

Degradation Processes in Corona-Charged Electret Filter-Media with Exposure to Ethyl Benzene

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ABSTRACT

The degradation of filtration performance for corona-charged electret filter media exposed to ethyl benzene was assessed. Nonwoven corona-charged polypropylene fiber mats were exposed to ethyl-benzene using a custom made apparatus. Evaluated scenarios included ethyl-benzene vapor and liquid exposures. The filtration performance was evaluated using DOP as a test aerosol to measure filtration performance. It was observed that significant filtration degradation occurred only when liquid ethyl benzene came into direct contact with the filter media. No significant changes in the pressure drop or filtration efficiency was observed for any of the exposure scenarios in which the fibers were only exposed to ethyl benzene in the vapor phase.

Keywords: electret filter, degradation, ethyl benzene

INTRODUCTION

An electret is a solid dielectric with a quasi-permanent electret moment. Electrets exhibit an external electric field in the absence of an applied field [1]. At the microscopic level, electrets can be classified as real-charge electrets or dipolar-charge electrets. Real-charge electrets consists of injected or embedded charges within the dielectric, while dipolar-charge electrets are composed of permanent dipoles at the molecular level. The situation becomes complex as an injected charge in a polymer will induce dipole moments in a region near the excess ion. The macroscopic shape of the electret can be a film or fiber. Dipolar-charged electrets can be formed by heating up the material, typically well above the glass transition temperature in the presence of a strong external electric field, and then cooling the material while the field is maintained, thus orienting the dipoles in a semi-permanent configuration. Real-charge electrets can be formed by exposing the material to a charged plasma or corona [2].

One of the benefits of electric filters is their ability to remove fine particles with high filtration efficiencies while maintaining a low pressure drop. This is especially important for particles in the .1 to .3 μm size range, or what is commonly denoted as the most penetrating particle size. The electrical charge plays a crucial role in the filtration mechanism, as the aerosol particles are attracted electrostatically to the fiber. One noticeable difference between charged and uncharged filter media is the distribution of captured particles on the fibers. For uncharged filter media, the particles are primarily distributed on the upstream side, whereas for electret filters, the particles are more uniformly distributed around the fibers. As particles are captured on the fibers, they tend to mask or neutralize the electrostatic field, resulting in a drop in filtration efficiency.

The distribution of injected sites for real-charge electrets has been an active area of research [3]. Bulk measurements of net charge, measured on the millimeter or larger scale tend not to give an accurate prediction of the effective field strength in the dielectric. The reason for this is that even though at the atomic level there could be regions of highly positive and highly negative charge, far away the effects cancel and the bulk charge could appear to be charge neutral. Furthermore, electrostatic capture of neutral particles is a function of the gradient of the electric field, and not the field itself [4]. The exact location of trapped charges in polypropylene electret media is still an open question, as is the exact distribution of charge. For particles of .3 μm or smaller, one needs to measure the electric field at the tens of nanometer scale, which is several orders of magnitude below most published results. Recently, Jasper and Hinestroza [5] have been able to measure the electric field on a 4 μm fiber to a resolution of a tens of nanometers using Electrostatic Force Microscopy, or EFM.

Although the exact charge distribution is not known, one can estimate the upper limit for the charge distri-

bution on the surface of the fiber using Gauss's Law.

$$\epsilon_0 \oint \mathbf{E} \cdot d\mathbf{S} = q \quad (1)$$

where q is the total charge enclosed by the surface \mathbf{S} . We can use Gauss's Law to calculate the maximum surface charge density by noting that the magnitude of the electric field can not exceed $3 \times 10^6 \text{ Vm}^{-1}$, the ionization potential for air [6]. If we choose as our surface a cylinder just larger than the fiber, we obtain

$$E = (Q_s \times S)/(S \times \epsilon_0) \quad (2)$$

therefore the maximum charge density becomes

$$Q_s = 3 \times 10^6 \times 8.85 \times 10^{-12} = 2.65 \times 10^{-5} \text{ C m}^{-2} \quad (3)$$

