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Sorption of oil from simulated seawater by fatty acid-modified pomelo peel

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ABSTRACT

A new, low-cost and locally available sorbent, pomelo peel (PP), was tested for its ability to remove spilled oil from simulated seawater. The experiments were conducted to evaluate the oil sorption capacities of PP modified by fatty acids (oleic acid and stearic acid). The effects of temperature, salinity, and oscillation frequency on the oil sorption capacity of the PP were studied in simulated seawater. It was found that the oil sorption capacity of the PP was greatly enhanced by the surface modification. The results showed that the PP modified by oleic acid had better oil sorption capacity than that treated with stearic acid. The sorption kinetics of unmodified and modified PP were well described by the pseudo-second-order kinetic model. The results indicated that PP was an attractive candidate for removing oily pollutants from seawater.

Keywords: Pomelo peel; Fatty acids; Oil spill; Sorption; Kinetics

1. Introduction

Crude oil is presently one of the most important energy sources in the world, and will hold its preeminent position as a fuel for several decades to come [1]. However, crude oil is unintentionally introduced into the environment during production, transportation, and refining process, and causes adverse effects on sea life and human economic activities [2]. When crude oil comes in contact with the water, it forms oil-in-water emulsion or floating film that needs to be removed before it is discharged into the environment [3].

The sorbent materials used for oil spill clean-up can be classified into three typical groups: synthetic polymers, natural fiber materials, and inorganic minerals. Synthetic polymers, such as polyurethane [4] and butyl rubber [5] have been widely used due to their hydrophobic and oleophilic characteristics. However, their slow degradation rate is a major disadvantage. Natural fiber materials usually show hydrophilic and relatively low sorption capacities, e.g. kapok [6], bagasse [7], and barley straw [8–10]. The inorganic minerals include fly ash [11] and exfoliated graphite [12], etc.

Pomelo (*Citrus grandis*), belongs to the Rutaceae, is native to southeastern Asia and China. Pomelo is widely planted in Guangdong, Guangxi, Fujian, Hunan, and Sichuan of China. It is the largest citrus fruit, growing as large as 30 cm in diameter and weighing as much as 10 kg; its rind is very thick but

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soft and easy to peel away. Pomelo peel (PP) is disposed and causes severe problem in the community. Zhou et al. [13] found that PP presented consistent macropore size distributions which centered at approximately 2–20 μm. Some literatures have reported that PP has been used as a sorbent to remove basic dye from aqueous solution [14], however, few literatures about the application of PP as an oil adsorbent material are found. PP tends to absorb water; its oil sorption capacity may be drastically reduced [15]. The primary objectives of this work are to (1) modify PP with oleic acid and stearic acid, and (2) evaluate the oil sorption capacity of the resulting PP.

2. Materials and methods

2.1. Materials

PP (the content of cellulose, hemicellulose, and lignin of PP in this study were 46.22, 18.84, and 10.24% by weight, respectively) was collected from nearby market as solid waste. The collected materials were washed with distilled water for several times to remove all the dirt particles, and dried in a hot air oven at 60℃ for 48 h, then stripped the yellow skin and cut into small pieces (1–2 cm), and dried in a hot air oven at 60℃ for 48 h, then stored in air tight container for further use.

Diesel (density at 20℃ = 0.8170 g cm^{-3}, viscosity = 6.51 $mm^2 s^{-1}$) was obtained from Minghe petrol station of Sinopec in Baoshan district, Shanghai; Lubricating oil (density at 20℃ = 0.7881 g cm^{-3}, viscosity = 47–57 $mm^2 s^{-1}$) was obtained from Shanghai Hasitai lubricating oil Co., Ltd. Oleic acid, stearic acid, and other reagents used were of analytical grades (Sinopharm Chemical Reagent Co., Ltd).

Simulated seawater for the experiments was deionized water mixed with sodium chloride to yield a 20, 25, 30, 35, and 40 parts-per-thousand saline solution.

2.2. Preparation of fatty acid-modified PP

One gram of PP was treated with 0.5 g of fatty acid (oleic acid or stearic acid) in the presence of 150 ml n-hexane and one drop of concentrated sulfuric acid (98% purity). The mixture was refluxed in a Dean–Stark apparatus at 65 ± 2℃ for 6 h. The treated PP were washed with ample amount n-hexane, then dried in an oven at 80℃ for 24 h, and stored in an air-tight container until use. The treated PP was denoted as oleic acid-treated pomelo peel (OAPP) (oleic acid-treated) and stearic acid-treated (SAPP), whereas for specific comparison purpose, the unmodified PP was denoted as unmodified pomelo peel (UMPP).

