

# A New Method to Prepare Oil Adsorbent Utilizing Waste Paper and Its Application for Oil Spill Clean-ups

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A cellulose-based oil adsorbent was developed utilizing the waste paper PS-16 (mixed kraft cuttings) via a simple modification process. Through mechanical treatment and spray with polyethylene wax and alkyl ketene dimer, it was found that the product was significantly more hydrophobic than the raw material, with a water contact angle of 125.6°. The capability of the product to absorb engine oil, kerosene, and xylene was studied and compared with other cellulose-based adsorbents. The adsorbent had an excellent performance with a high absorbing ability of 16 to 28 times its own weight. Up to 92.8% of the oil in the adsorbents could be easily recycled and collected by manual squeezing. The adsorbent could be reused over eight cycles, and the sorption capacity remained constant. Therefore, this adsorbent is expected to be a promising oil sorbent for potential applications like oil spill clean-ups.

*Keywords:* Waste paper; Hydrophobic modification; Oil adsorbent; Oil spill clean-up

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## INTRODUCTION

Clean-up of oil spills from water bodies has become a worldwide problem. To address the oil spills in a timely manner is a severe challenge. Currently, the major methods for handling oil spills are *in situ* combustion, chemical dispersants and flocculants, micro-biological degradation, physical interception, and various adsorbents (Suni *et al.* 2004; Al-Majed *et al.* 2012; McNutt *et al.* 2012; Hubbe *et al.* 2013; Xue *et al.* 2016). Among these methods, the use of adsorbents is sometimes the most effective and optimized treatment because other techniques do more harm than good, such as by wasting large amounts of resources (*in situ* combustion), taking a long time to work (micro-biological degradation), and causing secondary pollution (chemical dispersants and flocculants).

At present, the most commonly used oil-absorbing materials are generally classified as inorganic mineral materials, organic synthetic products, and organic natural materials (Lu and Zhou 2002). The cost of inorganic oil absorption materials is low, but the oil/water selectivity and reusability are poor, which limits their application. Recently, research about organic synthetic products has developed rapidly because of their excellent oil absorption capacity, oil/water selectivity, and reusability (Reyssat *et al.* 2010; Li *et al.* 2012; Pham and Dickerson 2014; Oribayo *et al.* 2017). However, the preparation process of organic synthetic products is sophisticated and the cost is high. Most importantly, these products cannot be degraded naturally, which causes secondary pollution issues.

Therefore, researchers have gradually shifted their focus to biodegradable organic natural materials, including stalk, plant fibre, cotton grass fibre, cotton grass mats, kapok fibre, *etc.* However, these materials generally exhibit hydrophilicity and poor oil/water

selectivity (Carmody *et al.* 2007; Karakasi and Moutsatsou 2010; Korhonen *et al.* 2011; Gui *et al.* 2013; Hu *et al.* 2013; Nguyen *et al.* 2013; Idris *et al.* 2014). The hydrophobicity of raw materials is improved by pyrolysis (Dimitrov *et al.* 2012), graft copolymerization, predeposition of lignin and alkenylsuccinic anhydride or other hydrophobic sizing agents on cellulosic fibers (Payne *et al.* 2012), and other methods. Recently, hydrogels and aerogels have become popular, though their preparation processes are relatively complex. The energy consumption of these processes is high, which makes these materials unsuitable for industrial production (Banerjee *et al.* 2006; Wang *et al.* 2010; Korhonen *et al.* 2011; Vlaev *et al.* 2011; Nguyen *et al.* 2013).

