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Microwave Aquathermolysis of Oil Shales: Enhancement of Light Hydrocarbon Yield and Modification of Gaseous Product Composition

The growing global demand for energy and the depletion of conventional light hydrocarbon resources necessitate the development of alternative and hard-to-recover feedstocks, among which oil shales represent a strategically important resource due to their vast reserves. In this study, the efficiency of conventional microwave thermolysis in an inert atmosphere is compared with an innovative microwave aquathermolysis approach employing activated steam for oil shale samples from three Azerbaijani deposits (Diyally, Guba, and Jangichay). The experiments were conducted under microwave irradiation (2.45 GHz, 600–800 W) at 300–400 °C with a treatment duration of 20 minutes, ensuring rapid and uniform heating. Microwave aquathermolysis was shown to enhance heat and mass transfer, promoting deeper degassing and partial kerogen conversion. Steam activation led to an increased yield of light hydrocarbons, a marked reduction of heavy resins and asphaltenes, and significant changes in gaseous product composition. Gas chromatographic and SARA analyses confirmed accelerated thermochemical reactions, suppression of secondary coking, steam-assisted conversion of carbon oxides, and partial hydrogenation of unsaturated compounds. Overall, the results indicate that microwave aquathermolysis is more efficient and energy-saving than conventional microwave thermolysis, demonstrating strong potential for processing Azerbaijani oil shales and other kerogen-rich unconventional resources.

Keywords: oil shale, microwave aquathermolysis, heat–mass transfer, enhanced light fraction yield, energy–efficient processing, microwave irradiation, kerogen conversion, steam activation, gaseous product modification

Introduction

The depletion of conventional oil reserves poses a challenge for the energy and chemical industries to identify alternative sources of hydrocarbon feedstock [1–10]. In this context, oil shales acquire particular importance, as their global reserves far exceed the combined resources of oil, natural gas, and coal [11–13].

The decline in conventional hydrocarbon production in Azerbaijan has also increased attention to heavy hydrocarbons, including oil shales, discovered in the eastern regions of the country and considered a secondary hydrocarbon resource [14, 15]. These shales belong to a stratigraphic sequence ranging from the Middle

Eocene to the Upper Miocene [16, 17]. Their thickness reaches 400–500 m, with individual layers measuring 10–20 m or more [18, 19].

Tectonic activity in the collision zone of the African and Arabian plates in Eastern Azerbaijan has led to the exposure of oil shales at the surface, both as natural outcrops and as mud volcano ejecta [17–19]. The distribution of mud volcanism provides favorable conditions for obtaining valuable information on the internal structure and geochemical–mineralogical characteristics of the volcanic system, as well as on the extent, composition, and distribution of oil shale layers in the subsurface, which can be traced over considerable distances at the surface [20–27].

Oil shales widespread on the land of Eastern Azerbaijan are characterized by a high organic matter content (up to 24 wt% total organic carbon — TOC) and low thermal maturity [17]. This necessitates the use of innovative methods for the production of synthetic hydrocarbons, employing technologies such as in–situ pyrolysis and surface processing [18].

Conventional processing methods based on convective heating (e.g., electric heating, heat carriers) require prolonged exposure, which induces secondary coking reactions, reduces liquid product yields, and promotes the formation of carbon oxides (CO and CO_2) [12, 28–31]. The low reactivity of kerogen and the high ash content of shales make the use of alternative energy–based methods reasonable, such as microwave irradiation, which provides rapid heating and uniform heat distribution [32]. In recent years, the conventional (dry) microwave thermolysis of oil shales has been extensively studied globally due to its ability to accelerate heating rates and alter the fractional yield of retorting products compared to traditional convective heating [33, 34]. Comprehensive reviews and three-dimensional numerical simulations have confirmed that microwave application effectively enhances hydrocarbon desorption and volumetric heating within the shale matrix [35, 36]. In this broader global context, previous studies have shown that microwave thermolysis of Azerbaijani shale samples is highly effective, confirming the promise of this approach [37]. However, conventional dry microwave processing still faces critical challenges, such as secondary coking and the limited hydrogen availability within the kerogen itself, which restricts the maximum achievable yield of light liquid hydrocarbons. Recently, independent studies exposing oil shales to high-temperature water vapor have shown promising results in improving micro-fracture evolution and permeability [38, 39]. By combining the rapid heating benefits of microwaves with the chemical reactivity of steam, in the present study, results are presented for a modified microwave aquathermolysis method, based on the treatment of samples with steam activated by microwave radiation, which enhances degassing intensity, increases the yield of light hydrocarbons, and reduces coking.

