Filter Performance Degradation of Electrostatic N95 and P100 Filtering Facepiece Respirators by Dioctyl Phthalate Aerosol Loading

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ABSTRACT

Polydisperse dioctyl phthalate (DOP) aerosols are employed for testing filter penetration with loading of R- and P-series particulate filters for National Institute for Occupational Safety and Health (NIOSH) certification. Penetration for filters must not exceed NIOSH allowed maximum levels during the entire loading of 200 mg DOP indicating no filter degradation. Degradation of respirators loaded with DOP by other aerosols as well as respirators exposed to both oil and non-oil aerosols found in some workplaces is not well studied. To better understand the degradation of respirators with electrostatic filter media, two models of N95 and P100 filtering facepiece respirators (FFRs) were loaded with polydisperse DOP aerosols up to 200 mg as employed for NIOSH certification testing with simultaneous measurement of filter penetration. In parallel experiments, both N95 and P100 FFRs were loaded with 10 to 200 mg DOP and challenged with polydisperse NaCl aerosol employed for NIOSH certification testing as well as monodisperse NaCl aerosol, and filter penetration was measured. Results showed that filter penetration for both N95 models increased with increasing amounts of DOP loading and exceeded NIOSH allowed maximum penetration (5%) by both DOP and NaCl aerosols indicating filter degradation. Monodisperse NaCl aerosols (20-400 nm) gradually increased the penetration and shifted the most penetrating particle size from ~40 nm to larger sizes. In the case of P100 FFRs, DOP aerosol penetration was below 0.03% for up to 200 mg DOP loading as required for NIOSH certification. Interestingly, one of the two P100 FFR models loaded with 10-50 mg DOP showed >0.03% penetration with polydisperse, as well as monodisperse, NaCl aerosol testing. Overall, the results obtained in the study indicate that some P100 models loaded with DOP at lower amounts may show higher penetration with other aerosols such as NaCl. Further studies are needed to better understand the filter degradation of DOP loaded respirators.

INTRODUCTION

The National Institute for Occupational Safety and Health (NIOSH) recommends the use of particulate respirators for protection against airborne inert and biological particles in many workplaces. NIOSH approves three categories, N (not resistant to oil), R (resistant to oil), and P (oil proof) of particulate filters based on resistance to filter efficiency degradation by dioctyl phthalate (DOP) [1]. Each category of filter is certified at three levels of filter efficiency 95%, 99%, and 99.97%, designated as class 95, class 99 and class 100, respectively. For example, a filter marked N100 would mean an N-series filter that is at least 99.97% efficient. The filtration efficiency of respirators is measured using a more challenging test method with charge neutralized aerosol particles at 85 L/min flow rate. To measure the filter efficiency degradation, filtering facepiece respirators (FFRs) are loaded with test aerosols, up to 200 mg, with a simultaneous filter penetration measurement. The maximum penetration must not exceed 5%, 1% and 0.03% for class 95, 99 and 100 FFRs, respectively, during the entire loading period of 200 mg test aerosols [1]. For certification of R- and P- designated respirators, polydisperse DOP aerosol with a count median diameter (CMD) of 185 ± 20 nm and a geometric standard deviation (GSD) of less than 1.60 is used. DOP aerosol is known to degrade N-series respirators and increase filter penetration. For this reason, N-series respirators are tested using polydisperse NaCl aerosols with a CMD of 75 ± 20 nm and a GSD of less than 1.86.

Filter media employed in particulate filtering facepiece respirators (FFRs) capture particles efficiently by a combination of electrostatic, diffusion, interception, impaction and other mechanisms with a relatively reduced level of breathing resistance. N-series respirator filter media contain electrostatic charge and show a most penetrating particle size (MPPS) in the ~50 nm size range [2-4]. The electrostatic charge in filter media shifts the MPPS to <100 nm size range from the >100 nm range for mechanical filters [5]. Many R- and P-series FFR models available on the market contain filter media with electrostatic charge as shown by their MPPS in the ~50 nm size range [3, 6, 7].