In this study, an efficient and environmentally friendly oil adsorbent was prepared from the waste paper PS-16 (mixed kraft cuttings). The modification process included pulverizing the waste paper into fluffy material having a loose and messy texture, using a grinder as a physical pretreatment, followed by spraying two modifiers successively, whose main components were polyethylene wax and alkylketene dimer. The idea was to enhance the hydrophobicity of PS-16 by grafting alkyl groups, which have a good affinity for oil and good resistance to water. The raw materials and modifiers used in this study are common low-cost materials. Furthermore, because the main raw material used was cellulose fibre, the adsorbent can be naturally degraded. This is advantageous because oil adsorbents are consumed in large amounts and therefore, secondary pollution of the environment is minimized. In contrast, as the modification process is particularly simple, it is suitable for industrial scale production. All of these advantages make this adsorbent unique when compared with previously studied chemically synthesized superhydrophobic oil-absorbing materials (Wei *et al.* 2003; Ceylan *et al.* 2009; Zhu *et al.* 2011; Hu *et al.* 2013; Lin *et al.* 2013; Zhou *et al.* 2013). The properties and performances of the adsorbent related to the clean-up of spilled oil were also investigated. This work could create a new perspective in recycling waste pulp and paper into useful products.

## EXPERIMENTAL

### Materials

The PS-16 (mixed kraft cuttings) was collected from Shanghai Weizhong Paper Co. Ltd. (Shanghai, China). The engine oil was obtained from a petroleum plant station (China Petroleum, Guangzhou, China). The main component of the first modifier was polyethylene wax, which was purchased from Longkou Excel Chemical Science and Technology Co. Ltd. (Longkou, China). The major ingredient of the second modifier was alkyl ketene dimer, which was bought from Lansen Chemical Products Co. Ltd. (Wuxi, China).

### Sample Preparation

The modification process mainly adopts a coating method to improve the fiber's hydrophobicity (Li *et al.* 2016). Firstly, the waste paper PS-16 was torn into 2-cm × 2-cm pieces, and then put them in the oven at 80 °C for 8 h. After drying, the raw material was ground into a fluffy state. The material obtained after physical pretreatment we defined as PP. Using an atomizing sprayer (JDT-05A, Nanshuiguangai, Dongguan, China), 0.35 g of the first modifier was sprayed onto 1 g of the PP, which was then oven-dried at 80 °C for 15 min. These adsorbents were referred to as FPP. Then, 0.15 g of the second modifier was sprayed onto the FPP and dried again. These adsorbents were referred to as SPP.

## Characterization

The surface morphology of the adsorbents was studied using scanning electron microscopy (SEM) (LEO1530VP, Zeiss, Jena, Germany). X-ray diffraction (XRD) was performed using a Rigaku SmartLab SE (Tokyo, Japan). A contact angle meter (OCA40MICRO, Dataphysics, Filderstadt, Germany) was used to measure the contact angle between the adsorbent and water by dropping a single drop of liquid from a syringe onto the adsorbent surface and photographing it immediately. Before the measurement, the adsorbent materials were pressed into a slice, which had a smooth surface for convenient testing.

## Measurements of Oil Absorption Capacity and Reusability

The absorption capacity of the adsorbent for various oils and organic solvents from the surface of water was tested by placing a pre-weighed ball of adsorbent in a beaker containing water and oil or organic solvents. Absorption occurred under stirring condition for 15 mins at room temperature (30 °C). Then, the adsorbent was removed from the beaker, placed on a copper mesh, allowed to drain for 5 mins, and weighed. The absorption capacity ( $Q$ ) was calculated using Eq. 1,

$$Q = \frac{M_1 - M_2}{M_2} \quad (1)$$

where  $M_2$  and  $M_1$  are the weights (g) of the adsorbent before and after absorption, respectively.

The reusability of the adsorbent was evaluated by repeated absorption and squeezing. The adsorbent was immersed in oil, and then the oil was collected by manual squeezing. The adsorbent became fluffy after a grinding process. The process was repeated several times, and the oil recovery ( $R$ ) was calculated with Eq. 2:

$$R = \frac{M_r}{M_1 - M_2} * 100 \quad (2)$$

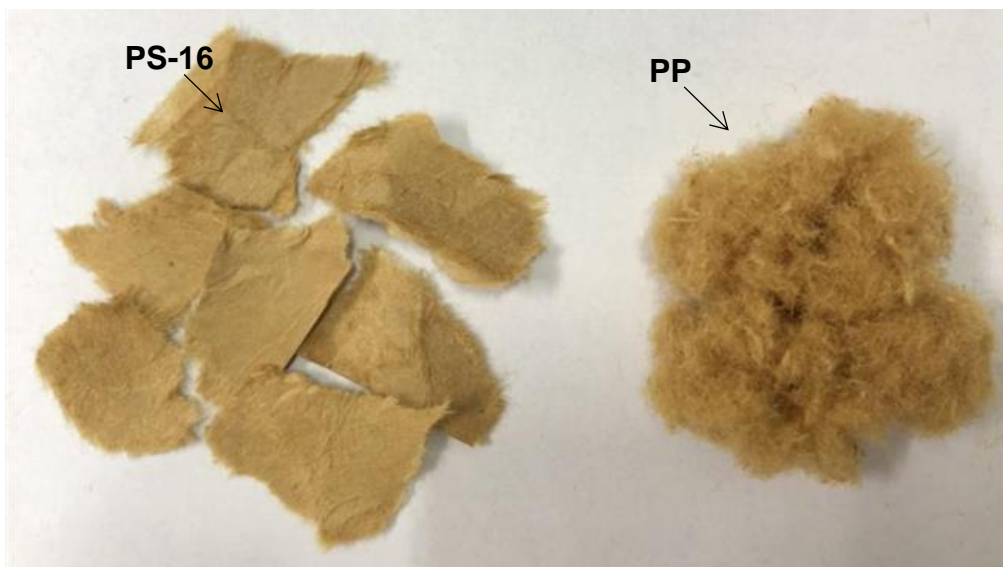
where  $M_r$  is the weight (g) of the recovered oil.

## RESULTS AND DISCUSSION

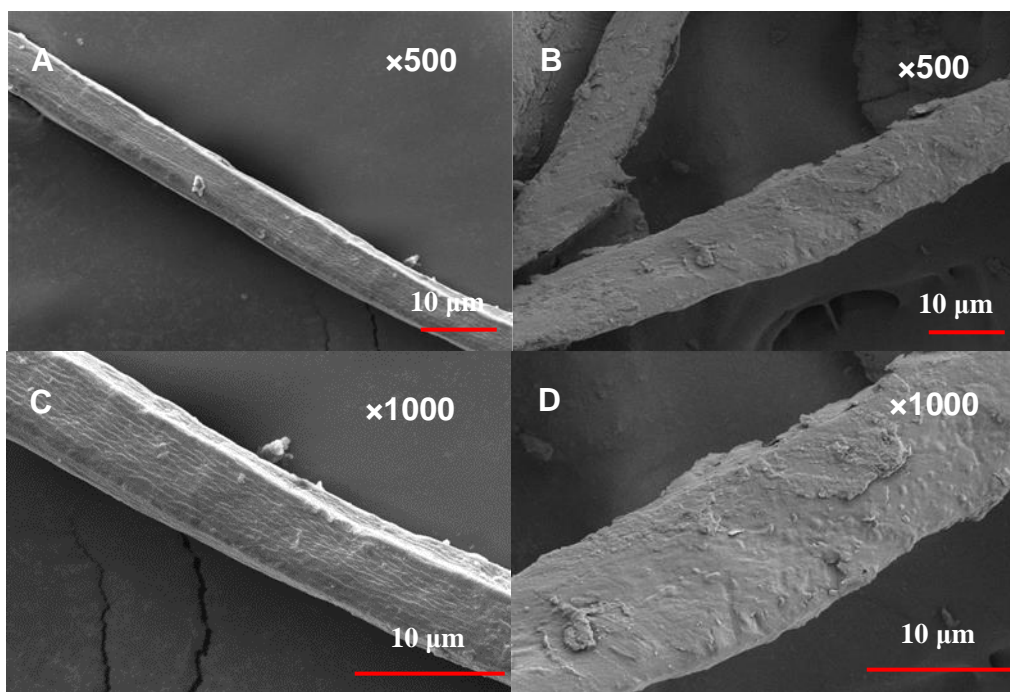
### Surface Morphology and XRD Analysis

The material obtained after physical pretreatment was defined as PP. Compared with the raw material PS-16, PP had a fluffy soft texture (Fig. 1). The morphologies of the PP and SPP were observed by SEM (Fig. 2). The raw material fibre had a relatively smooth surface (Fig. 2A and 2C). After attachment of the modifiers to the skeletons of the raw fibre material, the surface of the SPP fibre exhibited a rougher morphology, whose surface was covered by a large number of micro-scale protrusions (Fig. 2B and 2D). This indicated that the two modifiers were successfully adhered onto the skeletons of the raw fibre material.

