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Non-destructive evaluation of an activated carbon using microwaves to determine residual life

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ABSTRACT

This paper describes a novel technique for determining the residual life of activated carbon material through changes in response when placed inside a resonant cylindrical cavity excited at microwave frequencies. The technique has been validated through the co-ageing of two activated carbon batches at 40 °C and 80% relative humidity over a period of 7 months. Two regions in the microwave spectra, 7500 and 8595 MHz, have been identified to exhibit similar transmitted signal attenuation: 0.25 and 0.35 dBm/month, respectively. Breakthrough curve analysis using (CN)2, CNCl and HCN confirmed that the samples were indeed deteriorated over the experimental period. Since activated carbon derives its filtration properties from both porosity and impregnants contained in the structure, the results obtained here suggest that there is a strong correlation between the response of activated carbon at microwave frequencies and amount of time that the activated carbon is exposed to a humid atmosphere. As a result, it is possible to produce calibration curves which can be used to predict the residual life of the activated carbon material in real-time.

1. Introduction

Whether paint fumes, cleaning solvents, industrial or military toxic gases contaminate air, gas masks with activated carbon filters are needed to minimize adverse health effects and to protect personnel from possible exposures. Adsorption rate from flowing air is one of the parameters which determine breakthrough times (i.e. the service life) of carbon filters subjected to vapours and gases. Other parameters include gas or vapour properties, adsorption capacity, carbon properties and use conditions, such as relative humidity (RH), temperature, airflow rate, and vapour concentrations [1].

Activated carbons include a wide range of processed carbon based materials all of which have an amorphous structure. Activated carbon incorporates an extensive network of pores of various dimensions, which are categorised by the International Union of Pure and Applied Chemistry into three classes, namely macropores (diameter > 50 nm), mesopores (2 nm < diameter < 50 nm) and micropores (diameter < 2 nm) that have relatively strong potential wells in which foreign molecules can be adsorbed, namely physisorbed [2]. As a result they are excellent and versatile adsorbents used heavily for filtration applications, such as clean rooms,[1] high value archives,[2] personal protective equipment PPE [3] (e.g. gas masks) and water treatment.[3] Traditional sources include coal [4], lignite [5], wood [6] and coconut shells [7], but it is also possible to derive activated carbon products from waste

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[2] Emcel Filters. SONOXOCARB Product Information Sheet. 2002 [cited 1/06/2012]; Available from: http://www.emcelfilters.co.uk/.

[3] CARBTROL Corporation. Granular Activated Carbon for Water and Wastewater Treatment. 1992 [cited 28/04/2011]; Available from: www.carbtrol.com/.

agricultural solids [8] and waste tyre rubber [9]. They are also available in a number of different forms, including powder, fibres and granules. The type used would usually be application dependant: gaseous applications would normally use the material in the granule form whereas in liquid based applications fibrous forms are often used due to low hydrodynamic resistance and their ease of moulding to any shape in the adsorption equipment.

The extensive porosity of activated carbon implies that it has a high specific surface area, typically 1000–1500 m^2/g (but as high as 2500 m^2/g [10]), which makes it a perfect support to deposit a large amount of solid [11], e.g. catalyst or reactant, known as the impregnant. Activated carbons are known to adsorb compounds with low saturation pressures more easily than compounds with high saturation pressures [2].

The prediction of breakthrough behaviour has been addressed in essentially two different ways. One is by solving a set of partial differential equations (PDE) that accurately describe the different steps and processes involved as the adsorbate passes through the filter bed. The second is by application of semi-empirical models that are based on an approximated theory of dynamic adsorption and empirical knowledge. For complex adsorbents, such as activated carbon, the PDE method requires a larger number of input parameters and more computational power than semi-empirical models to achieve comparable results [12].

