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Research Article

Solvent-Assisted Thermal Extraction and Molecular Characterization of Natural Bitumen from Beke Oil Sands

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Abstract: Oil sands are a potential source of bitumen, and efficient recovery methods are essential for their utilization. In this study, bitumen was recovered from Beke oil sands in Kazakhstan using solvent-assisted thermal extraction at 40–50 °C, followed by filtration and atmospheric distillation. The residual bitumen content was 1.576 wt%, corresponding to a calculated bitumen removal efficiency of 86.9%. High-resolution negative-ion electrospray ionization Fourier-transform ion cyclotron resonance mass spectrometry (ESI FT-ICR MS) showed that the detected ions were mainly distributed over an m/z range of 200–700. The O_2 class was the most abundant heteroatom class, followed by O_3, O_4, and N_1. The O_2 compounds were mainly distributed over carbon numbers of 16–35 and double-bond equivalent (DBE) values of 1–4, whereas the N_1 compounds were mainly distributed over carbon numbers of 20–40 and DBE values of 10–18. These findings indicate effective bitumen removal under the investigated conditions and provide molecular-level information on the composition of Beke oil-sand bitumen

Keywords: *oil sands; bitumen extraction; solvent-assisted thermal extraction; ESI FT-ICR MS; heteroatom classes; DBE–carbon number distribution.*

1 Introduction

Oil sands, also known as bituminous sands or tar sands, are naturally occurring deposits composed mainly of bitumen, water, and mineral solids, including sand, silt, and clay [1], [2]. Oil sands constitute a major unconventional petroleum resource and occur in several regions worldwide, with particularly large deposits in Canada. Commercial oil-sands production began in Canada in the late 1960s with the Great Canadian Oil Sands operation in Alberta, followed by other major

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commercial projects, including Syncrude, in the 1970s [3], [4]. The main approaches to recovering bitumen from oil sands include hot-water-based extraction and nonaqueous solvent extraction; thermal treatment has also been investigated for bitumen recovery and upgrading. Hot-water extraction is well established, whereas nonaqueous extraction has been investigated as an alternative that can recover bitumen without using large quantities of process water. In nonaqueous extraction, oil sands are mixed with an organic solvent, followed by solid–liquid separation and solvent recovery to obtain a bitumen-containing product [5], [6]. Extraction efficiency depends on solvent type and operating conditions. Previous research has shown that solvent polarity and molecular structure influence compatibility with bitumen components, whereas the solvent-to-oil-sand ratio, extraction temperature, stirring speed, and extraction time also affect recovery [7]. Thus, solvent extraction is a potential approach to recovering bitumen from oil-wet oil sands.

Bitumen is a multicomponent system comprising asphaltenes, resins, aromatic hydrocarbons, and saturated hydrocarbons. At sufficiently high temperatures, thermal decomposition can cleave chemical bonds and generate lower-molecular-weight compounds. Products may include light gaseous hydrocarbons (C_1–C_4), liquid hydrocarbons (including paraffins, naphthenes, and aromatic compounds), and solid carbonaceous residue (coke). Asphaltene decomposition has been reported mainly at 350–420 °C, whereas resin decomposition occurs at approximately 300–400 °C; lighter fractions can evaporate at lower temperatures. At temperatures above approximately 450–500 °C, extensive secondary reactions can increase gas and coke formation. These high-temperature processes are distinct from the mild heating (40–50 °C) used for solvent-assisted extraction in the present study.

Although thermal, catalytic, solvent-based, and hydrothermal methods have been proposed for recovering bitumen or producing synthetic oil from oil sands, some approaches are energy-intensive, economically challenging, or dependent on specialized reagents [8].

