



Catalytic pyrolysis of waste lubricating oil into jet fuel using aloe vera-modified CaO catalyst derived from marble waste

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ABSTRACT

The improper disposal of waste lubricating oil (WLO) and marble waste poses serious environmental challenges, while their valorization into fuel-range products offers a promising circular-economy approach. Although marble waste can be used as a calcium-based catalyst precursor, its irregular and agglomerated morphology may affect the dispersion of active metal species and catalytic performance. This study developed an Aloe vera-assisted hydrothermal modification of marble-waste-derived CaO, followed by Ni and Mo impregnation, for the catalytic pyrolysis of WLO into fuel-range hydrocarbons. Marble waste was ground, sieved, dried, mixed with Aloe vera extract, and hydrothermally treated at 150 °C for 12 h. The resulting material was impregnated with 5 wt% Ni and 5 wt% Mo and calcined at 850 °C for 5 h to obtain the NiMo/MWAC catalyst. The synthesized materials were characterized using XRD, FTIR, FESEM-EDS, TEM, N₂ adsorption-desorption, XPS, and CO₂-TPD analyses. The Aloe vera-assisted treatment modified the surface morphology and facilitated the distribution of Ni and Mo species on the calcium-based support. XRD and XPS confirmed the formation of CaO, NiO, and CaMoO₄ phases and the presence of Ni²⁺ and Mo⁶⁺ species. NiMo/MWAC exhibited the best catalytic performance, producing a maximum liquid yield of 51.86 wt% and promoting hydrocarbons in the jet-fuel range, particularly C13–C16. Response Surface Methodology showed that reaction temperature and catalyst loading were the most influential variables, whereas reaction time had a limited effect. The developed NiMo/MWAC catalyst provides a low-cost and sustainable route for the simultaneous valorization of marble waste and WLO into jet-fuel range hydrocarbons.

1. Introduction

The global demand for energy has increased substantially over recent decades, driven by rapid population growth, industrialization, and the expansion of transportation activities (Zhang et al., 2025). Among the transportation sectors, aviation is one of the fastest-growing consumers of liquid fuels, with petroleum-derived jet fuel remaining its principal energy source. Continued dependence on fossil-based jet fuel accelerates the depletion of non-renewable resources and contributes significantly

to greenhouse-gas emissions. Global jet-fuel demand is projected to increase from approximately 106 billion gallons in 2019 to 230 billion gallons by 2050, while the aviation sector accounted for approximately 2% of global CO₂ emissions in 2022 (Umenweke et al., 2025). These trends emphasize the need to develop sustainable and waste-derived alternatives to conventional aviation fuels.

The conversion of waste materials into fuel-range products has attracted increasing attention as a strategy for simultaneously reducing environmental pollution and producing value-added energy carriers.

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Waste lubricating oil (WLO) is a promising feedstock because of its high hydrocarbon content and physicochemical similarity to heavy petroleum fractions. Improper disposal of WLO may therefore contaminate soil and water. Its widespread generation, particularly in regions with high vehicle use, further highlights the need for effective recycling technologies. For example, Thailand alone generates approximately 230 million litres of WLO annually (Nisamaneenate et al., 2023). Therefore, converting WLO into fuel-range hydrocarbons can reduce its environmental burden while supporting circular-economy and waste-valorization principles (García-Gutiérrez et al., 2025).

Pyrolysis is a promising thermochemical process for converting hydrocarbon-rich waste into liquid products, non-condensable gases, and solid residues. The process is generally conducted at elevated temperatures in the absence of oxygen and offers several advantages, including feedstock flexibility, relatively simple operation, and the ability to recover fuel-like hydrocarbons (Beims et al., 2019; Jovita et al., 2025). Nevertheless, non-catalytic pyrolysis commonly provides limited control over product distribution and may result in excessive gas or coke formation. The use of catalysts is therefore important to control cracking, dehydrogenation, hydrogen-transfer, deoxygenation, and hydrocarbon-rearrangement reactions, thereby improving liquid production and directing the product distribution toward jet-fuel-relevant hydrocarbons, generally within the C8–C16 range (Tamim et al., 2024, 2025).

