Volume 1 | Number 1 | Jan 2013 | Pages 1–100

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ARTICLE TYPE

Synthesis of Polyurethane Containing Carbon-Carbon Double Bonds to Prepare Functionalizable Ultrafine Fibers via Electrospinning

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Polyurethane (TPU) containing carbon-carbon double bonds are synthesized for use as novel materials with the ability to form functionalizable ultrafine fibers via electrospinning. By adjusting the molecular structure, a series of TPU products with different amounts of carbon-carbon double bonds are obtained. After investigating the reactivity of the TPU with 1H, 1H, 2H, 2H-Perfluorooctanethiol, all of the TPU
10 samples exhibit effective functionalizability. The more carbon-carbon double bonds contained in the molecular structure, the stronger the functionalizability. Besides, these TPUs can easily form uniform ultrafine fibers in electrospinning. Upon comparison, the functionalizability of the electrospun fibers is similar to that observed in the bulk TPU materials. This work suggests a feasible methodology to produce a functionalized ultrafine fibrous carrier. Accordingly, TPU containing carbon-carbon double bonds is
15 expected to be exploited as a fibrous carrier of solid catalyst in the future.

1 Introduction

As one of the most convenient techniques to swiftly produce fibers of nano- or micro- meter size, electrospinning[1-5] has attracted great attention from scientific researchers during the
20 past decade. Owing to their high ratio of surface area to volume, [6,7] electrospun fibers have exhibited huge potential for fabrication of functionalized materials. Even today, the particular micro-structure of electrospun fibers is continually exploited in new applications requiring functional materials.[8-14]

25 In the area of chemical transition and refinery, catalysts are indispensable functional materials that effectively reduce the time and improve the efficiency of the chemical reaction. However, as we know, the traditional homogeneous catalyst has inevitable drawbacks of hard separation and difficult recycle.[15-18] Therefore,
30 establishing a way to create immobilized catalyst is considered in order to optimize catalyst performance. Accordingly, many inorganic and organic materials such as nanoparticle, [19] nanowire, [20] carbon nanotube, [21] mesoporous material, [22] and ion exchange resin[23] are starting to be used as carriers of immobilized catalysts.
35 In spite of using these carriers, the balance between highly effective catalysis and easy post-processing separation is still elusive. Thus, it is necessary to produce an effective carrier for the immobilized catalyst.

The structural characteristics of electrospun fibers present
40 interesting advantages in fabricating a carrier for immobilized catalyst. The effect of catalysis can be controlled by increasing the surface area to volume ratio of the fiber, that is, to reduce the fiber diameter as much as possible. Besides, the essential fiber structure can effectively maintain the ability to be separated from
45 the reaction system or product. Specially, the establishment of a functionalizable electrospun fibrous carrier offers an excellent solution for preparing highly effective immobilized catalyst. In this work, we will focus on the preparation of polyurethane containing carbon-carbon double bonds (thermoplastic
50 polyurethane, TPU) as the functionalizable material. It is shown that functionalizable electrospun fibrous carriers can be fabricated for this application. Additionally, the potential for functionalization is discussed.

2 Experimental

2.1 Materials

55 Itaconic acid, 1,4-butanediol(BDO), 1,6-hexanediol, 1,10-decanediol, *p*-Toluenesulfonic acid (PTSA), hydroquinone, tetrabutyl titanate (TBOT), isophorone diisocyanate (IPDI), ditinbutyldilaurate (DBTL), dibutylamine, bromocresol green,
60 2,2-Dimethoxy-2-phenylacetophenone (DMPA) were obtained from the Aladdin Reagents (Shanghai) Co., Ltd. Chloroform, methanol, actone, toluene, 2-butanone, tetrahydrofuran (THF), *N,N*-Dimethylformamide (DMF), hydrogen chloride were obtained from Sinopharm Chemical Reagent Co., Ltd.
65 1H,1H,2H,2H-Perfluorooctanethiol (TESH-6) was purchased from Hengtong Fluorine Co., Ltd. All chemicals were used as received.

2.2 Synthesis of polyester glycol

Itaconic acid (0.15mol), 1, 10-decanediol (0.18mol), together
70 with PTSA (0.1427g) as esterification catalyst, and hydroquinone (0.1900g) as radical inhibitor, were mixed in a three necked flask and reacted at 120°C for 30min, 145 °C for 60min and 160 °C for 140min in sequence under dry nitrogen atmosphere. After that, the moisture in the reaction system was removed by mean of a
75 vacuum pump for 1h. With addition of TBOT (0.3mmol) as

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polycondensation catalyst, the reaction was processed under vacuum- using firstly a vacuum pump for 20min and then an oil pump for 3.5h. The produced polyester glycol (DPDO) was dissolved in CHCl$_3$ and precipitated in methanol. The dissolving-precipitating process was repeated for several times for purification. Finally, the DPDO was dried under reduced pressure at room temperature for 12h.

By similar way, 1, 6-hexanediol (0.26mol), itaconic acid (0.2mol), together with PTSA (0.1902g) and hydroquinone (0.2533g) were stirred in a three neck flask and heated up gradually in steps such as 120 oC for 30min, 145 oC for 60min, and 160 oC for 120min under dry nitrogen atmosphere. Then, a dehumidification process was followed using a vacuum pump for 1 h. After that, TBOT (0.4mmol) as polycondensation catalyst was added to the mixture and stirred for reaction at 160 oC under vacuum condition attained by a vacuum pump for 20min at first and then an oil pump for 3.5h. The produced polyester glycol (HPDO) was dissolved in CHCl3 and precipitated in methanol for several times before being dried under reduced pressure at room temperature for 12h.

