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Selective Adsorption of Neutral Nitrogen Compounds from Fuel Using Ion-Exchange Resins

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Adsorptive removal of hydrodesulfurization inhibitors, that is, nitrogen-containing compounds (N-compounds), was studied using model fuel which contains carbazole and dibenzothiophene in an *n*-dodecane−toluene mixture over ion-exchange resins (IXRs) in batch and in fixed-bed adsorption systems. The adsorptive capacity and selectivity for neutral N-compounds were examined. The high affinity for neutral N-compounds present in hydrocarbon feed was explained by hydrogen bonding between the chloride anion (with nonbonding electrons) and the neutral N-compounds. However, the presence of a flat charged cation, that is, N-alkylated pyridinium, is essential to ensure the adsorption of neutral N-compounds. The regeneration of the sorbent could be easily performed using polar organic solvent like methanol. Commercially available IXR bearing pyridinium chloride functionality (HPQ) was further evaluated with straight-run fuel oil. The N/S selectivity decreases, but analysis of the adsorbed compounds by gas chromatography/mass spectroscopy (GC/MS) shows that no paraffin and no monoaromatics were adsorbed. The results obtained provided new insight into the fundamental understanding of the adsorption mechanism of neutral N-compounds from hydrocarbon feed.

Introduction

The world refining industry has demonstrated its capability of adapting hydrotreatment processes to the new and stricter regulations on the heteroatom contents in their major products such as diesel and gasoline. However, the worldwide petroleum reserves are becoming heavier as a result of the depletion of traditional resources, increasing their sulfur and nitrogen content.[1] Removal of nitrogen-containing compounds (N-compounds) from fuel is essential to prevent negative kinetic effects on hydrotreating reactions such as hydrodesulfurization, on other hydrogenolysis reactions, and on hydrogenation reactions.[2–5]

N-Compounds are present predominantly in heterocyclic aromatic compounds classed as basic (pyridines, quinolines, acridines) and neutral (indoles, carbazoles, and benzocarbazoles). Nitrogen can be removed catalytically by hydrodenitrogenation, which is generally more difficult than desulfurization because N-compounds are much less reactive than sulfur-containing compounds.[6] Thus, researchers worldwide are seeking alternative approaches to achieve deep denitrogenation of liquid hydrocarbon streams. The elimination of N-compounds by noncatalytic processes has already been studied. N-Compounds can be removed using strongly acidic ion-exchange resin (IXR),[7] by liquid−liquid extraction using carboxylic acid,[8] and by adsorption on various supports including zeolite-based materials, supported metals, and activated carbons.[9–12] However, these methods are efficient for the removal of basic N-compounds, but none of them seemed to be efficient or selective enough to remove neutral N-compounds.

We have recently showed that neutral N-compounds could be effectively removed from straight-run fuel oil using ionic liquids (ILs) and the selectivity could be explain by hydrogen bonding between chloride anion and neutral N-compounds.[13] Furthermore, specific interactions of planar neutral N-compounds with planar charged moieties of ILs should also be considered.[13] However, a low solubility of the hydrophilic ILs used was measured in the hydrocarbon phase (lower than 10 $\mathrm{mg \cdot kg^{-1}}$).[13] The solubility of ILs of the feed is not only undesirable because of the potential effects of the ILs on quality of the fuel but also because of the relatively high cost of ILs compared to molecular solvents. Thus, a small solubility can be magnified to insurmountable losses in a chemical plant. Furthermore, there are only a few reports on regeneration and subsequent recycling of ILs, although this was of vital importance for industrial application.[14,15] As a result, loss and regeneration cost of spent ILs restrict the industrial application to some extent. To explore a way to mitigate these problems, we have studied polymer-supported ILs.[16] The best results were obtained when imidazole or pyridine were covalently bound to Merrifield's resin. Immobilization of IL moieties on resins has proved to be a highly suitable method for the extraction of neutral N-compounds from models and straight-run fuel oil. The covalent binding of IL moieties on polystyrene solved the problem of leaching of ILs to the raffinate.[16] However, the process suffers from several drawbacks, including relative high cost of Merrifield's resin, limited capacity as well as solvent, and the time-consuming procedure of immobilization.

