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Adsorption behavior and XPS analysis of nonylphenol ethoxylate on low rank coal

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Abstract: In this work, low rank coal was used for the removal of nonylphenol ethoxylate with fifteen ethylene oxide groups (NPEO₁₅) from aqueous solutions at different contact times, temperatures, and initial adsorbent concentrations. The adsorption isotherms showed good fit with the Langmuir equation. Maximum adsorption capacities calculated at 308, 318, and 328 K were 23.64, 29.41, and 35.71 mg g⁻¹, respectively. The changes in the free energy of adsorption (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) were calculated in order to predict the nature of adsorption. The results of the thermodynamic analysis indicated that a spontaneous process took place, driven synergistically by both enthalpy and entropy. The adsorption kinetics of NPEO₁₅ were consistent with a pseudo-second order reaction model. XPS results showed that the oxygen functional groups on the low rank coal surface were significantly covered by NPEO₁₅. Furthermore, while the content of C-C/C-H functional groups increased significantly, that of C-O functional groups decreased after absorption. These results clearly indicate that low rank coal is more hydrophobic and displays better floatability.

Keywords: Low rank coal, nonylphenol ethoxylate, adsorption isotherm, adsorption kinetics, XPS

1. Introduction

Sub-bituminous coal is the classic low rank coal. Fine low rank coals are difficult to float with normal oily collectors such as kerosene, diesel oil, and fuel oil (Jia et al., 2000; Dey, 2012; Xia et al., 2013; Zhou et al., 2015; Kim et al., 2015; Zhou et al., 2016; You et al., 2017). Their poor floatability is mainly attributed to the abundance of hydrophilic and polar surface functional groups such as phenol (-OH), carbonyl (C=O), and carboxyl (-COOH) groups (Somasundaran et al., 2000; Li et al., 2013; Lyu et al., 2018). These groups enhance the surface hydration of coal by increasing the number of sites which can form hydrogen bond with the water molecules. Therefore, the low rank coal is difficult to float with common collectors due to these large amounts of oxygen functional groups on its surface (Boylu and Laskowski, 2007; Cinar, 2009; Temel et al., 2010; Tian et al., 2016a; Tian et al., 2016b). The effect of the surfactants for flotation of low rank coal has been also studied by many researches (Chander et al., 1994; Celik et al., 1995; Harris et al., 1995; Jia et al., 2000; Vamvuka et al., 2001). The performance of nonionic surfactant is found to be more effective than the cationic/anionic surfactant (Dey, 2012). The nonionic surfactants possess effective hydrogen bonding at one end of the alkyl chain which confers on them the required water solubility. The interaction of the oily collector in the presence of a cationic surfactant that has a polar nature with the coal surface may be due to hydrogen bonding and the electrostatic attraction with the negatively charged coal surface. This type of adhesion will result in a tendency for a residual water film to be retained between the coal surface and oil. Therefore, the coal surface would be to a lesser extent, covered with the oil as obtained from nonionic surfactants. Similarly, the presence of anionic collector with oily collector was due to the hydrogen bonding as a result of their polar nature. However,

the electrostatic repulsion between the negatively charged oil droplets and coal surface exists. Therefore, the surface of coal would be, to a lesser extent, covered with kerosene. It is also possible that the residual water film remained between the coal surface and kerosene droplets. As a result of the combining of these effects, the yields were lower than those obtained from nonionic surfactants (Cebeci, 2002).

Polyethoxylated nonyl phenols constitute a major part of the nonionic surfactants (Furlong and Aston, 1982), which can be used during floatation as frother, collector, emulsifier, and surface modifier. It was found that these compounds were likely to improve the floatation of low rank/oxidized coal. Harris et al (Harris et al., 1995) investigated the collecting ability of polyethoxylated nonyl phenols with 0–6 ethoxy groups in the floatation of unoxidized, laboratory-oxidized, and naturally weathered coals. It was found that the surfactants were more effective collectors than the oily collector dodecane for both unoxidized and oxidized coals. Although the benefits of a series of nonylphenol ethoxylates for low rank/oxidized coal floatation have been proven before, most of studies have been focused on the macroscale content, such as contact angle and separation experiment. Few researchers have analysed the mechanism of interaction between nonionic surfactants and coal surface from microscopic point of view.

