Bioactive Nano-Composite Multifilament Yarns

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ABSTRACT
Physical, mechanical and antimicrobial properties of nylon 6 (polycaprolactam) doped with different amounts of silver nanoparticles were investigated in this study. Two series of filament yarns counts (20 and 70 Denier) were produced by melt spinning method with different amounts of silver nanoparticles contents (0, 0.5, 1 and 4 wt%). Elemental analysis of silver and titanium dioxide (present in polymer as delustering agent) was carried out by energy dispersive X-ray (EDX) and inductively coupled plasma mass spectrometry (ICP-MS) methods.

Tensile testing, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and differential scanning calorimetry (DSC) were used to characterize the yarns. Antimicrobial activities were quantitatively evaluated against Escherichia Coli (gram-negative) and Staphylococcus Aureus (gram-positive) bacteria. The doped nylon 6 fibers showed a well dispersed distribution of silver nano-particles. Yarns with 0.5-1.0 wt% of silver nanoparticle content were found to have improved physical and mechanical properties, as well as, significant antimicrobial activity.

Keywords: Bioactive; Nano-composite; Multifilament Yarn; Melt-Spinning; Antimicrobial; Nano-silver; Nylon 6

INTRODUCTION

The intrinsic properties of textiles provide suitable growth environments for microorganisms, especially in wet and warm environment. The growth of microorganisms on textiles inflicts a range of unwanted effects both on textiles (unpleasant odor, stains and discoloration) and the persons who wear them (cross infection by pathogens) [1-4].

Methods for incorporating antimicrobial agents in polymeric materials (like organic fibers) include surface coating, surface absorption, and melt blending.

Surface coating is a physical method in which the antimicrobial agent settles on the surface of polymeric materials. Vapor, sputter, and ion beam deposition methods are included in this category. This method generally suffers from many demerits such as poor adhesion, unevenness of deposited films on substrates, and difficulty in processing [5-8].

Surface absorption is based on the intrinsic tendency (electrostatic attraction) of antimicrobial agents toward the polymer surface. Surface absorption is an acceptable method when it is merged with other wet processes such as dyeing. Otherwise, it is a time and energy consumable process [9-13].

Introducing the antimicrobial agents inside a polymer matrix via melt blending is an efficient method [14-17]. The main advantages of this method are ease of process, durability and cost effectiveness [3].

Among the various antimicrobials agents used for producing antimicrobial textiles, silver has unique properties such as high thermal stability, low volatility and long-term activity. It is capable of killing 650 types of pathogens microorganism [16] and show little toxicity to mammalian cells and tissues. Consequently silver has been used in the textile industry for many years [18, 19].

The antimicrobial property of silver depends on its particle size. On the nanoparticle scale, this provides larger surface area and high dispersion ability (for better contact with microorganisms); thus higher degrees of antimicrobial activity are expected. These nanoparticles get attached to the denature bacteria Ribonucleic acid (RNA), and Deoxyribonucleic acid (DNA), thus preventing their replication [20-22].

Nylon 6 (poly caprolactam by ring opening polymerization) is one of the most successful commercial synthetic semi crystalline polymers. It has outstanding physical properties and high resistance to a wide spectrum of fuels, oils and chemicals. Consequently, it has been used in a wide

variety of applications including tire reinforcement cords, ropes, fishing lines, sport rackets, dental floss, and carpets. Due to its excellent elastic properties, it is used for socks, woman stocking, under wear, and health socks where antimicrobial activity plays an important role [23, 24].

In this research, bioactive nylon 6/silver nano-composite multifilament yarns were made by melt spinning. This method is easy, rapid, cost effective and friendly to environment for preparing polymer/silver nano-composite yarns on a large scale without using any solvents or carriers. It provides high durability because the active agent is physically embedded in the structure of the fiber polymer matrix and will be released slowly during usage. The majority of studies on polymer/silver nano-composite have focused on molded bulk materials. In this study, the nano-composite in its yarn form was considered for experimental work, and yarn characteristics and properties were investigated.

EXPERIMENTAL

Materials

Silver powder (spherically shape) with an average particle size of less than 10 nm was used for master-batch production. The specifications of nylon 6 granules for producing the master-batch and multifilament yarns are given in *Table I*.

