

Superhydrophobic Polyurethane Foam Modified by Graphene Oxide

Hai-dong Liu,¹ Zheng-ying Liu,¹ Ming-bo Yang,¹ Qi He²

¹College of Polymer Science and Engineering, State Key Laboratory of Polymer Materials Engineering, Sichuan University, Chengdu 610065, Sichuan, People's Republic of China

²China Bluestar Chengrand Chemical Co., Ltd., 4th Section Renmin South Road, Chengdu 610041, People's Republic of China
Correspondence to: M. Yang (E-mail: yangmb@scu.edu.cn) or Q. He (E-mail: heqi998@263.net)

ABSTRACT: Graphene with amine group was covalently grafted on the polyurethane foam with nitrile group to form superhydrophobic foam. Fourier transform infrared spectroscopy (FTIR), atomic force microscopy (AFM), thermogravimetric analysis (TGA), optical contact angle measuring device, and scanning electron microscopy (SEM) were used to characterize the foam, showing the dodecane diamine was successfully grafted onto graphene oxide and the graphene oxide modified by dodecane diamine was successfully grafted onto polyurethane foam. Moreover, the modified foam exhibited a high contact angle ($159.1 \pm 2.3^\circ$) compared to unmodified foam ($121.4 \pm 3.2^\circ$). And that is due to the foam modified by amidation of graphene oxide can enhance the surface roughness and reduce the surface energy of the foam. Owing to modified foam was extremely hydrophobic and preferentially adsorbed oil other than water, the sorption capacity of the modified foam for toluene, gasoline, and diesel is 41, 27, and 26 g/g, respectively. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2013

KEYWORDS: grafting; foams; adsorption; polyurethanes; functionalization of polymers

Received 28 February 2013; accepted 11 April 2013; Published online

DOI: 10.1002/app.39406

INTRODUCTION

Oil leaking during the process of oil exploring, transportation, storing, and using usually imposes a series of problems on the environment.^{1,2} To avoid the adverse impacts of oil spills on environment, there is an urgent need to develop a wide range of techniques for cleaning up spilled oil effectively. The conventional treatments of oil spill include usages of oil dispersant, oil gelling agent, and oil sorbent.^{3–7} Among the existing urgent techniques, the use of sorbents is generally considered to be one of the most promising countermeasures because it can efficiently remove/recover oil from the water surface.^{8,9} Thus, effective sorbent is of great interest. The traditional polyurethane foam has characteristics of high adsorption rate, low density, and easily scalable fabrication. However, it usually absorbs both water and oils.^{10,11} Recently, surfaces with superhydrophobic property have attracted considerable interest in the field of oil–water separation because they only absorb oil while repel water completely.^{11–13}

Graphene is a two-dimensional carbon nanofiller with a one-atom-thick planar sheet of sp^2 bonded carbon atoms densely packed in a honeycomb crystal lattice, has attracted much attention since its discovery by Novoselov et al.¹⁴ It is regarded as

“the thinnest material in the universe” with remarkable properties, such as high thermal conductivity, superior mechanical properties and excellent electronic properties.^{15–23} Except for the advantages mentioned hereinbefore, graphene materials have recently been reported to possess exciting hydrophobic properties.^{12,24–27} It is clear that the superhydrophobic surface can be obtained by modified graphene oxide, suggesting the potential application in superhydrophobic coating. However, to the best of our knowledge, few studies involving the graphene-based materials with superhydrophobic properties have been reported to date, only Tai and co-workers reported that foam was coated with a functionalized graphene oxide via a facile dip coating method to prepare superhydrophobic foam.¹¹

In this study, we first report an effective protocol to prepare superhydrophobic foam with graft polymerization. The functionalized graphene oxide with primary amine groups is directly grafted onto the foam with nitrile group by in situ amidation to prepare superhydrophobic foam. The foam was further characterized through infrared analysis (FTIR-ATR), scanning electronic microscopy (SEM), and contact angle meter. The hydrophobicity of the modified foam was enhanced because of grafting graphene oxide onto foam, which could increase the surface roughness and decrease surface energy of the modified

Additional Supporting Information may be found in the online version of this article.

© 2013 Wiley Periodicals, Inc.

Table I. The Recipe of the PU Foam Sample

Ingredients	Phr ^a	Remarks
HTBN	100	OH value = 0.56 mmol/g
Catalyst (Dabco 33LV)	3	Blowing catalyst
Catalyst (DABCO T-9)	0.5	Gelation catalyst
Surfactant (Niax L580)	4	Surfactant
Distilled water	2	Chemical blowing agent
Isocyanates	1.05 ^b	NCO (wt %) = 31.5

^aRepresents as parts per hundred of polyols by weight.

^bNCO index is 1.05.

foam. The hydrophobicity of the modified foam increased with the increasing of the surface roughness and decreasing of the surface energy,^{12,24} hence the performance of the modified foam will be improved as sorbents of oil in aqueous conditions.

