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# Activated carbon production from RDF and its use for dioxin removal from flue gas of waste incinerators

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ABSTRACT

**Aims:** The aims of this study were to produce activated carbon from refuse derived fuel (RDF) and to examine its efficiency in dioxins and furans (PCDD/F) removal for application in the adsorptive processes of waste incineration.

Materials and Methods: In the present study the RDF was carbonized at 600°C to produce char and the char was then steam activated at 900°C to generate activated carbon (AC). The efficiency of the produced AC in PCDD/F removal from simulated flue gas was investigated using a bench scale static-bed reactor. Experiments were conducted under flue gas stream at 275°C using a standard flyash (BCR-490) as a source of PCDD/F. The PCDD/F content of the exhaust gas was trapped by XAD-II resin, and after extraction and clean-up processes, were analyzed using a GC coupled with a triple quadrupole mass spectrophotometer (MS).

**Results:** The results showed an overall reduction of 85% and 43% in the flue gas concentration of PCDD and PCDF, respectively, which compared favourably with a commercial AC. In addition, reduction was varied throughout the congener groups of these compounds.

**Conclusions:** The AC derived from the pyrolysis of RDF therefore shows potential as a control material for dioxins and furans in waste combustion process.

Key words: Activated carbon, flue gas, incinerator, MSW, PCDD/F, RDF

# INTRODUCTION

Incineration of wastes are known as a major source of PCCD/F emissions to the environment.<sup>[1]</sup> A variety of end-of-pipe control techniques are used to continuously remove or reduce the emissions of these toxic pollutants

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to the recommended standard levels.<sup>[2]</sup> Applications of carbonaceous materials such as activated carbon, lignite or coke, either through injection or in a fixed-bed adsorber, are a commonly used method for the adsorption of PCDD/F from flue gas of waste incinerations. Several studies have been conducted to evaluate the performance of various activated carbon and their application methods on PCDD/F adsorption from waste incineration flue gas.<sup>[3-13]</sup>

For example, Everaert *et al.*, has examined the adsorption of PCDD/F from municipal and industrial waste incineration flue gases by activated carbon. The results showed an excellent PCDD/F removal efficiency either in the form of fixed-bed application or powder form injected into the

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waste gas.<sup>[5]</sup> In an Investigation using a laboratory-scale fixed-bed media adsorption system,<sup>[9]</sup> activated carbon has shown superior capacity in the adsorption of organo-chlorine compounds compared to activated coke and carbide wood. In a MSW incinerator, by applying a lime and activated carbon powder mixture (Surbalit) the dioxin level of the flue gas was reduced to less than 0.098 ng I-TEQ/N m<sup>3</sup>.<sup>[11]</sup>

Although the application of carbonaceous adsorbents for PCDD/F removal from waste gases has shown very good performance, the cost-effectiveness of the process should be taken into account. In this regard, waste-derived activated carbons may be a cheap alternative and have absorption properties similar to commercially available activated carbon. For example, a scrap tyre-derived activated carbon has shown a maximum BET surface area of 640 m<sup>2</sup>g<sup>-1</sup> with greater mesopore volume than micropore comparable to low grade commercial activated carbon.<sup>[14]</sup> In another study a refuse derived fuel (RDF) was pyrolyzed to produce char, and then the char was steam activated at 900 °C for 3 hours to produce activated carbon. The yielded activated carbon had a BET surface area of 500 m<sup>2</sup>g<sup>-1</sup> and total pore volume of 0.19 cm<sup>3</sup>g<sup>-1</sup>, comparable to low grade commercial one.<sup>[15]</sup>

The main objectives of this study were to produce activated carbon from RDF and to examine its PCDD/F removal efficiency for application in the adsorptive processes of waste incineration. The adsorption behavior of different PCDD/F congener groups on activated carbon and the influence of the physical characteristics of adsorbents on PCDD/F adsorption ability were also discussed. The PCDD/F adsorption efficiency of waste-derived activated carbon was compared with a commercial activated carbon (Norit 80) in the form of granules.

