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Polarize Edilmiş İzotaktik Polipropilen Monofilamentlerinin Voltaj Çıktıları Üzerine Çekim Oranının Etkisinin İncelenmesi

Derman VATANSEVER BAYRAMOL
Department of Textile Engineering, Namik Kemal University, Turkey

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INVESTIGATION ON THE EFFECT OF DRAW RATIO ON VOLTAGE OUTPUTS OF POLARISED ISOTACTIC POLYPROPYLENE MONOFILAMENTS

Derman VATANSEVER BAYRAMOL*

Department of Textile Engineering, Namik Kemal University, Turkey

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***ABSTRACT*:** Piezoelectric isotactic polypropylene (iPP) monofilaments were prepared by drawing and poling on a laboratory scale melt extruder. Results showed that the filament properties are affected by the drawing ratio. As expected, an increase in draw ratio caused lower filament counts (thinner diameters), higher tensile strengths and higher crystallinities. To invetigate the effect of draw ratio on voltage output of produced samples, a rotational mass was applied onto the samples. Results showed that filaments subjected to a draw ratio of 2:1 had highest voltage output as compared to others. Produced filaments are found to be suitable for use in smart textiles.

Keywords*:* piezoelectric, isotactic polypropylene, melt extrusion, voltage output

POLARİZE EDİLMİŞ İZOTAKTİK POLİPROPİLEN MONOFİLAMENTLERİNİN VOLTAJ ÇIKTILARI ÜZERİNE ÇEKİM ORANININ ETKİSİNİN İNCELENMESİ

ÖZET: Bu çalışmada laboratuvar tipi bir eriyikten lif çekim ünitesinde, çekim ve polarizasyon uygulanarak piezoelektrik izotaktik polipropilen (iPP) monofilamentler üretilmiştir. Araştırma sonuçları filament özelliklerinin çekim miktarından etkilendiğini göstermiştir. Beklendiği gibi, çekim miktarındaki artış daha düşük filament numaralarına (daha ince çaplara), daha yüksek mukavemetlere ve daha yüksek kristalin bölge miktarına neden olmuştur. Çekim oranının üretilen numunelerin voltaj üretimleri üzerine etkisini incelemek için dönen bir cisim numunelerin üzerine uygulanmıştır. Sonuçlar, kaydedilen en yüksek voltajın 2:1 oranında çekim uygulanarak üretilmiş filamentten yapılan numuneden elde edildiğini göstermiştir. Üretilen filamentlerin akıllı tekstil ürünlerinde kullanılmaya uygun olduğu sonucuna varılmıştır.

Anahtar Kelimeler*:* piezoelektrik, izotaktik polipropilen, eriyikten çekim, voltaj çıkışı/üretimi

**** Sorumlu Yazar/Corresponding Author:*** *dvbayramol@nku.edu.tr*
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1. INTRODUCTION

Same materials show extraordinary responses as a result of applied external stimuli. These materials are called as “smart materials” and they provide many advantages for a significant number of applications. Materials having piezoelectric properties are in this smart family because they can convert mechanical energy to electrical potential and vice versa. Therefore, piezoelectric materials can be used as both sensors and actuators.

The history of piezoelectric materials goes back to the last quarter of 19th century. Curie brothers (Pierre Curie and Jacque Curie) observed charges on the surface of quartz crystal when a weigth was placed on. They announced that the charge generated on the surface was proportional to the size of the stimulus [1]. This is called as “direct piezoelectric” which means the electricity produced by piezo (pressure in Greek). Soon after, they experimentally proved that this crystal material could undergo a shape deformation when an electrical charge was applied. This is called as “converse/reverse/inverse piezoelectric effect” which means the shape change resulted from an applied electrical charge.

Some crystals and some man-made ceramic based materials are prevalently used for a significant number of applications, such as submarine detectors [2], loud speakers [3], microphones [4], spark generators [5], buzzers [6] etc. Working with polymers for piezoelectric applications is a newer area of research as compared to crystal and ceramic based materials. The first work on piezoelectric behaviour of a polymer was conducted in 1969 [7], years later than the discovery of piezoelectric phenomenon. Cellular polypropylene is known to be one of those polymers which can convert mechanical energy to electrical charge [8-12].

