



The Production and Physical and Thermal Characterization of Polybutylene Succinate Multifilament Yarns

Kerim Kılınç¹  0000-0001-7409-0115Esra Karaca²  0000-0003-1777-3977¹Polyteks Textile Company, R&D Center, Bursa, Türkiye²Department of Textile Engineering, Engineering Faculty, Bursa Uludağ University, Bursa, Türkiye**Corresponding Author:** Kerim Kılınç, kerimkilinc16@gmail.com

ABSTRACT

There are significant problems in the production, use and waste management of petroleum-based polymers due to the increasing plastic waste problem, exceeding limit of the greenhouse gas emissions and decreasing fossil resources. The textile sector is the second sector that causes the plastic waste problem after the packaging sector. About 65% of the total yarn produced in the textile industry consists of yarns obtained from petroleum-based polymers. Biopolymers come to the fore in studies carried out within the scope of sustainability philosophies such as using of renewable raw materials, recycling at the end of their life and decomposition without harming the nature. In this study, it is aimed to produce biobased and biodegradable polybutylene succinate (PBS) based polymer into multifilament yarn by melt spinning method and examine the effect of different winding speeds on the textile values of PBS yarns. In this context, multifilament yarns with round cross-sections were produced at four different winding speeds. The linear densities (dtex), elongations at maximum load and tenacities of the produced yarns were determined. The thermal characterization of PBS yarns were carried out by differential scanning calorimetry (DSC). The cross-sectional images of PBS filaments were taken and their diameters were measured, on an optical microscope. The results showed that as the winding speed increased, the elongation and dtex values decreased, while the tenacity and crystallinity values increased. It was determined that PBS filaments have smooth round cross-sections.

1. INTRODUCTION

In recent years, there has been an increasing interest in the use of materials derived from natural renewable resources (bio-based) as well as decomposable into eco-friendly components (biodegradable) for the protection of nature. Green movements, initiatives, regulations and laws emerge in almost every developed country to reduce the volume of solid polymer waste produced by consumers [1]. With the Paris Agreement, it is aimed to strengthen the global socio/economic resilience against the threat of climate change, and reduce greenhouse gas emissions in 2020 and beyond [2]. Sustainability philosophies, production

ideologies and ecosystem efficiency, green chemistry and environmentally friendly engineering should be taken into account for the advancement of complex environmental problems [3-5]. Many important steps have been taken recently to create a more cost-effective and eco-friendly world. The best example of this step is the development of biobased materials [6].

The prefix "bio" in the term "biopolymer" indicates that these polymers are naturally produced from living substances (biobased) and/or are biodegradable [7]. The term biopolymer is used for such as biopolymers made from renewable raw materials (bio-based) and are

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biodegradable, or biopolymers made from renewable raw materials (bio-based) that are nonbiodegradable, or biopolymers made from fossil fuels that are biodegradable [8]. Human beings use only 3.5% of the average 17×10^{10} tons of biomass produced in nature every year [9]. According to the European Bioplastics Conference; biopolymers represent about 1% of the more than 368 million tons of polymers produced annually. Global biopolymer production capacity was 2.11 million tons in 2020 and is expected to increase to approximately 2.87 million tons in 2025. The biopolymer used in the textile industry in 2020 accounts for approximately 12% of the total biopolymer use (241 000 tons) [10]. In 2015, textile products including clothes, carpets, furniture coverings, bags, tarpaulins and similar products constitute an important slice of 14% of total plastic waste with a waste amount of 42 million tons [11, 12].

Due to the increasing global demand for textile products, traditional resources and methods are very limited and can not meet these needs. Studies have reported that textile waste in the European Union constitutes 10% of the total consumption waste and the recycling (1% by weight) and reuse (1% by weight) rates of these textile wastes are almost negligible. It is estimated that this destruction caused by the textile industry will increase by 50% by 2030 [13]. These alarming statistics encourage many researchers to make the textile industry greener in various ways. Resulting in various problems, the downsides of the production and use of petroleum-based synthetic yarns incorporate a high carbon footprint, non-renewable resources and high costs, the transition to greener options is accelerating day by day. Also, some traditional natural-based sources, such as cotton and wool, are not considered a promising solution for replacing synthetic yarns [14].

