

Chemical Fixation of Antimicrobial Substance within Polyester and Polyamide-6 Fibers

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Abstract

This work is aimed at preparing polyester (PET) and polyamide-6 (PA) fibers having antimicrobial properties by using simple and easily applicable method on pilot scale. To achieve this, PET fibers were activated by alkali hydrolysis for creation of carboxylic groups in the chains of PET macromolecules. The hydrolyzed PET and regular Nylon-6 fibers were finished with antimicrobial substance (AS) (quaternary ammonium salt under trade name katamine). Different factors (AS concentration, treatment temperature and duration) affecting the antimicrobial properties were investigated in order to find the optimal finishing conditions. The finished fibers have been converted into nonwoven fabrics and the effect of resin finishing on their antimicrobial activity was studied. The results revealed that the dilution of resin emulsion up to 12.5% by water paves the way for obtaining finished nonwoven fabrics having antimicrobial activity equal to that for unfinished ones.

Keywords: PET, Regular PA-6 Fibers, Alkali Hydrolysis, Antimicrobial Finishing, Resin Finishing.

INTRODUCTION

Over the last few years attention has been directed toward the imparting of the antimicrobial activities to polyester and polyamide fibers for increasing its fields of applications. The manufacture of bioactive polyester and polyamide fibers or of articles from them can be accomplished through:

1. Physical fixation of the antimicrobial substrates within the fine structure of polymers via addition of bioactive reagent to the polymer chips before fiber formation, or by impregnation of the fibers or the fabrics in bactericidal solutions [1-5].

2. Chemical bonding of the antimicrobial substances with textile materials. This can be achieved by modification of such materials for increasing the content or creating on the fibers new functional groups which are able to react with the bactericidal agents [6-20].

It is important to mention that most of the several attempts which were carried out for loading antimicrobial substances onto polyester and polyamide fibers and fabrics cannot be applied on industrial scale for imparting antimicrobial properties.

Textile materials are produced in the form of woven and nonwoven fabrics. Polyester and polyamide nonwoven fabrics are of considerable use in textile and other industries. Therefore, there is a requirement for the antimicrobial finishing of such textile articles is highly needed for a wide range of different applications.

The ultimate goal of this study is to produce antimicrobial polyester and polyamide nonwoven fabrics on pilot scale. In order to achieve this, the adaptation and development of laboratory conditions which have been, previously, set up [18-21] for imparting polyester and polyamide textile materials antimicrobial properties is highly requested for establishment of local technological package for the production of antimicrobial polyester and nylon nonwoven fabrics on pilot scale.

EXPREMENTAL WORK

Materials

- Polyester fibers used throughout this project were kindly supplied by Polyester company, Kafr El-Dowar, Egypt. The fibers were scoured at 80°C for 45 min. with solution containing 2 g/L nonionic detergent, washed with water, squeezed, and finally air dried.
- Nylon-6 fibers used throughout this project were kindly supplied by El Nasr Company, Elmeahalla, Egypt. The fibers were scoured using the same method applied for scouring polyester fibers.
- Antimicrobial substance (AS) under trade name " katamine " was in the form of 50% aqueous solution (Russia).
- NaOH (A.R grade).
- Resin under trade name APOMUL DR was kindly supplied by Egypt Tex Co.
- 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.
- Alkaline hydrolysis of PET fibers was carried out using the method described by shalaby et al [22]. 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 [23] 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 50:1. 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.

- The initial and final concentrations of AS solutions were determined by Recording Spectrophotometer (UV-Vis, 2401 Shimadzu) at $\lambda_{\text{max}} = 208 \text{ nm}$.
- The salt uptake on hydrolyzed PET and Nylon-6 fibers (SUT) was calculated as follows:
- $\text{SUT (g./g. fiber)} = [V(C_1 \times C_2) / W]$, where C_1 and C_2 are the concentrations (g/L) of AS solutions before and after treatments, respectively, V is the volume of AS solutions (cm^3) used in treatment, and W is the weight of fibers.
- Finishing efficiency of hydrolyzed PET and Nylon-6 fibers with AS solutions (FE%) was calculated as follows: $\text{FE(\%)} = [A/B] \times 100$, where A is the salt uptake on the fiber samples (mg/g. Fiber), and B is the amount of salt equivalent to the carboxylic content (mg/g. fiber) in fibers before treatment.