Experimental

For this study, oil shale samples were selected from the stratigraphic sequence ranging from the Middle Eocene to the Upper Miocene, obtained from three prospective deposits in Eastern Azerbaijan: Diyally (Middle Eocene), Guba (Middle–Upper Miocene), and Jangichay (Middle Eocene) [18, 40, 41]. The samples are characterized by high TOC contents: Diyally — 24 %, Guba — 20 %, Jangichay — 18 % (2). Petrographic and pyrolytic analyses showed that the organic matter in these shales is predominantly type II kerogen, formed in marine conditions from algal detritus, indicating a high potential for liquid hydrocarbon generation [2, 18].

The mineralogy of the rocks was analyzed via X-ray diffraction using a Rigaku Miniflex X-ray diffractometer (Rigaku, Japan). The measurements were performed to identify the crystalline phases and provide a semi-quantitative assessment of the inorganic matrix, which plays a vital role in the samples' interaction with microwave radiation. Table 1 presents the mineralogical composition of the studied oil shale samples.

Table 1

Semi-quantitative mineralogical composition of the studied oil shale samples (wt. %)

Sample	Clay minerals (illite, montmorillonite, kaolinite, chlorite)	Silicates (quartz, feldspar)	Carbonates (calcite, Mg-calcite)	Sulfur-bearing minerals (pyrite, jarozite)	Iron Oxides (hematite, rutile)
Diyally	33	44	12	2	9
Guba	45	40	8	1.8	5
Jangichay	43	34	8	15	0

Microwave irradiation experiments were conducted using a setup based on a Panasonic EM–G5593V laboratory microwave oven with a 23 L cavity. The magnetron output power ranged from 200 to 800 W at an operating frequency of 2450 MHz. Under the applied optimal microwave power settings (600–800 W), the sample temperature reached 500–600 °C over the 20-minute exposure period, which corresponds to an average actual heating rate of approximately 25–30 °C/min. Sample and steam line temperatures were monitored with a CEM DT–8858 infrared pyrometer (range 50–1300 °C), which recorded the surface temperature of the oil shale samples through the microwave-transparent quartz vessel. Shale samples with a particle size of 1–5 mm were placed in a 100 mL quartz reaction vessel, which is transparent to microwaves and equipped with inlet and gas outlet lines. To prevent oxidative reactions of desorbed products, the inlet line was saturated with steam delivered from a heated distillation water evaporator at a flow rate of 0.05–0.3 mol/min. To avoid local overheating of the samples, a water–shunting vessel with circulating cooling fluid was employed. The setup included a condensation and collection system for liquid thermolysis products. Gaseous compounds were collected in a coil trap cooled in a Dewar vessel filled with solid CO_2.

The liquid fraction of the desorbed hydrocarbons was separated using liquid adsorption chromatography (Saturates, Aromatics, Resins, Asphaltenes — SARA) with a column packed with KSK–grade silica gel, allowing the isolation of four compound groups: saturated hydrocarbons, aromatic compounds, resins, and asphaltenes [42]. The separation procedure followed ASTM D4124–09 standards.

Qualitative and quantitative analysis of the gaseous phase was performed using an LKhM-8MD gas chromatograph (column length 3.6 m, internal diameter 3 mm, stationary phase “Porapak QS”) under programmable thermostat heating from 30 to 650 °C at a rate of 3–5 °C/min.

