10th Eco-Energy and Materials Science and Engineering Symposium


On December 5-8, 2012
Sunee grand hotel,
Ubon-ratchathani

Organized by

Co-organized by
PREFACE:
Message from the President of
Rajamangala University of Technology Thanyaburi

Rajamangala University of Technology Thanyaburi (RMUTT), in conjunction with Kyoto University, is pleased to host the 10th Eco-Energy and Materials Science and Engineering Symposium (10th EMSES). This international conference is not only giving an opportunity for Thai and foreign researchers to present and discuss their research works and update their expertise but also to initially stimulate the development of research works on eco-energy and materials science and engineering. Our program consists of six research tasks: (1) Energy Technology, (2) Environmental and Social Impact, (3) Nanotechnology and Materials Science, (4) Energy Economics and Management, (5) New Energy technology and (6) Nuclear Technology.

I would like to take this opportunity to express our sincere gratitude to our two distinguished Plenary Speakers for kindly accepting our invitation. I deeply appreciate of the very strong support given by Kyoto University. Thanks to the tireless works of the Organizing Committee, the Technical Program Committee, the invited speakers and paper and poster contributors, and excellent program been assembled to cover a broad spectrum of interesting topic.

We warmly welcome you to the 10th EMSES on December 5-8, 2012, Ubon Ratchathani, Thailand.

Numyoot SONGTHANAPITAK, Ph.D.
President of Rajamangala University of Technology Thanyaburi and Conference Chairman of 10th EMSES 2012
PREFACE:
Message from the Director of Institute of Advanced Energy, Kyoto University

It is my great pleasure to have the 10th Eco-Energy and Materials Science and Engineering Symposium (EMSES) with Rajamangala University of Technology Thanyaburi (RMUTT) under the long-term collaboration between RMUTT and Kyoto University. The 1st EMSES was held in 2001 in Thailand and the symposium has been expanded in its scientific contents as well as the academic network. I believe that the 10th EMSES gives a good opportunity to all participants to exchange their knowledge and idea to realize eco-friendly energy system in society. I would like to express my welcome to all participants and sincere thanks to the 10th EMSES organizing committee and all supporting organizations to make us having this symposium.
I hope that the symposium will be successful and lead to further progress in energy science and technology and also in friendships of participants.

Professor Yukio Ogata, Ph.D.
Director of Institute of Advanced Energy, Kyoto University
PREFACE:
Message from the Former Dean of
Graduate School of Energy Science, Kyoto University
Program Leader,
Global COE “Energy Science in the Age of Global Warming”

I want to express my hearty welcome to all participants of Eco-Energy and Materials Science and Engineering Symposium (10th EMSES). This symposium is aiming the realization of importance of energy and materials technology through the academic, science and technology network among the world communities. The symposium gives an opportunity for researchers to discuss their research works and also to initially stimulate the development of research works on eco-energy and materials science and engineering. Once the cooperation among researchers has been created, the further cooperation work will be developed.

I would like also extend my sincere thanks to all who made the meeting possible, including the 10th EMSES organizers, the SEE forum committee members, and the Japanese Government, JSPS, for their kind support. I am looking forward to seeing you in Ubon Ratchathani, Thailand.

Professor Takeshi YAO, Ph.D.
Former Dean of Graduate School of Energy Science, Kyoto University
and Program Leader, Global COE “Energy Science in the Age of Global Warming”
Message from the Chairperson of
10th EMSES Organizing Committee

Rajamangala University of Technology Thanyaburi (RMUTT), in conjunction with Kyoto University, is pleased to host the 10th Eco-
Energy and Materials Science and Engineering Symposium (10thEMSES).

RMUTT has a major mission on encouraging and supporting all areas of research. One of the key reasons is to assist in developing capability in science and technology in order to cope with recent rapid change in this field. We have jointly set up an academic symposium on the 10thEMSES with the perception on the significance of exchanging knowledge and research experiences between researcher in the field of energy, materials technology and environmental science. This symposium is not only giving an opportunity for Thai and foreign researcher to present and discussion their research works and update their expertise but also to initially stimulate the development of research works on eco-energy and materials science and engineering. Once the cooperation among researchers has been created, the closer future cooperation incorporate with joint-research works will be developed. Thus, to support the aforesaid role, the symposium working committee would like to invite you to participate in this academic symposium.