TABLE I. Specifications of used nylon 6 granules.

Type of nylon 6 granules	Application	TiO_2 content (%)	*MFI (g/10 min)
I	master-batch production	0.03	48
II	nylon 6 multifilament yarns spinning	0.3	53

* melt flow index

Melt Spinning Process

Two similar series of yarn counts (20 and 70 denier containing three different numbers of filaments 7, 17, & 34) with four different amounts of silver nano-particles content (0, 0.5, 1 and 4 wt %) were spun by an industrial melt spinning apparatus.

The conventional nylon 6 pellets (containing 0.3% TiO_2 and melt flow index = 53g/10 min) and prepared master-batch pellets were introduced into the spinning apparatus equipped with an accurate master-batch dozing system. The heating profile of the screw was regulated between 260 °C and 265 °C and winding speed of 4100 m/min. The spin pack had five layers of filters with mesh number of 50/250/400/325/250. The produce fibers had round cross-sections and were classified as shown in *Table II*.

TABLE II. Classification of melt –spun fibers

Group No.	Sample Code	Number of Filament	Silver Content (%)
Group I (70 Denier)	*PA70-17-0	17	0
	PA70-34-0	34	0
	PA70-17-0.5	17	0.5
	PA70-34-0.5	34	0.5
	PA70-17-1	17	1
	PA70-34-1	34	1
	PA70-17-4	17	4
	PA70-34-4	34	4
Group II (20 Denier)	PA20-17-0	17	0
	PA20-7-0	7	0
	PA20-17-0.5	17	0.5
	PA20-7-0.5	7	0.5
	PA20-17-1	17	1
	PA20-7-1	7	1
	PA20-17-4	17	4
	PA20-7-4	7	4

*PA- Denier- No. of Filament- Silver Content

Scanning Electron Microscopy (SEM)

Melt-spun fibers were sputter coated with gold under vacuum for 5 minutes and visualized by SEM.

Elemental Analysis

Silver and titanium dioxide (TiO_2) contents of produced samples were measured by energy dispersive X-ray (EDX) spectroscopy, and inductively coupled plasma mass (ICP) spectroscopy. For ICP sample preparation, 0.5 gram of sample yarn was cured in electrical furnace at 600 °C (T_m silver: 961.78 °C, T_m Nylon 6: 215 °C) to remove the organic materials then the obtained ash acidified with nitric acid. The prepared solution was heated to 70-80 °C for 30-45 minute, and diluted to 50 ml for analyzing.

Transmission Electron Microscopy (TEM)

All filament samples were ultra-microtomed with a diamond knife at room temperature to generate sections with a nominal thickness of 100nm. The sections were transferred to 400 mesh Cu grids. Bright-field transmission electron microscopy (TEM) images of nylon 6/nano-silver nano-composite filament yarns were obtained at 120 kV under low-close conditions.

Thermal Analysis

Differential scanning calorimeter (DSC) was carried out under nitrogen flow (50 ml/min) in a pierced aluminum pan (ca.8mg sample) from 0 to 270 °C (T_g nylon 6: 53 ºC, T_m nylon 6: 215 ºC). The heating rate

was 10 °C /min, the sample was held at 270 °C for 2 minutes in order to remove their thermal history, and then cooled down to 0 °C at a cooling rate of 10 °C /min. Thermograms were evaluated by means of Proteus software. DSC curves were developed in a standard arrangement, showing the heat flow as a function of the sample temperature. For each sample, the crystallization temperature (T_C), heat of fusion (ΔH_f) and the percentage of crystallinity were obtained from the thermogram, the area of endothermic peak and through the Eq. (1), respectively:

$$\text{Crystallinity } (\%) = (\Delta H_f / \Delta H^0_f . w_f)\ 100 \qquad (1)$$

Where ΔH_f and W_f are the heat of fusion and the weight fraction of nylon 6 fibers, respectively and ΔH^0_f is the extrapolated value of the enthalpy corresponding to the heat of fusion of 100% crystalline nylon 6 taken as 190 j/g from the literature [25].