All quantitative experiments, including the SARA fractionation and gas chromatographic analyses, were performed in triplicate (n = 3) to ensure reproducibility. The experimental data are presented as the mean values ± standard deviation (SD).

Results and Discussion

The study of the heating dynamics of oil shale samples under aquathermolysis conditions, with varying microwave power in the oven cavity, revealed differences in their heating rates (Figure 1). This is likely associated with variations in dielectric losses, which are determined by the inorganic fraction of the shales, dominating over the organic matter (kerogen), and whose composition depends on their genesis [18]. As illustrated in Figure 1, while an applied power of 600 W is sufficient for all samples to surpass 400 °C (the onset of active thermolysis), the Diyally deposit sample required exposure at up to 800 W to reach the higher optimal temperature range of 450–500 °C. In contrast, samples from the Jangichay and Guba deposits more efficiently achieved these higher temperatures at only 600 W for the same duration.

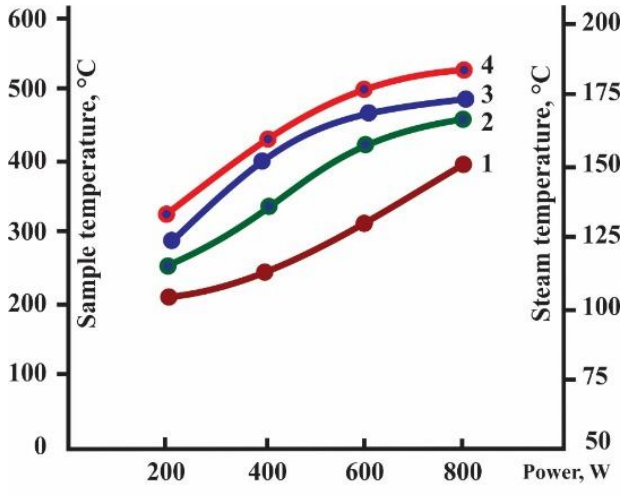

Figure 1. Temperature rise dynamics of the supplied steam (Y-axis, right) and oil shale samples (Y-axis, left) as a function of applied microwave power (X-axis) for the Diyally (2), Guba (3), and Jangichay (4) deposits. Exposure conditions: sample mass 50 g; duration 20 min; steam flow rate 0.2 mol/min; magnetron operating frequency 2450 MHz. The presented data points represent the mean values of three independent experiments (n = 3)

The selection of the 400–500 °C temperature range is fundamental for optimizing the yield of liquid products while minimizing secondary gasification. While some industrial processes targeting total gasification operate at 600 °C and above, such high temperatures promote the intensive secondary cracking of synthesized hydrocarbons and the undesirable decomposition of the inorganic carbonate matrix. In our microwave-assisted process, the presence of localized dielectric heating sites (e.g., pyrite and jarosite) ensures efficient kerogen transformation within the moderate 400–500 °C range, preserving the integrity of the liquid fractions.

The efficiency of microwave irradiation on heterogeneous materials, such as oil shales, is determined by their ability to absorb electromagnetic energy and convert it into heat [43]. Regardless of the electromagnetic radiation parameters, the measured result corresponds to the average power loss:

$$\Delta P_x = \frac{c \cdot m \cdot \Delta T}{0.24\tau} \text{ (J/s)},$$

where: 0.24 — the mechanical equivalent of heat; m — mass of the sample (g); c — specific heat capacity of the sample (kJ/kg·K); ΔT — measured temperature increase of the sample (K); τ — exposure time in the microwave oven (s).

Figure 2 shows that the dielectric losses of microwave radiation by oil shale samples from the three deposits are relatively high, facilitating the efficient attainment of the temperature required for microwave thermolysis. Power losses during aquathermolysis were found to exceed those in the absence of steam, likely due to the formation of adsorbed moisture layers on the shale surfaces, which enhance particle polarization [44].