EXPERIMENTAL

Materials

Graphene oxide (GO) was supplied by Nanjing Jcnano technology Co., China. Ethanol and cyclohexane were all analytically pure and purchased from Tianjin Damao Chemical Reagent Co., China. 1,12-Dodecane diamine (DADD) was obtained commercially from Zibo Guangtong Chemical Co., China. Deionized water was prepared in laboratory. Hydroxyl-terminated liquid butadiene–acrylonitrile rubber (HTBN, OH% = 0.56 mmol/g, nitrile group content $20 \pm 2.0\%$, provided by Zibo Qilong Chemical Industry Co., China). Polymeric MDI (Desmodur 44V20L, Polyurethane Raw Materials EMEA) was used as the isocyanates. Other ingredients used for the preparing of foam were surfactant (Niax L580), catalysts triethylenediamine (Dabco 33LV) and stannous octoate (DABCO T-9). Deionized water was employed as the chemical blowing agent. The recipe for the preparation of foam is shown in Table I.

Amidation of Graphene Oxide

First, 300 mg graphite oxide was dispersed by ultrasonication and mechanical stirring in 150 mL deionized water in a three-neck flask for 30 min at $25 \pm 3^\circ\text{C}$. Second, 150 mL ethanol containing 600 mg DADD was added into the three-neck flask. Afterward, the suspension was refluxed with mechanical stirring for 20 h at 90°C .^{12,24} The resulting product (GO–DADD) was diluted with 1000 mL ethanol and separated by filtration using a nylon membrane ($0.02 \mu\text{m}$, Whatman), and then washed 20 times with ethanol to remove the physically absorbed DADD. Finally, GO–DADD was dried at 80°C overnight in an oven.¹² The reaction equation is described in Figure 1.

Preparation of PU Foam Sample

According to the recipes as given in Table I, the ingredients were mixed in a high pressure foaming machine (JHPK-IIIB235, provided by company profile of Beijing JingHua Park Polyurethane Equipment Co., China.) at 45°C , and then injected into a wooden mold on site (dimension: length \times width \times height = 100 cm \times 50 cm \times 50 cm) via the mixing head of the high-pressure foaming machine (the injection pressure was adjusted to 40 kg/cm^2). The foam was taken out after 5 min, and allowed to cure for 10 min at room temperature.²⁸ The skin layer (10 mm) of the prepared foam was removed, and then cut sample with dimension of 2 cm \times 2 cm \times 2 cm. The amount of isocyanates required for the reaction with distilled water and hydroxyl-terminated liquid butadiene–acrylonitrile rubber was calculated from their equivalent weights. To ensure the success of the reaction, an excess amount of isocyanates (NCO/OH = 1.05) was used.²⁹ The preparation of polyurethane foam is shown in Figure 2.

Grafting Procedure

First, 300 mg GO–DADD was dispersed in 150 mL mixture of cyclohexane and ethanol (the volume ratio is 1 : 0) in a three-neck flask with the aid of ultrasonication for 5 min. Subsequently foam samples (0.5 g) was added, and the three-neck

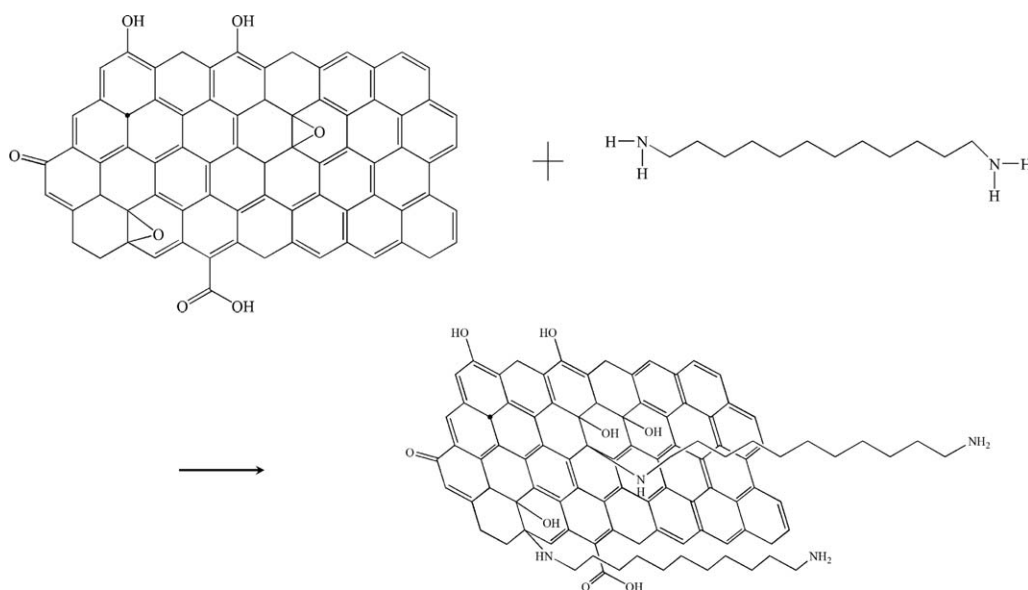


Figure 1. Schematic illustration of the reaction between GO and DADD.