I would like to express our sincere thanks to the organizing committee, participants and contributors for your kind corporation to this symposium. I wish this symposium proceeding will be a useful reference for future scientific research development.

Sommai PIVSA-ART, Ph.D.
Dean of Faculty of Engineering, RMUTT
Director of CoE on Sustainable Energy System (Thai-Japan)
Organizing Chairman of 10th EMSES 2012
10th EMSES 2012

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30 x 100 = 3000
### Conference Program of 10th Eco-Energy and Materials Science and Engineering

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<td>Keynote Speaker I: Japan Power Generation Mix and Energy Security after Fukushima Nuclear Accident, presented by Professor Dr. Keiichi N. Ishihara, Graduate School of Energy Science, Kyoto University, Japan</td>
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<td>Prof. Dr. Padungsak Rathnanacho</td>
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<td>Assoc. Prof. Dr. Seiichi Kawahara</td>
<td>Prof. Dr. DaeHee Park</td>
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<td>Dr. Wirachai Roynarin</td>
<td>Dr.Sorapong Pavasupree</td>
<td>Asst Prof. Dr. Warunee Arikarnayanan</td>
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<td>Prof. Dr. Narongrit Sombotsompop</td>
<td>Prof. Dr. Hideaki Ohgaki</td>
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<td>Dr. Leong Yew Wei</td>
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<td>Dr. Arthit Sode-Yome</td>
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<td>Chair</td>
<td>Prof. Dr. Chul-Su Kim</td>
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<td>Prof. Dr. Keiichi N. Ishihara</td>
<td>Assoc. Prof. Dr. Natha Kuptasithien</td>
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<td>Asst. Prof. Dr. Sommai Pivsa-art</td>
<td>Dr. Boonyang Plangklang</td>
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<td>Time</td>
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Effects of Melt Spinning Conditions on Cross-sectional Features of Poly(lactic acid) Fibers

N. Roungpaisan\textsuperscript{1}, N. Ocharoen\textsuperscript{1}, N. Srisawat\textsuperscript{1}, C. Prahsarn\textsuperscript{1}, S. Pavasupree\textsuperscript{1*}

\textsuperscript{1}Department of Materials and Metallurgical Engineering, Faculty of Engineering, Rajamangala University of Technology Thanyaburi, Pathumthani 12110, Thailand
\textsuperscript{2}Department of Textile Engineering, Faculty of Engineering, Rajamangala University of Technology Thanyaburi, Pathumthani 12110, Thailand
\textsuperscript{3}National Metal and Materials Technology Center, 114 Paholyothin Rd., Klong Luang, Pathumthani 12120, Thailand
\textsuperscript{*E-mail: sorapongp@yahoo.com}

Abstract— Melt spinning of Poly(lactic acid) was conducted, using two spinneret profiles—circular and 4-lobed shapes, to investigate effects of spinning conditions on cross-sectional features of the obtained PLA fibers. The studied spinning parameters included throughput rates, spinning temperatures, and take up speeds. Results showed that, with varying spinning conditions, PLA fibers spun from circular orifice could remain their circular shapes whereas those spun from 4-lobed orifice exhibited variation in cross-sectional shapes. Such shape variation was found increased with increasing spinning temperature and throughput rate, but with decreasing take up speed. This offered possibility to design fibers with desired cross-sectional shapes for different applications, through the same spinneret orifice, simply by manipulating fiber spinning conditions.

Keywords— Poly(lactic acid) fibers, Fiber melt spinning, Cross-sectional fibers, Die swell, Surface tension

1. INTRODUCTION

Poly(lactic acid) or PLA has been widely used in biodegradable plastic applications due to its naturally-derived lactic acid monomer [1-3]. In textiles, an introduction of poly(lactic acid) as a commercial polymer offers textile manufacturers with interesting properties that should prove valuable in a wide range of applications. Cross-sectional feature of fibers is known to closely relate to properties including surface area, hand, and luster. For example, the commercial deep-grooved fibers (4DG), having capillary channeled surface, showed excellent moisture transport. Recently, the fibers were also used to fabricate tissue engineering scaffolds with the potential for cell growth and guide tissue formation [4-6].