2.3. Batch kinetic studies

To the batch kinetic studies, samples were withdrawn at different time intervals up to the equilibrium. PP (0.1 g) was placed in a 50 ml beaker containing 30 ml of oil, (diesel or lubricating oil) maintained at 20℃, and left for 15 min without agitation. PP was then placed on the stainless steel mesh to drip away the sorbed oil from the sorbent for 10 min, then the oil-loaded sorbent was weighed. The sorption capacity (Q, in g g^{-1}) was calculated by

$$Q = \frac{m_f - m_0}{m_0} \tag{1}$$

where m_f is the weight of the wet material after draining (g) and m_0 is the initial weight of the dry material (g).

2.4. Effect of solution temperature

To investigate the effect of temperature on the oil sorption capacity, 0.1 g of sample was placed in a 50 ml beaker containing 2 ml of oil (diesel or lubricating oil) and 50 ml 20% simulated seawater maintained at 15, 20, 25, 30, 35℃, respectively, and left for 15 min with 24 rpm agitation.

2.5. Effect of solution salinity

To investigate the effect of salinity on the oil sorption capacity, 0.1 g of sample was placed in a 50 ml beaker containing 2 ml of oil (diesel or lubricating oil) and 50 ml simulated seawater (20, 25, 30, 35, 40%) maintained at 20℃ and left for 15 min with 24 rpm agitation.

2.6. Effect of oscillation frequency

To investigate the effect of oscillation frequency on the oil sorption capacity, 0.1 g of sample was placed in a 50 ml beaker containing 2 ml of oil (diesel or lubricating oil) and 50 ml 20% simulated seawater maintained at 20℃ and left for 15 min with 0, 24, 72, 120, 168 rpm agitation, respectively.

3. Results and discussion

3.1. FTIR

A stack plot of FTIR spectra of UMPP and OAPP is shown in Fig. 1. The identification of the important bands indicated in the spectra is based on previous studies of banana trunk fibers [16] and hemicellulose, cellulose, and lignin [17]. The very strong and broad band at 3,408 cm^{-1} is due to the stretching vibrations of hydroxyl groups present in cellulose, hemicellulose, and lignin of UMPP. The band at 2,921 cm^{-1} corresponds to C–H asymmetric stretching of CH_2-groups. The band at 1,633 cm^{-1} is attributed to the bending mode of the absorbed water [18]. The bands at 1,373 and 1,319 cm^{-1} represent OH bending and C–O skeletal vibrations, respectively. The band at 1,232 cm^{-1} corresponds to C–O stretching in hemicellulose. The band at 1,104 cm^{-1} corresponds to C–O antisymmetric bridge stretching and that at 1,053 cm^{-1} to C–O–C pyranose ring skeletal vibration. It can be seen from the FTIR spectrum of OAPP that the intensity of the bands at 3,408 and 1,633 cm^{-1} have considerably decreased. This is attributed to the replacement of the hydroxyl groups by the oxalate group ($C_{17}H_{33}COO^-$) of the oleic acid. OAPP also shows a weak band at 1,751 cm^{-1}, which provides further evidence that the fiber has been esterified [19].

3.2. Morphology analysis

Scanning electron micrographs (SEM) of untreated and treated PP are shown in Fig. 2. PP are porous and the pore can transport and hold oil (and/or water)(see in Fig. 2(a) and (b)). The micrographs in Fig. 2(a) and (c) show that UMPP are covered with a layer, whose composition is probably mainly waxy substances [20]. In addition, Fig. 2(a) and (c) demonstrates that UMPP have rougher surfaces than OAPP. This difference was likely due to the presence of crystallites in UMPP [21] and replacement of surface hydroxyl groups by oxalate group.

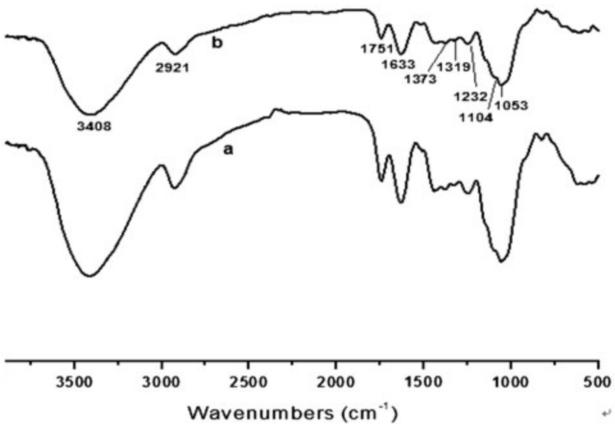

Fig. 1. FTIR spectrum of (a) UMPP and (b) OAPP.

