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Effect Of Additives On The Quality Of PLA Melt Electrospun Fibers As Oil Sorbents

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الملخص

تم تصنيع ألياف حمض البولي لكتيك النقي (pure polylactic acid) وهي ألياف فائقة النعومة بإستخدام أسلوب أو تقنية (melt differential electrospinning) لغرض إختبارها كمادة ماصة قابلة للتحلل في حالات إزالة أو تنظيف حوادث تسرب النفط . تم إستخدام ثلاث أنواع من الزيوت وهي (زيت المحركات – النفط الخام – زيت الديزل) وكانت قدرة الإمتصاص للزيوت الثلاثة 96,67,53 جم/جم على التوالي ، وكان متوسط قطر الألياف 6.57µm , وزاوية تلامس الماء 138 درجة . وقد أظهرت النتائج المتحصل عليها بأن الياف حمض البولي لأكتيك النقي المصنعة لم تكن مرشحا أو أختيارا موفقا لهذه المهمة ، وذلك لعدة أسباب منها : قدرة إمتصاص منخفضة ، قطر ألياف كبير ، و معدل تحلل أعلى . من أجل تحسين جودة الألياف المصنعة تم إستخدام (Sucrose fatty acid ester)كمادة مضافة وتم تصنيع ألياف فائقة النعومة بإستخدام نفس الأسلوب أو التقنية وكان تأثير المادة المضافة واضحا . النتائج أظهرت أن الألياف المغزولة كهربائيا تم تحسينها (refined) الى قطر قدره 1.73 µm في المتوسط مع زيادة قدرة امتصاص بمعدل 96، 61، 126جم/جم على التوالي وهو أعلى بكثير من ذلك الخاص بالحمض النقي ، وكذلك تحسنت الخواص الميكانيكية للألياف المصنعة ، زاوية تلامس الماء 135 درجة ، ولوحظ كذلك إنخفاض في معدل التحلل . وصل معدل الإمتصاص الإنتقائي للألياف مع المادة المضافة الى 1000، وهي كذلك تتميز بقدرة طفو عالية وقابلة لإعادة الإستخدام.

الكلمات المفتاحية : تسرب نفطي, قدرة إمتصاص الزيت, مادة ماصة, ألياف فائقة النعومة, تطبيقات بيئية.

Abstract

Ultra-fine fibers of pure polylactic acid (PLA) were fabricated using melt differential electrospinning technique, to be tested as biodegradable sorbent material in oil spill cleanup situations. Three different oils were used (engine oil – crude oil – diesel oil), and the sorption capacity of the three oils were 96, 67, 53 g/g respectively, the average fiber diameter was 6.57µm, water contact angle of 138 degrees. The obtained results showed that pure PLA fibers were not a good candidate for the job, because of many reasons, such as low sorption capacity, large fiber diameter, and higher degradation rate. Sucrose fatty acid ester (SE) was used as an additive in order to improve the quality of the produced fibers, and ultra-fine fibers were made using the same technique, the effect of additive was obvious , results showed that the electrospun fibers were refined to an average diameter of 1.73µm, with sorption capacity increase of 126, 96, and 61 g/g respectively which is significantly higher than that of pure PLA, also mechanical properties of the fabricated fibers were improved , water contact angle of 135 degrees, and reduction in degradation rate was observed. Water-oil selective absorption rate of PLA/SE fiber reached almost up to 1000, and is also of big flotage and is reusable.

