

Article

Thermocatalytic Upgrading of Polymer Waste to Hydrocarbon Fuel Fractions over Mo- and W-Modified Natural Taizhuzgen Zeolite

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Abstract

Plastic recycling, also known as chemical recycling, is widely promoted worldwide as a sustainable way to reduce polymer waste and produce alternative fuel sources. One of the promising areas in this field is the thermocatalytic decomposition of plastics into hydrocarbon fractions suitable for use as motor fuel. This study investigates the feasibility of using natural Taizhuzgen zeolite separately modified with molybdenum and tungsten and activated by an acid-free method for the thermocatalytic hydrogenation of polymer waste. The catalyst was prepared by modifying the activated zeolite with molybdenum ((NH₄)₆Mo₇O₂₄·4H₂O) and tungsten ((NH₄)₅H₅[H₂(WO₄)₆]·H₂O) salts. Thermal catalytic hydrogenation experiments were conducted under controlled temperature and barometric conditions, and the resulting liquid products were separated into fractions with boiling points of 180 °C, 180–250 °C, and 250–320 °C. The individual and group hydrocarbon compositions of the gasoline, diesel, and heavy gas oil fractions were determined using gas chromatography–mass spectrometry. The structural and surface characteristics of the synthesized composite catalysts were studied using electron microscopy and physicochemical analysis. The results show that Taizhuzgen zeolite modified with molybdenum and tungsten exhibits favorable textural properties and catalytic activity, which increases the yield of liquid fuel fractions. The developed catalyst is promising for use in the resource-efficient thermal catalytic processing of polymer waste into motor fuel.

Keywords: natural zeolite; composite catalysts; polymer waste; thermocatalytic recycling



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1. Introduction

The consumption of plastics has increased dramatically in recent decades due to their versatility, durability, lightweight nature, and low production costs. However, the widespread use of plastics has led to serious environmental problems associated with the accumulation of plastic waste. As a result, the conversion of plastic waste into valuable raw materials, known as chemical recycling, has attracted worldwide attention.

Among available chemical recycling technologies, catalytic hydrocracking is considered a promising method for converting plastic waste into high-quality liquid transportation fuels with low olefin and heteroatom content [1–6]. Many studies have examined the hydrocracking of virgin and waste plastics by direct liquefaction without the addition of

co-feedstocks. These studies examined the hydrocracking of individual polymer types, as well as mixed plastic waste, under various operating conditions [7–10].

Hydrocracking reactions have been studied in catalytic and non-catalytic systems, with particular emphasis on the influence of process parameters such as temperature, hydrogen pressure, reaction time, catalyst type, and catalyst amount. Catalytic cracking typically requires acidic catalysts, which allow operation at low temperatures and subsequently improves product yield and quality. When cracking is carried out in the presence of hydrogen, the process is defined as hydrocracking [11–13].

Comprehensive reviews of the hydrocracking of plastic materials by direct liquefaction without co-flow have been presented in the literature [14–19]. These studies show that, although increasing reaction time promotes hydrocracking, excessively long residence times reduce liquid product yield and increase gas formation. Under typical temperature and pressure conditions and in the presence of a suitable catalyst, reaction times exceeding 60 min are not recommended. Bifunctional catalysts with hydrogenation–dehydrogenation and cracking functions are considered the most effective for hydrocracking plastic materials [20–26]. However, further research is needed to develop improved catalytic systems, including metal-modified microporous, mesoporous, and mesoporous catalysts, in order to reduce the intensity of the process and improve fuel quality. In addition, the mechanisms of catalyst deactivation are still not fully understood and require detailed investigation [27,28]. In this regard, this study was aimed at using natural zeolite of Taizhuzgen geylandite–clinoptilolite modified with molybdenum and tungsten compounds as an effective bifunctional catalyst for the thermocatalytic processing of polymer waste. Unlike traditional approaches based on strong acid activation or complex catalyst synthesis methods, Taizhuzgen zeolite was acid-free activated followed by calcination, which represented a simple and environmentally friendly modification strategy. The addition of Mo(VI) and W(VI) compounds is aimed at introducing hydrogenation–dehydrogenation functionality while maintaining the cracking activity inherent in the natural zeolite structure. Molybdenum (Mo) and tungsten (W) were selected as modifiers for the zeolite catalyst due to their ability to form active metal–acid assemblies that enhance acidity and facilitate the efficient cracking and hydrogenation of high-molecular-weight hydrocarbons. Zeolite modification with transition metals such as Mo and W improves reagent access to active sites, enhances high-temperature stability, and contributes to an increased yield of target liquid fractions of polymer waste processing products. Recent studies have shown that zeolites modified with molybdenum and tungsten exhibit improved adsorption properties and catalytic activity in the thermocatalytic conversion of complex hydrocarbons (e.g., hydrocracking of heavy petroleum fractions and recycling of plastic pyrolysis oil), confirming the relevance and potential of the chosen modification for polymer waste recycling [29–32].

The novelty of this work lies in the systematic evaluation of modified Mo and W natural Taizhuzgen zeolite for the thermocatalytic conversion of polymer waste into hydrocarbon fuel fractions at a relatively moderate hydrogen pressure (up to 5 MPa). Special attention is paid to the correlation of the physico-chemical properties of modified catalysts with their efficiency in terms of product yield, distribution of hydrocarbon fractions and fuel quality. This approach provides a new insight into the potential of natural zeolites as effective and sustainable catalysts for plastic waste recycling [5].

2. Results and Discussion

Thermogravimetric analysis (TGA) was performed to study the thermal stability of composite catalysts based on natural Taizhuzgen zeolite modified with molybdenum and tungsten. TGA profiles for catalysts containing 2 wt.% Mo@Taizhuzgen and 2 wt.% W@Taizhuzgen are shown in Figure 1.

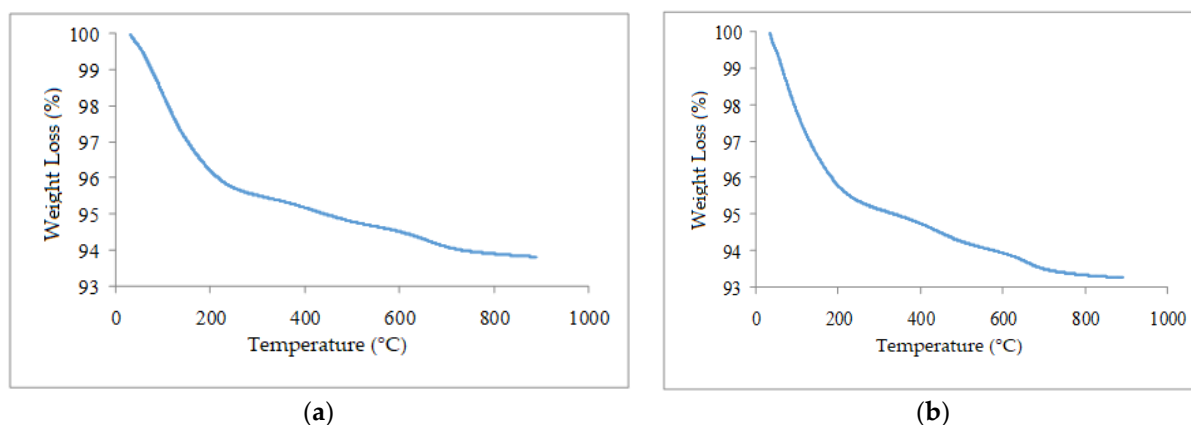


Figure 1. Thermogravimetric analysis (TGA) curves of composite catalysts based on natural Taizhuzhen zeolite: (a) 2 wt.% Mo@Taizhuzhen; (b) 2 wt.% W@Taizhuzhen.