Exposure to certain aerosols has been shown to degrade the filtration efficiency of electrostatic filters [8-11]. One study evaluated the filtration performance of charged filter media using solid NaCl and liquid dioctyl sebacate (DOS) aerosol loading [8]. With NaCl aerosol loading, filter efficiency showed an initial decrease followed by a rapid increase. The initial decrease in efficiency was attributed to neutralization of the filter charge while the subsequent increase in efficiency was attributed to additional mechanical capture mechanism of the particle deposits. With DOS loading, a continual decrease in efficiency was obtained with no pressure change. Scanning electron microscopy studies showed a uniform coating of DOS over fiber surface indicating charge neutralization [8]. Further studies with an electret filter loaded with charged as well as uncharged, liquid bis-ethylhexyl sebacate (BES) aerosols showed similar increase in filter penetration [9]. Their results indicated that electrostatic charge degradation was due to mechanisms other than direct neutralization. Another study showed significant effect of aerosol size, charge and composition on the degradation of electrostatically active filter material [10]. Their results were consistent with a charge screening process suggested previously [11]. The influence of various factors, including particle hygroscopicity and air humidity [12], surfactant treatment [13] and cake formation [14], on filter performance degradation has been reported.

Liquid organic solvents including isopropanol (IPA), xylene and toluene have been known to decrease the filtration performance of electrostatic filter media and respirators [3, 7, 15, 16]. Charge depleted filters showed higher filter penetration for different size particles with a shift in the MPPS toward larger sizes. Treatment with organic solvents has been shown to decrease or remove charge on electrostatic filter materials [17, 18]. Recent studies, using an electron force microscopy technique [19] confirmed that liquid isopropanol immersion removed the charge from electrostatic filter media.

To study filter efficiency degradation, many investigators loaded the filters with solid or liquid aerosols and measured the filter penetration simultaneously. However, filters loaded with one type of aerosol have not been challenged with a different aerosol to assess their filtration efficiency degradation. The goal of this study was to load two types of electrostatic filters, N95 and P100 FFRs with different masses of DOP and assess filter degradation by measuring penetration with liquid DOP aerosol, as well as with solid NaCl aerosol. N95 FFRs were included in the study for comparison purpose, although they are not tested with DOP for NIOSH certification.

MATERIALS AND METHODS

Test Respirators

Two commercially available NIOSH-approved N95 FFR models and two P100 FFR models were selected randomly from among those models tested previously in our laboratory. None of the FFR models tested in the study had exhalation valves.

DOP Loading of FFRs

FFRs were loaded with DOP using a TSI 8130 Automated Filter Tester (TSI, Inc. Shoreview, MN) similar to the method employed for NIOSH certification (NIOSH, 2007). First, the concentration of DOP aerosol produced by the TSI 8130 was determined using a Gelman 102 mm filter media (Type A/E glass filter media, Pall Corp., Ann Arbor, MI). The filter media was placed in between two Plexiglas (20 cm x 20 cm x 5 cm) plates with a hole (5 cm diameter) in the center. The Plexiglas plates with the filter in the center were positioned between the two filter chucks of the TSI 8130 and loaded with DOP for 40 min at 30 L/min flow rate. The amount of DOP (mg) loaded on the filter was divided by 1.2 to obtain the DOP aerosol concentration (mg/m^3). From the DOP concentration, the time required to load a known amount of DOP at 85 L/min can be calculated by the following formula.

$$T \text{ (min)} = (\text{mg load}).(1000 \text{ L/m}^3)/(C).(Q)$$

where, C is the concentration of DOP aerosol (mg/m^3) and Q is 85 L/min. The time (T in min) required to load respirators with 10, 30, 50, 100 and 200 mg DOP was calculated. This was verified by loading FFRs with different amounts of DOP at 85 L/min. The amount of DOP loading plotted against time showed a linear relationship. DOP loading of FFRs for different experiments was conducted at 85 L/min flow rate.

Polydisperse NaCl and DOP Aerosol Penetration Test

The effect of DOP loading on filter penetration of N95 and P100 FFRs was tested by the following methods:

1. Four FFR samples from each model were loaded with 200 mg DOP and penetration was measured simultaneously each minute during the entire period of loading. From the time required to load 10, 30, 50, 100 and 200 mg DOP, the penetration for each loading amount was obtained.
2. Four samples from each FFR model were loaded with DOP at 10, 30, 50, 100 and 200 mg amounts. Each FFR sample was challenged with polydisperse NaCl aerosol and penetration was measured for 1 min as described previously [4].

Monodisperse NaCl aerosol Test

A second set of FFRs (three samples from each model) loaded with DOP (10, 30, 50, 100 and 200 mg) were tested against monodisperse NaCl particles using a 3160 Fractional Efficiency Tester (TSI 3160) as described previously [4]. Initial penetration for ten different monodisperse aerosols (centered at 20, 30, 40, 50, 60, 80, 100, 200, 300 and 400 nm) was measured at 85 L/min flow rate.

