## **MELT-BLOWN FILTERS**

Page 157

## Characterization of meltblown filters made of polypropylene and polypropylene—antimicrobial blends

Microorganisms and other bioaerosols are common pollutants in indoor air, leading to ailments ranging from mild allergic reactions to severe illness. Drug-resistant strains of tuberculosis are a particular source of concern because this disease is caused by a accerium that is spread through the air.

Most bioaerosols are in the range of 0.5-10 µm. Airborne particles in this range can be collected using non-woven fibrous air filters. However, captured bioaerosols will grow under the right conditions. Air flow through the filter can dislodge particles of new growth, which then reenter the circulating air.

This study evaluated the possibility of producing bacteria-resistant filters by adding antimicrobial compounds to polypropylene resin and then extruding the material as a filter medium. The results suggest that biocide-impregnated fibers in indoor air filters could reduce concentrations of bioaerosols in recirculating indoor air.

MAUREEN DEVER,
WAYNE T. DAVIS,
ANDREW A. ARRAGE,
DAVID C. WHITE, AND
ROBERTO S. BENSON
University of Tennessee

## **NONWOVENS**

Page 16

## New glass-fiber geometry a study of nonwovens processability

A new form of glass fiber is now commercially available under the trade name Miraflex™. The product—a bicomponent fiber consisting of two glass formulations with different coefficients of thermal expansion—is formed using a proprietary glassfusion technology. This process produces a fiber with a unique three-dimensional geometry of irregular twists.

The new fiber was used in three major textile processes (carding, needling, and air laying) under a range of conditions. Results of studies on commercial equipment demonstrate that all three processes can produce fiber structures of 100% Miraflex. The authors present recommendations for specific equipment features and operating parameters that were found suitable for processing this fiber.

The new fiber offers an interesting alternative to synthetic fibers and could be a cost-effective option in high-performance nonwovens applications.

- MARYANN C. KENNEY AND SANDRA K. BARLOW Albany International Research Co.
- SHERI L. EIKLEBERRY
  Owens-Corning

## **COTTON NONWOVENS**

Page 179

Hydroentangled nonwovens made from unbleached cotton

Bleached cotton fibers are commonly used in nonwoven fabrics. Their softness and absorbency make them especially useful in fabrics that contact the skin and in wipes. These desirable attributes are preserved in fabrics where the fibers are bonded by hydroentanglement. This paper examines the use of lower-cost unbleached cotton as a raw material for hydroentangled nonwovens.

Carded, crosslapped webs of unbleached cotton were hydroentangled on a pilot-scale unit over a range of specific energies. The study identified the practical range of processing conditions to produce fabrics with high tear and tensile strengths. Moreover, the results show that the mechanical energy applied by the water jets during hydroentanglement removes surface oils and waxes from the unbleached fibers. The resultant fabric can be bleached at lower cost per pound than the raw fibers before processing. Hydroentangled raw-cotton nonwovens also can be dyed, and they have better fabric handle because of the lower level of hydrogen bond-

- THOMAS F. GILMORE AND NEELESH B. TIMBLE North Carolina State University
- GLENN P. MORTON Cotton Inc.



# Characterization of melt-blown filters made of polypropylene and polypropylene—antimicrobial blends

MAUREEN DEVER, WAYNE T. DAVIS, ANDREW A. ARRAGE, DAVID C. WHITE, AND ROBERTO S. BENSON

NDOOR AIR FREQUENTLY CONTAINS higher concentrations of microbes, bioaerosols, and volatile organic compounds (VOCs) than are found in the outside air.

#### **BIOAEROSOLS**

#### Bioaerosols in enclosed environments

Indoor-air bioaerosols are an accumulation of a variety of biological agents and/or their products in one or more source reservoirs, with the potential to become airborne near the source. Bioaerosols may include, but are not limited to, bacterial cells, fungal mycelia, viruses, protozoa, microtoxins, pollens, dust mites, skin scales or hair of mammals, residues or products of organisms, and excreta or fragments of insects. Bioaerosols are removed from an airstream by filters, electrostatic air cleaners, adsorbers, centrifugal separators, and air washes (1). Most bioaerosols of concern in the indoor environment are in the 0.5-10-μm range (2-4). Table I lists bioaerosols reported in indoor environments.

Bioaerosols are collected using the same principles used for other particulates, but the variety and viability of the organisms complicate their collection and counting (4). Bioaerosols impacted in and on an air filter can remain dormant and then grow when conditions become favorable. Problems develop when high counts are in the air or when the microorganisms are pathogenic

(4-7). Moreover, bioaerosols that penetrate the filter can deposit on interior building surfaces and grow into larger aggregates. Such sites become a new biocontaminant source when they shed particles that are re-entrained in the indoor air (2-4, 8-19). Static chambers with known environmental conditions are used to study the behavior of particulates in HVAC (heating, ventilation, and air conditioning) systems (5, 7-9).