Polypropylene is one of the most widely used polymer in technical applications due to its versatility and a number of advantages upon other polymers. It is easily available and less expensive as compared to other polymers which can be used for piezoelectric applications. There are a number of works but most of them concentrate on cellular polypropylene films [8-10]. In this experimental study, polypropylene monofilaments were successfully produced via a laboratory scale melt extruder. A polarization unit was built and placed between the slow and fast rollers in the draw area where the filament was reheated upto 90°C. Therefore, filaments underwent thermal, mechanical and electrical conditions simultaneously. To investigate the effects of draw ratio on the voltage output of iPP filaments, 50mmx30mm samples were produced and subjected to a ligth rotational impact. Results showed that the highest voltage response was recorded from the sample produced from 2:1 times stretched filaments. Surface morhologies of the filaments were also investigated under scanning electron microscopy (SEM).

2. MATERIAL AND METHODS

Higly isotactic polypropylene polymer (iPP) granules (HJ120UB) were received from Borealis AG, Austria with pleasure. HJ120UB is the commercial propylene homopolymer with a density of 0.905 g/cm^3, an MFR of 75 g/10 min. and a melting temperature of approximetly 165°C.

For filament production, a laboratory scale single-screw melt extruder originally constructed by Plasticisers Engineering UK was used for the melt extrusion of the filaments. The screw with a diameter of 22 mm and a maximum speed of 50 rpm was located in individually controlled three heating zones. Since the aim was to investigate the effect of draw ratio, other production parameters were kept the same. The feeding speed of the polymer was 2rpm for the all samples. The temperetures of the feeding zone, melting zone and die zone were controlled via integrated thermocouples. The temperature was set to 150°C at the feeding zone. The temperature was increased gradually to 165°C (zone-1), 175°C (zone-2) and 185°C (zone-3) at the melting zone and it was maintained at 200°C at the die.

There were six take-up (slow) rollers and two fast rollers on the melt extruder. The filament is cooled via first two slow rollers which are water-cooled and then reheated via other four slow rollers which are temperature controlled. The tempereture of these four slow rollers were 60°C, 70°C, 80°C and 90°C, respectively. After slow rollers, filament reaches to the fast rollers and between these slow and fast rollers the drawing of the filament occurs. The speed of take-up rollers (slow rollers) were 7.5 meter per minute (mpm) while the spped of fast rollers was increased from 7.5 mpm. up to 52.5 mpm. to apply different draw ratios. The filament is also polarized simultaneously during the drawing as aforementioned. A Spellman SL300 series high voltage power supply was used for polarization. 15kV was applied onto the iPP monofilament via two conductive plates which were located in the draw area. For detailed information on piezoelectric filament production from polymers, readers are recommended to read relevant literatures [13-14].

2.1. Characterisation of poled iPP filaments

7 lots of filaments were produced to investigate the effects of draw ratio. To avoid any confusions, produced filaments were named as given in Table 1. The TS presents “times stretched” while F stands for “filament”. Therefore, 7TSF presents “seven times stretched filamet”. The speeds of the slow and fast rollers are also given in the same Table. Since a laboratory scale melt extruder was use to produced the filaments, the speed of the rollers were checked frequently on the indicators as well as a tachometer to avoid the change of the draw ratio during the production.

Filament count of each produced filaments was investigated by weighing a certain lenght of the filament. The same measurement was carried out 5 times for each sample and the avarage value was taken as the count of that specific filament. Tensile strength and elongation properties of produced filaments were investigated under a Textechno Statimat M Tensile Test Equipment. A single filament was located between two clamps with a gauge length of 100mm. Top clamp was stable while bottom clamp moved with a speed of 300mm/min. The test was carried of 10 times for each filament sample. The printed results gave the avarage fo 10 measurements as well as the individual tensile strength and elongation results. The effect of drawing on the crystallinity of the filaments was investigated by using TA Instruments DSC Q2000 equipment. Each sample was weigthed and placed in the equipment where the reference sample sits. The samples were thermally scanned from −50°C to 200°C under nitrogen atmosphere with a heating rate of 10°C/min. Endotermic melting peaks of the samples were found to be in the region of 164–167°C. The crystallinity of the filaments (ΔX_c) were calculated from equation (1)

Table 1. Nomenclature of samples produced at different draw ratios

Sample ID	Speed of slow rollers (mpm)	Speed of fast rollers (mpm)	Draw ratio (Times of stretching)
1TSF	7.5	7.5	1:1
2TSF	7.5	15	2:1
3TSF	7.5	22.5	3:1
4TSF	7.5	30	4:1
5TSF	7.5	37.5	5:1
6TSF	7.5	45	6:1
7TSF	7.5	52.5	7:1

$$\Delta X_c(\%) = \frac{\Delta H_m(J/g)}{\Delta H_{m100}(J/g)}\ x100 \qquad (1)$$

where ΔH_m is the melting enthalpy of the filament and ΔHm_{100} is the melting enthalpy for 100% crystalline PP which was taken as 209 J/g [15-18].