3.3. Adsorption kinetics study

In this study, the batch kinetic studies were carried out, and the data obtained were shown in Fig. 3. Fig. 3 showed that the lubricating oil sorption capacity was better than diesel for UMPP, SAPP, and OAPP. This could be caused by higher viscosity of lubricating oil compared with that of diesel. The result also showed that the oil sorption capacity increased in the order of UMPP < SAPP < OAPP. The oil sorption capacity of OAPP was higher than that of SAPP, which might be the conversion of oleic acid was much more compared to stearic acid. Moreover, it might be the solubility of oleic acid which was higher than stearic acid in n-hexane. The solubility of oleic acid was 720 g/100 g of n-hexane at 10℃ while that of stearic acid was less than 0.5 g/100 g [22].

To determine relevant parameters, batch kinetic data obtained here were processed in conjunction with appropriate models mentioned in the literature [23]. In this connection, both the first-order and the second-order adsorption kinetic models are presented in the following. The first-order adsorption kinetics can be described as:

$$\frac{d_q}{d_t} = k_1(q_e - q) \tag{2}$$

where is the equilibrium amount of adsorbed contaminant (g) per unit mass (g) of the adsorbent, q is adsorbed amount at any time t, and k_1 is the first-order rate constant (1 min^{-1}). Integrating the above equation with the limit $q = 0$ at time $t = 0$ gives:

$$\ln \frac{q_e - q}{q_e} = -k_1 t \tag{3}$$

Eq. (3) can also be rewritten as:

$$\ln (q_e - q) = -k_1 t + \ln\ q_e \tag{4}$$

It is clear from Eq. (4) that a plot of ln (q_e–q) vs. time yields a straight line of slope $-k_1$ and the y-intercept as ln (q_e). The experimental data and fits are shown in Fig. 4.