Epidemiologists have documented the spread of measles and tuberculosis via building ventilation ducts. These studies have shown that disease transmission increases with the increase in recirculated air. Indoor air samples obtained at a university had 2-46 times the bacteria compared with the outside air in 10 of 23 HVAC systems (10). In hospitals, where the spread of resurgent tuberculosis is becoming an increasingly serious problem, the dental areas showed the highest bacterial fallout on blood agar, followed by the sterilization areas and operatories

Indoor air is often humidified, and humidifier reservoirs are often contaminated with microorganisms that are free to travel through air-distribution systems. Disinfection of humidifier reservoirs with silver and copper ions shifts the major bacterial contaminants from *Pseudomonas aeruginosa* to *P. paucimobilis* (12). In a survey of 14 buildings in the Atlanta area, the nine buildings

MELT-BLOWN FILTERS ABSTRACT tiganing and a property of the contract of the level south in the green in Augustriality (augustriality) and augustria en als the compensations are noticed មិនទី ម៉ូមីមីស្រី ម៉ាទី ទើបមានកម្មបំពុក សង្គេស विस्तित्र सम्बद्धाः । स्वतः । स्वतः । els Components had a composit នៃក្រៅមកដែលនាស់ក្រៅក្រសួលក្រសួលនេះ Artifel (2008) in synthesikejine, syn susince insiene mai ma egas Me អាមារជានេះមិន «មារស្រួនមារសារ នេះ ការដែ ទី២៥៩ មន្តិការនេះ ២០១៤១៣១ នាគ្រាក កែកមានមាន ខាងក្រៀបចំ មានជាប្រជាជា าห์บังได้โดย น้องรูปแล้งน้องสุด เกิด เพละร विद्रश्रह्म वार्षः वार्यस्थानस्य विद्राप्तिका प्रिंगहर्ग्यम्बर्धाः । ग्रेन्ड व्यक्तित्वः increased with increasing concentra-tions of antimicrobial compounds: Fil-tration efficiency, decreased with increasing fiber diameter. Application: Fortification of polypropylene resin with antimicrobial compounds shows promise as a method for producing aseptic air filters.

thought to have the "sick building" syndrome all had higher levels of fungi contamination in their indoor air than the five "healthy" buildings (13).

Airlines provide several interesting cases of biocontamination. For example, a jet with an inoperative ventilation system was grounded for four hours while the system was repaired. During that time, 72% of the passengers contracted influenza, with all cases traceable to a specific passenger. In another situation, 18 of 34 people in a Navy

lection efficiencies (at 17 m³/min with a pressure drop of 12-mm H<sub>2</sub>O) of a melt-blown electret HEPA filter over a two-year life span to be >99.9999 and 99.999, respectively, for 0.1-µm and 0.3-µm particles. While it is clear that HEPA filters and electronic air cleaners are more efficient than the typical residential air filters, they are also much more expensive.

#### **OBJECTIVE**

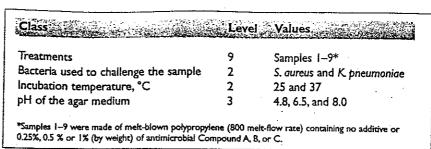
This study was carried out to evaluate the possibility of producing bacteria-resistant filters by adding antimicrobial compounds to polypropylene (PP) resin and then processing the material into a filter medium. In particular, the researchers sought to establish whether the antimicrobial additives would (a) migrate to the fiber's surface, where it could contact the bioaerosol and (b) remain effective after being subjected to the heat encountered in processing.

Three different antimicrobial compounds at three concentrations (0.25%, 0.50%, and 1% by weight) were compounded into PP, which was then processed into melt-blown nonwoven filter media. The presence of the antimicrobial compounds on fiber surfaces was determined using FTIR (Fourier-transform infrared spectrometry) and SEM- EDX (scanning-electron-micro-graphy-electron-diffraction X-ray). The efficacy of the antimicrobial compounds was determined using gram-positive and gram-negative bacteria. Thermal, morphological, and mech-anical properties of the nonwovens were determined along with the filtration efficiencies of the media.

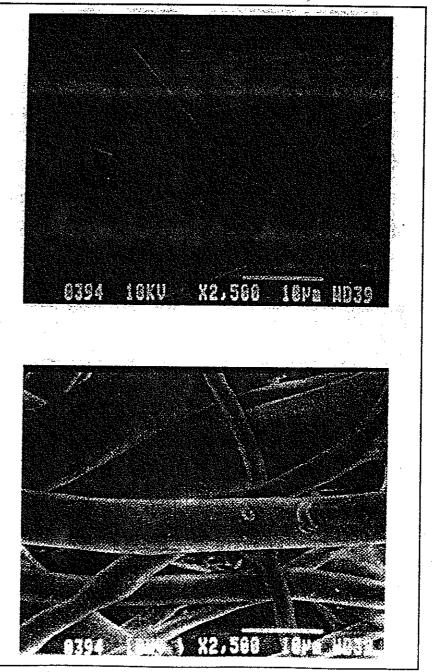
# EXPERIMENTAL TEST METHODS AND MATERIALS

Antimicrobial compound additives

Three antimicrobial compounds were added to PP (Exxon Chemical Co., Baytown, TX). Each antimicrobial compound was added at concentrations of 0.25%, 0.50%, and



