VOC Outgassing from Baked and Unbaked Ventilation Filters

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ABSTRACT

Volatile organic compounds (VOC) outgassing from newly manufactured baked and unbaked ventilation filters were examined using gas chromatography/mass spectrometry following thermodesorption (TD-GC/MS) technique. Filters were tested under both static and dynamic conditions. Under static conditions, the unbaked filter outgassed VOC (0.50 ± 0.18 μg) more than twice as much as the baked filter (0.23 ± 0.15 μg). 2-Propanol and nonanal were the components outgassed from both baked and unbaked filters and 4-piperidinol, 2,2,6,6-tetramethyl- was determined to be the main contaminant outgassed from the unbaked filters. Series of tunnel experiments were conducted by passing highly purified dry air through the filters and taking air samples upstream and downstream the filter. Under dynamic test conditions, the baked filter was not significantly distinguishable from the background with a downstream total VOC concentration increase only by 35.3%, which was almost 9 times lower than that of the unbaked filter (294%). 3-hydroxy-4,6-di-t-butyl-2H-pyran-2-one was the main VOC outgassed from the unbaked filter. In addition, a general trend of decreasing VOC outgassing with time from baked and unbaked filters was observed in both static and dynamic tests. It also suggested that baked filter behaved like a sink for certain VOC and unbaked filters from different batches may outgas different VOC. Overall, bake-out was proved to be an effective way of reducing VOC outgassing from ventilation filters.

Keywords: VOC; Outgassing; Baked ventilation filters.

INTRODUCTION

Filters are widely used for removal airborne particulate contaminates. Meanwhile, however, ventilation filters may outgas volatile organic compounds (VOC), which have adverse effects under specific applications. For instance, supply air filters have been identified as a major pollution source in heating, ventilation, and air conditioning (HVAC) systems, giving an adverse impact on perceived indoor air quality (Pejterson *et al*., 1989; Clausen, 2004). Later studies showed that the indoor air pollution emitted from used ventilation filters has negative impact on health, comfort, and occupant performance. Sick building syndrome symptoms such as nose irritation, perceived dryness, headache and dizziness have been reported (Clausen *et al*., 2002) after long time exposures of VOC contaminated air. The degraded indoor air quality may also significantly decrease office work productivity and the performance of call-center operators (Wargocki *et al*., 2004; Wyon, 2004). Laboratory and field tests have demonstrated that microbial provenance and growth on air-conditioner filters might be a

possible reason for VOC emission from filters (Simmons *et al*., 1995; Schleibinger *et al*., 1999). Other studies (Weschler, 1996; Hyttinen *et al*., 2003a) suggested that ozone reactions on ventilation filters contribute to odor production. Recent studies showed that odor released by ventilation filters was due to VOC originating in particles and dust layers collected onto the filters (Pejtersen, 1996; Hyttinen *et al*., 2001, 2002, 2003b, 2007). Moreover, in order to improve the air quality and to reduce the risk of airborne disease transmission in indoor environments, research on and practical use of personal ventilation (PV) have been gaining popularity in recent years (Melikov *et al*., 2007). Hence, filters are used in various PV systems and VOC outgassing from such filters becomes a significant health concern due to the proximity between them and the occupants. VOC outgassing from filters can also be a source of airborne molecular contaminants (AMC), which is a serious concern in clean room. It has been documented that organic contaminants adsorbed on a silicon wafer surface can cause various detrimental effects (Saga and Hattori, 1997; Ogata *et al*., 1998; Liu *et al*., 2002).

Although abundant research has been conducted to evaluate VOC outgassing from filters, most of them focused on used ventilation filters since early work concluded that the pollution contribution of a new filter to the indoor air was small while used filters may contribute significantly to pollute indoor air (Bluyssen, 1993;

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Pejetersen, 1996) and the VOC outgassed from the filter itself should be very low (Schleibinger and Ruden, 1999). Therefore, data are still scarce for VOC outgassing from new ventilation filters. Furthermore, bake-out, a term referring to the act of using heat to accelerate the process of VOC outgassing, has been proved to be effective in reducing building indoor VOC levels (Girman, 1989; Park *et al.*, 2007). We thus hypothesize to use bake-out as one way to control VOC outgassing from ventilation filters.