Mechanical Properties

Mechanical properties of the produced yarns were determined by a tensile testing apparatus. Testing speed and gauge length were chosen 5 cm/min and 20 cm, respectively. For each sample 15 tests were carried out. Analysis of variance (ANOVA) was used for determining the nano-silver effects on mechanical properties, and Duncan and Tukey's tests were applied for categorizing and grouping different samples.

Evaluation Of Antimicrobial Efficiency

A quantitative antimicrobial test method; the AATCC 100-1989 was used in this study for evaluating the antimicrobial activity of the produced samples [26]. Antimicrobial tests were performed with two types of bacteria: gram-positive bacterium (Staphylococcus aureus, ATCC 6538) and gram-negative bacterium (Escherichia coli, ATCC 25922). The initial count of cultured bacteria was 10^7 for both bacteria. The cultured bacteria were inoculated with both the control nylon 6 sample and the samples incorporating various silver contents.

First samples were inoculated with Escherichia Coli (gram-negative) and Staphylococcus Aureus (gram-positive) microbes. Then they were incubated in sealed jars for 24 hours. The grown bacteria on the fabric were collected by eluting and the eluted solution placed on the nutrient agar plates and after 24 hours incubation, the antimicrobial properties of sample obtained through Eq. (2):

Percent reduction of bacteria

$$(\%) = ((A-B)/A)\ 100 \qquad (2)$$

Where A and B are the numbers of bacteria on the pristine and the silver containing samples after 24 hours, respectively.

RESULTS AND DISCUSSION

Morphology

Figure 1 shows the morphology of nylon 6/silver nano-composite multifilament yarns containing 0, 0.5, 1 and 4wt% of silver content in equal denier and number of filaments. Those nanoparticles of silver which are present on the surface of samples are observable in SEM micrographs. The average particle size of silver nanoparticles is about 60 nm, and it implies that probably silver nanoparticles agglomerate due to the interaction forces between them. This phenomenon is more obvious in samples with higher amounts of silver content [17 and 27]. Besides, polymers containing more than 1wt% nano-silver show other problems such as breakage of yarn, excessive pressure behind filter and leakage of spin-pack.

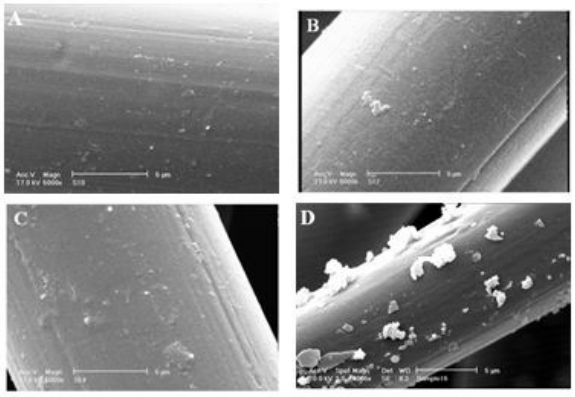

FIGURE 1. SEM micrographs of samples PA20-7-0 (A), PA20-7-0.5 (B), PA20-7-1 (C) and PA20-7-4 (D)

Figure 2 shows the TEM images of the nylon 6/silver nano-composite multifilament yarns containing 4wt% silver. The silver nano-particles are present on the fiber surface and also within the polymer composite. They are nearly spherical shape with diameters averaging about 60 nm. This is consistent with afore mentioned SEM results. The silver particles however tend to become irregular and non-spherical, which is attributed to the massive silver migration and aggregation. Probably, these incidents are largely caused by the instability of silver atoms due to their high surface free energy, and their aggregation would produce more thermodynamically stable particles.

