

Effect of trisilanol isobutyl polyhedral oligomeric silsesquioxane (TPOSS) in recycled PET (rPET) and polypropylene based compound: Investigation on mechanical properties

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Received date: 26 March 2023 Revised date 11 June 2023 Accepted date: 23 July 2023

Keywords:

Recycled polyethylene terephthalate; Polypropylene; Melt spinning; Compounding; Mechanical properties

Abstract

Recycled polyethylene terephthalate is successfully prepared. This project is involved for "Circular economy" policy. It is successfully prepared into powder form by nano-grinder technique. 10 phr of PP-g-MA and 1 phr to 5 phr of TPOSS are added for compatibilizer and mechanical properties enhancement, respectively. PP-g-MA can form the chemical linkage of the anhydride groups with the polyamide end groups. 20 wt% of recycled polyethylene terephthalate powder and polypropylene based compound is successfully fabricated by melt mixing. Then, it is fabricated by melting spinning technique. Fourier transform infrared presents Si-O-Si linkage in binary blend compound. X-ray diffraction pattern reports that single phase is obtained. Adding TPOSS can be induced to crystallinity enhancement. No significant change of thermal properties was observed from room temperature to 300°C. Scanning electron microscope reports that with the PP-g-MA provides smoothness of surface. Recycled polyethylene terephthalate is uniformly distributed. Melting and crystallization temperatures are reported to be 162°C to 167°C and 115°C to 120°C, respectively. All of throughput rate is reported to be 1.8 g-min⁻¹ to 2.3 g-min⁻¹. With the existence of TPOSS, the mechanical properties of compound are slightly increased. TPOSS are added for mechanical properties enhancement. The polymer compound presents the excellent properties. It is remarkable to note that recycled polyethylene terephthalate is considered as an excellent candidate for use as raw material for polymer compound production.

1. Introduction

Nowadays, with the rapid increment of worldwide population, numerous types of plastic have been developed in order to facilitate users. They are cheap, lightweight as well as ease of process. Plastic can be designed to numerous shapes of material with additional feature of transparency and flexibility. Therefore, it is considered as versatile material for modern economy [1]. The role of plastic is employed in various sectors of industry including food and beverage packaging [2], automotive part [3] as well as artificial textile for clothing [4]. Up to the present time, although utilization of plastic significantly offers various benefits in terms of engineering properties, the usage is still in trouble due to difficulty of degradation and risk of environmental issue. It was considered as a waste for landfill, providing any hazardous chemicals to soil, water and air pollution [5]. Up to the present time, in order to protect the world from plastic waste, numerous strategies are promoted such as to avoid single-use plastic, to replace with biodegradable plastic as well as to recycle plastic waste for various purposes [6,7]. The objective or plastic waste management is to ensure on the minimal of plastic use and to maintain the value-add to plastic waste. One of the most important policies for plastic waste management is typically referred to "Circular economy (CE)" [8,9]. This policy is regarded as a model of production and consumption involving reusing, repairing, leasing as well as sharing platform of plastic waste. It is remarkable to note that the target of circular economy is due to waste elimination, product and material circulation as well as regeneration to nature.

To achieve this goal, recycled polyethylene terephthalate (rPET) is considered as one of the most abundant plastic wastes in municipal area. It is presented as food and beverage packaging waste. After usage, recycled polyethylene terephthalate is useless and provided the difficulty to manage. One effective way to solve this issue is regarded to evaluate the strategy of recycling. From the fundamental point of view, it is structurally defined as a thermoplastic polymer of the polyester class [10]. The application is commonly employed as a fiber for clothing and food packaging. Recycled polyethylene terephthalate (rPET) is remarkably considered as outstanding candidate for circular economy. One of important tasks of recycled polyethylene terephthalate (rPET) is to employ as raw material for polypropylene compound preparation. With the presence of recycled polyethylene terephthalate (rPET), significant enhancement of mechanical properties of polypropylene is therefore obtained by using low-cost material.