### **MATERIALS AND METHODS**

### **Raw samples preparation**

A sample of RDF from Malaysian municipal solid waste was pulverized and sieved to produce the particle size of 8 mm. The RDF sample was composed of 40 wt% carbon, 7 wt% hydrogen, 7 wt% nitrogen and 0.1 wt% sulphur.<sup>[16]</sup> A commercial activated carbon (Norit 80) in the form of granules was purchased from Sigma-Aldrich, UK. The granules was crushed and sieved to make a particle size of 1-2 mm.

### Pyrolysis of the refuse derived fuel

Pyrolysis of the RDF was carried out using a static-bed stainless steel reactor, 25 cm in length and 30 mm internal diameter. The reactor was externally heated by an electrical cylindrical furnace equipped with temperature control. About 8-10 grams of each sample was placed on a support inside the reactor, heated at 600 °C under nitrogen and held for 2 hours to obtain char, oil and gaseous products. A condensation system consisted of a water cooled condenser; two solid CO<sub>2</sub>/acetone cooled condensers followed by a glass wool containing bottle were applied to trap the condensable products from exhaust gas.

### Activation of the chars produced via pyrolysis

The chars obtained from the pyrolysis of the RDF sample were ground, and sieved to obtain 1.5-3 mm size fractions and dried at 105°C, overnight. Steam activation was carried out using the same reactor which was applied for pyrolysis, but with additional modifications to provide the steam activating agent [Figure 1]. About 4-5 g of the char was placed inside the reactor supported by a support bed and nitrogen was passed through the reactor in order to purge air from the system. The sample was heated gradually to attain the final temperature of 900°C. Deionized water, as an activation agent, was then introduced into the nitrogen gas stream passing through a pre-heater maintained at 300°C to generate steam, before entering the reactor. A Sage instrument (model 255-2) syringe pump was used to inject the deionized water into the gas line. Activation was carried out for 3 hours using a molar flow rate of 0.03-0.32 mol.g<sup>-1</sup>h<sup>-1</sup>of the activating agent.

### **Characterization of the activated carbons**

The surface area and pore structure of the waste-derived activated carbon (AC) and a commercial AC were determined by the nitrogen adsorption method using a TriStar 3000 gas adsorption analyzer (Micromeritics Instrument Corporation, Norcross, GA, USA). Metals content of the activated carbons was analyzed by preliminary acid digestion of the samples using EPA method 3050b<sup>[17]</sup> and analysis of the solutions by atomic absorption spectrometry using a Varian AA240FS instrument.

# Experimental reactor for PCDD/F adsorption by activated carbons

The PCDD/F adsorption capacities of the activated carbons were examined using a laboratory scale static-bed reactor [Figure 2]. The experiments were carried out at two stages. First, 2 g of the reference flyash sample (BCR-490) was heated at 275°C under a constant flow of simulated flue gas for 96 hours without applying activated carbon for PCDD/F adsorption. Next, the activated carbon media was placed inside the reactor after the flyash sample bed isolated by a glass frit and on the way of exhaust gas for adsorption of dioxin being released to the gas phase. Reactions took place on the flyash under a constant flow of simulated flue gas (4.8 vol% O<sub>2</sub>, 9.6 vol% CO<sub>2</sub> and 20.0 vol% H<sub>2</sub>O in N<sub>2</sub>) and the released PCDD/F was expected to be adsorbed on the activated carbon at the temperature of 275°C for 96 hours. Each test was carried out in duplicate and the PCDD/F contents of the exhaust gas was trapped in the XAD-II resin, extracted, fractionated and analyzed for both stages of the experiments.

#### PCDD/F trapping from exhaust gas of the reactor

Any PCDD/F released to the exhaust gas from the reactor was collected by a tertiary trapping system [Figure 2]. The first trap was an empty dreschel bottle located in an ice/water bath to act as a water vapour condensation system which was connected directly to the reactor's outlet tube. The second trap was a glass tube containing pre-cleaned Supelco amberlite polymeric XAD-II resin, held in place with pre-cleaned Orbo Hajizadeh and Williams: RDF derived activated carbon for dioxin removal



Figure 1: Schematic diagram of the reactor used in char activation process

polyurethane foam (PUF) plug. The resin was cleaned by solvent extraction and spiked with  ${}^{13}C_{12}$ -labelled 2378-chloro substituted PCDD/F standards prior to application. An empty dreschel bottle was used as a third trap to completely condense the exhaust gas and to capture any escaped PCDD/F species. The resin trap was covered with aluminium foil to prevent UV degradation of the dioxins.