This is the maximum charge density that can be sustained on any surface in air. An elementary charge (proton or electron) has a value of $1.60 \times 10^{-19} \text{ C}$, which corresponds to 1.6×10^{14} extra charges per square meter on the surface. The atomic density of polypropylene, one radius of gyration¹ deep (or about 16 nm) can be shown [7] to be on the order of $3.5 \times 10^{18} \text{ atoms m}^{-2}$. Therefore, if the charge resides on or within a radius of gyration of the surface, only 1 in 22,000 atoms can at most be charged.

EXPERIMENTAL

Materials

Three different types of eletret filter media as well as one type of mechanical filter media were used in this study. Technical specifications for these materials are shown in Table I. All filter media were tested as received from the manufacturers. The eletret filter media was composed of melt-spun polypropylene nonwoven mats charged by means of corona treatment. The mechanical filter media consisted of a nonwoven mat of glass fibers without electrostatic charge. The chosen materials are typically used as components in commercially available particulate respirators rated by NIOSH as R, P and N.

ACS grade ethyl benzene, di-octyl-phthalate (DOP) (also known as diethylhexyl phthalate) was obtained from Sigma Aldrich (Milwaukee, WI) and used as received. Commercial grade nitrogen was used as the carrier gas, and it was provided by Machine and Welding Supply Company (Dunn, NC).

¹The square of the mean radius of gyration of a polymer chain or coil is defined as the average square distance of the chain segments from the center of mass of the chain or coil.

Table I: Filter Media Specifications

Test Code	Basis Weight (g/m^2)	Thickness (mm)	Fiber	Charge
E1	112	0.99	Meltblown Polypropylene	Corona
E2	185	1.78	Meltblown Polypropylene	Corona
E5	80	0.76	Meltblown Polypropylene	Corona
Mechanical	78	0.40	Microglass	N/A

Analytical Instrumentation

A CertiTest 8130 automated filter tester was used to determine the performance (percentage of penetration) and pressure drop across the filter media. The CertiTest 8130 is manufactured by TSI Incorporated in St. Paul, MN. In this work, DOP was used in the oil aerosol generator to produce particles with a count median diameter of $0.20 \mu\text{m}$ and a geometric standard deviation of less than 1.6. The filter media were tested as flat circular samples which were 15.2 cm in diameter. All the tests were done according to 42 CFR 84 [8] for challenging respirator filter media at a flow rate of 42.5 lpm.

A gas chromatograph (GC-17A Shimadzu Instruments, Columbia MD) equipped with a 30 m capillary column, 0.53mm ID (Stabilwax[®] crossbond[®] carbowax[®]-PEG) and a flame ionization detector was used to determine the concentration of ethyl benzene in the gas entering and leaving the exposure apparatus. The accuracy of the method was corroborated using calibrated mixtures certified by The Specialty Gas Group (Raleigh, NC). The maximum exposure concentration of ethyl benzene in nitrogen was found to be %1.275, which was in agreement with the saturation values predicted by simulations [9].

Filter Media Testing

Filtration evaluation methods used in this work are described in detail on 42 CFR 84, which establishes performance criteria for all negative pressure particulate air-purifying respirators and pre-filters. The filter media specimens were placed on the lower portion of the filter holder, exposing an area of 182.4 cm^2 to the test agent. The DOP aerosol traveled from the generator to the filter holder and down through the filter media.

Two solid-state laser photometers measured the aerosol concentration upstream and downstream of the tested specimens. Filter penetration was determined

from the ratio of the two aerosol upstream and downstream measurements. Electronic pressure transducers located upstream and downstream of the test holder were used to determine pressure drop across the filter media. All the signals and values were time stamped and stored in a SQL database.

