Oil Aquathermolysis are over five times of conventional light oil reserves, has oil (such as asphalt and oil sands) due to large reserves, which continuous reduction of light oil reserves, heavy or extra-heavy. With the rapid increase of global energy consumption and the transportation, and refinery challenging. Several interesting options are being considered to address this issue to reduce the viscosity and improve the fluidity of heavy oil, such as thermal, chemical, and microbial recovery. One of the most common and effective thermal options to recover heavy oil is steam stimulation (huff and puff), which uses steam to increase the temperature and break heavy molecular bonds of crude oil to reduce viscosity. The term “aquathermolysis”, proposed by Hyne et al., is used to describe the overall chemical reactions that occur between steam, crude oil, and rock minerals at a specific pressure and temperature. It should be emphasized that their studies revealed that this change is reversible because heteroatoms (S, N, and O) interact with other molecules due to van der Waals forces and thus polymerization, leading to reappearance with high viscosity. The addition of proper catalysts, on the other hand, helps to a large extent to promote the reactions of heavy feeds, leading to an effective and irreversible viscosity reduction. Researchers have recently conducted some studies on homogeneous and heterogeneous catalysts to enhance the performance of reducing crude oil viscosity in aquathermolysis. Homogeneous catalysts include aromatic sulfonate copper complexes and molybdenum oleate, while heterogeneous catalysts include heteropoly acids, zeolites, modified zirconia, etc. Among them, zeolites have attracted much interest due to their high abundance, suitable pore texture, acidity properties, and economy. Merissa et al. studied the activity of two natural zeolites (beudantite and clinoptylilite). They found that when the material was preground and
activated at 300 °C, although it negatively impacted the crystallinity of the catalyst during the gridding process, it effectively reduced the viscosity of crude oil. Junaid et al. found that it has a good effect on heteroatom removal when exploring the role of clinoptilolite in heavy oil pyrolysis. The researchers also found that the porous structure of zeolites can adsorb many substances that are not conducive to the reaction, and their acidity helps produce active hydrogen substances to prevent chain polymerization.

2. EXPERIMENTAL SECTION

2.1. Materials. Zeolites ZSM-5, Y, β, and MCM-41 were purchased from Nankai University Catalyst Co., Ltd. Sodium hydroxide was used to adjust the pH of the solution purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. FeSO₄·7H₂O and Fe(NO₃)₃·9H₂O(AR) were used for the preparation of Fe-containing catalysts purchased from ShangHai Hushi Laboratorial Equipment Co., Ltd. The experiment used two types of oil from the Xinjiang oilfield (light oil) and the Shengli oilfield (heavy oil) in China.

2.2. Preparation of the Fe-Containing Catalyst. The ZSM-5 zeolite (SiO₂/Al₂O₃ = 25, Nankai University Catalyst Co., Ltd., Tianjin, China) was loaded with Fe₂O₃ by the equal volume impregnation method. Iron nitrate solution was added dropwise to zeolite and stirred evenly at 25 °C. The mixture was allowed to stand undisturbed for 1 h, dried at 110 °C for 2 h, and then calcined in a 550 °C muffle furnace for 4 h to obtain the Fe₂O₃/ZSM-5 catalyst.

The preparation method of the Fe₂O₃/ZSM-5 catalyst was as follows: First, 4 g of zeolite was added to 100 mL of deionized water. After stirring for 30 min, a 0.5 mol/L sodium hydroxide (Shanghai Aladdin Biochemical Technology Co., Ltd) solution was added to the mixed solution until the pH reached 12.8. Then, FeSO₄·7H₂O (0.114 mol/L, AR, ShangHai Hushi Laboratorial Equipment Co., Ltd) and Fe(NO₃)₃·9H₂O (0.228 mol/L, AR, ShangHai Hushi Laboratorial Equipment Co., Ltd) were added to the mixed solution, and the pH of the mixture was adjusted to 11 by continuous stirring. Finally, the Fe₂O₃/ZSM-5 catalyst was obtained by drying at 50 °C.