The surface morphology of the filaments were examined by using a Hitachi S-3400N Scanning Electron Microscope (SEM), The microstructural images of poled filaments were captured at an accelerating voltage of 5 kV and at various magnifications. For obtaining clear surface images, all the samples were gold-coated for 45 seconds by using an EMS 7620 Mini Sputter Coater. Mechanical and thermal characteristics and surface morphologies of the filaments were tested by using produced filaments. However, measuring the voltage response of a single filamet is extremely challenging for now. Therefore, fifty pieces of each filament were aligned between two pieces of aluminium sheet to acquire fiber composite-like samples.

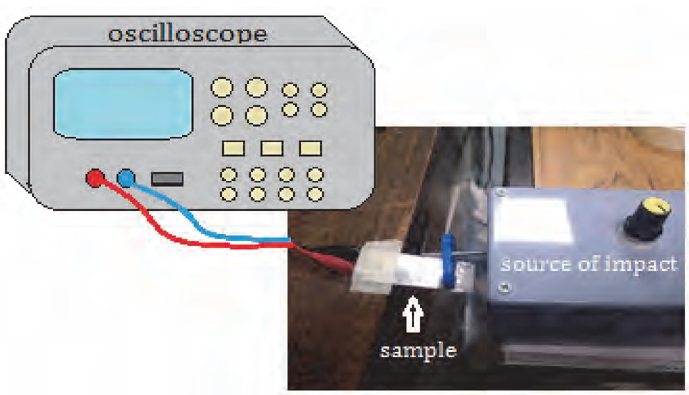

Figure 1. Visual presentation of the experiment carried out to investigate the voltage outputs of the samples when subjected to the same impact created by a rotational mass.

Prepared samples were immobilised from one end, where it was connected to a digital oscilloscope, and the other end was left free where a rotational mass was applied onto the material as shown in Figure 1. The samples started to oscillate as a result of applied impact crated by the rotational mass. The voltage responses of the samples were recorded by the digital oscillosscope and saved in an external pen drive.

3. RESULTS AND DISCUSSIONS

Poled iPP filaments with various draw ratios were produced to investigate the effect of draw ratio on tensile strength, crystallinity and voltage output of produced filaments. Obtained results from each test and experiment have been discussed in this section with the same order as explained in the previous section. Lineer density, tensile strength and elongation values of the filaments are given in Table 2 and a comparision diagram for tensile strength and elongation values for each filament is shown in Figure 2.

Table 2. Lineer density, tensile strength and elongation values of the filaments produced at different draw ratios

Sample ID	Filament count (tex)	Tensile strength (cN/tex)	Elongation at break (%)
1TSF	70.09	4.41	4.17
2TSF	37.06	10.39	180.25
3TSF	25.47	14.76	88.9
4TSF	19.25	18.96	14.23
5TSF	15.77	28.79	15.82
6TSF	13.40	39.10	14.67
7TSF	11.52	46.83	13.7

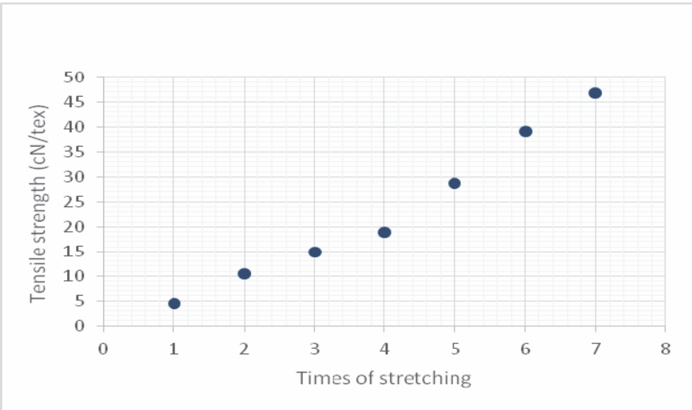

Figure 2. Comparative demonstration of tensile strength (cN/tex) values obtained from the Textechno Statimat M Tensile Test Equipment for each sample

As expected, tensile strength of the filamets increased with an increase in draw ratio. Therefore, the maximum value of tensile strength was 46.83 cN/tex recorded for 7TSF. The strength of the filamets decreased almost gradually as a result of decreased drawing. The lowest tensile strength was 4.41 cN/tex, which was recorded for non-drawn filaments (1TSF). Temperature dependent behaviours of the filaments were investigated under DSC. The tests conducted for each sample between -50°C and

200°C with 10°C increment per minute. Results such as mething temperature and melting enthalpy of the samples were obtained from the thermograms. The degree of crystallinity for each sample was calculated by equation (1). The results were as given in Table 3.