Oil-sand deposits occur in several regions of Kazakhstan, including the Beke, Munaily-Mola, and Dongeleksor deposits. Previous studies have investigated thermal recovery of natural bitumen from these deposits and shown that extraction behavior depends on deposit characteristics and processing conditions [9]. Characterization of natural bitumen from Munaily-Mola oil sands revealed oil, resin, and asphaltene fractions of 46.3, 47.5, and 6.2 wt%, respectively. These values indicate a substantial resin content and highlight the importance of developing suitable processing approaches for this unconventional resource [10].

Solvent-assisted thermal processes have been investigated to improve the mobility and recovery of heavy bitumen. Solvent addition can lower bitumen viscosity and improve flow and drainage, while solvent type and concentration can affect recovery behavior [11]. Combining solvent action with moderate heating therefore offers a potentially useful approach to recovering heavy bitumen. Bitumen is commonly separated into four fractions: saturates, aromatics, resins, and asphaltenes (SARA). The saturate fraction consists mainly of nonpolar aliphatic and alicyclic hydrocarbons, including branched chains. Its reported average relative molecular mass is approximately 200–900, and its H/C atomic ratio is approximately 2. Saturates are often among the more chemically stable bitumen fractions [12], [13].

The aromatic fraction contains hydrocarbons with one or more aromatic rings and may include alicyclic or aliphatic side chains. Polycyclic aromatic molecules can contribute to intermolecular interactions and thus influence bitumen viscosity [14]. Aromatics also act as a dispersing medium for other bitumen components; reported values include average relative molecular masses of approximately 400–2,000, H/C atomic ratios of 1.5–1.7, and mass fractions of 30–60%, depending on the bitumen source [15].

The resin content of bitumen is often reported to be approximately 15–30%, depending on its source. Resins are generally dark-colored compounds with reported densities of approximately 0.98–1.08 g cm^{-3}, average relative molecular masses of 700–4,000, and H/C atomic ratios of 1.4–1.5. During aging, resins can undergo chemical changes that alter the balance between the resin and asphaltene fractions. Resins help disperse or solubilize asphaltenes and can influence the ductility of bitumen [16].

Asphaltenes are chemically complex, high-molecular-weight constituents of petroleum systems. They typically contain aromatic structures, heteroatoms, and aliphatic side chains. Their compositions and molecular architectures vary with petroleum source, and multiple structural models have been proposed [17]. Asphaltene content likewise varies with the origin and composition of the bitumen or crude oil. Asphaltene aggregation influences the colloidal stability of bitumen. Asphaltene molecules may associate through π–π interactions, hydrogen bonding, dispersion forces,

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and acid–base interactions. The extent of aggregation and the size of the resulting aggregates can affect the colloidal properties and stability of bitumen [18]. Despite considerable progress in bitumen extraction and characterization technologies, the molecular composition of natural bitumen from Kazakhstan's oil-sand deposits remains insufficiently investigated, particularly at the molecular level. Most previous studies have focused on extraction methods, thermal behavior, and bulk physicochemical properties, whereas detailed characterization of heteroatom-containing compounds and their structural distributions remains limited. High-resolution electrospray ionization Fourier transform ion cyclotron resonance mass spectrometry (ESI FT-ICR MS) provides a powerful analytical approach for investigating the molecular composition of complex petroleum systems, enabling the identification of heteroatom classes and the determination of double-bond equivalent (DBE) and carbon-number distributions.

Therefore, the present study aims to investigate the recovery and molecular characteristics of natural bitumen from the Beke oil-sand deposit in Kazakhstan. Bitumen was recovered using solvent-assisted thermal extraction, and the extraction performance was evaluated by determining the residual bitumen content. The recovered bitumen was subsequently characterized using negative-ion ESI FT-ICR MS, with particular emphasis on heteroatom-class distributions and the molecular characteristics of O_2- and N_1-containing compounds. The findings provide insights into the molecular composition of Beke bitumen and contribute to a better understanding of its potential for further processing and utilization.

