# Determination of Activated Carbon Residual Life using a Microwave Cavity Resonator

**A Mason[1], S Wylie, A Shaw and A I Al-Shamma'a**
School of Built Environment, Liverpool John Moores University, Liverpool, UK.

**A Thomas and H Keele**
Dstl, Porton Down, Salisbury, SP4 0JQ, UK.

E-mail: A.Mason1@ljmu.ac.uk

**Abstract.** This paper presents the continuation of work conducted jointly between Dstl and LJMU. This unique body of work has been, largely, concerned with detecting the residual life of high performance filter materials using electromagnetic (EM) waves within a resonant cavity. Past work has considered both HEPA [1] and ASZM-TEDA[2] activated carbon filter materials. This paper continues the later work, considering the response of ASZM-TEDA activated carbon through the co-ageing of two distinct batches of the material. The paper briefly introduces activated carbon, discusses theory relevant to the work and the methodology used for investigation. A comprehensive set of results is included which seek to validate this technique for determining the residual lifespan of activated carbon.

## 1. Introduction

Activated carbon (sometimes also referred to as activated charcoal [3]) includes a wide range of processed carbon based materials, and has a microcrystalline structure. Its use in filtration is brought about by the fact that it has a high level of porosity and hence a large surface area where adsorption or chemical reactions may take place.

The preparation of activated carbons involves two main steps [4]: the carbonisation of raw material at temperatures less than 800°C in an inert atmosphere, and the activation of the carbonised product. During the carbonisation process, most of the non-carbon elements (e.g. oxygen, hydrogen and nitrogen) are eliminated as volatile gaseous species by the decomposition of the starting material. The residual elementary carbon atoms group themselves into stacks of flat sheets cross-linked in a random manner; the irregular arrangement of these sheets gives rise to pores. These pores are further enhanced through the activation process, which is usually carried out in an atmosphere of air, $CO_2$, or steam at 800-900°C. This results in an oxidation of some of the regions within the char in preference to others so that as combustion proceeds, a preferential etching takes place. This results in the development of a large internal surface area which can be as high as $2500m^2/g$ [5], although areas in the region of $500m^2/g$ to $800m^2/g$ are more typical [6].

Activated carbon is 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 ASZM-TEDA product under test in this work is in a granule form, and contains a number of inorganic impregnant. These are Copper, Silver, Zinc, Molybdenum and Triethlyenediamine, and are present in order to improve the effectiveness of the product against warfare gases [4, 7]. Figure 1 and 2 show a sample of the ASZM-TEDA product as viewed by a scanning electron microscope (SEM) at varying levels of magnification.

Activated carbons are excellent and versatile adsorbents, which inevitably leads to a wide number of commercial applications, including gas and water purification, sewage treatment as well as being used in air filtration for clean-rooms and respirators for volatile environments (i.e. gas masks). This later application was the initial driver for this work as issues has been raised in military circles relating to gas masks which utilise activated carbon filters. At present there is no convenient method for determining the residual life of a filter, which may age as a result of adsorbing harmful gases or simply as a result of being stored ready for future use. This has led to a conservative filter change policy which results in significant wastage due to useable filters being discarded.

Since beginning the work, the authors have also considered wider reaching applications of an activated carbon residual life indicator and it would appear that medium to large scale implementations (e.g. clean room and general building filtration) could benefit significantly. In these cases the filter material carries a high cost due to the volumes required, and therefore a technique which could reliably determine residual life could maximise cost effectiveness.

The aim of the research thus far has been to develop and validate an EM resonant cavity sensor for the purpose of residual life indication; the remainder of the paper focuses on this aim.
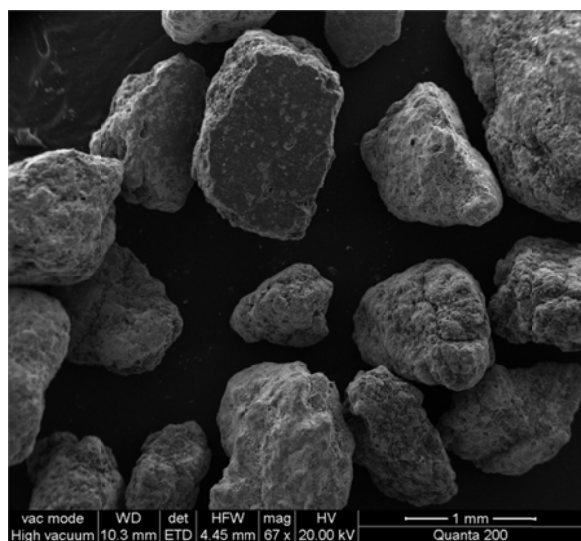