Itaconic acid (0.2mol), 1, 4-butanediol (0.27mol), together with PTSA (0.2122g) as esterification catalyst, and hydroquinone (0.2595g) as radical inhibitor were added into a three neck flask. The solvent mixture was stirred and heated in three steps while being purged by dry nitrogen in steps such as 140 °C for 60min, 150 °C for 60min, and 160 °C for 105min. Once the generated moisture was removed with a vacuum pump for 1h, TBOT (0.4mmol) as polycondensation catalyst was added to the mixture and stirred under vacuum (using a vacuum pump for 20min and switching to an oil pump for 2h). A cycling dissolution in CHCl$_3$ and precipitation in methanol process were carried out to purify the generated polyester glycol (BPDO) which was later dried under reduced pressure at room temperature for 12h.

2.3 Preparation of TPU containing carbon-carbon double bonds

TPU containing carbon-carbon double bonds were synthesized following FIGURE 1. Three types of polyester glycol prepared by 1, 10-decanediol, 1, 6-hexanediol, and 1, 4-butanediol and itaconic acid were respectively used to synthesize the TPUs (that is DTPU, HTPU and BTPU) containing different amounts of carbon-carbon double bonds. In the text below, the process of the synthesis will be elucidated in more detail.

IPDI and BDO were employed as hard segments for the fabrication of TPU with the use of DPDO as soft segments. Typically, the synthesis of TPU was carried out in a three necked flask equipped with a mechanical stirrer, a reflux condenser and a N2 inlet. Firstly, IPDI and polyester glycol together with DBTL as catalyst were dissolved in toluene and then stirred at 65 °C under dry nitrogen atmosphere. After 3h, the residual isocyanate (NCO) content was determined by titration according to DIN EN ISO 14896-2009. Sample liquid after dissolved in dry toluene reacts with excessive di-n-Butylamine, and back titrate residual di-n-Butylamine with hydrochloric acid up to the endpoint. The content of isocyanine is calculated from titration volume. Thereafter, an appropriate amount of BDO as chain extender was added drop by drop into the flask at 80°C. Once all NCO groups were reacted (the presence of NCO groups was checked through FTIR spectrometry by detecting the -NCO stretching absorbance band at 2230cm^{-1}), the mixture was concentrated by rotary evaporation under reduced pressure at room temperature and then purified three times by dissolving-precipitation in chloroform and methanol solution (50:50vol%). Finally, the purified TPU was dissolved in THF and poured into PTFE mould. Seven days was required for the solvent to evaporate thoroughly in the fume hood and the TPU to be shaped into a 100×100 mm^2 square sheet (with the thickness of 1mm) for later usage. Similar process was also conducted for HPDO and BPDO.

2.4 Reaction of the TPU with TESH-6

BTPU, HTPU and DTPU were respectively dissolved in THF for 8h. Excessive TESH-6 was added into the solution and stirred for 30min at first. Then, 2wt% DMPA was added into the solution under vigorous agitation for 20min to form a homogeneous mixture. The mixture was poured into a PTFE mould and exposed under a high-pressure mercury lamp (500W) at 365 nm for different irradiation times (5min, 10min, 30min, 60min,120min). After reaction, the TPUs are prepared to films for further characterization.

2.5 Electrospinning of the TPU

DTPU, HTPU and BTPU were respectively dissolved in a mixture solvent of DMF/THF (with the volume ratio of 2/3) at room temperature to form 20-35wt% solutions. The solutions were put into a 5mL syringe with a stainless needle (diameter=0.8mm) as the capillary. The syringe was then fixed on a pump (14-831-201, Fisher Scientific). Electrospinning was performed at a flow rate of 0.3mL/h. A high-voltage power supply (DW-P503-1ACDF, Dongwen High Voltage Power Plant, Tianjin, China) was used to produce voltages ranging from 5.5-8.0 kV and a drum covered with aluminium foil was used as the collector at the speed of 100rpm.

2.6 Reaction of electrospun TPU membranes with TESH-6

The electrospun membranes were cut into square pieces within 5 $\times$ 5cm^2, and then immersed in the mixture of TESH-6 and 2wt% of DMPA under the protection of nitrogen. After 2.5h, the immersed electrospun membranes were exposed immediately under the same UV lamp mentioned in section 2.4. The exposure time was set from 1min to 120min. After UV irradiation, the electrospun memberanes were washed with methanol for 5 times and dried at room temperature. A blank experiment of electrospun membranes without TESH-6 solution was conducted as well.

2.7 Characterization

The 1H NMR spectra of polyurethane were recorded on a Bruker AVANCE III 400M nuclear magnetic resonance spectrometer operating at 25°C and using acetone and CDCl$_3$ as solvents respectively. The FTIR spectra were collected with an Agilent Technologies Cary 600 Series Microscope FTIR. The morphology of the electrospun fibers was observed by a Hitachi S4800 field emission scanning electron microscopy. The chemical elements of the membranes were investigated by an AMETEK EDAX detector. The molecular weight was determined by a PL-GPC 220, and THF as the solution.

3 Results and discussions

3.1 Molecular structure of the TPU

FIGURE 1 shows the schematic process of the synthesis of TPU. Itaconic acid and three kinds of glycols are used to synthesize different types of polyester glycols. Consequently, after chain extension, three types of TPUs containing carbon-carbon double bonds are obtained from the experiment. The chemical structures of the TPUs are confirmed by FTIR. In FIGURE 2, two strong absorption bands at $1706cm^{-1}$ and $3330cm^{-1}$ are observed. It reflects the essential stretching vibration of carbonyl and amino groups in TPU. There is also a clear absorption band at $1643cm^{-1}$, which is attributed to the C=C double bond. It is clear that BTPU exhibits the highest absorption at $1643cm^{-1}$ while HTPU and DTPU show relatively weak absorption. This result indicates that BTPU has the highest quantity of carbon-carbon double bonds. In contrast, the amount of carbon-carbon double bonds in HTPU and DTPU is relatively low. 1H NMR spectra of TPUs are given in FIGURE 3. Accordingly, the practical structure and composition of TPUs can be calculated in detail. The related data are summarized in TABLE 1. BTPU has 3.60mmol/g carbon-carbon double bonds in its molecular structure. However, in HTPU and DTPU, only 2.22 and 1.94mmol/g of carbon-carbon double bonds are measured, respectively.