Both catalysts exhibit similar thermal behavior. The initial weight loss observed below 200 °C is attributed to the removal of physically adsorbed moisture and weakly bound surface hydroxyl groups. The gradual weight loss in the temperature range of 200–600 °C is associated with the release of structurally bound water and partial dehydroxylation of the zeolite framework. Above 600 °C, no significant additional weight loss is detected, indicating the high thermal stability of the catalyst structure. At 450 °C, both catalysts show a minimal weight loss of approximately 6–7%, confirming negligible thermal degradation and stable retention of active sites.

Morphological analysis revealed a composite structure with mesoporous regions and preserved crystalline zeolites, which provides efficient diffusion and accessibility during polymer conversion. This combination of thermal stability and pronounced porosity directly contributes to the selective formation of hydrocarbons and the preservation of catalytic activity at elevated temperatures. The structural properties of the catalysts, determined by nitrogen adsorption–desorption measurements, are listed in Table 1. Both samples have relatively low BET surface areas, which is typical for materials based on natural zeolites. However, the catalyst with 2 wt.% molybdenum from Taizhuzhen shows a higher BET surface area ($3.7 \text{ m}^2 \text{ g}^{-1}$), BJH desorption total surface area ($10.5 \text{ m}^2 \text{ g}^{-1}$), and pore volume ($0.033 \text{ cm}^3 \text{ g}^{-1}$) compared to the sample with 2 wt.% molybdenum from Taizhuzhen ($2.6 \text{ m}^2 \text{ g}^{-1}$, $8.4 \text{ m}^2 \text{ g}^{-1}$, and $0.026 \text{ cm}^3 \text{ g}^{-1}$, respectively).

Table 1. Structural properties of the catalysts.

Catalyst	Multipoint B2	BJH Cumulative Desorption Surface Area ($\text{m}^2 \text{ g}^{-1}$)	BJH Cumulative Desorption Pore Volume ($\text{cm}^3 \text{ g}^{-1}$)	BJH Desorption Pore Diameter (mode, Å)
2% Mo@Taizhuzhen	3.7	10.5	0.033	37.4
2% W@Taizhuzhen	2.6	8.4	0.026	21.3

The increased porosity of the Mo-modified catalyst can be attributed to the uniform dispersion of molybdenum particles in the zeolite matrix, which prevents pore clogging and promotes the formation of mesoporous channels during catalyst preparation. In contrast, tungsten particles tend to form larger oxide aggregates, which partially block the pore entrances, leading to a decrease in the available surface area and a decrease in the average pore diameter. This effect is reflected in the BJH pore diameter values, where 2 wt.% molybdenum has a larger pore size (37.4 Å) than 2 wt.% molybdenum (21.3 Å). Overall, the high surface area and mesoporous characteristics of the Mo-modified catalyst are expected to improve mass transfer and access to active sites during the recycling of polymer waste,

making it suitable for high-temperature catalytic applications. The composite nature of the catalyst is evident, as its morphology appears to exhibit a mesoporous phase. The small cubic particles indicate the presence of a zeolitic structure. The results of the catalyst analysis are shown in Table 1.

The results of the N₂ adsorption–desorption measurements are summarized in Table 1 and illustrated in Figure 2. Figure 2 presents the BJH pore size distributions and the corresponding N₂ adsorption–desorption isotherms of the catalysts. As shown in Figure 2, both catalysts exhibit a pronounced increase in nitrogen uptake in the adsorption branch at high relative pressures ($P/P_0 > 0.4$), which is characteristic of capillary condensation in mesoporous materials. The catalysts containing 2 wt.% molybdenum and 2 wt.% tungsten display type IV isotherms according to the IUPAC classification, indicating the presence of mesoporous structures and multilayer nitrogen adsorption. In addition, both catalysts exhibit an H4-type hysteresis loop, suggesting the presence of narrow slit-like mesopores, typically associated with aggregated zeolite crystallites and zeolite-based composite materials. The pore size distributions shown in Figure 2 further confirm the predominance of mesoporosity in both catalysts, in good agreement with the BJH pore diameter and pore volume values listed in Table 1. The Mo-modified catalyst exhibits a broader pore size distribution with relatively larger mesopores compared to the W-modified sample, which is consistent with its higher specific surface area and larger pore volume. Such mesoporosity is particularly advantageous for the catalytic pyrolysis of polymer wastes, as it facilitates the diffusion of bulky intermediates and enhances access to active sites.

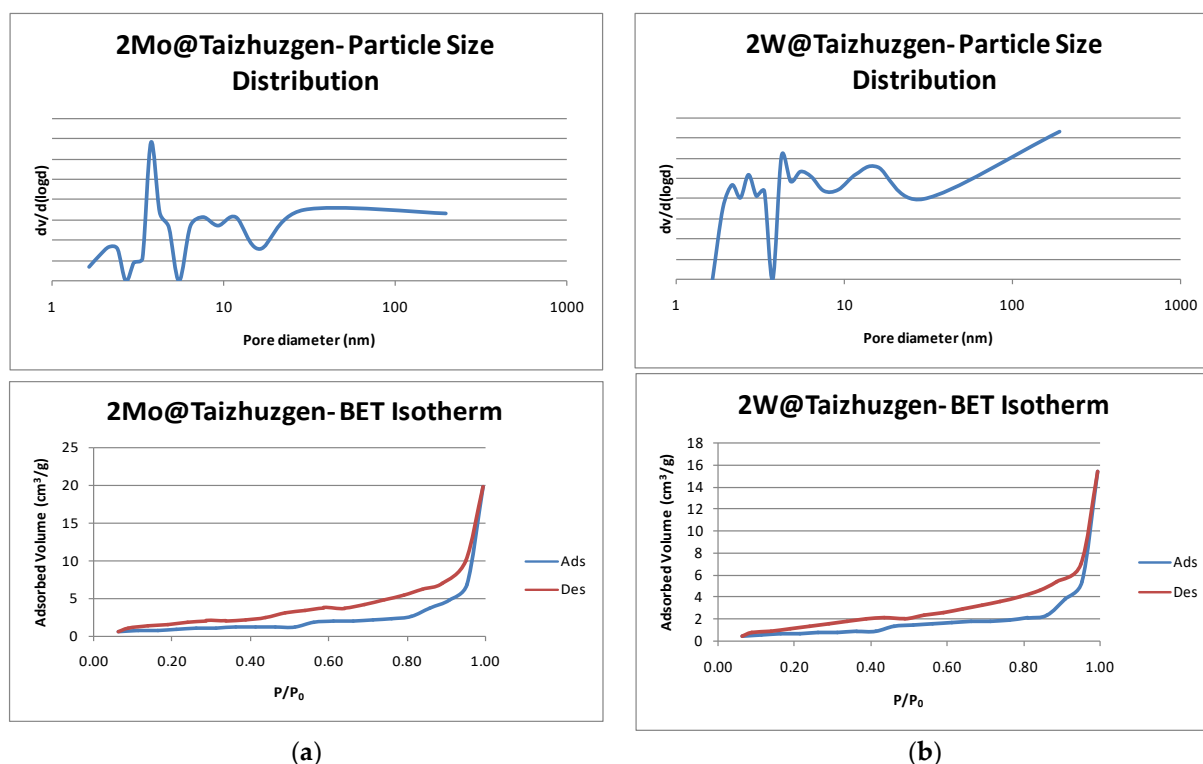


Figure 2. BJH pore size distributions and N₂ adsorption–desorption isotherms of the composite catalysts: (a) 2 wt.% Mo@Taizhuzgen; (b) 2 wt.% W@Taizhuzgen.