Keywords*: Oil spill, oil sorption capacity, sorbent material, melt differential electrospinning, ultra-fine fibers, environmental applications.*

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1. INTRODUCTION

The great and serious impacts of Heavy and crude oil spilled by any mean to the environment, especially to the ecosystem which could remain contaminated in unpredictable ways for years or even decades to come, the health effects to workers, volunteers, and local residents, call for an urgent need to find ways, techniques, and develop materials for cleaning up spills from the impacted areas, which still remain a challenge to scientists and decision makers of all kind .Many processes have been developed and applied to clean up oil spills from those impacted areas, such as mechanical extraction, in situ burning , and bioremediations[1- 3] , among which physical recovery by the concentration and transformation of oil from the liquid phase to the semisolid or solid phase with oil sorbents is considered to be one of the most efficient and cost effective countermeasures[4] . An ideal sorbent material for oil spill cleanup should have oleophilicity, hydrophobicity, high uptake capacity, high rate of uptake, and buoyancy [5]. At present, the sorbents used for oil sorption mainly include natural materials, inorganic mineral products, and organic synthetic fibers [6], natural materials include , milk -weed floss, cotton, and kapok, have shown low hydrophobicity, and a relatively high oil sorption capacity of approximately 20-50g/g [2,6,7,8], whereas wool, kenaf, and sisal had a low sorption capacity of 8-15g/g [6,10]. The mineral products (i.e., perlite and vermiculite) showed insufficient buoyancy and very low oil sorption capacity 6g/g [2]. Nonwoven polypropylene (PP) fibrous mats, have been widely used as synthetic fibers in oil spill cleanup, because of their oleophilic- hydrophobic properties, good oil /water selectivity, high buoyancy, and mass production, but they suffer from a low oil sorption capacity, approximately 15-30g/g [6], the reason is they are made of solid fibers with large diameters.

In recent years, some researchers utilize electrospinning technique to prepare Ultra-fine fibers and obtained oil absorption material with super high oil adsorption rate and good hydrophobicity, which are being applied into oil-spilled treatment applications ［11］. However, it still remains unresolved problem that synthetic oil absorption material is biologically non-degradable. Polylactic acid (PLA) it is biodegradable aliphatic polyester, which has an outstanding advantage over other polymers, and the four most attractive of those advantages are: renewability, biocompatibility, processability, and energy saving. It is easily degradable at high temperatures during the process of Melt electro spinning. Polylactic acid monomer, the raw lactic acid, can be extracted from renewable plant resources (such as maize, wheat) in a very large scale with no pollution [12, 17]. In the natural environment, under the action of micro-organisms, PLA will be completely degraded into carbon dioxide and water, then participate in process of photosynthesis and become the raw material of starch, realizing the nature cycle. So, it is environmentally friendly. PLA production process is shown in (Figure 1). PLA is well suited for melt-spinning into fibers. Compared to the solvent-spinning process required for synthetic cellulosic fibers, melt spinning allows PLA fibers to be made with both low cost and minimum environmental effects., and allows the production of fibers with a wider range of properties [18-24].

In this study, biologically degradable PLA/SE fibers and PLA fibers were produced were prepared by self-designed air assisted melt electrospinning device. The fibers were tested for the use as oil sorbent in oil spill cleanup situation, characterizing its morphology and hydrophobicity by contact angle and scanning electron microscope (SEM), and experiments were designed to analyze oil adsorption rate, flotage, and reusability of the electrospun fibers.

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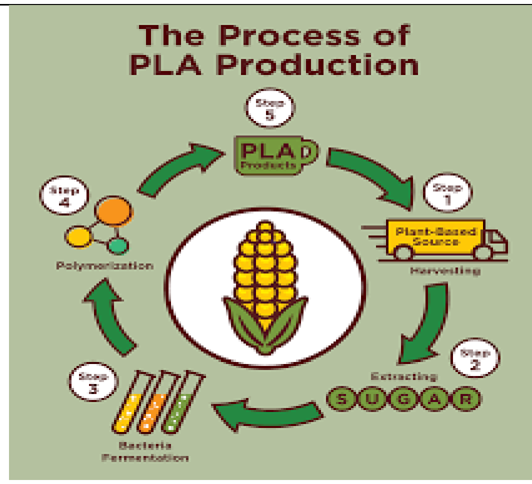