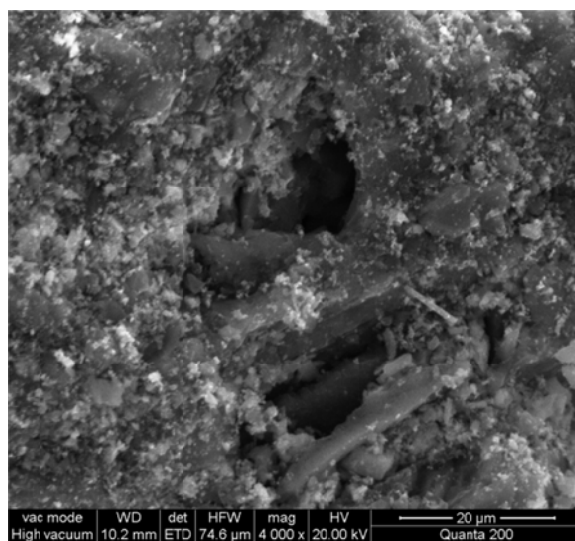**Figure 1.** Activated carbon granules as viewed with a SEM.



**Figure 2.** A highly magnified view of an activated carbon granule as viewed with a SEM, showing macroscopic surface pores.

## 2. Microwave Theory

Using microwave cavity resonator techniques with activated carbon began under the assumption that as the product is aged (i.e. through adsorption of material from the air) its relative permittivity ($\varepsilon_r$) must change.

One method which can be used to find the $\varepsilon_r$ of a substance is to monitor the effect it has on the resonant frequencies of an electromagnetic (EM) cavity. An EM cavity, consisting of a hollow structure with conducting walls, will resonate when it is excited at an appropriate frequency by a small

antenna placed inside the structure. The resonant modes occur when the electric and magnetic fields form standing waves, and so depend upon the internal dimensions of the cavity and the permittivity of any material placed inside. For a cylindrical cavity resonator the fundamental modes are $TE_{111}$ and $TM_{010}$. TE (Transverse Electric) modes have a magnetic component in the propagation direction and TM (Transverse Magnetic) modes have an electric component in the propagation direction. Each mode will generate a resonant peak with a quality factor (Q), which is inversely proportional to the power dissipated in the cavity for each applied EM oscillation. A high Q indicates a sharp resonant peak that will be more readily analysed and improve the accuracy of the sensor.

The resonant frequency for $TE_{nml}$ and $TM_{nml}$ modes in a cylindrical cavity [8] can be calculated using equation (1).

$$f_{nml} = \frac{c}{2\pi\sqrt{\mu_r \varepsilon_r}} \left[ \left( \frac{p_{nm}}{a} \right)^2 + \left( \frac{l\pi}{d} \right)^2 \right]^{1/2} \tag{1}$$

Where:

$c$ is the speed of light
$\mu_r$ is of the relative permeability
$\varepsilon_r$ is the relative permittivity
$p_{nm}$ is the $m^{th}$ root of the of Bessel function of the $n^{th}$ order for TM modes or the $m^{th}$ root of the first derivative of Bessel function of the $n^{th}$ order for TE modes
$a$ is the radius of the cavity
$d$ is the depth of the cavity

All EM modes therefore have the same dependence upon $\sqrt{\varepsilon_r}$, so when the cavity is excited by an appropriate range of frequencies and the resulting spectrum is captured, the resonant peaks corresponding to these modes will shift to lower frequencies as the permittivity is increased.


## 3. Experimental Setup

### 3.1. Sample preparation

In order to determine a microwave response to activated carbon relating to residual life, two batches (referred to separately in this document as Batch A and Batch B) of ASZM-TEDA activated carbon granules were artificially aged simultaneously at Dstl facilities.

Briefly, the ageing process included placing the carbon granules in shallow trays at a maximum depth of 2cm and exposing them to an atmosphere of 40°C and 80% relative humidity[1]. The carbon remained in this controlled environment for 7 months, being riffled on a weekly basis to ensure even exposure to the ambient air. Approximately 300g of carbon was removed from the trays each month and divided equally between two sealed containers. The material in one of the containers would then be used to fill a 15ml polypropylene centrifuge tube and sent to LJMU. The material weight varied with age due to water adsorption, although the majority of the weight increase was experienced within the first month as shown in Figure 3. The centrifuge tubes were filled via a "snowflake" filling method [9], which is designed to ensure homogeneity (i.e. minimal air voids and compression zones). This is noteworthy since compression of the carbon granules will inevitably increase EM wave attenuation due to a reduced skin depth [10].