These differences in pore structure are reflected in the surface morphology of the catalyst. The larger and more diverse mesopores of the Mo-modified sample indicate a more complex surface, which may affect the interaction with reagent molecules. Thus, SEM imaging provides additional insight into the structural features identified by pore analysis.

Figures 3 and 4 show SEM micrographs of the catalysts with 2% Mo@Taizhuzgen and 2% W@Taizhuzgen at 1000, 5000, and 30,000 times magnification. The magnification reveals a clear heterogeneity in the surface morphology of both catalysts.

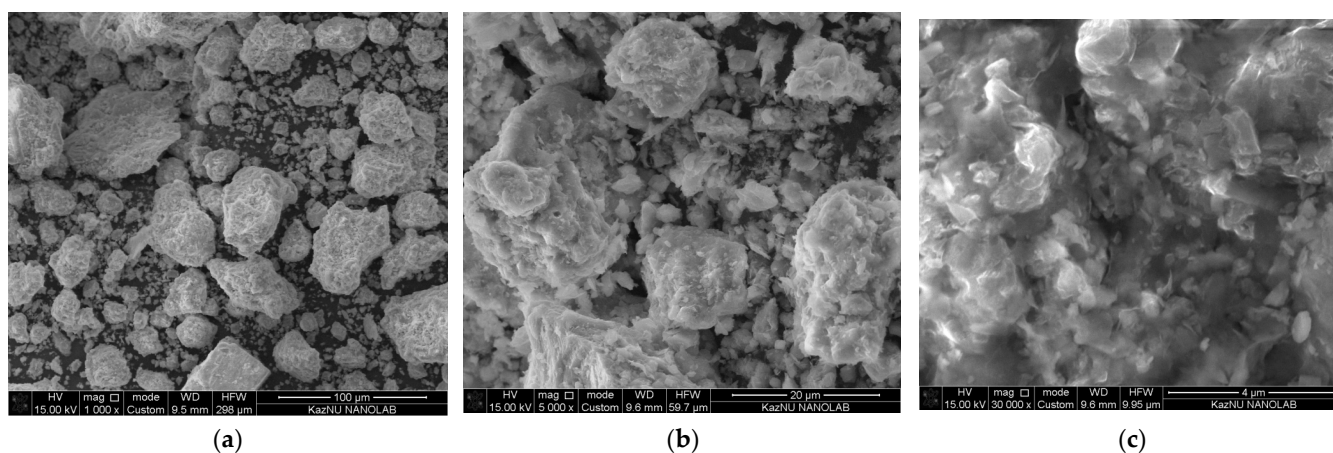


Figure 3. SEM micrographs of activated zeolite modified with 2% Mo@Taizhuzgen at different magnifications: (a) general morphology and particle size distribution at $\times 1000$; (b) agglomerated particles exhibiting a rough and heterogeneous surface after modification at $\times 5000$; (c) high-magnification image at $\times 30,000$ revealing the detailed surface texture and porous structure with finely dispersed Mo-containing species.

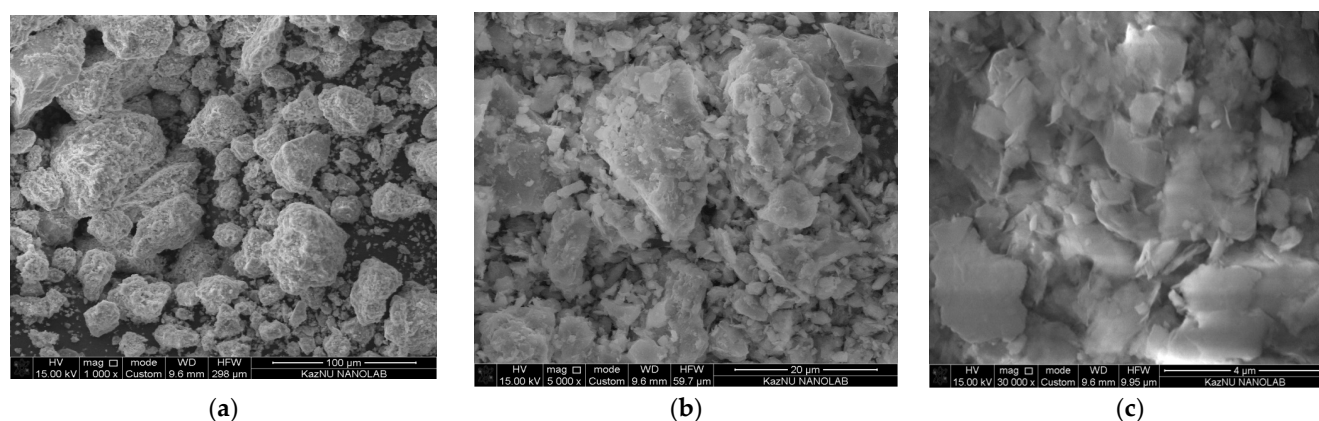


Figure 4. SEM micrographs of activated zeolite modified with 2% W@Taizhuzgen at different magnifications: (a) general morphology and particle size distribution at $\times 1000$; (b) agglomerated particles exhibiting a rough and heterogeneous surface after modification at $\times 5000$; (c) high-magnification image at $\times 30,000$ revealing the detailed surface texture and porous structure with finely dispersed W-containing species.

At low magnification ($\times 1000$), the catalyst particles appear as relatively large, irregularly shaped agglomerates, ranging in size from 3.51 to 23.08 μm . At $\times 5000$ and $\times 30,000$ magnifications, these agglomerates break down into smaller structural units ranging from 82.03 to 256.4 nm for Mo-modified samples and from 266.7 to 775.6 nm for W-modified samples. The catalysts have a predominantly porous structure consisting of closely packed particles, indicating a high level of texture. The modification of the activated zeolite surface with molybdenum and tungsten salts leads to a significant smoothing of the pore edges and an increase in pore size heterogeneity. This effect is more pronounced at higher magnifications, where partial filling of the pores and surface coverage by Mo(VI) and W(VI) particles are observed. These structural changes indicate a strong interaction between the metals and the zeolite structure. Modification of the zeolite with Mo(VI) and

W(VI) salts significantly affects the morphology of the catalyst, which can directly affect its performance. Differences in particle size distribution, surface roughness, and pore structure are expected to affect the availability of active sites and mass transfer processes. Consequently, these morphological differences can lead to changes in the yield and composition of liquid fractions during the thermocatalytic hydrogenation of hydrocarbons.

These morphological observations are further confirmed by X-ray diffraction analysis, which shows that despite the changes in surface structure and pore characteristics, the main diffraction peaks of the original Taizhuzgen zeolite remain virtually unchanged after the addition of metal. This indicates that the overall crystalline structure is preserved, indicating that the addition of molybdenum and manganese modifies the surface without damaging the zeolite structure. The combination of stable crystallinity and modified surface properties provides both structural strength and access to active sites, which is essential for efficient catalyst performance.