In order to design the compound based on plastic waste, the presence of polypropylene grafted maleic anhydride (PP-g-MA)

should be considered as a compatibilizer. It significantly provides the interfacial adhesion improvement by chemical bonding of the anhydride group throughout binary blend network. From structural viewpoint, it is expected to create the strong interaction by oxygencontaining functional groups, leading to good dispersion during melt process, as suggested by Li *et al.* [11]. Furthermore, in order to develop binary blend of compound, numerous techniques are involved. Recently, Zhou *et al.* [12] evaluated the role of cold plasma for recycling plastic waste modification. It can be tailored the adhesion for compound, and it evidently provided the ability for large scale of production. Silvia *et al.* [13] investigated the existence of banana starch onto recycled polyethylene terephthalate (rPET). The crystallinity and morphology of compound were then changed, resulting to ease of degradation process and time.

Furthermore, in order to enhance the mechanical properties of recycled polyethylene terephthalate (rPET), the integration of polyhedral oligomeric silsesquioxane (POSS) into compound is considered as one of the alternative routes for compound production [14]. It is structurally noted as a class of nanoparticles with a cage-like structure, offering high melt elasticity. Recently, Hu *et al.* [15] developed POSS and polyhydroxy urethane compound. It offered the thermal stability along with rubbery plateau modulus, resulting in excellent creep resistance at high temperature. Zhao *et al.* [16] prepared POSS and thioether compound. It exhibited ultrahigh selectivity of Ag(I) ions over a wide pH range.

In order to use recycled polyethylene terephthalate (rPET) with additional benefit, it was developed as a compound for polypropylenebased composite by melt spinning technique. TPOSS was integrated into compound for mechanical properties enhancement. Fourier transform infrared and X-ray diffraction technique were employed to evaluate the functional group and crystallinity. Thermogravimetric analysis and differential scanning calorimetry were used to investigate thermal properties. Scanning electron microscope was employed to investigate morphology. Mechanical properties were then reported.

2. Experimental

2.1 Materials and chemical reagents

Polypropylene (with the melt flow index (MFI) of 25 g·10 min⁻¹) was provided from IRPC Public Company Limited, Thailand. Recycled polyethylene terephthalate (rPET, of the I.V. grade of 0.80 dL·g⁻¹) was obtained from Thai Shinkong Industry Corporation Limited, Thailand. They were employed as starting for compound development. Fusabond P353 (with the melt flow index (MFI) of 22.4 g·min⁻¹)

was obtained from the Dow Chemical Innovation Company Limited, Thailand. Its chemical structure was noted as polypropylene-graftedmaleic anhydride call instead with (PPMA). It was employed as compatibilizer. TPOSS was purchased from Hybrid Plastic Inc., USA. All of chemical reagents were used as received without further purification.

2.2 Methodology

2.2.1 Preparation of polypropylene and recycled polyethylene terephthalate-based compound

Prior to compound preparation, polypropylene (PP), polypropylenegrafted-maleic anhydride (PP-g-MA) and TPOSS pellets were dried at 80°C for 12 h, while recycled polyethylene terephthalate (rPET) was dried at 170°C for 4 h. These techniques were conducted in order to prevent the moisture absorption onto pellets. After that, the mixture was prepared by hand shaking in a plastic bag in order to ensure the uniformity. Table 1 reports the formulation of polypropylene and recycled polyethylene terephthalate-based compound with the addition of TPOSS. After that, it was proceeded by using melt-spinning technique.

2.2.2 Fabrication of polypropylene and recycled polyethylene terephthalate-based compound by melt-spinning technique

After ensuring the uniformity of mixture, it was fabricated by melt-spinning technique. This technique composed of a lab-scale single-screw melt spinning process (Haake, Germany). The mixture was melted at temperature profiles of $220/240/250/260^{\circ}$ C with a screw speed of 4 rpm. The melted polymer was extruded through the 8-spinneret spin pack with a diameter of 0.32 mm of the round shape. The fibers were spun at the drawing unit with a take-up speed of 35 m·min⁻¹ and then collected at the winding unit with a speed of 40 m·min⁻¹. All of products were stored in desiccators in order to prevent the moisture absorption.

2.2.3 Surface modification of polypropylene and recycled polyethylene terephthalate-based compound by heat treatment

The polypropylene and recycled polyethylene terephthalatebased compound with an addition of TPOSS was conducted on heat treatment process. It was annealed by transferring into a rotating heat set unit at 10 min⁻¹. The temperature was set up at 140°C and then collected at winding unit with a speed of 40 m·min⁻¹.