3.2 Functionalizability of the TPU

The functionalizabilities of DTPU, HTPU and BTPU are detected by reaction with TESH-6 under UV irradiation. In FIGURE 4 which exhibits the FTIR spectra of the DTPU, HTPU and BTPU reacted without (a, c, e) and with (b, d, f) TESH-6 under UV irradiation, no matter with or without TESH-6, after UV irradiation, the absorption band at $1643cm^{-1}$ decreases obviously. The longer reaction time is applied, the more thorough reaction occurs. This phenomenon adequately exhibits the reactivity of the carbon-carbon double bonds in the TPU. Without using TESH-6, the reaction must take place between carbon-carbon double bonds. As a result, it produces the effect of crosslinking. After addition of TESH-6 in the TPU, the FTIR spectra, exhibits additional absorption bands at $1145cm^{-1}$, $1191cm^{-1}$ and $705cm^{-1}$, which are related to the stretching vibration of C-F and C-S, respectively. Therefore, although crosslinking between carbon-carbon double bonds is very easy to happen under UV irradiation, the TPU obtained in the experiment also has the ability to react with functional agent such as TESH-6. In other words, the synthesized polyurethane containing carbon-carbon double bonds exhibit strong functionalizability.

The total amount of fluoride element existing on the surface of TPU films is further determined by EDS (see FIGURE 5). BTPU film shows larger amount of fluorine element (about 12-14%) than that of the HTPU and DTPU films. These results are in agreement with the amount of carbon-carbon double bonds in TPUs before reaction. It can be concluded that introducing as much carbon-carbon double bonds as possible is an effective method to increase the functionalizability of the TPU. FIGURE 5 also suggests an optimum reaction time to functionalize the TPU. Too-long reaction time will lead to the breaking of breaking of C-S bonds the thus reduces amount of fluorine element. It relatively supports the principle of functional agent choice during the process of the functionalization of the TPU.

3.3 Electrospinnability of TPU and functionalizability of electrospun fibers

DTPU, HTPU and BTPU are further used in attempt to prepare ultrafine fibers via electrospinning by choosing the appropriate electrospinning conditions; all of the TPU can form ultrafine fibers with uniform morphology (see FIGURE 6). Fiber diameters are all around 700nm. Then, the functionalizability of electrospun TPU fibers was systematically tested. Firstly, the change in fiber morphology before and after reacting with TESH-6 is observed by SEM. In FIGURE 7, almost no obvious change can be found in fiber morphology without using TESH-6 before and after UV irradiation. As we discussed above, the carbon-carbon double bonds tend to crosslink together under UV irradiation. This characteristic undoubtedly results in the durable performance under UV light and heat. In addition, this phenomenon brings a limited change in fiber morphology. However, after adding TESH-6, because of the reaction between carbon-carbon double bonds and TESH-6, the effect of crosslinking is weakened. Under these conditions, long-time UV irradiation will initiate the melting of TPU fibers. In FIGURE 7(f) and 6(h), after 10-30min of UV irradiation, TPU fibers begin to melt. With increase of the time of UV irradiation up to 60-120min, the TPU fibers finally merge together. The fiber morphology on the surface is nearly lost.

FIGURE 8 further gives FTIR spectra of the electrospun TPU ((a, b) DTPU, (c, d) HTPU and (e, f) BTPU) fibers reacted without (a, c, e) and with (b, d, f) TESH-6. As narrated in section 3.2, the change of the absorption band at $1643cm^{-1}$ defines the change of C=C double bonds in the TPU. On the other hand, the absorption bands at $1145cm^{-1}$, $1191cm^{-1}$, and $705cm^{-1}$ reflects the change of C-F and C-S bonds. According to FIGURE 7(b, d and f), the reaction between carbon-carbon double bonds and TESH-6 is successfully achieved.

FIGURE 9 exhibits the amount of fluorine element existed in electrospun TPU fibers after reacting with TESH-6 under UV irradiation. The highest amount of fluorine element can be observed on BTPU fibers and the fluorine element of HTPU and DTPU is relatively low. This result is almost the same as the TPU materials, so that the functionalizability of electrospun TPU fiber can be recognized by the property of TPU materials. Besides, higher amount of fluoride element is achieved in almost all of electrospun TPU fibers than in TPU films. As we know, TPU and TESH-6 is firstly reacted in solution. During this process, some carbon-carbon double bonds in TPU react with TESH-6, the other reacts with each other. After preparing TPU into films, the fluoride element is relative low. However, as to the electrospun TPU fibers, the grafting reaction takes place in a heterogeneous system. Carbon-carbon bonds, which particularly exist at the surface of electrospun TPU fibers, should react with TESH-6 as much as possible. It induces a little high grafting amount of fluoride element on electrospun TPU fibers.

Considering the possibility of the electrospun TPU fibers in preparation of solid catalyst carrier, the experimental data from FIGURE 7, 8 and 9 suggest the feasibility and the optimum condition of fabrication. Synthesizing TPU containing carbon-carbon double bonds and with the ability to form ultrafine fibers via electrospinning is an effective way to produce functionalizable fibrous precursor. The functionalizability of the

electrospun fibers is easy to be controlled by the molecular structure of TPU materials. After that, within suitable reaction time, the final fictionalization of the electrospun TPU fibers can be achieved. However, prolongation of the reaction time under
5 UV irradiation to functionalize the electrospun TPU fibers is not helpful. Too-long reaction time will lead to the loss of fiber morphology and thus has a detrimental effect on the functionalization.