Analysis:

- Antimicrobial potency of PET and Nylon-6 fibers was quantified by diffusion [24] and by shake flask Method [25].
- Carboxylic content was determined according to the method described in [26].

RESULTS AND DISCUSSION

1. Chemical Fixation of AS within PET Fibers

1.1. Activation of PET Fibers

Surface activation of PET fibers with alkali before reaction with antimicrobial agents is essential for creation of additional carboxylic groups in PET macromolecules[21]. So, it is necessary to discover a suitable weight loss, which paves the way for acquiring polyester fibers better antimicrobial performance.

Based on the data tabulated in table 1 one can conclude that, the alkaline treatment under the following conditions: [NaOH], 1.5-2.0 mol/l, Reaction time, 45-60 min and Reaction Temperature, 90-100°C, pave the way for creating additional carboxylic groups in PET macromolecule. The amounts of these additional groups are found to be suitable for carrying out the antimicrobial finishing of PET fibers.

Table (1): Effect of Treatment Conditions of PET Fibers with Aqueous Solutions of NaOH on Weight Loss %

Temperature (°C)	Time(min.)	Weight Loss% (WL) at [NaOH], mol/l		
		1.0	1.5	2.0
80	30	1.5	2.1	2.9
	45	2.1	2.5	4.9
	60	2.4	3.8	6.4
	90	3.5	4.8	8.6
90	30	2.5	4.2	7.4
	45	3.8	6.2	10.5
	60	4.2	7.7	13.2
	90	5.7	10.8	18.7
100	30	4.5	5.4	15.2
	45	5.9	9.8	20.9
	60	7.2	14.0	23.2
	90	11.4	21.3	35.8

Material : Liquor Ratio (M:L) = 1:25.

1.2. Finishing of Activated PET Fibers with AS

In this article experiments were carried out to explore an efficient method for imparting PET fibers antimicrobial properties on bench scale. So, the previously partial hydrolyzed polyester fibers were introduced into thermostated aqueous solution containing AS. The suggested finishing method was carried out at suitable temperature and duration. It was found that there was a salt uptake loaded on the polyester fibers. A part of this salt was bonded to PET fibers even after five washing cycles with H₂O. The presence of fixed amount of salt on fibers is, mainly, due to the inclusion of the AS within PET fibers. This is in full agreement with our previous conclusions listed in [23].

1.2.1. Factors Affecting the Antimicrobial finishing of PET Fibers

The effect of treatment conditions, mainly AS concentration, treatment time and temperature, and the weight loss percent, on the salt uptake (SUT) and finishing efficiency (FE) was evaluated. The results in table 2 showed that:

- 1- The salt uptake on the fibers and finishing efficiency increased with increasing AS concentration up to 2.0 % (OWF). The increase in the concentration of AS above 2.0 % in the finishing solution did not practically, affect the salt uptake. This means that, a concentration of AS equal to 2% (OWF) in reaction medium is the optimum concentration for obtaining maximum salt uptake and finishing efficiency.

- 2- Carrying out the finishing process for 30 minutes led to a detectable salt increase in PET fibers, irrespective of the percentage of the weight loss. Further increase of the duration up to 60 minutes is accompanied by a noticeable increase in SUT and FE.
- 3- Increasing the finishing temperature from 70°C to 90°C led to increase in the salt uptake. The quantity of salt uptake was more detectable at finishing temperature close to the glassy transition temperature (T_g) of polyester fibers. The higher exhaustion rates of AS was attained at 90°C. So that, the optimum finishing temperature was chosen as 90°C.
- 4- No significant increase was noticed neither in the salt uptake nor in the antimicrobial potency of the finished PET samples with increasing the weight loss % from 10 to 20 %. Based on the obtained results a weight Loss of 10 % is quiet enough for imparting high biological activity to PET fibers.