In this paper, a detailed study was conducted for the adsorption of the nonionic surfactant nonylphenol ethoxylate (NPEO₁₅) on low rank coal. The thermodynamic functions such as Gibbs free energy, enthalpy, and entropy were calculated to understand the adsorption mode of the surfactant. XPS analysis and corresponding peak analysis were performed to investigate the distribution characteristics of NPEO₁₅ on the sub-bituminous coal, especially the various oxygen-containing functional groups. The results obtained from this study can be used to explain the competitive adsorption of NPEO₁₅ existed among the oxygen-containing groups. The findings of this study demonstrate the interaction mechanism between nonylphenol ethoxylate and low rank coal, which can be used to improve the floatability of low rank coal effectively.

2. Materials and methods

2.1. Materials

Low-rank coal obtained from Shenhua Shendong Coal Group Corporation Limited in China was selected for this study. The proximate analysis of coal samples presented in Table 1 reveals that the sample is a typical low rank coal. M_{ad} , V_{ad} , FC_{ad} , and A_{ad} refer to the moisture content, volatile content, fixed carbon content, and ash content on a dry basis, respectively. The sample is a typical sub-bituminous coal.

Table 1. Proximate analysis of the coal sample

Index	Proximate analysis / %			
	M_{ad}	A_{ad}	V_{ad}	FC_{ad}
Result	5.29	21.60	37.90	62.10

The nonylphenol ethoxylate with fifteen ethylene oxide groups (NPEO₁₅) was obtained from Union Carbide Chemicals, and used without further purification. The chemical structure of NPEO₁₅ used in this investigation is shown in Fig. 1.

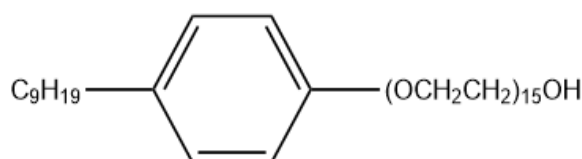


Fig. 1. Structure of NPEO₁₅

2.2. Surfactant adsorption

The coal sample was first crushed using a hammer, and then ground in an agate mill. The ground material was sieved to obtain particles with size fraction of -0.045 mm for adsorption experiments. The maximum adsorption density for NPEO₁₅ on the sample under investigation was measured. The adsorption experiments were carried out in scintillation vials. Each vial containing 25 cm³ of the surfactant solution of a known concentration and 0.5 g of the coal sample was agitated for 24 h in a constant temperature water bath oscillator, whereas the tests with initial concentration of 1.00 g/dm³ were effectuated during 5, 10, 20, 30, 60, 90, 120, 240, 480, 720, 960, 1200, and 1440 min. The adsorption experiments were carried out at 308, 318, and 328 K, respectively. After the adsorption process, the vials were centrifuged at 4000 rpm for 20 min to obtain clear supernatant solutions. The surfactant concentrations in the supernatants were then determined by using a spectrophotometer (Model UV757CRT) at the wavelength of 275 nm.

2.3. X-ray photoelectron spectroscopy measurements

The X-ray photoelectron spectroscopy (XPS) experiments were carried out at room temperature in an ultra-high vacuum (UHV) system using the surface analysis system (ESCALAB250 Xi, America). The survey scan and high-resolution spectra were recorded at pass energy of 100 and 20 eV with step sizes of 1.00 and 0.05 eV, respectively. Before the data analysis, the binding energies were corrected by setting the C1s hydrocarbon peak at 284.8 eV. Data processing (peak fitting) was performed with an XPS peak fit software, using a Smart type background subtraction and Gaussian/Lorentzian peak shapes.

3. Results and discussion

3.1. Adsorption isotherms

The adsorption isotherms of the nonionic surfactant on the coal are illustrated in Fig. 2 at 308, 318, and 328 K for NPEO₁₅.