Table 1. Formulation of polypropylene and recycled polyethylene terephthalate-based compound with the addition of TPOSS.

Samples		Co	Process		
	PP (wt%)	PPMA (phr)	rPET (wt%)	TPPSS (phr)	
PP	100	-	-	-	Melt-spinning
PP/PPMA	100	10	-	-	Melt-spinning
PP/PPMA/rPET	80	10	20	-	Melt-spinning
PP/PPMA/rPET/1TP	80	10	20	1	Melt-spinning
PP/PPMA/rPET/3TP	80	10	20	3	Melt-spinning
PP/PPMA/rPET/5TP	80	10	20	5	Melt-spinning
PP/PPMA/rPET/3TP-HT	80	10	20	3	Melt-spinning, Heat treatment

2.3 Characterizations

2.3.1 Mechanical properties determination

Mechanical properties of melt spun fibers were tested according to ASTM D2256-10 standard method using tensile testing machine (Instron, model 5566, USA). The extension rate of 20 mm min⁻¹ and gap length of 2.5 cm with a load cell of 50 N were employed. The tenacity and elongation at break were reported a statistical average and standard deviation of 20 specimens. The initial modulus and Young's modulus were presented as a statistical average and standard deviation of 5 specimens.

2.3.2 Scanning electron microscope (SEM)

The morphological properties of melt spun fibers were observed by using Field Emission Scanning Electron Microscope (FE-SEM, JEOL JSEM7800F, Japan). The fiber specimen was cut into 1 cm pieces and mounded on carbon tape. Then, it was sputtered with a thin layer of gold for 45 s at 23 mA in order to enhance the conductivity. The fibers were scanned at an acceleration voltage of 2 kV. The magnification was set to be 200X and 2kX, respectively.

2.3.3 Differential Scanning Calorimetry (DSC)

Thermal properties and crystallinity percentage of melt spun fibers were determined using Differential Scanning Calorimetry (DSC 3+, Mettler Toledo, Switzerland). The samples were weighted for 10 mg each, packed, and sealed in an aluminum pan. Samples were analyzed under nitrogen gas at a flow rate of 40 mL·min⁻¹. The samples were first heated from 25°C to 300°C at 10°C·min⁻¹, then held for 3 min to eliminate their thermal history. Subsequently, the samples were cooled down at cooling rate of 10°C·min⁻¹ to 25°C and held for 3 min. The second heating was from 25°C to 300°C at 10°C·min⁻¹.

Degree of crystallinity was calculated from the following Equation (1).

$$X = \frac{\Delta Hm - \Delta Hcc}{Wf \cdot \Delta Hm^{\circ}} \times 100 \tag{1}$$

where X is the degree of crystallinity (%), ΔHm is melting enthalpy (J.g⁻¹), *Wf* is weight fraction, and ΔHm° is melting enthalpy of pristine polypropylene which equal to 207.33 J.g⁻¹ [17], pristine polyethylene terephthalate which equal to 140.10 J.g⁻¹ [18].

2.3.4 Thermogravimetric analysis (TGA)

Thermal stability of melt spun fibers was investigated by Thermogravimetric analysis (TGA 2, Mettler Toledo, Switzerland). The samples were run under nitrogen gas at a flow rate of 40 mL·min⁻¹. The samples were weighted for 10 mg and transferred into the crucible and heated at 25°C to 800 °C with a heating rate of 10°C·min⁻¹.

2.3.5 X-ray diffraction (XRD)

The crystalline pattern and crystallinity percentage of samples were analyzed by X-ray Diffractometer (XRD, Bruker - D8 Advance model) with CuK α radiation. It was equipped with diffractive program. Fibers were aligned on a sample holder and then scanned from the 2 θ range of 5° to 80° at scan rate of 0.02° min⁻¹. The crystallinity percentage (Xc, XRD %) was calculated by the following equation.