#### **Chemical standards and organic solvents**

Method 1613 was applied for the analysis of PCDD/F in solid matrices.<sup>[18]</sup> A set of five calibration standard solutions  $(CS_1-CS_5)$  containing the 17 toxic congeners of native PCDD/F (2378-positional substitutes) and corresponding  ${}^{13}C_{12}$  isotope enriched PCDD/F with a purity of  $\geq$  99% were used. A <sup>13</sup>C<sub>12</sub> labelled standard stock solution containing 17 toxic congeners of PCDD/F was used to quantify the corresponding native compounds by the isotope dilution method. The XAD-II resin which was applied for trapping of PCDD/F from the exhaust gas of the reactor was spiked with the labelled standard prior to experimentation. Furthermore, an internal standard solution containing <sup>13</sup>C<sub>12</sub> labelled 1234-TCDD and 123789-HxCDD was added to the purified extract prior to GC injection as standards for the determination of recovery percentages of the labelled PCDD/F. All the standards were purchased from LGC Standards, UK; (supplied by CIL Inc. MA, USA). All the organic solvents such as Acetone, Toluene, Dichloromethane, n-Hexane, Ethyl acetate, used for extraction, clean-up and glassware rinsing, were certified grade for dioxin analysis and purchased from Sigma-Aldrich, Ltd. UK.



Figure 2: Schematic diagram of the modified reactor for PCDD/F adsorption tests

# Extraction of the resin trap, clean-up and PCDD/F analysis

At the end of each experiment the XAD-II resin with PUF plugs were extracted with 50/50% acetone/hexane at 100°C and pressure of 1500 psi using the Dionex ASE-100 extraction system.<sup>[19,20]</sup> The condensate in the first trap was liquid extracted three times with toluene and the washings added to the XAD-II resin extract. The extracts were evaporated in a rotary evaporator to reduce the solvent volume and the solvent exchanged to hexane before the sample clean-up procedure. All the glassware, dreschel bottles and reactor lining were washed with detergent, rinsed with toluene, hexane and acetone, and cleaned in an ultrasonic bath before use.

Isolation of PCDD/F from interferences was performed using an automated Power-Prep<sup>TM</sup> Fluid Management System (FMS), based on sequential use of acidic-basic multilayer silica, basic alumina and activated carbon adsorbents, pre-packed in disposable Teflon columns and hermetically sealed. The default dioxin clean-up method was set up and applied for the purifying the samples.<sup>[21]</sup> The collected fraction was evaporated and micro-concentrated by nitrogen blow-down to  $10 \,\mu$ L in nonane prior to instrumental analysis.

The PCDD/F analyses were performed using a gas chromatograph (GC) attached to a triple quadrupole mass spectrometry (GC-MS/MS QqQ) system. The operational characteristics and procedure adopted for the instrument have been described elsewhere.<sup>[22]</sup> The Varian 450-GC coupled with a Varian 320-QqQ-MS equipped with a Varian 8400 auto-sampler was

applied. The separation of PCDD/F was carried out using a Varian CP-Sil 88 capillary column for dioxins (50 m×0.25 mm I.D., 0.25  $\mu$ m film thickness). Samples were injected into the GC in split/splitless injection mode with the amount of 2  $\mu$ l. The retention time was categorized into several segments and the corresponding scan method for each segment was introduced in order to avoid the co-elution of PCDD/F isomers.<sup>[22,23]</sup>

Quantification of PCDD/F was carried out according to US-EPA method 1613 procedures using internal standard and isotope dilution methods.<sup>[18]</sup> Relative response (RR) and response factors (RF) for each individual compound was calculated with the corresponding calibration standard solutions. The results are reported in ng g<sup>-1</sup> and ng I-TEQ g<sup>-1</sup> of the sample dry mass. All the experiments were carried out in duplicate and the averages of the results were considered.

# RESULTS

The waste generated AC was characterized and the results was compared with a commercially available activated carbon which are shown in Table 1.

In Table 2 the concentrations of predominant metals in  $mg g^{-1}$  dry mass of carbons are presented.