Despite their great potential on properties and applications, shaped fibers were difficult to prepare. In their melt spinning, it is difficult to maintain fiber cross-sectional shape the same as its original hole orifice due to the effects of Newtonian fluid on die swell and surface tension [7-8]. There were reports on studying melt flow through star-shaped spinneret hole, using hydromechanical simulation software (CFD) [9-10]. It was found that fluid shear strain rate of melt through star-shaped spinneret hole was non-uniform. The largest shear strain rate occurred at the center of shaped hole, causing greater shape deformation than that at the extended arms. Therefore the spinneret hole should be re-designed to obtain balance shear rate that helped reducing the gap between polymer melt and spinneret hole, and thus reducing the shape changing.

In this study, PLA fibers were spun through circular and 4-lobed orifice profiles, under varied spinning temperatures, throughput rates, and take up speeds in order to investigate spinning behaviors as well as roles of these influencing parameters on cross-sectional features of PLA fibers.

2. PROCEDURE

2.1 Materials
Polyactic acid resins (PLA 3052D) (MFI = 14 g/10 min, density 1.24 g/cm\textsuperscript{3}) obtained from NatureWorks LLC were used as received.

2.2 Method
To prepare PLA fibers with two different cross-sectional shapes (circular and 4-Lobed), PLA resins were melt spun through 10-hole spinnerets, using laboratory fiber spinning machine (Fig. 1). Profiles of the two spinneret orifices employed were shown in Figure 2. Different spinning conditions: spinning temperatures (220, 240 and 260°C), throughput rates (0.34, 0.52 g/hole/min), and take up speeds (300 and 500 m/min.) were used (Table 1).

2.3 Characterizations
Characteristics of the obtained circular and 4-lobed PLA fibers such as cross-sectional features, perimeter, area, compactness were investigated, using optical microscope and Imagent software. Thermogravimetric Analyzer (TGA/SDTA851e) was used to determine thermal stability of PLA resins. The samples were heated at a rate of 5 °C per minute from 50°C to 800 °C. The chamber was continuously swept with nitrogen at a rate of 50 ml/min. The corresponding weight change was noted. Melt viscosity of PLA resins was measured at varied temperatures (220, 240, and 260°C), using rotational rheometer (ARE, Rheometric Scientific).
Polymer melt viscosities of PLA resins were measured at temperature 220, 240, and 260°C, and results were shown in Figure 4. The viscosity of PLA measured at 220°C was the highest, followed by those measured at 240 and 260°C. At 220 °C, polymer viscosity changed with shear rate, and dropped rapidly at very high shear rate (100/s). On the other hand, at 240°C and 260°C the polymer melt viscosities were observed less dependent on shear rate than that of 220°C. The polymer melt exhibited newtonian behavior at extreme shear rates (100/s) where the viscosity is approximately constant [12-13]. This showed that, under different spinning temperatures, the polymer melt exhibited different fluid behavior, which could be related to variation in cross-sectional shapes in the obtained PLA fibers.

3.2 Cross-sectional features
Cross-sectional features of PLA fibers obtained from different spinning conditions were shown in Figs. 5-6 and 7-8 for circular and 4-Lobed shaped fibers, respectively. PLA fibers spun from circular orifice could remain their circular shapes at all spinning conditions, whereas those spun from 4-lobed orifice exhibited variation in cross-sectional shapes and seemed to lose their grooved feature. In circular orifice, shear strain acting on polymer melt was more uniformly distributed, compared to that in the complex 4-Lobed orifice. As shown in Figs. 7 and 8, the features of 4-Lobed fibers changed with spinning parameters.

The final fiber profile was suspected to be related to surface tension of polymer melt. The adhesive forces between polymer melt and the walls of orifice center tended to decrease with increasing temperature. This affected shape forming of 4-Lobed fibers that low cohesive forces lead to transfiguration of cross-sectional feature so as the fibers showed variation shapes[16]. Yao et al. [7] reported numerical simulation on trilobal cross-sectional shape. Newtonian die swell and surface tension were considered to be the main causes for shape change such that surface tension made the shape become almost circular, in stead of trilobal shape, and die swell effect tended to enlarge the curvature in the center section.