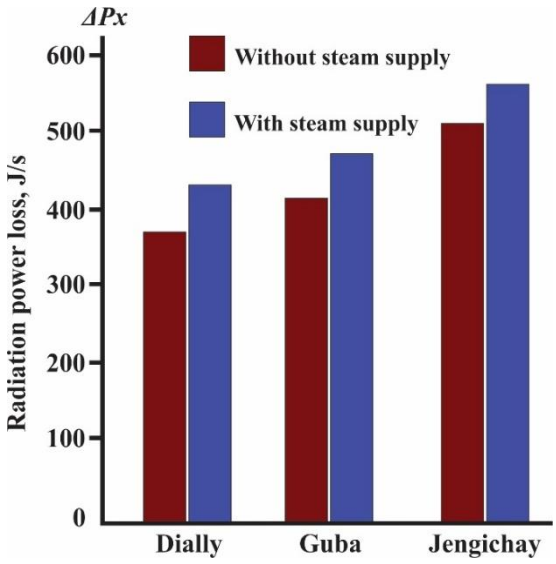

Figure 2. Comparative microwave power losses (ΔP_x, J/s) for different oil shale deposits under inert and steam-supplied conditions. Exposure conditions: sample mass 50 g; magnetron power 600 W; operating frequency 2450 MHz; exposure time 10 min; steam flow rate 0.3 mol/min. The presented data points represent the mean values of three independent experiments (n = 3)

After the exposure in the oven cavity, a relatively large variation in weight loss among the samples was observed. The maximum weight loss, due to degassing of organic matter and removal of volatile components, occurred for the Jangichay sample. It should also be noted that the highest ratio of desorbed liquid phase to gaseous phase, characteristic of this sample, is determined by the geochemical conditions of its formation (e.g., the kerogen composition, predominantly derived from marine lipid matter, relatively low influence of reducing conditions, and a certain degree of geothermal maturity) [18]. Considering this, from a practical standpoint, it is reasonable to conduct further microwave thermolysis experiments using the Jangichay deposit sample. While the overall multi-stage kinetic trends for the Diyally and Guba samples were broadly similar, plotting the data for all three deposits on a single graph would have severely cluttered the visualization. Therefore, the Jangichay shale was selected as the most representative optimal model to visually illustrate the comparative weight loss dynamics between inert thermolysis and aquathermolysis, as its mass-loss transitions are the most pronounced and visually distinct.

As evident from the aquathermolysis results of the Jangichay shale, weight loss during the desorption of hydrocarbon components, present both in the kerogen and adsorbed by the inorganic matrix of the sample [18], occurs in stages, as in an inert atmosphere (Fig. 3). Three main temperature regions of weight loss are observed. It is important to note that oil shale thermolysis is a complex process involving parallel and partially overlapping thermochemical transformations. Therefore, while dominant temperature intervals can be clearly identified for each sequence, precise and strict absolute boundaries between primary gas desorption, liquid phase evolution, and the coking stage cannot be rigidly defined.