2 Materials and methods

2.1 Solvent-Assisted Thermal Extraction of Bitumen from Oil Sands

Oil sands from the Beke deposit were used as the raw material. The extraction procedure is illustrated in Figure 1. The raw oil sands were mechanically ground to a particle size of 1–3 mm. Toluene was used as the extraction solvent at a solvent-to-oil-sand ratio of 3 mL g^{-1} (equivalent to 3 mL of toluene per gram of oil sand). The mixture was heated to 40–50 °C and stirred continuously at 300 rpm using a magnetic hotplate stirrer for 30 min to promote solvent contact with the oil-sand matrix and release bitumen from the mineral phase.

The heated mixture was subjected to three-stage filtration. It was first passed through a metal sieve with an aperture of 0.5–0.9 mm, followed by fine-mesh filter cloth with an aperture of 0.3–0.4 mm and, finally, ashless filter paper to remove mineral particles of different sizes. The resulting bitumen–toluene solution was collected, and the toluene was removed by atmospheric distillation to recover bitumen. Distillation continued until the recovered product reached a constant mass, indicating that volatile solvent components had been substantially removed. The recovered bitumen was used for subsequent physicochemical and molecular characterization.

The initial bitumen content of the Beke oil sands was determined gravimetrically. After extraction, the solid residue was re-extracted to determine the residual bitumen content. The initial bitumen content was 12.0 wt%, and the residual bitumen content was 1.576 wt%. Across three independent experiments, the residual bitumen content was 1.576 ± 0.034 wt% (mean ± standard deviation, n = 3). Bitumen removal efficiency was calculated as follows:

$$\eta = \frac{C_0 - C_r}{C_0} \times 100\%$$

where C_0 is the initial bitumen content and C_r is the residual bitumen content after extraction. The calculated bitumen removal efficiency was 86.9%.

All extraction experiments were performed in triplicate under identical conditions; extraction performance was calculated from the three independent runs.

2.2 ESI FT-ICR MS Analysis

The molecular composition of the recovered bitumen was analyzed by high-resolution ESI FT-ICR MS using a 9.4 T Apex Ultra FT-ICR mass spectrometer (Bruker, Germany). A stock solution was prepared by dissolving the bitumen in toluene at a concentration of 10 mg mL^{-1}. A 10 µL aliquot was diluted with a toluene/methanol mixture (1:3, v/v) to a final concentration of 0.1 mg mL^{-1}, followed by gentle mixing to prepare the solution for negative-ion ESI analysis. To enhance ionization in negative-ion mode, 15 µL of ammonium hydroxide solution (NH_4OH) was added per milliliter of sample solution. Before analysis, the FT-ICR MS instrument was calibrated using well-characterized N_1-series compounds, and the mass error was maintained within 1 ppm. Peaks with a signal-to-noise ratio (S/N) greater than 5 were selected for analysis. The acquired data were imported into Microsoft Excel and processed further using an in-house laboratory program. Molecular formulas were assigned from

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accurate masses, with the relative intensities of neighboring isotope peaks considered during identification. Assigned formulas were grouped by heteroatom class, and double-bond equivalent (DBE) and carbon-number distributions were determined to characterize the detected molecular species.



Figure 1. Experimental procedure for solvent-assisted thermal extraction of bitumen from oil sands.

3 Result and discussion

3.1 Extraction Performance of Bitumen from Oil Sands

Solvent-assisted extraction performance was assessed by measuring the bitumen remaining in the treated sand after subsequent re-extraction. Beke oil sands were treated at 40–50 °C, and the residual bitumen content was determined.

The residual bitumen content was 1.576 wt%. Compared with an initial bitumen content of 12.0 wt%, this corresponds to a calculated removal efficiency of approximately 86.9%. The relatively low residual content indicates that most of the initially measured bitumen was separated from the mineral matrix under the investigated conditions.

The remaining bitumen may have been retained on mineral surfaces or trapped in fine pores and microcracks. Thus, a small fraction of the organic phase remained associated with the mineral matrix after treatment. Overall, the results indicate substantial bitumen removal from Beke oil sands under the applied extraction conditions.