The material in the second sealed container was retained by Dstl and tested for toxic gas breakthrough times. A variety of gases are used in order to test both the adsorptive and catalytic properties of the activated carbon. Figure 4 shows indicative breakthrough times for the co-aged

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[1] Further samples have been aged at 22°C but are not discussed in this paper.

samples. The results here are not as linear as one might hope, however they do at least show that the ageing method used appears to be working as intended since the performance of the carbon degrades with time.

## 3.2. Data acquisition

The apparatus used for testing of the carbon samples is shown in Figure 5. It comprises a Marconi 6200A microwave test set, capable of power magnitude measurements between 20MHz and 20GHz, and a cylindrical microwave cavity. The cavity allows for measurement of reflected ($S_{11}$) and transmitted ($S_{21}$) power. Both measurements are taken over the full frequency range of the test set using a bespoke software application which acquires data in defined frequency intervals in order to maximise data resolution. For the purposes of these experiments data was captured at 500MHz intervals, with 1601 data points per interval. Each carbon sample was tested 6 times consecutively and then the average of these measurements taken. In order to minimise the effect of temperature variation over the course of the 8 month test period, the apparatus and samples were kept in an environmental chamber which maintained a temperature of 20°C. It is assumed that once the carbon is placed into the polypropylene tubes little or no further ageing occurs, which has been proven by recapturing the sample spectra on a monthly basis.
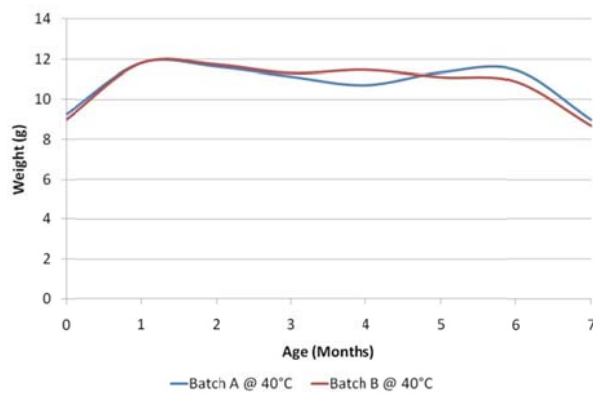


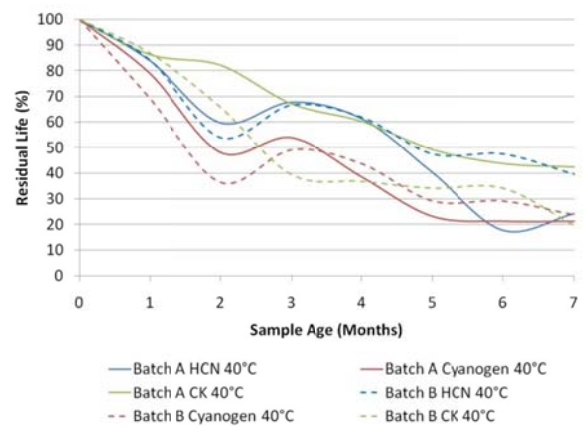**Figure 3.** Weight of activated carbon samples based on their age.



**Figure 4.** Indicative breakthrough times of the activated carbon samples from both co-aged batches.



**Figure 5.** Experimental setup for the resonant cavity sensor.

## 4. Results

Responses shown by materials exposed to microwave radiation in a resonant cavity are often specific to that material. Thus far, the aim of data analysis has been to seek logical patterns in the microwave spectrum recorded during the data acquisition phase. This has been a non trivial process considering the large amount of data per sample that which has been collated. For this purpose a bespoke SQL application has been developed which allows processing of the data in terms of variance calculations, normalisation of data and other methods of statistical analysis.

