



# Heel formation during volatile organic compound desorption from activated carbon fiber cloth



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## ABSTRACT

Heel formation on three activated carbon fiber cloths (ACFC10, ACFC15, and ACFC20, with increasing pore widths and volumes) was assessed after cyclic adsorption/resistive heating regeneration using 1,2,4-trimethylbenzene (TMB). Following regeneration at 288 °C, cumulative heel for ACFC10, ACFC15, and ACFC20 was 3.5 wt%, 4.1 wt%, and 4.2 wt%, respectively. Adsorption capacity of ACFC10; however, dropped by 83% compared to only 9% and 5% for ACFC15 and ACFC20, respectively. To prevent these capacity losses, regeneration temperature was increased to 400 °C. However, this increased cumulative heel and adsorption capacity losses to 3.7 wt% and 93% (ACFC10), 10.4 wt% and 51% (ACFC15), and 18.9 wt% and 50% (ACFC20), respectively. It is proposed that these observations result from carbon deposition due to thermal degradation of TMB during regeneration and rapid adsorbent heating. Increasing regeneration temperature enhances the endothermic decomposition processes, increasing heel buildup. Because the pores in ACFC10 are narrower than in ACFC15 and ACFC20, even small amounts of heel obstruct ACFC10's pores, explaining its notably larger decrease in adsorption capacity. These results suggest that rapid heating could be associated with reduced adsorbent performance. Regeneration conditions could be optimized to allow fast desorption with minimal adsorbate decomposition.

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## 1. Introduction

Automotive painting booths are the main source of volatile organic compounds (VOCs) in automotive manufacturing industries [1,2]. VOC emissions from painting and coating operations include aromatic hydrocarbons, esters, ketones, alcohols, and other organic compounds, with a wide range of molecular weights and boiling points (<240 °C) [1–3]. Adsorption is a common control technology for these emissions because it is efficient, low cost, allows for adsorbate recovery, and can control low VOC concentrations (ppb level) [4–9].

Activated carbon fiber cloth (ACFC) is an ash-free, porous carbon that provides high surface area, micropore volume, and adsorption capacity, making it an effective VOC adsorbent [5,10]. ACFC's internal micropores are connected to its external surface area,

decreasing mass and heat transfer limitations [5,11]. Higher mass and heat transfer rates improve adsorption/regeneration rates and decrease the risk of ignition inside the adsorption bed, respectively [12]. ACFC is more electrically conductive than other activated carbons because it consists of continuous fibers, making it a suitable material for electrothermal swing adsorption (ESA) [8,12–15]. ESA uses resistive heating to provide more rapid heating than more traditional conductive heating and steam regeneration methods, and it consumes less purge gas [8,9,16]. Additionally, unlike steam regeneration, ESA requires minimal condensate water separation after regeneration, and unlike hot gas regeneration, the adsorbent heating rate is independent of carrier gas flow rate, decreasing gas consumption [16].

Heel build-up is the unwanted accumulation of adsorbate on an adsorbent surface or in adsorbent pores after regeneration. It is a challenge associated with using activated carbon for VOC adsorption because heel decreases the adsorbent's capacity and corresponding lifespan, increasing operation and maintenance costs. Established heel formation mechanisms include strong physical

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adsorption [17], chemical adsorption [4,18–20], oligomerization [21–24], and adsorbate decomposition [25–27]. Adsorbate decomposition depends most prominently on the regeneration conditions used for desorption. Change et al. confirmed that thermal decomposition of aromatic hydrocarbons can occur when they are exposed to high temperatures (450 °C) [28]. Ania et al. found that pore blockage attributed to high temperature decomposition of adsorbed phenols occurs during microwave regeneration of activated carbons at 850 °C [26]. Pore blockage in a powder activated carbon was similarly attributed to adsorbate (promethazine hydrochloride) decomposition during microwave regeneration at moderate temperature (300 °C–500 °C) [25].

Adsorbate decomposition is a justified concern when heating adsorbents for regeneration, but the limited number of studies in the literature are inconclusive regarding the specific cause. For example, while activated carbon fiber cloth is a widely studied VOC adsorbent and resistive heating is an extensively tested carbon regeneration method, there has been no description of heel formation associated with adsorbate decomposition during their use. Identifying the adsorbent and adsorbate properties and the regeneration conditions that favor adsorbate decomposition is essential, but has not yet been described. The main objective of this research, therefore, is to identify reason(s) and mechanism(s) for heel formation resulting from the adsorption and desorption of organic vapors on ACFC.