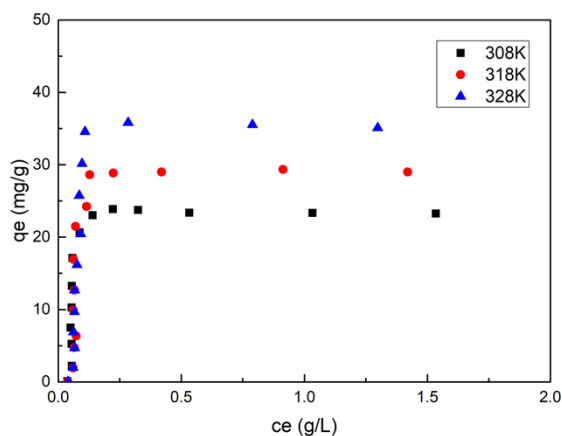


Fig. 2. Adsorption isotherms of NPEO₁₅ over coal sample at 308, 318, and 328 K

These isotherms show that the adsorption of NPEO₁₅ is temperature sensitive (McCracken and Datyner, 1977; Klimenko, 1980). The linearized Langmuir equation (1) and linearized Freundlich equation (2) can be represented as follows (Silva et al., 2004; Vadivelan and Kumar, 2005; Liu et al., 2013; Cheng et al., 2014; Wang et al., 2014; You et al., 2019):

$$C_e/q_e = 1/bQ_m + C_e/Q_m \quad (1)$$

$$\lg q_e = \lg k + (1/n) \lg c_e \quad (2)$$

where b is the Langmuir constant, related to the affinity of the adsorption sites, and Q_m (mg/g) represents the maximum amount of NPEO₁₅ that can be adsorbed per unit weight of the adsorbent. Q_m and b were calculated from the slope and intercept of the straight lines of the plot C_e/q_e vs. C_e .

Adsorption isotherms were obtained in terms of Eq. (1) and Eq. (2) by using results from adsorption experiments. The different values for Q_m , b and n are summarized in Table 2. The corresponding linear regression correlation coefficients (R^2 values) are given in the same table. As summarized in Table 2, the R_1^2 values of the Langmuir equation are 0.993, 0.996, and 0.993 at 308, 318, and 328K respectively all higher than the R_2^2 values of the Freundlich equation, suggesting that the adsorption of NPEO₁₅ onto low rank coal closely follows the Langmuir model. The maximum adsorption capacities for NPEO₁₅ onto the coal sample at 308, 318, and 328 K were found to be 23.64, 29.41, and 35.71 mg g⁻¹, respectively. Thus, the maximum adsorption capacities of the low rank coal increased with increasing temperature.

Table 2. Langmuir and Freundlich isotherm constants for NPEO₁₅ onto coal sample coal at different temperatures

T, k	Langmuir				Freundlich	
	Q_m , mg g ⁻¹	b , L mg ⁻¹	b , L mol ⁻¹	R_1^2	n	R_2^2
308	23.64	105.75	9.31×10^7	0.9993	2.26	0.7404
318	29.41	85.00	7.48×10^7	0.9996	1.92	0.7898
328	35.71	70.00	6.16×10^7	0.9993	1.70	0.8263

The adsorption free energy (ΔG°) can be calculated using the following equation (Crini et al., 2007):

$$\Delta G^\circ = -RT \ln b \quad (3)$$

where R is the ideal gas constant and T is the absolute temperature. The adsorption free energy (ΔG°) calculated on a molar basis for NPEO₁₅ used at 308, 318, and 328 K was determined to be -46.99 kJ mol⁻¹, -47.93 kJ mol⁻¹, and -48.91 kJ mol⁻¹, respectively. The negative values of ΔG° are as expected for a spontaneous process under the experimental conditions. A decrease in the ΔG° values with increasing temperature indicates more efficient adsorption at higher temperatures.