Polyamides, polyesters and (linear) polyolefins are commonly used polymer materials for melt spinning yarn production [15]. The main point in the melt spinning method is that the polymer can melt below its decomposition temperature [16]. Moisture negatively affects the processability and causes degradation of the

polymer during the extrusion process. Therefore, the drying process of the polymer before extrusion is very important [16]. A significant decrease in molecular weight is observed by hydrolytic degradation (hydrolysis) of the moisture-containing polymer during the extrusion process (in molten state). Hydrolytic degradation generally takes place in polyesters containing ester bonds such as polyethylene terephthalate (PET) or polylactic acid (PLA) [17]. PET is the most widely used polymer for yarn production due to its advantages in end-use properties, production economy, and ease of physical and chemical modification [18,19]. It is known that there are studies and research on yarn production from different biopolymers by melt spinning methods as an alternative to petroleum-based PET. Although there are some studies on yarn production of polybutylene succinate (PBS) by melt spinning method, there are significant deficiencies in the literature and industrial scale production.

PBS is gaining more and more attention due to the advantage of being synthesized from both petroleum and biobased sources as well as its biodegradability. PBS is a linear aliphatic polyester that can be synthesized by melt polycondensation of succinic acid (SA) and butanediol (BDO) (Figure 1) [20]. PBS is a semi-crystalline biopolyester and its mechanical properties such as tensile strength, impact strength and flexibility, transparency and biodegradability depend on the crystal structures and crystallinity contained in the polymer [21]. PBS crystallizes from the melt phase at 70–95°C and forms spherulites. The melting temperature and glass transition temperature of the PBS homopolymer are observed to be around 112–116°C and -44°C, respectively. The actual viscosity of the PBS homopolymer is in the range of 0.14 to 0.64 dl/g, depending on the amount of -OH and -COOH groups [21-25]. PBS biopolymer can be degraded through hydrolytic degradation, enzymatic degradation and biodegradation in environmental conditions such as burying, activated sludge and compost. The biodegradation rate of PBS depends on various parameters such as chemical structure, crystallinity, macroscopic shape of the material and degradation conditions [21].

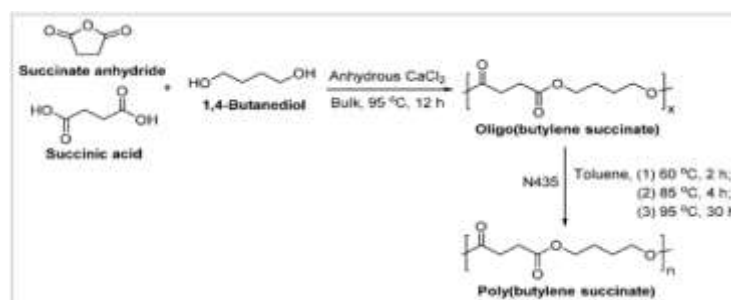


Figure 1. Synthesis of polybutylene succinate [26]

Due to the highly transparent surface and hardness of PBS, it finds use in a wide variety of applications including mulching films, compostable bags, nonwovens and textiles, catering and foams. In some studies, it has been determined that PBS can be widely used in sectors such as agriculture, fisheries, forestry and civil engineering. PBS can also be used in the monofilament, injection molded products and textile industries [27-30]. Due to its biodegradability and biocompatibility, PBS can be used at different points in the biomedical field [31]. Showa HighPolymer, one of the main producers of PBS, expects strong growth in the packaging industry (57.5% of total production), agricultural mulch films (15%) and automotive industry (10%) in 2020 [32].