The purpose of this study was to identify and quantify VOC outgassed from newly manufactured ventilation filters under both static and dynamic conditions. In addition, the effect of bake-out on filter VOC outgassing was investigated.

METHODS

Test Filters and Experimental Plan

Three newly manufactured pleated High Efficiency Particulate Air (HEPA) filters, one baked and two unbaked, of the same size 25.4 cm × 17.8 cm × 3.18 cm were used for the VOC outgassing test. The baking condition for the filter was set to be at 121°C for 24 hours. The tests were performed under two conditions, namely, the static condition and the dynamic condition. For the static test, filters were kept in a chamber (without air flow passing by) while for the dynamic test, filters were installed in a tunnel and were blown using highly purified dry air. The baked filter was tested under both static and dynamic conditions. However, for the two unbaked filters, one was used in static test while the other was used in dynamic test. For both tests, air samples were first collected by adsorption tubes and then analyzed by gas chromatography/mass spectrometry (GC/MS) to characterize the VOC outgassed from the filters. Before and after testing, the filters were stored in their original cardboard boxes under room temperature.

Static and Dynamic Tests

For the static test, the unbaked filter was incubated in a front opening-unified pod (FOUP) (Entegris, MN) for 3 days with the temperature held at 22 ± 2°C. A FOUP is an enclosure designed specially for transporting and storing 300 mm wafers in clean rooms. Since the wafer storage environment requires a low VOC concentration, using a FOUP ensures a relatively low VOC contamination from the test environment (background). To collect air samples, a freshly conditioned adsorption tube (Tenax TA stainless

steel prepacked sample tubes, product No. N9307005, PerkinElmer Instruments, MA) was carefully put above the filter with a sampling time of 3 days. The baked filter was tested in the same manner to determine the effect of baking on filter VOC outgassing. Furthermore, to monitor possible VOC interference from the background, the empty FOUP (without filters) was examined in the same way. After each test, compressed nitrogen was used to purge the FOUP at a flow rate of 25 LPM for 20 min to minimize the VOC residues inside the FOUP. To verify the results, the described static test was repeated five times for the background, the baked filter, and the unbaked filter.

For the dynamic test, the unbaked or baked filter was installed in a stainless steel tunnel. (Fig. 1) To minimize the introduction of VOC contaminants from the background, the inner surface of the test tunnel was first scrubbed using isopropanol and then rinsed with water. Building compressed air was dried and purified by a clean air generator (Breathing air system Ultrapure ALG 35 S–375 S, Donaldson, GA). The clean dry air was then blown through the tunnel to the test filter at a flow rate of 150 LPM, measured by a mass flow meter (Model 4043, TSI, MN). After measuring the air flow rate, the mass flow meter was removed from the test line to avoid possible introduction of VOC contamination. The tests were conducted at a flow temperature of 23°C ± 2°C and relative humidity of ~0.1%, measured by an indoor air quality monitor (Q-Trak Model 7565, TSI, MN). Two gaskets made of red silicone rubber were used to seal the filter to the filter holder frame. Air samples were collected simultaneously upstream and downstream of the filter using Tenax TA adsorption tubes at a rate of 0.2 LPM with a sampling time of 3 hours. Moreover, to evaluate VOC contamination from the test background (e.g. filter gaskets), the same test was conducted without installing the filter. The dynamic test was repeated once for the background, the unbaked and baked filter.

Analysis of Air Samples by Gas Chromatography/Mass Spectrometry

The VOC were desorbed from the Tenax TA tubes in a thermal desorption system (ATD 400, PerkinElmer, MA), separated by gas chromatography (HP 6890, Agilent, CA), and directed to a mass selective detector (MSD 5973, Agilent, CA). Helium was used as the carrier gas with a flow rate of 1 mL/min. J&W Scientific HP-5MS column (0.25 μm film thickness, 30 m × 0.25 mm I.D.) was used
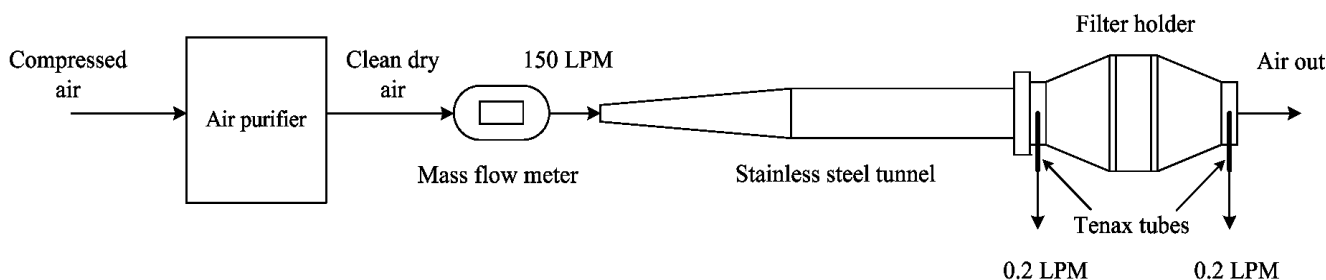