The modification transformed the SPP adsorbent into a hydrophobic and oleophilic adsorbent. The two modification processes introduced roughness and strength to the surface. Thus, the wettability of the SPP adsorbent changed because of its interaction with a wetting or non-wetting liquid (Zhu *et al.* 2014; Brown and Bhushan 2015).



**Fig. 1.** Digital photos of the PS-16 and PP



**Fig. 2.** Surface morphologies of the PP (A and C) and SPP adsorbents (B and D)

The crystal structures and crystallinity changes of the PS-16, PP, FPP, and SPP were analyzed by XRD (Fig. 3). The PS-16 had characteristic diffraction peaks at the  $2\theta$  values  $22^\circ$  and  $15^\circ$ , which were characteristic of natural cellulose. The peak intensity at  $22^\circ$  from the cellulose diffraction peak of the (002) crystal plane was high, which indicated that the cellulose existed in a highly ordered aggregation state in the PS-16, with a strong hydrogen bond between the hydroxyl groups so that it was difficult for the oil molecules to enter. Therefore, the absorption capacity of the PS-16 was low. At the value of  $15^\circ$ , which represents the diffraction peak of the (101) crystal plane, the width of the crystal plane is large and often appears as a side-by-side peak. When comparing the curves of the

PS-16 and PP, the diffraction peak of the PS-16 decreased dramatically after physical pretreatment. This was not only beneficial to subsequent chemical modification, but also improved the entry of oil molecules. After the two chemical modifications, the crystallinity increased.