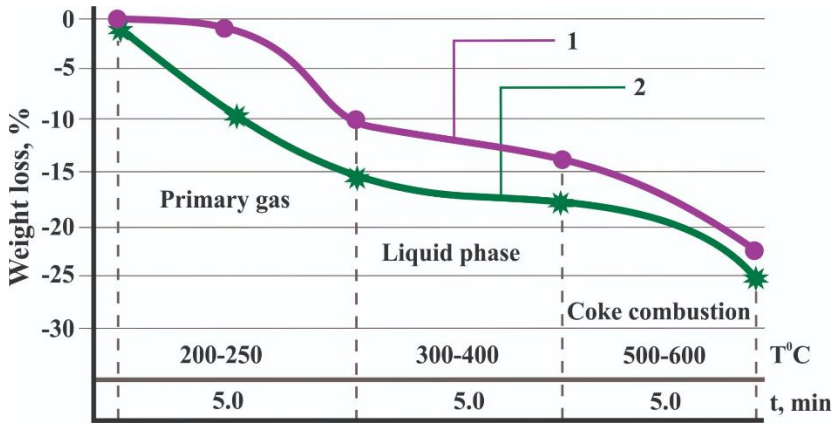

Figure 3. Comparison of the weight loss dynamics of a shale sample from the Jangichay deposit during microwave thermolysis in an inert atmosphere (1) and microwave aquathermolysis (2). Conditions: magnetron power 800 W, sample mass 50 g (fraction 3–5 mm), steam flow rate 0.5 mol/min. The presented data points represent the mean values of three independent experiments (n = 3)

In the first exposure region, during 5 minutes of irradiation at a temperature of ~200–250 °C, desorption of primary gaseous components occurs [15]. It was observed that under aquathermolysis conditions, with the activating effect of steam, desorption of gaseous components proceeds more intensively. Continued exposure of the samples for the next 5 minutes raises the temperature to ~300–400 °C, during which condensation of the heavier hydrocarbon components is observed in the trap. In the final stage of microwave treatment, the sample temperature rises to 600 °C and above. In this temperature range, desorption of gaseous products is again observed, associated with the combustion of coke–like residues formed during thermolysis.

Comparison of the weight–loss dynamics of the Jangichay shale sample during thermolysis in an inert nitrogen atmosphere and under aquathermolysis showed that weight change during the primary gas formation stage occurs more intensively under the influence of steam. In contrast, the intensity of weight loss associated with the release of the liquid fraction is slightly lower compared to the process conducted in an inert atmosphere.

The results of the SARA analysis of thermally desorbed high-molecular-weight hydrocarbon products (Table 2) show that under microwave aquathermolysis, the yield of saturated and aromatic hydrocarbons increases, while the content of high-molecular-weight resins and asphaltenes decreases. The observed decrease in resins and simultaneous increase in saturates are attributed to the hydrogen-assisted cracking (hydrocracking) of heavy organic macromolecules. The in-situ generated hydrogen, derived from steam activation, effectively stabilizes thermally generated organic radicals and promotes the scission of long-chain hydrocarbons into lighter, saturated fractions, thereby preventing secondary condensation into coke. It is important to clarify that “steam activation” under 2.45 GHz microwave irradiation does not imply the direct photolytic generation of free H• or OH• radicals, as the radiation is non-ionizing. Instead, activation occurs through intense dipole polarization and rotational excitation of the water molecules. When this rotationally excited steam interacts with micro-scale “hot spots”, generated by the strongly microwave-absorbing inorganic shale matrix, it vigorously drives localized hydrogen-generating pathways. The primary mechanism is the steam-carbon reaction ($C + H_2O \rightarrow CO + H_2$) occurring at these extreme-temperature solid-gas interfaces. The newly generated molecular hydrogen, potentially catalyzed by inherent shale minerals such as pyrite, stabilizes the thermally cracked organic radicals. This in situ generated hydrogen facilitates partial hydrogenation and completely suppresses secondary condensation and coking, as directly evidenced by the increased yield of saturated and aromatic fractions [45].

Table 2

Content of high–molecular–weight organic products in shale samples desorbed under microwave irradiation during thermolysis in an inert atmosphere and aquathermolysis. Magnetron power 800 W; sample mass 50 g; exposure time in the oven cavity 20 min; steam flow rate 0.5 mol/min

Sample	Content of organic products (% by mass)			
	Saturate	Aromatic	Resins	Asphaltenes
Microwave thermolysis in an inert atmosphere				
Diyally	2.2 ± 0.4	6.5 ± 0.4	8.6 ± 0.3	3.2 ± 0.4
Guba	3.2 ± 0.3	5.2 ± 0.4	8.7 ± 0.4	3.5 ± 0.5
Jangichay	2.7 ± 0.3	8.5 ± 0.5	8.8 ± 0.5	3.1 ± 0.4
Microwave aquathermolysis				
Diyally	3.4 ± 0.5	7.0 ± 0.4	7.4 ± 0.4	2.8 ± 0.5
Guba	3.8 ± 0.5	5.7 ± 0.4	7.2 ± 0.4	2.5 ± 0.5
Jangichay	3.6 ± 0.5	9.4 ± 0.4	6.9 ± 0.4	2.3 ± 0.5
Note: Data are presented as mean ± standard deviation (n = 3).				