Conclusions

10 Three types of polyurethanes with different amounts of carbon-carbon double bonds are synthesized. Characterization via FT-IR, EDS and SEM are conducted, and the functionalizability and electrospinning potential of the synthesized TPU are clearly exhibited. By means of UV irradiation, the functional agent
15 (TESH-6) can effectively react with the carbon-carbon double bonds in TPU. Systematic study of the relationship between the reaction time of functionalization and the amount of fluorine element showed the optimum conditions for functionalized electrospun TPU membranes. This research has established a
20 feasible way to produce functionalized ultrafine fibrous carrier materials. TPU containing carbon-carbon double bonds is expected to be exploited as a fibrous carrier of solid catalysts in the future.

Acknowledgements

30 Notes and references

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FIGURE Captions

FIGURE 1 Schematic process of the synthesis of TPU.

FIGURE 2 FTIR spectra of TPU

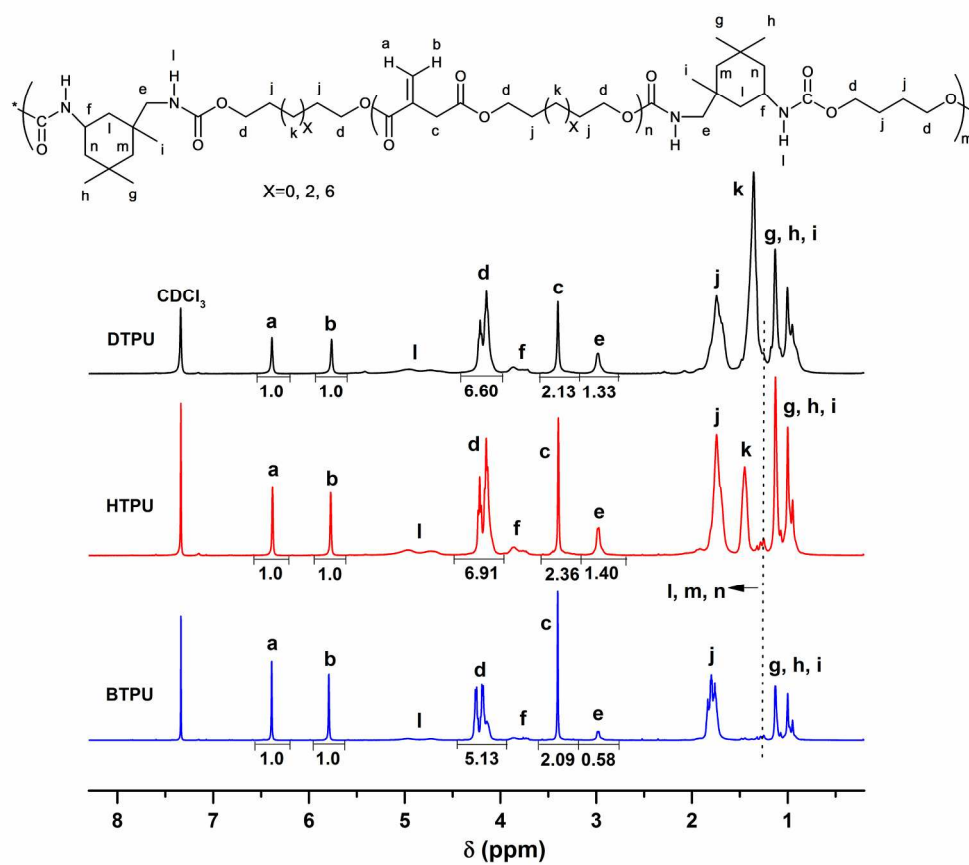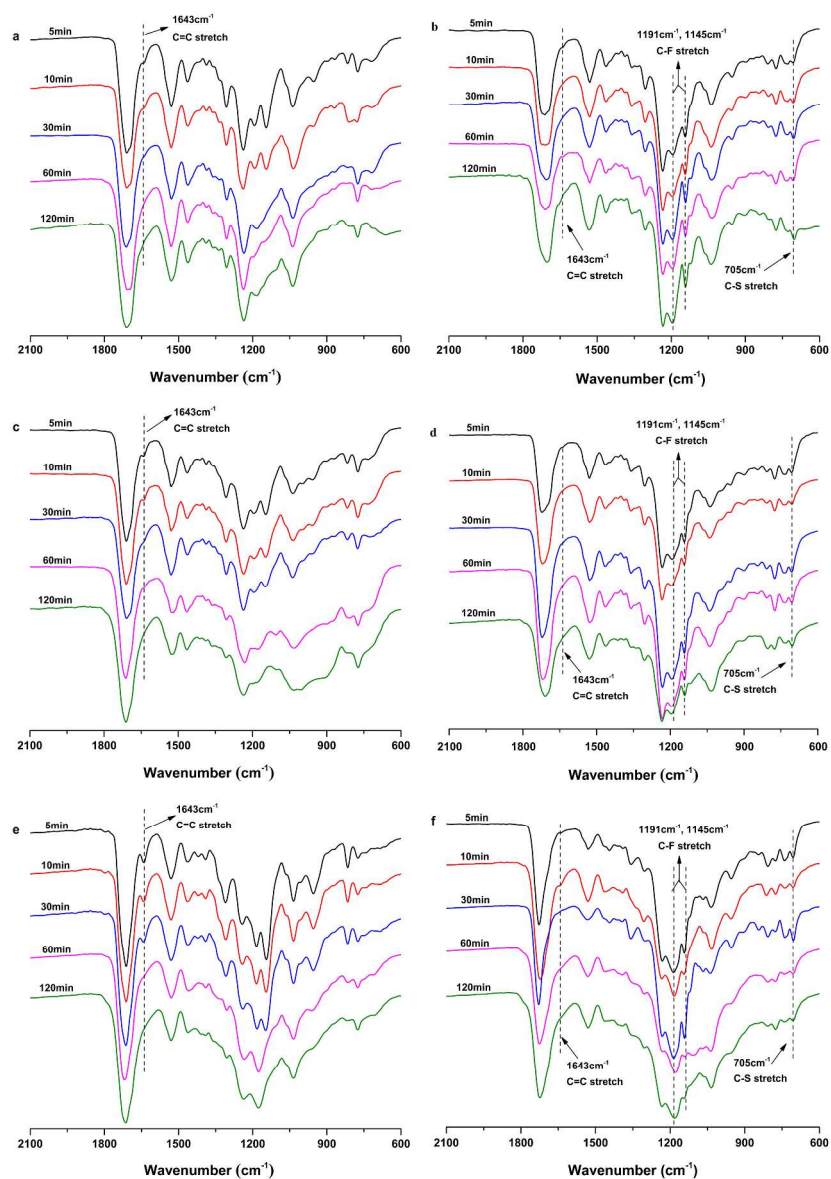**FIGURE 3** NMR spectra of TPU.