PBS is used as a raw material in the production of monofilaments for fishing. Mechanical properties such as tensile strength, elongation and softness are important in this application area, and it has been observed that the mechanical properties of PBS monofilament are maximized by being mixed with 5% measured by weight polybutylene adipate-co-terephthalate. In addition, good durability of PBS monofilaments for outdoor use has been demonstrated [33,34]. In the study on PBS homopolymer; bulk continuous filament (BCF) and continuous filament (CF) fibers were obtained while extruder temperatures were stated to be in the range of 200-230oC. The tenacity of CF fibers was obtained in the range of 0,16-0,20 cN/dtex, while the tenacity of BCF fibers was obtained as 0,11-0,22 cN/dtex. It has also been reported that PBS fibers have better characteristics than PLA fibers in terms of crimp values [35].

Studies on filament yarn production from PBS biopolymer have been reported in detail in the patent number WO2014173055A1. In this patent, three different types of yarn were produced partially oriented yarn (POY), highly oriented yarn (HOY) and fully oriented yarn (FDY). There is information about raw material preparation method, running temperature, winding speed, drawing rate and other parameters. However, these parameters are in a very wide range and there is no information about yarn analysis [36]. There are also melt spinning fiber studies made by mixing PBS with other biopolymers at different rates. As an example of this study, multifilament fiber was obtained by mixing PBS with PLA biopolymer in different ratios, and it was concluded that the 90:10 weight ratio of PLA/PBS blended fiber gave the most suitable properties for textile fiber application [37, 38].

In the literature review, it has been observed that there is no research or study on the production of yarn from PBS biopolymer by melt spinning method at high winding speeds, and these studies were carried out by laboratory scale melt spinning machines. In addition, the lack of analysis in terms of textile physics has also attracted attention in these studies.

It was determined that the information such as the number of filaments, cross-section type and cross-sectional image of the PBS yarns were not included in these studies in the literature. In this study, first of all, temperature and time trials at the drying process were carried out to determine its optimum conditions. Later, standard multifilament yarn production was carried out by experimenting with different four winding speeds by keeping other parameters constant on a large-scale pilot machine. Lastly, in the final step, the changes in yarns with increasing winding speed have been investigated in terms of textile physics. Textile values such as linear density, elongation (at Fmax) and tenacity of the produced yarns were obtained by performing analyses, and also cross-sectional images of the filaments were also taken by the optical microscope. The thermal characteristics of the PBS yarns were determined using DSC device. It will be the first study in the literature not only in terms of the scale of the machine used, but also in terms of applied tests and filament information.

2. MATERIAL AND METHOD

2.1 Material

In this study, the thermoplastic polymer used for the melt spinning process was biopolymer PBS chips. The properties of the PBS biopolymer and chips used in this work are presented in Table 1. The commercial biopolyester polybutylene succinate (BioPBS™ FZ78TM) was obtained from PTT MCC Biochem Company Limited (Bangkok, Thailand).

Table 1. Properties of the PBS polymer and chips

Intrinsic viscosity of the chips (IV)	1,5 g/dL
Moisture of the chips	>800 ppm
Melt flow rate of the polymer (MFR)	22 g/10min
Carboxyl end groups (CEG)	25,7 pcs
Density of chips	1,26 g/cm ³
Melting temperature of the polymer (T _m)	115°C
Grain size	70 pcs/gr
Renewable content	%51

2.2 Method

Polyesters have ester bonds, and therefore hydrolytic degradation (hydrolysis) occurs in processes involving high temperature and humidity. Before the melt spinning process, drying trials were carried out at different times and temperatures to reduce the moisture contained in PBS to the lowest levels and to determine the optimum drying conditions. Optimum parameters were determined with 7 different drying trials, which were carried out by keeping the temperature and time constant. The moisture contents of the PBS chips at the drying trials were measured using the Sartorius Moisture Meter MA 100 device according to ASTM D6980-17.

This device is a thermogravimetric moisture meter that features the highest possible measuring accuracy. Polybutylene succinate chips were dried at 80 degrees for 10 hours according to results of the drying trials.

In this study, multifilament yarn production by melt spinning method was made in V&B Set brand large scale pilot spinning machine by using process parameters in Table 2. After the drying process, PBS biopolymer was melted by the extruder at between 200 and 260°C. The molten polymer was delivered to the spin pump which rotates at constant speed and the spin packages by means of pump. The filament formation from molten polymer was carried out using 48-hole and round-section type spinnerets. In the quench cabinets located below the spinnerets, the filaments were quenched with air at 17°C along 120 cm. After the quench process, the spin finish process to the multifilaments was done at certain dosage. The multifilament yarns were produced with 4 different godets and winding speeds given in the Table 3.

