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# Facile stearoyl chloride grafted cotton filter fabric and its application in oil-water separation

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Abstract Surface hydrophobic modification for oil– water separation based on membrane filtration is essential but still challenging in dealing with various industrial and environmental problems like sewage treatment and oil spill. Chemical graft technology can arm membranes with more efficient durability for applications due to the stronger chemical interaction

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T. Jin e-mail: jintao@gic.ac.cn between modified layers and surfaces compared with physical coating technology. As cellulose-based material, cotton filter fabric is a flexible and degradable material with abundant hydroxyl groups, providing great possibility for graft reactions. Stearyol chloride has both hydrophobic alkyl chain and active acyl group, making it easily to be grafted onto cotton filter fabrics. Therefore, we propose to use stearoyl chloride as hydrophobic layer and one-step modify cotton filter fabric after activation. The obtained hydrophobic modified cotton filter fabric exhibits high contact angles ( $CA_{\text{max}} = 147^\circ$ ,  $CA_{\text{avg}} = 141.8^\circ$ ), which only decrease slightly after 5 h droplet holding time  $(CA_{\text{max}} = 141^{\circ}, CA_{\text{avg}} = 126.3^{\circ})$ . Besides, experiments also confirm that this modified membrane displays excellent anti-fouling property against different drinks as well as anti-washing property for 10 cycles. Much more surprised is that the obtained membrane displays outstanding anti-abrasion performance that the CA values can remain above  $131^\circ$  after 390 cycles abrasion with hektogram counterweight. As for filtration application, the modified membrane also shows hydrophobic durability after three times gravitydriven oil–water separation ( $CA_{\text{max}} = 137^{\circ}$ ,  $CA_{\text{avg}} =$ 132.6°). This hydrophobic modified cotton filter fabric may have potential application in future oilwater separation and the stearoyl chloride graft technology is an both effective and efficient way in surface hydrophobic modification.

## Graphic abstract



Keywords Stearoyl chloride graft - Hydrophobic modified cotton filter fabric - Oil–water separation

## Introduction

Oil–water separation is an inevitable process in many industries to protect environment and ecology. Sewage treatment, oil spill and other industrial fields need oil–water separation. Due to cost-efficiency and facile operation, membrane filtration method has caught wide attention of both academia and industry. One of the oil–water separation way is to fabricate hydrophobic membranes that only oil could permeate but water would be hold. There are various eco-friendly hydrophobic materials, such as natural waxes (Toruna et al. [2019;](#page-7-0) Guan et al. [2018\)](#page-6-0), fatty acids (Heale et al. [2018;](#page-6-0) Dong et al. [2019\)](#page-6-0), proteins (Liu et al. [2019](#page-7-0); Shome et al. [2019](#page-7-0)), cellulose derives (Huang et al. [2018;](#page-6-0) Guo et al. [2016\)](#page-6-0), biomass and agricultural waste (Anitha et al. [2018](#page-6-0); Shishodia et al. [2019](#page-7-0)). Within these, cellulose based fabrics is a flexible, degradable and cost-less material suitable for further development into useful hydrophobic membranes.

As shown in Scheme [1,](#page-2-0) physical coating is one of the hydrophobic modification methods of cellulosebased materials. Nanoparticles with low surface energy such  $SiO<sub>2</sub>$  (Wang et al. [2019\)](#page-7-0), TiO<sub>2</sub> (Ren et al. [2020](#page-7-0)) and ZnO (Thi et al. [2017\)](#page-7-0) exhibit hydrophobic properties. For example, the hexadecyl polysiloxane modified  $SiO<sub>2</sub>$  could act as a kind of superhydrophobic coating on glass slides, which exhibits a contact angle  $(CA)$  of 163.9 $^{\circ}$  (Zhao et al. [2019\)](#page-7-0).  $\text{Al}_x\text{Ni}_x\text{(Bi}_2\text{O}_3)_z$  coating synthesized by electrophoretic deposition and perfluorodecyltriethoxysilane (FAS-17) modification showed high CA of  $169^\circ$ 

(Guo et al. [2017](#page-6-0)). Epoxy resins @ stearic acid- $Mg(OH)$ <sub>2</sub> coating (Si et al. [2016\)](#page-7-0), benzoic acid incorporated Ag thin film coating (Sarkar et al. 2010) and other coatings (Biswas et al. [2021](#page-6-0); Bayer [2020;](#page-6-0) Beshkar et al. [2020a](#page-6-0), [b](#page-6-0); Joshi et al. [2020;](#page-6-0) Yu et al. [2019](#page-7-0)) also show excellent hydrophobicity.