## 2. Experimental

### 2.1. Adsorbent and adsorbate

The adsorbent was activated carbon fiber cloth (ACFC) prepared from phenol-formaldehyde resin precursor (Nippon Kynol Company). Three different ACFCs were used (ACC-5092-10 (ACFC10), ACC-5092-15 (ACFC15), and ACC-5092-20 (ACFC20)), having different levels of activation and corresponding to different surface area and pore size distributions. Prior to use, all ACFCs underwent 2 h of heat treatment at 288 °C or 400 °C, depending on the selected regeneration conditions, to remove adsorbed water vapor and

cartridge, an adsorbate generation system, a gas measurement system, a heat application module, and a data acquisition and control system (DAC) (Fig. 1). The cartridge consisted of a 3-layer annular ACFC cartridge (1.65 cm inner diameter, 10 cm length) attached to two stainless steel electrode tubes (1.65 cm outer diameter). Because the ACFC samples have different areal densities, different masses were required to prepare the 3-layer cartridges (5.5 g, 4.5 g, and 3.5 g for ACFC10, ACFC15, and ACFC20, respectively). The prepared cartridge was mounted inside a quartz tube (7.3 cm inner diameter, 40 cm length).

The inlet adsorbate concentration was 500 ppm<sub>v</sub> for all experiments. The adsorbate generation system used a syringe pump (New Era Inc., NE-300) to inject liquid organic compound into a dry air stream maintained at 10 SLPM (standard conditions are 25 °C and 1 atm) with a mass flow controller (Alicat Scientific). VOC concentrations were measured using a flame ionization detector (FID, Baseline-Mocon Inc., series 9000). The VOC concentration in the reactor's outlet stream was measured continuously during adsorption (2 min sampling frequency), and the FID was calibrated before each adsorption test using the same gas generation system.

The heat application module for adsorbent regeneration included a silicon-controlled rectifier (SCR) to apply voltage to both sides of the ACFC cartridge. A 1.6 mm outer diameter, ungrounded, type K thermocouple (Omega) was used to measure the temperature at the center of the ACFC cartridge during adsorption and regeneration. The DAC system consisted of a LabVIEW program (National Instruments) and a data logger (National Instruments, Compact DAQ) equipped with analog input/output modules. The data logger was interfaced to the FID and thermocouple to record outlet VOC concentration during adsorption and temperature during adsorption and regeneration. A proportional-integral-derivative control algorithm and SCR were used to control the voltage applied to the cartridge to achieve and maintain the set-point temperature.

After FID calibration, the gas stream containing organic vapor was introduced to the reactor, and outlet VOC concentration measurement began, generating a breakthrough curve. Adsorption was done at 25 °C and continued until the ACFC cartridge was saturated

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$$\text{Amount adsorbed}(\text{wt}\%) = \frac{\text{Cartridge weight after adsorption} - \text{Cartridge weight before adsorption}}{\text{Weight of heat treated ACFC}} \times 100$$


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VOCs. These samples are labeled as “heat treated ACFC”. 1,2,4-trimethylbenzene (TMB, 98%, Acros Organics) and acetone (99.8%, Fisher Scientific) were the adsorbates. Acetone is small (kinetic diameter = 0.44 nm [29]) with a low boiling point (56 °C), while TMB is bulkier (kinetic diameter = 0.61 nm [30]) with a higher boiling point (171 °C).

### 2.2. Experimental set-up and methods

The experimental set-up includes an adsorption/regeneration

(approximately 2 h). Adsorption capacity was calculated as follows:

Regeneration was completed by heating the ACFC cartridge at 288 °C or 400 °C for 100 min and then cooling for 30 min while purging with 1 SLPM N<sub>2</sub>. These temperatures are similar to temperatures used to regenerate beaded activated carbon treating VOCs from automotive painting operations [1]. When cool (25 °C), the cartridge was again weighed. The difference in the cartridge mass before adsorption and after regeneration is the amount of heel build-up during that cycle. Cumulative heel after five adsorption/regeneration cycles is described as:

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$$\text{Cumulative heel}(\text{wt}\%) = \frac{\text{Weight after 5}^{\text{th}} \text{ regeneration cycle} - \text{Weight before 1}^{\text{st}} \text{ adsorption cycle}}{\text{Weight of heat treated ACFC}} \times 100$$


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