The X-ray diffraction patterns of the Taizhuzgen materials containing Mo and W are shown in Figure 5. Comparison of the diffraction patterns of the zeolites modified with Mo or W with those of the original Taizhuzgen zeolite confirms that the addition of Mo and W does not significantly change the structure. These results, together with the observed morphological changes, indicate that the catalysts possess a balance of structural integrity and surface functionality, which is important for optimizing selectivity and productivity in hydrocarbon conversion reactions.

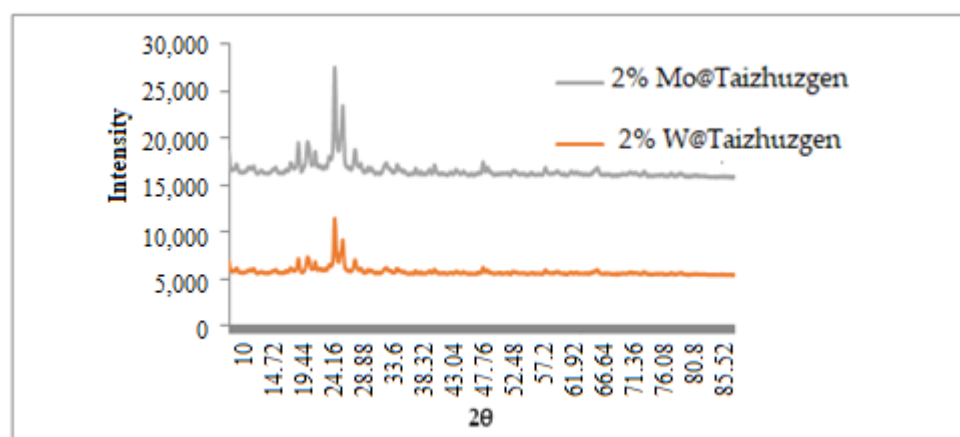


Figure 5. XRD pattern of the catalysts: 2% Mo@Taizhuzgen; 2% W@Taizhuzgen.

However, after metal loading, a slight increase in the intensity of the diffraction peak at 2° is observed, which is approximately equal to 28° . According to Wu et al., the main diffraction peaks of MoO_2 and MoO_3 appear at approximately 26° and 28° , respectively [33–38]. These peaks overlap with the characteristic diffraction peaks of Taizhuzgen zeolite located at 26.74° and 28° . Thus, the increase in the intensity of the peak at 28° in the Mo-containing sample is explained by the presence of molybdenum oxide. Similarly, the increase in the peak near 28° in the X-ray diffraction pattern of the 2% W@Taizhuzgen catalyst is associated with the formation of hexagonal WO_3 [39].

To further determine the chemical state of molybdenum and tungsten, X-ray diffraction analysis of the catalysts containing 2% molybdenum and 2% tungsten was carried out. For each sample, the overview and high-resolution spectra of O1s, C1s, Mo3d, and W4f were obtained, as shown in Figure 6. The X-ray diffraction results are consistent with the X-ray diffraction results and confirm that both metals are mainly in the oxide form.

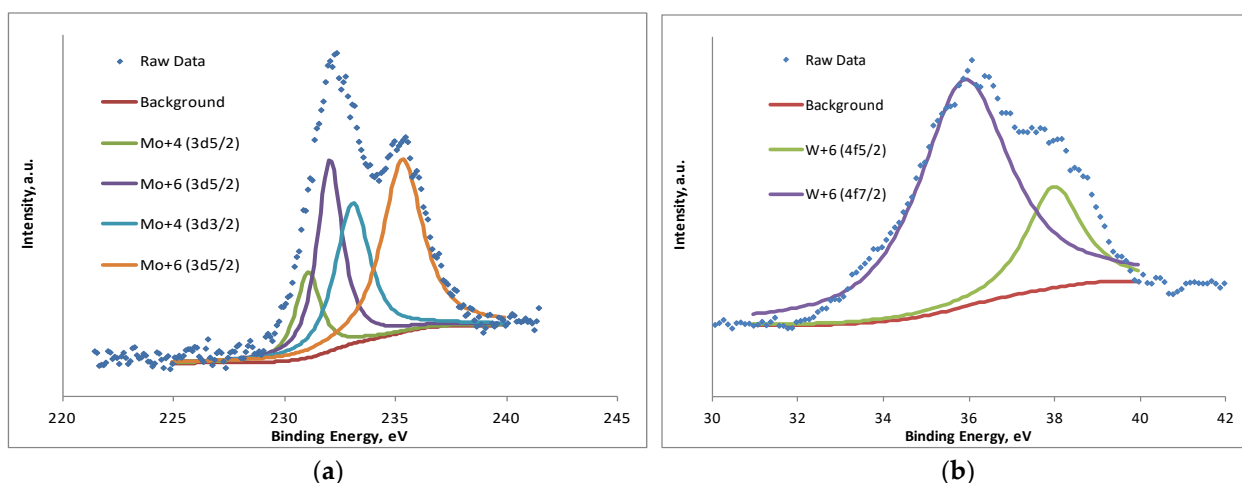


Figure 6. XPS spectra of the catalysts: (a) 2% Mo@Taizhuzgen; (b) 2% W@Taizhuzgen.

Detailed X-ray diffraction analysis not only confirms the presence of molybdenum and tungsten oxides, but also allows us to understand the distribution of their oxidation states on the catalyst surface. This information is very important, because the oxidation state directly affects the electronic environment, surface reactivity, and the presence of active sites, which in turn affect the catalytic performance. Combining these results with the structural information obtained from X-ray diffraction analysis allows us to fully understand the chemical composition of the catalyst surface.

In the XPS spectrum of 2% Mo@Taizhuzgen, two peaks at 231.1 eV and 233.1 eV correspond to the Mo^{4+} oxidation state, while additional peaks at 232.0 eV and 235.3 eV belong to the Mo^{6+} oxidation state [40]. These binding energy values are in good agreement with published data for MoO_2 and MoO_3 species. The peaks observed at 35.9 eV and 38.0 eV for the 2% W@Taizhuzgen catalyst correspond to the W^{6+} oxidation state characteristic of WO_3 and are very close to the literature values [33].

Combined X-ray diffraction and IR spectroscopy provide a comprehensive understanding of the chemical composition and surface properties of the catalysts. XPS reveals the oxidation states of Mo and W, confirming the presence of $\text{Mo}^{4+}/\text{Mo}^{6+}$ and W^{6+} compounds, while FTIR reveals the stability of acidic sites on the zeolite surface. The synergy between metal content and surface acidity is expected to play a crucial role in regulating catalytic activity and selectivity in hydrocarbon conversion reactions.

The FTIR spectra of catalysts containing 2 wt.% molybdenum and 2 wt.% tungsten supported on natural zeolite are shown in Figure 7. The initial natural zeolite exhibits a characteristic absorption band at approximately 1031.5 cm^{-1} , which is attributed to asymmetric stretching fluctuations of Si–O–Si and/or Si–O–Al bonds in the zeolite framework. The wide absorption band centered at about 3400 cm^{-1} corresponds to stretching fluctuations of surface hydroxyl (–OH) groups and adsorbed water, while the band observed at about 1650 cm^{-1} corresponds to bending fluctuations of molecular water. Absorption bands in the range of $2920\text{--}2850\text{ cm}^{-1}$ are associated with fluctuations in the stretching of C–H bonds of aliphatic groups. The bands located approximately in the range of $800\text{--}780\text{ cm}^{-1}$ are associated with symmetrical fluctuations in the stretching of Si–O bonds in the zeolite structure. Moreover, the appearance and/or enhancement of absorption bands in the low-frequency range of $600\text{--}450\text{ cm}^{-1}$ can be attributed to fluctuations in metal–oxygen bonds (Mo–O and W–O), which confirms the successful incorporation of molybdenum and tungsten compounds into the zeolite matrix.

