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INTEGRATED CROSS-REGIONAL ANALYSIS OF THERMAL REACTIVITY AND SPECTROSCOPIC-PHYSICOCHEMICAL CHARACTERISTICS OF CRUDE OILS

Ulviyya J. Yolchuyeva[*1,2,3], Rena A. Jafarova[1], Nazli A. Mehdiyeva[1,2], Ayaz M. Mammadov[1,4], Saida F. Ahmadbayova[1], Emin İ. Azizbayli[1], Elnur E. Baloglanov[5], Maliha B. Huseynova[1], Aynur Xuduyeva[6], Mohamed Alhayek[7], Ruslan V. Akhundov[5], Naila Q. Karinova[8]

[1]Institute of Petrochemical Processes of Ministry of Science and Education Republic of Azerbaijan, Baku, Azerbaijan
[2]Department of Chemical Engineering, School of Engineering and Applied Science, Khazar University, Baku, Azerbaijan
[3]Azerbaijan University of Architecture and Construction, Baku, Azerbaijan
[4]Sumgayit State University, Department of Chemistry, Sumgayit, Azerbaijan
[5]Institute of Geology and Geophysics, Ministry of Science and Education of the Republic of Azerbaijan, Baku, Azerbaijan
[6]Azerbaijan State Oil and Industry University, Baku, Azerbaijan
[7]Sivas Cumhuriyet University, Institute of Science and Technology, Department of Chemical Engineering Major, Sivas, Turkey
[8]Department of Pharmaceutical Toxicology and Chemistry, Azerbaijan Medical University, Baku, Azerbaijan

e-mail: u.jeyhunzade@gmail.com

Abstract: The energy potential and oxidative reactivity of high-density hydrocarbon systems are fundamentally linked to their thermal behavior under oxygen-rich environments. This investigation examines the thermo-oxidation dynamics of Kazakhstani (Korchagin) and Azerbaijani (Zaghli and Balakhani) petroleum samples using a synergistic approach of differential thermal analysis (DTA-TG) and infrared spectroscopy (FTIR). The results illustrate a multi-phase reaction sequence governed by a radical-chain pathway, encompassing the initial thermal-oxidative stage (LTO), molecular structural reorganization, and the subsequent high-temperature oxidation (HTO) phases. Korchagin crude exhibited significant early-stage reactivity between 155 and 476 °C, yielding an enthalpy of $\Delta H = 2443$ J g^{-1}. Conversely, the Zaghli sample demonstrated the highest energy-generation potential, reaching $\Delta H \approx 5093$ J g^{-1} in the 507-787 °C range, whereas Balakhani oil showed a consistent oxidation trend at 439-628 °C ($\Delta H = 2870$ J g^{-1}). FTIR validated the evolution of H_2O, CO_2, and CO, confirming the stepwise nature of the oxidation process. These findings highlight a distinct correlation between chemical composition and thermal output,

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emphasizing the promising utility of these regional heavy crudes for thermal enhanced oil recovery applications.
Keywords: Crude petroleum; staged thermal oxidation (LTO/HTO); DTA-TG analysis; IR spectroscopy; oxidation at low and high temperatures; heat effect; mass loss.

1. Introduction

Oil is one of the main natural raw materials of strategic importance in today's global infrastructure and power networks and is widely used in manufacturing indispensable derivatives such as motor spirits, jet fuels, middle distillates, heating oils, lubricating agents, as well as asphaltic substances and solid carbon residuals [1-4]. Compared to other fuel sources, the advantage of oil is characterized by the technological feasibility of its extraction, transportation, and processing [5, 6]. Fossil fuels still occupy a dominant position in the international energy matrix, and given the rising worldwide consumption requirements, the efficient utilization of oil resources acquires particular relevance [7-11]. In this context, especially the comprehensive investigation of heat-induced transformation pathways of dense petroleum systems with high flow resistance is of significant importance both from fundamental and applied perspectives [12, 13].