Fig. 1. Experimental setup for the dynamic test of VOC outgassing from unbaked and baked filters.

and the oven temperature was programmed as 35 to 300°C at 15°C/min and kept at 300°C for 7.5 min. Samples were injected in the splitless mode at 250°C (inlet temperature) and analyzed in the scan mode with scanning range from 38 to 550 m/z. Mass spectrum of each peak in the GC chromatogram was searched through and compared with the mass spectral library (Wiley275) by the ChemStation software and the compound with the highest quality match was reported as the detected component. The total mass or concentration of the VOC was measured as the sum of the peak areas and compared to the area responses of known amount of heptane.

RESULTS AND DISCUSSION

Filter VOC Evaluation under Static Condition

Fig. 2 shows a comparison of total VOC mass detected from the background, the baked filter and the unbaked filter under static test conditions with corresponding values of 0.34 ± 0.13 µg, 0.23 ± 0.15 µg, and 0.50 ± 0.18 µg, respectively. The unbaked filter gave a mean VOC mass more than twice as high as that of the baked filter, suggesting that bake-out is an effective way to reduce VOC outgassing from filters. Surprisingly the total VOC mass detected for the baked filter was even lower than that of the background. Possible explanation could be that the background (FOUP) had a higher VOC outgassing than the baked filter and/or the baked filter behaved like a VOC sink in the test. Also note that the static test results showed considerable variation, which is indicated by the large standard deviation. This may be due to the random errors of repeated measurements. Previous study (Hyttinen, *et al*., 2007) using thermodesorption and GC/MS techniques also showed a variation as large as 38% of the measured total VOC emission from filters. The large standard deviation more likely results from the general nature that VOC decreases with time. To make it clear, we plotted the total VOC mass detected from the background, the baked filter, and the unbaked filter as a function of time in Fig. 3. The maximum VOC production appeared within the first two weeks. The highest VOC mass detected was 0.50 µg, 0.48 µg, and 0.77 µg for the background, the baked filter, and the unbaked filter, respectively. A general trend of decreasing VOC outgassing with time from background and both filters was observed.