Filter Media Composition

Hydrophilic and hydrophobic filter media composition for both N95 and P100 FFR models was analyzed. Two coupons (3 x 3 cm^2) from each respirator model were cut with a scissors. Filter media layers from each FFR were physically separated with a forceps. To determine the hydrophilicity or hydrophobicity, a 10-µl droplet of water was placed on each filter media layer and visually examined for 5 min. The deposited water droplet spread over and wet the hydrophilic filter media layer, while it remained as a droplet in the case of hydrophobic media.

RESULTS AND DISCUSSION

The two N95 model FFRs loaded with increasing mass of DOP (10 to 200 mg) showed increasing aerosol penetration (*Figure 1*, N95-A and N95-B, open bars) when measured simultaneously. DOP aerosol penetration exceeded NIOSH allowed maximum penetration (5%) at 100 and 200 mg DOP loads. DOP loaded FFRs also showed a similar trend in the filtration performance degradation when tested with polydisperse NaCl aerosol (*Figure 1*, N95-A and N95-B, hatched bars). DOP aerosol penetration was higher than NaCl aerosol penetration at all loads. The increase in penetration with increasing DOP load indicates filter degradation. The result obtained in the study is similar to the filter degradation of electret charged filter media loaded with liquid aerosols [8, 9, 20]. The above studies reported different mechanisms of interaction of liquid aerosols with the electrostatic charge of the filter media. Results from one study indicated that liquid DOS aerosol degrades electrostatic filter by charge neutralization [8]. In another study, electrostatic filter treated with charged as well as neutralized liquid BES showed similar increases in penetration [9]. Based on the results, these authors suggested that electrostatic filter degradation by liquid aerosols was not due to direct neutralization of the charge on the fibers by the net charge of test particles. Oil aerosol was believed to discharge the fibers by ionic conduction through an oil film on the fibers.

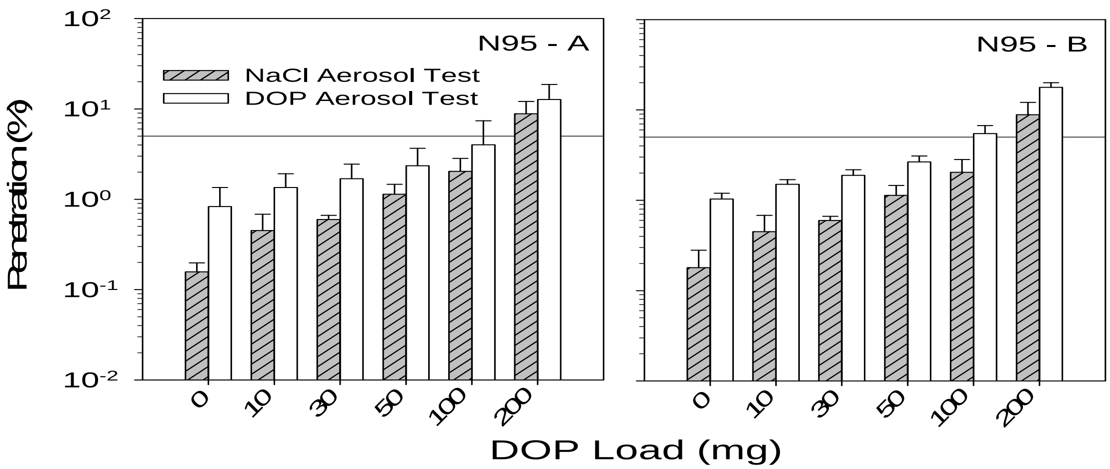

FIGURE 1: Penetration of polydisperse aerosol through two N95 FFR models loaded with different amounts of DOP. Penetration obtained using NaCl (shaded bars) and DOP (open bars) test aerosol for N95-A (left) and N95-B (right) at 85 L/min using a TSI 8130. The solid horizontal line indicates maximum allowed penetration (5%) for N95 respirators. Error bars represent 95% confidence interval.