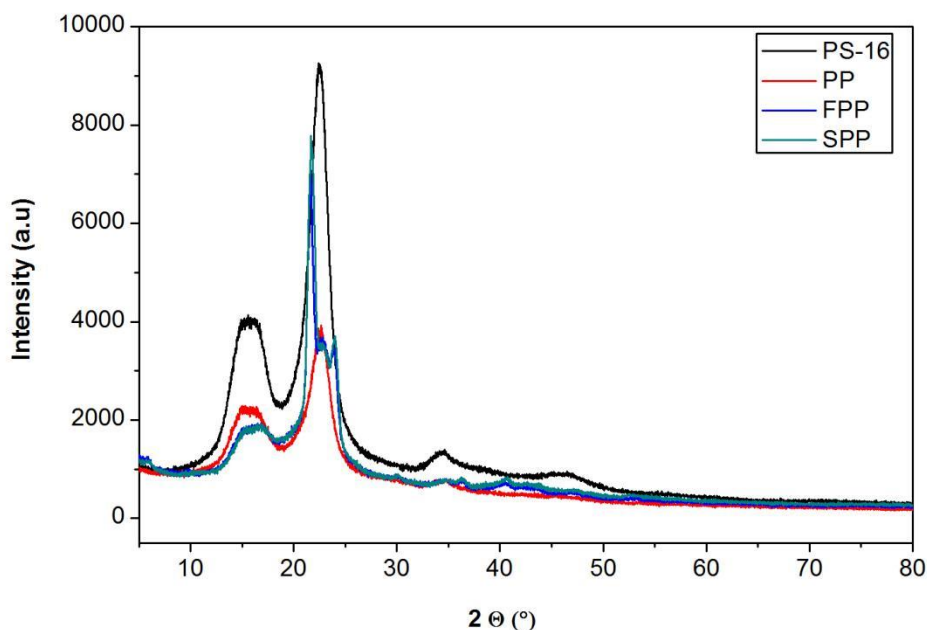
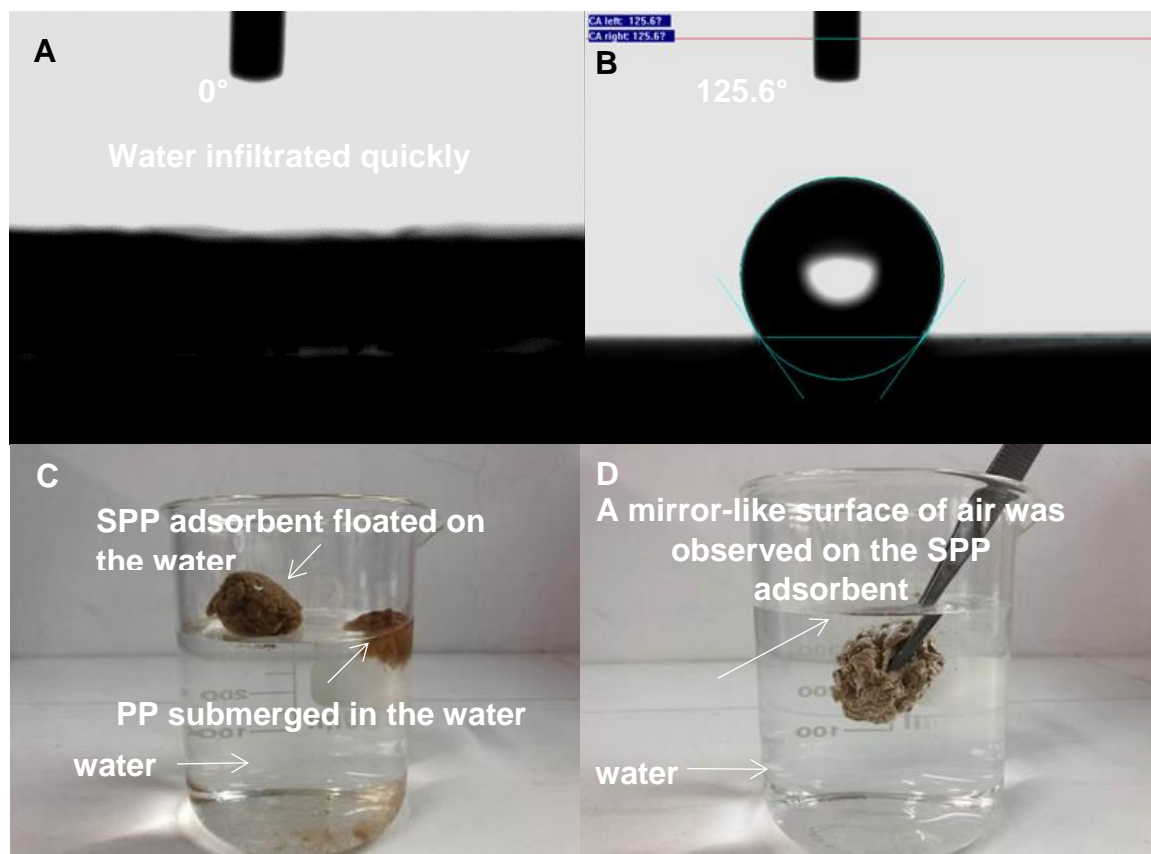


Fig. 3. XRD patterns of the PS-16, PP, FPP, and SPP adsorbents

### Surface Wettability of the Adsorbent

The surface wettability results for the PS-16 and SPP are shown in Fig. 4. Parts 4A and 4B show the water contact angles on the surface of the PS-16 and SPP adsorbents, respectively. The PS-16 exhibited a hydrophilic surface, such that the water could easily penetrate within 1 s and the water contact angle was measured to be 0°, while the SPP adsorbent showed a hydrophobic surface with a water contact angle of 125.6°.

When the adsorbents were placed on the water surface (Fig. 4C), the SPP adsorbent sample always floated on the water surface, while the PP was submerged in the water. Furthermore, when the SPP adsorbent was forced to enter the water under an external force, an air layer was observed on the adsorbent surface (Fig. 4D). The SPP adsorbent immediately returned to the water surface when the applied external force was released without the absorption of water, as was confirmed gravimetrically afterwards, which indicated its excellent hydrophobicity.