Table 1 shows the mean and standard deviation of each volatile component found by GC/MS under static test conditions. In total 9 volatile organic compounds were detected. As discussed before, the large standard deviation is probably due to the decreasing VOC outgassing with time and random errors of repeated measurements. Decane and dodecane (0.167 µg and 0.129 µg) were the two main components found from the background. All the components (except octane) found from the background also appeared in both the baked and unbaked filter tests. Decane (0.106 µg) was the main components found in the baked filter test. In all the measurements, two components, 2-propanol and nonanal were only observed in the baked and unbaked filter tests, but not from the background. Thus, they may be regarded as the VOC outgassed from the both filters. In addition, another two components, 4-piperidinol, 2,2,6,6-tetramethyl- and octamethyl cyclotetrasiloxane were only found in the unbaked filter tests. Since 4-piperidinol,2,2,6,6-tetramethyl- was found neither from the background nor in the baked filter tests, and it was the main component (0.130 µg) detected in the unbaked filter test, therefore, we believe that 4-piperidinol 2,2,6,6-tetramethyl- was the VOC outgassed from the unbaked filter. However, for octamethyl cyclotetrasiloxane, though not detected in either background or baked filter tests, we cannot claim it as VOC outgassed from the unbaked filter, since cyclotrisiloxane's are often attributed to the bleeding of column (Lestremau *et al*., 2004)
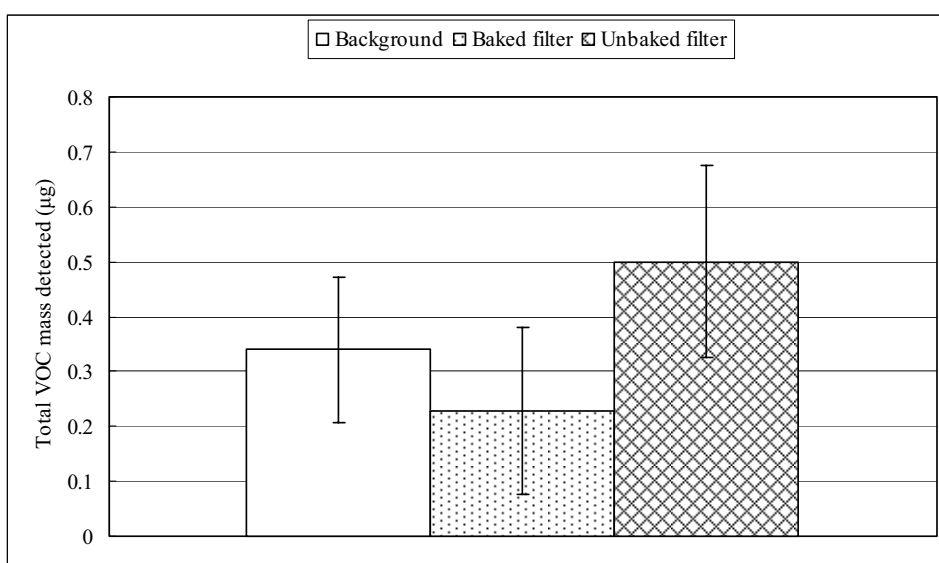

Fig. 2. Total VOC mass measured in the incubation chamber from background (empty column), baked filter (dotted column), and unbaked filter (meshed column). Each bar represents the mean ± one standard deviation of total VOC mass detected for n = 5 measurements.
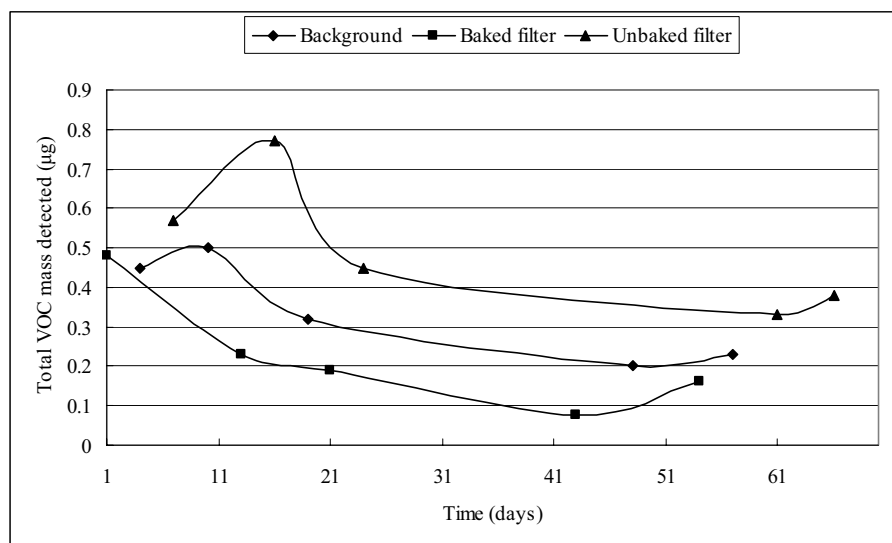
Fig. 3. The time history of total VOC mass detected in the incubation chamber from the background, the baked filter, and the unbaked filter.

Table 1. Mean volatile components mass and its standard deviation (μg, quantified as heptane) for n = 5 measurements under static test conditions.