3.2 Molecular Characterization of Extracted Bitumen by ESI FT-ICR MS

High-resolution negative-ion electrospray ionization Fourier-transform ion cyclotron resonance mass spectrometry (ESI FT-ICR MS) was used to characterize the molecular composition of the recovered Beke bitumen. The mass spectrum is shown in Figure 2. The detected ions were mainly distributed over an m/z range of 200–700, consistent with a broad range of ionizable molecular species in the extracted bitumen.

High-resolution negative-ion electrospray ionization Fourier-transform ion cyclotron resonance mass spectrometry (ESI FT-ICR MS) was employed to investigate the molecular composition of the recovered Beke bitumen. The corresponding mass spectrum is presented in Figure 2. The detected ions were predominantly distributed within the *m/z* range of 200–700, indicating the presence of a complex mixture of ionizable molecular species with different molecular masses. The spectrum exhibited numerous closely spaced peaks, reflecting the substantial molecular heterogeneity characteristic of petroleum-derived bituminous materials. The highest signal intensities were concentrated mainly between *m/z* 250 and 500, whereas the abundance of detected ions decreased progressively toward higher mass-to-charge ratios. This distribution suggests that the negative-ion ESI response was dominated by relatively low- to intermediate-molecular-mass constituents, while higher-mass ionizable

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compounds were detected at comparatively lower intensities. Several prominent signals were observed at *m/z* 255.23288, 279.23290, 319.26418, 391.35807, 475.45175, 517.49869, and 601.59222. Among these, the peak at *m/z* 319.26418 exhibited the highest signal intensity, making it the base peak of the spectrum. Other relatively intense signals, particularly those at *m/z* 255.23288 and 391.35807, further demonstrate that certain ionic species contributed more strongly to the measured signal than others. The presence of numerous additional peaks across the investigated mass range indicates considerable molecular complexity, potentially arising from variations in carbon number, heteroatom content, and molecular structure. Such complexity is characteristic of natural bitumen, which contains diverse petroleum-derived compounds, including resinous and asphaltenic constituents with different polarities and molecular architectures. Consequently, the obtained spectrum provides information primarily about the negative-ion-forming fraction of Beke bitumen rather than its complete molecular composition.

The distribution of signals across a broad mass range suggests the presence of multiple families of ionizable compounds, potentially differing in carbon number, heteroatom composition, and degree of unsaturation. However, the identification of individual molecular formulas and compound classes requires accurate-mass assignment, appropriate mass-calibration tolerances, and consideration of elemental constraints. Further analysis of heteroatom classes (such as O_1, O_2, N_1, and S_1O_x, where supported by molecular-formula assignments), double-bond equivalent (DBE) distributions, carbon-number distributions, and Kendrick mass defects would provide a more detailed understanding of the molecular characteristics of the recovered bitumen.