IXRs are commercially available in bulk, in the form of small beads. Among them, HPQ is an IXR, bearing a pyridinium

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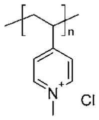
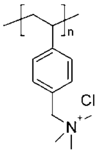
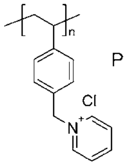
Table 1. Composition and Physical Properties of Arabian Light Straight-Run Fuel Oil

density (at 293 K)	870.3 $g\cdot L^{-1}$
sulfur	11540 $mg\cdot kg^{-1}$
total nitrogen	270 $mg\cdot kg^{-1}$
basic nitrogen	< 5 $mg\cdot kg^{-1}$
aromatics	$w = 0.356$

Table 2. Particle Size and TAEC of the Resins Studied

	Particle size	TAEC
Resin	μm	$meq\cdot g^{-1}$
 HPQ	250-600	4.1 [a]
 A4200Cl	700	1.7 [a]
 PSPyCl	300-1200	3.6 [b]

[a] From ref 17. [b] From ref 16.

chloride functionality, used in hydrometallurgy[17,18] or as support of molecular catalyst used in industrial process.[19] We now report the use of HPQ for the selective adsorption of neutral N-compounds from hydrocarbon feed. The result obtained will be compared to IXRs bearing trimethylamonium chloride functionality (Amberjet 4200 Cl) and polymer-supported ILs.[16] In addition, the regeneration of the adsorbent was studied, and the extracted compounds were characterized to confirm the observed selectivity.

Experimental Section

Materials. Analytical grade toluene and *n*-dodecane with a mass purity of 99 % were used as received. Carbazole (CAS 86-74-8) (purity of 95 % by GC) and dibenzothiophene (CAS 132-65-0) (purity superior to 99 % by GC), both from Aldrich, were used as received. Model fuel containing 150 $mg\cdot kg^{-1}$ of sulfur and 150 $mg\cdot kg^{-1}$ of nitrogen was prepared by dissolving an appropriate amount of dibenzothiophene and carbazole in a mixture of toluene−*n*-dodecane ($w = 0.8$). Arabian light straight-run fuel oil was kindly provided by TOTAL (Feluy-Belgium) and was used as feedstock. The relevant properties of the oil are summarized in Table 1.

HPQ and Amberjet 4200 Cl IXRs (Table 2) were obtained from Vertellus Specialties Inc. and Rohm & Hass, respectively. These anionic resins are supplied in chloride form and contain water and sodium chloride. They need to be suitably washed before use. To this end, a volume V of resin, previously swollen in deionized water, was placed on a column equipped with a sintered filter of porosity 0. The residual impurities were removed by washing with a volume $2V$ of deionized water. The column was then percolated with $5V$ of aqueous 2 N sodium hydroxide, washed with deionized water until neutral pH, washed with $5V$ of aqueous 2 N HCl, and finally washed with deionized water until neutral pH. The total anion exchange capacity (TAEC) of the resins was determined by back-titration of NaOH standard solutions as previously described[17] and is reported as milli-equivalents per gram of the oven-dried resin (Table 2). For adsorption, the resins were subsequently washed with methanol ($2V$), acetone ($2V$), pentane ($2V$), and toluene ($2V$) to obtain dry swollen resins. The polymer-supported IL, PSPyCl, was synthesized as described in previous research.[16] All of the resins were equilibrated in the toluene−*n*-dodecane mixture ($w = 0.8$).

Adsorption. Equilibrium experiments were performed using a batch procedure. A fixed mass of Büchner dried resin (1.5 g) was weighed and introduced into 25 mL glass flasks. Six grams of model fuel containing 150 $mg\cdot kg^{-1}$ of N and 150 $mg\cdot kg^{-1}$ of S or 15 g straight-run fuel oil were added, and the suspension was shaken mechanically with a Vibrax 100 shaker (Biobloc Scientific) at 313 K for 15 min. The distribution coefficient, the selectivity, and capacity were determined according to eqs 1 to 3 after measuring the initial and final S and N concentrations of the feed (C_i and C_f, respectively) by using an Antek 9000 series N/S analyzer, and M_{feed} and M_{ads} are the mass of hydrocarbon solution and resins, respectively.

$$D = \left(\frac{C_i}{C_f} - 1\right)\cdot\frac{M_{feed}}{M_{ads}} \tag{1}$$

$$S_{S/N} = \frac{D_S}{D_N} \tag{2}$$

$$C = \frac{M_{feed}(C_i - C_f)}{M_{ads}} \tag{3}$$

Regeneration. After equilibrium experiments, used HPQ were vacuum-filtered using a Büchner funnel. HPQ was then washed with $4V$ of pentane to remove the adsorb feed. The vacuum was stopped, and HPQ was suspended in the Büchner funnel with $4V$ of methanol. Methanol was removed using vacuum, and HPQ was again suspended in $4V$ of methanol. After filtration under vacuum, the regenerated HPQ thus obtained could be reused after equilibration in the toluene−*n*-dodecane mixture.

