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Establishment of Technological Package for Production of Antimicrobial PET and PA Nonwoven Fabrics on Pilot Scale

Shalaby, S. E.[1]; Saad, M. M.[1]; Balakocy, N. G.[1]; Ibrahium, Y. H.[2]

[1]Textile Research Division; [1,2] National Research Centre, Dokki, Cairo, Egypt.
(Scopus Affiliation ID: 60014618)

Correspondence Author: Shalaby, S.E.,

([2]Enviromental Research Division)

Abstract

The scope of this technological process is producing polyester (PET) and polyamide-6 (PA) nonwoven fabrics having antimicrobial properties on pilot scale. The hydrolyzed PET and regular PA fibers were finished with antimicrobial substance (AS) on pilot scale. The suggested technological processes was carried out under the optimum conditions obtained from our previous work on bench scale. The finished PET and PA fibers have been converted into nonwoven fabrics using mechanical needle punched process. The effect of production parameters such as opening, carding, cross layering, mechanical bonding and finally, needle punching on their antimicrobial properties were investigated. The produced antimicrobial nonwoven fabrics showed high antimicrobial activity even after five washing cycles. The physico-mechanical properties of the produced antimicrobial PET and PA nonwoven Fabrics were measured. The quality and in-application performance assessment of the produced antimicrobial nonwoven fabrics as air filters and antimicrobial surgical masks was evaluated.

Keywords: PET, Regular PA-6 Nonwoven fabrics, Alkali Hydrolysis, Antimicrobial Finishing, Physico-Mechanical Properties, Air Filters, Face Masks.

INTRODUCTION

Polyethylene terephthalate (PET) and polyamide-6 (PA) fibers are widely spread. They find applications in several areas including protective clothing, sports, apparel, active wear, medical textiles and in many technical applications**.** Among the textiles applications, nonwovens are one of the fastest-growing segments of the textile industry and constitute roughly one third of the fiber industry according to the latest estimates, taking into account the official INDA (Association of Nonwoven Fabrics Industry) [1].

Filters made of textiles can be classified in two main groups, woven and nonwoven. The nonwoven filters have superior properties in contrast with woven counterpart: (a) available pores per unit area which imparted higher permeability and filtration efficiency, (b) no yarn slippage as that in woven media. In recent years, the new trends of nonwoven filters focus on lower cost, improved temperature resistance and lower pressure drop at a fixed efficiency to expand their applications [2].

In filtration, nonwoven fabrics can be generally described as a random fibrous web, formed by either mechanical, wet or laid means. They have interconnecting open area throughout the cross section, and able to remove a percentage of particulate from liquid or gaseous fluid streams flowing through it. Typically, nonwoven fabric filtration media have 1-500 micron mean flow pore (MFP) ratings. Below 10-15 micron, the fabrics must be calendared in order to achieve the finer micron rating [3].

The use of nonwoven in medical community has greatly expanded in recent years as evidenced by the fact that half of the drapes used in surgery are nonwovens. Technology increasingly requires room or work areas free of microbes, making it necessary to filter air in industrial activities. Pure air is essential in operating the pharmaceutical laboratories, clean rooms for electronics, museums, libraries and food industries [4].

The ultimate goal of the present work is to produce PET and PA nonwoven fabrics having durable antimicrobial activity. The optimum conditions for manufacturing on pilot scale antimicrobial PET and PA nonwoven fabrics are determined. Quality and in-application performance assessment of produced fabrics have been also investigated.

EXPREMENTAL WORK

Materials

- PET fibers (deniers 9 and 15) used throughout this work were kindly supplied by Polyester Fibers Company, Kafer El-Dawar, Egypt. The fibers were scoured at 80°C for 45 minutes, with solution containing 2.0g/L nonionic detergent, washed with water, squeezed, and finally air dried.
- PA fibers (denier 14) used throughout this work were kindly supplied by El Nasr Company, Elmehalla, Egypt. The fibers were scoured using the same method applied for scouring PET fibers.
- Antimicrobial substance (AS) under trade name " katamine " was in the form of 50% aqueous solution (Russia).
- NaOH (A.R grade).
- Microorganisms: *Bacillus mycords* (*B.m*) (Gram positive bacterium), *Echerichia coli (E.c)* (Gram negative bacterium) and *Candida alibcans* (*C.a*) (Non filamentous fungus) were used for estimation of antimicrobial potency of

control and treated polyester and polyamide fibers and nonwoven fabrics.