Table 2. Melt spinning parameters

Parameters	Values
Spin pump speed	15 rpm
Spinning pressure	75 bar
Spinning temperature	260°C
Extruder temperature (Zone 1-2-3-4)	200-210-220-240°C
Quench air speed	0,55 m/sec
Quench air temperature	17°C
Intermingled pressure	1,5 bar
Spinfinish pump speed	5 rpm

Table 3. The speeds of godets and winder at PBS yarn trials

Godet speed (m/dk)			Winding speed (m/dk)
1	2-3	4	
1000	1005	1010	1015
1500	1505	1510	1515
2000	2005	2010	2015
2800	2805	2810	2815

The cross-section images of PBS filaments were examined for each bobbin produced with different winding speeds by the Olympus BX51 optical microscope. The mean diameter and standard deviation values of ten filaments were calculated by using a special software in the microscope's computer.

Linear density (dtex) of PBS yarns were measured using a winding reel and precision scale according to DIN EN ISO 2060. Tensile properties (elongation at maximum load and tenacity) of PBS yarns were determined by Textechno brand Statimat Me+ model tensile tester according to DIN EN ISO 2062. The analyses were repeated at 5 times for each bobbin produced with different winding speeds. Thermal analysis of PBS multifilament yarns were performed via heating of the samples from 30 to 300°C at 10°C/min under nitrogen atmosphere by using Hitachi 7020 differential scanning calorimeter (DSC) according to

ASTM E 1356-03. The heat of fusion (enthalpy, ΔH) and the melting point (T_m) of the PBS multifilament yarns were measured from the DSC curves and the percentage crystallinity values was determined according to Equation 1 (39, 40).

$$\text{Crystallinity} = \frac{\text{Heat of fusion of the yarn}}{\text{Heat of fusion of the pure crystal for PBS (210 J/g)}}$$

3. RESULTS AND DISCUSSION

3.1. Drying Trials of PBS Chips

The results of the drying trials before melt spinning are given on moisture meter in Table 4. When the results are evaluated; the optimum drying conditions under which the polymer chips can operate without hydrolysis are 80°C for 10 hours that the minimum moisture content remains. Since PBS has a low melting temperature (115°C), it has been determined that polymer chips stick to each other and there is a clumping problem in trials above 100°C.

Table 4. Drying trials and results

Time (h)	Temperature (°C)	Moisture (ppm)
4	50	800
4	65	750
4	80	500
4	95	250

Time (h)	Temperature (°C)	Moisture (ppm)
4	80	500
6	80	350
8	80	200
10	80	<50

3.2. Physical Characterization of PBS Multifilament Yarns

First of all, the linear density of PBS yarns produced at winding speeds of 1015, 1515, 2015 and 2815 m/min are 475, 328, 252 and 171 dtex (gr/10000m), respectively. It is observed that dtex value decreases with increasing winding speed at constant spin pump speed (flow rate).

Secondly, when the elongation (at F_{max}) results are examined; it is observed that there are decreases in elongation (at F_{max}) values with increasing winding speed (Figure 2). It is understood that PBS yarns produced at winding speeds below 2000 m/min exhibit elongation properties similar to conventional polyester yarns produced at winding speeds between 2000 – 3500 m/min. While the speed increase in winding speed trials was 500, 500 and 800 m/min, the changes in the elongation of PBS multifilament yarns were calculated as 13,00, 14,51 and 9,04 %, respectively. In particular, we observe that the decrease in elongation with the increase in the winding speed of 800 m/min should be higher than the decrease in the increase in the winding speed of 500 m/min, but it is lower than others.