Among various heterogeneous catalysts, basic oxides modified with transition metal, such as CaO and MgO, have attracted considerable attention because of their low cost, thermal stability, ease of separation, and ability to promote the adsorption and conversion of oxygen-containing compounds (Brahma et al., 2022; Sharifi et al., 2025). Marble waste is a promising precursor for CaO-based catalysts because it is an abundant industrial by-product composed predominantly of calcium carbonate. Converting marble waste into a catalytic material can reduce the environmental burden associated with its disposal while providing a low-cost and sustainable source of CaO. Nevertheless, CaO obtained directly from marble waste commonly consists of compact, irregular, and highly agglomerated particles. These morphological characteristics may restrict surface accessibility, reduce the effective contact between the catalyst and large WLO molecules, and hinder the uniform distribution of deposited metal species. Therefore, morphological and textural modification of marble-waste-derived CaO is required to improve active-site accessibility and facilitate the dispersion of additional catalytic components.

Natural plant extracts offer a greener alternative to conventional synthetic modifying agents. Aloe vera is particularly attractive because it contains polysaccharides, phenolic compounds, organic acids, and oxygen-containing functional groups that may interact with calcium during catalyst preparation (Shahinuzzaman et al., 2024). During hydrothermal treatment, these organic constituents may influence nucleation, particle growth, and particle organization. Their subsequent decomposition during calcination may promote surface restructuring, reduce particle agglomeration, and generate interparticle voids that improve the accessibility of the catalyst surface. Aloe vera modification may also influence the interaction between the CaO support and subsequently deposited metal species, thereby promoting a more uniform distribution of the active components.

Although CaO provides strong basic sites for adsorbing oxygenated compounds, WLO upgrading also requires metallic sites for C–C and C–O bond cleavage and hydrogen-transfer reactions. Therefore, transition metals were incorporated into Aloe-vera-modified marble-waste-derived CaO to provide complementary catalytic functions. Ni is widely used because of its high activity toward cracking, dehydrogenation, and hydrogen transfer. However, monometallic Ni may undergo agglomeration and sintering and may have limited oxygen-removal capability. To overcome these limitations, Mo can be combined with Ni because its redox and oxophilic properties promote C–O bond activation and improve metal dispersion, reducibility, and active-phase stability. Thus,

the Ni–Mo combination integrates the cracking activity of Ni with the oxygen-removal function of Mo. The effectiveness of Ni–Mo catalysts for waste-oil upgrading has been demonstrated in previous studies (El-sawy et al., 2020) applied Ni–Mo catalysts in the hydrogen-assisted co-processing of WLO-containing feeds. Similarly, (Ding et al., 2021) showed that the physicochemical properties of the support strongly influenced NiMo dispersion, reducibility, and catalytic performance. More recently, (Yuan et al., 2026) reported that NiMo supported on mesoporous aluminosilicate produced the highest conversion, liquid yield, and C8–C16 hydrocarbon selectivity among the investigated catalysts. These findings confirm the effectiveness of Ni–Mo while highlighting the important role of the support.

The novelty of this study is the use of Aloe vera extract as a bio-derived modifying agent for marble-waste-derived CaO prior to Ni and Mo incorporation to produce a multifunctional catalyst for WLO conversion under an inert atmosphere. The influence of Aloe vera treatment on particle morphology, crystallite size, and textural characteristics was systematically examined, followed by evaluation of the effects of Ni/Mo incorporation on catalytic activity and product distribution. Response Surface Methodology was then applied to determine the individual and interactive effects of reaction temperature, catalyst loading, and reaction time, as well as to identify the optimum conditions for maximizing WLO conversion and liquid yield.