The change in enthalpy of the adsorption process (ΔH°) for the interaction of NPEO₁₅ with the low rank coal surface can be calculated by using the following form of the Clausius-Clapeyron equation (Fan et al., 2011):

$$\frac{d \ln b}{d(1/T)} = -\frac{\Delta H}{R} \quad (4)$$

Thus, a plot of $\ln b$ vs $1/T$ should be a straight line (Fig. 3). The slope of the plot can be used to calculate ΔH° as shown in Table 3. The entropy change (ΔS°) of the adsorption can be calculated using Eq. (5), which is also given in Table 3.

$$\Delta H^\circ = \Delta G^\circ + T\Delta S^\circ \quad (5)$$

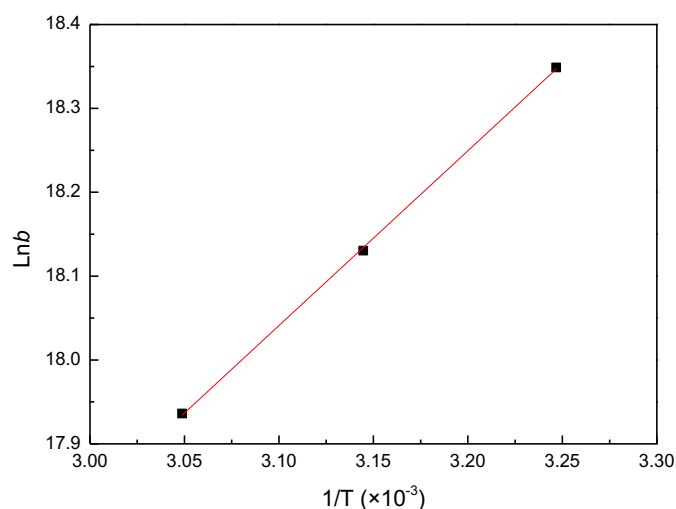


Fig. 3. Plot of $\ln b$ vs $1/T$ for calculating the enthalpy change of the adsorption

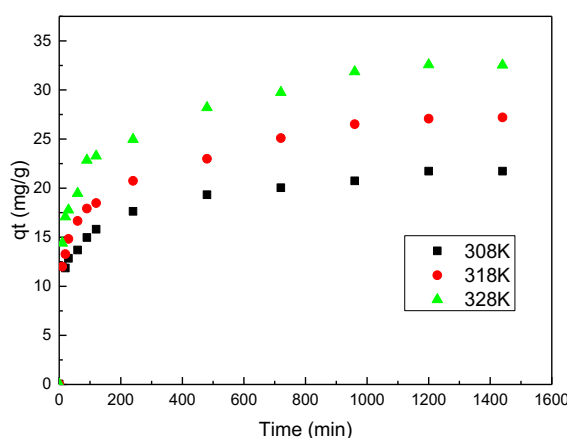
Table 3. Thermodynamic functions for the adsorption of NPEO₁₅ onto coal

T, K	ΔG° , kJ mol ⁻¹	ΔH° , kJ mol ⁻¹	$-\Delta S^\circ$, kJ mol ⁻¹
308	-46.99		-29.6559
318	-47.93	-17.33	-30.6039
328	-48.91		-31.5818

Thus, ΔH° of the adsorption process was evaluated to be -17.33 kJ mol⁻¹. The negative value of ΔH° indicates that the adsorption process is exothermic. As ΔS° is slightly higher than $|\Delta H^\circ|$, it may be concluded that the adsorption of NPEO₁₅ is driven by both enthalpy and entropy in a synergistic manner (Celik, 1989; Celik and Yoon, 1991).

3.2. Adsorption kinetics

The applicability of the pseudo-first and pseudo-second order models was tested for the adsorption of NPEO₁₅ onto the low rank coal. The time-concentration profiles of NPEO₁₅ over coal (with initial concentration of 1.00 g/dm³) at different temperatures are presented in Fig. 4. As seen from Fig. 4, the adsorption of NPEO₁₅ at different temperatures increases with the adsorption time and eventually reaches an equilibrium. Pseudo-first and pseudo-second order kinetic equations are the most widely used models for describing the adsorption of an adsorbate from an aqueous solution. Accordingly, in this work, these two kinetic models were used to predict the mechanism of adsorption.