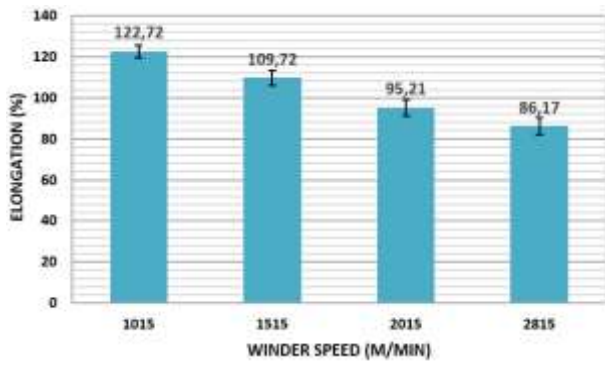


Figure 2. Elongation (at F_{max}) values of the PBS multifilament yarns

When the strength values of the yarns are examined; it is observed that increasing winding speeds have a positive effect on the strength values and cause significant increases. While the speed increase in winding speed trials was 500, 500 and 800 m/min, the changes in the strength of PBS multifilament yarns were calculated as 0,22, 0,19 and 0,24 cN/dtex, respectively (Figure 3). As expected, it was observed that the tenacity values of PBS multifilament yarns increased when the winding speed increased, inversely proportional to the elongation values.

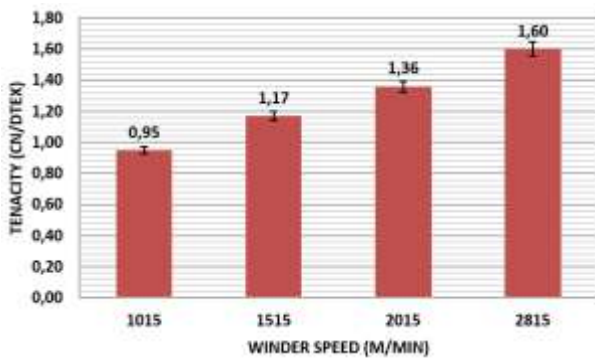


Figure 3. Tenacity values of the PBS multifilament yarns

3.3. Thermal Characterization of PBS Multifilament Yarns

DSC curves of PBS multifilament yarns were given in Figure 4. The data for enthalpy, melting point and crystallinity of the yarns were given in Table 5.

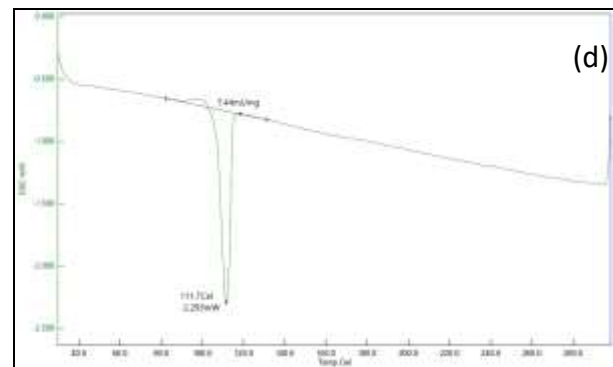
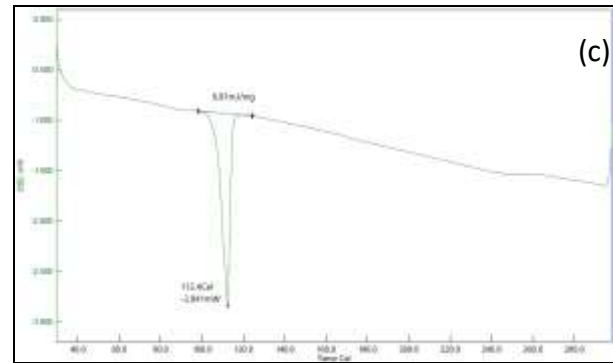
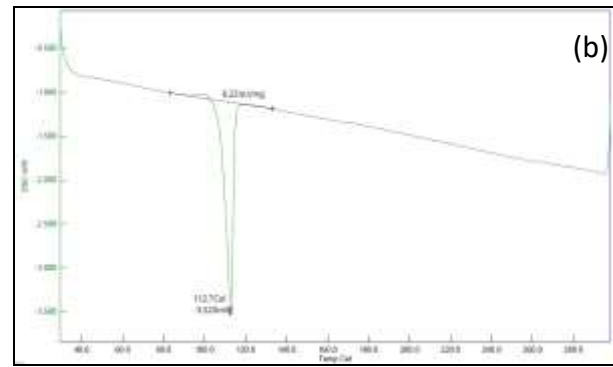
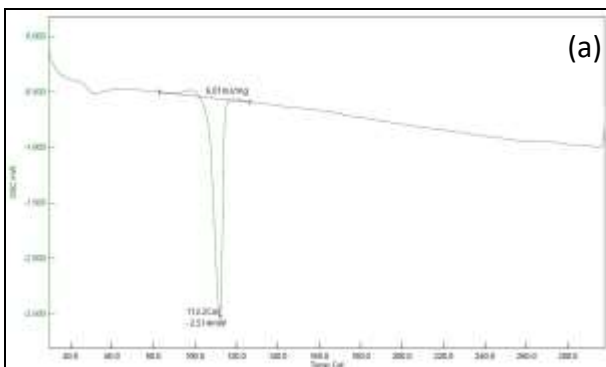