Exposure Apparatus

Nitrogen was bubbled through a three nozzle rounded vessel containing liquid ethyl benzene in order to generate a saturated vapor. Gas flow through the saturator was controlled at $1000 \text{ cm}^3 \text{ min}^{-1}$ using a flow-meter. The stream of saturated vapor was connected to the exposure chamber using Tygon tubing. By measuring the gas concentrations up and down stream of the filter sample and the flow rate, one can calculate from mass conservation the molecular fraction of gas in the exposed chamber and the rate of liquid ethyl benzene deposition onto the filter media. This value is temperature dependent, as the ethyl benzene in the gas phase precipitates onto the fiber when cooled below its dew point temperature.

The vapor exposure chamber was a custom design that was fabricated by Prism Research Glass, Inc. (Research Triangle Park, NC). The vessel was a round tube closed on one end with a spherical bottom. A base was molded to the tube so that the chamber would be free standing. A shelf was pressed into the edges of the glass tube that would serve, in conjunction with a matching glass ring, as a means to suspend the sample materials in the middle of the vessel. Two nozzles were attached to the vessel on opposite sides, one above and one below the shelf. The top closure was achieved by adding a flange and grinding the surface to match a ground-glass lid. The interface was sealed with inert grease. The exhaust of the exposure chamber was connected to a common ventilation system and was intermittently sampled with the gas chromatograph to verify the ethyl benzene concentration. The temperature of the exposure apparatus and the gas bubbler was controlled at 25 degrees Celsius. A schematic of the experimental setup is shown in Figure 1.

Exposure Experiments

The purpose of this work was to evaluate the performance of electret filter media after being exposed to ethyl benzene in gas and liquid phases. In order to generate two-phase flow, ice packs were placed at the entrance nozzles of the exposure chamber. Four sets of experimental conditions were devised to decouple the effect of exposure to the gas and liquid phases of ethyl

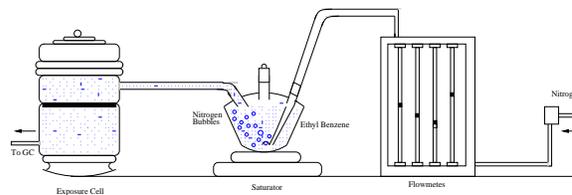


Figure 1: Experimental Setup for Chemical vapor exposure tests.

benzene.

The first set of experimental conditions introduced the saturated gas into the exposure apparatus using the upper nozzle and exhausting through the lower one. This set of experimental conditions was labeled as *downward flow*. The second set of experimental conditions introduced the saturated gas into the exposure chamber through the lower nozzle and exhausted through the upper nozzle, and will be referred in this paper as *upward flow*. The third experimental condition resembles the downward flow experimental condition with the difference that ice packs were placed around the outside of the upper chamber in order to generate a two-phase flow. This condition is referred to as *ice-bath downward flow*. The fourth experimental condition introduced the saturated gas into the exposure chamber using the lower nozzle and exhausted through the upper nozzle like condition two, except that ice packs were placed around the lower chamber. This condition is referred to as *ice-bath upward flow*.

During experimental conditions three and four (ice-bath downward and ice-bath upward flow), liquid ethyl benzene was observed at the bottom of the exposure chamber confirming the presence of a two-phase flow. It was evident that only during experimental condition three (ice-bath downward flow) was the filter media directly exposed to liquid ethyl benzene.

RESULTS

A total of 80 experiments were carried out using DOP aerosol as a test agent. Four repetitions were performed at each one of the four experimental conditions for the filter media selected for this study, plus 16 baseline tests for filters not exposed to ethyl benzene.

Baseline Tests

Figure 2 illustrates the baseline performance and pressure drop for filter media not exposed to ethyl benzene. As expected, electret filter media E1, E2, and E5 ex-

hibited lower pressure drops than the mechanical filter media. The filtration performance of specimen E5 showed a linear time dependence.