2.3. Characterization of Catalysts. The crystal structure and relative crystallinity of zeolites were determined by X-ray diffraction (XRD, X’pert PRO MPD, PANalytical B.V.). The strong acid and weak acid distribution and total acid content of zeolite were measured by NH₃ temperature-programmed desorption (NH₃-TPD) using a FINESORB-2010 automatic temperature-programmed chemisorption instrument. The infrared spectrum of pyridine adsorbed by the sample was recorded by a Nicolet NEXUS 670 Fourier transform (FT-IR, Thermo Nicolet Corporation) spectrometer to determine the acid type of zeolite samples, and the stereoscopic morphology and size of iron oxide and zeolite samples were measured by a transmission electron microscope (TEM, JEM-2100UHR).

2.4. Characterization of Oil. Oil viscosity was measured by a viscometer (BROOKFIELD DV-II + PRO) at 50 °C. Elements of crude oil and oil after the reaction with zeolites were analyzed by an elemental analyzer (EL, Vario ELIII). The functional groups in oil were determined by an elemental analyzer (EL, Vario ELIII). The functional groups in oil were determined by a NEXUS Fourier transform spectrometer, and the scanning range was 400–4000 cm⁻¹.

2.5. Catalytic Aquathermolysis of Oil. The experiment used two types of oil from the Xinjiang oilfield (light oil) and the Shengli oilfield (heavy oil) in China. The steps of the catalytic aquathermolysis experiment were as follows: 40 g of oil and 10 g of deionized water were mixed in an autoclave (CQF, Dalian Jingyi Autoclave Co., Ltd.), and then 2.5 g of the catalyst was added to the autoclave (initial pressure: 3 MPa). The temperature was set to gradually increase to 88, 140, 200, 250, 270, 275, and 280 °C, and the reaction was carried out at 280 °C for 6 h. Finally, the oil samples were cooled down and removed for further analysis, while the viscosity of the treated oil at 50°C was measured.

Here, the viscosity reduction (Δη%) was calculated as follows: Δη% = (η₀ − η)/η₀ × 100% where η₀ and η (mm²/s) are the viscosities of the oil before and after the catalytic aquathermolysis, respectively.

3. RESULTS AND DISCUSSION

3.1. Effect of Zeolite Species on Catalytic Aquathermolysis Performance. 3.1.1. Physical—Chemical Properties of Zeolite Catalysts. Figure 1 shows the XRD patterns...
of ZSM-5 (Figure 1a), Y (Figure 1b), β (Figure 1c), and MCM-41 (Figure 1d) zeolite catalysts. Among them, ZSM-5 should have the characteristic peaks of the MFI-type zeolite crystalline phase, which is one of the typical characteristic structures of high silica zeolite with a structural framework containing two intersecting pore systems. From the XRD patterns (Figure 1a), it can be seen that the diffraction peaks of the sample at 7.75, 8.66, 22.91, 23.10, and 23.86° corresponded to (011), (020), (051), (511), and (31), respectively, and the sample has no stray peaks, confirming that it is ZSM-5 zeolite.

Y zeolite has an octahedral zeolite cage structure, which has a β-cage as a structural unit. Such adjacent β-cages are connected by hexagonal cages, while β-cages and hexagonal cages are interconnected to form octahedral zeolite cages. From the XRD patterns (Figure 1b), it can be seen that the crystal planes 111, 220, 311, 331, 511, 440, 533, 642, and 555 appeared at diffraction peaks 6.2, 10.14, 11.92, 15.7, 18.74, 20.42, 23.72, 27.16, and 31.52°, respectively, which confirms that the sample is a Y support.

β zeolite is a three-dimensional (3D) 12-ring structure composed of cross-channels, formed by the intersection of a sizeable 12-membered oxygen ring channel and two 12-
Table 1. Analysis of Acid Properties of Various Types of Zeolite Catalysts

| catalysts | total     | strong | weak    | Brønsted/Lewis acid peak area |
|-----------|-----------|--------|---------|------------------------------|
| ZSM-5     | 11,226.63 | 5739.91| 5486.72 | 7060.77 4165.86 1.05 0.59  |
| HY        | 13,729.98 | 6232.09| 7497.89 | 4198.77 9531.21 0.83 2.27  |
| Hβ        | 16,471.36 | 7222.99| 9248.37 | 6806.34 9665.02 0.78 1.42  |
| MCM-41    |           |        |         |                              |

As can be seen from Figure 1c, the strongest diffraction peaks appear at 7.75° and 22.52°, where 7.75° corresponds to the diffraction peak with a small angle, while 22.52° corresponds to the narrow diffraction peak with a large angle, and there are no obvious stray peaks. Therefore, the sample was confirmed as a membered oxygen folded channels. As shown in Figure 1d, the sample shows a strong diffraction peak at 2−3°, corresponding to (100), and two small peaks at 3−5°, corresponding to (110) and (200), respectively. The absence of obvious stray peaks indicates that the sample is MCM-41 zeolite. The pore structure of MCM-41 zeolite is typically a honeycomb: one-dimensional (1D) hexagonal dense arrangement and parallel order in the pore size direction, which indicates that it has long-term stability.