- Mask Outer Material
- The outer layer is a polyester spun lace nonwoven fabric having the following characteristics:
  1- Basic weight, 20 gm/m^2.
  2- Thickness, 0.15 mm.
  3- Rate of air-permeability: 320 cm^3/cm^2. sec @ 20 mmWG.

Methods

- Alkaline hydrolysis of PET fibers was carried out using the method described by shalaby et al [5].
- The fixation of antimicrobial substance on hydrolyzed polyester fibers and on Nylon-6 fibers on bench scales was carried out using the methods described by Shalaby et al [6] as follows:
- The fixation of AS was carried out using a high-temperature high-pressure laboratory dyeing machine. Required amounts of AS solutions were placed in stainless-steel bowls, hydrolyzed samples were immersed in the solutions, and the sealed bowls were rotated in a closed bath containing ethylene glycol at the desired temperature. The material: liquor ratio (M:L) was 1:50. The bath temperature increased at rate of 28°C/minute. After the predetermined durations, the samples were removed from the bath, rinsed repeatedly with distilled water and allowed to dry in the open air.
- Scouring, alkaline treatment, and antimicrobial finishing of PET and PA fibers on pilot scale were carried out in Misr El Bieda Dyers Co., Kafr El Dawar, Egypt. English tops machine. Shell-y 42 working under pressure at maximum temperature 100°C, volume 1000 liter and load 100 kg was used for carrying out the above mentioned technological processes under its optimal conditions as described in our previous investigations [7] .

Analysis:

- Antimicrobial potency of PET and PA fibers was quantified by diffusion [8] and by shake flask Method [9].
- Carboxylic content was determined according to the method described in [10].
- Physico-mechanical properties of finished PET and PA nonwoven fabrics were investigated in NRC central laboratory for testing textile fibers and fabrics.
- Assessment the Application in Air Conditioning and Ventilation Sectors was carried out in the NRC campus located in Dokki, Giza, Governorate, Egypt.

<u>a- Air Sampling</u>

Bioaerosol sample collection was accomplished with a one stage Andersen sampler [11] (Tisch Environmental Cleves, OH, USA). The sampler was placed upon a tripod, 1.5m above the floor level, and operated at the manufacturer recommended flow rate of 28.3l/min, for 5-10 minutes. This sampler divides particles which can deeply penetrate into the lungs into fine (≤ 2.5 µm) and (≤ 7.0 µm). Nutrient agar medium supplemented with cycloheximide [12] and Malt extract agar medium supplemented with streptomycin [13] were used for counting bacterial and fungal aerosols, respectively. The sampler was loaded with 100×15 mm Petri dishes with 20 ml of the collection media.

b-Sample Analysis

Total bacterial and fungal plates were incubated at 28°C for 48 hours, and 5-7 days, respectively. The plates of Gram negative bacteria were incubated at 37°C for 48 hours. Using positive holt conversion tables, the CFU/m^3 concentrations were calculated by CFU counts divided by sampling flow rate and sampling time [11]. Fungal isolates were, mainly identified by the direct observation on the basis of micro and macro morphological features on Sabouraud dextrose agar, Czapek Dox agar and malt extract agar, reveres and surface coloration of colonies. Cultivable isolates were identified to the genus level, using various Literatures [14-15].

RESULTS AND DISCUSION

The previously produced on pilot scales antimicrobial PET and PA fibers [7] have been used for the manufacturing of corresponding nonwoven fabrics. This was carried out using mechanical needle punched process on the production line of Egyptex Co., in the 6th October city, Giza, Egypt.