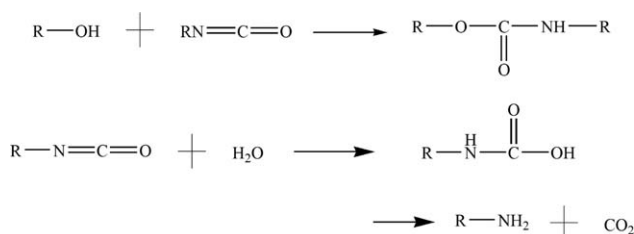


Figure 2. The chemical equation of the preparation of polyurethane foam.

flask was transferred to a oil bath (95°C) with magnetic stirring and reflux condensation for 24 h to carry out the reaction.³⁰ The end point of the reaction was determined by FTIR monitoring. When the absorption band of nitrile group at 2237 cm⁻¹ was disappeared, the reaction was ended. The modified foam was washed for several times by the method of adsorption–disadsorption with ethanol, and then dried at 60°C under vacuum for 48 h. The modified foam was named as PU–DADD–GO. The grafting reaction scheme was shown in Figure 3.

Characterization

Structural Analysis. The FTIR characterization was performed at ambient temperature with an infrared spectrometer (Thermo Scientific Nicolet 6700). Thermogravimetric analysis (TGA) was measured on a thermogravimetric analyzer (Shimadzu DT-40, Japan). Contact angle measurements were performed at ambient conditions using DSA100 optical contact angle measuring instrument (Germany cruise Co.). Scanning electron microscopy (SEM; JSM-5900LV) was used to investigate the surface morphology. The samples were covered with gold, and then the samples were observed in a SEM instrument at an acceleration voltage of 20 kV.

Sorption Capacity Test. The method developed for the measurement of solvent and oil sorption capacity of the sorbent was based on ASTM F726-99: Standard Test Method for Sorbent Performance of Adsorbents. For solvent or oil sorption tests, toluene, gasoline, and diesel (50 mL) was poured into three 100 mL beakers, respectively. The sorbent (foam) was weighed

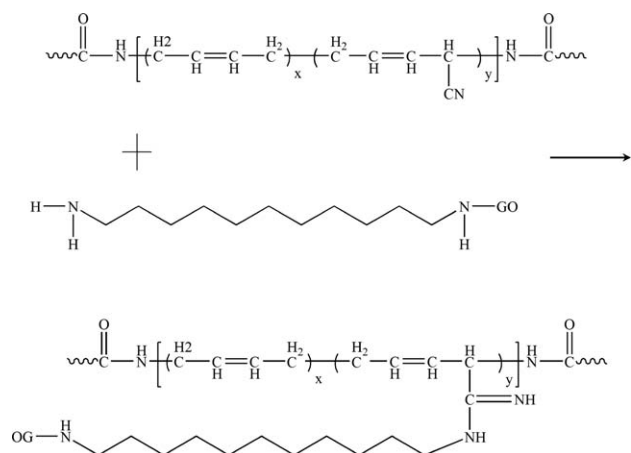


Figure 3. Reaction scheme for grafting polymerization of DADD–GO onto foam surface.

and the value was recorded, then it was immersed into the oil. In general, after 10 min ± 10 s of immersion, the sorbent was removed and allowed to drain for 60 ± 3 s. The saturated sorbent was then immediately transferred to a pre-weighed weighing bottle and weighed.⁸ The solvent or oil sorption of sorbent was calculated using the following equation:

$$\text{Sorbed pollutant (g/g sorbent)} = \frac{m_t - m_{\text{dry}}}{m_{\text{dry}}}$$

where m_{dry} is the initial dry weight of a sorbent and m_t is the weight of sorbent with solvent or oil absorbed. For water sorption tests, the sorbent was first weighed then placed into a 100 mL beaker with 50 mL deionized water. In general, after 60 min ± 10 s of immersion, the sorbent was removed and allowed to drain for 60 ± 3 s, then immediately transferred to a pre-weighed weighing bottle and weighed.⁸

Oil–Water Separation Capability of Foam. The oil–water mixture was prepared as follows according to the method described in Refs. 5 and 31. First, 8.42 g (10 mL) diesel oil was poured into a 100 mL container with the volume of one liter which contains ca. 50 mL NaOH solution (0.01 mol/L) as tracer reagent, respectively. Second, the foam (modified foam or blank foam) was first weighed and immersed into the oil–water mixture for 10 min. Third, the total mass of the foam was recorded. Fourth, the liquid absorbed by foam was detached by centrifugation at a speed of 1000 rpm for 10 min. Finally, the concentration of water in the detached liquid was determined by acid-based titration.

RESULTS AND DISCUSSION

Synthetization of GO–DADD

The nucleophilic substitution reaction between the primary amine groups of DADD and the epoxide groups of GO occurred during the refluxing.^{12,24} Figure 4 depicts the FT-IR spectra of GO, DADD, and GO–DADD. The typical peaks of GO appear at 1738 cm⁻¹ (C=O carboxyl stretching vibration),