2. Experimental

2.1. Materials

Waste lubricating oil (WLO) used as the feedstock was collected from a motorcycle repair workshop in Semarang, Indonesia. Marble waste (MW) were collected from Tulungagung, East Java, Indonesia, and were calcined at 850 °C before use. Aloe vera was collected from Surabaya, Indonesia. Ammonium heptamolybdate tetrahydrate ((NH₄)₆Mo₇O₂₄·4H₂O, 99.99%), nickel(II) nitrate hexahydrate (Ni(NO₃)₂·6H₂O, 99.99%), and n-hexane (>98%) were purchased from Sigma-Aldrich. Nitrogen gas (N₂, 99% purity) was used during the pyrolysis reaction, while n-hexane (>98% purity) was used as a solvent for GC–MS analysis.

2.2. Catalyst preparation

The Aloe vera's gel (50 g) was blended with 100 mL of deionized water, then heated and stirred at 80 °C for 2 h before filtration to obtain the Aloe vera extract. Marble waste (MW) obtained from Tulungagung was ground, sieved through an 80-mesh sieve, and dried at 105 °C for 24 h to remove moisture. Subsequently, MW was mixed with 157 mL of Aloe vera extract and stirred for 45 min, followed by hydrothermal treatment at 150 °C for 12 h to produce MWA. Bimetallic modification was then performed via the impregnation method using nickel(II) nitrate hexahydrate (Ni(NO₃)₂·6H₂O) and ammonium heptamolybdate tetrahydrate ((NH₄)₆Mo₇O₂₄·4H₂O) as metal precursors with a loading of 5 wt% each. The metal salts were dissolved in 10 mL of deionized water and stirred for 15 min, after which the uncalcined MWA catalyst was added and the mixture was stirred for 45 min at room temperature (25 °C). The suspension was further heated at 80 °C until a slurry formed, dried in an oven for 24 h, and finally calcined at 850 °C for 5 h to obtain the NiMo/MWAC catalyst.

2.3. Catalyst characterization

The crystal structure was characterized using X-ray Diffraction (XRD) using a PHILIPS-binary Xpert with MPD diffractometer with Cu K α radiation ($\lambda = 1.5405 \text{ \AA}$). The functional group was analyzed using Fourier Transform Infra-Red (FTIR) measurement was recorded using FTIR Shimadzu Instrument Spectrum One 8400S in the wave number range 4000–500 cm⁻¹. The specific surface area of catalysts was

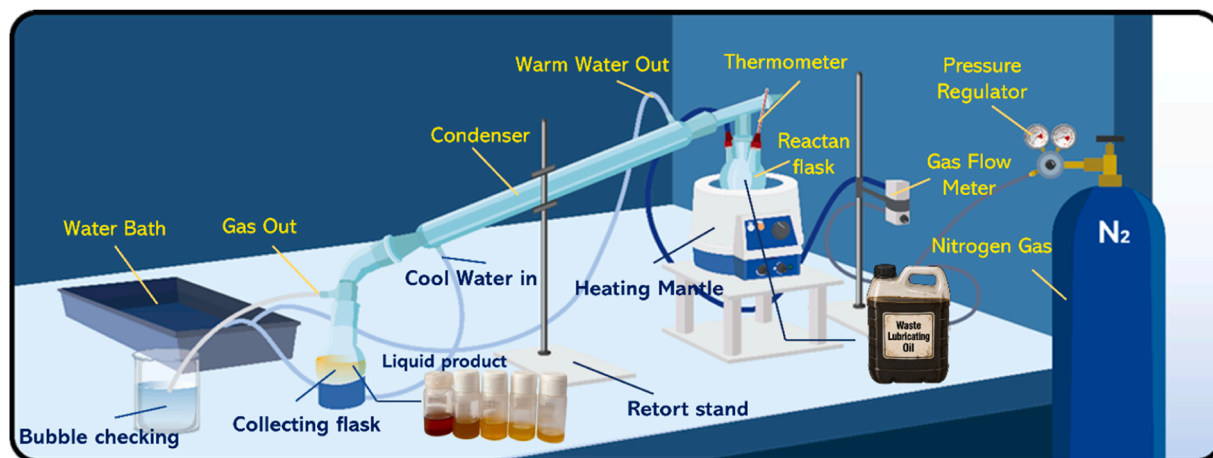


Fig. 1. Reactor designed for low-temperature pyrolysis of waste lubricant oil.