The methanol fraction could be concentrated under reduced pressure using a rotary evaporator, and 17 mg of extracted compounds per grams of adsorbent were obtained as viscous brown oil. Extracted compounds were analyzed on a gas chromatograph/mass spectroscopy (GC-MS, Agilent, model 6890 GC with a 5973 N mass selective detector) equipped with a CPSil5CB low bleed fused silica capillary column.

Packed-Bed Breakthrough Experiments. The fixed-bed experiments were performed in a jacketed glass-made cylinder with an inner diameter of 30 mm. The bed volume was approximately 220 mL of buchner dried resin, with a bed length of 300 mm. The column was first rinsed with $2V$ of a mixture of toluene−*n*-dodecane ($w = 0.8$), and the temperature of the organic phase inside the column was maintained at 313 K using a water−glycol thermocirculator HAAK K20. The loading step was carried out down-flow with a constant loading flow of 172 $cm\cdot h^{-1}$. During loading runs, samples were taken at given time intervals for S and N analysis. N and S loading curves were plotted against the number of bed volumes.

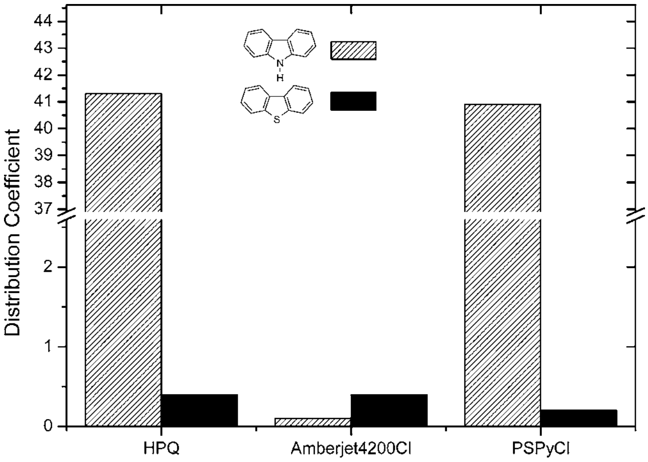


Figure 1. Evaluation of different resins with model fuel which contain 150 $mg\cdot kg^{-1}$ of N and 150 $mg\cdot kg^{-1}$ of S in toluene−*n*-dodecane ($w = 0.8$). Resin/model fuel: $w = 0.25$, 15 min at 313 K.

Table 3. Comparison of the Resins with Model Fuel Which Contain 150 $mg\cdot kg^{-1}$ of N and 150 $mg\cdot kg^{-1}$ of S in Toluene−*n*-Dodecane ($w = 0.8$)[a]

		capacity	
		$mmol\cdot g^{-1}$	
resin	selectivity (N/S)	S	N
HPQ	> 100	< 0.002	0.086
Amberjet4200Cl	0.2	< 0.002	< 0.002
PSPyCl	> 100	< 0.002	0.086

[a] Mass ratio resin/model fuel: 1/4, 15 min at 313 K.

Results and Discussion

Evaluation of IXRs as Adsorbents. We have previously shown that the extraction of heterocyclic aromatic compounds from a hydrocarbon mixture by ILs was strongly dependent on the composition of the feed and in particular on the aromatic content.[13,15] Lower extractions have been observed when toluene was used as a solvent instead of *n*-alkane. The diesel feed contained from 0.25 to 0.35 mass fraction aromatics, so we decided to evaluate the IXRs with a model fuel containing a high content of aromatics. Furthermore, carbazole was sparingly soluble in *n*-alkanes. Thus, model fuel was prepared by dissolving carbazole and dibenzothiophene in a toluene−*n*-dodecane mixture ($w = 0.8$). Solid−liquid equilibrium data were obtained at 313 K with a ratio resin/model fuel of 1/4. The results are presented in Figure 1 and were compared to the results obtained by supported pyridinium chloride-based ILs (PSPyCl).[16]

HPQ showed a high distribution coefficient for carbazole with a high carbazole/dibenzothiophene selectivity ($S_{carb/DBT} > 100$) (Figure 1). The results obtained could be compared to the results obtained by supported ILs (PSPyCl) since particle size and TAEC are comparable (Table 2). Under those conditions, no differences could be noticed between commercially available HPQ and supported pyridinium chloride-based ILs synthesized from Merrifield's resin[16] (Table 3).