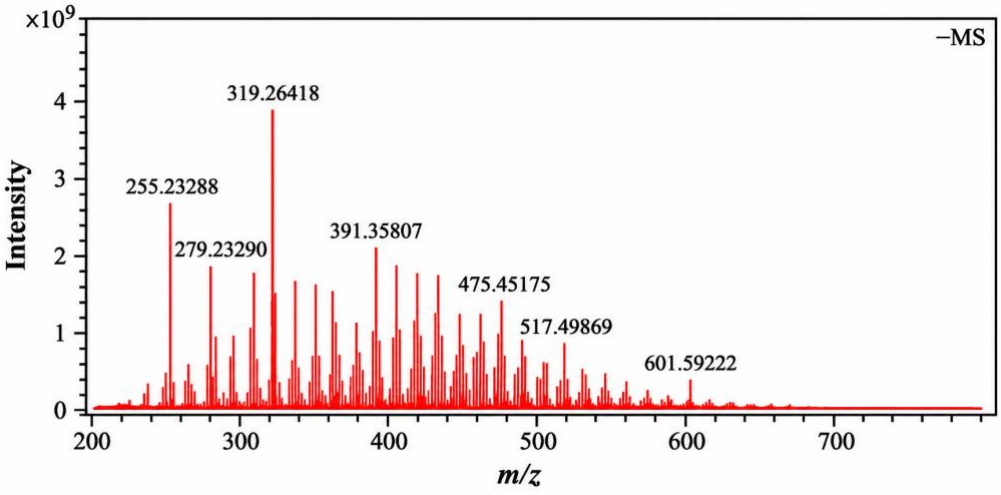


Figure 2. Negative-ion ESI FT-ICR mass spectrum of Beke bitumen.

Figure 3 presents the distribution of heteroatom-containing molecular classes identified in Beke bitumen using negative-ion ESI FT-ICR MS. The molecular composition was strongly dominated by the O_2 class, with a summed ion intensity of approximately 8.9×10^{10} a.u., substantially exceeding those of the N_1, N_1O_1, N_1O_2, O_1, O_3, and O_4 classes. This predominance suggests that oxygen-containing compounds, particularly species potentially associated with carboxylic acids, constitute a major fraction of the negative-ion-responsive components of Beke bitumen. The O_3 and O_4 classes exhibited considerably lower intensities, while nitrogen-containing classes contributed relatively minor signals. The distribution of double-bond equivalent (DBE) values further indicates molecular diversity within the detected heteroatom classes, reflecting variations in the degree of unsaturation and ring structures.

The pronounced dominance of the O_2 class is consistent with the preferential detection of acidic oxygen-containing compounds under negative-ion electrospray conditions. Such compounds may influence the polarity, interfacial behavior, and interactions of bitumen with mineral surfaces and extraction solvents. However, the measured ion intensities are influenced by differences in ionization efficiency and should not be interpreted as absolute molecular concentrations. These results demonstrate the prevalence of negative-ion-responsive oxygenated species in Beke bitumen and provide valuable molecular-level information for understanding its chemical heterogeneity and potential extraction behavior.

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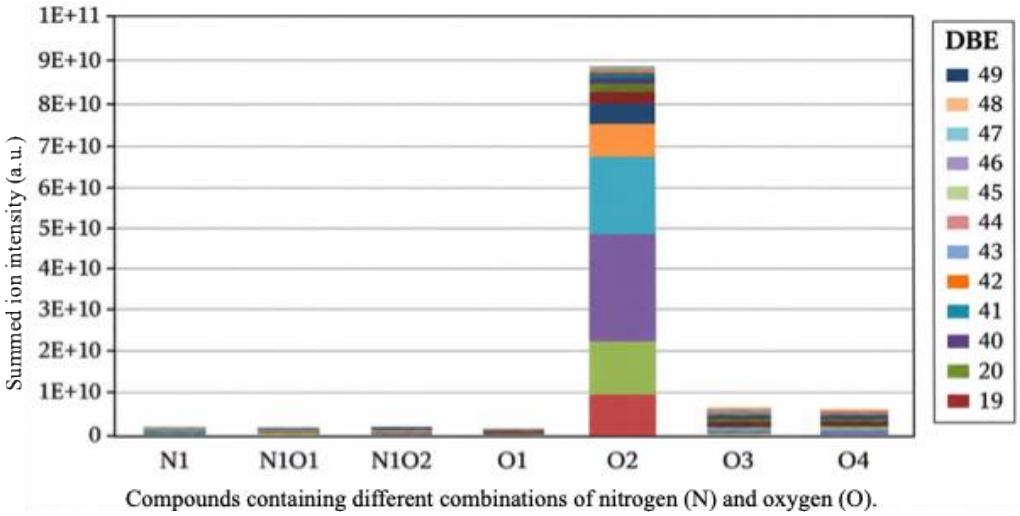


Figure 3. Relative abundances of heteroatom classes detected by negative-ion ESI FT-ICR MS in Beke bitumen, grouped according to double-bond equivalent (DBE) values.