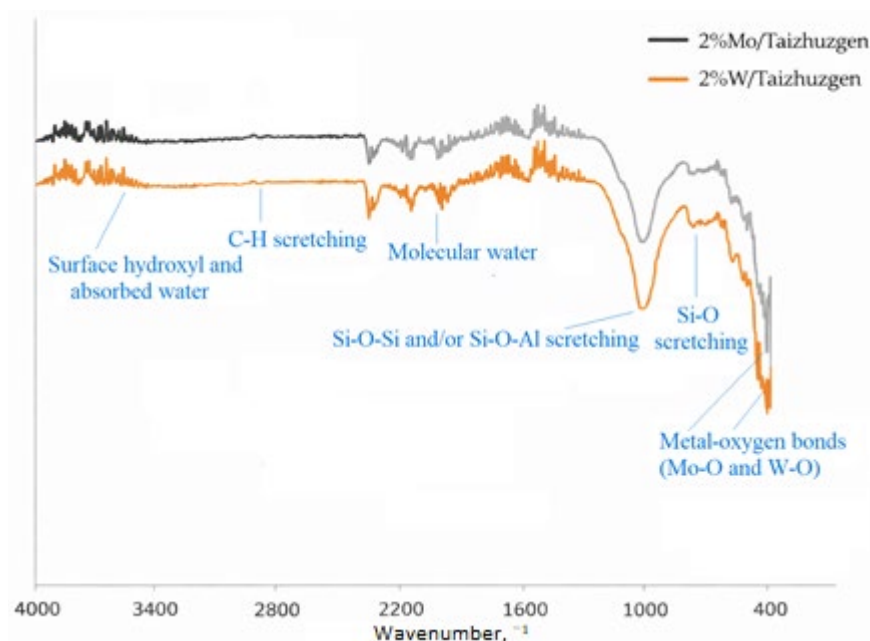


Figure 7. FT IR spectroscopy of the catalysts: 2% Mo@Taizhuzgen; 2% W@Taizhuzgen.

Minor shifts in the positions and intensities of the absorption bands after metal deposition indicate changes in surface acidity, suggesting the presence of both Brønsted and Lewis acid centers. These changes are related to the interaction between metal particles and the zeolite framework and contribute to an increase in the catalytic activity of zeolite-containing materials during the catalytic cracking of carbonaceous raw materials, which leads to the formation of liquid products enriched with short-chain paraffin, naphthenic and aromatic hydrocarbons [5].

To determine the optimal catalyst for the thermocatalytic conversion of polymer residues, a series of control and metal-modified catalysts based on natural Taizhuzgen zeolite were systematically investigated. The tested catalysts included unmodified activated Taizhuzgen zeolite, as well as Mo- and W-modified zeolites with metal loadings of 0.5, 1.0, 1.5, and 2.0 wt.%. The product distribution obtained over different catalysts is summarized in Table 2. The non-catalytic experiment resulted in a low yield of liquid products, with a significant fraction of solid residue, indicating incomplete conversion of polymer materials. The use of unmodified Taizhuzgen zeolite slightly increased the liquid product yield; however, its catalytic activity remained limited, demonstrating that the natural zeolite itself provides only a minor contribution to the overall conversion process. For Mo-modified catalysts, a clear dependence of catalytic performance on metal loading was observed. At low Mo contents (0.5–1.0 wt.%), the yield of liquid products increased only moderately. A substantial enhancement was achieved when the Mo loading was increased to 1.5 wt.%, resulting in a liquid product yield of 53.25 wt.%. The highest performance was obtained for the 2.0 wt.% Mo/Taizhuzgen catalyst, which produced 61.56 wt.% liquid products, accompanied by a pronounced increase in the high-boiling fraction (250–320 °C). This behavior indicates improved cracking efficiency and intensified secondary reactions promoted by higher molybdenum content. A similar trend was observed for W-modified catalysts. Catalysts containing 0.5–1.0 wt.% W exhibited relatively low liquid product yields. Increasing the tungsten loading to 1.5 wt.% led to a noticeable increase in liquid yield, while the 2.0 wt.% W/Taizhuzgen catalyst achieved 60.14 wt.% liquid products. These results confirm the important role of tungsten species in promoting thermocatalytic degradation and stabilization of liquid hydrocarbons. Overall, the results demonstrate that both the presence of metal species and their loading levels strongly affect catalytic

performance. Catalysts with metal loadings below 1.5 wt.% exhibited insufficient activity, whereas catalysts containing 2.0 wt.% Mo or W showed optimal performance in terms of liquid product yield and reduced solid residue formation. Therefore, 2 wt.% Mo/Taizhuzgen and 2 wt.% W/Taizhuzgen catalysts were selected as the most effective systems for further investigation. The product distribution obtained from separate thermocatalytic processing of polymer and heavy oil residue is presented in Table 3.

Table 2. Effect of catalyst type and metal loading on product distribution during thermocatalytic conversion of polymer residues.

Catalyst	Gas Output, %	Yield of Liquid Products, %			Solid Residue, %	Loss, %
		0–180 °C	180–250 °C	250–320 °C		
Without a catalyst	7.42	6.32	8.78	10.56	55.60	11.32
Taizhuzgen zeolite	24.29	8.23	10.08	11.56	33.89	11.95
0.5% Mo@Taizhuzgen	23.26	9.21	11.2	14.11	30.13	12.09
1.0% Mo@Taizhuzgen	20.19	11.52	12.79	16.79	26.7	12.01
1.5% Mo@Taizhuzgen	18.19	15.07	16.74	21.44	15.57	12.99
2% Mo@Taizhuzgen	15.31	12.44	19.12	30.00	10.8	12.33
0.5% W@Taizhuzgen	20.02	10.56	11.52	20.02	26.63	11.25
1.0% W@Taizhuzgen	20.95	10.68	11.6	20.95	18.31	17.51
1.5% W@Taizhuzgen	18.62	12.8	15	18.62	16.84	18.12
2% W@Taizhuzgen	16.30	20.34	21.96	18.10	9.43	13.87

Run conditions: T = 450 °C, p = 0.5 MPa, m (polymer):m (heavy oil residue) = 1:1, m (catalyst) = 2% of total mass.

Table 3. Product distribution obtained from separate thermocatalytic processing of polymer and heavy oil residue over Taizhuzgen-based catalysts.

Catalyst	Gas Output, %	Yield of Liquid Products, %			Solid Residue, %	Loss, %
		0–180 °C	180–250 °C	250–320 °C		
Polymer	39.55	11.50	19.46	5.42	16.88	7.19
Heavy oil residue	22.65	8.65	12.33	6.72	45.81	3.84

Run conditions: T = 450 °C, p = 0.5 MPa, m (polymer):m (heavy oil residue) = 1:1, catalyst loading = 2 wt.% of the total feed mass.

As shown in Figure 8, the main part of the liquid products is alkanes, with a maximum content of about 62.49% when using 2% W@Taizhuzgen catalyst in the temperature range of 250–320 °C. The simultaneous presence of aromatic compounds, cycloalkenes, cycloalkanes and isoalkanes indicates that the reaction system contains aromatization, cyclization and cracking pathways, in accordance with the expected reaction mechanisms.