However, physical bonding between coating and substrates is usually too weak to bear many injuries in applications. Thus, researchers put forward to replace this weak function by stronger chemical bonding. According to the difference in releasing groups, there are three major kinds of chemical grafted methods (Scheme [1\)](#page-2-0). The first kind is non releasing group, mainly including radical addition and ring-opening reaction. For example, -SH can convert to -S- under UV radiation and then add with  $C = C$  group in organofunctional polysiloxanes, but in this situation, - OH on cellulose has to be changed into -SH in pretreatment process (Sun et al. [2016\)](#page-7-0). Our group also used to modify cotton filter fabrics via activators regenerated by electron transfer atom transfer radical polymerization (ARGET-ATRP) (Li et al. [2019](#page-7-0); Luo et al. [2020\)](#page-7-0). Epoxy unit can directly reacts with the - OH on cellulose, but it will produce -OH after ringopening reaction, which is not favor the hydrophobicity (Shang et al. [2010](#page-7-0); Muresan et al. [2013;](#page-7-0) Ma et al. [2018\)](#page-7-0). The second kind is  $H_2O$  releasing. For example, carboxylic group can react with hydroxyl on cellulose fiber and release  $H_2O$  (Huang et al. [2010](#page-6-0); Bretel et al. [2018\)](#page-6-0). The third kind is t-BuOH releasing. Using tertbutyl acetoacetate as co-reagent and releasing t-BuOH can make the low active acetoxyacetic group react with -OH. As the acetoxyacetic group is not a good hydrophobic layer, another compound such as octadecyl amine is needed to be further grafted onto cellulose via serial process (Rong et al. [2019](#page-7-0)). But it will create hydrophobic -NH group on the graft compound, which has passive impact on hydrophobicity. The forth kind, HCl releasing, is a novel kind we put forward in this work intrigued by the efficient reaction between -OH and -COCl (Wolfe [1997](#page-7-0); Sano et al. [1999](#page-7-0)). High reactive efficiency can not only graft hydrophobic compounds onto cellulose surface easier and more uniform, but also reduce energy wastage.

Herein, we propose to use stearoyl chloride as hydrophobic layers via one-step graft on cotton filter fabrics after activation. Instead of multiple process or high reaction temperature, this hydrophobic modification can occur at room temperature, and the alkyl

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Scheme 1 Classification of major hydrophobic modification methods of cellulose-based materials

chains can be grafted onto cellulose via one activation process and one efficient reaction, which is both energy cost-less and time cost-less. Apart from fundamental properties such as anti-fouling, washing and abrasion performance characterization, we also evaluate the potential filtration application in oil– water separation of the obtained hydrophobic modified cotton filter fabric.

The mechanism scheme is shown as Fig. 1. In this experiment, N,N,N',N'-tetramethyl ethylene diamine (TEMED) is acted as base to firstly react with stearoyl chloride and produce nitrenium ion precursor. Subsequently, hydroxyl on the glucose monomer reacts with nitrenium ion precursor via nucleophilic addition reaction. Finally, alkyl chains with 18 carbon atoms are grafted onto cotton filter fabrics. From the result, it is stearoyl chloride reacts with -OH and produces HCl, which will be neutralized by TEMED. After activation, the whole process occurs via only one step that put all reactants together and the reaction will finish at room temperature overnight. Details can be found in the supporting information. Of course, hydroxyl on the secondary carbon atom is more easier to react with stearoyl chloride than those on the third carbon atom due to less steric hindrance. But we calculate the stearoyl chloride dosage according to the ideal mole ratio in order to graft alkyl chain as more as possible. Table S1 shows different mole ratios and concentration of reactants and the resulted CA values are compared in Fig. S1. As can be seen, stearoyl chloride graft achieves nice hydrophobic modification compared with the blank cotton filter fabric because the blank sample shows hydrophilicity after few minutes droplet holding time but the modified samples can keep hydrophobicity for hours. Among the modified samples, it is found that too low concentration of stearoyl chloride and TEMED (sample 1) presents unexpected hydrophobic durability that the CA value drops to  $120^{\circ}$  after 3 h droplet holding time, although the initial CA can be larger than  $140^\circ$ . This suggests a low graft effect of stearoyl chloride on cotton filter fabric. On the other hand, too high concentration of hydrophobic layer and base (sample 3) is also not suitable for graft effect that the initial CA is only around  $130^\circ$  though it does not slide down too much after 5 h droplet holding time. When under a