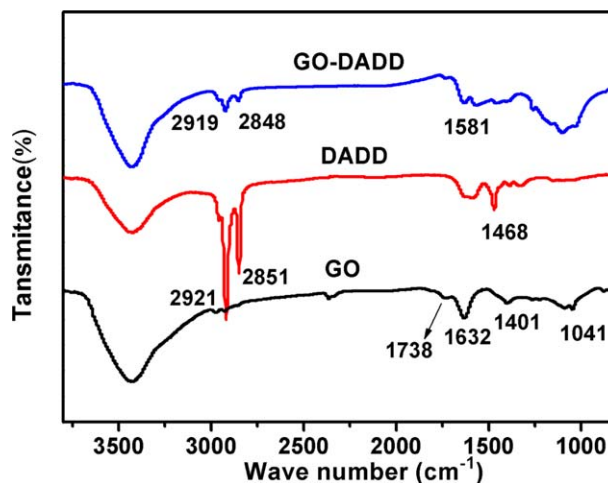


Figure 4. The FTIR spectra of the GO, DADD, and GO–DADD. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

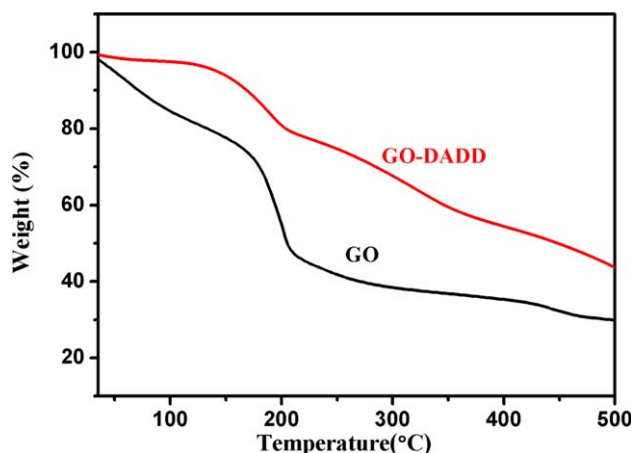


Figure 5. TGA curves of GO and GO-DADD. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

1632 cm^{-1} (C=C in aromatic ring), 1401 cm^{-1} (C–OH stretching), and 1041 cm^{-1} (C–O–C in epoxide).^{12,24} In addition, the wide peak appearing at $3000\text{--}3500\text{ cm}^{-1}$ could be assigned to the hydroxyl groups (–OH). While in the FT-IR spectrum of GO-DADD, the two new peaks at 2919 and 2848 cm^{-1} resulting from the –CH₂ stretching of the dodecyl chain implies the existence of the dodecyl chain on GO-DADD.^{12,24} Furthermore, a new peak at 1581 cm^{-1} (N–H stretching vibration) appears, indicating a new chemical bond was formed (–C–NH–C–) due to the reaction between the epoxide group and the amine group.¹² These results suggest that the chemical reaction of DADD with GO.^{12,24} The schematic illustrating the functionalization of GO with DADD is shown in Figure 1.

Figure 5 shows the TGA curves for GO and GO-DADD. Below 100°C , GO showed a gradual weight loss of $\sim 16\%$ due to desorption of water, while, GO-DADD showed nearly zero weight loss. The phenomenon indicates that GO modified by DADD grafting can enhance the hydrophobicity of GO, that minimizes the amount of absorbed water.^{12,24}

GO-DADD Grafting onto Foam

The reaction scheme for the grafting polymerization of GO-DADD onto foam is shown in Figure 3. The FT-IR characterization can help determine the end point of the reaction at which the signal of the nitrile group (C≡N) disappears. The IR spectra of blank PU foam and modified PU foam are shown in Figure 6. In Figure 6, the nitrile group signal of the blank foam sample (PU) appears at around 2237 cm^{-1} , whereas in the modified foam sample (PU-DADD-GO), the peak at 2237 cm^{-1} disappears and a new peak appears at around 1645 cm^{-1} which is the characteristic peak of (N–C=N) in PU-DADD-GO.³⁰ All these results support the reaction of GO-DADD with foam, which undoubtedly indicates that the GO-DADD was grafted onto foam.

In order to study the GO dispersion in PU foam, the morphology of all the foam samples was characterized by scanning electron microscopy (SEM). The SEM images of foam samples are shown in Figure 7. The surface characteristics of the modified foam (PU-DADD-GO) rendered homogeneous dispersion of

amidation of graphene oxide sheets (GO-DADD) in foam surface as shown in Figure 7(c), resulting from the successful grafting on foam by functionalized GO with DADD chains. The bright regions are attributed to the GO sheets for their high conductivity.³² It is obviously inspected that the bright regions are well-distributed in foam surface (dark region) without congregation. To compare the effects of grafting copolymerization on the GO dispersion, foam sample was modified by coating with GO using a facile dip coating method as reported in Ref. 11, and the foam was named as GO-coated foam. In Figure 7(b), there exists obvious aggregation in GO-coated foam surface, due to the unmodified GO has a poor compatibility with the foam and unevenly disperses in foam surface.^{22,23} While, the homogeneous dispersion of functionalized GO on modified foam (PU-DADD-GO) is ascribed to graft copolymerization between foam and amidation of graphene oxide, which enhanced the interfacial interaction between GO and foam matrix. What's more, the enhanced interfacial interaction between GO and foam matrix may be beneficial for the foam to maintain its hydrophobicity.