The formation of certain hydrocarbon groups was found to be strongly dependent on both the catalyst type and the reaction temperature. In particular, the 2% Mo@Taizhuzgen catalyst promoted the formation of aromatic compounds, reaching 27.13% at high temperature conditions (250–320 °C). In contrast, the tungsten-based catalyst showed significantly higher selectivity towards alkanes. These observations indicate significant differences in catalyst selectivity and highlight the important role of the active metal in achieving the desired amount of hydrocarbon in the reaction. Overall, the results indicate that metal selection and thermal conditions are the main factors affecting the product distribution during catalytic conversion. Overall, the characterization analysis and catalytic tests show

that the addition of Mo and W to Taizhuzgen zeolite significantly affects the structural and chemical properties, which in turn determine the performance of the catalyst. The Mo-modified catalyst exhibits high surface area, large mesoporosity, and improved active site accessibility, which contributes to the formation of aromatic-rich liquid fractions, while the W-modified catalyst exhibits increased selectivity toward alkanes. The thermal stability, preserved crystallinity, and the presence of acidic sites on both catalysts provide efficient mass transfer and active site accessibility, demonstrating the important role of metal species, porosity, and surface acidity in controlling product distribution and selectivity in the thermal-catalytic conversion of polymeric heavy oil blends.

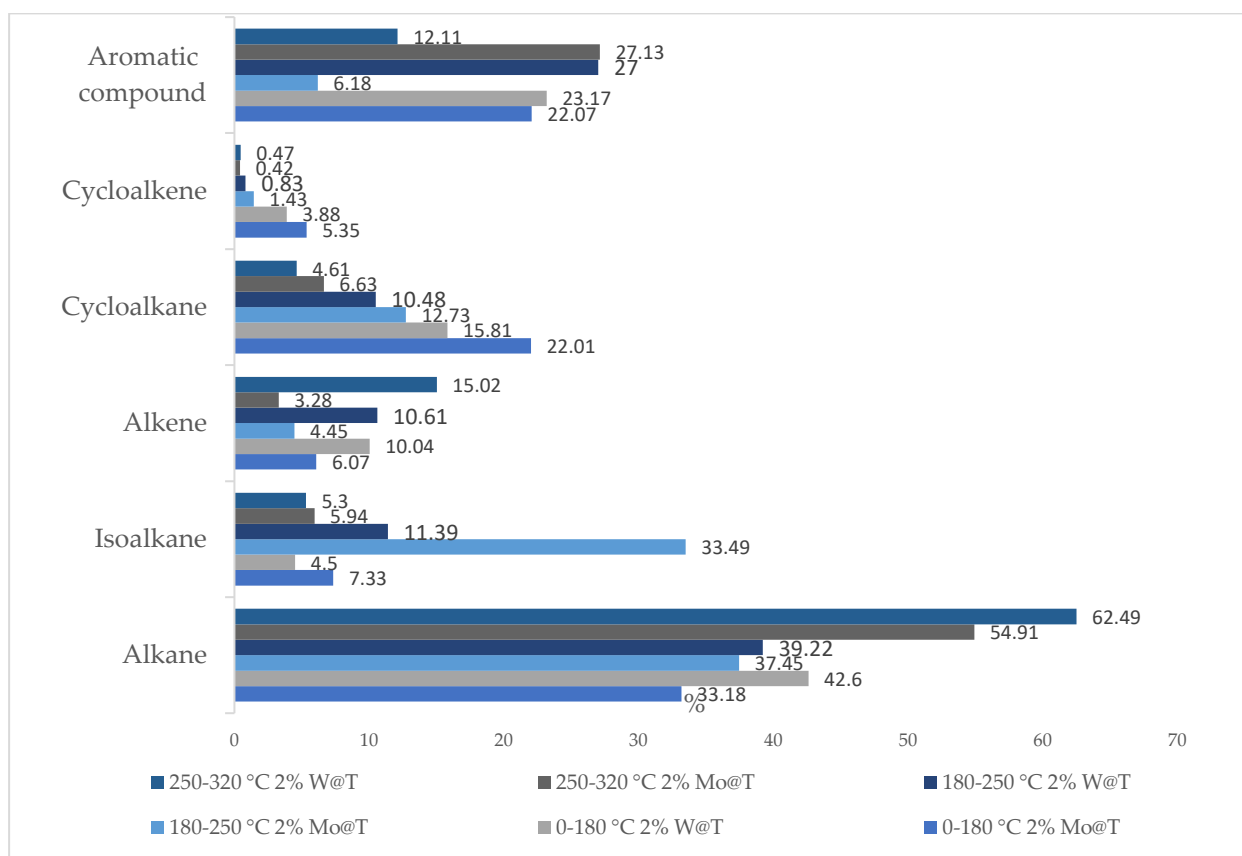


Figure 8. Chemical composition of liquid fractions obtained in relation to 2% Mo@Taizhuzgen and 2% W@Taizhuzgen catalysts.

GC–MS results showed that they were mainly composed of paraffinic, isoparaffinic, naphthenic, and aromatic hydrocarbons, which are typical of traditional petroleum fuels. The W-modified catalyst enhanced the formation of alkanes suitable for diesel and gas oil fractions, while the Mo-modified catalyst enhanced the formation of aromatic compounds suitable for gasoline fractions and petrochemical feedstocks.

Figure 9 shows a schematic diagram of the thermocatalytic treatment of polymer waste based on Mo and W-modified Taizhuzgen zeolite. The polymer and heavy oil waste undergo cracking, cyclization, aromatization, and isomerization reactions in the mesoporous structure of the zeolite. Mo compounds provide the formation of aromatic fractions (for gasoline and petrochemical feedstocks), while W compounds provide the increase in alkanes and diesel/gas oil fractions. The final products are separated by fractional distillation into gasoline (0–180 °C), diesel (180–250 °C), and heavy gas oil (250–320 °C) fractions, which confirms the ability of the catalysts to effectively convert polymer waste into useful hydrocarbon components.