In our study, it was noted that fibers exhibited shape transition from 4-Lobe to tetragonal shape when take up speed was decreased from 500 to 300 m/min, especially at spinning temperature 240°C and throughput rate 0.34 g/hole/min (Figs. 7-8). At lower take up speed, polymer
melt were slightly stretched and had more time to relax, thus exhibiting die swell.

Fig. 5 OM micrographs of circular PLA fibers at various temperatures (top-down) and throughput rates (left-right) 0.34 and 0.52 g/hole/min (TP0.34, TP0.52) at take up speed 300 m/min (S:300) (magnification=40x)

Fig. 6 OM micrographs of circular PLA fibers at various temperatures (top-down) and throughput rates (left-right) 0.34 and 0.52 g/hole/min (TP0.34, TP0.52) at take up speed 500 m/min (S:500) (magnification=40x)

Fig. 7 OM micrographs of 4-lobed PLA fibers at various temperatures (top-down) and throughput rates (left-right) 0.34 and 0.52 g/hole/min (TP0.34, TP0.52) at take up speed 300 m/min (S:300) (magnification=40x)

Fig. 8 OM micrographs of 4-lobed PLA fibers at various temperature (top-down) and throughput rates (left-right) 0.34 and 0.52 g/hole/min (TP0.34, TP0.52) at take up speed 500 m/min (S:500) (magnification=40x)
3.2.1 Effect of spinning temperatures

At lower spinning temperature (220°C), fiber spinnability of the 4-Lobed fibers was poor. Better spinnability was obtained when higher spinning temperature was increased to 240°C. However, at higher temperature 260°C, fibers breakage occurred from time to time. Such fiber breakage was suspected to be related to rapid decrease in polymer melt viscosity at 260°C even at lower shear rate (Fig. 4). However, the fibers could be collected, especially at higher take up speed (500m/min).

To compare between circular and 4-Lobed fibers, their perimeter and areas were determined (Figs. 9-10 and 11-12). From results, Circular fibers area and perimeter tended to increase with increasing spinning temperatures. On the other hand, area of 4-Lobe tended to increase with increasing spinning temperatures, whereas their perimeter showed no clear trend (Figs. 11 and 12). It was noticed that the groove of 4-Lobed fibers tended to disappear when spun at higher temperature.

![Fig. 9 Cross sectional area of circular PLA fibers obtained from different spinning conditions (Temperature 220, 240, 260 °C Throughput rates, TP:0.34, 0.52 g/hole/min and Take up speeds S:300, 500m/min) (magnification= 40x)](image)

![Fig. 10 Cross sectional perimeter of circular PLA fibers obtained from different spinning conditions (Temperature 220, 240, 260 °C Throughput rates, TP:0.34, 0.52 g/hole/min and Take up speeds S:300, 500m/min) (magnification= 40x)](image)

![Fig. 11 Cross sectional area of 4-Lobed PLA fibers obtained from different spinning conditions (Temperature 220, 240, 260 °C Throughput rates, TP:0.34, 0.52 g/hole/min and Take up speeds S:300, 500m/min) (magnification= 40x)](image)

![Fig. 11 Cross sectional perimeter of 4-Lobed PLA fibers obtained from different spinning conditions (Temperature 220, 240, 260 °C Throughput rates, TP:0.34, 0.52 g/hole/min and Take up speeds S:300, 500m/min) (magnification= 40x)](image)

3.2.2 Effect of throughput rates

As shown in Figs 9-12, both circular and 4-Lobed fibers obtained at high throughput 0.52 g/min/hole tended to have greater perimeter and area than those obtained from lower throughput 0.34 g/min/hole, for all take up speeds. Teke et al [14] reported influence of volumetric flow rates and take up velocities on surface tension of polyester melt. They found that such spinning conditions caused rapid acceleration and rapid attenuation for solidification of fiber near the orifice spinneret, resulting in different diameter profile of the obtained fibers. In our study, we found that throughput rate was related to take up speed in fibers of 4-Lobed profile. Figures 7 and 8 compared different fiber profiles in relation to throughput rates and take up speeds at various temperatures. It was noticed that at low throughput rate (0.34g/hole/min) and high take up speed (500m/min), most fibers tended to remain their grooves, which were preferable for fiber spinning.
3.2.3 Effect of take up speeds

Two take up speeds, 300 and 500 m/min, were used to study effect of take up speed on cross-sectional feature of PLA fibers. From results, both circular and 4-lobed fibers showed decrease in perimeter and area with increasing take up speed, for all throughput rates (Figs 9-12).