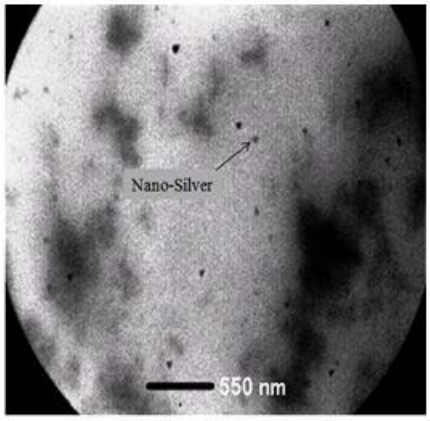

FIGURE 2. TEM image of sample PA70-17-4

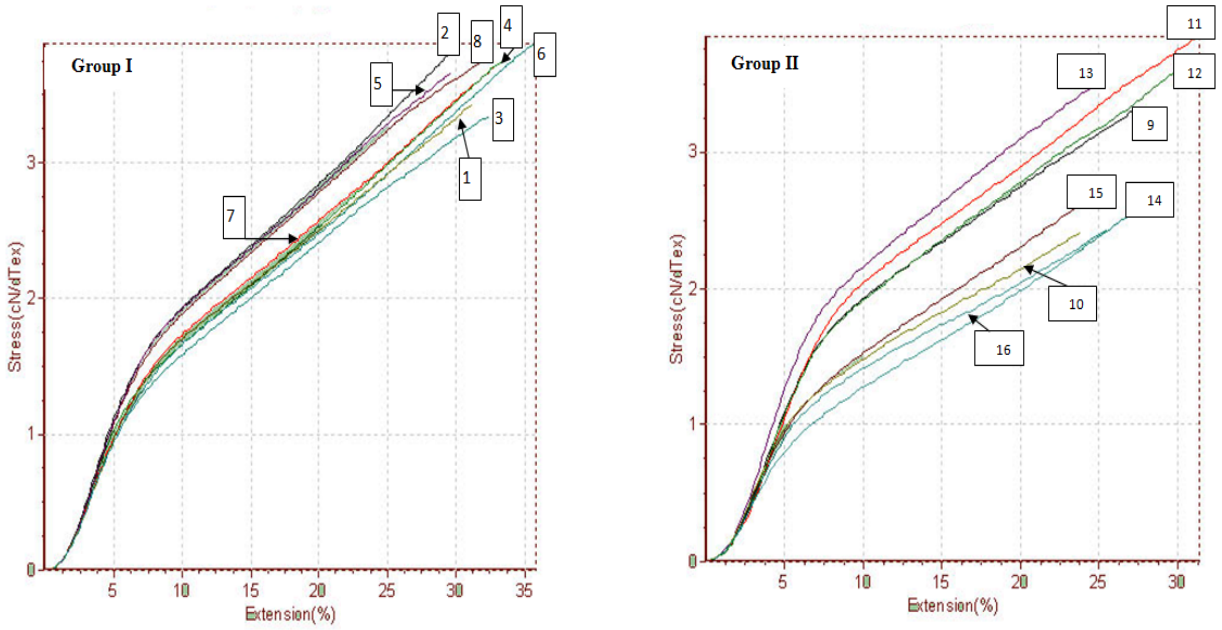

FIGURE 3: Stress-Strain behavior of (A) Group I and (B) Group II
Group I: 1- PA70-17-0; 2-PA70-34-0; 3- PA70-17-0.5; 4- PA70-34-0.5; 5-PA70-17-1; 6- PA70-34-1; 7-PA70-17-4; 8-PA70-34-4
Group II: 9-PA20-17-0; 10-PA20-7-0; 11-PA20-17-0.5; 12-PA20-7-0.5; 13-PA20-17-1; 14-PA20-7-1; 15-PA20-17-4;16-PA20-7-4

Stress-Strain Behavior

Essentially, antimicrobial processes should not negatively affect the quality (physical properties) or appearance of the textiles. *Figure 3* shows the stress-strain curves of various nylon 6/nano-silver multifilament yarns containing 0, 0.5, 1 and 4 wt% of silver. The pure nylon 6 yarns used in the tensile test as the reference, was subjected to the same sample preparation conditions as the other yarn samples were.

The values of investigated mechanical properties of nano-composite multifilament yarns are summarized in *Table III*. Based on standard deviation of samples, finer sample yarns (Group II) show more unevenness in comparison with other samples (Group I) which leads to show lower investigated mechanical properties values. Furthermore, inside of each groups with increasing the number of filaments the samples show more evenness.

One way analysis of variance (*Table IV*) proved that there are significant differences between yarn samples.