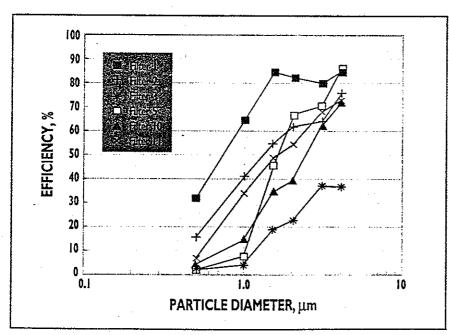
ll. General linear model



2. SEM-EDX of the control (top) and Sample 4 (bottom) containing 1% Compound A

Organism'	Size, µm 🖓 - 🛬 👙
Bacteria	· 16 4 5 4 5 5 5 13 11 11 11 11 14 45 15 16 16 16 16 16 16 16 16 16 16 16 16 16
Escherichia coli	$1.1-1.5 \times 2-6$
Pseudomonas aeruginosa	0.5-0.7 x 1.5-3.0
Staphylococcus aureus	0.5–1.0 (diam.)
Bacillus subtilis	0.7-0.8 × 2-3
Klebsiella	$0.3-1.0 \times 0.6-6.0$
Legionella pneumophila	$0.3-0.9 \times 2-20$
Staphylococcal pneumonia	0.5-1.5 (diam.)
Streptococcai pneumonia	0.5-1.5 (diam.)
Micrococcus luteus	0.9-1.8 (diam.)
Staphylococcus epidermus	0.5–1.5 (diam.)
Bacillus circulans	0.6-1.0 × 2-5
Bacillus anthrasis	1.0-1.2 x 3-5
Fungi	
Thermoactinomyces vulgaris	I-2 (diam.)
Acanthamoli spp., Naegleria spp.	• • • • • • • • • • • • • • • • • • • •
Penicillium	4-6 (diam.)
Aspergillus	4-6 (diam.)
Coccidioidomycosis immitis	60–200 (diam.)
Cladosporium	8–15 (diam.)
Viruses	• •
Rhinovirus	0.02-0.03 (diam.)
Influenza virus	0.08-0.12 (diam.)
Lymphocytic choriomeningitis	0.2-0.3 (diam.)
Coxsackie virus	0.02-0.03 (diam.)

## I. Bioaerosols reported in the indoor environment (3, 4)



1. Efficiency of six residential HVAC air filters at air flow of 2.34 mls (460 ft/min)

squadron caught flu while traveling in a military DC-9 with all the freshair ventilating systems working perfectly. Tuberculosis, a bacterium that spreads through the air, is a source of concern among those who use public transportation systems. To date, the U.S. Centers for Disease Control has investigated four cases of suspected tuberculosis outbreaks on airplanes. Only one of these investigations established transmission of this disease. In that case, it was found that a flight attendant infected 13 coworkers over the course of several months (14).

# Removing bioderosols from enclosed environments

Commercially available residential air filters span the range from standard fibrous mats to filters containing electrostatic fibers (electrets) that enhance particulate removal to carbon-impregnated filters that enhance removal of organic vapors. In a previous study (15), 11 commercially available filters were evaluated to determine their efficiency at removing particulates between 0.5-4.0 µm. The filters were evaluated in a simulated home heating system at velocities of approximately 450-750 ft/min to determine their initial efficiencies before loading. An optical particle counter with a multichannel analyzer was used to simultaneously measure the concentration of particles in six particle-size increments. The measured efficiencies of the filters were compared with values predicted by using filtration theory and the measured filter characteristics (fiber diameter, thickness, packing density, and filtration velocity). In general, the residential filter efficiencies ranged from 0-32% for 0.5-µm particles up to 35-86% for 4-µm particles, as seen in Fig. 1.

Most residential furnace air filters have been found to provide negligible removal of particles in the size range of 0.1-1 µm, i.e., those that penetrate deeply into the human lung (5, 15). Airborne bioaerosols in this size range can cause problems in susceptible people either by causing an infection or an allergic reaction. Gravitational settling has little effect on such particles, which are light enough to be carried by air currents and circulated through HVAC systems. Highefficiency particulate air (HEPA) filters remove small airborne contaminants at flow rates typically found in commercial and residential buildings. Tani (16) determined the colo o > fc W el ci fi e: T at te m le th

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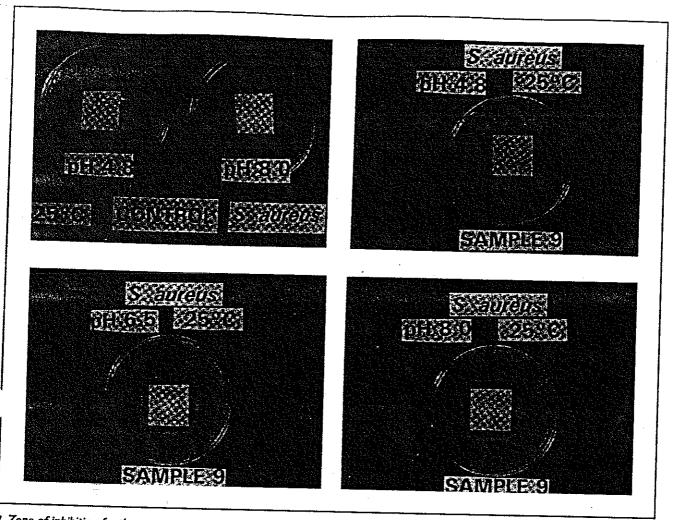
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3. Zone of inhibition for the gram-positive bacteria (S. aureus) at 25°C and three levels of pH for Sample 9 (0.5% Compound C)