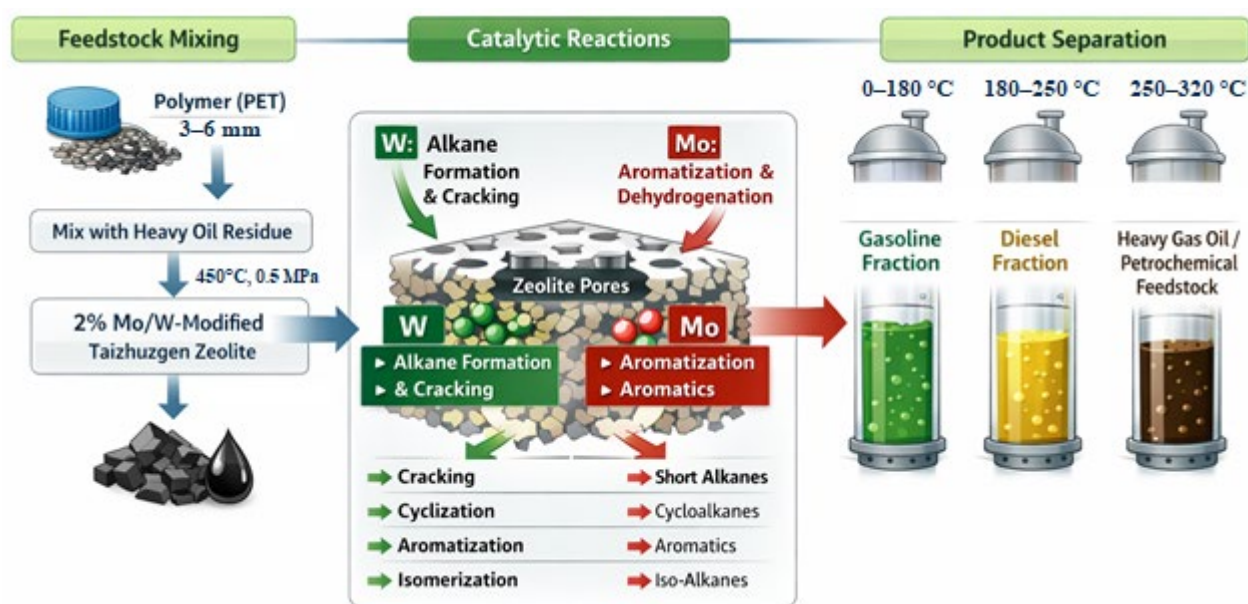


Figure 9. Schematic illustration of the thermocatalytic conversion of polymer waste over 2% Mo@Taizhuzgen and 2% W@Taizhuzgen catalysts into gasoline-, diesel-, and heavy gas oil-range fractions.

For comparison, the catalytic performance of the developed Mo and W modified Taizhuzgen zeolite was evaluated against representative zeolite catalysts commonly reported in the literature for plastic hydrocracking, such as ZSM-5 and Y-type zeolites.

As shown in Table 4, several synthetic zeolite catalysts reported in the literature have demonstrated high activities in the catalytic conversion of plastics to liquid hydrocarbon fuels under various conditions. For instance PdAg/HZSM-5 catalysts have achieved liquid hydrocarbon yields of ~69% under relatively mild temperatures, and hierarchical Y-type zeolites have delivered high conversion and selectivity to gasoline- and diesel-range products [41,42]. In contrast, the Mo- and W-modified natural Taizhuzgen catalysts developed in this work show competitive liquid fuel yields (~60 wt.%) without the need for external hydrogen and under significantly lower pressure. This comparison highlights the practical advantages of Taizhuzgen-based natural zeolite catalysts, including simpler activation procedures, reduced process severity, and lower catalyst cost, while maintaining efficient polymer waste conversion.

Table 4. Comparison of Taizhuzgen-based catalysts with representative zeolite catalysts reported in the literature for plastic hydrocracking.

Catalyst Type	Zeolite Nature	External H ₂	Typical Conditions	Liquid Fuel Yield, wt. %	Main Advantages
PdAg/HZSM-5	Synthetic, MFI	+	230 °C	~69.18	High liquid yield under mild T with bimetallic metal
Hierarchical Y	Synthetic, MFI	+	280 °C	~90.8	High conversion and fuel selectivity
2% Mo@Taizhuzgen	Natural, heulandite-clinoptilolite	—	450 °C, 0.5 MPa	61.56	Acid-free activation, competitive yield, low cost
2% W@Taizhuzgen	Natural, heulandite-clinoptilolite	—	450 °C, 0.5 MPa	60.4	Mild conditions, alkane-rich products

3. Experimental Part

3.1. Catalyst Preparations

Natural zeolite obtained from the Taizhuzgen deposit, located in the Tarbagatai District (administrative center: Akzhar, East Kazakhstan Region, Kazakhstan), was mechanically ground in a mortar and sieved to obtain a particle size fraction below 0.25 mm. Ion exchange was carried out to convert the zeolite into its ammonium form. For this purpose, 15 g of the sieved zeolite was mixed with 350 mL of a 1.0 M aqueous NH_4Cl solution, prepared by dissolving 53.5 g of NH_4Cl in 1000 mL of distilled water. The suspension was placed in a round-bottom flask and treated in a water bath at 80 °C under continuous mechanical stirring for 6 h. The ion exchange procedure was repeated three times using a fresh NH_4Cl solution each cycle, resulting in a total exchange time of 18 h. After each cycle, the solid was separated by filtration. Following ion exchange, the zeolite was thoroughly washed with distilled water to remove residual chloride ions. The completeness of chloride removal was monitored using an AgNO_3 test, and washing was continued until no white AgCl precipitate was observed. The NH_4^+ form zeolite was dried at 100 °C to constant weight and subsequently calcined in a muffle furnace at 450 °C for 4 h with a heating rate of 10 °C min^{-1} in static air to obtain the H-form zeolite.

The powdered H-form zeolite was promoted with molybdenum or tungsten using the incipient wetness impregnation method. Aqueous solutions of ammonium heptamolybdate $((\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O})$ and ammonium metatungstate $((\text{NH}_4)_5\text{H}_5[\text{H}_2(\text{WO}_4)_6]\cdot \text{H}_2\text{O})$ were used as metal precursors. The amount of precursor was calculated to achieve a metal loading of 2 wt.% relative to the catalyst mass. Impregnation was performed under continuous stirring at 80 °C until a homogeneous paste-like mass was obtained. The impregnated samples were dried at 100 °C for 2 h and used for further characterization and catalytic experiments.

The catalysts were not pre-treated with hydrogen prior to catalytic testing. They were used after ion exchange, drying, and calcination in air at 450 °C. The active form of the catalyst was formed during the reaction through the interaction of Mo and W oxides with the products of the thermal catalytic decomposition of the feedstock. The catalyst was used as a powder with a particle size of less than 0.25 mm, obtained by mechanical grinding and sieve analysis. The particles were uniformly mixed with the feedstock (polymer and fuel oil) before loading into the reactor, ensuring uniform distribution of the catalyst throughout the reaction mixture.

3.2. Catalyst Characterizations

The natural zeolite used in this study was obtained from the Taizhuzgen deposit (Kazakhstan) and is predominantly classified as a heulandite–clinoptilolite-type zeolite, which is typical for this geological formation. The elastic (Young's) modulus was not determined, as mechanical properties were beyond the scope of this work and do not directly affect catalytic performance under the applied conditions. The crystallinity of the zeolite was confirmed by XRD analysis, which shows that the crystalline framework is preserved after activation and Mo or W modification. The acidity of the zeolite was evaluated qualitatively by FTIR spectroscopy, indicating the presence of both Brønsted and Lewis acid sites.

The morphology of natural zeolite Taizhuzgen and composites on their basis were investigated in solution of low vacuum scanning electronic microscope. The accelerating voltage of the electronic optics varied from 200 V to 30 kV. The resolution in the low vacuum mode was 10–130 Pa. The elemental compositions of composite zeolite catalysts were determined using a low-vacuum scanning electron microscope, which is equipped with an energy-dispersed x-ray spectrometer and detector.

Specific surfaces of catalyst samples were carried out on a specific surface analyzer using the low-temperature nitrogen adsorption method by the BET method.

The powder XRD analysis using a Rigaku Ultima IV diffractometer (Rigaku Corporation, Tokyo, Japan) equipped with a Cu K α ($\lambda = 0.15406$ nm) radiation source was performed to determine the crystalline nature of the materials. XPS analysis was made by a SPECS EA300 instrument (SPECS GmbH, Berlin, Germany). The XPS analysis data were corrected for charge shifting using standard C 1s binding energy of 284.5 eV. FTIR spectrum and DRIFTS analysis was performed with a JASCO FT-IR/4700 spectrometer (JASCO, Tokyo, Japan).