Petroleum represents an intricate, multi-constituent, and evolving matrix; beyond the diverse categories of hydrocarbons, it encompasses entrained gases, moisture, inorganic salts, and solid particulates. The physical and chemical attributes of the oil exhibit extensive variability based on its fractional and structural make-up, and rather than possessing a specific boiling point, they are defined by a wide distillation window. Key parameters such as gravity, flow limits, and flash point show a direct correlation with the paraffinic concentration and the relative abundance of volatile components [14-16]. Its high solubility in organic solvents and its ability to form stable emulsions in water directly affect its technological behavior in extraction, transportation, and processing processes.

Investigating the molecular makeup and heat-induced response of petroleum is crucial in terms of its genesis, geochemical transformations, and the optimization of industrial processing strategies. During the catagenesis stage, a complex chemical system is formed from the conversion of precursor organic materials, where specific biological markers and their derivatives play a fundamental role in clarifying these evolutionary pathways. The analysis of hydrocarbon composition enables the typological classification of oils; in particular, saturated hydrocarbons act as indicators of geochemical processes [17, 18].

To elucidate the reaction pathways and kinetic data associated with the heat-induced breakdown of petroleum, along with its distillates, the integrated use of thermo-analytical instrumentation, encompassing thermogravimetric analysis (TG) alongside differential thermal and scanning calorimetry (DTA and DSC), is a common practice [19-21]. According to the literature data, the thermal oxidation process comprises LTO and HTO stages, and these stages are characterized by heat release, mass loss, and combustion kinetics [22]. Various kinetic approaches indicate that the activation energy varies over a wide range and depends on the selected calculation model [23-25].

Lately, the thermophysical response of spent lubricants, pyrolytic oils from polymers, and bio-based oils has been explored using TG–DSC techniques to assess their enthalpic profiles, auto-ignition propensities, and kinetic variables [26, 27]. Under atmospheric conditions, the oxidative degradation of transformer, engine, and hydraulic fluids has shown mass reductions between 73% and 89%, with motor oil specifically exhibiting the maximum activation energy (110.5 kJ/mol) [28]. Such findings validate the precision of thermal analysis for assessing the stability and safety of these systems.

Escalating global power requirements and the progressive exhaustion of conventional, low-complexity reservoirs have heightened the strategic value of heavy hydrocarbon deposits [29, 30]. Effective strategies for upgrading dense crudes include thermochemical cracking, in-situ combustion,

supercritical water processes, and steam-based recovery methods [31]. While isoconversional models like OFW, Friedman, and KAS are frequently utilized for evaluating breakdown kinetics, the integration of Py-GC/MS offers supplementary data regarding the chemical profile of released vapor-phase products. Specifically, combined TG and Py-GC/MS assessments of Iranian crudes from Lavan and Koroosh indicated that their activation energy fluctuations span from 35.9 to 193.8 kJ mol^{-1} [32, 33].

However, most existing studies have been conducted at atmospheric pressure, which does not fully reflect real reservoir conditions. Intensive evaporation at low pressure distorts the oxidation mechanism and hinders the correct interpretation of thermal effects. Analyses performed under high-pressure conditions show that pressure significantly affects thermo-oxidation kinetics, heat release, and auto-ignition characteristics [34]. Under these conditions, since evaporation is minimized, the oxidation process can be modeled in closer accordance with actual reservoir conditions [35, 36].

During thermo-oxidative cracking (300–450°C), the decomposition of heavy components and coke formation occur in parallel; as a result, viscosity decreases and production is facilitated. Given that crude oil comprises a vast array of chemical entities, the oxidation process proceeds as a complex combination of parallel and consecutive reactions, and simplified kinetic approaches based on SARA fractions are applied in modeling these processes [56-58]. Techniques operating under elevated pressure and near-adiabatic conditions, including HP-DSC and ARC, provide a more robust estimation of self-ignition points, exothermic reaction timeframes, and fundamental kinetic constants (E_a, A_0) [37, 38].