Following Duncan and Tukey tests show that adding silver nano-particles up to 1wt% did not change the investigated mechanical properties. But adding 4wt%

nano-silver content decreased mechanical properties significantly. This may be due to agglomeration of nano-particles in high concentrations and these agglomerations can act as stress concentration centers during tensile testing and hence the mechanical properties of the matrix [17, 28 and 29].

These negative effects of silver nano-particles in high concentration on some mechanical properties of bioactive nano-composite multifilament yarns are confirmed by DSC results which indicate decreasing of crystallinity with increasing the nano-silver content of fiber polymer matrix.

Thermal Analysis

The crystallization behavior of nylon 6/nano-silver multifilament yarns was examined using the differential scanning calorimetry (DSC) method. *Figure 4* displays DSC thermogram curves of the nylon 6 fibers containing different amounts of silver. The heating scans were used to determine the melting behavior of the fiber such as the melting temperature (T_m) and the heat of fusion (ΔH_f).

TABLE III. Mechanical properties of control and modified Fibers.

Group No.	Sample	Elongation at Break (%)	Std. Deviation	Work of rupture (cN/Tex)	Std. Deviation	Tenacity (cN/Tex)	Std. Deviation
Group I (70 Denier)	PA70-17-0	34.4	1.6	5.7	0.9	38.1	3.4
	PA70-34-0	38.4	2.0	6.1	0.5	37.4	2.1
	PA70-17-0.5	37.3	2.6	5.8	0.9	36.8	4.1
	PA70-34-0.5	38.4	1.6	6.0	0.6	37.7	2.9
	PA70-17-1	33.7	1.9	5.4	0.5	35.9	2.2
	PA70-34-1	41.4	2.2	6.5	1.0	38.6	4.0
	PA70-17-4	37.1	2.4	5.3	1.0	34.5	4.2
	PA70-34-4	37.4	1.2	5.3	0.6	33.4	2.5
Group II (20 Denier)	PA20-17-0	32.2	3.5	4.4	0.8	30.9	2.6
	PA20-7-0	28.1	5.9	3.2	1.3	25.2	5.5
	PA20-17-0.5	33.7	2.5	5.7	0.5	35.7	2.0
	PA20-7-0.5	34.0	3.8	5.5	1.4	35.6	6.1
	PA20-17-1	29.0	2.2	4.4	0.8	32.7	3.6
	PA20-7-1	31.8	4.9	3.5	1.4	25.5	6.3
	PA20-17-4	27.7	2.8	2.9	0.7	23.8	3.4
	PA20-7-4	29.0	4.9	3.3	1.2	24.4	5.7

TABLE IV. ANOVA results (Group II)

		DF	sum of square	Mean square	F value	sig
ELONGATION	Between Groups	7	676.4	96.6	6.0	.000
	Within Groups	112	1793.5	16.0		
	Total	119	2469.9			
TENACITY	Between Groups	7	2831.4	404.5	18.4	.000
	Within Groups	112	2462.4	22.0		
	Total	119	5293.8			
WORK OF RUPTURE	Between Groups	7	117.9	16.8	14.5	.000
	Within Groups	112	129.8	1.1		
	Total	119	247.7			

From *Table V* it can be concluded that the incorporation of the silver nano-particles decreases the crystallization temperature (T_C), heat of fusion (ΔH_f) and crystallinity. The reduction of crystallinity is due to the silver nano-particles interfering with the crystallization process of nylon 6, because they acted on a type of impurity in the nylon 6 matrix [15]. On the other hand the silver nano-particles did not act as nucleating agents in the polymer matrix and decelerated the crystallization rate of the polymer. It is also concluded that aggregation prevented crystal growth, and hence, reduced the crystallinity as the nano-silver content increased. Many types of filler are inert, they do not interact with the polymer segments and their coefficient of thermal expansion is different from polymer matrix. As a result, they can be sources of stresses during cooling [30].

TABLE V. DSC heating and cooling results of neat and silver- nylon 6 fibers.