Figure 2 shows that the total area of the diffraction peaks of these three zeolites are β, Y, and ZSM-5 in descending order, with ZSM-5 having the highest sites of Brønsted acid and strong/weak value, which may affect its aquathermolysis performance.

3.1.2. Viscosity Reducing Properties of ZSM-5, HY, Hβ, and MCM-41 Zeolite Catalysts. The viscosity reduction performance of different zeolite catalysts in crude oil (54.64 mm²/s) was compared (Figure 3). The experimental results show that the best catalyst for viscosity reduction is ZSM-5 zeolite, about 22.75%, followed by HY and Hβ, while the MCM-41 zeolite catalyst increased the viscosity of crude oil.

Figure 4 shows the infrared spectra of the crude oil before and after the reaction with different catalysts. The absorption peaks at 1222, 1380, 1604, 869, and 813 cm⁻¹ are considered to be PAHs (polycyclic aromatic hydrocarbons). By comparing the spectra of crude oil before and after the reaction, the peak with a large angle, and there are no obvious stray peaks. Therefore, the sample was confirmed as a β support.

As shown in Figure 1d, the sample shows a strong diffraction peak at 2−3°, corresponding to (100), and two small peaks at 3−5°, corresponding to (110) and (200), respectively. The absence of obvious stray peaks indicates that the sample is MCM-41 zeolite. The pore structure of MCM-41 zeolite is typically a honeycomb: one-dimensional (1D) hexagonal dense arrangement and parallel order in the pore size direction, which indicates that it has long-term stability.

The NH₃-TPD patterns and typical Py-IR spectra of different zeolites are shown in Figure 2, and the detailed results are shown in Table 1. The peaks at 1470−1430 and 1570−1640 cm⁻¹ are assigned to Lewis acid sites and Brønsted acid sites coordinated with pyridine, respectively, and L/B represents the ratio of the two areas. The acidity properties of ZSM-5, HY, and Hβ are mainly measured (MCM-41 zeolite is pure silicon with weak acidity). It can be seen that the total acid sites of these three zeolites are β, Y, and ZSM-5 in descending order, with ZSM-5 having the highest sites of strong Brønsted acid and strong/weak value, which may affect its aquathermolysis performance.

Table 2. Elemental Analysis of Oil after Aquathermolysis with Different Catalysts

| catalyst | C%  | H%  | N%  | S%  | H/C |
|----------|-----|-----|-----|-----|-----|
| crude oil| 85.00| 12.16| 0.29| 1.45| 1.71|
| ZSM-5    | 85.65| 12.64| 0.22| 1.33| 1.77|
| HY       | 85.68| 12.44| 0.22| 1.47| 1.74|
| Hβ       | 85.74| 12.53| 0.23| 1.34| 1.75|
| MCM-41   | 85.39| 12.46| 0.24| 1.37| 1.74|

The elemental analysis before and after the reaction of the oil with different zeolites is shown in Table 2. It can be seen that the ZSM-5 catalyst, as one of the best viscosity reduction catalysts, resulted in a higher H/C molar ratio (1.77) and the lowest S and N contents in the reacted oil. The higher H/C molar ratio indicates that more saturated hydrocarbons are generated during the aquathermolysis process, which leads to lower oil viscosity, and the lower S and N contents indicate that more C−S bonds are destroyed in catalytic aquathermolysis, thus improving the oil quality. In addition, MCM-41 is the worst effective catalyst for viscosity reduction, resulting in a slightly higher H/C molar ratio and lower N content than ZSM-5 treated oil. Compared with the crude oil catalyzed by the other two catalysts, the H/C molar ratios of HY and Hβ are similar, but the N content of the Hβ catalyst is lower, which also showed better viscosity reduction performance. This suggests that N content may affect the viscosity of crude oil.