Despite the substantial body of existing research regarding the thermo-oxidation of petroleum systems, there is a lack of rigorous, comparative investigations into the phased (LTO-HTO) thermal signatures of light versus heavy crudes across various reservoirs in an oxidative atmosphere. In particular, the temperature shift of exothermic maxima, the distribution of the heat effect, and the relationship between mass loss and the physicochemical parameters of oil (density, viscosity, molecular weight, acid number) have not been evaluated in an integrated manner. Furthermore, experimental data confirming the parallel progression of oxidation and pyrolysis processes as derived from the analysis of volatile and gaseous effluents remains scarce and inconclusive.

The central aim of this study is to carry out a side‑by‑side investigation into thermal‑oxidative signatures of low and high-density crude oils sourced from various reservoirs within an oxygen-enriched atmosphere. The study focuses on assessing the enthalpic shifts and weight fluctuations during the LTO and HTO phases, while examining the link between these variables and the intrinsic oil properties. Simultaneously, the work seeks to empirically validate the sequential nature of oxidative and pyrolytic pathways through the infrared spectroscopy of gaseous effluents.

In the current investigation, the thermal response of diverse crude oils under oxidative conditions was evaluated through a consistent experimental methodology. It was demonstrated that with increasing molecular weight and the proportion of heavy fractions, the exothermic maxima shift to higher temperature regions and heat release during the HTO stage becomes more intense. The IR analysis results confirmed that oxidation and pyrolysis reactions proceed in parallel, and that alkanes, alkenes, aromatic and carbonyl-containing compounds, as well as CO and CO_2 gases, are formed during the process.

These findings organize the interconnectedness of a sample's heat-induced response with its structural composition. This establishes a theoretical framework for enhancing fuel ignition and downstream refining operations.

2. Materials and Methods

Petroleum specimens were sourced from the Korchagin reservoir in Kazakhstan and various locations across the Absheron petroleum region in Azerbaijan, specifically the Zaghli, Balakhani, and Korchangin fields. Table 1 details the fundamental physicochemical characteristics of these samples. The selected oils represent light and heavy crude oil types, differing in density, viscosity, acidity, and molecular weight.

Table 1. Key physicochemical attributes of the investigated crude oils

Oil	Mass density, 20 °C (kg/m³)	Autoignition point (°C)	Viscosity (kinematic), 40 °C (mm²/s)	S (wt. %)	Solidification temperature (°C)	Neutralization capacity (mg KOH/g)	Residual carbon content (%)
Korchagin	740	+20	6.8	0.39	−20	0	0.92
Zaghlı	847.9	+9	5.03	0.159	−30	1.31	1.04
Balakhani	911.5	+36	30.45	0.329	−40	3.5	1.54

Characterization of the oxidative thermal response of the petroleum samples was conducted on a LINSEIS STA PTI1600 (STA Platinum Series) unit operating within an ambient air atmosphere. The experiments were performed by subjecting each sample to a linear temperature ascent from 20 °C up to 1000 °C, with the temperature raised at a uniform pace of 10 °C every minute. Specimens weighing roughly 20 mg were loaded into alumina (Al_2O_3) crucibles, with a constant stream of air supplied throughout the analytical procedure.

The experimental setup relied on the simultaneous operation of a thermobalance (TGA) and a differential scanning calorimeter (DSC), making it possible to register in parallel the temperature‑driven enthalpy changes and the gravimetric trend throughout the oxidation process. The TGA component served to distinguish between phases of volatile release, oxidative reactions, and final structural breakdown by tracking continuous weight reduction. Simultaneously, DSC data facilitated the identification of thermal stability limits, reaction onset points, and heat-flow transitions, encompassing both endothermic and exothermic events.

In addition, the volatile products formed during the thermo-oxidation process were characterized using FTIR spectroscopy. FTIR analysis enabled identification of evolved gaseous species and functional groups formed at different temperature intervals, providing complementary information on reaction pathways and confirming the staged oxidation mechanism observed in DSC-TG experiments.