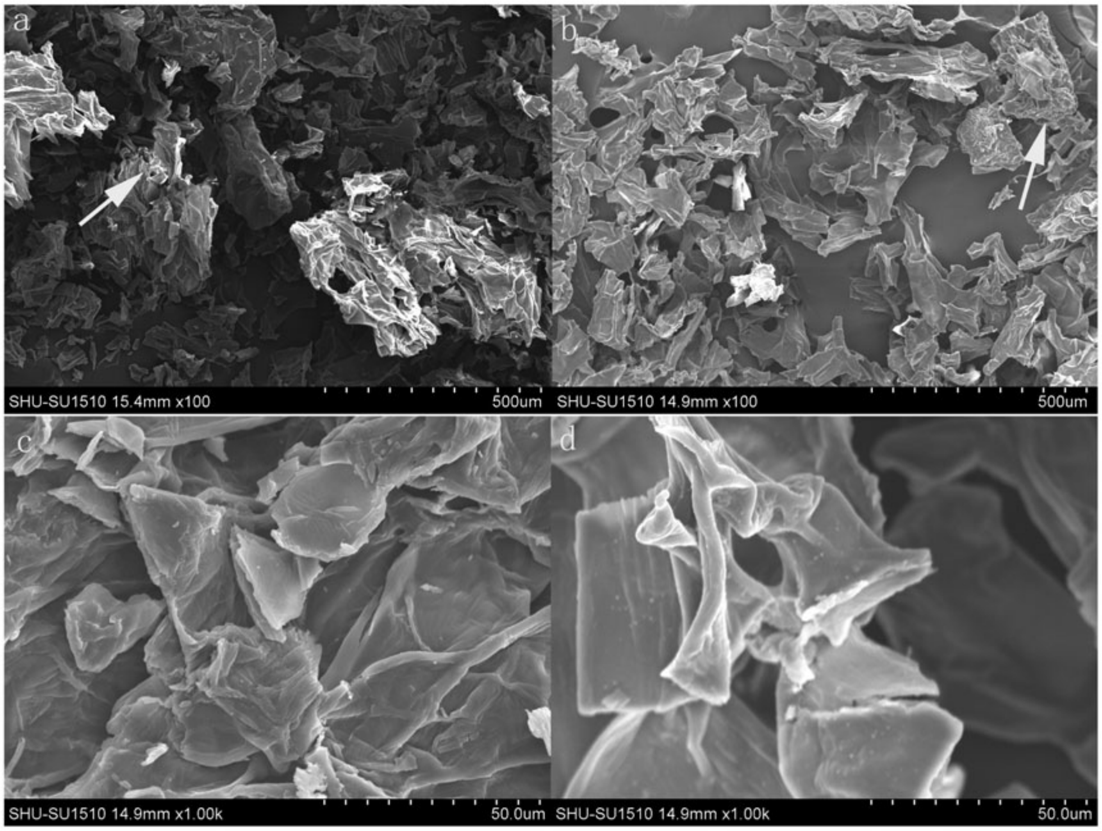

Fig. 2. SEM micrographs of UMPP and OAPP: (a) UMPP (100 ×), (b) OAPP (100 ×), (c) UMPP (1,000 ×) and (d) OAPP (1,000 ×).

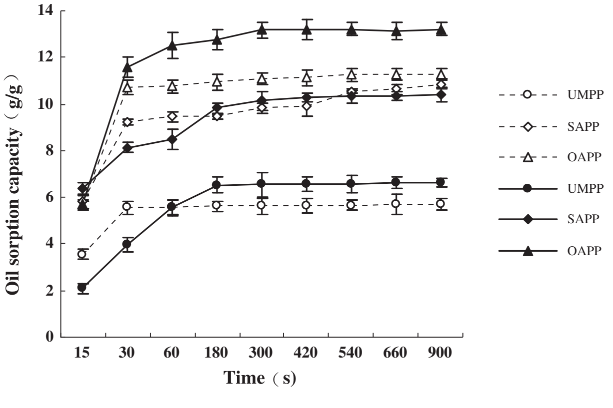

Fig. 3. Kinetic curves of sorption. The solid shape means lubricating oil, the hollow shape means diesel.

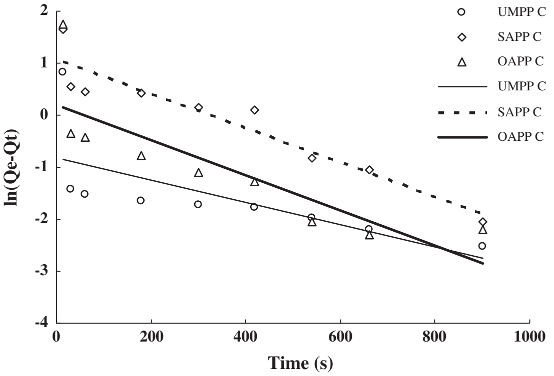

Fig. 4. Dynamic fitting results for the first order equation of soption. The hollow shape means diesel.

The agreement is not good in the case of diesel and lubricating oil. The parameter values, i.e. the rate constant k_1 and q_e, are computed from the slope and the intercepts of the fitted curves and reported in Table 1.

On the other hand, the second-order adsorption kinetics is represented as:

$$\frac{d_q}{d_t} = k_2(q_e - q)^2 \tag{5}$$

Table 1
Summary of the first order kinetic parameters for the oil sorption on UMPP, SAPP and OAPP

Sample	Oil	Slope	Intercept	R^2	q_e (g g^{-1})	k_1 (1 min^{-1})
UMPP	Diesel	−0.002	−0.813	0.4876	0.44	0.126
	Lubricating oil	−0.005	0.401	0.7504	1.49	0.270
SAPP	Diesel	−0.003	1.072	0.8965	2.92	0.198
	Lubricating oil	−0.004	0.736	0.8499	2.09	0.240
OAPP	Diesel	−0.003	0.196	0.6986	1.22	0.204
	Lubricating oil	−0.004	0.371	0.6400	1.45	0.240

Here, k_2 is the second-order rate constant (g adsorbent per g contaminant per min). The integration of Eq. (5) with the limit $q = 0$ at time $t = 0$ gives:

$$\frac{1}{q_e - q} - \frac{1}{q_e} = -k_2 t \tag{6}$$

Rearrangement of the above equation yields

$$\frac{t}{q} = \frac{t}{q_e} + \frac{1}{k_2 {q_e}^2} \tag{7}$$

Therefore, a plot of (t/q) vs. time yields a straight line with the slope of $(1/q_e)$ and the y-intercept of $(k_2 {q_e}^2)^{-1}$. In this case, the results of dynamic equation are fitted with the second-order equation of adsorption (Fig. 5). For example, the values of the slope are 0.174, 0.092 and 0.088 while the intercept values are 0.852, 1.759, and 0.652 for UMPP, SAPP, and OAPP to diesel, respectively. Parameter values obtained for all cases along with the goodness of fit, R^2 for diesel and lubricating oil are summarized in Table 2. The result reveals that fatty acid-modified PP (10.38–13.21 g g^{-1}) are efficient sorbent for oil spill recovery, and are apparently superior than fatty acid-grafted sawdust (6.15–6.50 g g^{-1}) [24] and fatty acid-grafted Banana Trunk fibers (9.58–10.78 g g^{-1}) [16].