Sample Code	DSC		
	T_C (℃)	ΔH_f (J/g)	Crystallinity (%)
PA20-17-0	188.9	73.09	38.46
PA20-17-0.5	183.2	62.49	32.88
PA20-17-1	186.6	68.94	36.28
PA20-17-4	188.0	70.64	37.17

T_C: crystallization temperature; and ΔH_f: heat of fusion .

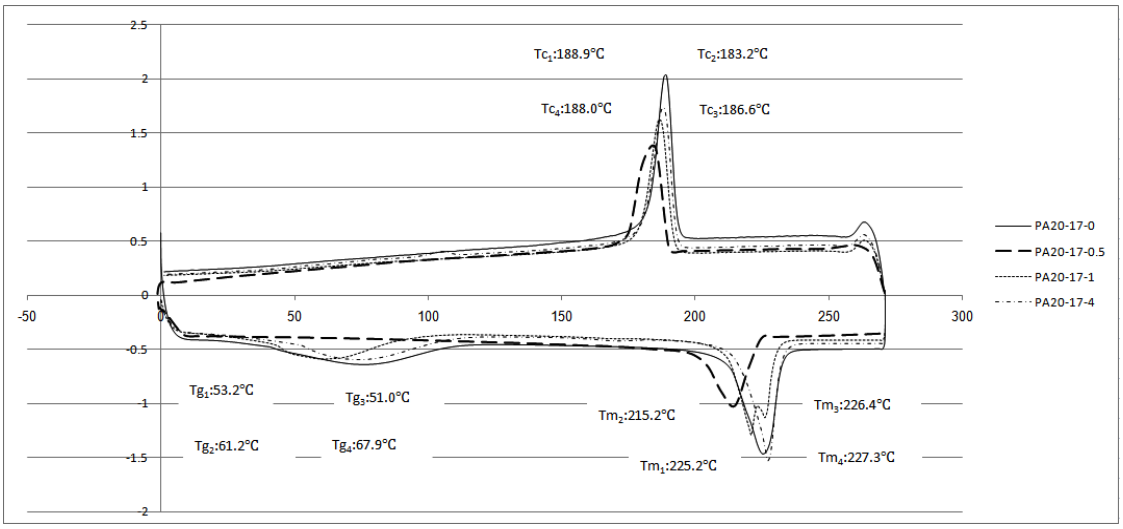

FIGURE 4. DSC, heating and cooling curves of neat nylon 6 and silver-nylon 6 fibers.

Silver And Titanium Dioxide Content

The existence of silver and titanium dioxide (TiO_2) on fibers was confirmed by EDX and ICP-MS which it was shown in *Table VI*.

The discrepancies between these two quantitative methods is related to the sample preparation method of ICP-MS. In which the sample completely will be solubilized in appropriate solvent, so all present nanoparticles in the specimen will be released. But EDX method just measures the particles percent up to 500 nm depth on the surface of a solid substrate [31].

Antimicrobial Efficiency

Table VII shows the antimicrobial properties of pristine samples (0 wt% nano-silver content) and samples with different amounts of nano-silver. The compounds containing maximum amounts of silver nano-particles however exhibited almost perfect reduction against both kinds of bacteria. This is related to high amounts of silver in the surface and silver ion release. These findings are in agreement with the other results [17, 19 and 32].

Furthermore it was shown that the release of silver ions has an inverse relation to composite thickness Eq. (3),

$$\frac{M_t}{M_\infty} = 4\sqrt{\frac{Dt}{\pi d^2}} \quad [33, 34] \qquad (3)$$

Where M_t/M_∞ is the fractional silver release, t, D and d are the release time, diffusion coefficient and sample thickness, respectively [33, 34]. It can now be concluded that finer fibers (Group II, 20 Denier) have higher antimicrobial potency compared to coarse fibers (Group I, 70 Denier).

In the case of samples with the same denier (70) and a different number of filaments, such as PA70-17-1 and PA70-34-1, it was observed that the one with fewer filaments exhibited more antimicrobial effect. This is again related to sample thickness.

Silver ions are generated from elemental silver particles only in the presence of water. Many factors

will influence the silver ion release of substrate including silver powder concentration, specific surface area of the silver particles, the nature of filler, water uptake, soaking time, water concentration in the matrix, composite morphology, changes in the physical state of the composite specimen as a result of the water diffusion, crosslinking and degree of crystallinity [18-19, 32, 35-38].