Pilot Scale Production of Antimicrobial PET and PA Nonwoven Fabrics

This process includes the following production steps:

1- Opening: The objective of an opening stage is to reduce the size of tuft fibers and mobilizing or bagging in bals.
2- Carding: The main objective of carding is to separate small tufts into individual fibers and blending different opening bals to deliver the fibers in the form of webs.
3- Cross Layering: Web formations can be made into desired web structure by the layering of webs from the card. Cross lappers device, vertical and horizontal cross lapper and pressed webs by metallic cylinder coated by rubber.
4- Mechanical bonding: Needle punching is a process of bonding the web which produced by cross lapper by first set of barbed needles boards (1600 needles/m^2).
5- Final Punching stage: In this stage the web is drawn to the final punching (upper and lower). The needle density per square meter is 18000/m^2.

Production Parameters:

A. 100 kg antimicrobial PET fibers with fiber denier 15.
B. 65 kg antimicrobial Nylon-6 fibers with fiber denier 14.

C. Fiber length: 90nm.

D. Needle density: 50/ inch^2.

E. Needle count: 32.

F. Punching rate: 250 punches/ Cm^2.

Quality and in Application Performance Assessment of the Produced Antimicrobial Polyester and PA-6 Nonwoven Fabrics

1. Physico-Mechanical Properties

Physico-mechanical properties and antimicrobial activity of produced antimicrobial PET and PA nonwoven fabrics have been investigated.

In general, nonwoven fabrics are characterized by tensile, burst strengths, elongation -at-break, air permeability, drape-ability, weight, thickness, porosity and debility to heat and chemicals. These properties of prefect fabric composition and structure. Most properties can be determined with standardized test procedures (ASTM).

Table (1): Physico-Mechanical Properties of Antimicrobial PET and PA Nonwoven Fabrics

Test	Test Method		PET Nonwoven fabrics		PA Nonwoven Fabrics
			Denier 9	Denier 15	Denier 14
Mass per unit area (g/m^2)	ASTM D5261		195	181.5	221.5
Free thickness (m.m.)	ASTM D5199		2.54	2.32	2.45
Thickness under pressure 2KN/m^2	ASTM D5199		1.87	1.62	1.88
Fabric density (g/m^3)	ASTM D4914-08		0.077	0.079	0.092
Air-permeability @12.5mm WG (cm^3/cm^2.sec)	ASTM D737-04(2012)		364.2	337.9	402<
Bursting strength (Kpa)	ASTM D5199		800.0	736.5	965.0
Apparent opining size (micron)	ASTM D4751		360	350	500.0
Tear strength (N)	ASTM D2261		240	210	300
Tensile Strength (KN/m)	ASTM D5034-09(2013)	XMD	0.295	0.19	0.295
		CD	0.355	0.23	0.340

XMD: Machines Direction CD: Crosswise Direction

Table (1) shows the physico-mechanical properties of antimicrobial PET and PA nonwoven fabrics manufactured by needle punching technique. As the produced fabric samples are intended to be used for air filtration, the rate of air- permeability is of prime interest. The range of air- permeability mentioned in table (1) is quite enough to cover the needed air capacity that any filter media may need to cover according to the literature. The values of pore size are much higher than the normal values of filter media used for the same purpose in the market. However, the targeted value of porosity could be reached by a multi-layer structure. The values of mechanical properties such as: bursting strength, tensile strength and elongation at break are within the range of market filter element and are adequate to resist the mechanical stresses when used in the air stream.

2- Antimicrobial Activity

The antimicrobial activities of produced on pilot scales PET and PA nonwoven fabrics were determined by diffusion and shake flask methods (Tables 2 and 3). The activity by diffusion is quantified by measurement of millimeters of the width of the zones of inhibition around the sample. The activity by shake flask method was determined as a % of CFU reduction. Based on the data listed in the above mentioned tables one can conclude the following:

1- PET Nonwoven Fabric bounded with AS showed high antimicrobial activity against *B.m, E.c* and *C.a* In fact the inhibition zones for finished PET samples are significant, whereas it is null for the unfinished ones (Table 2). The data given in tables also indicate that, washing durability of such fabrics exceeds more than 5 Launder-Ometer washes. The PET nonwoven fabrics could still provide more than 83% bacterial reduction against *B.m, E.c* and *C.a* (Table 2).