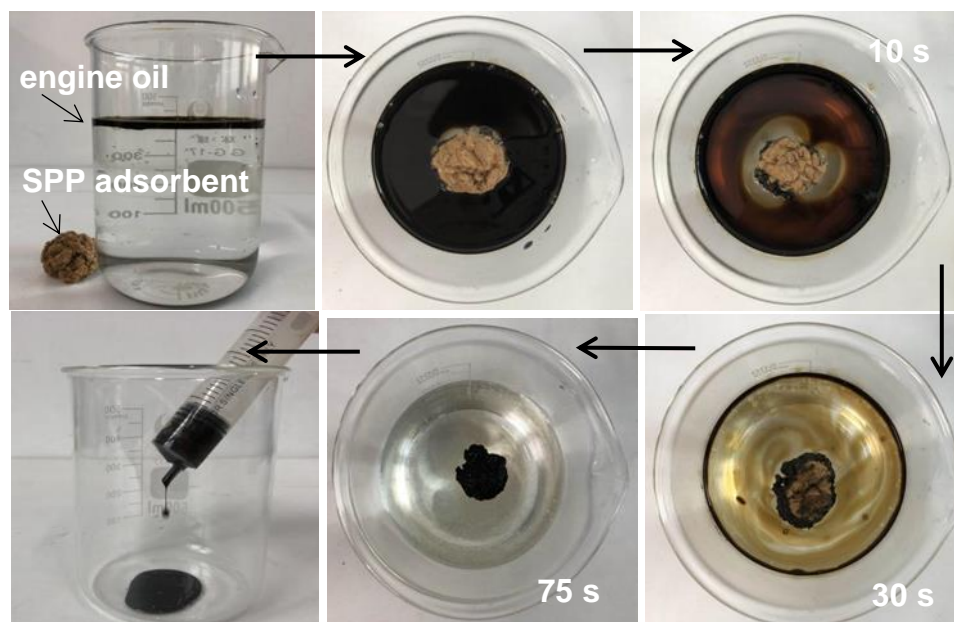
**Fig. 4.** Surface wettability of the adsorbent: water contact angles on the surface of the PS-16 and SPP adsorbents (A and B); PP and SPP adsorbents on the water surface (C); and the SPP adsorbent immersed in water under an external force (D)

### Application and Performance of the Adsorbent

To investigate the oil absorption process on the water surface, some of the adsorbent was placed in a beaker that contained water with dispersed engine oil. As shown in Fig. 5, the adsorbent immersed in the oil quickly and the oil layer appeared to be thin. 75 seconds later, the adsorbent was saturated *via* capillary force and it still floated on the water surface, which indicated that the adsorbent had an excellent sorption capacity for oil and it also had a very good floating performance.

Oil spills happen not only at sea, but also in filling stations, kitchens, and laboratories. The SPP adsorbent has a unique structure and uses capillary force to absorb oil. Hence, the selectivity for oil is low and the adsorbent can remove a variety of oils and other organic solvents from water. The absorption capacity of the adsorbent for various oils and some water-immiscible organic solvents was also tested. Table 1 listed the properties of oils and organic solvents.

It was observed in the experiment that the viscosity and density of these oils and organic solvents affected the absorption rate of the SPP adsorbent. When the viscosity of the oil was high, the absorption rate was low and the SPP adsorbent took a few minutes to reach the saturation point because the mobility of oil was slower when the viscosity was higher.