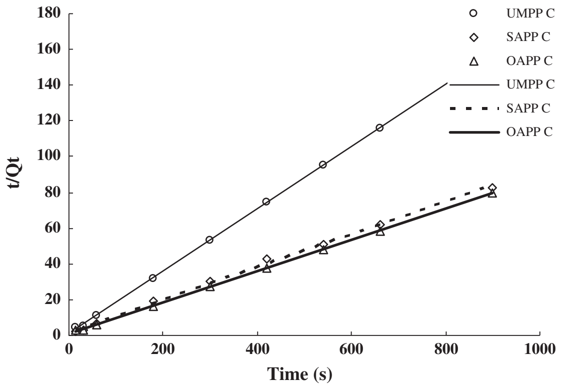

Fig. 5. Dynamic fitting results for the second order equation of soption. The hollow shape means diesel.

3.4. Effect of solution temperature

The temperature of seawater varied with the change of season and location. The effect of the solution temperature on the oil sorption capacity was investigated. Fig. 6 shows the oil sorption capacity of UMPP, SAPP, and OAPP at different temperature. The overall trend is that the oil sorption capacity gradually declines with the temperature rising. It is observed that the maximum sorption capacities of UMPP, SAPP, and OAPP are 5.96 (7.59), 11.33 (10.43), and 11.49 (14.17) g g^{-1} of diesel (lubricating oil) at 15–20˚C, respectively. The reason might be that the oil sorption process of PP was exothermic, temperature rising was not conducive to the sorption. Some researchers [25,26] found that when the temperature rose, the hydrophobicity of diesel was abated, and the solubility of diesel molecules in water increased, which made diesel harder to be absorbed from water. In addition, with the temperature rising, the viscosity of diesel reduced, the liquidity of diesel increased, and the Brownian motion of diesel particle accelerated, which could promote diesel particles and the adsorbent to collide with each other. On the other hand, it could also be the case that the diesel particle moved too fast, and reduced the chances that was adsorbed, and then reduced the diesel adsorption quantity.

3.5. Effect of solution salinity

Different location of the water had different salinity [27]; the effect of the salinity on the oil sorption capacity was investigated. Fig. 7 shows that oil sorption capacity of PP first rise and then declines when the salinity varies from 20 to 40%. It is observed that

Table 2
Summary of the second order kinetic parameters for the oil sorption on UMPP, SAPP and OAPP

Sample	Oil	Slope	Intercept	R^2	q_e ($\mathrm{g\,g^{-1}}$)	k_2 ($\mathrm{g\,g^{-1}\,min^{-1}}$)
UMPP	Diesel	0.174	0.852	0.9999	5.73	2.142
	Lubricating oil	0.147	2.735	0.9994	6.82	0.474
SAPP	Diesel	0.092	1.759	0.9981	10.93	0.288
	Lubricating oil	0.095	1.049	1.0000	10.53	0.516
OAPP	Diesel	0.088	0.652	0.9998	11.39	0.708
	Lubricating oil	0.075	0.619	0.9997	13.35	0.546

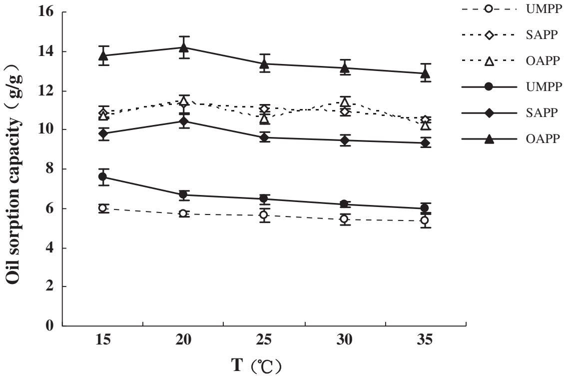

Fig. 6. The influence of temperature on sorption. The solid shape means lubricating oil, the hollow shape means diesel.