Zhou et al. [10] employed 3D simulation to adjust the value of surface tension coefficient until the predicted shape of as-spun polyester fiber was close to the actual observation. The depth of fiber grooves were chosen as primary standard, and the overall similarity in cross-section as secondary standard for comparison. Surface tension as a function of temperature and draw ratio were found to be a major factor to determine change of fiber cross-section. Temperature difference between outer surface and center of nozzle hole affected solidification of fibers. Fiber cross-section tended to have circular shape under high temperature and low draw ratio. On the other hand, the groove of polyester fiber could be maintained when increasing draw ratio although they were spun at high temperature. This result corresponded to our observation in 4-Lobed fiber. As shown in Figs. 7 and 8, fibers obtained at higher take up speed had sharp grooved profile, compared to those obtained at lower take up speed. This suggested that the 4-lobed PLA fibers preferably be spun at high take up speed.

3.3 Compactness of shaped fibers

Compactness is defined as feature extraction from shape [15]. It represents smoothness or roughness of the shape. Circular shape is considered most compact, and is given the lowest value. For rough or more edge shape, the value is higher. Value of compactness can be calculated from perimeter and area as shown in equation (1). In this study, the compactness value of the shape of spinneret orifice was determined to compare with those of actual spun shape fibers. As shown in Table 2, the compactness value of circular spinneret shape was equal to 1.13 and that of 4-Lobed shape was 12.43.

\[
\text{compactness} = \frac{\text{perimeter}^2}{4\pi a}
\]

(1)

Table 2 Compactness value of circular and 4-Lobed spinneret orifice shapes (OM= 40x)

<table>
<thead>
<tr>
<th>Measurement</th>
<th>Circular</th>
<th>4-Lobed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compactness</td>
<td>1.13</td>
<td>12.43</td>
</tr>
<tr>
<td>Perimeter(μm)</td>
<td>1666</td>
<td>7086</td>
</tr>
<tr>
<td>Area (Squm)</td>
<td>195847</td>
<td>321418</td>
</tr>
</tbody>
</table>

Figure 13 Compactness values of circular and 4-Lobed PLA fibers obtained from different spinning conditions (Temperatures 220, 240, 260 °C. Throughput rates: TP-0.34, 0.52 g/hole/min and Take up speeds S:300,500m/min)

Compactness values of the two profiled fibers: Circular and 4-Lobed, obtained from different spinning conditions were shown in Fig. 13. For circular fibers, compactness values were approximately 1.13 for all spinning conditions. Notice that shape areas were very similar among these circular shapes. For 4-lobed fibers, higher compactness values were observed with variations for difference spinning conditions. Fibers obtained at low temperature (220°C), low throughput rate (0.34g/hole min) and high take up speed (500m/min) showed highest compactness value (approximately 2.75). This indicated that their features were rougher than those of circular fibers and they were more similar to their original 4-lobed orifice. However, fiber breakage tended to occur at this spinning condition. Higher spinning temperature could be used to obtain better spinnability but it should not exceed 240°C, beyond which compactness started to decrease, i.e. less grooved feature. This may be due to newtonian fluid behavior at high temperature that caused loss in surface tension [7-10]. At this point, it seemed that spinning temperature was the most significant parameter which affected compactness, or cross-sectional features of 4-Lobed PLA fibers, compared to take up speed and throughput rate.

4. CONCLUSION

Circular and 4-Lobed shaped PLA fibers were prepared via melt spinning to investigate effects of spinning parameters on cross-sectional features of the obtained fibers. From results, it was found that spinning temperature was a major parameter that determined cross-sectional shapes of fibers, compared to take up speed and throughput rate. With increasing spinning temperature, the groove features of 4-Lobed fibers tended to lose or disappear. This was suspected to be related to loss in surface tension of polymer melt at high spinning temperature. In circular shaped fibers, such effect was found less that circular shape could still be maintained. In this study, the compactness value was used to indicate deviation of fiber shape from its original spinneret.
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REFERENCES