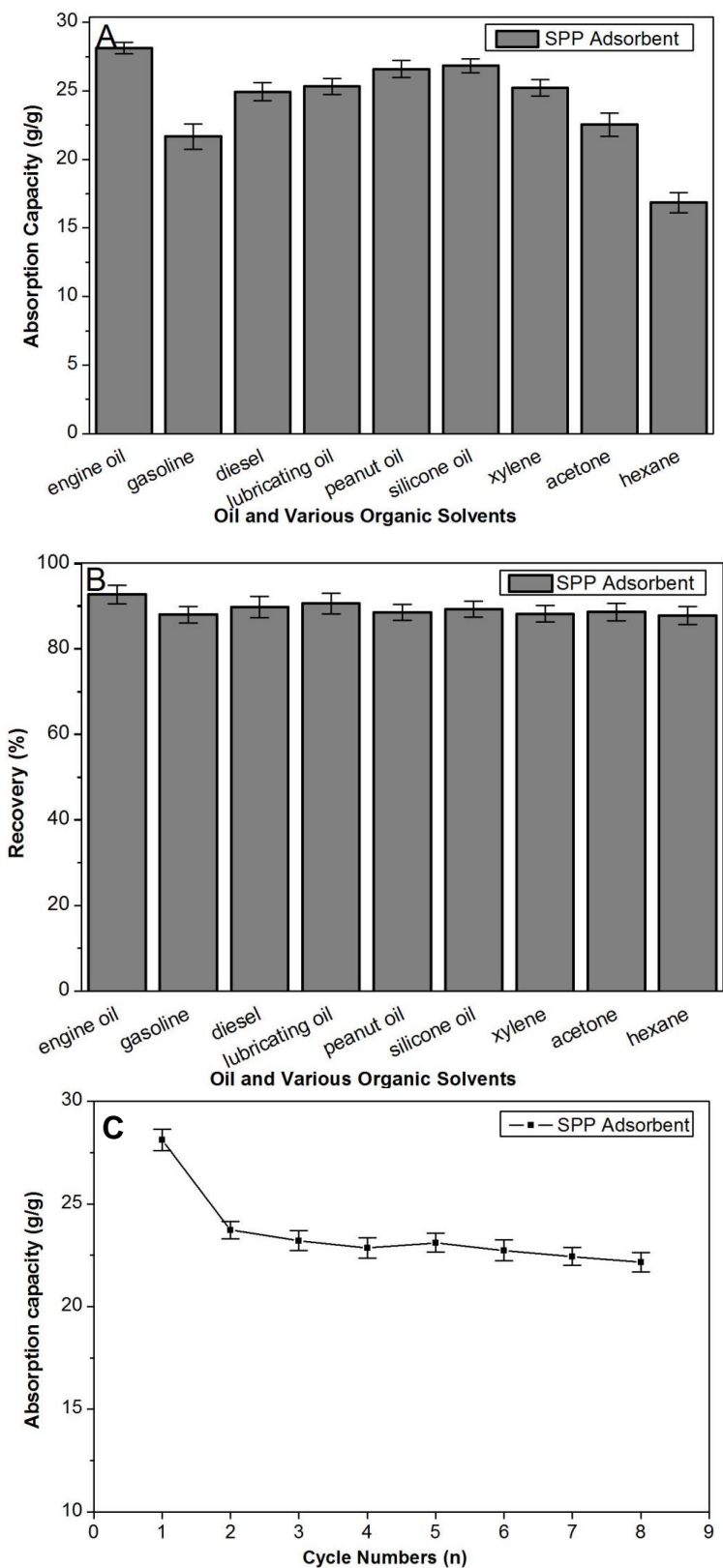
**Fig. 5.** Digital pictures showing the absorption and recovery of engine oil from water using the SPP adsorbent

**Table 1.** Properties of Oils and Organic Solvents

Sample	Viscosity ( mPa•s )	Density ( g/cm <sup>3</sup> )
engine oil	241.5	0.932
gasoline	0.57	0.711
diesel	6.2	0.847
lubricating oil	16.2	0.875
peanut oil	139.5	0.911
silicone oil	210.7	0.955
xylene	0.75	0.861
acetone	0.32	0.784
hexane	0.31	0.655

**Table 2.** A Comparison of Cellulose-based Oil Adsorbents

Reference	Oil/Solvent	Absorption capacity (g/g)
Mamat <i>et al.</i> (2010)	Soybean oil	16.2
	Xylene	14.3
	diesel	13.7
Arken <i>et al.</i> (2011)	dichloromethane	24.6
Keshawy <i>et al.</i> (2013)	methylbenzene	29.7
	Crude oil	18.6
Li <i>et al.</i> (2012)	diesel	52.65
Sun <i>et al.</i> (2003)	engine oil	18.8
Zhu <i>et al.</i> (2012)	diesel	20.12