In order to filter the gases that do not adsorb on activated carbons in the quantities required for gas mask applications, it is necessary to impregnate the activated carbon with compounds that will bind or complex (chemisorb) the targeted gas or alternatively break it down into nontoxic byproducts. The density of activated carbon is typically 0.5 g/mL and the loading of useful impregnant can be 3 mM of impregnant per gram of activated carbon [2].

Hydrogen cyanide (HCN) is a common chemical used in various industrial syntheses. It is a colourless, very volatile and very poisonous liquid, which boils at 26 °C. The derivatives of hydrogen cynanide are also acutely poisonous, and might enter the human body by breathing contaminated air. The HCN vapours are commonly released to the air from various sources including vehicle exhaust emissions, chemical processing, extraction of gold and silver from low-grade ores, metal plating, steel, iron, and finishing industries, petroleum refineries, and waste disposed of in landfills [13].

HCN is also known chemical warfare agent, so there is a risk of using it in case of terrorist acts. Because of risks of exposure to HCN, its vapour in air must be removed by appropriate gas mask filters. Since physical adsorption of HCN onto non-impregnated activated carbons is weak and, hence, reversible at room temperature, canisters for modern-day gas masks and collective protection filters are usually filled with impregnated activated carbons. The best-known adsorbents for the removal of HCN and cyanogen chloride from air are the metal salts impregnated activated carbons. Although the composition of impregnants and the process of impregnation vary, typical impregnants are salts of chromium, copper, silver, zinc, molybdenum, cobalt and organic compounds such as pyridine and triethylenediamine (TEDA) [14]. The nature of the metal ions is important not only for protection against the HCN breakthrough of the samples but it also impacts on the samples structure.

Activated carbon filters are often exchanged on a conservative basis since atmospheric exposure leads to ageing or deterioration, leading to inappropriate filter utilisation. If a filter is underutilised, its replacement may be premature therefore leading to waste and unnecessary cost. On the other hand, if a filter experiences an abnormal environmental condition then it may age more quickly than expected and thus not be replaced in time for safe use.

Thinking practically on the ageing of activated carbon filters, in military, fire-fighting or industrial accident situations mean that personnel have to respond quickly to mitigate the consequences of possible CNCl, HCN or $(CN)_2$ exposure. In such a case gas masks will be taken "from the shelf". These are not previously used, but could have been in storage or on site for a considerable time, or some event unknown to the personnel in charge could have resulted in their premature ageing. Although there are standard operating procedures in place across a range of organisations that would normally prevent this from happening, it would be beneficial to have some means of assessing canister life in real time. Since there is no technical means currently available to check the integrity of the gas masks immediately before their usage, it is just assumed that they are suitable, which is correct in most cases. However, there is a need to have a real-time portable system capable of confirming this assumption and this is the scope of this paper.

For end users of activated carbon filters there is no convenient non-destructive method for determining their residual life. The current means of experimentally predicting the life of an activated carbon product is to perform a breakthrough curve measurement [15], which involves measurement of how long it takes for undesirable gases to penetrate a filter. However, this method destroys the activated carbon filter and only gives an indication of the residual life once the filter has failed. Therefore the destructive nature of breakthrough curve excludes it from use as a real-time testing method.

The use of a novel real-time microwave technique based on the interaction of electromagnetic (EM) waves and a material under test is proposed. Microwaves are EM waves in the region of 300–100 GHz (or wavelength of 300 mm down to 3 mm), and are well known for use in sensing applications due to their response to the changing dielectric properties of materials in a range of applications such as healthcare [16–21], oil pipeline monitoring [22,23] and waste water monitoring [24–26]. An activated carbon filter ages over time due to atmospheric exposure since it adsorbs various gases including toxic from the medium it is present in. The composition of the activated carbon will change as a result of this, therefore leading to a dielectric change, which can be inferred through measurement of microwave power reflected from, and transmitted through, a sample of activated carbon. Microwave measurement techniques have been shown to offer excellent precision and speed, in addition to being versatile enough to accommodate a range of application requirements making them adaptable to a broad range of filter configurations and sizes.