5   **FIGURE 4** FTIR spectra of the DTPU, HTPU and BTPU reacted without (a, c, e) and with (b, d, f) TESH-6 under UV irradiation.

FIGURE 5 Amount of fluoride element in TPU films after reaction with TESH-6.

FIGURE 6 SEM images of electrospun TPU fibers. (a) DTPU, (b) HTPU, (c) BTPU.

FIGURE 7 SEM images of electrospun BTPU fibers reacted without (a, c, e, g, i, k) and with (b, d, f, h, j, l) TESH-6 under UV irradiation within different time of (a, b) 0min, (c, d) 5min, (e, f) 10min, (g, h) 30min, (i, j) 60min, (k, l) 120min.

10   **FIGURE 8** FTIR spectra of the electrospun TPU ((a, b) DTPU, (c, d) HTPU and (e, f) BTPU) fibers reacted without (a, c, e) and with (b, d, f) TESH-6 under UV irradiation.

FIGURE 9 Amount of fluoride element in electrospun TPU fibers after reaction with TESH-6.

TABLE 1 Molecular structure of TPU.

TABLE 1 Molecular structure of TPU.

Synthesized TPU	Number-average molecular weight of polyester glycol section (g/mol)	Number-average molecular weight of TPU (g/mol)	Content of polyester glycol* (Wt %)	Amount of C=C double bond* (mmol/g)
BTPU	1870	0.69×10^4	69.6	3.60
HTPU	578	3.45×10^4	58.5	2.22
DTPU	714	2.34×10^4	69.3	1.94

* calculating from NMR spectra
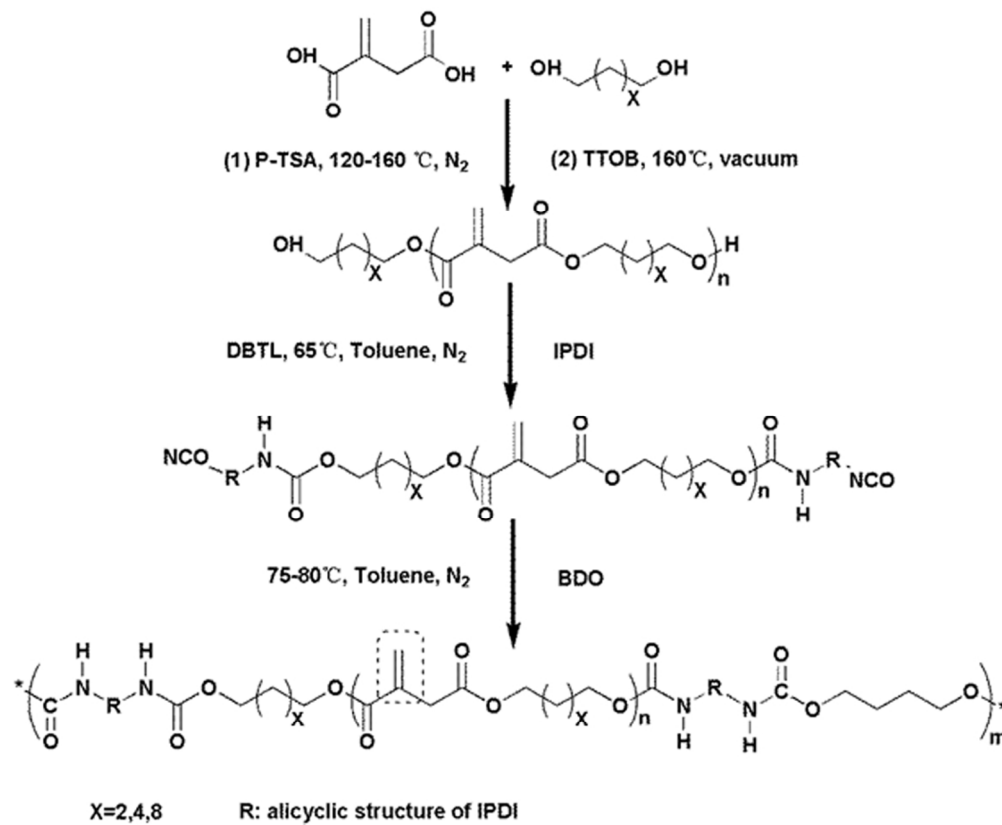
FIGURE 1 Schematic process of the synthesis of TPU
99x81mm (300 x 300 DPI)
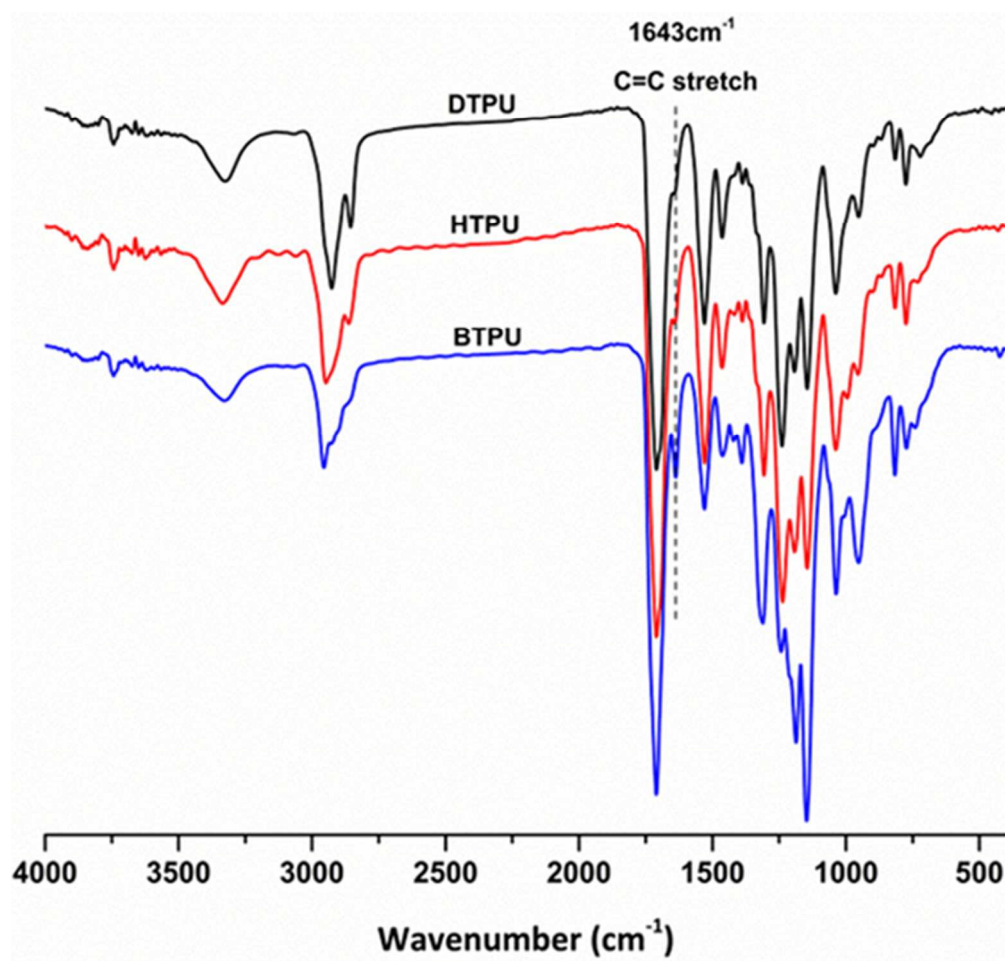
FIGURE 2 FTIR spectra of TPU
47x45mm (300 x 300 DPI)