The Hydrophobicity of the Modified Foam

The surface energy and surface roughness has greatly influenced the hydrophobicity of materials.^{12,24,33} However, it is difficulty to measure the value of the surface energy and surface roughness of the foam due to its porosity. So, the pure polyurethane elastomer and the modified polyurethane elastomer by functionalized GO is prepared as reported in Refs. 34–36 to study the effect of GO on the hydrophobicity of polyurethane elastomer. The surface energy of polyurethane elastomer was measured by DSA100 optical contact angle measuring instrument, the value of the pure polyurethane elastomer and the modified polyurethane elastomer is 48.2 and 29.7 mJ/m^2 , respectively. To determine the average roughness, we used atomic force microscope (AFM) to measure the pure polyurethane elastomer and the modified polyurethane elastomer by GO. Figure 8 shows the 3D image of the pure polyurethane elastomer and the modified polyurethane elastomer. The modified polyurethane elastomer showed an average mean square roughness (Ra) of $\sim 0.73\text{ nm}$.

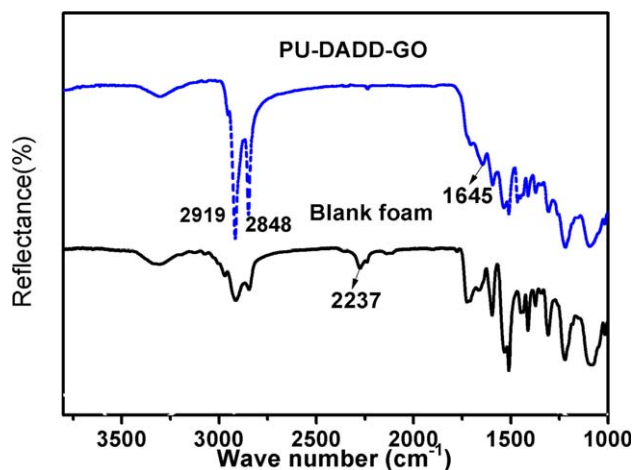


Figure 6. The FTIR spectra of the blank foam and modified foam (PU-DADD-GO). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