Gas chromatographic analysis of the gas phase desorbed at the initial stage of microwave treatment (Table 3) also revealed significant differences in the thermolysis products under an inert atmosphere versus aquathermolysis. Treatment in a steam flow leads to an increase in carbon dioxide and hydrogen sulfide yields, accompanied by a simultaneous decrease in carbon monoxide content. Methane yield remains stable, while hydrogen concentration shows a slight increase.

Table 3

Content of gaseous products in shale samples desorbed under microwave irradiation at 800 W; sample amount — 50 g; exposure time in the oven cavity — 20 min; steam flow rate — 0.5 mol/min

Sample	Content of gaseous products (% by mass)				
	CO	CO_2	H_2S	CH_4	H_2
Microwave thermolysis in an inert atmosphere					
Diyally	0.8 ± 0.1	1.6 ± 0.2	1.8 ± 0.2	2.2 ± 0.2	1.7 ± 0.1
Guba	0.6 ± 0.1	0.8 ± 0.1	1.3 ± 0.2	2.5 ± 0.2	1.5 ± 0.1
Jangichay	0.5 ± 0.1	0.4 ± 0.1	0.7 ± 0.1	3.1 ± 0.3	2.3 ± 0.2
Microwave aquathermolysis					
Diyally	0.7 ± 0.1	1.8 ± 0.2	2.0 ± 0.2	2.2 ± 0.2	1.8 ± 0.1
Guba	0.4 ± 0.1	0.9 ± 0.1	1.4 ± 0.2	2.5 ± 0.2	1.6 ± 0.1
Jangichay	0.3 ± 0.1	0.6 ± 0.1	0.9 ± 0.1	3.1 ± 0.3	2.5 ± 0.2
Note: Data are presented as mean ± standard deviation (n = 3).					

Interestingly, the mineralogical composition data presented in Table 1 serves as a critical foundation for interpreting these gaseous yields. The presence of sulfur-bearing minerals in all studied samples, specifically pyrite in Diyally and Guba, and a significant amount of jarosite (15 %) in Jangichay, correlates with the detection of H_2S in the gaseous phase (Table 3). This suggests that H_2S originates from a multi-component mechanism: the hydrothermal decomposition of both sulfide (pyrite) and sulfate (jarosite) minerals, alongside the intensified thermal cracking of sulfur-containing organic macromolecules (organosulfur) within the kerogen structure [46]. Furthermore, while Table 1 identifies the presence of carbonates in the shale matrix, their thermal decomposition typically requires temperatures well above 700 °C. Therefore, the significant amounts of CO_2 generated at our operating temperatures (400–500 °C) are primarily driven by the decarboxylation of the organic matter itself, rather than inorganic matrix decomposition.

The observed changes in the composition of gaseous products can be explained by the combined effect of activated steam on the progression of thermochemical processes. Steam activated by microwave irradiation intensifies the oxidative conversion of organic oxygen-containing functional groups and heavy fractions, manifested by an increased CO_2 yield. Simultaneously, the steam-mediated conversion of carbon monoxide (the water-gas shift reaction) shifts the equilibrium toward the formation of carbon dioxide and hydrogen [47]. The slight increase in hydrogen concentration (by 0.1–0.2 %) confirms the in-situ generation of hydro-

gen at the activated solid-gas interfaces, where it facilitates partial hydrogenation and suppresses secondary coking [48].