Figure 1. Production process of PLA

2. EXPERIMENTAL

2.1. Materials

PLA (2002D - Nature Works LLC company) a specific gravity of 1.24g/cm3, melting point 210℃, melt flow rate of 8 g/10 min, and dried at around 80℃ for about 3 hours before spinning. Three oil samples were used as some of the major water pollutants in oil spill situations (Engine oil, crude oil, and diesel oil). The physical characteristics of oil at 25 ± 1 °C and a relative humidity (RH) of 25 % ± 1 % are shown in Table 1. Artificial seawater (3.5 wt. % NaCl). High purity sucrose fatty acid ester, SE-1G, total ester content≥92%;

Table 1. Physical characteristics of oil used

Properties	Motor oil	Diesel Oil	crude oil
Density. g/ cm3 (at 25 ± 1 °C)	0.871	0.803	0.865
Viscosity. mPa s (at 25 ± 1 °C)	229	12	132

2.2. Melt differential electrospinning device

Self-designed air assisted melt differential electrospinning device (Figure 2) was used in this experiment, which composed of: high-voltage power supply, heating system, an air pressure device, a needleless cone-shaped nozzle, and a collecting device. The high-voltage supply device could provide a maximum output of 100 kV and a maximum current output of 2 mA. The unique features of our device are: the generation of multiple Taylor cones around the bottom edge of the nozzle, resulting in a high throughput; the air current driven by an air pressure system further strengthened the stretching force acting on the jets. More importantly, it could contract the flying jets with the pressure difference between the air current and the atmosphere to prevent the polymer jets from being attracted by the inner circle of electrode A; and two kinds of collecting devices could be applied whenever needed, namely, the nonwoven and electrode B. The nozzle was

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grounded to prevent any kind of interference between the high-voltage supply and temperature sensors;

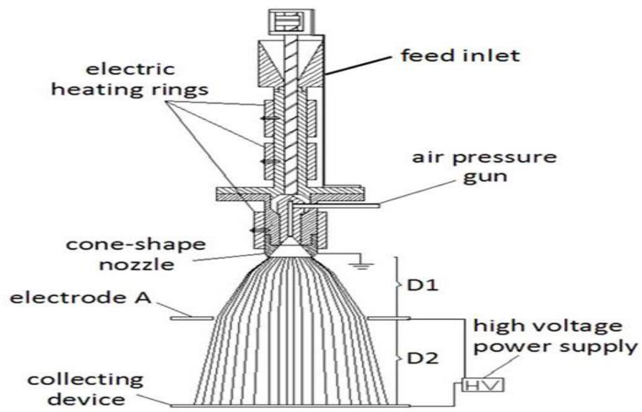

Figure 2. Schematic of the melt differential electrospinning device

2.3. Electrospinning process:

Pure PLA grains and the melt blending granular materials of PLA and SE of 8% mass fraction were fed through the feeding port at a rate of almost 5.5 g/h, as it was calculated from the weight of the spun fiber per unit of time. Heating temperature control device was set on to start the heating process. The process of melt differential electrospinning started to obtain pure PLA and PLA / SE ultrafine fibers respectively. The spinning voltage was 60 kV, the distance between the tip and collector was 12 cm, the heating temperature was 210℃.

3. CHARACTERIZATION

Several characterization techniques including (but not limited to) SEM, contact angle were used in this experiment. The fiber diameter and surface morphology of the electrospun PLA fibers were examined using SEM (S4700, Japan Hitachi Company). Before SEM observation, all of the fibers were coated with layer of platinum before observation, and scanning voltage was 20 kV. Mean diameter and standard deviation of the fiber was taken. Four microliters of deionized water droplets were dropped on the surfaces of 0.1 g of sample in five different positions. Measurements and calculations of contact angle done by using OCA20 contact angle tester (Germany Data physics company) 5 times and use the average as the contact angle.