Compound	Background	Baked filter	Unbaked filter
		Mean ± Standard Deviation	
2-Propanol	nd	0.013 ± 0.028	0.064 ± 0.062
Octane	0.007 ± 0.016	0.014 ± 0.031	nd
Hexamethyl cyclotrisiloxane	0.070 ± 0.038	0.035 ± 0.036	0.071 ± 0.051
Octamethyl cyclotetrasiloxane	nd	nd	0.004 ± 0.004
Decane	0.167 ± 0.149	0.106 ± 0.110	0.080 ± 0.116
Nonanal	nd	0.011 ± 0.024	0.042 ± 0.027
4-Piperidinol, 2,2,6,6-tetramethyl-	nd	nd	0.130 ± 0.016
Dodecane	0.129 ± 0.035	0.030 ± 0.029	0.056 ± 0.032
Decanal	0.009 ± 0.020	0.019 ± 0.029	0.058 ± 0.037

nd = not detected

and/or artifacts of Tenax TA, especially considering its low amount (0.004 μg) compared with other components. After baking, the mean outgassing values of 4-piperidinol,2,2,6,6-tetramethyl- was reduced to a level below its detection limit. Meanwhile, the mean outgassing masses of another two volatiles that were only detected from filters, 2-propanol and nonanal, were reduced by 80% and 74%, respectively. All these facts lead us to the conclusion that the effect of baking on reducing filter VOC outgassing under static conditions was significant. Note also that for some organic components (e.g. dodecane for the baked filter and decane for the unbaked filter), the mean mass detected in the filter tests were much lower than that from the background, suggesting that the filters might behave like sinks for certain VOC under static conditions.

Filter VOC Outgassing under Dynamic Condition

Fig. 4 presents the total VOC concentration measured upstream and downstream of the filter holder, the baked filter, and the unbaked filter under dynamic test conditions. As expected and was observed in all the experiments, the downstream total VOC concentration was higher than the upstream. Filtration increased the total VOC concentration (Hyttinen, 2007). For the baked filter tests, the total VOC concentration changed from 4.31 ± 1.38 μg/m³ (upstream) to 5.83 ± 1.96 μg/m³ (downstream), with an increase by 35.3%; for the unbaked filter tests, the total VOC concentration increased from 4.58 ± 2.55 μg/m³ (upstream) to 18.05 ± 5.10 μg/m³ (downstream), with an increase by 294%. The mean VOC concentration downstream of the unbaked filter was almost 9 times as high as that of the baked filter. Therefore, it was clear that bake-out helped to reduce filter VOC outgassing significantly under dynamic conditions. Note that for the background, the total VOC concentration detected upstream and downstream of the filter holder was 3.89 ± 0.20 μg/m³ and 4.44 ± 0.20 μg/m³, respectively, with a mean VOC concentration increase of less than 15%. This suggested that the background (e.g. the wall of the tunnel and the filter gasket) did not introduce too much VOC noise during the tests. Note also that the mean upstream total VOC concentration in the background test, the baked filter test, and the unbaked filter test was similar, indicating that by
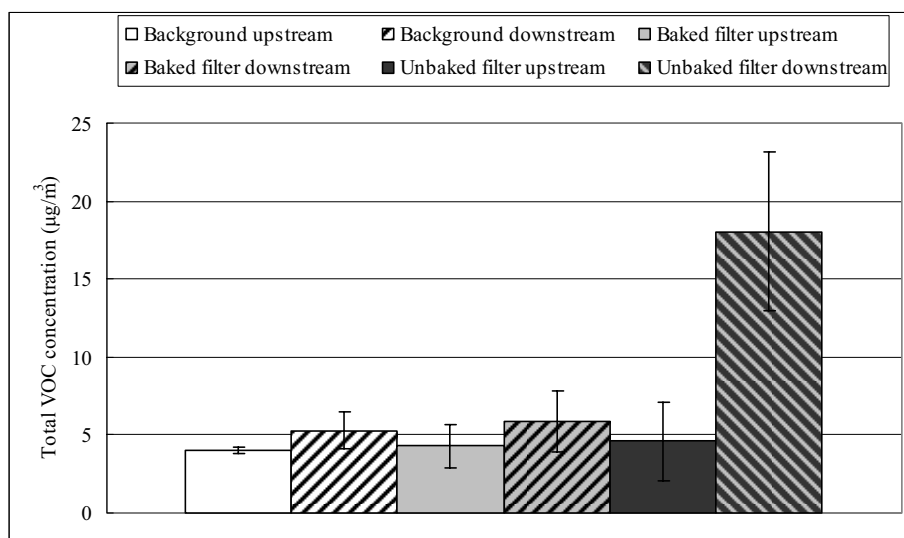
Fig. 4. VOC concentration detected upstream and downstream of the filter holder from the background, the baked filter, and the unbaked filter. Each bar represents the mean ± one standard deviation of total VOC concentration detected for n = 2 measurements.

using pre-cleaned stainless steel tunnel and highly purified dry air, we were able to maintain a relatively stable background for the filter VOC outgassing tests.