The reduction of crude oil viscosity by the catalytic aquathermolysis of crude oil results from the breakage of macromolecules bonds, and then broken-bond molecules and H are combined to avoid viscosity regression. But MCM-41 is a
substance without acidity, which cannot act as a hydrogen donor. Therefore, some macromolecules are recombined, resulting in increased oil viscosity, which is in line with the report in the literature. In contrast to the other three zeolites, ZSM-5 has the most Bronsted acid content, which can provide the most H protons, so its viscosity reduction performance is the best. The HY zeolite catalyst has a high total acid content, and it has been reported in the previous literature that its performance is not very good. In this experiment, it is found that the amount of Bronsted acid is less than that of ZSM-5, which may be the reason for the poor viscosity reduction effect. In addition, the content of Bronsted acid in the HY catalyst is less than that in HZ, but the effect is better, which may be due to different zeolite configurations.

3.2. Effect of Fe/ZSM-5 on Catalytic Aquathermolysis Performance. Iron has good hydrothermal stability and magnetism, which exhibits good activity and stability in aquathermolysis. It is effective to improve the catalytic performance of zeolite and make it magnetic and thus recoverable. Therefore, it is considered to combine iron with the selected ZSM-5 zeolite with a certain viscosity reduction performance to obtain better catalytic effects of zeolite. Here, FeO and FeO were loaded on the ZSM-5 support, separately, and their activity and viscosity reduction performance in zeolite were studied.

3.2.1. Characterization of the FeO/ZSM-5 and FeO/ZSM-5. Figure 5 shows the XRD patterns of ZSM-5, FeO/ZSM-5, and FeO/ZSM-5 catalysts. It is found that they all have typical diffraction peaks of ZSM-5 topology, and the strongest diffraction peaks appear at 7.75, 8.66, 23.10, and 23.86°. In addition, the peaks of FeO are seen at 2θ values of 30.23, 36.03, 43.13, and 56.32° are (220), (311), (400), and (511) respectively. The characteristic peaks of FeO appear at 35.6, 49.5, and 54.1°, confirming that FeO particles have been deposited on the support successfully. From the XRD patterns, it is also evident that the intensities of FeO/ZSM-5 and FeO/ZSM-5 are lower than that of pure ZSM-5. This is because the loading of FeO and FeO may have led to the formation of iron on the surface of ZSM-5, thereby reducing the crystallinity of zeolite. On the other hand, the synthesis of FeO particles needs to be carried out in an alkaline environment, which may result in the destruction of the zeolite framework and greatly reduce the crystallinity as well.

The TEM image of FeO/ZSM-5 obtained by the impregnation method is shown in Figure 6. It can be seen that the average diameter of the obtained FeO particles is ~31.9 nm, which has good interaction with ZSM-5 zeolite and can be deposited and dispersed on it well.

Figure 7 shows the magnetic properties of pure ZSM-5 and FeO/ZSM-5 catalysts in air and crude oil. The good magnetic properties of FeO/ZSM-5 catalysts in both air and oil suggest that they can be separated in crude oil. It is known that conventional zeolites usually require high-speed centrifugation for separation, which is expensive and time-consuming. In contrast, magnetic FeO/ZSM-5 catalyst magnetic separation has obvious advantages, such as simplicity, efficiency, and economy.

3.2.2. Viscosity Reducing Properties of ZSM-5, Fe-Loaded Zeolite Catalysts. To further explore the viscosity reduction performance of modified ZSM zeolite in heavier crude oil, which is closer to the actual situation and broadens its application range, crude oil with a viscosity of 347 mm²/s was used here. The viscosity of oil before and after treatment is shown in Figure 8. Results show that pure ZSM-5 reduced the viscosity of crude oil by 19.41%, and FeO/ZSM-5 reduced it by ~16%, even not as good as ZSM-5. FeO/ZSM-5 had the best viscosity reduction performance of 20.30%.

The infrared spectra of oil after different zeolite reactions are shown in Figure 9. It is known that the absorption peaks at 1222, 1380, 1604, 869, and 813 cm⁻¹ are assigned to polycyclic aromatic hydrocarbons, the peak at 2924 cm⁻¹ corresponds to the C–H stretching of methyl and methylene groups, and the peak at 3392 cm⁻¹ corresponds to hydrogen bonds. By comparing the IR spectra, it is found that the C–H absorption peak at 2924 cm⁻¹ becomes slightly stronger after the reaction, which is mainly due to the hydrogenation of unsaturated groups and the breakage of long-chain alkanes, while the hydrogen bond becomes weaker after the reaction, indicating that the hydrogen bonds in resin and asphaltene are dissociated, which reduces the intensities of hydroxyl and amine groups effectively.