Fiber diameters were determined using a scanning electron microscope (SEM). Each sample was placed on an aluminum stub, secured with silver paint, and coated with 3 nm of gold. Photographs of each melt-blown web were taken at 400X magnification using a field-emission SEM (Hitachi®). These photos were examined to detect the average fiber diameter. The standards for all fiber-diameter measurements were photographs of 1-µm polystyrene spheres taken at the same SEM settings used throughout the study.

To measure the average fiber —ameter of each sample, a line was drawn from opposite corners through the center of the picture. This was repeated, resulting in a large "X" drawn on the picture. The

photographs were scanned into a computer, and fiber diameters were measured using an image-analysis software program. Diameters of all fibers that crossed the "X" were measured.

### Collection efficiency

Collection efficiency of the filter samples was determined using 1- $\mu$ m polystyrene spheres at 30 cm/s velocity, in accordance with the procedures described in ASTM F 1215-89 (17, 18). An optical particle counter determined the number of particles upstream ( $C_{\rm u}$ ) and downstream ( $C_{\rm d}$ ) of the sample. These values were used to calculate the collection efficiency (E):

$$E = [(C_u - C_d)/C_u] \times 100$$
 (1)

#### Thermal analysis

A differential scanning calorimeter (Perkin-Elmer®, DSC-7) was used for the thermal analysis of pure PP and blends of PP-antimicrobial compound. The DSC was calibrated for temperature as described in the user's manual using pure indium  $(T_m)$ = 156.60°C) as the reference standard. The baseline calibration was performed using two empty aluminum sample pans. Nitrogen continuously flowed through the sample chamber. The samples were weighed on an analytical balance (Mettler®) and crimp-sealed in aluminum pans. Sample weights ranged from 2.5 mg to 5.0 mg. All samples were heated from 40°C to 200°C, held at 200°C for one minute, and then cooled to 40°C. The heating

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Sam No.	ple Treatment	25	ALVEN OH OS	830.	465	आ (द हो   हो	80	41 <b>5</b>	2596 pH = 65	801	7 <u>.</u> 7.	97 (C 19 <b>.9</b> 19.5	8.0
1	Control	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E
2	0.25 wt.%												
	Compound A	Ε	E	Not E	E	E	Not E	Not E	Not E	Not E	Not E	Not E	Not E
3	0.50 wt.%												
	Compound A	E	E	Not E	E	E	Not E	Not E	Not E	Not E	Not E	Not E	Not E
4	1.00 wt%												
	Compound A	Ε	E	Not E	E	E	Not E	Not E	Not E	Not E	Not E	Not E	Not E
8	0.25 wc%					-							
	Compound C	E	E	É	Ε	Ε	E	E	Ε	E	Ē	Ε	E
9	0.50 wt.%					-							
	Compound C	E	Ε	Ε	Ε	E	Ε	Ε	E	E	Ε	Ε	Ε

III. Contact inhibition\*

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Sampl No:	e Treasment	<u> </u>	##€ 17 <b>:</b> 17:	30	ÇIE.	011€ 13 <b>4</b> 65		45	7). (C 1) (C	6.0	<b>/</b> j.	ον/6 19#1 15	GÜ	
1	Control	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	
2	0.25 wt.%													
	Compound A	Not E	Not E	Not E	Е	E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	
3	0.50 wt.%												:. <u>-</u>	
	Compound A	Not E	E	Not E	E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	
4	1.00 wt.%													
	Compound A	Not E	Not E	Not E	E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	Not E	
8	0.25 wt.%											_	_	
	Compound C	Ε	E	E	Ε	Ε	Ε	Ε	Not E	Not E	E	E	E	
9	0.50 wt.%													
	Compound C	Ε	E	E	Ε	E	Ε	Ε	Not E	Ε	Е	E	E	

IV. Zone of inhibition\*

1.00% by weight. The materials were then spun into melt-blown nonwoven filter media. A sample containing no antimicrobial compound served as the control for comparison throughout the study.

# PP resin and extrusion conditions

An 800 melt-flow-rate PP (Escorene<sup>16</sup>, Grade PD-3545-G) was used throughout the study. The melt-blowing trial was conducted on the 0.1524-m (6 in.)-wide melt-blowing line in the Textile Processing Labora-

tory at the University of Tennessee, Knoxville. The line consists of a 0.01905-m (0.75 in.) screw extruder (10:1 L/D), two-position screen changer, a horizontal die with hundreds of spinnerette orifices, a furnace for heating the air, and a rotary-screw air compressor with a maximum capacity of 18.4 m³/min (650 std. ft³/min). Air flow rates were 2.8 m³/min (100 std. ft³/min). The die-to-collector distance was 30.5 cm (12 in.). Polymer throughput was 0.4 g/hole/min. The collector speed was

varied to produce webs of  $33.9 \text{ g/m}^2$  (1 oz/yd<sup>2</sup>).