Under those conditions, the capacity for carbazole is low (less than 0.09 $mmol\cdot g^{-1}$), but the selectivity toward dibenzothiophene is high (up to 100). As expected from previous results,[13] the adsorption of neutral N-compounds could be explained by hydrogen bonding between the chloride anion (with nonbonding electrons) with proton donor molecules (i.e., carbazole). However, a very low adsorption of carbazole as well as a low selectivity was observed with Amberjet4200Cl bearing a trimethylammonium chloride functionality (Figure 1). Thus, specific interaction between planar, electron-rich neutral N-compounds, and planar π-acceptor molecules (i.e., N-alkylated pyridinium) could explain the selective adsorption.[5] It demonstrates that both interactions might play an important role in the adsorption of neutral N-compounds[20] and could explain the extraction properties of ILs.[13,15]

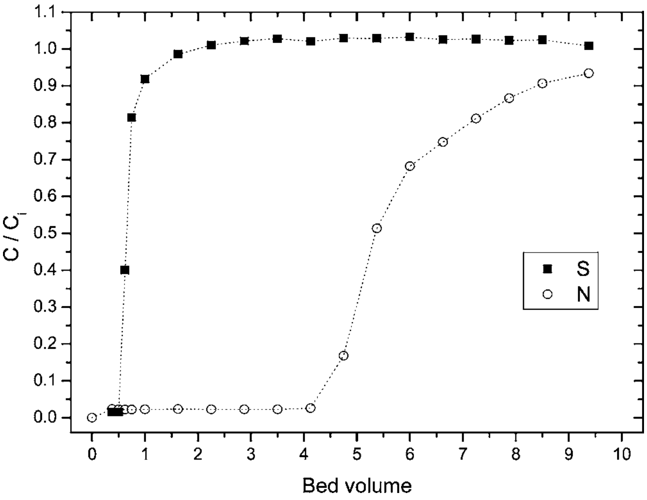


Figure 2. Breakthrough curves for dibenzothiophene and carbazole. Composition of synthetic solution, 1000 $mg\cdot kg^{-1}$ of S, 150 $mg\cdot kg^{-1}$ of N; toluene−heptane: $w = 0.8$; HPQ: 220 mL; $u = 172\ cm\cdot h^{-1}$ at 313 K.

Regeneration of the Adsorbent. After adsorption, HPQ was recovered by filtration. The adsorbent could be easily regenerated using polar solvents like methanol and then re-equilibrated with toluene−*n*-dodecane solution. Adsorption experiments were repeated, and no difference could be noticed. Furthermore, no adsorbed compounds could be detected by Fourier transform infrared (FT-IR) and elemental analysis of the regenerated HPQ resin.

Packed-Bed Breakthrough Experiments. The HPQ was further evaluated in column flow-through experiments using model fuel containing a large excess of dibenzothiophene compared to carbazole, that is, 1000 $mg\cdot kg^{-1}$ of S and 150 $mg\cdot kg^{-1}$ of N. Figure 2 gathers the experimental dibenzothiophene and carbazole breakthrough curves over HPQ at 313 K at a flow rate of 172 $cm\cdot h^{-1}$. Breakthrough data was acquired by plotting the C/C_0 value (ratio of the outlet concentration of dibenzothiophene or carbazole to the initial concentration of dibenzothiophene or carbazole) against the number of bed volumes of the feed that passed through the column (Figure 2).

The experimental results show a formation of steep breakthrough curves for dibenzothiophene, and the C/C_0 value for dibenzothiophene reaches 1.0 after 2.5 bed volumes. As expected from the high N/S selectivity obtained from equilibrium studies, the carbazole breakthrough occurred after 4.5 bed volumes, and the C/C_0 value increased gradually, showing that carbazoles are selectively adsorbed by HPQ. The amount of model fuel corresponding to the saturation point was superior to 10 bed volumes.

Evaluation of HPQ as Adsorbents with Straight-Run Fuel Oil. Figure 3 shows the concentration of S-compounds and N-compounds in straight-run fuel oil during a multistage liquid−solid equilibrium batch procedure using HPQ (Figure 3). The initial concentration of S-compounds and N-compounds was 11540 $mg\cdot kg^{-1}$ and 270 $mg\cdot kg^{-1}$, respectively.