Crystallinity percentage was calculated from the following Equation (2).

$$Xc = \frac{Ac}{A_{c+A_{s}}} \times 100 \tag{2}$$

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where Xc is the crystallinity percentage, Ac is area below crystalline peaks in the 2θ range of 10° to 30° and Aa is area below amorphous halo.

2.3.6 Fourier transform infrared (FTIR)

The chemical structure of samples was determined by using Fourier Transform Infrared Spectroscopy (FTIR, Perkin Elmer, model 2000). The samples were recorded in the wavenumber between 4000 cm⁻¹ to 400 cm⁻¹ at a resolution of 4 cm⁻¹ and 64 scans.

3. Results and discussion

Polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS was successfully prepared. The purpose of recycled polyethylene terephthalate integration into polypropylene compound was due to value-added concept. With the presence of recycled polyethylene terephthalate, less amount of neat polypropylene was used. The presence of TPOSS was due to mechanical properties enhancement.

Figure 1 illustrates the FTIR spectra of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS. No significant change on functional group was observed based on variation of composition and surface heat treatment. The pristine polypropylene and polypropylene-grafted-maleic anhydride were provided for comparison. No peak due to O-H stretching was observed, suggesting that surface of fiber did not adhere with water molecule.



Figure 1. FTIR spectra of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS.

It was remarkable to note that the peak position at 1375 cm⁻¹ was observed due to the C-H stretching (CH₃) [19]. This was probably referred to polypropylene. Furthermore, the peaks were observed at the wavenumber of 1242 cm⁻¹ and 728 cm⁻¹. These peaks were attributed to the vibration of the C-C-O and C-H stretching respectively [20]. It may refer to recycled polyethylene terephthalate. After that, the prominent peaks at 2800 cm⁻¹, 1650 cm⁻¹, and 1075 cm⁻¹ were observed due to the vibration of the C-H stretching, C=O stretching and Si-O-Si stretching, respectively [21,22]. These peaks were corresponded to TPOSS.

Figure 2 exhibits the XRD pattern of polypropylene and recycled polyethylene terepthalate-based compounded with an addition of TPOSS. The peaks at 20 of 14°, 17°, 19°, 21°, and 22° were presented. These peaks were corresponded to planes of (110), (040), (130), (111), and (041) respectively [23]. These miller indexes were belonged to polypropylene. However, the peak position at 20 of 26° was presented. This peak was corresponded to (100) [24]. It can refer to recycled polyethylene terephthalate. It was significantly noted that the peaks belonged to polypropylene exhibited strong intensity. It can be therefore observed that polypropylene was the main component of compound. No peak related to TPOSS was observed due to insufficient amount.

To investigate the successful binary blend of polypropylene and recycled polyethylene terephthalate-based compound with an addition of TPOSS, DSC thermogram was therefore employed. This technique allowed us to imply the compatibilizer among all compositions. Figure 3 reports the DSC thermogram of binary blend of polypropylene and recycled polyethylene terephthalate-based compound. The experiment can be classified into two stages. 1st heating and cooling experiments were employed to indicate the melting temperature and crystallization temperature region, respectively. All characteristic curve presented in the similar feature. The melting temperature was reported in the range of 162°C to 167°C, due to melting temperature of PP and glass transition temperature of PET comes near each. Therefore, melting temperature and glass transition temperature of two different polymer has also been previously reported [25]. While the crystallization temperature was found in the range of 115°C to 120°C. This was in agreement with previous work of Zhang et al. [26]. Due to small portion of recycled polyethylene terephthalate, no significant change due to both temperatures cannot be observed. The existence of polypropylenegrafted-maleic anhydride (PP-g-MA) can be successfully linked between polypropylene and recycled polyethylene terephthalate throughout network. It can be induced to decrease the crystallization temperature of rPET. As a result, it effectively provides the excellent compatibility, as indicated in FESEM analysis [26]. In addition, the presence of TPOSS did not provide any effect on thermal properties. This was associated with previous work of Li *et al.* [27].

Table 2 exhibits the thermal properties of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS. Melting and crystallization temperatures were still similar to Figure 3. Enthalpy of binary blend was still in the similar region. This data can be used to an energy-like property or state function, as suggested by Morais *et al.* [28]. Furthermore, it can be noted that the degree of crystallinity is typically related 45% to 70% for polypropylene and 4% to 16% for recycled polyethylene terephthalate. The degree of crystallinity is similar to XRD analysis reported in Table 3. The presence of TPOSS can be induced for crystallization in binary blend.