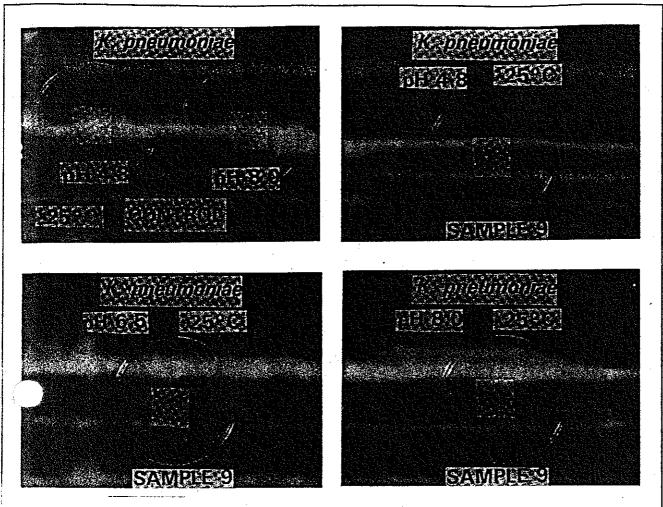
# Characterization of the air-filter

The thicknesses of the melt-blown webs were measured with an Ames<sup>™</sup> thickness gauge (Testing Machines, Model 85-0071) according to ASTM Test Method D 1777-64 (18). Basis weight was measured according to ASTM D 3776-79 (18). Air permeability was determined using a Frazier<sup>®</sup> air permeometer according to ASTM D 737-75 (18).

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4. Zone of inhibition for the gram-negative bacteria (K. pneumoniae) at 25°C and three levels of pH for Sample 9 (0.5% Compound C)

and cooling rate was 10°C/min.

#### Morphology

A light microscope (Olympus® BF22) with video output to a 12-in. television and VCR was used to record and study the morphology of the pure PP and PP-antimicrobial blends. Thin sections of the samples were heated to 200°C and cooled in a Mettler FP-80 hot stage at a rate of 10°C/min. The samples were positioned in the viewing path of the microscope.

#### Tensile properties

The tests were performed according to ASTM D 882-83 using a tensile tester (Instron, model 1122) (18). The sample dimensions were approximately 5 mm x 70 mm x 0.1 mm. Gauge length was 50 mm; maxima.

mum load was 2000 g; crosshead speed was 100 mm/min; and chart speed was 50 mm/min. The mechanical properties such as tensile modulus, tensile strength, stress-at-break, and elongation-at-break were determined from the mechanical data. The modulus was measured at 10% elongation. A total of seven samples were tested for the pure PP and for each of the PP-antimicrobial blends.

#### FTIR spectroscopy

An FTIR (Digilab, model FTS-40) equipped with a microscope (VMA-300) was used to collect the spectra. A total of 1024 scans and a resolution of 4 cm<sup>-1</sup> were used to collect the spectra.

#### Surface analysis

Surface analysis was conducted with

an FTIR spectrophotometer (Perkin-Elmer\*) and SEM-EDX.

#### Testing procedures

The efficacy of the antimicrobial compounds impregnated within the PP air-filter media was determined using a modification of AATCC Test Method 147-1982, "Detection of antibacterial activity of fabrics: parallel streak method" (19). The agar plates were streaked to yield confluent bacterial growth instead of the five parallel streaks described in the test method.

## Bacterial strains and test condi-

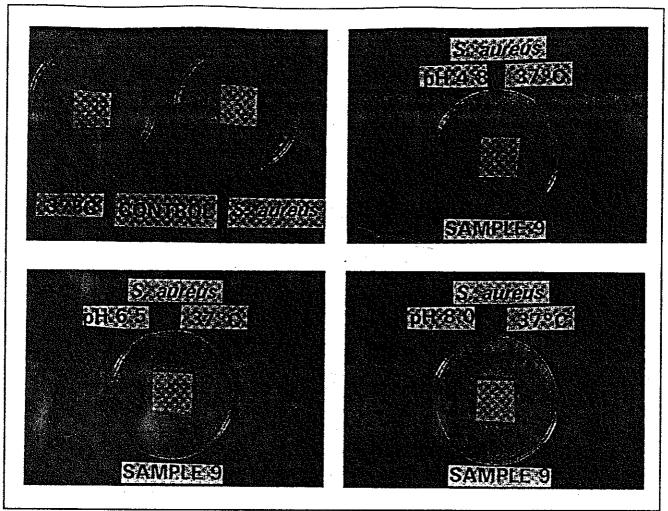
Staphylococcus aureus (ATCC 6538) and Klebsiella pneumoniae (ATCC 4352)—gram-positive and gram-negative bacteria, respec-

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5. Zone of inhibition for the gram-positive bacteria (S. aureus) at 37°C and three levels of pH for Sample 9 (0.5% Compound C)

tively—were maintained on agar plates at 37°C. Liquid cultures were prepared by inoculating 10 mL of nutrient broth with 5-6 isolated bacterial colonies from the stock plates and incubated at 37°C for 18 h prior to use.