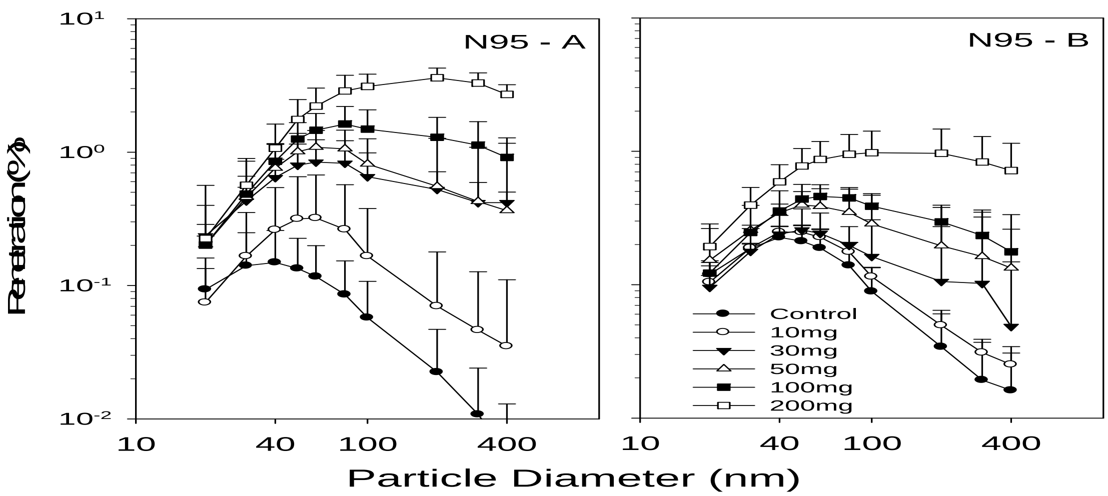

FIGURE 2: Penetration curves for different size monodisperse NaCl aerosols for two N95 FFR models (N95-A, left and N95-B, right). Penetration for FFRs loaded with increasing amounts of DOP (0-200 mg) (symbols) were measured at 85 L/min using a TSI 3160. Error bars represent 95% confidence interval.

DOP loaded N95 FFRs also showed a dose-dependent increase in monodisperse NaCl aerosol penetration for different size particles (*Figure 2*, N95-A and N95-B). The MPPS was ~40 nm for both N95 FFR models, which gradually shifted to larger particle sizes with increasing DOP loading. The increase in filter penetration and the shift in the MPPS to larger particle sizes with increasing DOP load showed filter degradation of N95 FFRs. The effect of DOP on the MPPS and penetration was similar to those obtained for N95 FFRs exposed to organic solvents [7, 16, 21].

In the case of P100 FFRs loaded with DOP up to 200 mg, filter penetration measured simultaneously was within NIOSH allowed maximum penetration level (0.03%) (*Figure 3*, P100-A and P100-B, open bars). Penetration was approximately similar at 10 to 200 mg DOP loads for each P100 model. The results agree with the flatter penetration obtained for filter media loaded with increasing amounts of DOP [22]. Electrostatic P-type filter media resistant to DOP degradation were designed to meet NIOSH certification test criteria. Penetration results for P-type filter media indicate that the manufacturing process somehow shielded the charge against DOP interaction.

P100 FFRs loaded with different DOP loads when tested with NaCl aerosol, showed higher penetration than DOP aerosol penetration, with some exceptions. NaCl aerosol penetration for P100-A FFRs was

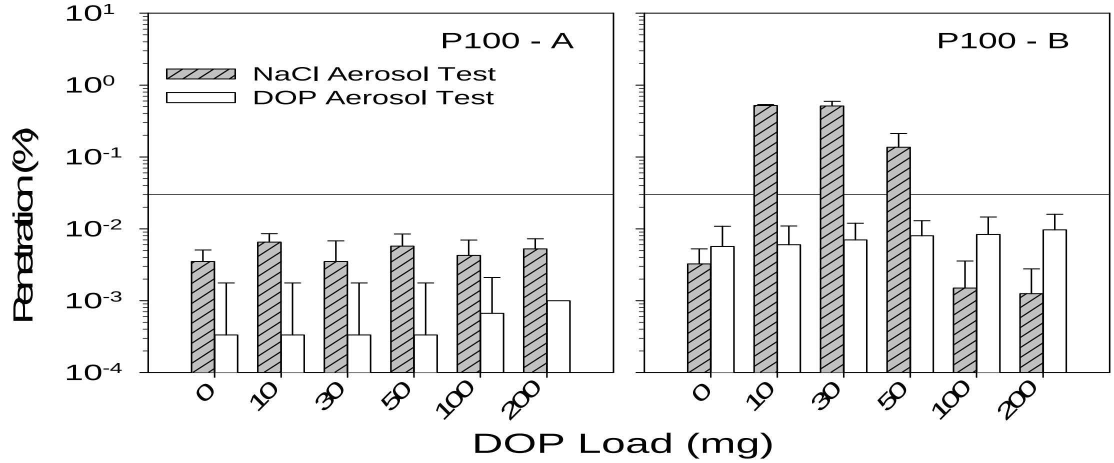

FIGURE 3: Penetration of polydisperse aerosol through two P100 FFR models loaded with different amounts of DOP. Penetration obtained using NaCl (shaded bars) and DOP (open bars) test aerosol for P100-A (left) and P100-B (right). The solid horizontal line indicates maximum allowed penetration (0.03%) for P100 respirators. Error bars represent 95% confidence interval.