Figure 2. XRD pattern of polypropylene and recycled polyethylene terepthalatebased compound with an addition of TPOSS.



Figure 3. The DSC thermograms of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS.

Table 2. Thermal properties from DSC first heating and cooling of polypropylene and recycled polyethylene terephthalate-based compound with an addition of TPOSS.

Samples	1 st Heating			Cooling	Degree of crystallinity (%)	
	T _{cc}	Tc	ΔH_m	Te	X _{PP}	Xrpet
PP	-	163.4	99.9	119.7	46.9	-
PP/PPMA	-	164.4	91.5	117.2	47.9	-
PP/PPMA/rPET	117.5	167.8	73.5	116.8	48.8	4.6
PP/PPMA/rPET/1TP	116.9	164.7	94.9	115.8	50.0	12.9
PP/PPMA/rPET/3TP	117.3	161.1	98.8	115.9	56.2	14.9
PP/PPMA/rPET/5TP	116.2	162.7	105.0	116.0	57.4	15.7
PP/PPMA/rPET/3TP-HT	112.2	166.3	117.4	118.8	69.6	15.0

Table 3. Crystallinity percentage of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS fibers from XRD.

Samples	Crystallinity percentage (%)		
PP	42.4		
PP/PPMA	43.5		
PP/PPMA/rPET	47.6		
PP/PPMA/rPET/1TP	50.5		
PP/PPMA/rPET/3TP	54.5		
PP/PPMA/rPET/5TP	56.3		
PP/PPMA/rPET/3TP-HT	78.8		



Figure 4. Morphological properties of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS (a) polypropylene, (b) polypropylene and recycled polyethylene terepthalate binary blend, (c) with 1 phr of TPOSS, (d) with 3 phr of TPOSS, (e) with 5 phr of TPOSS, and (f) with 3 phr of TPOSS after heat treatment.

Figure 4 illustrates the morphological properties of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS. The microstructure investigated by magnification of 200X and 2kX was presented on the left side and right side, respectively. It was observed that excellent compatibility can be occurred for binary blend composite. With low magnification, it was reported as a long fiber chain with random orientation. Meanwhile, with high magnification, it presented only surface of fiber. It reported the smoothness of surface. With the presence of TPOSS, it was observed as the white spot distributed onto the surface of binary blend. The red arrow was employed to indicate the position of white spot. Furthermore, it could be disappeared when binary blend was undergone with heat treatment process. The white-spot is referred to flake of rPET. When, re-heat treatment process was conducted. It may disappear [29].

One of the most important properties for polypropylene and recycled polyethylene terepthalate-based compound with an addition

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of TPOSS was focused on thermal decomposition behavior. This property can be used to imply the presence of TPOSS in binary blend compound. Figure 5 shows the thermal decomposition behavior of binary blend compound with an integration of TPOSS. It was observed that all of curves typically presented in the similar feature. From ambient temperature to 300°C, no weight loss was observed. This can be explained that binary blend compound presented high thermal stability within this temperature region. It can be implied that the application prepared by this compound can be used without the concern over thermal degradation. After that, in the temperature region of 300°C to 500°C, broad range of thermal decomposition behavior was occurred. This was probably due to the fact that pyrolysis of organic compound occurred. It can change to volatile gas. Furthermore, when the temperature was increased to region of 500°C to 800°C. No weight loss was observed. It was indicative that binary blend with TPOSS was completely decomposed. However, the inset figure was also presented. The enlargement of temperature region between 300°C and 450°C was provided. The decomposition was slightly changed with respect to the amount of TPOSS. It can be implied that TPOSS was existed, however, no significant change due to thermal decomposition behavior was reported.