3. Results and Discussion

DTA-TG analyses conducted in an atmospheric environment in the presence of oxygen have shown that the thermo-oxidative process of the investigated Korchagin, Zaghli, and Balakhani crude oils is complex and multistage in nature. The correspondence observed between the heat-flow and mass-change curves indicates that the thermal transformation is determined by the fractional composition of the oils, the degree of aromaticity, and, in particular, the molecular structural characteristics of the resin–asphaltene components.

As the temperature increases, hydrocarbons of different structural types sequentially enter oxidation reactions, and these processes are recorded as resolved heat effects on the DTA/DSC curves and as stepwise mass loss on the TG curve.

The thermal decomposition profile displayed by the Korchagin petroleum specimen reveals two primary exothermic phases followed by HTO (Figure 1). The initial exothermic event takes place within the 155-476°C temperature window, where the bulk of the weight reduction (roughly 5.8 mg) is detected. During this interval, the peak enthalpic release was documented at 358.6°C (ΔH = 2443 J/g). This stage corresponds to the LTO stage and is associated with the evaporation of light-middle fractions, oxidation of aliphatic chains, and the formation of hydroperoxide-type intermediate products.

The second exothermic stage is observed in the 544-631°C interval (ΔH = 459 J g^{-1}, $\Delta m \approx 0.75$ mg). The decrease in enthalpy indicates the oxidation of more stable polyaromatic structures. In this temperature range, the decomposition of resin–asphaltene aggregates and aromatic condensation processes predominate.

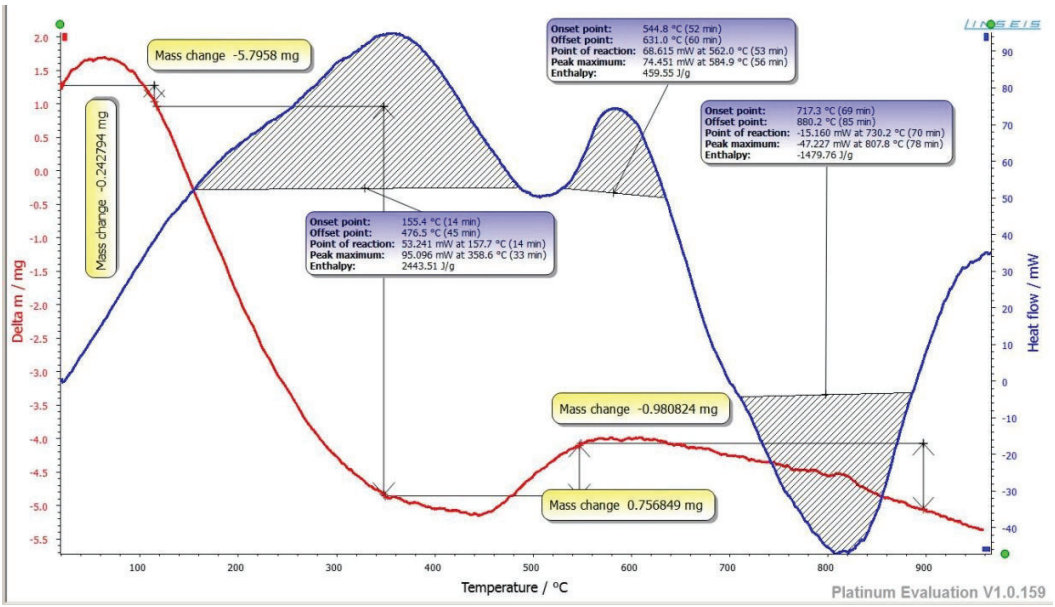

Figure 1. Thermal profiles (DTA-TG) for the Korchagin sample recorded in an oxidizing atmosphere.