Fig. 4. Time-concentration profiles of NPEO₁₅ over low rank coal at 308, 318, and 328 K

A linear form of the pseudo-first order model has been described by as follows (Li et al., 2016; Cao et al., 2016; He et al., 2018; Hua et al., 2018; You et al., 2018):

$$\ln(q_e - q_t) = \ln q_e - k_{pf} t / 2.303 \quad (6)$$

where q_t is the amount adsorbed at time t , and k_{pf} is the equilibrium rate constant of pseudo-first order adsorption. The value of q_t can be calculated as follows:

$$q_t = q_e \left(1 - e^{-k_{pf} t}\right) \quad (7)$$

The plots of q_t versus t based on pseudo-first order kinetics are compiled in Fig. 5. The calculated q_e , k_{pf} , and the corresponding linear regression correlation coefficient R_1^2 values are shown in Table 5. The correlation coefficients at different temperatures of 308, 318, and 328 K were only 0.7734, 0.7898, and 0.7980, respectively. These values indicate that the pseudo-first order model cannot accurately predict the kinetics of NPEO₁₅ adsorption onto the coal.

On the other hand, a pseudo-second order equation based on adsorption equilibrium capacity may be expressed as follows:

$$t/q_t = 1/k_{ps} q_e^2 + t/q_e \quad (8)$$

where k_{ps} is the equilibrium rate constant of the pseudo-second order adsorption process. The plots of t/q_t versus t based on pseudo-second order kinetics are compiled in Fig. 6, and the calculated values of q_e , k_{ps} , and the corresponding linear regression correlation coefficients R_2^2 are summarized in Table 4. The correlation coefficients (R_2^2) of the pseudo-second order kinetics were higher than 0.99, which suggests that the adsorption of NPEO₁₅ on coal slime is best described by the pseudo-second order kinetic equation.

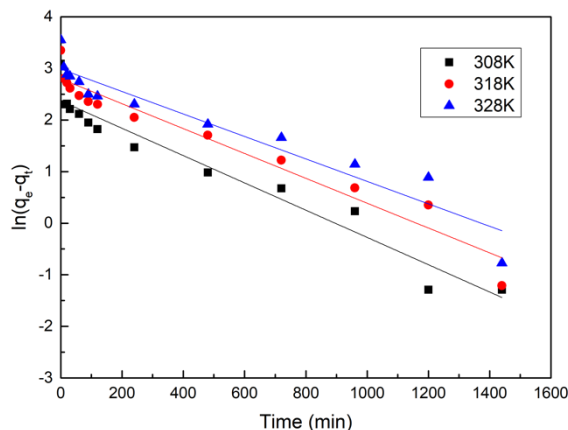


Fig. 5. Simulation of NPEO₁₅ adsorbing onto sub-bituminous coal by using pseudo-first order kinetics at 308, 318, and 328 K

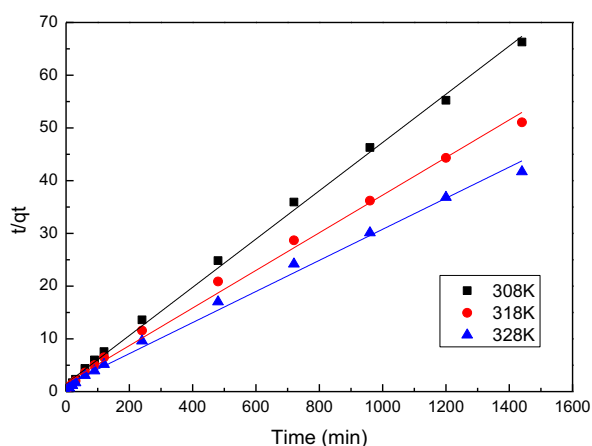


Fig. 6. Simulation of NPEO₁₅ adsorbing onto sub-bituminous coal by using pseudo-second order kinetics at 308, 318, and 328 K

