



Solid membrane electrode assembly for on board detection of peroxides based explosives



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ABSTRACT

The threat to human populace from the use of improvised explosive devices (IEDs) is increasing at an alarming rate. Many devices for IEDs detection lacks sensitivity, reliability, portability: a major issue to be addressed. In this work, we fabricated a disposable solid phase electrolyte/electrode (SPEE) interfaces for the detection of trace quantities of peroxide based explosives, namely, triacetone triperoxide (TATP) and hexamethylene triperoxide diamine (HMTD) which were synthesized without recrystallization, and their precursors: H₂O₂ and acetone via electrochemical technique. The SPEE device was able to detect 8.66 ± 1.048 ng of TATP at its vapor pressure of 4.80 Pa at 298 K. However, the device only gives good result during the first time usage. As suggested by the electrochemical cell impedance test, mass transfer limitations caused by the solution and charge transfer resistances at the electrode interface impedes reaction kinetics. Nevertheless, the ease of fabrication, response and speed make SPEE sensor an excellent choice and offer opportunity for the development of miniature sensors for screening of IEDs and its precursors at various screening sites.

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1. Introduction

Technologies for explosives detection are often required to have a number of attributes which include that they be low cost, portable and specific. At the most fundamental level sensors contain an immobilized active species which can selectively recognize a material of interest. A transduction step which can be optical, electrochemical or some other technique is then required to convert the recognition event into a measurable change, such as the production of electrons which can be measured at an electrode. For example, the principle of chemical sensors is to have a chemical which undergoes a selective chemical reaction with an explosive vapor, leading to an observable product such as a change in color or conductivity. However, sensitivity is often an issue, and many sensors cannot simply detect the low levels of material required. This is particularly true when attempting to detect low vapor pressure material; such as explosives. Work in the field of explosives sensor development needs to focus on improved sensitivity, reliability and reproducibility.

One sensor that should be considered for explosives vapor detection is an electrochemical sensor. Electrochemical sensors can be categorized into potentiometric (measurement of potential difference/voltage); amperometric (measurement of current) and conductometric (measurement of conductivity) based devices according to the transduction approach adopted [1]. These types of sensor are fast, inexpensive, have high sensitivity, and are a viable option for miniaturization [2]. Electrochemical detection techniques can respond to redox substances; the electrical output results from a flow in electrons or ions caused by the chemical reaction that takes place at the surface of the electrode. Because of this, the redox properties of peroxide explosive substances ideally lend themselves to electrochemical detection. The detection of peroxide based explosives is often difficult because the materials do not fluoresce, have minimal UV absorption and a lack of nitro groups – all necessary parameters upon which many traditional detection techniques are based [3]. Electrochemical sensors in this context offer an opportunity to detect peroxide based explosives that would otherwise prove problematic.

The development of single use processes to enable disposable electrochemical sensing has become more desirable for portable explosive sensors. The use of a Nafion (TM) coated, pre-anodized screen-printed carbon electrode that increased the sharpness of peaks and, therefore, the ease of identification of substituent groups

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upon the nitro-aromatic compound has been reported [4]. Recovery of TNT from spiked lake water was 95.7%, with this complex matrix only affecting the sample slightly.

Electrochemical instrumentation has evolved broadly since the early work of Nicholson and Shain on electroanalytical chemistry. In this present study, we fabricated an amperometric sensor to detect trace amounts of peroxides based explosives, namely, hexamethylene triperoxide diamine (HMTD) and triacetone triperoxide (TATP), and TATP precursors-hydrogen peroxide (H_2O_2) and acetone. Detection of peroxides, H_2O_2 , using electrochemistry is not a new concept [5,6]. In fact, an effective electrode was recently fabricated to detect 6 ppbv of H_2O_2 using a Prussian blue based electrode [7]. Also, previous work had attempted to electrochemically detect TATP using a solid-state sensor coated with a layer that selectively forms a bond with the vapor [8]. The exact limit of TATP detection was not established in that study.

Cyclic voltammetry is the most popular and convenient electrochemical technique, and usually the first experiment of this kind to be performed. It owes its versatility to its ability to deduce reaction mechanisms with relatively low cost equipment and quick experimentation, and together with chronoamperometry establishes undisputable technique that find useful in sensing trace amounts of explosive materials.

Staircase voltammetry becomes useful where surface confined effects are important. In this mode, the data is sampled throughout the duration of the step and averaged thereby eliminating the differences between a staircase and a true ramp. Typically, in staircase voltammetry, a current reading is acquired immediately prior to the next step [9]. This method of sampling discriminates against any capacitive or surface bound reactions. The current due to any capacitive charging or Faradaic current confined to the surface, decays in the initial part of the step and does not contribute to the measured current. The limiting currents which are proportional to concentration of analyte under study, resulting from electroactivities, are studied by double step chronoamperometry.