FIGURE 3 NMR spectra of TPU
210x185mm (300 x 300 DPI)

FIGURE 4 FTIR spectra of the DTPU, HTPU and BTPU reacted without (a, c, e) and with (b, d, f) TESH-6 under UV irradiation
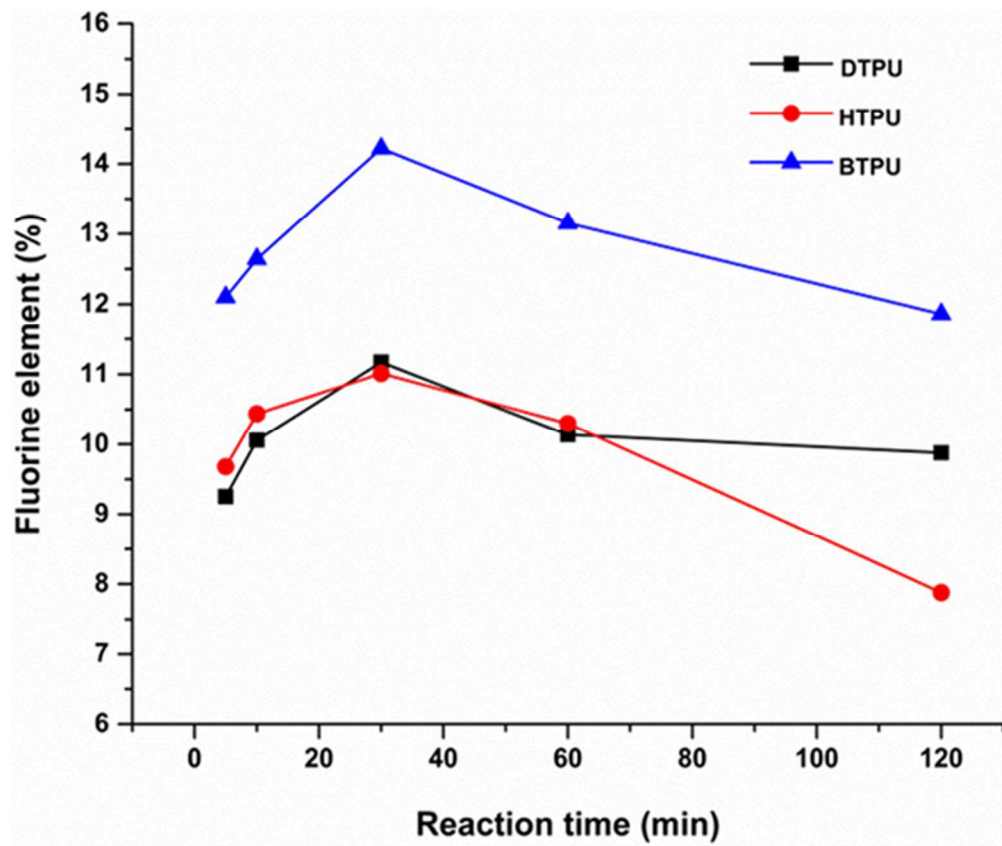124x173mm (600 x 600 DPI)