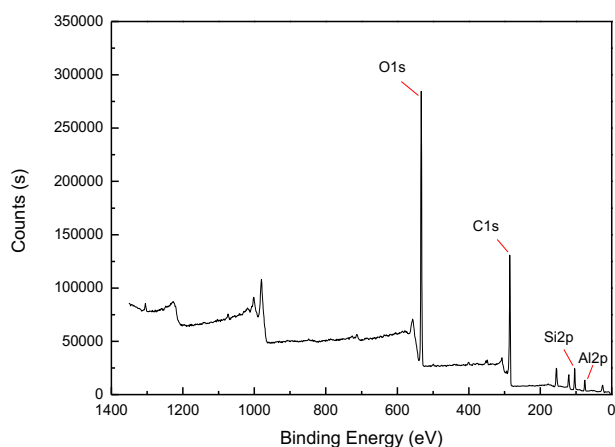
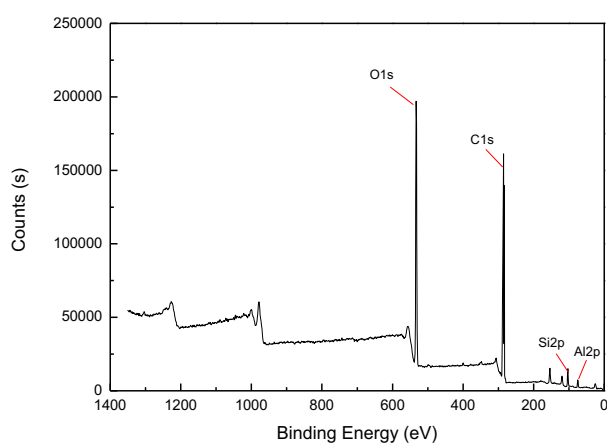
Table 4. Adsorption kinetic parameters for the adsorption of NPEO₁₅ onto sub-bituminous coal

T, K	q_e^a , mg g ⁻¹	Pseudo-first order kinetics			Pseudo-second order kinetics		
		k_{pf} , min ⁻¹	q_e^b , mg g ⁻¹	R_1^2	k_{ps} , g mg ⁻¹ min ⁻¹	q_e^b , mg g ⁻¹	R_2^2
308	21.73	0.05184	18.58	0.7734	0.00141	21.86	0.9977
318	27.20	0.03213	23.60	0.7898	0.00080	28.03	0.9951
328	33.51	0.03367	28.58	0.7980	0.00067	33.92	0.9940

^aExperimental; ^bCalculated.

3.3. XPS measurements

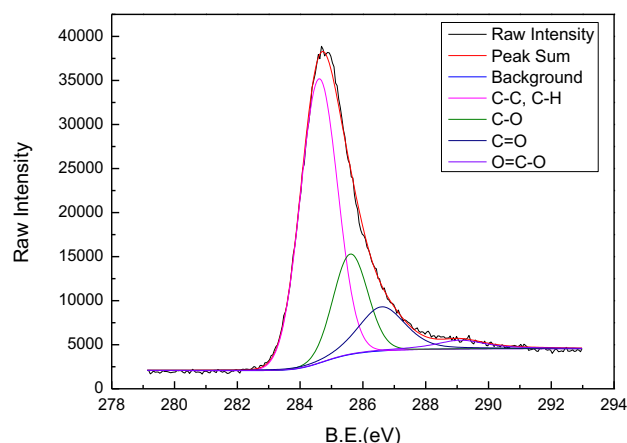
Wide scan spectrum in the binding energy range of 0–1400 eV was obtained to identify the surface elements present in the coal sample and perform quantitative analysis. A typical XPS wide scan spectrum of demineralized lignite coal is seen in Fig. 7.

(a) Before adsorption of NPEO₁₅(b) After adsorption of NPEO₁₅Fig. 7. XPS wide energy spectrums of coal surface before and after adsorption of NPEO₁₅Table 5. Contents of C1s, O1s, Si2p, and Al2p on coal surface before and after adsorption of NPEO₁₅

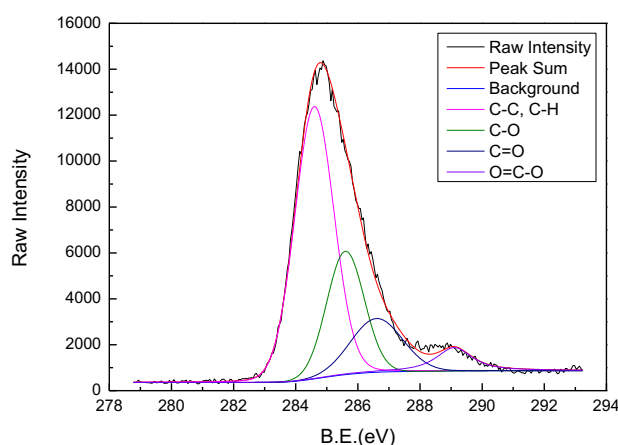
Types	Before adsorption	After adsorption
	Contents/%	Contents/%
O1s	39.64	26.61
C1s	45.47	67.14
Si2p	8.60	3.51
Al2p	6.29	2.74
O/C	0.87	0.40