Table (2): Effect of Treatment Conditions of PET Fibers with AS on salt uptake (SUT) and Finishing Efficiency (FE)

Treatment Conditions	SUT (mg/g. Fiber)		FE (%)	
	W.L (%)		W.L (%)	
	10*	20**	10*	20**
[AS] ^(a) % (OWF)				
1	1.00	1.26	28.0	23.0
2	1.26	2.00	35.3	36.4
3	1.21	1.87	33.9	34.1
Treatment Time (min) ^b				
30	0.67	0.98	18.8	17.9
45	0.81	1.66	22.3	30.3
60	1.26	2.00	35.3	36.4
Treatment Temperature ^(c) (°C)				
70	0.98	1.68	27.5	30.6
80	1.20	1.86	33.6	33.9
90	1.26	2.00	35.3	36.4

Treatment Conditions:

(a) pH, 11; Treatment Temperature, 90°C; Treatment Time, 60 min.; M: L, 1:25.

(b) pH, 11; [AS], 2% (OWF); Treatment Temperature, 90°C; M: L, 1:25.

(c) pH, 11; [AS], 2% (OWF); Treatment Time, 60 min.; M: L, 1:25.

Carboxylic content:

*23.3 (meq/100gr fibers) which is equivalent to 3.57 mg AS/g fibers.

**35.8 (meq/100gr fibers) which is equivalent to 5.49 mg AS/g fibers.

1.3. Antimicrobial Properties of Finished PET Fibers

The percentages of colony forming unites (%CFU) of hydrolyzed and finished with AS PET fibers against *B. m*, *E. c* and *C. a* was evaluated using the shake flask method. The obtained results are listed in table 3. These data are pointed to the following:

- 1- Polyester fibers finished with antimicrobial substance have outstanding antimicrobial efficiency against the abovementioned three microorganisms. Actually the % of CFU reduction for all hydrolyzed and finished polyester fibers are significant, while, the parent or unhydrolyzed PET fibers have no activity.
- 2- The increase of weight loss % from 10 to 20, practically, did not affect the antimicrobial activity of the tested fibers against the above mentioned microorganisms.

Table (3): Effect of Weight Loss (%) on Antimicrobial Activity of Hydrolyzed and Treated with AS PET Fibers

Tested Fibers	Weight Loss (%)	% CFU Reduction		
		<i>B. mycoides</i>	<i>E. coli</i>	<i>C. albicans</i>
PET (Blank)	0	0	0	0
PET→H→ T (AS)	10	98.0	98.4	96.0
	15	98.0	98.4	96.5
	20	98.5	98.1	96.5

Treatment Conditions:

[AS], 2% (OWF); pH, 11; Treatment Temperature, 90°C; Treatment Time, 60 min.; M:L, 1:25. *PET→H: Hydrolyzed polyester fibers

2. Chemical Fixation of AS within Nylon-6 Fibers

In our previous study [27] an effective chemical pretreatment method has been developed for imparting durable antimicrobial properties to Nylon-6 fibers. The results showed that, both regular and hydrolyzed fibers acquired appropriate antimicrobial activity. Therefore, this study is based on a direct antimicrobial treatment of regular nylon-6 fibers.

2.1. Finishing of Nylon-6 Fibers with AS

Regular Nylon-6 fibers were finished with AS aqueous solution of AS at temperature higher than the glassy transition temperature (T_g) of the nylon polymer. The treatment was proceed for the desired reaction time. It was found that some amount of antimicrobial salt are existing on the surface of fibers. A part of this salt was still existing on nylon fibers even after five washing cycles with H_2O . Nylon-6 fibers have carboxylic end groups which have the ability to react, in alkaline medium, with cationic groups in AS with forming carboxylate ions [18]. Stemming from this, the existing after extraction with water salt in fibers is pointed to chemical interaction between antimicrobial substance and nylon-6. This is in full agreement with our previous conclusions listed in [21, 23, 28].

2.2. Factors Affecting the Antimicrobial finishing of Nylon-6 Fibers

Factors that affect the antimicrobial treatment of Nylon-6 fibers with AS on bench scale were investigated:

- 1- Table 4 shows the dependence of the salt uptake estimated on Nylon-6 fibers and the finishing efficiency (FE) with AS concentration. As seen in table 4 both of the salt uptake on the fibers and reaction efficiency enhanced with increasing the AS concentration from 1% to 3% (OWF). It should be noted that, at low AS concentration from 1% to 2% (OWF) the SUT and FE continue to increase with increasing AS concentration. Further increase in concentration i.e. from 2% to 3% has a little effect on both salt uptake and reaction efficiency. This indicates that the AS concentration of 2% (OWF) seems to be the optimum concentration for imparting maximum bioactivity to Nylon-6 fibers.
- 2- The relation between the duration of finishing and the salt uptake and reaction efficiency is tabulated in Table 4. It is clear that the both values increase steadily with the increase in the reaction time. Moreover, at 45 min the increase in both salt uptake and reaction efficiency is significant. Further increasing in the reaction time (60 min) is accompanied by additional increase in the above mentioned properties. Based on the results listed in table 4 one can conclude that, 60 minutes is the optimum treatment time for obtaining Nylon-6 fibers with higher bioactivity.
- 3- The results in Table 4 also revealed that, both values were increased as the temperature increased from 70°C to 90°C . At 90°C these properties have reached its higher values under the chosen conditions.

Table (4): Effect of Treatment Conditions of Nylon-6* Fibers with AS on Salt Uptake (SUT) and Finishing efficiency (FE)

Treatment Conditions	SUT (mg/g fibers)	FE (%)
[AS] ^(a) % (OWF)		
1	1.28	36.7
2	1.76	50.4
3	1.79	51.3
Treatment Time (min) ^(b)		
30	0.79	22.3
45	1.45	41.5
60	1.79	56.3
Treatment Temperature °C ^(c)		
70	1.41	40.4
80	1.68	48.1
90	1.79	56.3

Treatment Conditions:

(a) pH, 11; Treatment Temperature, 90°C; Treatment Time, 60 min;
M: L, 1:25.

(b) pH, 11; [AS], 2% (OWF); Treatment Temperature, 90°C; M: L, 1:25.

(c) pH, 11; [AS], 2% (OWF); Treatment Time, 60 min.; M: L, 1:25.

*Carboxylic content: 22.8 (meq/100g fibers) which is equivalent to
3.49 mg AS/g fibers.

3. Effect of Resin Finishing on Antimicrobial Activity Of PET and Nylon Nonwoven Fabrics

PET and nylon-6 antimicrobial fibers can be used in the form of woven or/and nonwoven fabrics. These fabrics are of considerable use in textile and other industries. In recent years the nonwoven fabrics are increasingly used in different areas. Therefore, the resin finishing of such fabrics seems to be of great importance.

The resin used in this study was under trade name APOMUL DR which belongs to polyvinyl acetate emulsion, especially designed to be used as a multipurpose adhesive and textile stiffening as recommended by the supplier.

Antimicrobial PET and nylon-6 nonwoven fabrics produced on pilot scale were dipping in finishing mixture containing different concentrations of resin APOMUL

DR, and squeezed to a pick up 80% (OWF). These samples were dried at 100°C for 3 min., and cured at 130°C for 3 min. Finally the antimicrobial activity of finished with resin PET and Nylon-6 nonwoven fabrics were determined (tables 5-8).

Based on the data listed in the above mentioned tables it can be conclude that:

1. Antimicrobial activity of PET and Nylon-6 nonwoven fabrics depends, mainly, on the concentration of resin in finishing emulsion.
2. The application of concentrated resin emulsion (50%) in finishing process leads to a drastic decrease in the values of inhibition zone diameter, and, consequently, in the antimicrobial activity of nonwoven PET (tables 5 and 6) and Nylon-6 (tables 7 and 8) finished fabrics. The PET provide only 44,4%, 47,1% and 42.9% of its antimicrobial activity for unfinished fabrics against *B. m*, *E. c* and *C. a*, respectively. This compared with 65.9%, 65.5%, and 66.7% in case of Nylon-6 nonwoven fabrics (table 8).
3. The continuous decrease in resin concentration by addition of water to the finishing mixtures leads to progressive increase in antimicrobial activity of finished PET and Nylon-6 nonwoven fabrics.
4. It seems that the dilution of resin emulsion up to 12.5% by water paves the way for obtaining finished nonwoven fabrics having antimicrobial activity equal to that for unfinished nonwoven fabrics.

Table (5): Effect of Resin (R) Finishing on Antimicrobial Activity of Polyester Nonwoven Fabrics, Using Diffusion Method

No.	Nonwoven Fabrics	Finishing Mixture (Resin Emulsion/ Water) part/part	Inhibition Zone Diameter (mm) in case of tested microbes		
			<i>B. mycoides</i>	<i>E. Coli</i>	<i>C. albicans</i>
1	PET (Blank)	Without	- ve	- ve	- ve
2	PET→ T(AS)	Without	18	17	14
3	PET→ T(AS)	100/0	8	8	13
4		75/25	15	13	11
5		50/50	16	16	12
6		25/75	18	17	14

Resin Finishing Conditions:

Resin, APOMUL DR; [Resin], 50%; Pick-Up, 80%; Drying Temperature, 100°C; Drying Time, 3min.; Curing Temperature, 130°C; Curing Time 3 min.