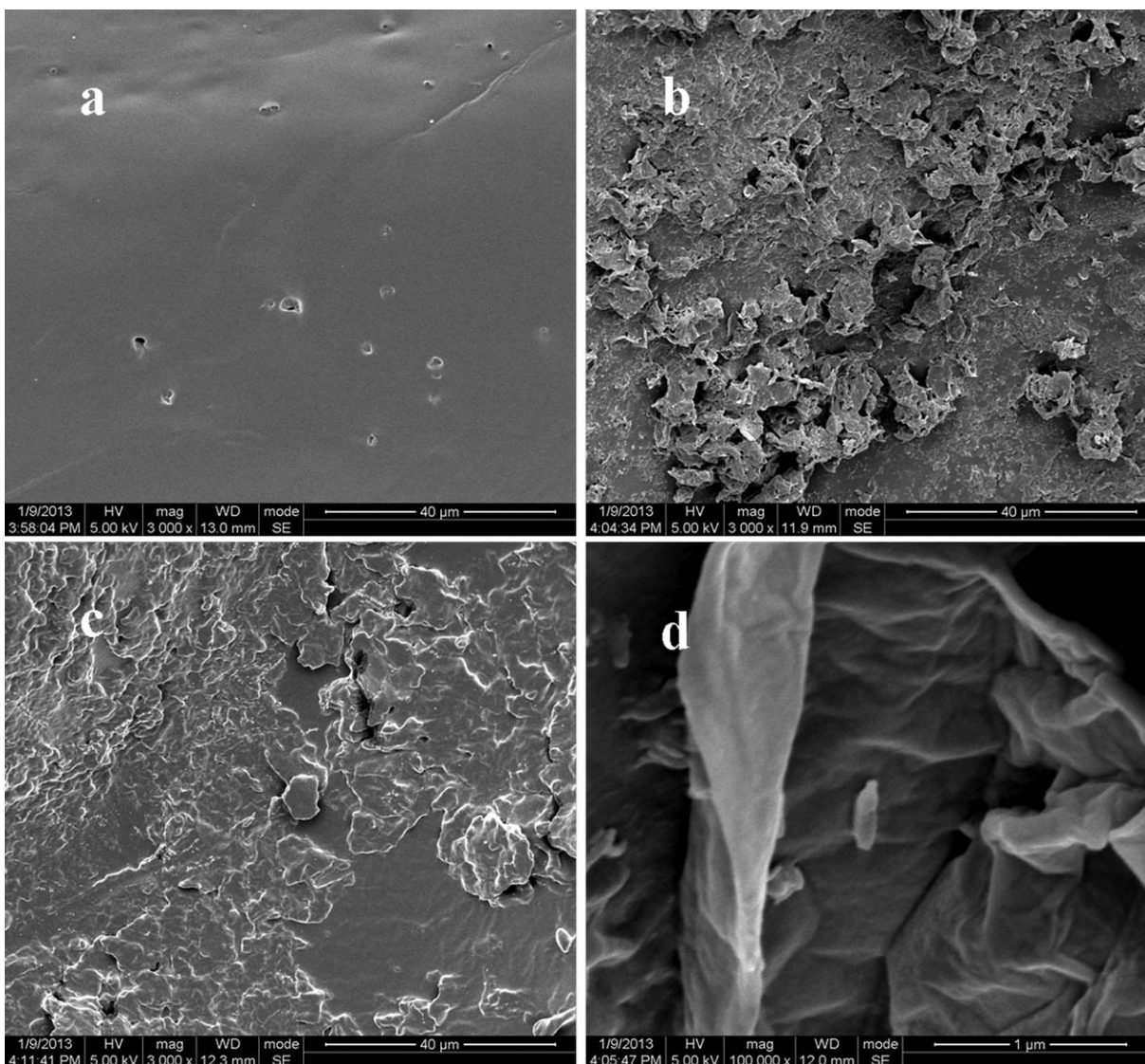


Figure 7. Typical SEM images of (a) the blank foam, (b) the GO-coated foam at a loading of 14.2%, (c) the modified foam (PU-DADD-GO) at a loading of 12.1%, and (d) the GO grafted on the modified foam (PU-DADD-GO) with higher magnification.

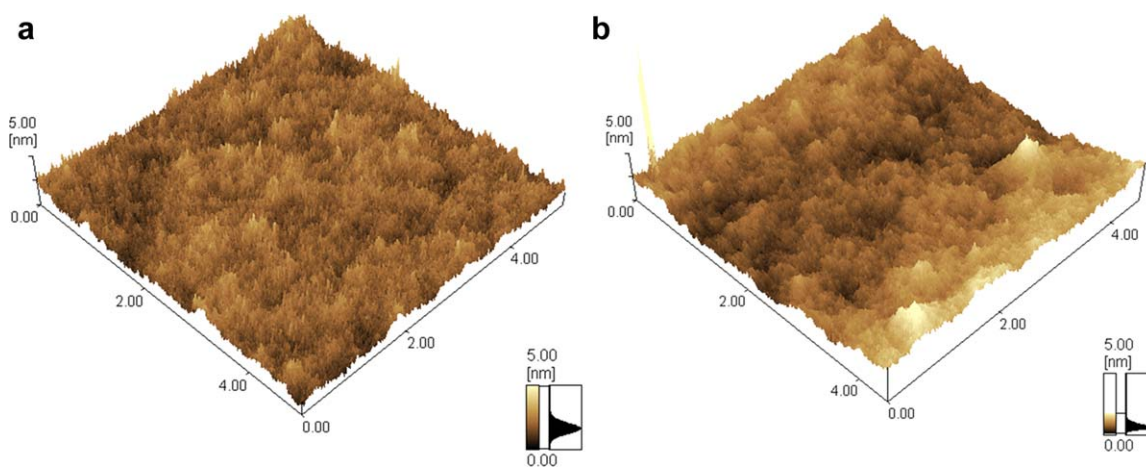


Figure 8. The AFM images of (a) pure polyurethane elastomer and (b) modified polyurethane elastomer. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

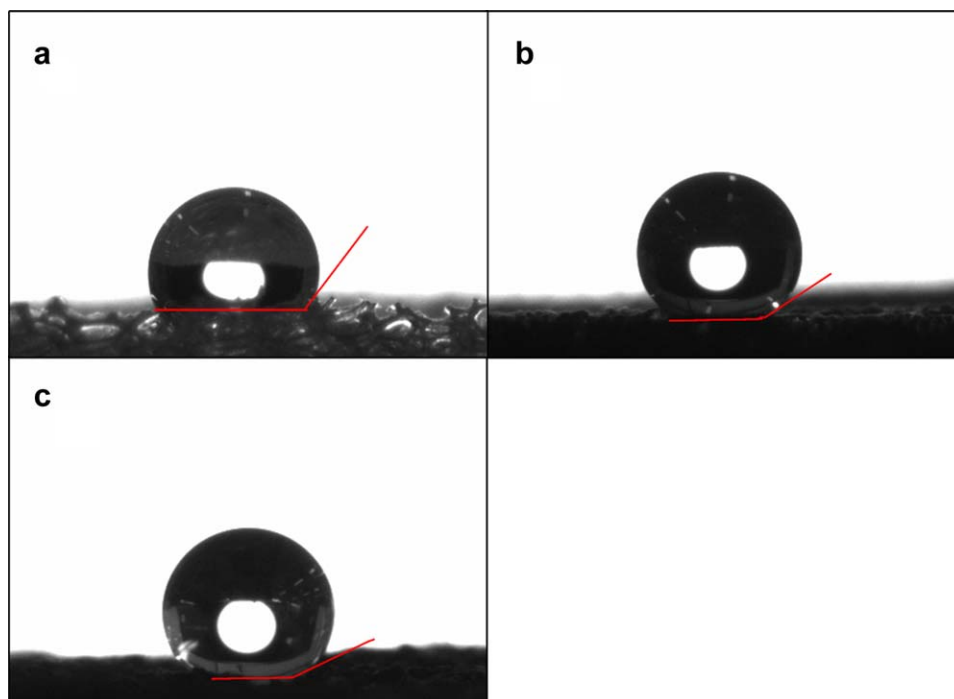


Figure 9. The optical image of a water droplet on (a) blank foam, (b) the GO-coated foam, and (c) the modified foam (PU-DADD-GO). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

This surface roughness is much higher than that of the pure polyurethane elastomer, which has a R_a of ~ 0.36 nm. The above results indicate that the hydrophobicity of the modified polyurethane elastomer was enhanced due to the adding of GO into polyurethane elastomer, which could increase the surface roughness and reduce the surface energy.