The terminal oxidative phase, identified between 717 and 880 °C (ΔH = –1479 J/g, Δm ≈ 0.98 mg), is defined by the ignition of carbonaceous residuals and suggests a high concentration of aromatic components. Based on this enthalpy profile, the Korchagin specimen demonstrates a reliable capacity for energy generation, which is beneficial for subsurface combustion (ISC) techniques.

The Zaghli sample exhibits the clearest multistage oxidation behavior (Figure 2). The initial stage is observed in the 183-255°C interval (ΔH=150 J g^{-1}, Δm= -3.30 mg) and is associated with the release of volatile components. The second stage occurs in the 348-487°C interval, and the heat effect increases sharply (ΔH=1220 J g^{-1}, Δm≈6.4 mg). In this region, radical chain oxidation reactions predominate. The main heat-generation stage was recorded in the 507-787°C interval. The peak maximum was 567°C, and the enthalpy was ΔH=5093 J g^{-1}. This value indicates the highest energy release among the investigated oils and is explained by the intensive oxidation of aromatic nuclei, resin–asphaltene structures, and the carbon skeleton (Δm≈ -10.05 mg). In the final stage (869-913°C, ΔH=282 J g^{-1}), combustion of residual carbon is observed. Thus, Zaghli crude oil is distinguished by maximum heat generation at high temperature.

Balakhani crude oil exhibits the classical LTO–HTO oxidation sequence (Figure 3). The initial stage occurs in the 70-222°C interval (ΔH=85 J g^{-1}, Δm≈1.5 mg) and corresponds to the radical initiation stage. The second stage develops in the 306-432°C interval (ΔH=862 J g^{-1}, Δm≈ -10.2 mg).

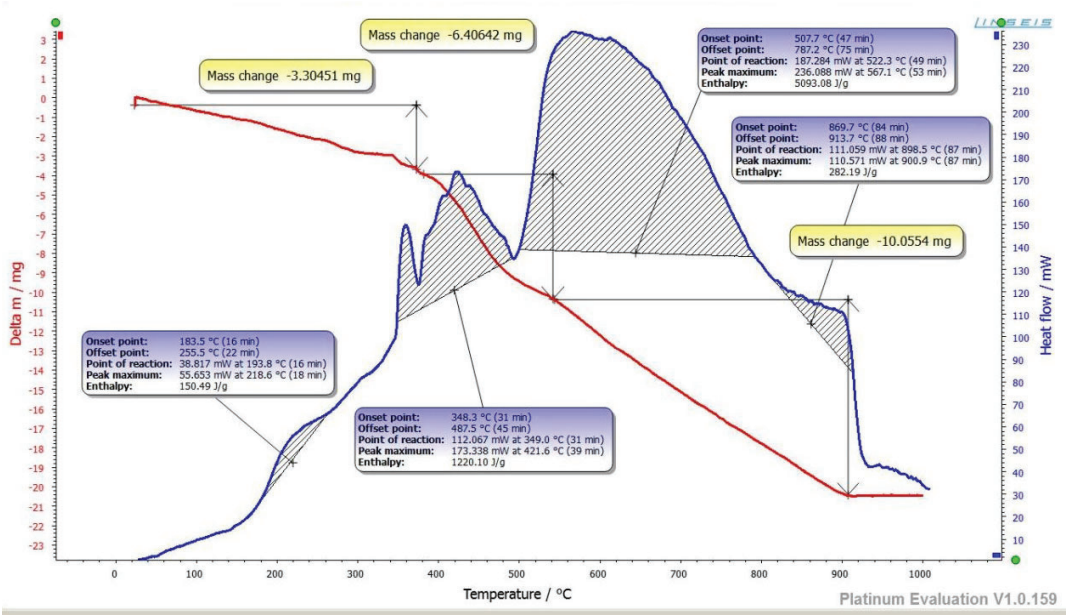

Figure 2. Thermal profiles (DTA–TG) for the Zaghli petroleum specimen recorded within an oxidizing atmosphere.