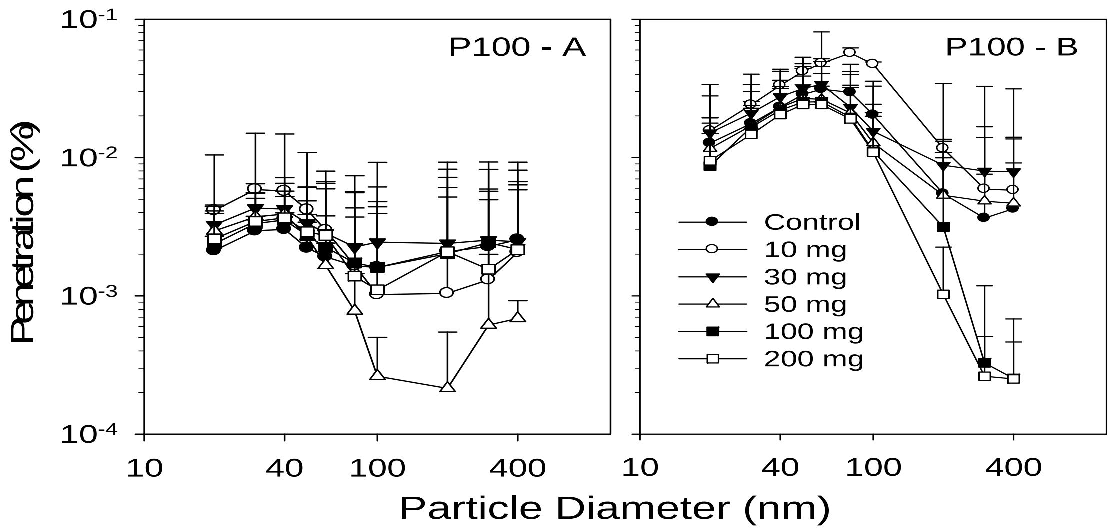

FIGURE 4: Penetration curves for different size monodisperse NaCl aerosols for two P100 FFR models (P100-A, left and P100-B, right). Penetration for FFRs loaded with increasing amounts of DOP (0-200 mg) (symbols) were measured at 85 L/min using a TSI 3160. Error bars represent 95% confidence interval.

higher than the DOP aerosol penetration at different DOP loads (*Figure 3*, P100-A, hatched bar). P100-B (*Figure 3*, hatched bar) showed a dramatic increase in NaCl aerosol penetration at low DOP loads (10-50 mg) but not at higher loads. The higher penetration may be explained by the deposition of NaCl aerosol on the electrostatic charge of the filter media. NaCl aerosol loading of an electrostatic filter media initially decreases filter efficiency, followed by an increase [8]. The initial decrease in filter efficiency has been attributed to the neutralization of the filter media charge by the charged NaCl particles depositing on the filter. Similar finding has been reported for TiO_2 aerosol loading of an electrically active fibrous filter [10]. At higher DOP loads, NaCl showed no degrading effect on filtration performance. This may be because DOP, at higher loads may form a heavy coat on the filter media and prevent NaCl interaction with the charge.

Figure 4 shows the penetration curves for different size monodisperse NaCl aerosols for P100-A and P100-B loaded with different amounts of DOP. Penetrations for different size particles were higher than those for the control P100-A FFRs. The MPPS remained at ~40 nm for the DOP loaded N95-A FFRs. Penetration for P100-B FFRs loaded with 10 and 30 mg DOP showed higher penetration than the control. At 10 mg DOP load, a shift in the MPPS from 60 nm to 80 nm was obtained. The increase in penetration, however, was not remarkable compared to that obtained with the polydisperse aerosol test. This may partly be explained by the differences in the FFRs tested at different times. Another point is the sensitivity of the two aerosol penetration tests employed in the study. The monodisperse aerosol test based on particle number is more accurate than the polydisperse aerosol test, which depends on the mass of the particles [6]. Monodisperse NaCl aerosol penetration results at 10-30 mg DOP load indicate filter degradation.

