



Characterization of PET fibers incorporated with bacteria spores

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ABSTRACT

The demand for high-tech textiles with special functionalities is currently increasing. This has led to the continuous effort to modify conventional polymeric textile materials like Polyethylene terephthalate (PET). Previous studies have proved that bacteria spores can be incorporated in PET fibers during melt extrusion. However, the effects of extruding spores in the fibers on the resulting fiber's tensile properties have not been studied deeply. In this work, tensile tester, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and optical microscopy (OM) were used to characterize the properties of PET/spores fibers. Results indicated that tensile strength, Young's modulus and elongation at break were dependent on spore concentration. Nevertheless, the properties of the resulting fibers were found to be as of same tensile quality as normal PET fibers. © 2015 Trade Science Inc. - INDIA

KEYWORDS

Bacteria spores;
PET fibers;
Extrusion;
Tensile properties.

INTRODUCTION

With the current switch from conventional use of textile to use of textile with additional superior functionalities, the textile industry has witnessed a tremendous increase in demand for functional textile materials which can basically be defined as materials tailored and engineered to give specific properties that are suitable for a given application^[1]. Due to this current demand, efforts have been made to transform the traditional textile materials by incorporating different additives to add new values or functionalities or to improve the chemical, physical and biological properties. This has resulted to production of innovative high performance textile materials for various specific end use which include

personal protective equipment (PPE), medical applications and packaging^[1, 2]. One of the traditional textile materials that are being transformed into functional textiles is Poly Ethylene Terephthalate (PET) popularly known as polyester^[3]. Polyester is a long-chain semi-crystalline thermoplastic polymer that is widely produced worldwide due to its superior mechanical properties like high tensile strength, low cost and ease of production as well as its ability to be recycled^[4-6]. Up to date, many functional additives have been incorporated into PET polymer among them being nucleating and antimicrobial additives, enzymes, colorants, cross-linking as well as matting agents^[7-10]. In most cases, functionalizing additives are usually applied on the material surface as a post-treatment process. This means that the

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additives are physically or chemically bound on the material surface^[10, 11]. The disadvantage with this technique is that resistance to abrasion and washing is never guaranteed as well as some finishing methods like spraying can be detrimental to the environment due to spills during spraying^[11, 12]. Additionally, poorly applied coatings may lead to partial loss during use, care and storage^[12]. On the contrary, applying functionalizing additives during fiber production can entrap the additive in the polymer matrix during solidification of the fiber forming polymer and may take part in the crystallization process resulting in a strong bond between the polymer and additives as well as providing high resistance to abrasion^[4, 11]. Moreover, due to the porous nature of the fibers, the functionalizing additive can slowly migrate to the surface acting as a slow release mechanism that can result to an extended period of additive activity^[13]. This means that the release rate will be influenced by the physical and chemical characteristics of the polymer in relation to the characteristics of the additive^[14, 15]. A recent study demonstrated that bacteria spores can be incorporated in PET fibers during extrusion^[16]. However, the extent to which the spores can be incorporated without fundamentally changing the properties of PET fibers needs further investigation. The present study looks into the tensile properties of PET fibers with incorporated spores. This is aimed at checking if the produced fibers will be different from normal PET fibers in terms of their tensile properties. The PET fibers incorporated with spores (0%, 2%, 4% and 6%) were extruded in a single screw extruder. The survival of PET was first tested and later spores-PET interaction and dispersion was studied with Scanning electron microscopy (SEM), Transmission electron microscopy (TEM) and optical microscopy (OM). Afterwards, the tensile properties like tensile strength, Young's modulus and elongation at break were studied on Favimat tensile fiber testing machine.

EXPERIMENTAL

Extrusion

The PET pellets were initially dried in an oven for two days at 70°C^[17] after which pure bacteria

spores were added by gravimetric dosing during spinning to obtain a 0 (control), 2, 4 and 6% concentration of spores to PET polymer. This mixture was then extruded into multi-filament fibers by a single screw extruder (General extrusion technology, China). The three barrel heating zones were set to 270°C for the feed stock (transport of material), 270°C for plasticizing (compression) and 275°C for pumping (metering). The die was heated at 275°C; the pressure was around 6.5 Mpa while the average residence time was approximately 4 minutes. The resulting fibers had an average linear density of about 7.5dtex.