In this region, oxidation of resin components and destabilization of asphaltene aggregates occur. The main heat-generation zone is observed in the 439-628°C interval (ΔH=2870 J g^{-1}, $\Delta m \approx -3.65$ mg). This phase involves the oxidative transformation of polyaromatic networks and the formation of carbonaceous residuals, with the oxidation step at elevated temperatures, denoted HTO, is terminated once the solid carbon‑containing remnants have been entirely burned off.

Comparative analysis of the DTA-TG results of Korchagin, Zaghli, and Balakhani crude oils shows that, although their thermo-oxidative behavior is similar in terms of the general mechanism, the distribution of heat while the specific levels of chemical responsiveness are determined by the internal molecular architecture of the oils. Korchagin crude oil exhibits higher reactivity at the early oxidation stage, with a high heat effect and the primary reduction in specimen weight recorded within the low-temperature region. Zaghli crude oil, on the other hand, is distinguished by the maximum heat generation recorded in the high-temperature interval ($\Delta H \approx 5093$ J g^{-1}), indicating the predominance of intensive oxidation of heavy aromatic and resin-asphaltene components. Compared with the other samples, Balakhani crude oil exhibits more stable thermal behavior and is characterized by a transformation mechanism consistent with the classical LTO-HTO kinetic sequence. Linking enthalpic transitions with weight fluctuations demonstrates that across every specimen, the oxidative process has a structurally dependent staged character.

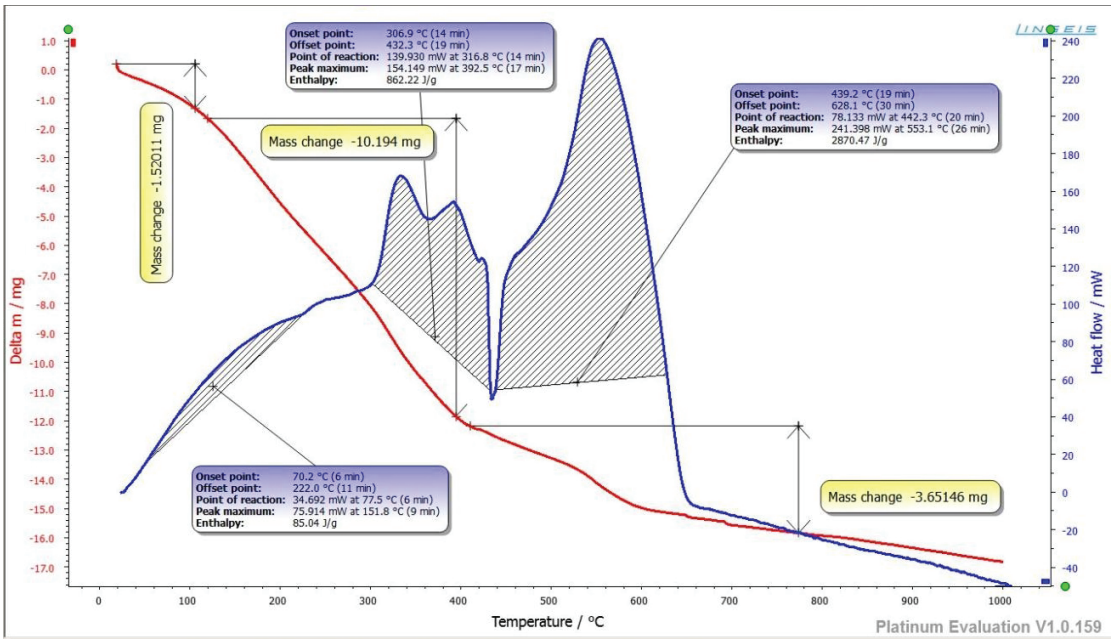

Figure 3. Thermal profiles (DTA-TG) for the Balakhani petroleum specimen recorded within an oxidizing atmosphere.