Double-bond equivalent (DBE) values were calculated from the molecular formulas assigned to the FT-ICR MS signals. DBE expresses the total number of rings and π bonds in a molecular formula and can be used to infer the degree of unsaturation. For a general formula CcHhNnOoSs, DBE was calculated as follows:

$$\mathrm{DBE} = c - \frac{h}{2} + \frac{n}{2} + 1 \qquad (1)$$

where c, h, and n denote the numbers of carbon, hydrogen, and nitrogen atoms, respectively. DBE values were plotted against carbon number to obtain DBE–carbon number distributions.

The DBE–carbon number distribution of the O_2 class is shown in Figure 4. O_2 species were mainly detected at carbon numbers of 16–35 and DBE values of 1–4. This dominant oxygen-containing class is consistent with the presence of acidic, potentially petroleum-acid-type constituents; however, specific functional groups cannot be identified from molecular formulas alone.

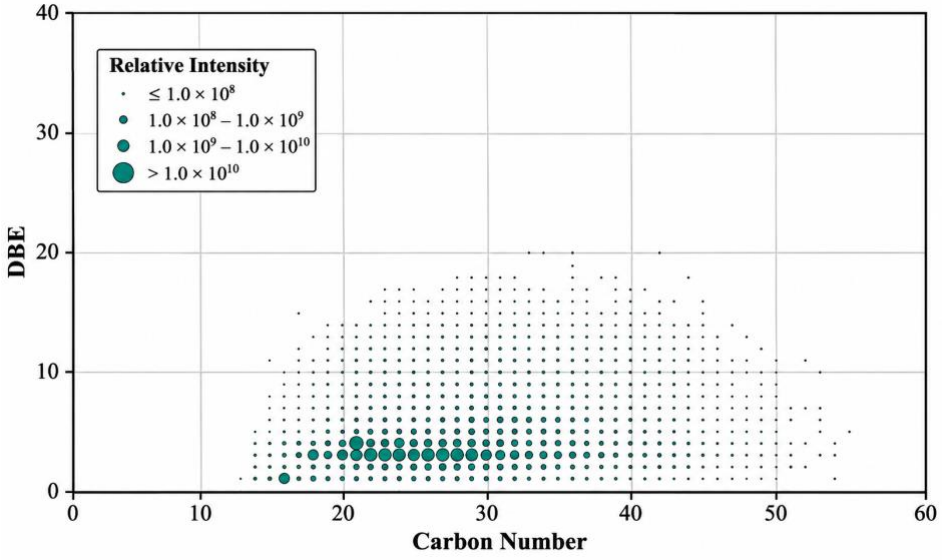


Figure 4. DBE–carbon number distribution of O_2-class compounds in Beke bitumen.

Figure 5 presents the double-bond equivalent (DBE) versus carbon number distribution of N_1-class compounds detected in Beke bitumen by negative-ion ESI FT-ICR MS. The identified molecular species span approximately C_{15}–C_{46}, with DBE values predominantly ranging from 9 to 19, indicating considerable structural diversity among nitrogen-containing compounds. The highest ion

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intensities are concentrated around C_{18}–C_{30} and DBE values of 9–13, suggesting the predominance of relatively low-carbon-number species with moderate to high degrees of unsaturation within the detected N_1 class. These molecular characteristics are consistent with nitrogen-containing aromatic compounds, potentially including carbazole-type structures commonly reported in petroleum systems. Compared with the O_2 class (Figure 4), the N_1 compounds exhibit a distribution shifted toward higher DBE values, suggesting greater unsaturation and potentially higher aromaticity. However, definitive structural identification requires additional analytical evidence, and the observed ion intensities reflect the negative-ion-responsive fraction rather than absolute molecular abundances.