The percentage removal of each of the 2378-substituted PCDD/F with respect to the activated carbon type at 275°C under the simulated flue gas stream are presented in Table 3.

Figure 3 shows the total toxic PCDD/F removal efficiency of RDF-AC in comparison with the commercial AC.

### DISCUSSION

### **Properties of the produced activated carbon**

The characteristics of the waste generated AC and their comparison with a commercial AC are shown in Table 1.



Figure 3: Total toxic PCDD/F removal efficiency of RDF derived AC in comparison with the commercial one

Although the properties of waste-derived AC were not comparable to the commercial one, they can be improved by developing the process of activation.<sup>[25]</sup> Nagano *et al.* (2000) carbonized an RDF by partial combustion at 350°C for 8 hours, then pre-treated by HNO<sub>3</sub> (3.3 N) for 3 hours and activated under N<sub>2</sub>-H<sub>2</sub>O flow at 850°C for 2 hours. The obtained AC had a BET (Brunauer, Emmit and Teller) surface area of 828 m<sup>2</sup>g<sup>-1</sup>, a micropore volume of 0.43 cm<sup>3</sup>g<sup>-1</sup> and a mesopore volume of 0.30 cm<sup>3</sup>g<sup>-1,[24]</sup> In spite of having surface area and pore volume lower than the commercial AC, the waste derived AC showed comparable potential of PCDD/F removal from flue gas stream.

### Metals analysis of the activated carbon by AAS

The concentrations of predominant metals in mg/g dry mass of carbons are presented in Table 2. The analyzed activated

Table 1: Properties of the compared to the commercia	RDF activated	carbon
Properties	Commercial	RDF
Activation Time (min)	NA*	180
Activation Temperature (°C)	NA	900
BET Surface Area (m <sup>2</sup> /g)	1019	333
Mesopore Volume (cm <sup>3</sup> /g)	0.034	0.07
Micropore Volume (cm <sup>3</sup> /g)	0.436	0.14
Total Pores (cm <sup>3</sup> /g)	0.47	0.21
Mean Particle Size (mm)	1.2-2	1.4-2.8

\*NA: Not available

Table 2: Met	al contents	of the	examined	activated
carbon samp	les (mg/g of	dry ma	ss)	

Metals	Commercial-AC	RDF-AC	
Aluminium	14.59	42.19	
Copper	0.03	0.13	
Cadmium	0.24	0.28	
Iron	2.94	11.16	
Mercury	4.34	4.42	
Magnesium	1.30	5.51	
Manganese	0.07	1.09	
Molybdenum	1.34	1.97	
Nickel	0.36	0.43	

carbons from exhaust gas (% w/w in I-TEQ values)				
Commercial-AC*	RDF-AC*			
87.6	86.7			
90.2	86			
89.6	83.3			
88.1	80.4			
90	77.5			
84.5	74.1			
76.6	69.8			
58.9	55.7			
81.2	79.1			
24.4	29.5			
69.3	65.7			
77	69.5			
90.8	88.8			
73	60.8			
62.8	49.8			
37.2	33.7			
70.3	82.2			
	as (% w/w in 1-TEC   Commercial-AC*   87.6   90.2   89.6   88.1   90   84.5   76.6   58.9   81.2   24.4   69.3   77   90.8   73   62.8   37.2   70.3			

\*AC = Activated carbon

carbons have got notable amounts of aluminium, mercury and iron. The presence of some catalytic metals such as nickel and a small amount of cupper were detected in the tested activated carbons. This may result in an increase potential of dioxin formation on the AC surface beside its adsorption from the gas stream.

# PCDD/F removal efficiency from flue gas by activated carbons

To assess the PCDD/F removal ability of the activated carbon the experimental reactor as described above was run without and with application of the carbon adsorbents and the PCDD/F contents of the exhaust gas were analyzed and the reduction by AC were calculated. Table 3 presents the percentage removal of each of the 2378-substituted PCDD/F with respect to the activated carbon type at 275°C under the simulated flue gas stream. The results are based on the difference between the concentrations of PCDD/F in the exhaust gas (vapour phase) with and without activated carbon adsorption. Thus the vapour phase PCDD/F removal efficiency of the applied activated carbons in wt% was calculated as follows:

PCDD/F removal efficiency (%) = [(PCDD/F<sub>WAC</sub>-PCDD/F<sub>AC</sub>)/PCDD/F<sub>WAC</sub>] × 100

Where, PCDD/F<sub>WAC</sub> is the concentration of PCDD/F in exhaust gas without activated carbon and PCDD/F<sub>AC</sub> is that with activated carbon use.