The effects of surface energy and surface roughness on the hydrophobicity of the modified foam are confirmed by the contact angle measurements. In Figure 9, the water contact angle of blank foam is $121.4 \pm 3.2^\circ$. While the contact angle of GO-coated foam is $155.3 \pm 2.4^\circ$ due to the foam coated by amidation of graphene oxide, which can enhance the surface roughness and reduce the surface energy of the foam as mentioned above. However, for PU-DADD-GO, one interesting phenomenon worth noting is that the water droplet could not fall down during our experiment due to the hydrophobicity of the modified foam is too strong to absorb the water droplet. To get the contact angle value, we knock the needle to let the drop fall down (Supporting Information Video). In Figure 9(c), the contact angle is $159.1 \pm 2.3^\circ$, higher than blank foam and GO-coated foam, which is due to the well-distributed hydrophobic graphene nanosheets in the micro-porous of the foam. Meanwhile, the micro/nano-textured structure of the hydrophobic graphene nanosheets in conjunction with the micro-porous structure of the foam creates a dually roughened surface; this special structure is benefit for the formation of superhydrophobic surface.

Oils Sorption Capacity of Foam

Modified foam (PU-DADD-GO) shows superior performance in absorbing toluene, gasoline, and diesel (Figure 10). The

sorption capacity of the modified foam for toluene, gasoline, and diesel is 41, 27, and 26 g/g, respectively. Compared with blank foam, the sorption capacity of modified foam (PU-DADD-GO) increased 7.9% for toluene, 3.8% for gasoline, and 8.3% for diesel oil. This phenomenon is a result of an increased the interaction force between the modified foam and oil compared with blank foam.

Oil–Water Separation

In Table II, the modified foam (PU-DADD-GO) exhibited little water pickup in oil–water system, this is due to the high hydrophobic property of the modified foam. The result demonstrates that the modified foam has excellent hydrophobicity compared to blank foam, indicating that the modified foam could be applied as efficient oil sorbent for spill emergency treatment.

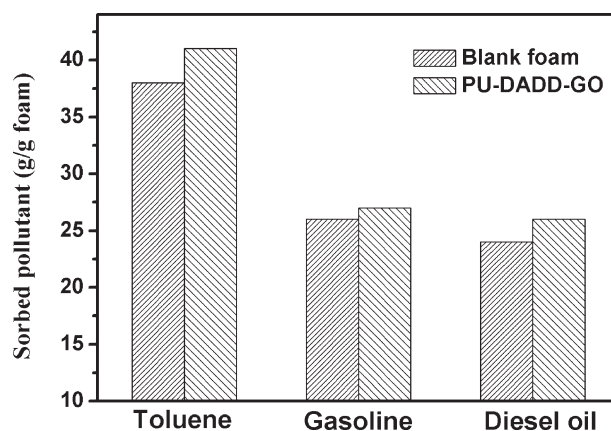


Figure 10. The maximum sorption capacities of blank foam (PU) and modified foam (PU-DADD-GO) for various pollutants.

Table II. The Separation Effect of Foam

Samples	Liquid collected from foam		Reclaim ratio of oil (%)
	Quality (g)	Oil content (wt %)	
PU-DADD-GO (0.52 g)	8.73	98.6	93.8
Blank foam (0.49 g)	11.32	53.5	68.9

CONCLUSIONS

In this study, the possibility of using foam modified by grafting with amidation of graphene oxide as a sorbent material for the recovery of oil spills was investigated. In contrast to the blank foam, the water sorption of modified foam (PU-DADD-GO) was reduced due to its superhydrophobic surface, meeting the requirements for environmental protection of aqueous system. The sorption capacity of modified foam for toluene, gasoline, and diesel is higher than blank foam. Therefore, the modified foam might be a promising substitute for the conventional sorbents used in the large-scale removal of oil spills or organic solvents from water surfaces.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the financial support from the National High Technology Research and Development Program of China (Contract No: 2008AA06A409).