3.1. Measurements of oil sorption capacity (OSC)

In order to study the oil-sorption capacity of the electrospun PLA fibers, the dried sample (0.1 g) was put into a stainless-steel mesh weighed beforehand and immersed in oil at room temperature. The sample and the mesh were taken out from the oil together after an hour (60 min), drained for 1 min, and wiped with filter paper to remove excess oil from the bottom of the mesh. The oil sorption capacity of the sample was determined by weighing the samples before and after the sorption using a digital balance, and calculated by the following equation:

$$Q = (W_t - W_i - W_w) / W_i$$

Where Q is the oil sorption capacity of the sorbents calculated as grams of oil per gram of sample, W_t is the weight of the wet sorbents after draining (g), W_i is the initial weight of sorbents (g) and W_w is the weight of water absorbed in the sorbents (g). All tests were triplicate and the average of the three runs was taken for calculation. In pure oil without any water, W_w is equal to zero.

3.2. Evaluation of cyclic sorption/desorption characteristics (Reusability Test)

This experiment evaluated reusability of the PLA and PLA/SE fibers for cyclic oil sorption/desorption. The oil recovery process was performed by squeezing out the absorbed oils from the oil saturated sorbents. The squeezed sorbent was used again in the sorption process. The weights of samples and the oil squeezed out were measured in each cycle. The sorption/desorption process was repeated for the desired number of cycles which was in our case six cycles to evaluate the reusability of the PLA and PLA/SE fibers.

4. RESULTS AND DISCUSSION

4.1. Morphology and hydrophobicity analysis of pure PLA and PLA /SE fiber

The SEM images of the melt electrospun PLA and PLA/SE fibers are shown in (Figure 3), from which we can see that the surface of the fibers were smooth, indicating that fibers had higher tensile strength compared with those fibers that have poor mechanical properties from solution electrospinning. The mean diameter of PLA / SE fiber is 1.73µm while the pure PLA fiber is 6.57µm, which indicated that adding SE has significant effect on fibrous refinement. The reason behind that is because the SE has improved melt flow ability, promoted the slip and untwisted of polymer chain, thinner the melt jet in electric field. PLA/SE melt with better flow ability stay the shorter time at the end of umbrella nozzle, reducing the thermal degradation of the melt. Different from the traditional pinhole-type nozzle, under the action of electric force melt at the end of nozzle spontaneously form cycle of “Taylor cone” and will not degrade because no fraction with the hole wall. PLA ultrafine fibers can be obtained by using melt differential electrospinning nozzle, and addition of SE plasticizer, low degradation rate of PLA can improve and ensure the mechanical properties, which are of great benefit in oil absorption.

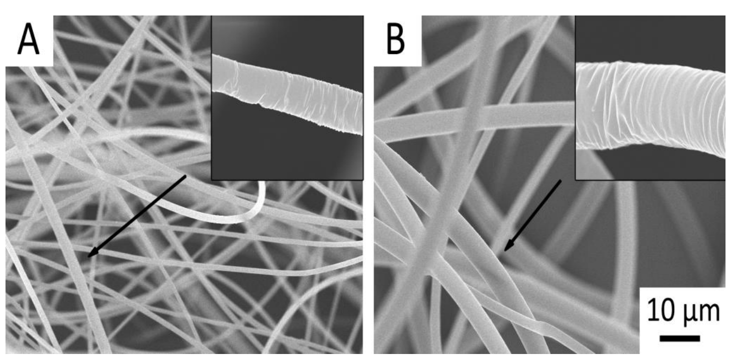

Figure 3. SEM images of PLA/SE fiber (A) and pure PLA fiber (B)