FIGURE 5 Amount of fluoride element in TPU films after reaction with TESH-6
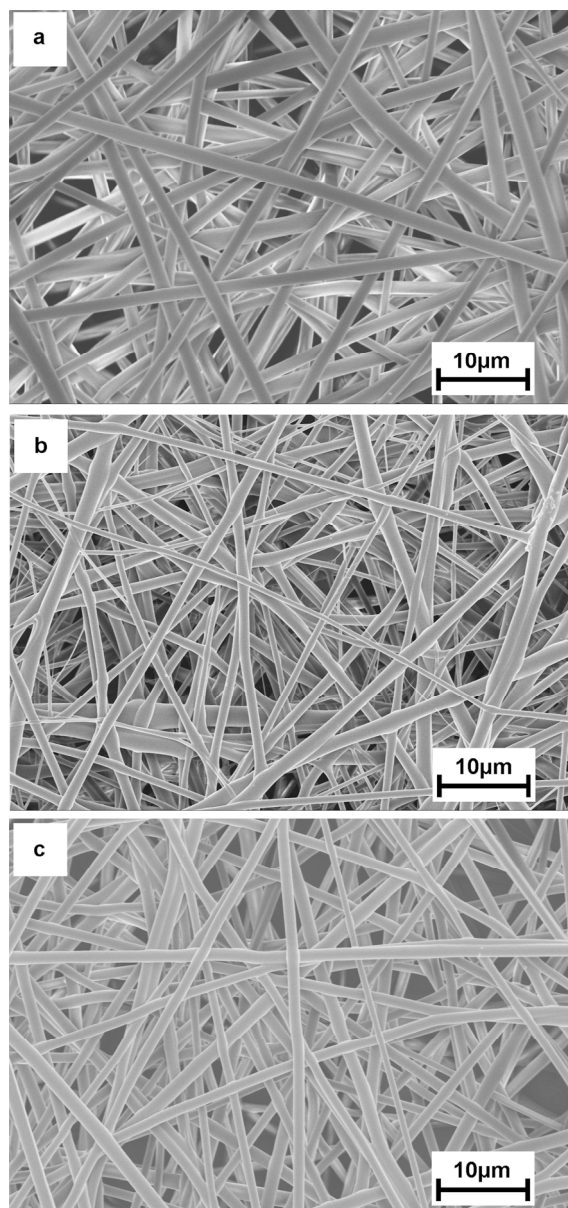50x42mm (300 x 300 DPI)

FIGURE 6 SEM images of electrospun TPU fibers. (a) DTPU, (b) HTPU, (c) BTPU
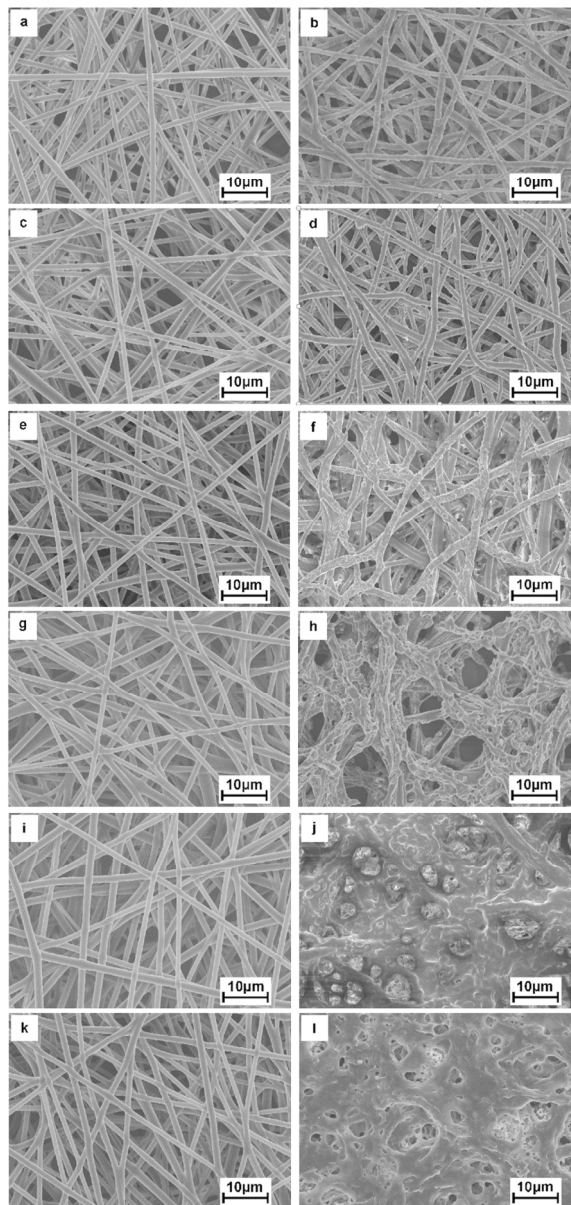127x270mm (300 x 300 DPI)