As seen in Table 3, DSC thermograms and calculated crystallinites proved that an increase in stretching/draw ratio induced an increase in the degree of crystallinity. These results were supported by tensile strength values. It can be said that the tensile strength was contributed further by the amount of crystalline structure in a filament. However, for further phase identification of the filaments an XRD analysis could be carried out. XRD provides the information about the materials including but not limited to crystallinity, crystal structure of the material with interatomic distances and bond angles of the atoms, phase identity, phase purity etc. Therefore, the effect of draw ratio on the crystal and amorphous structures of the filaments can be investigated further by XRD.

Table 3. Thermal characteristics of the filaments; melting temperature and melting enthalpy values were obtained from DSC, degree of crystallinity was calculated by the equation 1.

Sample ID	Melting Temperature Tm (°C)	Melting Enthalpy ΔHm (J/g)	Degree of Crystallinity Xc (%)
1TSF	163.80	80.01	38.3
2TSF	164.61	82.58	39.5
3TSF	163.47	88.69	42.4
4TSF	165.91	91.11	43.6
5TSF	164.28	92.77	44.4
6TSF	163.60	99.08	47.4
7TSF	166.82	101.20	48.3

Surface morhologies were investigated via SEM. Figure 3 presents the SEM images for each sample. 1TSF and 2TSF had high amount of voids on their surfaces. As the filament stretched further, these voids started to decrease and disappear. The filaments produced with a draw ratio of 7:1 had the smoothest surface as compared to others produced with lower draw ratios.

Voltage response of the filaments were investigated by applying a ligth impact onto the prepared fiber composite samples created by a rotational mass as shown in Figure 1. Samples were immobilized from one end, where it was connected to the oscilloscope, and the other end was left free for oscillation. The voltage responses of the samples were recorded into an external storage device. The voltage output results are given in Figure 4 (a-g).

Rotational impact test results showed that, draw ratio had an effect on voltage generation of the poled iPP filaments. This can be explained that existing voids started collapsing as a result of increasing draw ratio that may affect adversely to the material for carring the charge generated by the filaments. The data obtained from oscilloscope was recorded in an external data storage device. The voltage generation characteristic of each sample was given in Figure 4 (a-g) for comparetive evaluation. The comparison was done on the peak voltage output values of the samples. It was observed that the highest peak voltage (264mV) was generated by the sample prepared from 2TSF.

In the morphological images given in Figure 3, 1TSF and 2TSF had voids on their surfaces and it is assumed that these voids also existed in the filaments. It was reported earlier that these air voids are charged during the polarization and they also help the charge transfer when the material is subjected to an external sitimulus [19-21]. However, 1TSF (nonstretched) sample showed lower voltage generation than 2TSF. It would be assumed that 1TSF sample would have more voids into its structure since it wasnot stretched. It can be explained that 1TSF was the thickest filament which affected the oscillation ability of the whole sample structure. This is an agreement with some earlier works reported in the literatrure [11-12, 20-21]. Since 2TSF was much thinner that 1TSF, it could easily oscilate as a result of applied impact. The numerical peak voltage values of the samples recorded were as; 184mV for 1TSF; 264mV for 2TSF; 256mV for 3TSF; 200mV for 4TSF; 168mV for 5TSF; 144mV for 6TSF and 138mV for 7TSF. As the draw ratio of the filamets increased, the voids started to collapse, therfore the peak voltage values generated by the samples started to decline. These filaments can be used for applications where to detect small impacts.

Figure 3. Surface morphologies of produced filaments; images were captured at 5kV via SEM