Fig. 1 Mechanism scheme of stearoyl chloride grafted on cotton filter fabrics

suitable concentration, the modified cotton filter fabric presents expected hydrophobic durability with high CA values before  $(CA_{\text{max}} = 147^\circ, CA_{\text{avg}} = 141.8^\circ)$  and after 5 h droplet holding time ( $CA_{\text{max}} = 141^{\circ}$ ,  $CA_{\text{avg}} =$ 126.3°). Besides, we tried different solvent at the optimal condition to confirm the adaptability of this graft method (Table S2). As can be seen from Fig. S2, the sample modified in DMF shows comparable hydrophobic durability (CA<sub>initial</sub> =  $139^\circ$ , CA<sub>5h</sub> =  $128^\circ$ in average) with that in  $CH<sub>2</sub>Cl<sub>2</sub>$ . But the sample modified in THF seems much worse than in the above two solvent, showing unexpected hydrophobicity  $(CA<sub>initial</sub> = 123<sup>o</sup>)$  and durability  $(CA<sub>3h</sub> = 102<sup>o</sup>)$ . That means THF is not a suitable solvent for this system.

Figure 2a is the Fourier transform infrared spectroscopy (FTIR) of cotton filter fabrics before and after hydrophobic modification. There exist two characteristic peaks for alkyl chains after stearoyl chloride graft. Compared with the blank fabric, CH asymmetrical vibration at 2916 cm<sup>-1</sup> and CH symmetrical vibration at  $2848 \text{ cm}^{-1}$  can be seen clearly from the grafted fabric, indicating a well grafted result. Thermal gravimetric analyzer (TG) under  $N_2$  was also involved to investigate the graft effect. There are two steps in the grafted fabric curve but only one in the blank fabric curve (Fig.  $2b$ ). The first step around 350 C represents the decomposition of glucose, which points out the higher decomposition temperature of the cotton filter fabric after grafted, suggesting that alkyl graft can improve the thermal stability of fabrics. At around 380 °C, glucose is completely decomposed. The second step only existing in the grafted fabric curve at about  $530 \degree C$  is the alkyl chain decomposed process, which is another evidence for the successful graft of alkyl chains. Apart from structure characterizations and thermal analyses, morphological measurements were also conducted to confirm the graft effect via this method. As can be seen from the scanning electronic microscopy (SEM) images (Fig. S3), the surface morphology of cotton filter fabrics in low magnification is similar before (Fig. S3a) and after (Fig. S3b) graft reaction. But in high magnification, the roughness of the grafted fiber surface (Fig. S3d) is obviously larger than that of the blank fiber (Fig. S3c). This also indicates the well graft effect via the stearoyl chloride graft technology, which is corresponding with the FTIR and TG results. Energy dispersive spectrometer (EDS) was performed to analyse the element distribution of the cotton filter fabrics. Fig. S4 displays the EDS spectra of the cotton filter fabrics before and after hydrophobic modification. After the modification, the carbon atom content was improved from 62.6 At% to 66.4 At%, suggesting the existence of alykl chains on the grafted fabric. Insets are the corresponding mapping, showing the uniform distribution of carbon and oxygen element.

To evaluate this stearoyl chloride graft technology more objectively, we compared the CA values both among our previous methods and the research of other groups. Figure [3](#page-4-0)a displays the hydrophobic durability among the three period studies of our group. The initial CA values can all be above  $140^\circ$ , but their CA values show more and more obvious distinguish as the droplet holding time increases. As for our first work, hydrophobic modification via ARGET-ATRP mechanism, the CA drops quickly below  $120^{\circ}$  within 1 h due to low alkyl chain density. In fact, the -OH on glucose monomer is active to be reacted after special treatment, but they are always tied with each other due to hydrogen bond interaction in macromolecule (Lee et al. [2020a,](#page-7-0) [b\)](#page-7-0). Relative researches has pointed out that  $CS_2$  can dissolve cellulose and make it



Fig. 2 a FTIR spectra and b TG curves under  $N_2$  of the cotton filter fabrics before and after hydrophobic modification