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Usually, if the contact angle of water is less than 30°, the surface is designated hydrophilic since the forces of interaction between water and the surface nearly equal the cohesive forces of bulk water, and water does not cleanly drain from the surface. If water spreads over a surface, and the contact angle at the spreading front edge of the water is less than 10°, the surface is often designated as super hydrophilic provided that the surface is not absorbing the water, dissolving in the water or reacting with the water. On a hydrophobic surface, water forms distinct droplets. As the hydrophobicity increases, the contact angle of the droplets with the surface increases. Surfaces with contact angles greater than 90° are designated as hydrophobic. The theoretical maximum contact angle for water on a smooth surface is 120°. Micro textured or micro-patterned surfaces with hydrophobic asperities can exhibit apparent contact angles exceeding 150° and are associated with super hydrophobicity and the "lotus effect." Results of fiber contact angle were 135° for PLA/SE; and138°for pure PLA, which indicated that the two fibers have better hydrophobicity which is attributed to lower surface energy and micron fibrous diameter of PLA. For most thermoplastic polymers have lower surface energy, hydrophobicity could be improved by increasing its surface roughness. One way is to further refine fiber, producing 500 nm nanofibers. Another way is to prepare fiber with multistage structure like micro pore and micro groove.

4.2. Oil sorption capacity of pure PLA fiber and PLA /SE fiber

As shown in (Figure 4), oil sorption capacity of the PLA/SE fiber on engine oil, crude oil, and diesel oil are 126, 96, and 61 g/g respectively, while the oil adsorption rate of pure PLA fiber on this oil is 96, 67, and 53 g/g respectively. Thin fiber has a higher oil absorption rate can be attributed to the higher specific surface area and porosity. The oil adsorption rate of these two kinds of samples declines as the following trend: engine oil > crude oil> diesel oil. For the same kind of oil sorbents, the oil sorption capacity increases with the increased oil viscosity. High viscosity oil, will lead to two opposite results: increase the oil adsorption force on the fiber surface to increase sorption capacity, and preventing oil to enter fiber pores to reduce sorption rate. The former plays a more important role for the electrospun fiber being porous material. Oil sorption capacities of PLA/SE and pure PLA fibers used in this study and other reported oil sorbent fibers are compared in Table 2. Oil selectivity and water flotage are two important parameters in evaluating oil sorbents. In this experiment the oil sorption capacity in an oil-water system was tested, among which maximum oil sorption capacity of PLA/SE fibers on engine oil, crude oil, and diesel oil were 125, 93, and 66 respectively. Considering the experimental error, fiber oil sorption capacities almost have no difference in pure water and oil-water system. Water sorption capacity of PLA / SE and pure PLA was 0.07 and 0.11 g/g respectively, oil/water selectivity up to 1000 times. High flotage can make the material float in the sorption process, which is beneficial to oil sorbents recycling. In a static condition, the fiber samples can float absorbed after 60 min placed in surface. Under dynamic conditions, (oscillation rate 300 r/min), fiber samples showed good floating capacity and oil sorption capacity, which is attributed to low density, high porosity and lipophilicity-hydrophobicity of electrospun fibers.

Oil sorption/desorption curve of electrospun fiber is shown in (Figure 5). After the first oil desorption cycle, oil sorption capacity of fiber samples were dropped, only2/3 to 3/4to the new. The oil sorption capacity remained steady in second, third time. In the fourth time, pure PLA fiber partially turned into powder, leading to the sharp decrease of oil sorption capacity again. The pure PLA fiber completely turned into powder in consequent experiments, almost unable to absorb oil.

The reusability of PLA/SE fiber is slightly better, in the sixth time the oil sorption capacity maintained in 30 g/g, but some fiber also presents obvious fracture and powder. Thinner PLA/SE composite fiber has better reusability, which can be attributed to: (1) the flow rate of the melt in spinning process is higher, which shorten the time melt stay in the barrel and nozzle and lower the degradation rate; (2) the full stretch fiber jet improves crystal degree of the electrospun fiber.