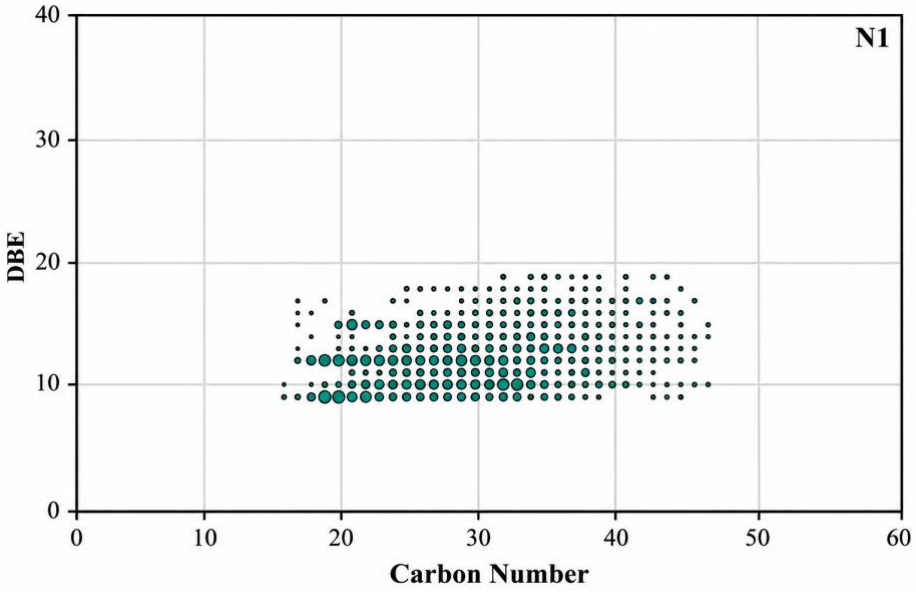


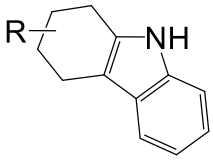
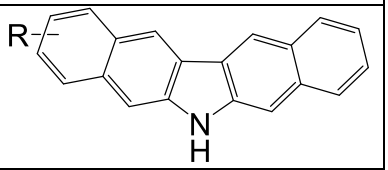
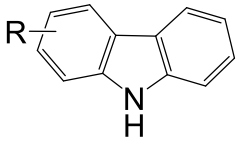
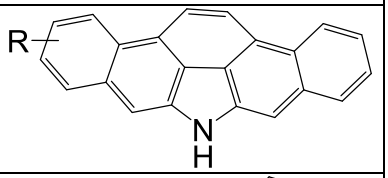
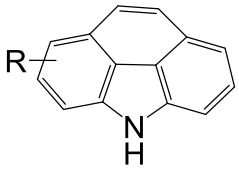
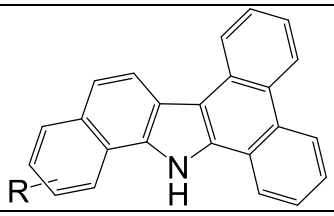
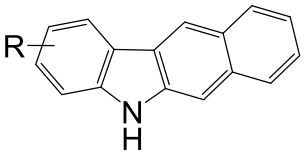
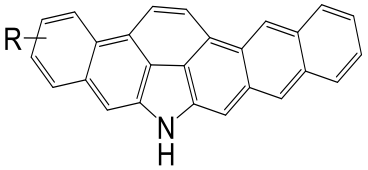
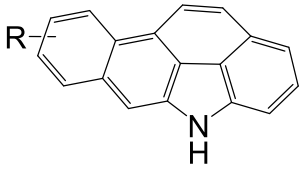
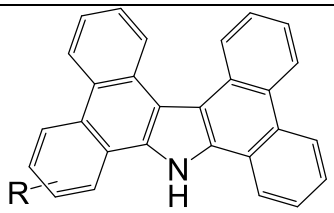
Figure 5. DBE–carbon number distribution of N_1-class compounds in Beke bitumen.