The original agar pH was 6.5. Two additional pH levels were prepared by adjusting the agar pH to 4.8 with HCl and 8.0 with NaOH. The agar plates were heavily streaked with a sterile cotton swab soaked in the 18-h broth culture to yield a confluent lawn of bacterial growth. The plates were incubated at 25°C and 37°C.

Immediately after streaking each bacterial strain onto identical sets of agar plates, a 1-in.<sup>2</sup> sample of melt-blown nonwoven filter medium was placed in the center of each plate.

Triplicates were tested against each bacterial strain at the three pH levels and two incubation temperatures for a total of 36 plates per sample.

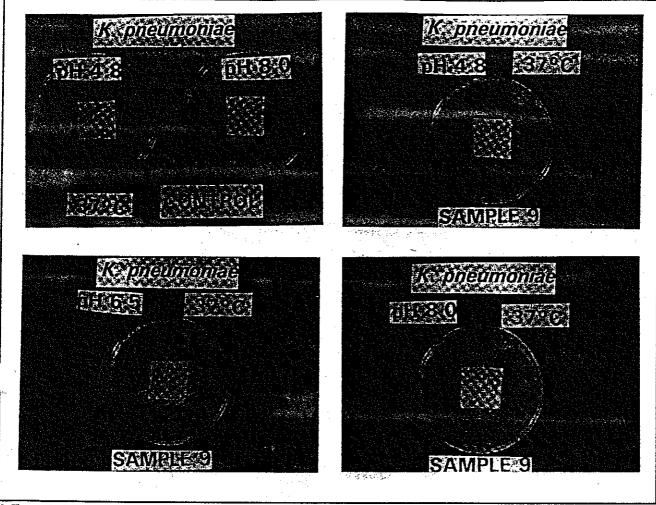
The plates were examined for indications of antibacterial activity after incubation times of 24 h and 48 h. Two different types of activity were measured: contact growth (i.e., growth on or under the sample) and leaching inhibition (i.e., inhibition of growth in a zone around the sample). Contact growth was quantified on a scale of 0-4, with zero indicating no antimicrobial effectiveness (100% bacterial growth on or under the sample) and 4 indicating no growth. A score of 1 indicated 50-99% growth; 2 indicated 10-50% growth; and 3 indicated 1-9% growth. Leaching inhibition was demonstrated as a zone of clearing beyond the material

sample measured in millimeters.

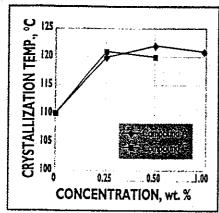
#### **Statistics**

The general linear model for the study is listed in **Table II**. A one-way analysis of variance (ANOVA) was used to determine if the response variables—contact growth and leaching inhibition—were statistically significant at p 0.01, which indicates that the probability of the event's occurrence by chance is less than 1%. The analysis of variance was conducted based on three assumptions:

- The values for the response variables are not dependent on each other.
- 2. The observations are sampled from a normal distribution.
- 3. The groups have equal variance.



6. Zone of inhibition for the gram-negative bacteria (K. pneumoniae) at 25°C and three levels of pH for Sample 9 (0.5% Compound C)



T. Crystallization temperature as a funcn of the concentration of antimicrobial compounds in PP matrix. (Results obtained from differential scanning calorimetry of PP and PP—antimicrobial blends.)

These three assumptions were met. If the analysis-of-variance *F*-test statistic was significant, then the least-square means for each dependent variable were calculated.

#### RESULTS

#### Preparation of melt-blown airfilter media

Ten melt-blown samples were made and characterized. Sample 1, the control, contained no antimicrobial compound. Samples 2, 3, and 4 contained 0.25%, 0.50%, and 1% Compound A, respectively. Samples 5, 6, and 7 contained 0.25%, 0.50%, and 1% Compound B, respectively. Samples 8, 9, and 10 contained 0.25%, 0.50%, and 1% Compound C, respectively. Melt-blown processing of PP with the 1% concentration of Com-

pound C was not completed because of processing problems.

#### Surface analysis

FTIR analysis showed that the three samples containing 0.25%, 0.50%, and 1.00% of Compound A (Samples 2, 3, and 4, respectively) contained the theoretically expected concentration of Compound A. Although the amount of the compound in the sample was not quantified, the FTIR analysis showed no trace of any antimicrobial compounds in the control (Sample 1). There was a direct correlation between the FTIRmeasured amount of Compounds A and C in the nonwoven and the amount added to the PP before processing. Compound B was not detected in any of the melt-blown samples.