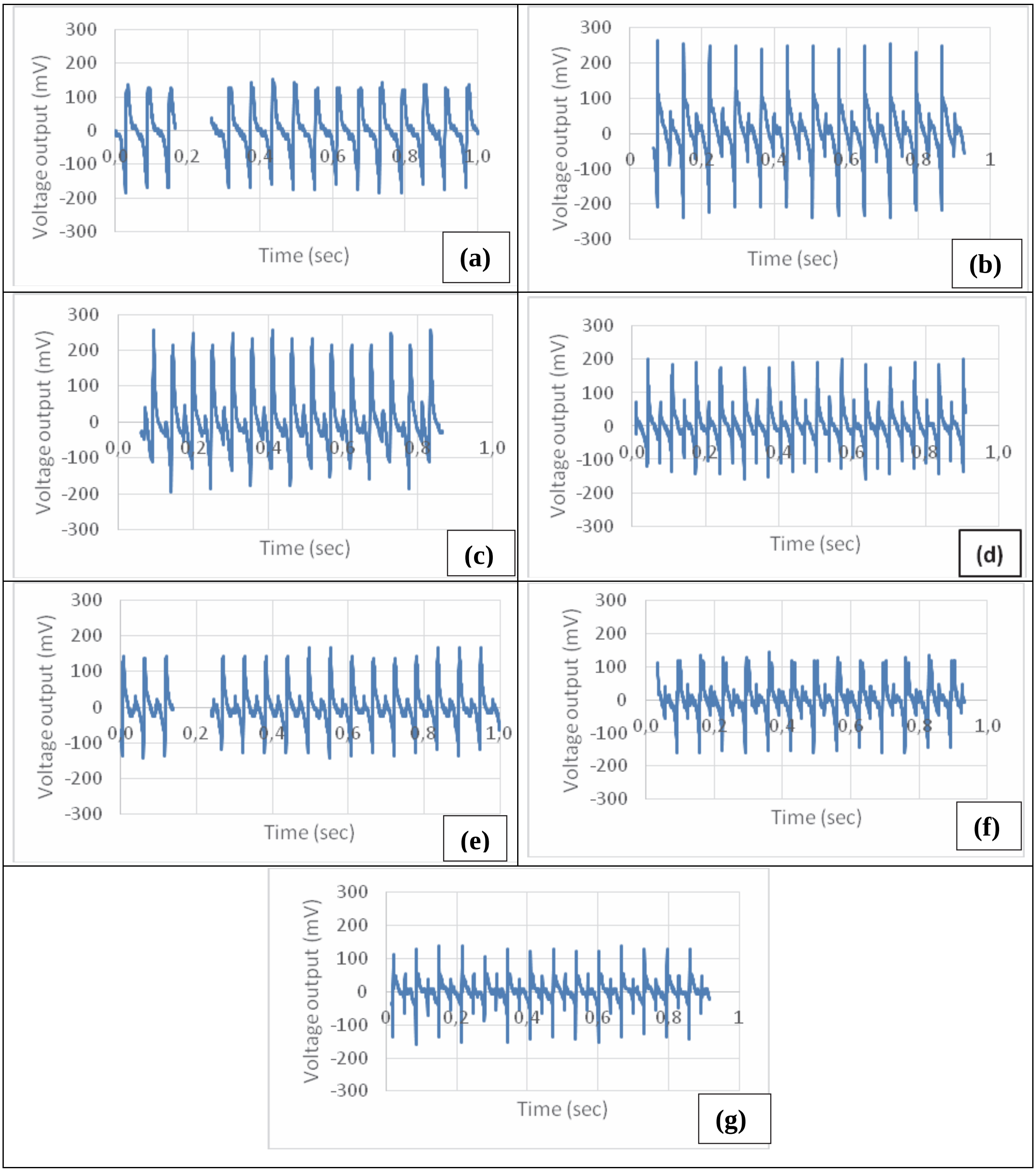

Figure 4. Voltage output results of the samples prepared from; (a) 1TSF, (b) 2TSF, (c) 3TSF, (d) 4TSF, (e) 5TSF, (f) 6TSF, (g) 7TSF poled iPP filaments

4. CONCLUSION

In this study, highly isotactic polypropylene monofilaments were produced via a laboratory scale melt extruder. Filaments were also polarized during the production by a polarization unit placed in the draw zone. Production parameters were kept the same except for draw ratio. Tensile properties, crystallinity behaviour and surface morphologies of the filaments were investigated. An increase in draw ratio caused lower filament counts (thinner diameters), higher tensile strengths and higher crystallinities. Therefore, it can be stated that all these parameters were affected by the draw ratio. Comparetive study on voltage outputs of the samples showed that the highest peak voltage generation recorded was generated by the sample produced at 2:1 draw ratio. Further strecthings caused unformed air voids which have the charge seperation during the polarization. 1TSF samples showed as many voids as 2TSF had. However, the voltage output of 1TSF was lower than that of 2TSF. It was because 1TSF was much thicker than 2TSF, the applied impact was not enough to oscillate the sample as much as 2TSF. It can be concluded that it is possible to produce voltage generating piezoelectric iPP. However, for each specific application, mechanical and voltage response charcteristics should be evaluated.

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