In this present study, staircase cyclic voltammetry (CV) and chronoamperometry techniques were utilized to investigate the ability to detect trace amounts of some peroxide based explosives and their precursors, namely, TATP and HMTD, acetone, and H_2O_2 .

2. Materials and methodology

2.1. Instrumentation and reagents

Amperometric measurements (chronoamperometry and voltammetry) were performed with a 1000 Potentiostat-Galvanostat-ZRA, electrochemical interface analyzer (Gamry Instruments, USA) along with carbon patterned electrode transducers and a $2 \times 10^{-5} m^3$ electrochemical cell from Pine Instruments. The patterned transducer cards consists of disk screen printed conductive carbon inks as the indicator and auxiliary electrodes, and screen printed silver/silver chloride, Ag/AgCl, inks as the reference electrodes. Overall card dimensions are $15 \times 61 \times 0.36$ mm. The thickness of the carbon, silver, and insulating layers is typically 7–13 μm .

Stock solutions of 30 and 50 w% H_2O_2 standard solution were purchased from ACROS ORGANICS and used as received, agarose type I–A low electroendosmosis (EEO) – a movement of liquid through the gel (Sigma–Aldrich), KBr $\geq 99.99\%$ (Sigma–Aldrich), strengthening nail polish remover as source of acetone (CVS Pharmacy), TATP and HMTD were prepared in-house, ultrapure water ($0.06 \mu S cm^{-1}$ or $18.2 M\Omega cm$. Neither terminology is exclusive).

2.2. Preparation of solid electrolyte

Alkali metal bromides, are typical ionic salts, and are fully dissociated at near pH 7 in aqueous solution. For this reason, KBr serves as source of Br^- ions in acidic electrolyte. A $1.0 \times 10^{-4} kg$ of agarose I–A was dissolved under stirring in a $5 \times 10^{-6} m^3$ 0.2 M KBr solution. The mixture was slowly heated until all the agarose were solubilized. Immediately, at that state, $1.03 \times 10^{-7} m^3$ agarose droplets were coated on the electrodes surfaces covering the indicator, auxiliary and reference electrodes. Thus, the solid phase electrode/electrolyte (SPEE) interfaced was fabricated in this manner. The droplet was allowed to cool to room temperature and rapidly solidified to form a thin layer gel covering the three electrodes. It is important when heating the solution to dissolve the agarose I–A that evaporation be avoided so that the molarity of the initial KBr solution is preserved.

2.3. Device fabrication for alkali metal bromide amperometric cell

Voltammetric and chronoamperometric measurements were performed using the Gamry electrochemical interface analyzer mentioned previously. To generate vapors of representative test materials: TATP, HMTD, acetone, bottled H_2O and H_2O_2 , each sample was sealed in a $2 \times 10^{-5} m^3$ air tight vial bottle and allowed to equilibrate for 1.8×10^3 s. In order to compare the response of the peroxides based explosives, equal mass of TATP and HMTD were initially charged into the vial bottle. In our previous finding [10] an 8.66 ± 1.048 ng TATP has a vapor pressure of 4.80 Pa at 298 K for TATP synthesized using HCl catalyst as given by a GC–MS headspace approach. Since our present measurement is based on headspace approach, as in our previous work, this mass of TATP was part of the measurement carried out in this study. Subsequently, the SPEE was introduced into the $2 \times 10^{-5} m^3$ vial cell through an opening in its cap and sealed in place with tape. Agarose gel matrix facilitates the transport of charges and enables diffusion of materials through it [11–15]. Also, current and potential measurements were performed following 60 s exposure of the electrode to the vapor. This exposure time was to enable the SPEE to equilibrate with the test vapor. Digital staircase voltammetry and double step chronoamperometry were performed.

3. Results and discussion

3.1. Amperometry

The aim of this study is to develop a miniature sensor for peroxide based explosives for application at screening locations. In order to achieve this goal electroactive behavior of some peroxide explosives and their precursor species have been studied. The redox of TATP, HMTD, acetone (contained in the strengthening nail polish remover) and H_2O_2 in a KBr solid electrolyte environment were chosen for this purpose.

Preliminary investigation was conducted in order to set the redox potential by voltammetric experiment as shown in Fig. 1 Subsequently, potential for the double step chronoamperometry was stepped from +0.70 V to +0.96 V/Ag/AgCl where electrode reaction occurs corresponding to the diffusion-limited reduction–oxidation. There was no electroactive specie involved using the electrode by itself and as such, no redox peak was observed. When the solid electrolyte, 0.2 M KBr, was incorporated into the electrolytic cell, the resulting voltammograms produced reduction peaks. In the voltammetry experiment, the fast sweep rate produced more current as expected. This was due to larger concentration gradients formed because of reduced time for the bulk solution to diffuse

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