The concentration range of each volatile component detected by GC/MS outgassing from the background, the baked filter and the unbaked filter are presented in Table 2. In total 4 components were detected from the background, which also appeared in the baked filter and unbaked filter tests as expected. For the background tests (the left part of Table 2), hexamethyl cyclotrisiloxane and decanal were the two main components detected. Nonanal was only found downstream of the filter holder. The hexamethyl cyclotrisiloxane concentration before the filter holder ranged from 2.55 up to 2.70 μg/m^3, after the filter holder from 2.56 up to 3.07 μg/m^3. The mean ratio (concentration after the filter holder/concentration before the filter holder) is 1.07, indicating that overall the hexamethyl cyclotrisiloxane concentration slightly increased after the filter holder. The mean ratio of octamethyl cyclotetrasiloxane is 1.05, which also indicates a slight increase after the filter holder. However, the decanal concentration downstream the filter holder is more than 50% higher than the upstream (mean ratio = 1.51), which was probably due to the filter gasket outgassing. This large increase of decanal concentration was also confirmed in the baked and unbaked filter tests, but with even larger mean ratios of 1.90 and 2.53, respectively.

For the baked filter tests (the middle part of Table 2), hexamethyl cyclotrisiloxane and decanal were again the two main components detected. In addition, nonanal was again only found in the downstream of the baked filter, but with a higher concentration than that of the background. Therefore, under dynamic conditions, there was no significant difference between the VOC outgassing from the baked filter and that from the background in terms of either VOC components or total VOC mass (see Fig. 4). For the unbaked filter tests (the right part of Table 2), 3-hydroxy-4,6-di-t-butyl-2H-pyran-2-one was found to be

the main component. Since its mean concentration (10.04 μg/m^3) was much higher than other components and it was not detected in either background or baked filter, we believe that 3-hydroxy-4,6-di-t-butyl-2H-pyran-2-one was the VOC outgassed from the unbaked filter. As seen in Table 2, bake-out can remove certain VOC below detection limit and thus reduce the total VOC outgassing from filters significantly. Moreover, note that all mean ratios of the baked filter were smaller than that of the unbaked, e.g. the mean ratio of decanal was reduce from 2.53 to 1.90, suggesting that bake-out can also reduce the outgassing ability of certain VOC. Note that the mean ratio of hexamethyl cyclotrisiloxane and octamethyl cyclotetrasiloxane for the baked filter were less than unit (0.98 and 0.70, respectively), suggesting that the baked filter was a sink for these two components. Previous studies also found similar behavior for ventilation filters. Schleibinger (1999) reported that the prefilter in a convention center was a sink for acetaldehyde and Hyttinen (2007) found that nonanal mean concentration upstream of the filters was exceptionally higher than downstream when the humidifier was on.

For the baked filter, some consistency is observed when we compare the static test results (Table 1) with the dynamic test results (Table 2) of the baked filter. First, the baked filter behaved like a sink of hexmethyl cyclotriloxane in the dynamic test, which was also observed in the static test with a "baked filter/background" mean ratio of 0.50. Second, the static test showed that nonanal was the component outgassed from the baked filter and in the dynamic test, nonanal was only detected downstream of the baked filter. However, since nonanal was also detected downstream of the filter holder in the background test, we are not sure whether the baked filter or the background (e.g. filter holder) was the source of nonanal. In addition, 2-propanol, the component found to outgas from the baked filter in the static test, was not detected in the dynamic test,

Table 2. Volatile components concentration ranges (μg/m^3, quantified as heptane) and mean ratios (upstream concentration/ downstream concentration) for n = 2 measurements under dynamic test conditions. Bold type: main components found from both the background and the baked filter; Underlined type: volatile component outgassed from the unbaked filter.