2- PA-6 nonwoven fabrics are characterized before washing, by quite strong biocide effects on the above mentioned microbes (Table 3). This was demonstrated by quite large zones of stunned microorganism growth. tables 3 also show that washing durability of the biological functions exceeds more than 5 Launder Ometer washes. The finished nylon-6 nonwoven fabrics could still provide more than 88.7%, 85.8% and 94.5% (Table 3) of its antimicrobial activity against *B. mycoides, E. coli*, and *C. albicans*.

Table (2): Antimicrobial Activity of PET Nonwoven fabrics, Determined by Diffusion and Shake Flask Methods

No	Nonwoven Fabric	Inhibition Zone Diameter (mm) * in Case of Tested Microbes			% CFU Reduction*		
		B. m	*E. C*	*C. a*	*B. m*	*E. C*	*C. a*
1	PET (D15) Blank	0.0	0.0	0.0	0.0	0.0	0.0
2	PET Hydrolyzed**	0.0	0.0	0.0	0.0	0.0	0.0
3	PET (D15) Hydrolyzed + Treated with AS	18	17	14	-	-	-
4	PET (D15) Hydrolyzed + Treated with AS + Washed	15	13	13	83.0	83.3	90.1

Pilot Scale Production Conditions:
Alkaline Hydrolysis:
[NaOH], 2.0 mol/l; 60 minutes; Temperature, 100^{o}C; M: L, 1:7.
Treatment with AS:
[AS], 2.0 % (OWF); Time, 60 minutes; Temperature, 90^{o}C; M: L, 1:7.
(D15): Denier, 15
* After 5 Times Launder- Ometer washings; AATCC Test Method (61 –1989).
** Carboxylic Content: 11.0 meq/100g fabrics which is equivalent to 3.17 mg AS/g fibers.

Table (3): Antimicrobial Activity of PA nonwoven Fabrics, Determined by Diffusion and Shake Flask Methods

Fibers	Inhibition Zone Diameter (mm) * in Case of Tested Microbes			% CFU Reduction*		
	B. m	*E. C*	*C. a*	*B. m*	*E. C*	*C. a*
PA Blank (D 14) [(a)]	0	0	0	.0	0.0	0.0
PA Blank Treated	29	28	18	-	-	-
PA Blank Treated with AS + Washed [(b)]	25	23	17	88.7	85.8	94.5

Pilot Scale Production Conditions:
Treatment with AS:
[AS], 2% (OWF); Time, 60 minutes; Temperature, 90^{o}C; M: L, 1:7.
(a) Carboxylic content, 22.8 meq/100gr fibers which is equivalent to 3.67 mg AS/g fibers.
(b) *After 5 Times Launder- Ometer washings; AATCC Test Method (61 – 1989).

3- Assessment the Application of Antimicrobial Nonwoven Fabrics in Air Conditioning

We have mentioned before that, the use of nonwoven in medical community has generally expanded in recent years. Technology increasingly requires room or work areas free from microbe, making it necessary to filter air in industrial activities. Pure air is essential in operating the pharmaceutical laboratories, clean rooms for electronics, museums, libraries and food industries. Based on the above mentioned it is of great importance to assess the applicability of antimicrobial PET and PA nonwoven fabrics as filters in air conditioning sector.

Tables 4-6 illustrate the results obtained after the filtration of air using the abovementioned antimicrobial nonwoven fabrics. It is important to mention that *Gram negative* bacteria lack the resistance to the environmental stress due to their cellular structure, especially their phospholoipid membrane [14] and its sensitively to solar radiation and other environmental stress [15]. Gram negative bacteria mainly present when a direct source is found. According to that, in our results there is no presence of Gram negative due to their sensitivity and absence of source near the working place.

Based on the data listed in tables (4, 5, and 6) one can conclude the following:

1. Antimicrobial PET and PA nonwoven fabrics seem to have high efficiency in decreasing microbes (Table 4). This efficiency ranged from 71.0 % to 68.9 %, respectively (Table 5).
2. The efficiency of Filtration in case of fungi reached 59.0 % and 45% in case of PET and Nylon-6, respectively (Table 5).
3. The identification, concentration and percentages of airborne fungi show that, PET and PA nonwoven fabrics are efficient in the filtration of *Aspergillas*. Concerning the other isolated airborne fungi, the efficiency of the filters ranged from 5% to 26% or completely undetectable (Table 6).