The contribution of the water-gas shift (WGS) reaction ($CO + H_2O \rightarrow CO_2 + H_2$) can be qualitatively differentiated from the oxidative conversion of coke residues by analyzing the stoichiometry of the carbon oxide changes. In our experiments (Table 3), the decrease in CO concentration (0.1–0.2 % by mass) aligns with the simultaneous increase in H_2, confirming the activity of the WGS pathway. However, the total increase in CO_2 yield often exceeds the stoichiometric equivalent of CO consumed, particularly in the Diyally and Jangichay samples. This indicates that the oxidative conversion of oxygen-containing organic functional groups and coke residues remains a primary source of CO_2 under microwave aquathermolysis conditions.

To evaluate the energy efficiency of the proposed method, the specific energy consumption (SEC) was calculated. The total energy input during the 20-minute exposure at 800 W was 0.267 kWh. The specific energy consumption per unit mass of the shale sample was determined using the relation:

$$SEC_{shale} = \frac{P \cdot \tau}{m} = \frac{0.8\ \text{kW} \cdot (20/60)\text{h}}{0.05\ \text{kg}}$$

This calculation directly justifies the energy density required to reach the target thermolysis temperatures within the microwave cavity, providing a quantitative basis for comparing the efficiency of the different processing regimes. For a 50 g shale sample, the specific energy consumption per mass of raw material (SEC shale) is 5.34 kWh/kg. However, the true efficiency is reflected in the energy required to produce a unit of the desired light hydrocarbon fraction (saturates and aromatics). As shown in Table 4, microwave aquathermolysis reduces the energy/product ratio for light fractions by approximately 14–16 % compared to inert thermolysis, confirming its superior energy efficiency.

Table 4

Energy efficiency and specific energy consumption (SEC) for the Jangichay shale sample

Parameter	Microwave thermolysis (Inert)	Microwave aquathermolysis
Total Energy Input (kWh)	0.267	0.267
Specific Energy (SEC shale), kWh/kg shale	5.34	5.34
Light Product Yield (Saturates + Aromatics), wt%	11.2	13.0
Energy/Product Ratio (SEC product), kWh/kg light product	47.7	41.1

Conclusions

The study demonstrated that microwave aquathermolysis is an effective approach for the thermochemical conversion of the organic fraction in oil shales. Treatment with steam activated by microwave irradiation provides rapid sample heating, enhances heat and mass transfer, and increases the degree of hydrocarbon desorption at 400–600 °C. Thermal analysis confirmed the staged nature of the process and a higher rate of gaseous and liquid product release compared to microwave thermolysis in an inert atmosphere.

Quantitative analysis confirmed the method's effectiveness: SARA data showed a consistent increase in the content of saturated and aromatic hydrocarbons (e.g., up to 1.2 % for saturates), accompanied by a noticeable decrease in resins and asphaltenes (e.g., up to 1.9 % for resins). In the gas phase, an increase in hydrogen and hydrogen sulfide yields up to 0.2 % was observed, while methane yield remained stable and carbon monoxide content decreased to 0.3 %.

Thus, microwave aquathermolysis provides a high degree of conversion of the organic fraction in shales, reduces the intensity of coking, and increases the yield of light hydrocarbon fractions in a short time, confirming its superior energy efficiency with a 14–16 % reduction in energy/product ratio compared to inert thermolysis.

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. CRediT: **Peri Aghaguseyn Muradova** — conceptualization, methodology, supervision, investigation, writing — original draft, writing — review & editing; **Yuriy Nikolayevich Litvishkov** — conceptualization, data curation, formal analysis, validation, writing — review & editing; **Elnur Eyvaz Baloglanov**, **Ruslan Vagif Akhundov** and **Elmar Surkhay Samedov** — investigation, data curation, visualization, resources, writing — review & editing; **Ulviyya Jeyhun Yolchuyeva** — methodology, investigation, data curation, visualization, writing — original draft.

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Conflicts of Interest

The authors declare no conflict of interest.

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