Characterization methods

Survival tests

The resistance of the extruded spores was determined by the growth/germination biological assay as explained by Ciera *et al*^[16]. The samples were sterilized by subsequently soaking first in a sodium hypochlorite solution, followed by a Dettol solution and finally in an ethanol solution for 10 minutes each, where after they were inoculated on Nutrient Agar plates and incubated at 40°C for 24 hours. Finally, the plates were visually checked for colony forming spots^[16].

Tensile testing

The tensile properties, like tensile strength, modulus and elongation at break of the resulting extruded spores/PET fibers were determined with the FAVIMAT-ROBOT (Textechno, Germany). All samples were kept in a conditioned room of 55% humidity and a temperature of 20°C for 24 hours before testing. An average of 50 single filaments was tested for each spore concentration level. The two-sample Kolmogorov-Smirnov statistical test was used to determine how significantly the spores affected the tensile properties at all the spore concentration level (0%, 2%, 4% and 6%).

RESULTS AND DISCUSSION

Survival tests

Dormant bacteria spores are more resistant to extreme environmental conditions than their vegeta-

tive cell counterparts. Among the factors that contribute to the spores resistant includes saturation of the spore chromosome with a group of small, acid-soluble proteins (SASP) of the a/b type, low water content in the spore protoplast/core, decreased spore permeability, high levels of minerals in the spore core, etc.^[18-21]. The survival test results of spores extruded in PET fibers is shown in Figure 1. The control sample of pure PET fibers with no spores (Figure 1a) shows no growth around the fibers, while the PET fibers extruded with 4% spores shows bacterial growth all around the fibers (Figure 1b). The growth observed in Figure 1b confirms that spores survived the extreme melt extrusion parameters (High temperature, pressure, shear stress and residence time). As explained by previous studies, the resistance of spores to extreme melt extrusion conditions could have been due to low operating pressure, short residence time and possible agglomeration of spores in the extruder^[16-22].

Morphological structure of PET incorporated with bacteria spores

The morphological properties of the fibers were studied using an optical microscope, a scanning electron microscope and a transmission electron microscope.

Scanning electron microscopy (SEM)

The SEM micrograph of PET fibers incorporated with 4% spores showed visible spore agglomerates

on the surface (Figure 2). Two main factors may have caused the agglomeration of spores in the polymer matrix. Firstly, the high temperature and shear stress which are the main process parameters during extrusion. Spores can form agglomerates in suspensions during heat treatment due to increased surface hydrophobicity caused by denaturing of the protein coats^[21, 23].

On the other hand, force originating from shear stress possibly impacted a higher energy on the spores which overcame the spores' electrostatic repulsive force leading to formation of agglomerates. The second factor that possibly caused the agglomeration of spores in the fibers is poor dispersion bond between the spores and the polymer matrix influenced by spore size and loading. In most cases, small sized particles tend to agglomerate more than larger particles while chances of forming agglomerates increases with loading^[14, 27].

Optical microscopy (OM)

The optical micrographs of PET-fibers extruded with 4% spores are characterized by cracks parallel to the fiber axis (Figure 3). These cracks suggest that spores may have been heterogeneously distributed throughout the fiber. These cracks could have been caused by the observed agglomerates of spores that formed in the polymer matrix (Figure 2). Additionally, the cracks can also be associated with decreased macromolecule arrangement of PET along the fiber axis possibly caused by incorporating the