Table (4): Effect of the Nature of Nonwoven Filter on the Residual Concentrations of Total Airborne Bacteria and Airborne Fungi (CFU/m^3)

No	Nonwoven Fabrics	Denier of Fiber (D)	Total Airborne Bacteria (CFU/m^3)	Total Airborne Fungi (CFU/m^3)
1	Ambient air	-	1442.3	254.4
2	PET	9	413.4	156.1
3			425.8	88.9
4		15	399.3	113.1
5			420.5	58.9
6	PA-6	14	464.1	143.1
7			431.1	137.2

Table (5): Percentages of CFU Reduction of Total Airborne Bacteria and Airborne Fungi (CFU/m^3) in Ambient Air After Filtration Using Antimicrobial PET and PA Nonwoven Fabrics

No	Nonwoven Fabrics	Denier of Fiber (D)	Total Airborne Bacteria (CFU/m^3)	Total Airborne Fungi (CFU/m^3)
	PET	9	71.3	38.6
2			70.5	65.0
3		15	72.3	55.5
4			70.8	76.8
5	PA-6	14	67.8	43.8
6			70.1	46.1

Table (6): Identification, Concentrations, and Percentages of the Isolated Airborne Fungi

No	Nonwoven Fabrics	Denier of Fiber (D)	*Aspergillus*	
			CFU/m^3	%
1	Ambient Air	-	160.18	-
2	PET	9	64.7	59.6
3			53.0	66.9
4		15	130.7	18.4
5			58.8	63.3
6	PA-6	14	94.2	41.2
7			88.9	44.5

Effect of the Filter Thickness

In order to increase the efficiency of filters under investigation, the number of layers was increased to two instead of one (Tables 7-8). It was found that doubling the layer leads to significant improvement in filtration efficiency not only with microbes (98 %) but also with fungi (62.3 – 72.9%).

Table (7): Effect of Filter Thickness of Nonwoven Fabrics on Concentrations of Total Airborne Bacteria and Airborne Fungi (CFU/m^3)

No	Nonwoven Fabrics	Denier of Fiber (D)	Number of Filter Layers	Total Airborne Bacteria (CFU/m^3)	Airborne Fungi (CFU/m^3)
1.	Airborne	-	-	1442.3	173.7
2.	PET	15	1	399.3	113.1
			2	29.4	47.1
3.	PA-6	14	1	464.1	143.1
			2	23.6	65.4

Table (8): Effect of the Number of Filter Layers on % of CFU Reduction of Total Airborne Bacteria and Airborne Fungi (CFU/m^3) in Ambient Air After Filtration Using Antimicrobial PET and PA Nonwoven Fabrics

No	Nonwoven Fabrics	Denier of Fiber (D)	Number of Filter Layers	% CFU Reduction in:	
				Total Airborne Bacteria	Airborne Fungi
1.	PET	15	1	72.5	34.9
			2	97.9	72.9
2.	PA-6	14	1	67.0	27.6
			2	98.0	62.3

4- Assessment the Application of Antimicrobial PET and PA Nonwoven Fabrics in Medical Sector

A surgical mask, is intended to be worn by health professionals during surgery and during nursing to catch the bacteria shed in liquid droplets and aerosols from the wearer's mouth and nose. They are not designed to protect the wearer from inhaling airborne bacteria or virus particles and are less effective than respirators, masks which provide better protection due to their material, shape and tight seal. Surgical masks are popularly worn by the general public in many countries to reduce the chance of spreading airborne diseases.

The treated nonwoven fabrics have been utilized as a functional part in different designed shapes of face masks. All treated nonwoven samples were thermally treated to enhance surface side’s integrity such as: material thickness and air-permeability required to achieve best comfort properties for the mask wearer.

Different profiles and shapes were selected using the treated nonwoven samples as inter-lining to design the face mask:

1- Rectangular with sharp edges.