The results of the elemental analysis of oil after the reaction are shown in Table 3. It is found that FeO/ZSM-5 is more ideal than other catalysts in both the H/C molar ratio and S content, which is consistent with the change in crude oil viscosity. The high H/C molar ratio of FeO/ZSM-5 may be attributed to the hydrogenation of unsaturated bonds, and the lower S content indicates that the C–S bonds in crude oil are easier to destroy during catalytic aquathermolysis.

In a word, the viscosity reduction rate of FeO/ZSM-5 is ~16%, whose performance is lower than that of pure ZSM-5. This may be because the hydrothermal effect of FeO in this oil sample is not good, which makes the catalytic effect of FeO/ZSM-5 less obvious.
ZSM-5 support worse and inhibits its viscosity reduction performance. In contrast, the viscosity reduction effect of \( \text{Fe}_2\text{O}_3/\text{HZSM-5} \) increases to 20.30%. This is mainly because the existence of \( \text{Fe}_2\text{O}_3 \) leads to single-bond breakage, which reduces the heavy components (resin and asphaltene) and increases saturated hydrocarbons and aromatics. In addition, the loaded \( \text{Fe}_3\text{O}_4 \) makes ZSM-5 zeolite magnetic and easy to separate from heavy oil. \( \text{Fe}_3\text{O}_4/\text{HZSM-5} \) has the advantages of simple preparation, low cost, recyclability, etc., and it also has a good industrial application prospect.

4. CONCLUSIONS

This paper investigated the fundamental research on the performance of synthetic zeolite catalysts in catalytic aquathermolysis. We first chose ZSM-5, HY, H\( \beta \), and MCM-41 zeolites for the primary aquathermolysis work and then investigated the effects of different zeolite catalysts on the viscosity reduction performance. The results showed that ZSM-5 had the best viscosity reduction performance among the four selected zeolites, which was 1.7 and 4.1 times higher than those of HY and H\( \beta \), respectively, while MCM-41 had the worst effect and increased the crude oil viscosity by \( \sim 5\% \) instead. This suggests that the acidity of zeolites is an important parameter influencing the reaction. However, a high total acid content does not always imply improved performance; Brønsted acid content is the deciding factor. The selected ZSM-5 support was then loaded with Fe, and the performance of the two Fe-modified catalysts was compared. It was found that \( \text{Fe}_2\text{O}_3/\text{ZSM-5} \) had superior viscosity reduction performance, which could reduce the viscosity of heavy oil by more than 20%. Moreover, the \( \text{Fe}_3\text{O}_4/\text{ZSM-5} \) catalyst is magnetic and can be separated from crude oil by a magnet. As a result, this performance provides a possibility for separating and recycling zeolite catalysts and crude oil.

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**Table 3. Elemental Analysis of Oil after the Reaction with Different Catalysts**

| Catalyst    | C%  | H%  | N%  | S%  | H/C|
|-------------|-----|-----|-----|-----|-----|
| crude oil   | 83.19 | 10.78 | 0.27 | 4.94 | 1.5550 |
| ZSM-5       | 82.99 | 10.81 | 0.26 | 4.89 | 1.5631 |
| \( \text{Fe}_2\text{O}_3/\text{ZSM-5} \) | 83.49 | 10.87 | 1.5623 |
| \( \text{Fe}_3\text{O}_4/\text{ZSM-5} \) | 83.31 | 10.86 | 0.27 | 4.77 | 1.5642 |

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Figure 7. Magnetic conditions of (b) pure ZSM-5 and (b) \( \text{Fe}_3\text{O}_4/\text{ZSM-5} \) catalyst in air and (c) \( \text{Fe}_3\text{O}_4/\text{ZSM-5} \) in crude oil.

Figure 8. Viscosity of crude oil and oil after the reaction for ZSM-5, \( \text{Fe}_2\text{O}_3/\text{ZSM-5} \), and \( \text{Fe}_3\text{O}_4/\text{ZSM-5} \) catalysts.

Figure 9. FT-IR spectra of crude oil and oil after the reaction with different catalysts.
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