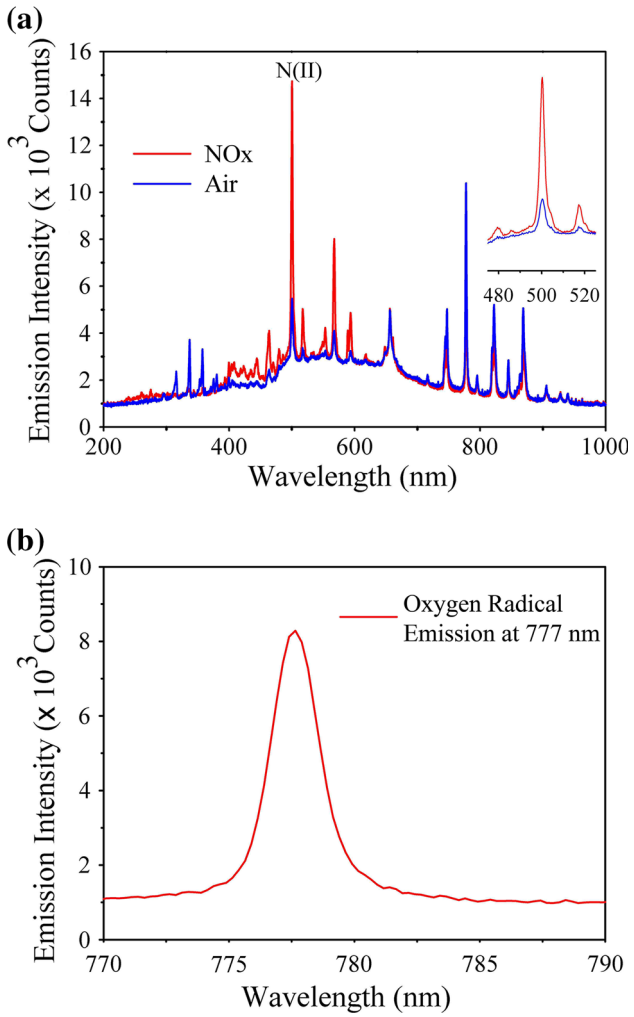

Fig. 8 Detection of toxic NO_x (**a**) and highly reactive oxygen radical (**b**), using our setup

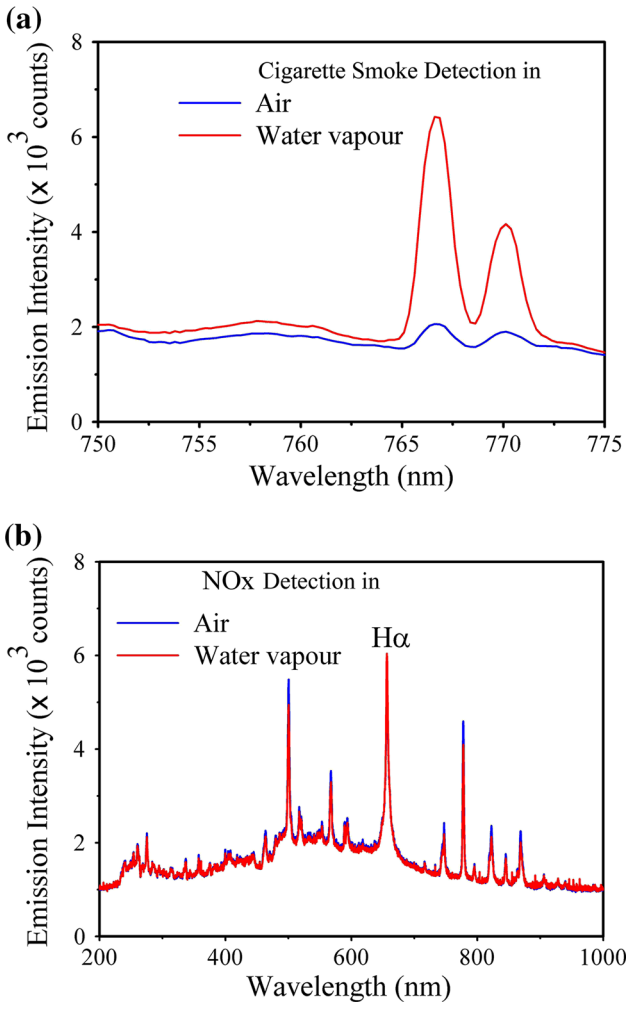

Fig. 9 Detection of cigarette smoke (**a**) and NO_2 in presence of water vapor (**b**). Enhancement of detection sensitivity in the case of cigarette smoke is clearly evident from panel

as compared to that of Hα. Thus, the host water cluster is expected to absorb energy from the excitation source and hinder the excitation of the digested NO_2 in the water cluster. Ease of excitation of metal ions due to solvation of water cluster is reported in the literature (Partanen et al. 2013). Our Observation of enhanced detection of K^+ line in the presence of water vapor is consistent with the theoretical prediction (Zhang et al. 2011). Recently, the solvation of potassium salts in water clusters at nanoscale is studied by photoelectron spectroscopy using synchrotron radiation, and the results are compared with the salts in dry condition (Partanen et al. 2013). The study clearly showed that the potassium salts are completely dissolved in water clusters as ions. The study found that K 3p binding energies of the dissolved potassium ions are at least 0.8 eV lower than that in the dry cluster of the salts. Relatively lower ionization energy of potassium in water cluster as observed in our experiment is also consistent with other earlier experimental studies (Winter et al. 2005; Zhang et al. 2012).

Conclusion

We present design and construction of a low-cost portable spectroscopic instrument for monitoring air quality in various environmental conditions. Apart from the detection of oxygen, nitrogen, and water vapor, the instrument is shown to monitor toxic fumes including cigarette smoke, NO_x, and the reactive oxygen radical. We have also developed a software for the online monitoring of the toxic fumes in the environments. The role of the presence of water vapor in the sensitivity of detection has also been explored. General use of the instrument for any gas detection in the environment has also been discussed. In the present study, we have made a prototype for the low-

cost automated detection of toxic fumes (from the cigarette smoke) using the K^+ emission lines at 767.67 nm. We have also calibrated the prototype with respect to optical densities at 632.8 nm (He–Ne laser), of the cigarette smoke in a designed gas chamber. In principle, our methods can be modified to design low-cost automated gas detection systems for any toxic fumes including NO_x, SO_2, and O_3 by calibrating the system at 400–600 nm, 7.28 μm and 7.35 μm, and nearly 350 nm, respectively (Francis et al. 1995; Marcq et al. 2008; Puckrin et al. 1996; Mérienne et al. 1995; Coquart et al. 1995; Fukuchi 2009; Martin 2008; Bachmann et al. 1993). The instrument can be a cheap alternative for costly methods of detection of indoor air pollution (e.g., cigarette smoking) as well as for outdoor air quality management studies. The portability of the unit facilitates its placement at potentially dangerous places that are unfit for human presence like volcanic eruption sites and jungle fire. The tentative cost of the prototype for detection of cigarette is tentatively 100 USD including the filter. The long-term and short-term stability of the instrument are found to be reasonably good.

Supporting information

The measurement strategy for simultaneous detection of OD and K^+ line intensity has been shown in a video in the supporting information.

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