2- Rectangular with round corners.

3- Circular shaped masks.

400 pieces of such shaped masks were sewn using a simple sewing machine to combine different parts together. The sample masks were designed with three corrugations of both outer sides in opposite directions in order to ease entire stretching of the mask to fit with face area without deformation and prevent leakage around the edges when the users inhale.

Efficiency of Masks in Removing Bacteria and Fungi Aerosols

A number of questions have been raised regarding the use of respirators against biological agents. The primary question is which the particulate size can respirator remove? taking in mind that, biological particles may be suspended individually or associated with other particles.

According to the data listed in tables (9 - 10) we can conclude that:

1. Concentrations of airborne bacteria and fungi were significantly reduced by respirators (masks), ~50-80%.
2. The variations in efficiency of removal are attributed to: Filtration, Microorganism survival on the filter, potential reaerosolization of the bioaerosol.
3. Interpretation of microbial air quality from the particle size point of view is important as smaller particles are more likely to penetrate respiratory system.
4. Generally coarse particles were significantly removed, because bacteria are mainly attached to particulate matter/or present as aggregates (Schulzet al., 2011) [16], and fungal spores are individually found in the environment and aerodynamic diameters of most fungal spores ranged between 2 and 8 µm (Yamamoto et al., 2012) [17].
5. Masks under investigation showed significant removing of airborne microorganisms, higher for removing bacteria than fungi.

Table (9): Airborne Bacterial Concentrations (CFU/m^3) Before and After Using Different Types of Masks and The Efficiency (%) of Masks Under Investigation in Removing Airborne Bacteria

Mask Type	Airborne Bacterial Concentration (CFU/m^3)						% CFU Reduction of Bacteria		
	Before (C_o) Filtration			After (C_i) Filtration					
	Coarse	Fine	Total	Coarse	Fine	Total	Coarse	Fine	Total
Non-treated	592	1130	1722	330	948	1278	44.3	16.0	25.8
PET Physically Treated with AS	449	667	1116	42	474	516	90.9	28.9	53.8
PET Chemically Treated with AS	397	859	1256	88	476	564	77.9	44.6	55.1

Table (10): Airborne Fungal Concentrations (CFU/m^3) Before and After Using Different Types of Masks and The Efficiency (%) of Masks Under Investigation in Removing Airborne Fungi

Mask Type	Airborne Fungal Concentration (CFU/m^3)						% CFU Reduction of Fungi		
	Before (C_o) Filtration			After (C_i) Filtration					
	Coarse	Fine	Total	Coarse	Fine	Total	Coarse	Fine	Total
Non-treated	25	193	218	15	150	175	40	22	20
PET Physically Treated with AS	23	136	159	5	61	66	79	55	58
PET Chemically Treated with AS	28	244	272	6	120	126	79	55	53

Estimation of the Antimicrobial Activity of Masks

Table 11 shows the estimated Zones of Inhibition (Cm) of *Bacillus subtilis* and *Staphylococcus aureus* Obtained with antimicrobial PET and PA nonwoven fabrics used as surgical masks in medical sector.

Table (11): Zones of Inhibition (Cm) of *Bacillus subtilis* and *Staphylococcus aureus* Obtained with Different Types of Masks

Mask types	*Bacillus subtilis*	*Staphylococcus aureus*
Non-treated	0.0	0.0
PET Physically Treated with AS	1.7	1.73
PET Chemically Treated with AS	1.85	1.53

CONCLUSION

The technological process for the production of PET and PA nonwoven fabrics having durable antimicrobial properties on pilot scale was carried out. The hydrolyzed PET and regular PA fibers were finished with antimicrobial substance (AS) on pilot scale. The produced PET and PA antimicrobial fibers were converted into nonwoven fabrics by using mechanical needle punched process. The antimicrobial properties of nonwoven PET and PA were investigated. The produced nonwoven fabrics appeared high antimicrobial activity even after five washing cycles. The physico-mechanical properties of the produced nonwoven fabrics were measured. The evaluation of PET and PA nonwoven fabrics as air filters and surgical masks showed their applicability in these fields.

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