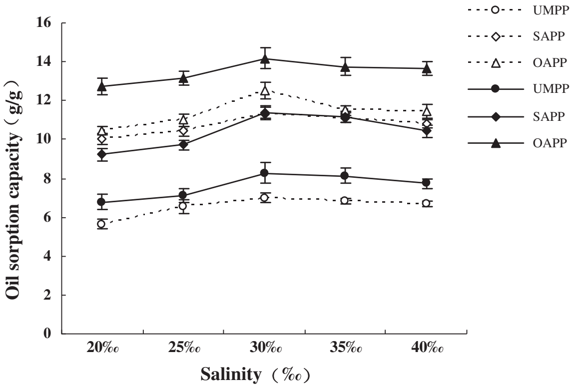

Fig. 7. The influence of salinity on adsorption. The solid shape means lubricating oil, the hollow shape means diesel.

the maximum sorption capacities of UMPP, SAPP, and OAPP are 6.99 (8.26), 11.33 (11.41), and 12.53 (14.17) $\mathrm{g\,g^{-1}}$ for diesel (lubricating oil) at 30% salinity, respectively. When the salinity was 20–30%, oil became more hydrophobic due to electrostatic interactions and salting out effect, the oil sorption force of PP became larger, thus increased the sorption amount [28]. When the salinity was between 30 and 40%, the oil sorption capacity of PP decreased, the reason might be the physical and chemical properties of PP which were influenced by higher salinity.

3.6. Effect of oscillation frequency

Fig. 8 shows the oil sorption by UMPP, SAPP, and OAPP at different oscillation frequency. The overall trend is that the oil sorption capacity firstly rises and then declines with the oscillation frequency rising. It is observed that the maximum sorption capacities of UMPP, SAPP, and OAPP are 6.82 (7.66), 11.33 (10.43), and 11.49 (14.17) $\mathrm{g\,g^{-1}}$ of diesel (lubricating oil) at low oscillation frequency (24–72 rpm). Chen et al. [29] noted that low oscillation frequency was able to promote the sorption process. With the stirring frequency increasing, the oil particles accelerated, and had more opportunity to contact with material, then was beneficial to surface adherency on oil sorption material. When the frequency was bigger than 72 rpm, the oil particles moved too fast and affected the contact with

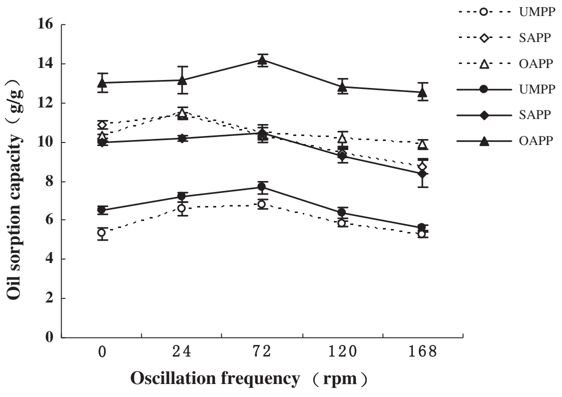

Fig. 8. The influence of oscillation frequency on sorption. The solid shape means lubricating oil, the hollow shape means diesel.

the oil sorption material. With a larger flow shear, the oil particles were more likely to be washed back into the sea, leading to the decreased oil sorption capacity of the sorption material.

3.7. Economic viability

Synthetic fibers, in particular polypropylene and polyurethane [30] are playing an important role in oil spill cleanup, and are available for approximately USD 100 kg^{-1}. The cost of the polypropylene is relatively high even though the oil sorption capacity is reported to be at 10 g g^{-1} [31]. OAPP has equal, even slightly better, sorption capacity than polypropylene, however, the cost of transport, chemicals used for the surface modification, electrical energy, and labor would be approximately USD 8 kg^{-1}. Therefore, the developed fatty acid-grafted PP may be considered as an alternate sorbent to commercially available synthetic materials due to better oil sorption capacity, cost-effectiveness and biodegradability.

4. Conclusion

This work proved that the oil sorption capacities of PP modified by oleic acid and stearic acid increased markedly compared with that of the unmodified PP. The sorption was depended on solution temperature, salinity, and oscillation frequency. OAPP was found to have better oil sorption capacity for lubricating oil and diesel in simulated seawater. The adsorption kinetics of unmodified and modified PP were well described by the pseudo-second-order kinetic model equation. Moreover, compared to polymers which are used for oil spillage, PP is biodegradable and will not lead to secondary pollution. Thus, fatty acid-modified PP can be potential sorbents for oil spillage.

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