Table 2. Comparison of sorption capacities of various oil-absorbing fibers

Fiber	Oil Type	Oil sorption capacity (g/g)	Ref
Raw cotton	Vegetable oil	30	3
Wool fiber based non-woven	Diesel oil	10.6	11
Melt electrospun PP fibers	Motor oil	129	4
Pure PLA fiber PLA/SE fiber	Crude oil Engine oil	67 126	This study

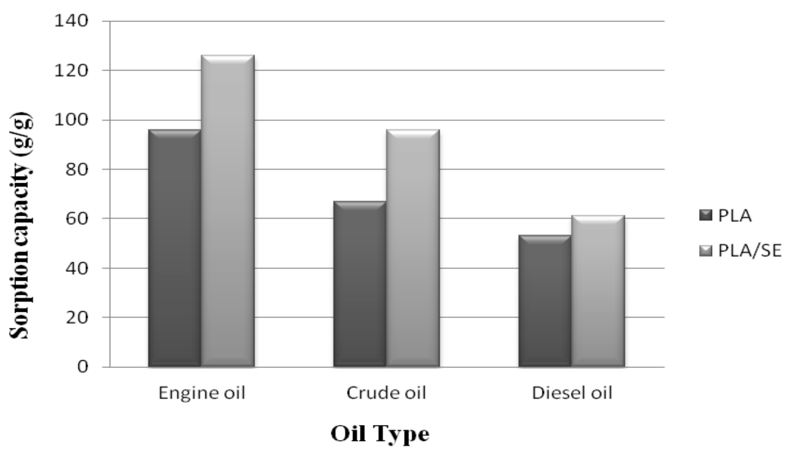

Figure 4. Oil sorption capacity of fiber samples for oils used

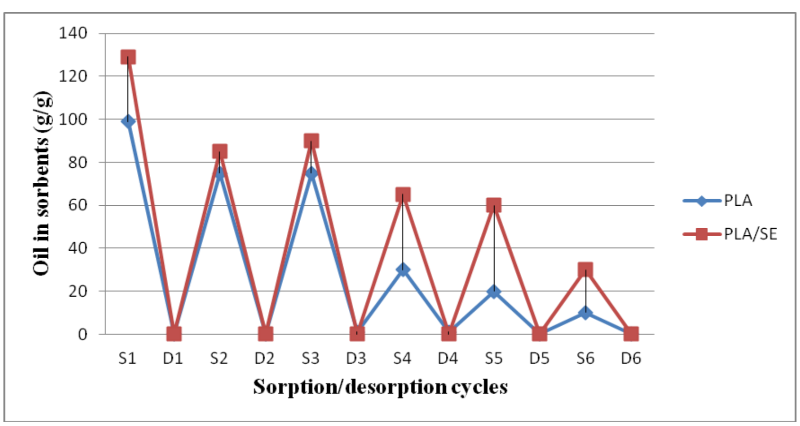

Figure 5. Sorption/desorption cycle curve. S1–S6 oil-sorption. D1–D6 oil desorption.

5. CONCLUSION

By the use of self designed melt differential electrospinning device, PLA/SE ultra- fine fibers and PLA fibers were produced. The fibers were tested for the use as oil sorbent in oil spill cleanup situation. Results showed , the average diameter of pure PLA fiber prepared through melt technique was 6.57 μm, and had hydrophobic structure, and that sorption capacities of pure PLA fiber with respect to engine oil, crude oil, diesel oil were 96, 67, and 53 g/g, which indicated low sorption capacity, large fiber diameter, and higher degradation rate. Addition of Sucrose fatty acid ester (SE) was necessary for the refinement of the produced fibers characteristics, and ultra-fine fibers were made using the same technique, the results showed that the electrospun fibers were refined to an average diameter of 1.73μm, with an increase in sorption capacity of 126, 96, and 61 g/g, a lot higher than pure PLA, also degradation rate was reduced. Water-oil selective absorption rate of PLA/SE fiber was very high, and is also of big flotage and is reusable. The applying of oil sorbent material that uses natural fibers with a safe, efficient method like melt differential electrospinning may bring up a great chance to mitigate the present environmental crisis issues, in particular the ones dealing with the serious water pollution all over the globe as a direct consequence of oil spill disasters and other industrial accidents.

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