Although, different P100 FFR models certified by NIOSH meet the test criteria, their penetration property may be different because of some variations in the manufacturing process. The difference among the two P100 models tested in the study is also supported by the variation in the filter media composition (*Table I*). In general, N95 models had more hydrophilic media layers than P100 models. P100-A had hydrophobic outer and middle layers with a hydrophilic inner layer. All three layers of P100-B model were hydrophobic with an outer mesh support. The results indicate that NaCl interaction with DOP loaded P100 FFRs may be influenced by factors including filter media polarity (hydrophobic and hydrophilic) and composition.

TABLE I. Hydrophobic and hydrophilic filter media composition of N95 and P100 FFRs.

Model	Outer Layer	Middle Layer	Inner Layer
N95-A	Hydrophobic	Hydrophilic Hydrophobic	Hydrophilic
N95-B	Hydrophilic	Hydrophobic	Hydrophilic
P100-A	Hydrophobic	Hydrophobic	Hydrophilic
P100-B	Hydrophobic	Hydrophobic	Hydrophobic

DOP loading showed varying filter degradation effects among the electrostatic N95 and P100 FFRs. With increasing DOP load, filter penetration gradually increased for N95 models with relatively no change for P100 models. The difference in the penetration with DOP loading of N95 and P100 FFRs confirms earlier findings [23]. In that study filter penetration was measured during DOP loading of several electrostatic and one mechanical filter media [23]. The mechanical filter media showed the lowest rate of filter degradation throughout the loading up to 5 mg/cm^2. Among the electrostatic filters, filter degradation occurred in two regimes, with an initial fast period followed by a slow second phase. Some filters were degraded rapidly because DOP liquid masked the electrostatic charge and showed increased penetration with DOP loading as shown for N95 FFRs. The other group of electrostatic filters behaved like mechanical filters, with a single slow degradation regime resembling the slow degradation of P100 respirators showing the difference in the filter degradation among respirators containing electrostatic filter media. P100 respirators also showed relatively high resistance to degradation by organic solvents [7].

Limitations of the study include that only two models of N95 and P100 FFRs were used for DOP loading and NaCl aerosol penetration. Additional models need to be tested to confirm the results obtained in the study. Variations between polydisperse and monodisperse NaCl aerosol penetrations for the FFR models were observed. The variation may be due to the use of FFRs obtained from the vendors at different times. This problem can be reduced by conducting the different set of experiments using FFRs from a single batch. One other point is that degradation of electrostatic FFRs in the study has not been correlated with the charge density of the filter media. Quantification of the filter charge density at different amounts of DOP loading and NaCl testing may unravel the mechanism of filter degradation of the N95 and P100 respirators. Nonetheless, the filter degradation of DOP loaded P100 FFRs tested with NaCl aerosol may indicate that non-oil aerosols could influence DOP resistance to filter degradation. This may have important implication on the filtration performance of some P100 model FFRs exposed to both oil and non-oil aerosols in workplaces. Further research on the filter degradation of P100 FFRs loaded with DOP is needed to address respiratory protection in workplaces.

CONCLUSIONS

Polydisperse DOP and NaCl aerosol penetration for both N95 model respirators increased with increasing amounts of DOP loading and reached higher than 5% with >100 mg loading. With increasing amounts of DOP loading, monodisperse NaCl aerosol penetration for both N95 model FFRs increased, with a shift in the MPPS from 40 nm to larger particle sizes. The results showed filter performance degradation of N95 respirators by DOP loading. In the case of P100 models, DOP loading up to 200 mg did not increase DOP aerosol penetration, indicating no filter degradation. Interestingly, one of the two P100 FFR models (P100-B) loaded with 10-50 mg DOP showed >0.03% penetration with polydisperse as well as monodisperse NaCl aerosol testing, with some exceptions. Data obtained in the study indicate that some P100 models loaded with lower amounts of DOP may show higher filter penetration when exposed to other aerosols such as NaCl. The results suggest that further studies are needed to better understand filter degradation of DOP loaded filters.

ACKNOWLEDGEMENTS

The authors acknowledge NIOSH colleagues including Jay Snyder, Colleen Miller and Mike Bergman for their useful suggestions and critical review of the manuscript. This research work was supported by NIOSH funding.

DISCLAIMER

Mention of commercial product or trade name does not constitute endorsement by the National Institute of Occupational Safety and Health. The findings and conclusions of this report are those of the author(s) and do not necessarily represent the views of the National Institute for Occupational Safety and Health.

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