As seen in Fig. 7, the coal surface peaks of O1s, C1s, Si2p, and Al2p changed after the adsorption of NPEO₁₅. The peak of C1s is greatly enhanced after adsorption of NPEO₁₅. While the peak corresponding to O1s is significantly weakened, those of Si2p and Al2p are slightly weakened. It was observed in Table 5 that the contents of O1s, C1s, Si2p, and Al2p were 39.64, 45.47, 8.60, and 6.29% on coal surface before adsorption and 26.61, 67.14, 3.51, and 2.74% after adsorption in terms of atomic percentage. The content of C1s increased from 45.47% to 67.14% after adsorption, and the content of O1s decreased from 39.64% to 26.61%. O/C ratio decreases from 0.87 to 0.40, which mainly results from polar interactions between the ethoxylated group of NPEO₁₅ and the hydrophilic sites (oxygen functional groups) at the surface of coal. Change in carbon and oxygen content indicate that the oxygen functional groups on the low rank coal surface are significantly covered by NPEO₁₅.

A typical curve fitting of the C1s peak region at 284.80 eV of the coal surface before and after adsorption of NPEO₁₅ is shown in Fig. 8.



(a) Before the adsorption of NPEO₁₅



(b) After the adsorption of NPEO₁₅

Fig. 8. C1s peaks for coal surface before and after adsorption of NPEO₁₅

Fig. 8 shows the curve fitting results of C1s of coal sample. It can be seen that the fitting curve is close to the original curve. Furthermore, XPS measurements revealed that C-C/C-H, C-O (alcohol, phenol, or ether), C=O (carbonyl or chinone), and O=C-O (carboxyl) are the four forms of carbon present on the surface of the samples, and their binding energies are 284.60, 285.60, 286.60, and 289.10 eV, respectively (Pietrzak, 2009; Liu et al., 2017). The content of the C-C/C-H, C-O, C=O, and O=C-O groups was calculated by the analysis of C1s peak (Table 6). C-C/C-H is the main form of the carbon element present on the surface of the samples.

Table 6. The fraction of C on coal surface before and after adsorption of NPEO₁₅ (relative % of C1s)

Groups	Contents /%	
	Before adsorption	After adsorption
C-C/C-H	56.16	61.97
C-O	23.65	20.69
C=O	14.22	13.83
O=C-O	5.96	3.52

As seen in Table 6, the content of C-C/C-H groups which is hydrophobic functional groups is 56.16% in the original coal sample, while the total content of hydrophilic functional groups (C-O, C=O,

and O=C-O groups) is 43.84%. Compared with the original coal sample, the content of C-C/C-H functional groups increased and that of the C-O functional groups decreased significantly after the adsorption of NPEO₁₅, which made the coal surface much more hydrophobic and improved the floatability of low rank coal.

4. Conclusions

In this study, a low rank coal was used for the removal of the typical endocrine-disrupting chemicals such as NPEO₁₅ from aqueous solutions. The adsorption of NPEO₁₅ on jet coal, a non-caking coal, follows the Langmuir-type isotherm. The maximum adsorption capacity at different temperatures of 308, 318, and 328 K was determined to be 23.64, 29.41, and 35.71 mg g⁻¹, respectively. The thermodynamic functions determined from the adsorption isotherms show that the entire adsorption process is spontaneous, being driven synergistically by enthalpy and entropy. The adsorption kinetics of NPEO₁₅ could be described by the pseudo-second order reaction model. XPS results showed that the oxygen functional groups on the low rank coal surface were significantly covered by NPEO₁₅. After adsorption, while the content of the C-C/C-H functional groups increased greatly, that of C-O functional groups decreased significantly. These results indicate better hydrophobicity and floatability for the low rank coal.

Acknowledgments

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