While E1, E2 and the mechanical filter media samples exhibited negligible changes in penetration as a function of time, E5 exhibited a monotonic increase in penetration as a function of time. This behavior was expected, as E5 is not designed to filter liquid aerosols. What is important to note, is that after 1 minute of exposure to DOP at a flow rate of 42.5 lpm, the penetration did not exceed 3% with a standard deviation of less than 0.2 for all the filters.

Exposure Tests

Figures 3-5 show the performance of filter media as a function of exposure time under the four experimental conditions stated previously. For all three electret filter media specimens, E1, E2, and E5, a marked decrease in performance (increase in penetration percentage) was noted for experiments performed under experimental condition three (ice-bath downward flow). Negligible changes in performance were noted for experiments carried out under experimental conditions one, two and four.

Experimental condition three, ice-bath downward, was the only one in this study in which liquid ethyl benzene was in direct contact with the filter media. Liquid ethyl benzene was observed at the bottom of the exposure chamber during these experiments as the presence of two phase flow was also predicted by thermodynamic simulations [9]. During experimental condition four, liquid ethyl benzene was observed at the bottom of the exposure chamber, but due to the direction of the flow, the liquid was not in direct contact with the filter media.

Since an increase in aerosol penetration was only noted when the filter media was directly exposed to ethyl benzene in the liquid phase, the decrease in performance could be attributed to a reduction in the filter media's static electric field caused by the creation of a thin layer of liquid ethyl benzene over the surface of the charged polymer fibers. All samples were air dried for 24 hours before testing, and no measurable increase in weight due to ethyl-benzene exposure was noted.

Based on Figures 3-5, it is clear that ethyl benzene in the gas phase did not alter the performance of electret filter media for exposure times up to eight hours. This behavior may be explained considering that the sorption and diffusion process for ethyl benzene into polypropylene occurs at different time scales with diffusion being the rate-limiting step.

Table II: Performance evaluation of filter media as received (Baseline Test).

Material	Flow-rate lpm	Penetration (%) (DOP @ 100 mg)	Pressure Drop (Pa)	Fiber Diameter (μm)
E1	42.5	<1	150	1.7 - 6
E2	42.5	<3	150	1.7 - 6
E5	42.5	<15	100	1.8 - 6.5
Mechanical	42.5	<0.02	500	0.5 - 9.0