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(b)  $180$ Contact angle (°) 160  $155.6$ 152.5 152 149.2 150 145 143.6 141.8 140  $120$ 100 **HDTMS MS DTES** RCOCI **5 1580 DMS**  $\overline{O^{OP}}$  $\overline{\xi^{p}}$ ဇာ

Fig. 3 CA comparison among (a) previous works and (b) other groups' works of grafted modification of cotton filter fabrics. HDTMS (Huang et al. [2010](#page-6-0)), ODTES (Shang et al. [2010\)](#page-7-0), GS

(Muresan et al. [2013\)](#page-7-0), KH-580 (Sun et al. [2016\)](#page-7-0), CPDMS (Ma et al. [2018](#page-7-0)), ODA (Rong et al. [2019\)](#page-7-0), FAS (Yu et al. [2019](#page-7-0))



Fig. 4 a Contact angles of the RCOCl-grafted cotton filter fabric after each oil-water separation. **b** Separation efficiency and oil flux of the RCOCl-grafted cotton filter fabric of each cycle. Insets are optical photos of the liquid before and after

decrystalization (Arce et al. [2020;](#page-6-0) Lee et al. [2020a,](#page-7-0) [b](#page-7-0)), which may help to break up the hydrogen bond between hydroxyls on glucose monomers, making them exposed to reaction system and be more active to be reacted with hydrophobic layers. Therefore, in our second work, the hydrophobic durability was improved largely that the CA can remain above  $120^{\circ}$ after 2.5 h droplet holding time. However, it is hard to

filtration. The Blue phase is  $CuSO<sub>4</sub>$ -dyed aqueous phase and the transparent phase is  $CHCl<sub>3</sub>$  oil phase. c The first filtration process of oil–water separation

control active sites through radical polymerization, resulting in less uniform grafted alkyl chains. Besides, ARGET-ATRP method is too complex and time costly. To further improve hydrophobic durability and simplify the modification process, we utilize an efficient reaction between acyl chloride and hydroxyl. Unlike radical reaction, this reaction relies on the excessive TEMED and the activated -OH. Thus, the

active sites distribution can be more uniform. As can be seen from Fig. [3a](#page-4-0), the modified sample via RCOCl graft in this work exhibits the improved hydrophobic durability, whose CA can remain above  $130^{\circ}$  after 3 h and  $120^{\circ}$  after 5 h droplet holding time. The corresponding CA images are shown in Fig. S5. RCOCl is also comparable with other graft compounds. Many hydrophobic modified fabrics display high initial CA values above 140°, such as ODTES (Shang et al. [2010\)](#page-7-0), GS (Muresan et al. [2013](#page-7-0)), CPDMS (Ma et al. [2018\)](#page-7-0) and FAS (Yu et al. [2019](#page-7-0)) modified samples (Fig. [3](#page-4-0)b). As can be seen, all the materials shown in Fig. [3b](#page-4-0) including our sample display similar hydrophobicity. However, these reported materials have various unexpected aspects. Such as FAS, it is a fluorine-contained material, which is environmental unfriendly. Others like ODTES and GS, the graft compounds have to be synthesized firstly before graft reaction, which is not as convenient as the directed used RCOCl. Moreover, CPDMS and ODA methods involved high temperature reaction above  $100^{\circ}$ C, which is energy costly compared with RCOCl method in this work. Without the above mentioned drawbacks, the modified sample via RCOCl method can be facile synthesized and maintain comparable hydrophobic properties with other reported materials at the same time. Although it is not superior to other graft compounds, RCOCl can reach their levels. Besides, the cellulose-based substrate of the sample is both ecofriendly and costless.