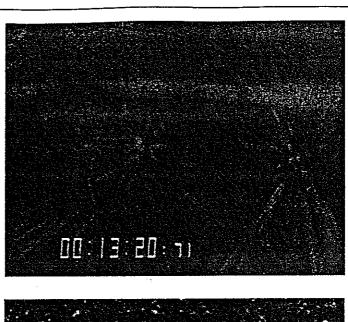
The two micrographs in Fig. 2 were taken using SEM-EDX. The top picture is the control (Sample 1), which contains no antimicrobial compound. The background, at about 1 keV, was recorded and subtracted from the compound levels found in the sample containing 1% Compound A (lower photo in Fig. 2). The white specks on the fiber surfaces were identified as antimicrobial Compound A using SEM-EDX.

The micrographs in Fig. 2 provide at least a partial answer to one of the questions posed by the authors at the onset of this research study. Initially, it was not known if the antimicrobial compounds would migrate to the surface of the PP fibers, where they would be most effective at preventing bacterial growth. The SEM-EDX and FTIR surface-analysis techniques showed the presence of antimicrobial Compounds A and C on the fiber surfaces. However, the quantitative amount on the surface vs. the amount remaining within the fiber is not known.

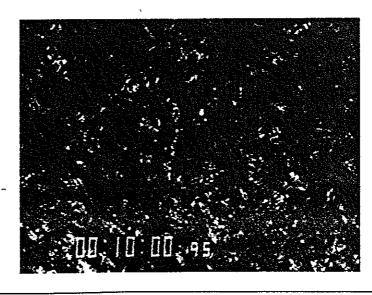
#### **Statistics**

The large F-test statistics generated by the ANOVA for the two response variables—contact growth and zone of inhibition—were statistically significant at the p <0.01 level. Least-square means were calculated for the test conditions evaluated in the study, and the results are reported in **Tables III and IV**. For cases where the test statistics were statistically significant at the p < 0.01 level, an "E" is reported to indicate that the treatment was effective. "Not E" indicates the treatment was not effective.

Both the gram-positive (S. aureus) and gram-negative (K. pneumoniae) bacteria grew on the control sample containing no antimicrobial additive. Bacterial growth did not appear to be affected by the pH of the agar medium or by the incubation temperature. The gram-positive (S. aureus) bacteria did not



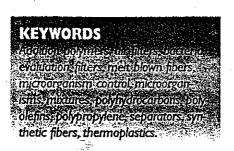




8. PP spherulites (top), PP-Compound A spherulites (middle), and PP-Compound C spherulites (bottom). Spherulites were obtained by cooling the melted melt-blown filter medium.

Property		2	SAMPLE	NO.	8	9
Antimicrobial compound	None	A	Α .	<b>A</b>	С	С
Concentration, wt.%	•••	0.25	0.5	ł	0.25	0.5
Decomposition temp., °C		464	4 <del>64</del>	464	300	300
Melt-blown filter			•			
Weight, g/m²	, 33.56	31.75	29.02	28.12	31.75	39.91
Thickness, cm	0.0382	0.0355	0.035 <del>4</del>	0.0349	0.036 <del>4</del>	0.0416
Air permeability, ft3/min/ft2	99.38	115.80	155.00	144.00	172.00	168.00
Fiber diameter, µm	2.85	2.65	3.59	4.12	2.76	3.60
Filtration efficiency, %	48.2	35.6	33.7	30.3	31.1	26.1
Modulus, MPa 🥻	33.3	32.7	35.1	33.5	<del>4</del> 7.2 <del>4</del>	45.92
Tensile strength, MPa	2.65	2.66	2.51	2.89	9.78	9.20
Stress at break, MPa	2.60	2.66	2.39	2.81	9.78°	9.20
Elongation at break, %	28.2	14.8	17.9	16.5	27.5	31.6

V. Characterization of the melt-blown air-filter samples



grow on samples containing antimicrobial Compound A at agar with pH 4.8 or 6.5, as seen in Table III. However, at pH 8, the gram-positive bacteria grew on the sample. The same trend was seen at both incubation temperatures. Compound A did not prevent growth of the gram-negative (K. pneumoniae) bacteria at any pH. Compound C was effective at preventing contact growth of both the gram-positive and gramnegative bacteria on all of the samples at all three agar pH levels and at both incubation temperatures.

The same trends reported for bacterial growth on the samples were observed for the zone of inhibition. Table IV shows that Compound A was effective against only the gram-positive (*S. aureus*) bacteria at the low agar pH levels of 4.8 and 6.5 and was ineffective against the gram-negative (*K. pneumoniae*) bacteria under any of the conditions evaluated. Compound C was effective against both the gram-positive

and gram-negative bacteria at both incubation temperatures and at all three levels of pH. Figures 3–6 show the zone-of-inhibition results for samples containing 0.5% Compound C.

#### Mechanical properties

The crystallization behavior of pure PP and PP-antimicrobial blends was studied. As seen in Fig. 7, the crystallization temperature ( $T_c$ ) of PP increased from 110°C to 122°C with the addition of Compound A. Since the presence of nucleating agents increases the  $T_c$ , these results suggest that the biocide acts as a nucleating agent for PP during the solidification process.