REFERENCES

- Sokker, H. H.; El-Sawy, N. M.; Hassan, M. A.; El-Anadouli, B. E. *J. Hazard. Mater.* **2011**, *190*, 359.
- Cloud, R. M.; Choi, H. M. *Environ. Sci. Technol.* **1992**, *26*, 772.
- Atta, A. M.; El-Hamouly, S. H.; Al Sabagh, A. M.; Gabr, M. M. *J. Appl. Polym. Sci.* **2007**, *104*, 871.
- Shimizu, T.; Koshiro, S.; Yamada, Y.; Tada, K. *J. Appl. Polym. Sci.* **1997**, *65*, 179.
- Su, C. H. *Appl. Surf. Sci.* **2009**, *256*, 1413.
- Tanobe, V. O. A.; Sydenstricker, T. H. D.; Amico, S. C.; Vargas, J. V. C.; Zawadzki, S. F. *J. Appl. Polym. Sci.* **2009**, *111*, 1842.
- Yang, J. S.; Cho, S. M.; Kim, B. K.; Narkis, M. *J. Appl. Polym. Sci.* **2005**, *98*, 2080.
- Li, H.; Liu, L.; Yang, F. *Mar. Pollut. Bull.* **2012**, *64*, 1648.
- Ceylan, D.; Dogu, S.; Karacik, B.; Yakan, S. D.; Okay, O. S.; Okay, O. *Environ. Sci. Technol.* **2009**, *43*, 3846.
- Li, A.; Sun, H.-X.; Tan, D.-Z.; Fan, W.-J.; Wen, S.-H.; Qing, X.-J.; Li, G.-X.; Li, S.-Y.; Deng, W.-Q. *Energy Environ. Sci.* **2011**, *4*, 2062.
- Duc Dung, N.; Tai, N.-H.; Lee, S.-B.; Kuo, W.-S. *Energy Environ. Sci.* **2012**, *5*, 7908.
- Wenjuan, L.; Xiu-Zhi, T.; Hao-Bin, Z.; Zhi-Guo, J.; Zhong-Zhen, Y.; Xu-Sheng, D.; Yiu-Wing, M. *Carbon* **2011**, *49*, 4724.
- Si, F.; Zhang, L.; Zhao, N.; Chen, L.; Xu, J. *Prog. Chem.* **2011**, *23*, 1831.
- Novoselov, K. S.; Geim, A. K.; Morozov, S. V.; Jiang, D.; Zhang, Y.; Dubonos, S. V.; Grigorieva, I. V.; Firsov, A. A. *Science* **2004**, *306*, 666.
- Jun, C.; Yu, W.; Kai, K.; Yong, L.; Wei, Y.; Bang-Hu, X.; Ming-Bo, Y. *Polym. Int.* **2012**, *61*, 1031.
- Balandin, A. A.; Ghosh, S.; Bao, W.; Calizo, I.; Teweldebrhan, D.; Miao, F.; Lau, C. N. *Nano Lett.* **2008**, *8*, 902.
- Goyal, V.; Balandin, A. A. *Appl. Phys. Lett.* **2012**, *100*, 73113.
- Balandin, A. A. *Nat. Mater.* **2011**, *10*, 569.
- Shahil, K. M. F.; Balandin, A. A. *Solid State Commun.* **2012**, *152*, 1331.
- Shahil, K. M. F.; Balandin, A. A. *Nano Lett.* **2012**, *12*, 861.
- Balandin, A. A. *IEEE Nanotechnol. Mag.* **2011**, *5*, 15.
- Bin, L.; Olson, E.; Perugini, A.; Wei-Hong, Z. *Polymer* **2011**, *52*, 5606.
- Bin, L.; Wei-Hong, Z. *J. Mater. Sci.* **2011**, *46*, 5595.
- Lin, Z.; Liu, Y.; Wong, C.-p. *Langmuir* **2010**, *26*, 16110.
- Dong, X.; Chen, J.; Ma, Y.; Wang, J.; Chan-Park, M. B.; Liu, X.; Wang, L.; Huang, W.; Chen, P. *Chem. Commun.* **2012**, *48*, 10660.
- Ying-Kui, Y.; Cheng-En, H.; Wen-Jie, H.; Lin-Juan, Y.; Ren-Gui, P.; Xiao-Lin, X.; Xian-Bao, W.; Yiu-Wing, M. *J. Nano-part. Res.* **2011**, *13*, 5571.
- Fei-Peng, D.; Jing-Jing, W.; Chak-Yin, T.; Chi-Pong, T.; Xing-Ping, Z.; Xiao-Lin, X.; Yong-Gui, L. *Nanotechnology* **2012**, *23*, 475704.
- Harikrishnan, G.; Patro, T. U.; Khakhar, D. V. *Ind. Eng. Chem. Res.* **2006**, *45*, 7126.
- Thirumal, M.; Khastgir, D.; Singha, N. K.; Manjunath, B. S.; Naik, Y. P. *J. Appl. Polym. Sci.* **2008**, *108*, 1810.
- Che, A. F.; Nie, F. Q.; Huang, X. D.; Xu, Z. K.; Yao, K. *Polymer* **2005**, *46*, 11060.
- Rajakovic-Ognjanovic, V.; Aleksic, G.; Rajakovic, L. *J. Hazard. Mater.* **2008**, *154*, 558.
- Xu, Z.; Gao, C. *Macromolecules* **2010**, *43*, 6716.
- Zhang, L.; Zhao, N.; Li, X.; Long, Y.; Zhang, X.; Xu, J. *Soft Matter* **2011**, *7*, 4050.
- Menes, O.; Cano, M.; Benedito, A.; Gimenez, E.; Castell, P.; Maser, W. K.; Benito, A. M. *Compos. Sci. Technol.* **2012**, *72*, 1595.
- Nawaz, K.; Khan, U.; Ul-Haq, N.; May, P.; O'Neill, A.; Coleman, J. N. *Carbon* **2012**, *50*, 4489.
- Nguyen, D. A.; Raghu, A. V.; Choi, J. T.; Jeong, H. M. *Polym. Polym. Compos.* **2010**, *18*, 351.