After 15 min of contact with HPQ with a mass ratio of HPQ/straight-run fuel oil of 0.1, 112 $mg\cdot kg^{-1}$ (41 %) of nitrogen

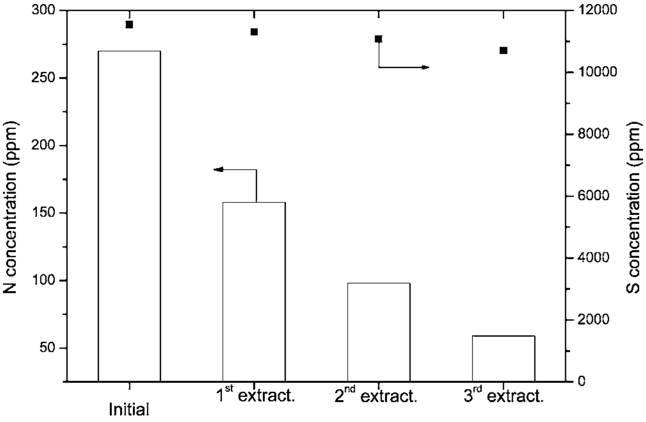


Figure 3. Multistage extraction of straight-run fuel oil by HPQ. Mass ratio HPQ/straight-run fuel oil: $w = 0.1$, 15 min at 313 K.

Table 4. Elemental Analysis of Compounds Extracted by HPQ

	total N content	total S content	
	$mg \cdot kg^{-1}$	$mg \cdot kg^{-1}$	ratio S/N
straight-run fuel oil	270	11540	42.7
extracted compounds	16200	57800	3.6

are removed, although the sulfur concentration was almost constant (decrease of less than 2 %). With real hydrocarbon feed, a nitrogen/sulfur selectivity higher than 32 was obtained (Table 5) and could be compared to the carbazole/dibenzothiophene selectivity higher than 100 obtained with synthetic solution (Table 3). The decrease of selectivity was already explained by the presence of other aromatic compounds in the real feed that can act as competitive binders.[15]

After extraction of straight-run fuel oil, HPQ was regenerated by filtration using a Büchner funnel and washed with pentane to remove the adsorbed feed and then with methanol. The methanol fraction was concentrated under reduced pressure, and 17 mg of extracted compounds per g of adsorbent were obtained as viscous brown oil. Under those conditions, the mass of the extracted correspond represents less than 0.2 % wt of the feed. The elemental analysis of the extracted compounds is given Table 4. The N-content was raised by a factor of 60, although the S-content was raised by a factor 5.

The selectivity of the extraction process could be illustrated by the GC/MS analysis of the extracted compounds (Figure 4). In straight-run fuel oil, trace polar compounds like N- and S-compounds could not be directly analyzed by GC/MS because of the dilution and the lack of resolution due to crowded GC spectra. However, our process selectively extracted and concentrated trace polar compounds from paraffin present in straight-run fuel oil, and thus GC/MS analysis could be used.

The chromatogram emphasized the selectivity of the HPQ for trace polar compounds and polyaromatics versus paraffin. Alkylated carbazole and dibenzothiophene could be identified by MS as well as alkylated triaromatics. We were pleased to see that no paraffin and no monoaromatics could be identified in the adsorbed compounds, indicating that the adsorption process was very selective.

Treated straight-run fuel oil containing 11 300 $mg \cdot kg^{-1}$ of S and 158 $mg \cdot kg^{-1}$ of N was contacted with regenerated HPQ under the same conditions, and 60 $mg \cdot kg^{-1}$ of nitrogen (38 %) are removed (Figure 3). The process was carried out a third time. Under those conditions, the global yield of the three-step process was promising: more than 98 wt % of the feed is recovered after extraction, and 78 % of N-compounds are removed, whereas less than 7 % of S-compounds are adsorbed (Table 5). It should also be noticed that refractory S-compounds (i.e., alkylated dibenzothiophene) have been identified in the extract (Figure 4).

Conclusions

The adsorption performance of IXR bearing a chloride anion associated with an *N*-methyl pyridinium functionality for the selective removal of neutral nitrogen compounds was evaluated in a batch adsorption system and in a flowing fixed-bed adsorption system. Commercially available HPQ was proven