It was shown that there exists a relationship between the silver ion release amounts and the antimicrobial properties of nano-silver/polymer composites. A steady and prolonged release of the silver biocide in a concentration level (0.1 ppb) is capable of rendering an acceptable antimicrobial efficacy [39].

TABLE VI. Silver (Ag) and titanium dioxide (TiO_2) amounts in silver-nylon 6 Fibers.

Group No.	Sample Code	EDX (Ag,TiO_2)		ICP(Ag,TiO_2)	
		Ag (%)	TiO_2 (%)	Ag(ppm)	TiO_2(ppm)
Group I (70 Denier)	PA70-17-0	-	0.67	-	
	PA70-34-0	-	0.93	-	
	PA70-17-0.5	0.94	0.61	0.13761	1.4422
	PA70-34-0.5	0.40	0.44	0.38710	3.1049
	PA70-17-1	1.12	0.46	0.25496	1.0909
	PA70-34-1	1.04	0.31	0.44187	1.0135
	PA70-17-4	1.65	0.50	0.55496	1.9695
	PA70-34-4	1.31	0.59	0.54455	0.45558
Group II (20 Denier)	PA20-17-0	-	0.68	-	
	PA20-7-0	-	0.54	-	
	PA20-17-0.5	1.46	0.76	0.072553	2.0430
	PA20-7-0.5	1.88	0.47	0.038929	1.1076
	PA20-17-1	1.66	0.53	0.086277	0.90427
	PA20-7-1	1.77	0.34	0.21697	1.3517
	PA20-17-4	2.19	0.32	0.36250	5.2086
	PA20-7-4	2.74	0.58	0.31495	3.4975

The antimicrobial activity of silver is dependent on the silver cation, which binds strongly to electron donor groups in biological molecules containing sulphur, oxygen or nitrogen. Hence the silver-based antimicrobial polymers have to release the silver ion to a pathogenic environment in order to be effective. The oxidation of the metallic silver to the active species silver ion is possible through an interaction of the silver with the water molecules [18].

TABLE VII. Antimicrobial Results.

Group No.	Sample Code	Antimicrobial	
		Escherichia Coli	Staphylococcus Aureus
Group I	PA70-17-0	0%	0%
	PA70-34-0	0%	0%
	PA70-17-0.5	95%	93%
	PA70-34-0.5		
	PA70-17-1	90%	82%
	PA70-34-1	80%	60%
	PA70-17-4	-	-
	PA70-34-4	88%	80%
Group II	PA20-17-0	0%	0%
	PA20-7-0	0%	0%
	PA20-17-0.5	99.6%	99.6%
	PA20-7-0.5		
	PA20-17-1	98.4%	97.4%
	PA20-7-1		
	PA20-17-4	100%	100%
	PA20-7-4		

CONCLUSION

Bioactive nylon 6/nano-silver nano-composite multifilament yarns have been successfully prepared by an industrial melt spinning apparatus. SEM micrographs show that produced multifilament yarns with 0.5-1.0 wt% silver content exhibit a finer and more uniform morphology. From DSC results, it was concluded that the silver nano-particles hardly exert any acceleration effect on the crystallization of nylon 6, because by increasing the silver content in nylon 6 fibers, the heat of fusion, crystallization temperature, and crystallinity decreased. Fibers containing 0.5-1.0 wt% of silver seem to be more desirable because they have higher elongation, elasticity and modulus compared to the fibers containing a higher silver concentration. By an increase in silver content (≥1wt %), the fibers lose strength and toughness. An increase in the amount of silver causes a decrease in elongation and toughness which is due to the existence of weak structure in fibers. Also by increasing silver content (≥1wt %), the fibers become yellow. This change of color is attributed to an agglomeration of nano-silver particles and a plasmon effect. The samples showed good antimicrobial efficiency against gram-positive and gram-negative bacteria. Nano-composite multifilament yarns containing maximum amounts of silver nano-particles exhibited perfect reduction against both kinds of bacteria but in this case agglomeration was formed which caused breakage of yarns and leakage in the spinning process.

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