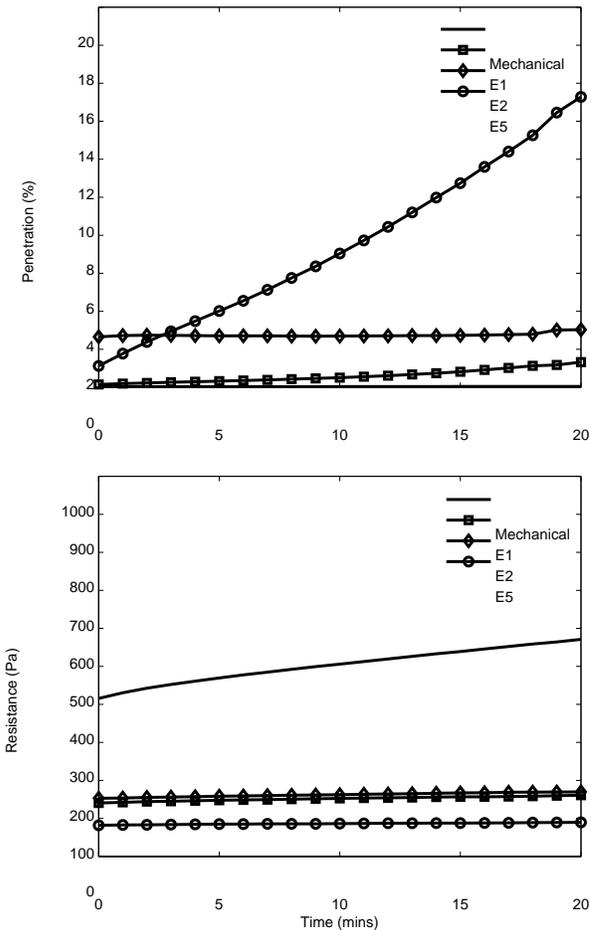


Figure 2: Baseline Performance Data

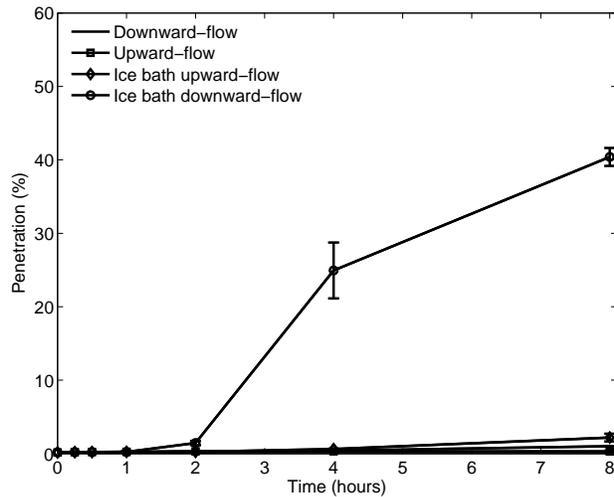


Figure 3: Experimental Results of E1 specimen exposure to ethyl benzene.

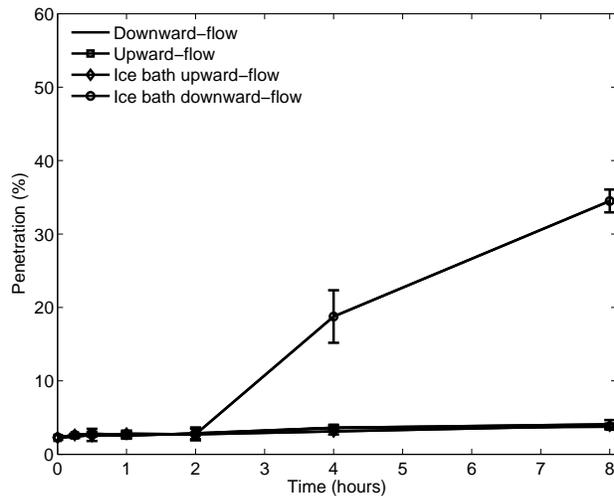


Figure 4: Experimental Results of E2 specimen exposure to ethyl benzene.

CONCLUSIONS

A set of experiments were conducted to determine the effect of exposure to ethyl benzene on the performance of electret filter media. It was established that the penetration percentage of DOP aerosol through the electret filter media was not affected by exposure to ethyl benzene vapors for up to eight hours.

Direct contact of electret filter media with ethyl benzene in the liquid phase caused a decrease in filtration performance up to 40%. The decrease in performance was attributed to changes in the static electric field caused by the presence of liquid ethyl benzene on the surface of the fiber.

The performance of the mechanical filter media was unaffected by the exposure to ethyl benzene in either the liquid or gas phases.

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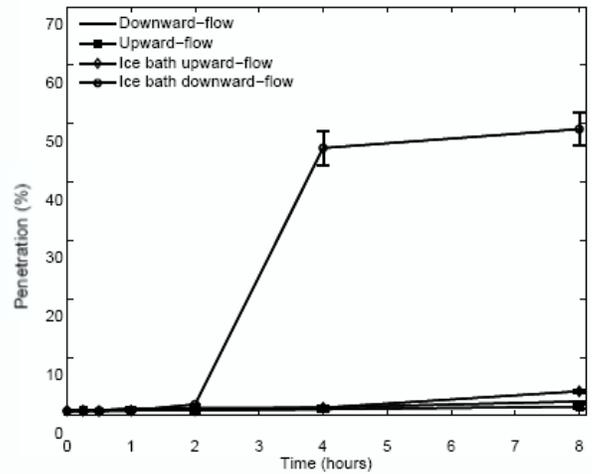


Figure 5: Experimental Results of E5 specimen exposure to ethyl benzene.