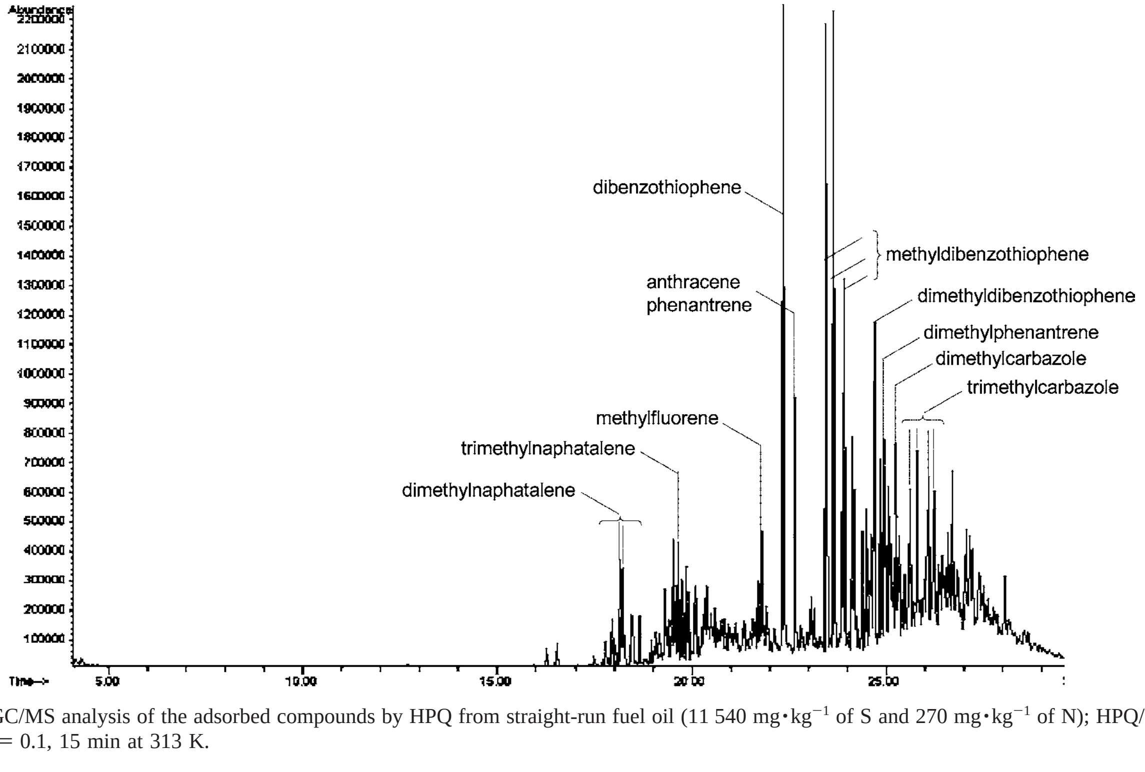


Figure 4. GC/MS analysis of the adsorbed compounds by HPQ from straight-run fuel oil (11 540 $mg \cdot kg^{-1}$ of S and 270 $mg \cdot kg^{-1}$ of N); HPQ/straight-run fuel oil: $w = 0.1$, 15 min at 313 K.

Table 5. Influence of the Composition of Straight-Run Fuel Oil during Multistage Extraction by HPQ[a]

composition of straight-run fuel oil		distribution coefficient		selectivity	capacity	
S	N				*S*	N
$\mathrm{mg \cdot kg^{-1}}$	$\mathrm{mg \cdot kg^{-1}}$	S	N	N/S	$\mathrm{mmol \cdot g^{-1}}$	$\mathrm{mmol \cdot g^{-1}}$
11540	270	0.21	7.09	33.8	0.075	0.080
11300	158	0.20	6.12	30.6	0.068	0.043
11080	98	0.23	6.60	28.7	0.078	0.028

[a] Mass ratio HPQ/straight-run fuel oil: $w = 0.1$, 15 min at 313 K.

to be an effective adsorbent for neutral N-compounds with a high selectivity versus paraffin, mono- and diaromatic hydrocarbons, and sulfur compounds. The adsorption capacity and selectivity for nitrogen compounds was correlated with the presence of a planar charged heterocyclic moiety, that is, pyridinium chloride. This selectivity was illustrated not only by packed-bed breakthrough experiments with model fuel but also by multistage batch adsorption experiments using straight-run fuel oil containing 270 $\mathrm{mg \cdot kg^{-1}}$ of nitrogen. In three steps, 78 % of nitrogen was removed, whereas less than 7 % of sulfur was removed. In addition, a method for regeneration of the saturated adsorbent was proposed and evaluated using methanol. The selectivity of the adsorption process was emphasized by the GC/MS analysis of the extracted compounds. Complementary experiments are necessary to find appropriate conditions (sorbent bed height, hydrodynamic conditions, and temperature) to efficiently remove N-compounds in the fixed-bed flow system from straight-run fuel oil.

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