Possible structures corresponding to selected DBE values of the N_1 class are illustrated in Table 1. These structures are representative structural possibilities inferred from the DBE–carbon number distributions rather than definitive identifications. Table 1 presents representative proposed molecular structures of N_1-class compounds at selected double-bond equivalent (DBE) values ranging from 7 to 21. The illustrated structures are predominantly based on carbazole-type nitrogen-containing aromatic frameworks, with increasing DBE values generally corresponding to greater aromatic ring condensation and molecular structural complexity. These proposed structures are consistent with the relatively high DBE values observed for the N_1 class in Figure 5, suggesting the presence of highly unsaturated nitrogen-containing molecular species in Beke bitumen. The identification of such molecular families is important because nitrogen-containing aromatic compounds can contribute to the polarity, intermolecular interactions, and aggregation behavior of heavy petroleum fractions, potentially influencing bitumen viscosity, colloidal stability, and interactions with extraction solvents. In particular, highly condensed aromatic structures may participate in π–π stacking and hydrogen-bonding interactions, depending on their functional groups and molecular environment. Understanding the distribution of these compounds could therefore provide valuable molecular-level insights into the physicochemical behavior of Beke bitumen and support the development of more effective extraction, separation, and upgrading strategies. Nevertheless, the illustrated structures should be regarded as hypothetical molecular configurations rather than experimentally confirmed compounds, because molecular formulas and DBE values obtained from ESI FT-ICR MS cannot independently establish specific structural arrangements or distinguish structural isomers. Additional structural characterization would be required to confirm their identities and evaluate their contributions to the macroscopic properties of bitumen.

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Table 1. Representative hypothetical molecular structures of N_1-class compounds corresponding to selected double-bond equivalent (DBE) values.

DBE	Possible molecular structures	DBE	Possible molecular structures
7		15	
9		17	
11		18	
12		20	
14		21	

4 Conclusion

This study investigated the recovery and molecular composition of bitumen from Beke oil sands in Kazakhstan using solvent-assisted thermal extraction followed by high-resolution ESI FT-ICR MS. The oil sands were treated at 40–50 °C with toluene, and the extracted bitumen was recovered by filtration and atmospheric distillation. The residual bitumen content was 1.576 wt%, corresponding to a calculated removal efficiency of 86.9% under the investigated conditions.

Negative-ion ESI FT-ICR MS showed that the detected ions were mainly distributed over an m/z range of 200–700. The O_2 class was the most abundant heteroatom class, followed by O_3, O_4, and N_1. The O_2 and N_1 classes displayed distinct DBE–carbon number distributions that provide insight into the molecular characteristics of the recovered bitumen.

The N_1 class was mainly detected at DBE values of approximately 10–18 and carbon numbers of 20–40. These features are consistent with carbazole-type and other condensed nitrogen-containing aromatic structures, although the assignments remain tentative rather than definitive molecular identifications. The findings indicate that the investigated solvent-assisted thermal extraction procedure removes most of the measured bitumen from Beke oil sands. ESI FT-ICR MS provides complementary information about the molecular composition of the recovered product, supporting further evaluation of its chemical characteristics and processing potential.

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Author Contributions
U.K.: Methodology, Software, Writing – Original Draft.
Y.T.: Supervision, Conceptualization, Writing – Review & Editing.

Ethics Approval and Consent to Participate
This study did not involve human participants or animals. Therefore, ethical approval and informed consent were not required.

Data Availability Statement
The data supporting the findings of this study are available from the corresponding author upon reasonable request.

Supporting Information
Not applicable.

Conflict of Interest
The authors declare no conflict of interest.

AI Use Disclosure
No AI tools were used to generate scientific results, data, figures, or interpretations. All analyses, conclusions, and scientific content were developed by the authors.

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