In addition, relevant properties such as anti-fouling, washing and abrasion performance were also conducted in this work. As for anti-fouling property, we conducted three experiments. Fig. S6 compares the nano-TiO<sub>2</sub> suspension droplets sliding down on the hydrophobic modified cotton filter fabric and the raw sample at a tilted angle of  $20^{\circ}$ . The raw sample absorbed the droplet as soon as it touched the surface. Instead, droplet slid down quickly on the modified sample surface and we have to slow down at  $\times$  0.2 speed, or we can not snapshot the sliding process pictures. Commercial aqueous drinks were also used to test the anti-fouling property of the modified sample. As shown in Fig. S7, tea, cola, vinegar and juice droplets can be held stably on the modified sample, while they are collapsed or adsorbed on the unmodified surface. These four drink droplets also slide down quick on the modified sample surface (Fig. S8). The above phenomena consistently confirm the good anti-fouling property of the hydrophobic modified cotton filter fabric. Washing and abrasion test were conducted to evaluated the mechanical durability of the hydrophobic modified cotton filter fabric. As can be seen from Fig. S9a, the CA values of the modified sample can remain at around  $140^\circ$  after washing for ten times, indicating a nice anti-washing performance. Fig. S9b displays the abrasion test for the modified sample. In the situation of 200 g counterweight put on the slide, the CA values can remain at around  $140^{\circ}$  after 180 cycles. When the counterweight was changed to 500 g, the CA values drops slightly after abrasion and finally maintains at around  $132^{\circ}$  after 210 cycles. In another word, the modified sample still exhibits well hydrophobicity after 390 cycles abrasion, displaying an outstanding anti-abrasion performance. These two performances both confirm a well mechanical property and durability of the modified sample, which is suitable for practical applications.

After fundamental characterizations, we performed a membrane filtration application research on the hydrophobic modified cotton filter fabric. Oil–water separation was conducted on an extraction filter with effective area of  $3.14 \text{ cm}^2$ . For each turn, oil phase was 5 mL CHCl<sub>3</sub> and aqueous phase was 5 mL CuSO<sub>4</sub>dyed water. The above mixture was poured into the asmentioned extraction filter and filtered drop by drop by gravity. The gravity-driven oil flux can be calculated by the formula shown in the supporting information. Figure [4](#page-4-0)a is the average CA values after each filtration, from which can be seen that there is only few angles declined compared with the initial one. After three times filtration, the average CA value can still be above 130°, suggesting a fine recyclability of the RCOCl-grafted membrane. Figure [4](#page-4-0)b displays the separation efficiency and oil flux of each cycle. Separation efficiency can remain at 99 % after each cycle. However, the oil flux decreases after every cycle from the beginning of 522. 08 L m<sup>-2</sup> h<sup>-1</sup> to 282.33 L m<sup>-2</sup> h<sup>-1</sup> then further decreases to 117.69 L  $m^{-2}$  h<sup>-1</sup>. This can be explained by the hydrophobic effect that the oil droplets will adhere during each separation process, making it fewer and fewer holes for oil pass through. Insets are the optical photo of the liquid before and after filtration, showing nearly 5 mL aqueous phase recovery. Figure 4c is the screenshots of the first oil–water separation video recording. We started to record as we poured the mixture into the <span id="page-6-0"></span>extraction filter. The first oil drop filtered was observed at 13 s and the oil phase finished filtration at 2 min 03 s. And the aqueous phase could be hold for at least one and a half minutes by the obtained hydrophobic modified cotton filter fabric. The second and third filtration processes are also provided in Fig. S10 and S11. Similar with the first process, the aqueous phase holding times are also about one and a half minutes, indicating a maintaining aqueous phase holding property after three times separation.

In conclusion, we reported a facile hydrophobic modification method of  $CS_2$  activation and RCOCl graft on cotton filter fabric. Through a serial optimizing experiments, alkyl chains were successfully grafted on the glucose monomers, which is confirmed by FTIR and TG. The obtained fabric exhibits both high hydrophobicity ( $CA = 141.8^\circ$ ) and hydrophobic durability ( $CA_{5h} = 126.3^{\circ}$ ), which is comparable with those of other reported grafted fabrics. Besides, the modified sample also exhibits excellent anti-fouling property against various aqueous drinks as well as anti-washing property with almost no CA decrease after 10 cycles washing. Much more surprised is that this RCOCl-grafted fabric displays outstanding antiabrasion performance that it can bear 180 cycles abrasion with 200 g counterweight and 210 cycles under 500 g counterweight totally. The average CA value can remain above  $131^\circ$  finally. For membrane filtration application, the RCOCl-grafted fabric shows expective oil–water separation with 99 % separation efficiency and 522. 08 L m<sup>-2</sup> h<sup>-1</sup> gravity-driven oil flux. This cost-effective hydrophobic modified cotton filter fabric has potential application in future oil– water separation. And this time cost-less and energy cost-less chemical graft technology may rich the surface hydrophobic modification routes of cellulosebased substrates.

#### Declarations

Conflict of interest The authors declare that they have no conflict of interest.

Human or animal rights The work described in this article did not involve human participants and or animals.

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