The thermo-oxidative mechanism is explained as staged radical oxidation reactions based on the combined analysis of heat effects and mass change. In the LTO stage, hydroperoxide- and peroxide-type intermediate products are formed as a result of the incorporation of oxygen molecules into aliphatic hydrocarbon chains, and the process is accompanied by an exothermic effect [39, 40]. As the temperature increases, the decomposition of these unstable compounds leads to the formation of free radicals, and chain reactions intensify. In the intermediate-temperature region, structural reorganization of asphaltene aggregates occurs as a result of the weakening of π-π interactions [41], whereas in the HTO stage, condensed polyaromatic structures undergo carbonization to form coke-like residues, and their reaction with oxygen constitutes the main heat-generation stage [42]. Thus, for all three crude oils, the thermo-oxidative process generally proceeds through the sequence of radical initiation→decomposition of oxygen-containing intermediate products→aromatic condensation→carbonization and combustion.

Volatile products released under isothermal conditions were identified by FTIR spectroscopy (Table 2).

Table 2. Absorption bands recorded during the DTA of crude oil based on the FTIR spectrum

Recorded maxima (cm^{-1})	Bonds	Identified compounds
3556	O-H	H_2O
3079, 3078 1550, 1540, 1610	C-H C=C	aromatic compounds
3018 1625, 1633, 1670	C-H C=C	alkene
2968, 2920, 2850	CH_3 and CH_2	alkane or alkyl substituents
1705, 1710, 1720, 1750, 1755	C=O	ketone or aldehyde
2250, 2350, 2361	O=C=O	CO_2

2119	C=O	CO

With increasing temperature, the absorption bands intensified considerably, which reflected the accumulation of larger amounts of volatile and gaseous matter; conversely, the subsequent drop- off in these signals attested to the near- complete consumption of the fuel feedstock.

The absorption signal centered at 3556 cm^{-1} stems from the O–H stretching mode, thereby confirming the production of water vapour. The 2400-2224 cm^{-1} and 2180-2108 cm^{-1} regions are associated with the formation of CO_2 and CO gases, respectively. The bands at 2968, 2920, and 2850 cm^{-1} indicate CH_3 and CH_2 groups, the 3076-3079 cm^{-1} bands indicate aromatic =C–H bonds, and the 1610–1540 cm^{-1} region indicates aromatic C=C bonds.

The observation of carbonyl groups in the 1705-1755 cm^{-1} region confirms the formation of aldehydes, ketones, and esters as a result of oxidation. The formation of alkenes, in turn, is explained via the scission of aliphatic lateral substituents and pyrolysis reactions.

Integrating the data from DTA and FTIR investigations reveals that thermo-oxidative process develops sequentially according to the pathway LTO→intermediate stage→HTO, and the caloric capacity of high-molecular-weight constituents is primarily released within the elevated thermal zone.

4. Conclusion

In the current investigation, the heat-induced oxidative response of Korchagin, Zaghli, and Balakhani crude oils was comparatively evaluated based on DTA-TG and IR analyses. The results showed that all samples undergo transformation via a staged radical mechanism consisting of LTO, intermediate structural transformation, and HTO stages.

In Korchagin crude oil, the main reactivity was observed in the 155-476°C interval and was accompanied by a high heat effect (ΔH=2443 J g^{-1}). Zaghli crude oil, in contrast, exhibited the highest heat-generation potential, showing the maximum enthalpy value (ΔH≈5093 J g^{-1}) in the 507–787°C temperature range. Balakhani crude oil exhibited more stable thermal behavior, with the main oxidation stage occurring in the 439-628°C interval (ΔH=2870 J g^{-1}).

FTIR results confirmed the absorption bands corresponding to 3556 cm^{-1} (H_2O), 2400-2224 cm^{-1} (CO_2), 2180-2108 cm^{-1} (CO), and aromatic C=C bonds during oxidation, and demonstrated the temperature-dependent development of carbonization and combustion processes. In totality, the outcome of this investigation points toward heavy crude oils from Azerbaijani fields possess their main heat-generation potential in the high-temperature interval and are promising energy sources for ISC technologies.

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