Figure 4. DSC curves of PBS multifilament yarns at different winding speeds; (a) 1015 m/min, (b) 1515 m/min, (c) 2015 m/min, (d) 2815 m/min

Table 5. The heat of fusion, melting point and crystallinity of the PBS multifilament yarns

Winding Speed (m/min)	Melting Point (°C)	Enthalpy (J/g)	Crystallinity (%)
1015	112,2	6,01	2,86
1515	112,7	6,22	2,96
2015	112,4	6,81	3,24
2815	111,7	7,44	3,54

In the DSC curves of PBS multifilament yarns, only the endothermic peak due to the melting temperature was observed. The melting temperatures of the all yarns produced at different winding speeds were identical (approximately 112 °C). The enthalpy values that is a function of the interchain forces, and the crystallization values calculated depending on enthalpy increased as the winding speed increased, similar to the tenacity values.

3.4. Cross-Section Analyzes of PBS Multifilament Yarns

In the melt spinning process, multifilament yarn production was made from PBS biopolymer by using 48-hole round section type spinnerets. The cross-section analysis was performed on the yarn samples taken from each of the yarns produced with different winding speeds. If we examine all the cross-section images, we observe that the filament diameters are the same as the other filament diameters. It is also observed that the filaments have smooth round cross-sections (Figure 5). When we compare with the studies in the literature; it is understood that the cross-sectional images of PBS yarns produced at high winding speeds have characteristic features similar to the cross-sections of conventional yarns such as polyester filaments. These results demonstrate that we can produce PBS yarns has uniform cross-section property at high speed.