Table (6): Effect of Resin (R) Finishing on Antimicrobial Activity of Polyester Nonwoven Fabrics, Using Shake Flask Method

No.	Nonwoven Fabrics	Finishing Mixture (Resin Emulsion/ Water) part/part	Antimicrobial Activity (%)		
			<i>B. mycooides</i>	<i>E. Coli</i>	<i>C. albicans</i>
1	PET (Blank)	Without	-ve	-ve	-ve
2	PET→T(AS)	Without	100.0	100.0	100.0
3	PET→T(AS)	100/0	44.4	47.1	42.9
4		75/25	83.3	76.5	78.6
5		50/50	88.9	94.1	85.7
6		25/75	100.0	100.0	100.0

Resin Finishing Conditions:

Resin, APOMUL DR; [Resin], 50%; Pick-Up, 80%; Drying Temperature, 100°C; Drying Time, 3min.; Curing Temperature, 130°C; Curing Time 3 min.

Table (7): Effect of Resin (R) Finishing on Antimicrobial Activity of Nylon-6 Nonwoven Fabrics, Using Diffusion Method

No.	Nonwoven Fabrics	Finishing Mixture (Resin Emulsion/ Water) (part/part)	Inhibition Zone Diameter (mm) in case of testes microbes		
			<i>B. mycooides</i>	<i>E. Coli</i>	<i>C. albicans</i>
1	Nylon-6 (Blank)	Without	-ve	-ve	-ve
2	Nylon-6→ T (AS)	Without	29	28	18
3	Nylon-6→ T (AS)	100/0	19	19	19
4		75/25	24	22	14
5		50/50	28	27	17
6		25/75	29	28	18

Resin Finishing Conditions:

Resin, APOMUL DR; [Resin], 50%; Pick-Up, 80%; Drying Temperature, 100°C; Drying Time, 3min.; Curing Temperature, 130°C; Curing Time 3.0 min.

Table (8): Effect of Resin (R) Finishing on Antimicrobial Activity of Nylon-6 Nonwoven Fabrics, Using Shake Flask Method

No.	Nonwoven Fabrics	Finishing Mixture (Resin Emulsion/ Water) part/part	Antimicrobial Activity (%)		
			<i>B. mycooides</i>	<i>E. Coli</i>	<i>C. albicans</i>
1	Nylon-6 (Blank)	Without	-ve	-ve	-ve
2	Nylon-6→ T (AS)	Without	100.0	100.0	100.0
3	Nylon-6→ T (AS)	100/0	65.5	65.5	66.7
4		75/25	82.8	78.6	77.8
5		50/50	96.6	96.4	94.4
6		25/75	100.0	100.0	100.0

Resin Finishing Conditions:

Resin, APOMUL DR; [Resin], 50%; Pick-Up, 80%; Drying Temperature, 100°C; Drying Time, 3min.; Curing Temperature, 130°C; Curing Time 3.0 min.

CONCLUSION

The adaptation and development of laboratory conditions for establishment know-how for the production of antimicrobial PET and PA-6 fibers on pilot scale were carried out. PET fibers were activated by alkali hydrolysis under the following conditions: [NaOH], 1.5-2.0 mol/l, Reaction time, 45-60 min and Reaction Temperature, 90-100°C in order to create carboxylic groups in PET macromolecules. The hydrolyzed PET and regular PA fibers were finished with antimicrobial substance (quaternary ammonium salt). It was concluded that, a weight Loss of 10 % is quiet enough for imparting high antimicrobial properties to PET fibers. The optimal finishing conditions were determined for both two types of fibers. Moreover, the effect of resin finishing on the produced antimicrobial PET and Nylon-6 nonwoven fabrics on pilot scale was studied. The results revealed that the dilution of resin emulsion up to 12.5% by water paves the way for obtaining finished nonwoven fabrics having antimicrobial activity.

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