FIGURE 7 SEM images of electrospun BTPU fibers reacted without (a, c, e, g, i, k) and with (b, d, f, h, j, l) TESH-6 under UV irradiation within different time of (a, b) 0min, (c, d) 5min, (e, f) 10min, (g, h) 147x308mm (300 x 300 DPI)
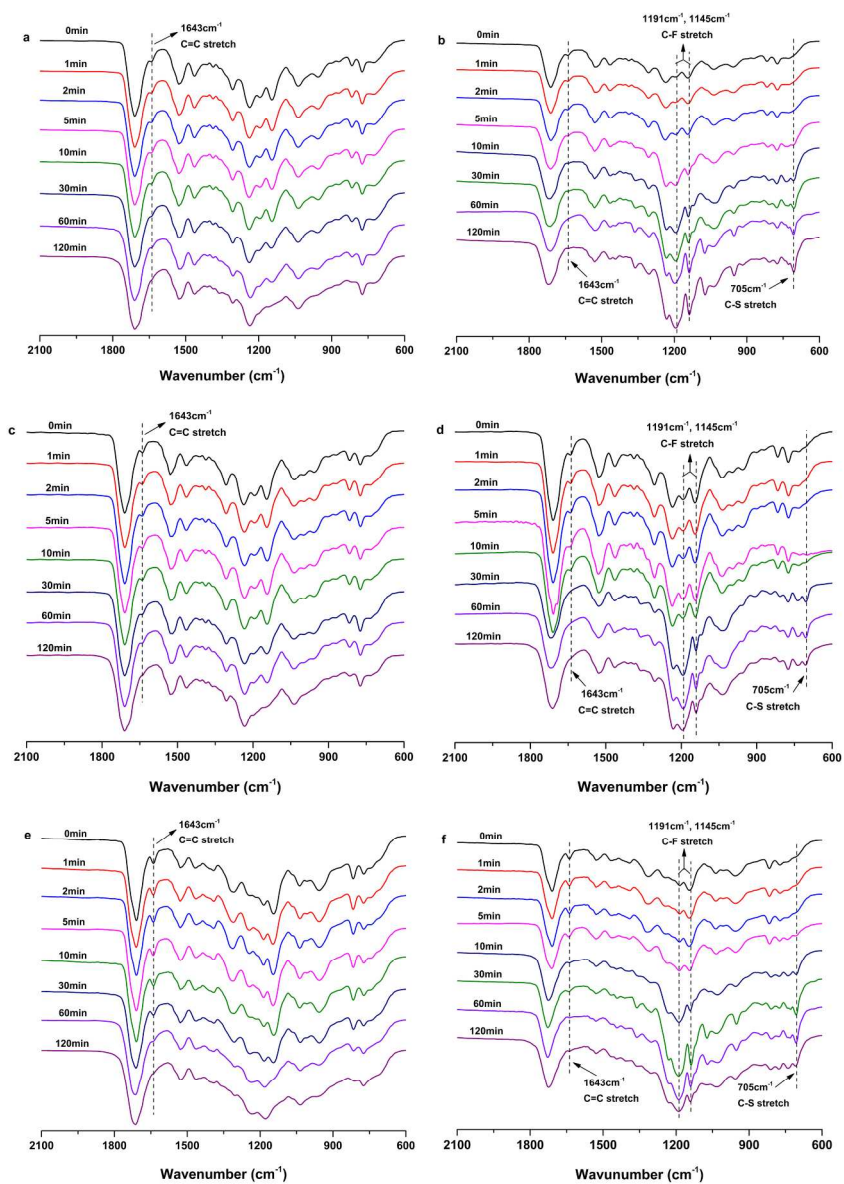
FIGURE 8 FTIR spectra of the electrospun TPU ((a, b) DTPU, (c, d) HTPU and (e, f) BTPU) fibers reacted without (a, c, e) and with (b, d, f) TESH-6 under UV irradiation
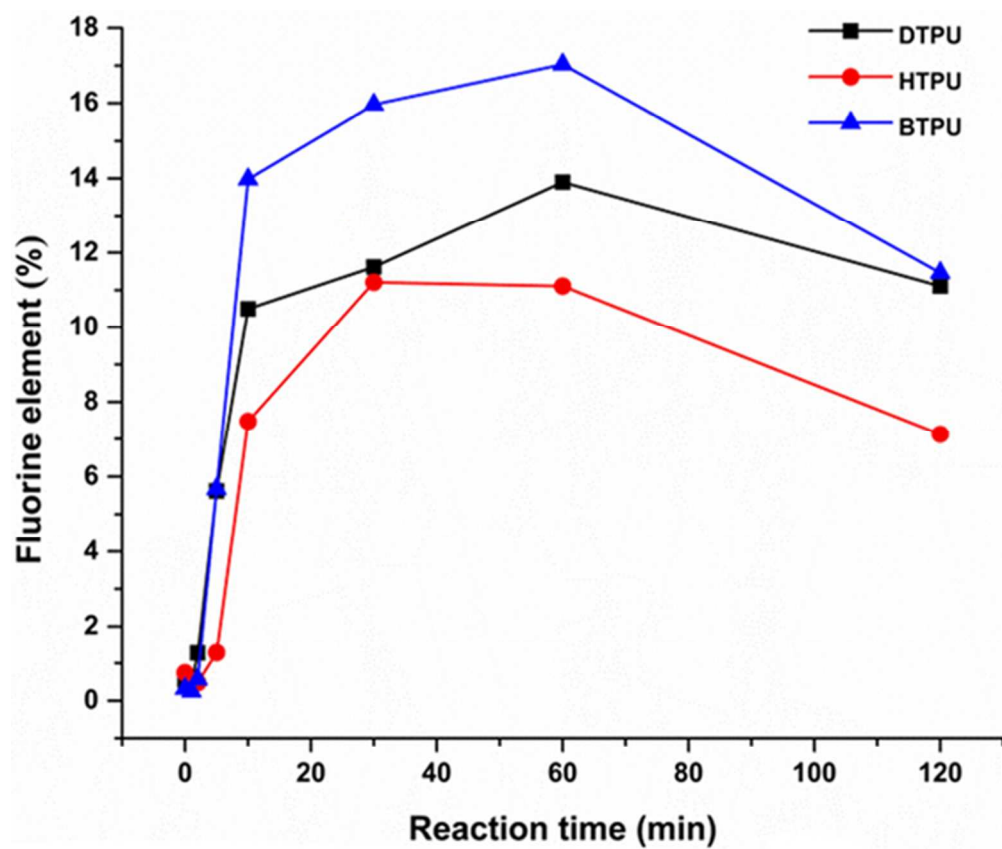134x191mm (600 x 600 DPI)

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FIGURE 9 Amount of fluoride element in electrospun TPU fibers after reaction with TESH-6
50x42mm (300 x 300 DPI)

This work suggests a feasible methodology to produce a functionalized ultrafine fibrous carrier with the capacity to fabricate solid catalyst in the future.