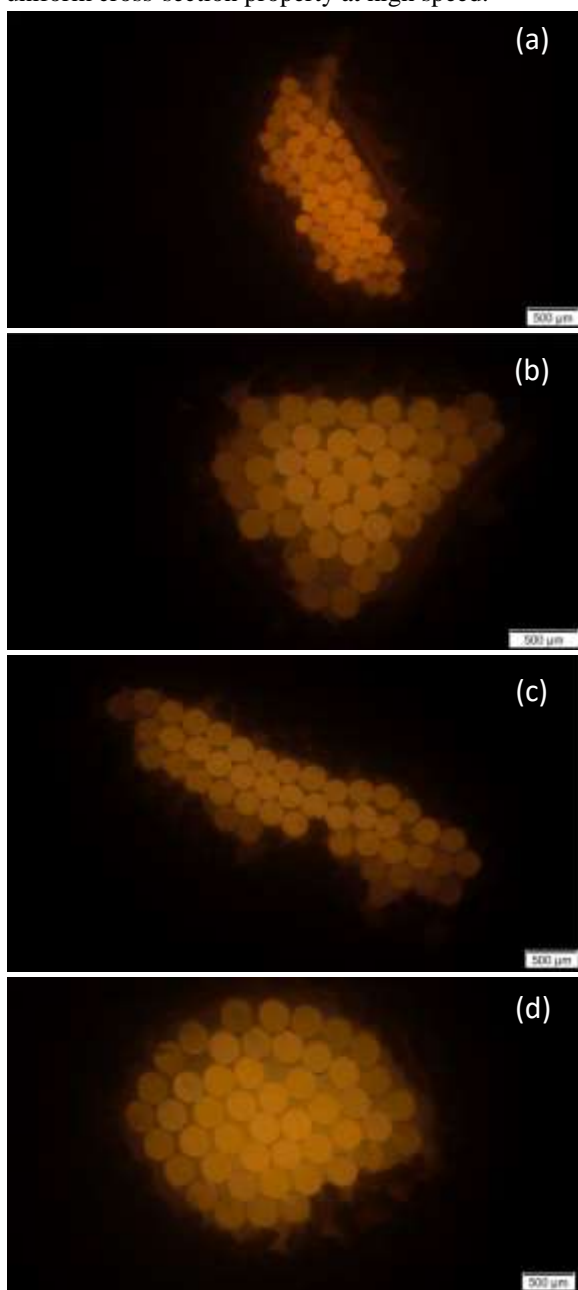


Figure 5. Cross-sectional views of multifilament yarns; (a) 1015, (b) 1515, (c) 2015, (d) 2815 m/min

The diameters of the PBS filaments were calculated by using computer and a special software which integrated to optical microscope. The means and standard deviations of the diameter of PBS filaments were given in Table 6. The decrease in the linear density of PBS multifilament yarns is directly proportional to the decrease in the diameter of the filaments. Increasing the winding speed causes the orientation of the amorphous polymer chains and the free volume in the polymer filament to decrease [41]. It was observed that the filament diameters decreased due to the reduction of free volumes with the orientation of the polymer chains.

Table 6. The filament diameter (mean \pm standard deviation)

Winding Speed (m/min)	Filament Diameter (μm)
1015	321,37 \pm 3,13
1515	260,26 \pm 3,24
2015	229,39 \pm 1,67
2815	189,94 \pm 3,15

4. CONCLUSION

This study was carried out in order to offer an eco-friendly and green alternative to petroleum-based polymer raw materials used in melt spinning fiber production method. The lack of information in terms of working parameters, raw material information and textile physics analyzes of PBS multifilament yarns produced in these studies was determined by literature reviews. In addition, multifilament yarn production from PBS raw material was carried out for the first time in a large-scale melt spinning machine, and a study was carried out for the first time at high winding speeds in terms of winding speeds. In this context, biopolymer PBS multifilament yarns with 48 filaments and round cross-section type were produced at 1015, 1515, 2015 and 2815 m/min winding speeds.

It is concluded that the linear density value decreases with the increase of the winding speed. Since the melt density of the polymer and the spin pump speed are constant, it is usual for the linear density to decrease with the increase of the winding speed.

It is observed that with the increase of the winding speed, there is a decrease in the elongation (at Fmax) value as well as the linear density. The lowest elongation (at Fmax) value was obtained in PBS multifilament yarn which was produced a winding speed of 2815 m/min compared to yarns produced with other speeds. Additionally, the elongation of PBS multifilament yarn produced with a winding speed of 1015 m/min had the highest value among others. Another result obtained from the tensile test is the tenacity value of the yarns. There was a significant increase in the tenacity value of PBS multifilaments with increasing winding speed.

The highest tenacity value was obtained in PBS multifilament yarn which was produced at winding speed of 2815 m/min among others. In addition, the elongation of PBS multifilament yarn produced with a winding speed of 1015 m/min had the lowest value compared to yarns produced with other speeds.

The heat of fusion and the crystallinity of PBS multifilament yarns gradually increased as the winding speed was increased. It was determined that PBS multifilament yarn produced at 2815 m/min had the highest value for the heat of fusion and crystallinity.

In the cross-section images obtained by the optical microscope, it was concluded that there are smooth round sections. Also, the diameters of PBS filaments was

calculated, and it was observed that filament diameters decreased with increasing winding speed.

In the next study, PBS multifilament yarn production trials will be carried out at higher winding speeds in order to produce yarn under conditions similar to the production of conventional polyester multifilament yarns.

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