**Fig. 6.** Absorption capacities of the SPP adsorbent for oil and various organic solvents (A); recovery rates of the SPP adsorbent (B); and absorption capacity of the engine oil over 10 sorption-squeezing cycles for the SPP adsorbent (C)



Figure 6A shows that the SPP adsorbent exhibited a sorption capacity of 16 to 28 times its own weight. The absorption rate and absorption capacity changed with the density and viscosity of the tested organic solvents and oils. The solvent and oil absorbed into the SPP adsorbent could be easily collected by manual squeezing, and the percentage recovery was more than 85%, which is shown in Fig. 6B. The reusability of the adsorbent was stable and remained over 80% of its maximum oil absorption after eight cycles of sorption-squeezing, which is shown in Fig. 6C.

Table 2 listed the performance of some cellulose-based oil adsorbents as compared to the SPP adsorbent in this work. Except for modified kapok fiber (Li *et al.* 2012), which has excellent absorption capacity reaching 52.65 g/g, for most of them the absorption capacities were under 20 g/g. Their hydrophobic modification process was graft copolymerization, and this method was complicated. Furthermore, these cellulose-based oil adsorbents were in powder form, which was very unfavorable to the oil spill in water bodies because it would be quite difficult to recycle. In contrast, the raw material in this work realized waste utilization and the modification was simple and didn't need purification. For the vantage, the SPP adsorbent had good absorption capacity, it held great promise as adsorbent material for clean-up and recovery of spilled oils.

These tests demonstrated that the adsorbent showed an excellent and stable absorption capacity for a wide range of applications. Most importantly, the raw material is low cost and the modification and recovery process was simple. Hence, the adsorbent presented great promise for clean-up and recovery of oil spilled on water bodies.

## CONCLUSIONS

1. An efficient and environmentally friendly oil adsorbent was prepared from the waste paper PS-16 (mixed kraft cuttings). The advantages of using this adsorbent were its low cost because of the abundance of waste paper, and the simple modification process. The method for preparing the adsorbent included mechanical treatment and coating. Therefore, it is suitable for industrial production.
2. The resulting exterior of the adsorbent was similar to dry pulp and it exhibited a high absorption capacity for oil/organic pollutants in water bodies. Up to 92.8% of the oil could be easily recycled and collected by manual squeezing, and the reusability performance remained essentially the same after eight cycles of oil sorption-squeezing experiments. It was concluded that the oil adsorbent in this work is a promising material for the clean-up of oil spills in water bodies.

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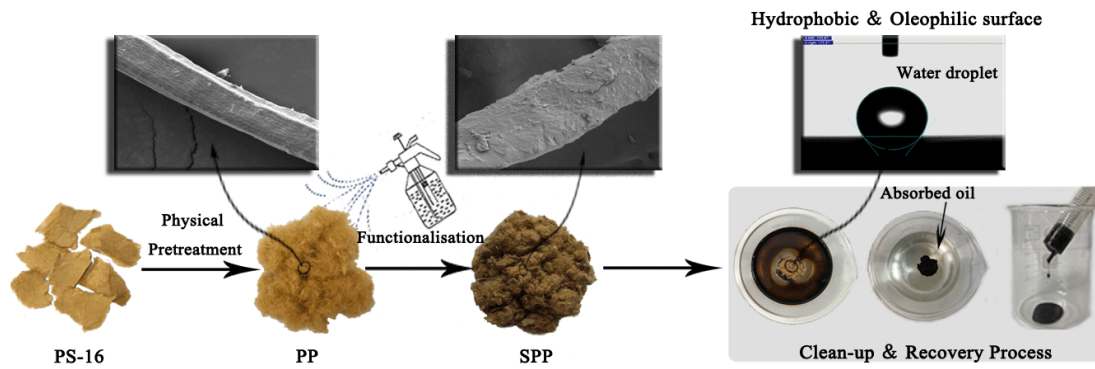
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## APPENDIX

## SUPPLEMENTARY INFORMATION



**Fig. S1.** The preparation process of the adsorbent and its hydrophobic and oil absorption performance