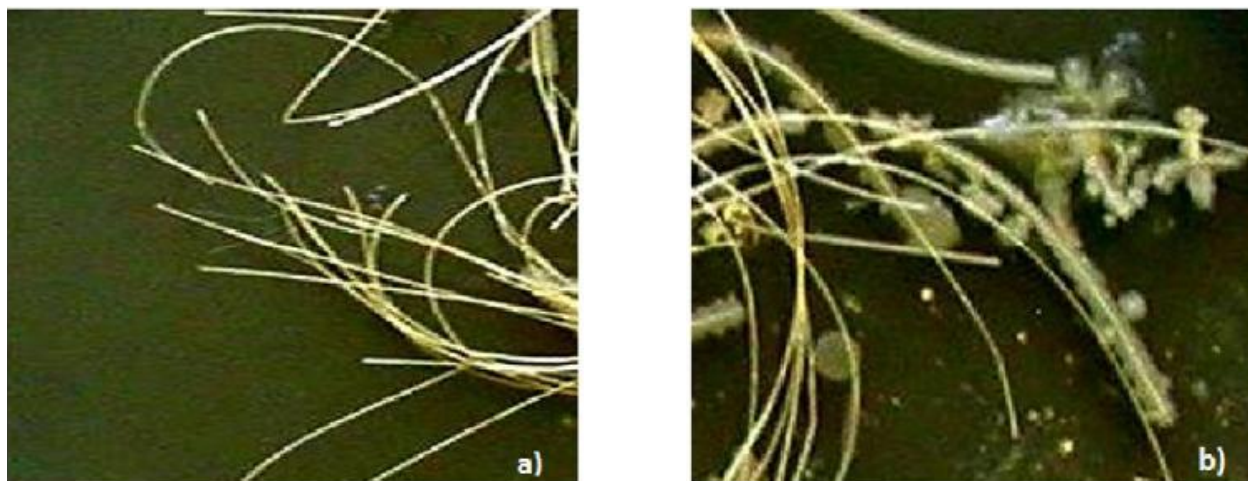


Figure 1 : Viability tests (PET fibers inoculated in nutrient medium and incubated for 24 h at 40°C; a) Control PET fibers with no spores showing no growth, b) PET fibers extruded with 4% spores showing bacterial growth

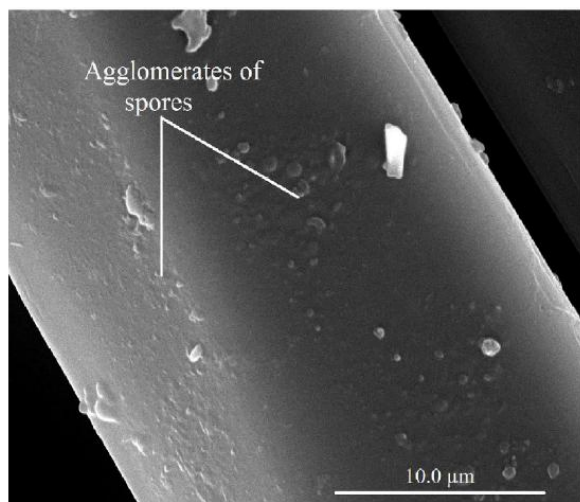


Figure 2 : SEM micrographs for PET incorporated with 4% spores characterized with agglomerates of spores on the fiber surface (arrow showing)

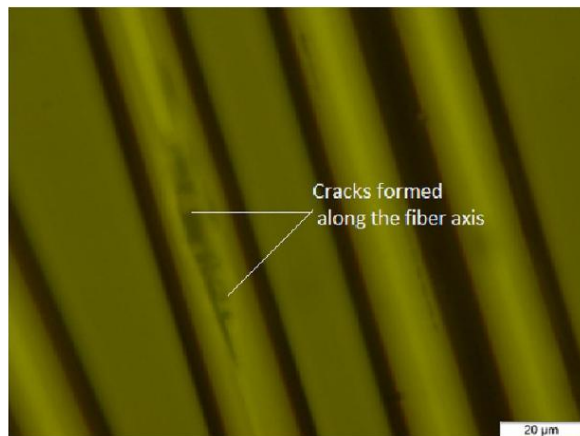


Figure 3 : Representative optical micrographs of PET fibers extruded with 4% spores showing cracks parallel to the fiber length (arrow showing)

spores in the structure^[28, 29]. This suggests poor dispersion of spores in the polymer matrix and poor spore-matrix interface adhesion bonding^[29].

Transmission electron microscopy (TEM)

The TEM microscopy confirmed the presence of spores in the PET fibers. The spores seem to have no preferential orientation in the fibers suggesting a heterogeneous distribution. Moreover, there seems to be a good adhesion bond between the spores and the polymer matrix (Figure 4).