In order to investigate the feasibility of flow behavior of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS, throughput rate was considered as one of the important parameters to predict the rheological behavior [30]. This technique allowed us to investigate the flow behavior of polymer melt with respect to viscosity and molecular weight. The flow behavior of compound could be used to predict the compatibility of polypropylene and recycled polyethylene terephthalate, as suggested by Faizi et al. [31]. It was observed that the data of throughput rate was presented in the similar level. It was estimated to be 2 g min⁻¹. No significant change was observed compared to pristine polypropylene. It was therefore noted that with the presence of recycled polyethylene terephthalate, it was successfully grafted by polypropylene-graftedmaleic anhydride. This was the identical for TPOSS integration. It was therefore remarked that polymer compound was successfully prepared with excellent compatibility.

One of the most important features of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS was focused on mechanical properties. Tenacity is typically referred to the ability of material to resist the effect of tensile forces without any rupture. It can be explained that with the presence of recycled polyethylene terephthalate, the tenacity was in the similar level compared to pristine polypropylene. No significant change of tenacity was observed. It can be implied that recycled polyethylene terephthalate can be replaced portion of pristine polypropylene. Furthermore, with the integration of TPOSS, the tenacity was slightly shifted to be higher. It can be explained by the presence of silica atom. This atom can be crosslinked throughout binary blend network. This was in agreement with previous literature of Wang et al. [32]. Furthermore, it can be noted that with heat treatment process, it affected on the orientation of fiber, significant enhancement was therefore obtained. Due to the heat treatment process can change the structure of fiber and improve the properties [33]. Which results present by XRD, and tenacity can be used to investigate the structural properties of material after heat treatment process.

Next to tenacity, elongation at break was considered as one of important parameters in order to determine the mechanical properties. This data was typically presented the rupture point of fiber. It can be used to indicate the flexibility [34,35]. With the presence of recycled polyethylene terephthalate, the elongation at break of binary blend was significantly shifted to lower. This was probably due to the incompatibility between matrix and interface in binary blend composite [36], which result in elongation at break of binary blend was lower. Also, with the existence of TPOSS, so significant reduction was observed compared to binary blend. The presence of silicon atom may interfere the pathway of load direction during tensile measurement. Moreover, it was remarkable to note that with the heat treatment process, elongation at break was evidently decreased. This technique may provide the molecular chain movement for binary blend.







Figure 6. Throughput rate of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS.



based compound with an addition of TPOSS.





Figure 8. Elongation at break of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS.



Figure 9 Initial modulus at 1% strain and Young's modulus of polypropylene and recycled polyethylene terepthalate-based compound with an addition of TPOSS

Figure 9 reports the initial modulus at 1% strain and Young's modulus of polypropylene and recycled polyethylene terephthalatebased compound with an addition of TPOSS, respectively. It was observed that recycled polyethylene terephthalate could be replaced into polypropylene. The data of modulus were presented in the similar region. With the presence of TPOSS, modulus was slightly shifted to higher. It can be induced for the crystallization in binary blend. Furthermore, by strategic technique of heat treatment, it affected to the re-orientation of crystallization. The order of crystal exhibited in good form. It resulted in superiority of modulus.

4. Conclusion

Recycled polyethylene terephthalate was successfully prepared as a powder form by nano-grinder technique. It is fabricated as a polymeric compound with polyethylene. The PP-g-MA and TPOSS are added for compatibilizer and mechanical properties enhancement, respectively. Fourier transform infrared confirms the presence of TPOSS. X-ray diffraction presents the single of compound. Heat treatment can induce the crystallinity as observed by strong intensity. Scanning electron microscope illustrates the smoothness of surface, indicating that compound exhibits the excellent miscibility. No crack is observed. Furthermore, it was thermally stable at room temperature. PP-g-MA and TPOSS do not significantly provide the change of melting and crystallization temperature. Mechanical properties of compound are therefore increased. It is remarkably noted that recycled polyethylene terephthalate powder can be employed as a raw material for plastic production. This project provides the benefit for "Circular economy" policy.

Author contribution

Rujira Phumma: Conceptualization, Methodology

Chutima Vanichvattanadecha: Validation

Sarute Ummartyotin: Writing-original draft, Writing-Review& Editing, Supervision, Resources

Acknowledgement

This study was supported by Thammasat University Research Fund, Contract No TUFT 354 5/2566. The author would like to acknowledge the support from Thammasat University Research 355 Unit in Textile and Polymer Chemistry.

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