The application of AC produced from the pyrolysis of RDF and the commercial AC (Norit 80) showed significant reduction of PCDD/F concentrations in the exhaust gas. In general, the average PCDD/F toxic equivalency removal efficiencies of 58% and 57% were obtained respectively for the Norit 80 and RDF derived AC. These values appear low compared to the work of Abad *et al.*,<sup>[6]</sup> however, there is a huge difference in the initial concentrations of PCDD/F in the flue gas used in the two studies.

The adsorption capacity of activated carbon is highly dependent on its properties, particularly the surface area and pore size distribution. As can be seen in Table 1, even though the commercial activated had a higher porosity and a higher BET surface area, its performance in the adsorption of PCDD/F from the flue gas was similar to the RDF derived AC. By improving the activation methods, there is a possibility for the improvement of the properties of waste derived activated carbon.<sup>[25]</sup>

The results of this study are comparable with those obtained in the field using commercial AC. Application of a fixed-bed activated carbon filter filled with commercial AC with a BET surface area of 1000 m<sup>2</sup> g<sup>-1</sup> and a particle size of 4-6 mm in a hazardous waste incinerator led to an average dioxin removal efficiency of about 56%.<sup>[7]</sup> Within the PCDD congener groups the removal efficiency of activated carbons slightly decreased by increasing the level of chlorination. However throughout the PCDF congeners, although the minimum amount of 23478-PeCDF and 1234789-HpCDF (18-38%), and the maximum amount of 12378-PeCDF, 123789-HxCDF and OCDF (70-90%) were adsorbed, no clear trend in relation to their adsorption on the activated carbons was observed.

The average removal efficiencies of the total PCDD/F, PCDD and PCDF are illustrated in Figure 3 in wt/wt% I-TEQ values. In general, the activated carbons were much more effective and consistent in the removal of PCDD compared to the PCDF. Comparing the removal efficiencies of PCDD and PCDF, it can be seen that the average concentration of PCDD congeners adsorbed on activated carbon and thereby removed from the gas phase was approximately two times higher than that of PCDF. For example, only about 30% each of 23478-PeCDF and 1234789-HpCDF were removed in the presence of the activated carbons, whereas the lowest removal efficiency for any PCDD was in the region of 70%. However, as there were no appreciable differences in PCDD/F adsorption capacity between the waste derived activated carbon and the commercial one, their use could be a cost-effective option in PCDD/F removal from waste incinerator flue gases.

In relation to the results achieved in this study, it should be noted that the temperature of maximum formation of PCDD/F via de-novo synthesis is in the range of 200-400°C.<sup>[26]</sup> However, injection of activated carbon in full scale waste incinerator plants is generally at lower temperatures of around 180°C. Therefore the higher temperature of 275°C used in our experiments might influence the efficiency of PCDD/F adsorption onto the activated carbon. Thus, at lower temperature a PCDD/F removal efficiency of higher than our work for RDF derived AC can be predictable.

# **CONCLUSIONS**

The results have indicated that the RDF can be significantly converted to activated carbon and applied for flue gas treatment which is cost-beneficial than commercially available ones. The examined waste RDF derived activated carbon can remove the PCDD/F released to the exhaust flue gas by 57%. This result compared favourably with the result from the commercially available (Norit-80) activated carbon. The results also revealed that the removal efficiency for PCDD (average 85%) were much higher than the removal of PCDF (average 41%). The adsorption trends varied with the dioxin and furan congener groups. The results suggest that waste derived activated carbon shows potential as a considerably low-cost control material for PCDD/F in waste incinerator flue gases compared to the commercial carbon. However, it is important to bear in mind that this method not destroys the dioxins but transfers the gas phase dioxins to the solid phase (AC) in the flue gas cleaning systems. This would therefore require further advance treatments prior to its disposal to the environment.

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