Tensile properties of PET fibers incorporated with bacteria spores

The tensile properties of a textile fiber like the strength, elongation and Young's modulus greatly

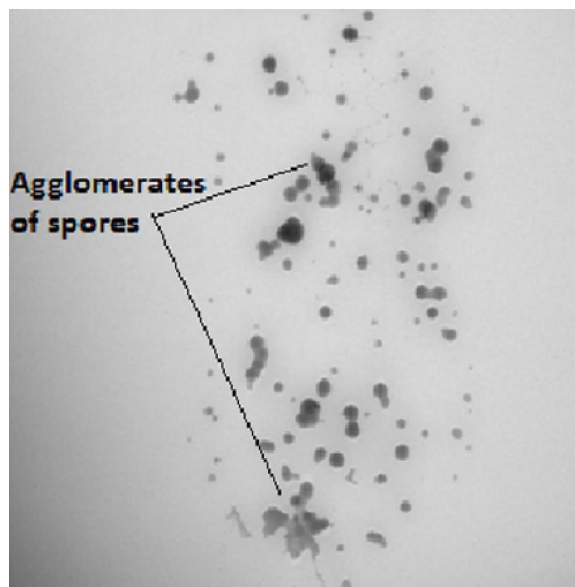


Figure 4 : TEM micrographs for PET fibers incorporated with 4% spores showing agglomerates of spores in the polymer matrix

determines its applications. For this reasons, it is important to develop fibers of a specific quality for a given application. The fibers tensile properties are generally influenced by the processing technique used, type of polymer, additives incorporated, size and distribution of the additives, etc.

A negative trend on the tensile properties can be noted whereby an increase in spore concentration results in a decrease in tensile strength and elongation at break. However, the Young's modulus increased with spore concentration (TABLE 1). This trend can be attributed to the observed spore agglomerates in the polymer matrix.

Spore agglomerates could have induced cracks on the fiber surface, obstructing stress transfer between the spores and the polymer matrix creating weak points in the fibers which resulted in earlier failure^[15, 30-33]. The mechanical properties of the PET fibers extruded with 2-6% spores were compared with the mechanical properties of blank PET fibers (Control). The p-values presented in TABLE 2 indicate that incorporating the bacteria spores in the PET fibers shows a significant effect on tensile strength and Young's modulus at all concentration levels. However, incorporating spores didn't have a significant effect on the young's modulus at all spores concentrations ($p > 0.05$).

Generally, fibers normally have a tensile strength

TABLE 1: The tensile properties of PET fibers incorporated with bacteria spores

Spore content (% w/w)	Tensile properties		
	Tensile strength (cN/dtex)	Young's modulus (cN/dtex)	Elongation at break (%)
0	4.86±0.3	87.74±7.4	50.24±6.9
2	4.26±0.4	88.88±5.8	46.99±5.2
4	4.0±0.1	89.36±6.5	42.90±2.5
6	3.76±0.3	90.97±7.2	40.72±5.2

TABLE 2 : P-values for the tensile properties of PET fibers incorporated with bacteria spores

Spore content (% w/w)	P-Values		
	Tensile strength (cN/dtex)	Young's modulus (cN/dtex)	Elongation at break (%)
2 vs. control	0.0010	0.0001	0.527
4 vs. control	0.0001	0.0001	0.182
6 vs. control	0.0001	0.0001	0.177

of around 3-7 cN/dtex, elongation of about 20-50% and Young's modulus of 77-87 cN/dtex^[24, 29]. Taking these values as the standard, the properties of PET fibers produced in this study are within the acceptable range. However, our fibers have an additional advantage in that functionalizing additives have been incorporated that can give them special properties.

CONCLUSIONS

The tensile strength, modulus and elongation at break were dependent on the spore concentration. The study shows that incorporating spores in PET fibers resulted in decrease of the tensile properties. This decrease in the tensile properties of the resulting PET/spore fibers was associated with the observed spore agglomerates and formed cracks along the fiber axis. However, the resulting properties of the produced fibers were within the acceptable range thus they are of equal quality as normal PET fibers.

Recommendations for future research

Agglomeration of spores and inhomogeneous distribution of spores in the polymer matrix lead to the decrease in the tensile properties of the resulting PET/spore fibers. Therefore, it is important to identify good techniques that can promote homogenous dispersion of spores and that can prevent spores from forming agglomerates. By solving these two problems, the cracks observed along the fiber axis

will be eliminated and there will be efficient load transfer between the spore and the polymer matrix resulting in improved quality of the functionalized fibers.

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