The composition of the fractions was determined using the Agilent 7890A/5975C gas chromatographic mass spectrometer (Agilent Technologies, Santa Clara, CA, USA). In the chromatographic analysis, helium gas was used as the mobile phase (grade A, the volume fraction of helium is not less than 99.995%). The mass spectra were obtained in the Scan mode. Chromatographic separation was performed on a capillary column HP-5MS, column length—30 m, internal diameter is 0.25 mm, fixed phase-dimethylpolysiloxane 95%, methylphenylpolysiloxane 5%. The thickness of the fixed phase layer is 0.25 microns.

3.3. Activity Tests

Hydrocracking experiments were conducted in a laboratory setup (Figure 10) operating in batch mode at a pressure of 0.5 MPa and a temperature of up to 450 °C with continuous stirring for 15 min. The setup consisted of a stainless steel reactor (X18N10T) with a working volume of 150 mL. The reactor was heated by an AC electric heater controlled by a transformer and an ammeter. The temperature inside the reactor was monitored using a chromel–alumel thermocouple. Temperature readings were recorded using a KSP-4 instrument calibrated using standard reference points corresponding to the boiling point of water (100 °C) and the melting points of tin (232 °C), lead (327 °C), and zinc (427 °C). The operating pressure in the hydrogenation setup was generated using nitrogen and measured with a pressure gauge. A mixture consisting of polymer feedstock (7 g), catalyst (0.28 g), and a paste-forming agent (7 g) was loaded into the reactor. After sealing, the reactor was leak-tested, purged with nitrogen, and pressurized to 0.5–0.6 MPa. The heating system was then turned on. Plastic bottle caps produced by Bericap Kazakhstan LLP (collected from beverages produced by Coca-Cola Almaty Bottlers LLP) were used as the polymer feedstock. The plastic was crushed to a particle size of 3–6 mm. The paste-forming agent was a heavy residue obtained from crude oil from the Kumkol oil field after distillation to 350 °C. During the hydrogenation process, pressure changes were continuously monitored using a pressure gauge, reflecting temperature increases, gas evolution, and the presence of volatile components. Heating was stopped 30–40 min after the start of the process, when a decrease in pressure was observed, and the system was allowed to cool to room temperature. The gaseous products formed during the reaction were collected in a gasometer filled with a saturated NaCl solution. The volume of gas formed was determined by the pressure difference. The liquid products were then distilled and separated into fractions with boiling ranges of 0–180 °C, 180–250 °C, and 250–320 °C using the same setup (Figure 10). For this purpose, a condenser with a receiver was connected to the reactor outlet after cooling to room temperature. During distillation, the reactor was reheated, and the corresponding fractions were collected. After completion of the experiment and cooling of the system, the solid residue was removed from the reactor. Each experiment was performed three times under identical conditions. The deviation between experimental results did not exceed 2%.

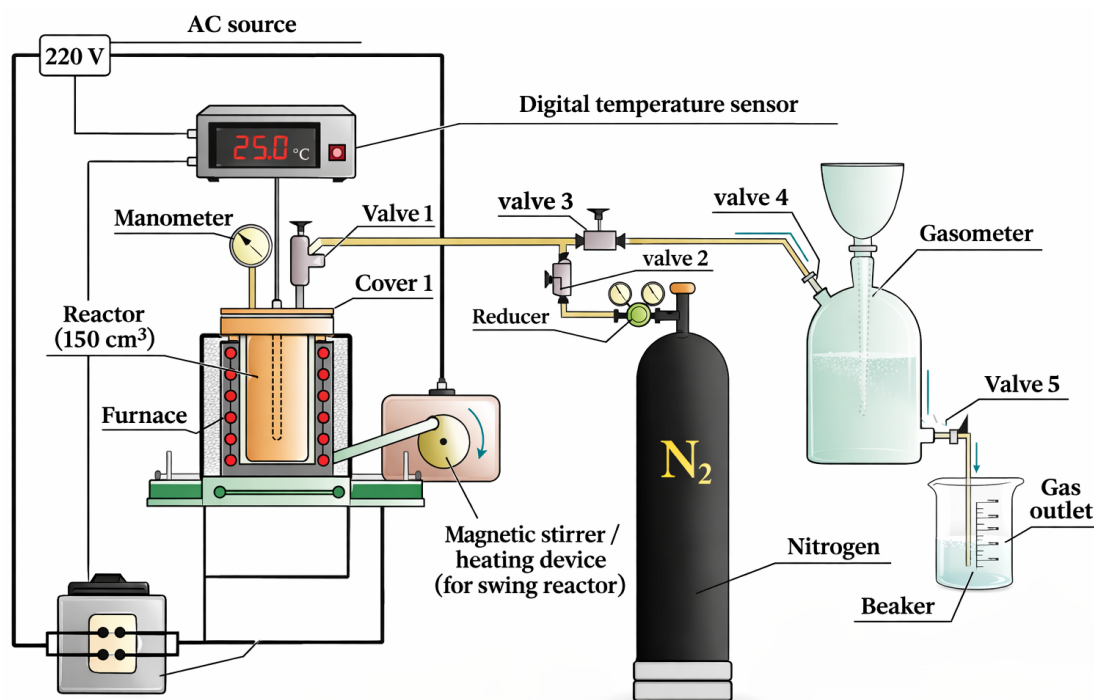


Figure 10. Scheme of the installation used for catalytic hydrogenation of polymer waste.

In the presented study, molecular hydrogen was not supplied externally. The process took place under thermal catalytic processing conditions in an inert environment, and the hydrogen involved in the hydrogenation reactions was generated through the thermal decomposition of heavy petroleum residues. Therefore, there was no external hydrogen consumption, further emphasizing the resource-saving nature of the proposed approach.

4. Conclusions

Composite catalysts based on natural Taizhuzgen zeolite modified with 2 wt.% molybdenum (2% molybdenum in Taizhuzgen) and tungsten (2% molybdenum in Taizhuzgen) were successfully synthesized using an acid-free activation method and tested for thermal catalytic hydrogenation of polymer waste in the presence of heavy oil residues. Physico-chemical characterization using BET, SEM, XRD, XPS, and FTIR confirmed that the addition of Mo(VI)/Mo(IV) and W(VI) compounds preserves the crystalline structure of the zeolite, while creating mesoporous materials with increased surface area, pore volume, and accessible active centers. SEM revealed a heterogeneous but well-developed porous morphology, X-ray diffraction confirmed stable crystallinity with small peak shifts consistent with metal oxides, X-ray diffraction confirmed the oxidation state of Mo⁴⁺/Mo⁶⁺ and W⁶⁺, and FTIR revealed the presence of stable acidic sites required for catalytic activity. Catalytic testing showed that 2% molybdenum, especially in the high-boiling fraction (250–320 °C), gave the highest total liquid yield of 61.56%, and 2% molybdenum with high selectivity towards alkanes was 60.40%. GC-MS analysis showed that the thermal catalytic process proceeds through cracking, cyclization, isomerization, aromatization and dehydrogenation, resulting in hydrocarbon fractions rich in paraffinic, naphthenic and aromatic compounds. These results indicate that the type of metal modifier, mesoporousness and surface acidity together determine the product distribution and selectivity, and that Taizhuzgen-based Mo-W composite catalysts are efficient, thermally stable and suitable for the resource-saving conversion of polymer waste and heavy oil residues into valuable hydrocarbon fuel fractions under optimal conditions at 450 °C, 0.5 MPa pressure and 2 wt.% catalytic loading.

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