A material that functions as a nucleating agent must fulfill certain requirements: It must be insoluble in the polymer that is being nucleated; it must be wetted or absorbed by the polymer; and its melting temperature must be higher than the polymer. Moreover, the activity of a nucleating agent increases with the density of the nucleus, with higher densities translating to greater increases in  $T_c$ .

Morphological studies of the filter media support the proposition that Compounds A and C served as nucleating agents in the PP matrix. These studies showed a decrease in the size of the PP spherulites with addition of biocide, as seen in Fig. 8. The usual dependence of  $T_{\rm c}$  on the concentration of nucleating agent is observed in these studies, albeit only at the lowest concentration of 0.25%. The initial addition of 0.25% of Compound A increased  $T_{\rm c}$  by 10°C, while subsequent additions (0.5% and 1.0% by weight) increased  $T_{\rm c}$  by only an additional 2°C.

The effects of additions of Compounds A and C on the mechanical properties of PP are summarized in Table V. The presence of Compound A in the PP reduced the elongation-at-break but had a negligible effect on the modulus, tensile strength, and stress-at-break. The decrease in elongation-at-break while other properties remained constant could be the result of a reduction in the average size of the amorphous regions.

#### Filtration efficiency

The control sample containing no antimicrobial compounds had the highest filtration efficiency (48.2%) and contained small-diameter fibers (avg. 2.85 µm), as seen in Table V. The collection efficiencies of the three samples containing 0.25%, 0.50%, and 1.0% Compound A were 35.6%, 33.7%, and 30.3%, respectively. Fiber diameters increased with increasing concentration of Compound A (avg. 2.65 µm, 3.59 µm, and 4.12 µm, respectively). As expected, filtration efficiency

decreased with increasing fiber diameter (and thus with increasing concentration of antimicrobial compound). The same trend was also seen with Compound C. Filtration efficiencies for samples containing 0.25% and 0.5% Compound C were 31.1% and 26.1%, respectively. The average fiber diameters in the samples containing Compound C were 2.76 µm and 3.60 µm, respectively. In all samples, fiber diameter increased with increasing concentration of antimicrobial compound. as seen in Table V. This suggests that the antimicrobial additive was a nucleating agent that increased the rate of crystallization, thereby limiting the reduction in diameter of the melt-blown fibers. Because filtration efficiency is related to the fiber diameter, this finding will need to be addressed in future studies.

#### CONCLUSIONS

Polypropylene (PP) resin is used to produce melt-blown nonwoven filter media. This paper describes initial research efforts to develop an aseptic air filter by adding antimicrobial compounds to PP resin.

Three antimicrobial compounds were tested. Addition of antimicrobial compounds led to an increase in fiber diameter and thus a decrease in filtration efficiency. Fiber diameter also increased with additive concentration.

The three compounds varied in their effectiveness against gram-positive (Staphylococcus aureus) and gram-negative (Klebsiella pneumoniae) bacteria at three levels of pH (4.8, 6.5, 8.0) and two levels of incu-

bation temperature (25°C and 37°C).

The effectiveness of antimicrobial Compound A increased only slightly as its concentration increased from 0.25% to 0.50% to 1.00% by weight. This suggests that 0.25% is sufficient and may be excessive. Consequently, the effectiveness of Compound A at lower concentrations will be investigated in future studies. Compound A was effective against the gram-positive bacteria at the lowest level of pH tested (pH 4.8). It was less effective at pH 6.5 and ineffective at pH 8.0. Incubation temperature had little effect on the results, although bacterial growth was slightly higher at the higher temperature. Compound A was ineffective against the gram-negative bacteria at all conditions studied.

Fibers containing Compound B had no effect on bacterial growth. Interestingly, Compound B was the only one of the three additives that was not detected in the melt-blown fibers, even though all three compounds were added using the same procedures. Decomposition apparently was not a factor, since the three antimicrobial compounds had similar decomposition temperatures.

Compound C was effective against both the gram-positive and gram-negative bacteria under all conditions tested.

#### **FUTURE STUDIES**

Future studies will investigate (a) the effectiveness of lower concentrations of antimicrobial compounds, (b) the effectiveness of the

antimicrobial compounds - on microorganisms commonly found in sick buildings, (c) the development of more efficient melt-blown air filters by producing thicker samples containing smaller fibers, and (d) the stability of antimicrobial agents at the elevated processing temperatures. TJ

Dever is a research assistant professor; Davis is a professor in the Environmental Engineering Dept.; Arrage is a technician and White is a professor in the Center for Environmental Biotechnology, and Benson is an associate professor in the Materials Science Engineering Dept. All are affiliated with the University of Tennessee, Knoxville, TN 37996.

The authors note the contributions of numerous parties that made it possible to conduct the research described herein. The melt-blowing processing facilities of TANDEC (Textiles and Nonwoven Development Center), sponsored by Exxon Chemical Co., was used to generate the filter media. Filter efficiencies were done by Wayne T. Davis. Characterization of the filter media were done by Roberto S. Benson in the Materials Engineering Department. Efficacy of the antimicrobial compounds to bacteria were done by Andrew A. Arrage and David C. White at the Center for Environmental Biotechnology. All research was done at the University of Tennessee, Knoxville.

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