This statistical analysis is an on-going element of this work. However, searching for patterns in the data which correlate with sample age has proven fruitful. A number of regions, particularly in the $S_{21}$ data, have been discovered which indicate an approximately linear relationship between sample age and some characteristic feature of the acquired spectrum. The pattern search is configured such that it allows for inconsistent non-linearity in order to accommodate potential experimental error, signal noise or other potential anomaly. Two example regions are shown in Figure 6 and 7. The spectrum within each region is shown for batch A and B. Visual inspection of the Figures confirms that the response from each batch is similar, and follows a trend which can be correlated with sample age.
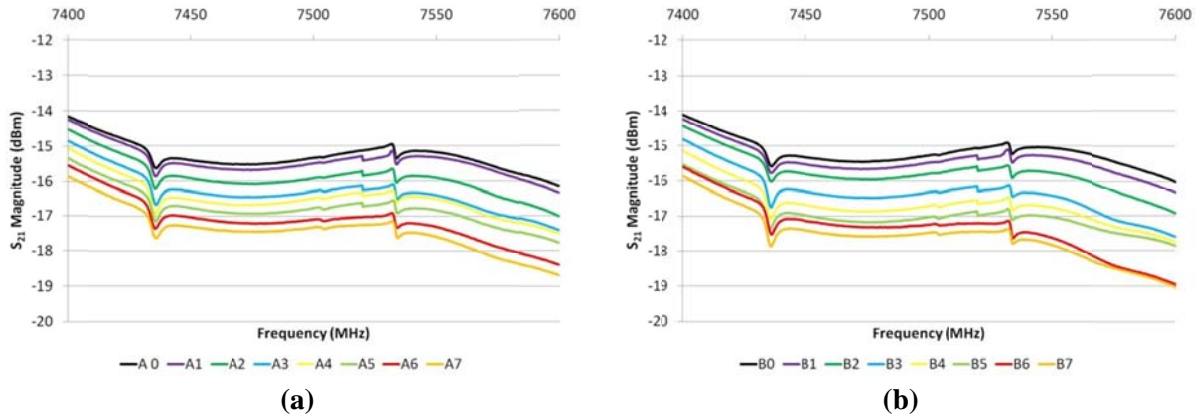


**Figure 6.** ASZM-TEDA activated carbon $S_{21}$ spectra in the range of 7400-7600MHz for (a) Batch A and (b) Batch B
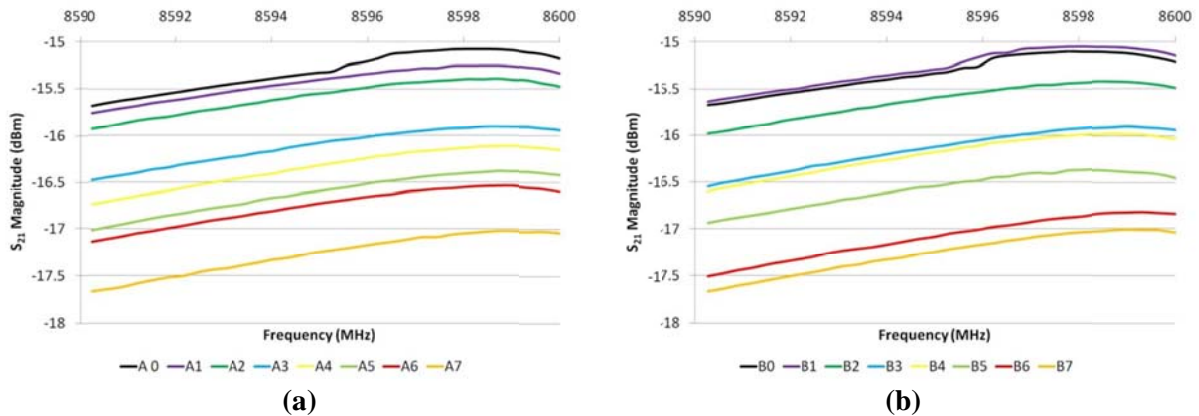


**Figure 7.** ASZM-TEDA activated carbon $S_{21}$ spectra in the range of 8590-8600MHz for (a) Batch A and (b) Batch B

Initially it was thought that the response shown might simply be due to increased water content in the carbon samples. During the ageing of the samples they are exposed to high humidity, therefore take on moisture from the atmosphere. However, the samples do this relatively quickly; the samples increase in weight significantly between months 0 and 1, but in subsequent months the weight remains relatively stable. This feature is highlighted in Figure 3. Therefore the measured response must be as a result of some other feature of the carbon, which appears to be linked to its ageing. It is known that the carbon is ageing due to the reduced gas breakthrough times shown in Figure 4. Since we know the useful lifetime of activated carbon from gas breakthrough testing, it is possible to correlate this with residual (or useful) remaining life. From the perspective of gas masks, the activated carbon material used in this work would not provide a significant amount of end user protection from harmful gases after 8 months of ageing; it would therefore require discarding.

## 5. Conclusions and Future Work

This paper describes a technique for determining the residual life of ASZM-TEDA activated carbon material through changes in EM response when placed inside a resonant cylindrical cavity. The paper builds upon previous work in this field [2], and has used a co-ageing method in order to demonstrate cross batch repetition; Figure 6 and 7 show this to be the case. In addition, we know that although the ageing process of activated carbon causes a significant weight gain (in the order of 70%), much of this occurs in the first month of ageing and therefore the sensor response is not simply to water. Ongoing work with the results obtained hope to discover correlations between EM response and carbon ageing at lower frequencies – principle component analysis (PCA) is being investigated as a potential tool in this process.

## 6. References

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