	background			baked filter			unbaked filter		
	Up	Down	Mean ratio	Up	Down	Mean ratio	Up	Down	Mean ratio
Hexamethyl cyclotrisiloxane	2.55–2.70	2.56–3.07	1.07	2.40–3.25	2.55–3.01	0.98	2.78–2.89	2.94–3.42	1.12
Octamethyl cyclotetrasiloxane	nd–0.66	nd–0.69	1.05	nd–0.84	nd–0.59	0.70	nd	nd–1.01	na
Nonanal	nd	nd–1.09	na	nd	nd–1.51	na	nd–1.12	nd–2.68	2.40
Decanal	0.90–0.96	1.02–1.78	1.51	0.93–1.19	1.31–2.70	1.90	nd–2.39	1.92–4.12	2.53
<u>3-hydroxy-4,6-di-t-butyl-2H-pyran-2-one</u>	nd	nd	na	nd	nd	na	nd	9.58-10.5	na

Up = upstream; Down = downstream; nd = not detected; na = not applicable, since the upstream or downstream concentration was below the detection limit

suggesting that levels of certain VOC outgassed from filters may reduce over time. Similar phenomenon was also observed in the static tests (see Fig. 4).

Since two different unbaked filters were used in the static and dynamic tests, a comparison of unbaked filter results is impossible. However, we note that 4-piperidinol, 2,2,6,6-tetramethyl- was determined to be the main component from the unbaked filter in the static tests, while it was not found in the dynamic tests. Instead, a high concentration of 3-hydroxy-4,6-di-t-butyl-2H-pyran-2-one was detected. This indicated that unbaked filters manufactured from different batches could outgas different VOC. This behavior was verified in all the measurements, but no explanation for this has yet been found.

Filter VOC outgassing commonly occurs from filter binders (e.g. polyurethane), as well as the filtration media and/or chemical treatments of the filtration media (George *et al.*, 1996). For example, some glass fiber filters contain organic binder, up to 5% by weight, which interferes with the analysis of certain organic compounds (Hinds, 1999). In addition, TD-GC/MS analysis of various components of HEPA filters has showed that Fyrol PCF, a compound commonly used as a flame retardant, was present in the polyurethane potting material used to secure and seal the filter media (Lebens *et al.*, 1996). It has been proved that boron, a well known contamination on wafer surfaces, is a major component in the filter media of HEPA filters used in clean rooms (Stevie *et al.*, 1991). In our study, 4-piperidinol, 2,2,6,6-tetramethyl-, the main VOC outgassed from the unbaked filter, may originate from the filter binders since filter binders usually contain strong adhesives such as thermosetting epoxy resin and 4-piperidinol, 2,2,6,6-tetramethyl- is widely used as an epoxy resin cross-linking agent.

One of the limitations of the experiment is using a FOUP for the static tests. We tested the background VOC outgassing as soon as the FOUP was received (without any pre-cleaning like nitrogen purge) and found a total VOC mass of 0.95 μg (data not shown), which was even higher than the maximum VOC mass (0.77 μg) detected for the unbaked filter in all the five measurements. Actually, there are concerns about the presence of emitted VOC from a FOUP, which may lead to the degradation of the electrical

properties in circuits on wafers (Hu and Tsao, 2006). Therefore, the FOUP may not be a good incubation chamber for the filter VOC outgassing test. Instead, generally low outgassing materials like stainless steel or glass may be better. It should be noted that no standards were used when analyzing compounds outgassed from the background, the baked filter, and the unbaked filters and the mass and concentration of these VOC were quantified as heptane equivalents and thus were only estimates for the amounts of these compounds. Moreover, though Tenax TA is widely used for sampling VOC in ambient air, it can only be used for analytes such as aromatics except benzene, apolar components (boiling temperature > 100°C) and less volatile polar components (boiling temperature > 150°C) (EPA, 1997). To cover a wider range of VOC, other sorbents should be selected. For example, 2,4-dinitrophenylhydrazine (DNPH) cartridges with liquid chromatography are generally used to cover the aldehydes with low molecular weight.

CONCLUSIONS

Newly manufactured baked and unbaked ventilation filters were tested in both a FOUP and a stainless steel tunnel to investigate VOC outgassed from filters and the effect of baking on VOC reduction. The present study indicates that methods like baking, by removing certain VOC below detection limit and by decreasing the outgassing levels of certain VOC, was an effective way of reducing VOC outgassing from ventilation filters under both static and dynamic conditions. Additional work, however, needs to be conducted to investigate the effect of different baking temperature and time on VOC reduction and on filter collection efficiencies. In conclusion, baking is recommended for newly manufactured ventilation filters to reduce VOC outgassing significantly and thus minimize specific concerns.

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