measured on a Micromeritics TriStar II Plus 3.03 by N_2 adsorption-desorption test using the Brunauer-Emmet-Teller (BET) method. The catalysts were pretreated at $150\text{ }^\circ\text{C}$ for 4 h under a nitrogen atmosphere to remove moisture from the catalyst surface. The Barrett-Joyner-Halenda (BJH) method was used to determine the pore size distribution profiles. Morphological analysis was characterized. The reducibility and basicity of the catalyst were analyzed using CO_2 temperature programmed reduction (CO_2 -TPD) on Quantachrome ChemBET Pulsar TPD/R/O) equipped with a thermal conductivity detector. The morphology of catalysts was characterized by Transmission Electron Microscopy (TEM) images obtained on a Hitachi HT-7700 at a voltage of 200 kV. X-ray photoelectron spectroscopy (XPS) was implemented to determine the elemental composition of the catalyst surface using a SPECS electron spectrometer with a PHOIBOS 100 SCD analyzer and a non-monochromatic Al/Mg twin X-ray source (SPECS GmbH, Germany). on a Hitachi SU8010 field emission scanning electron microscope (FE-SEM), operated at an accelerating voltage of 15 kV.

2.4. Pyrolysis of WLO

Waste lubricating oil (WLO) was employed as the feedstock for the catalytic pyrolysis reaction. The pyrolysis process was conducted under an inert nitrogen atmosphere to prevent unwanted oxidation reactions. A distillation system was connected to the reactor to continuously collect the condensed liquid products formed during the reaction as shown in Fig. 1. Various marble waste-based catalysts were evaluated, namely MWC, MWAC, Ni-MWAC, and Ni/Mo-MWAC. For each

experiment, 10 g of WLO was introduced into a three-necked round-bottom flask containing the catalyst and heated in an oil bath at a controlled temperature. The reaction mixture was continuously stirred and heated to $380\text{ }^\circ\text{C}$ for 4 h under an N_2 flow. The liquid products were subsequently analyzed by GC-MS for further characterization.

2.5. Liquid product analysis

Liquid products were analyzed using gas chromatography-mass spectroscopy (HP 6890 GC). The analysis was conducted using HP-5MS as a capillary column (length: 30 m x inner diameter: 0.25 mm x film thickness: 0.25 mm). The samples were diluted with GC-grade n-hexane. The peak area from GCMS analysis was used to calculate the yield of various products and to confirm the components of the liquid products. The selectivity of hydrocarbon was calculated using Eq. (1). The percentage of liquid yield, char yield, gas yield, hydrocarbon yield, and cyclic yield were determined using Eqs. (2)–(6), respectively. The hydrocarbon selectivity was calculated using Eq. (7), aromatic selectivity using Eq. (8).

$$\text{Selectivity of WLO} = \left(\frac{\text{peak area of desired hydrocarbon}}{\text{peak area of total hydrocarbon}} \right) \times 100\% \quad (1)$$

$$\text{Liquid Yield} = \left(\frac{\text{weight of liquid product}}{\text{weight of reactant}} \right) \times 100\% \quad (2)$$

$$\text{Char Yield} = \left(\frac{\text{weight of char product}}{\text{weight of reactant}} \right) \times 100\% \quad (3)$$

$$\text{Gas Yield} = 100 - (\text{oil yield} + \text{char yield}) \quad (4)$$

$$\text{Hydrocarbon yield (\%)} = \left(\frac{\text{total area of hydrocarbon}}{\text{total area of liquid product}} \right) \times 100\% \quad (5)$$

$$\text{Cyclic yield (\%)} = \left(\frac{\text{total area of cyclic}}{\text{total area of liquid product}} \right) \times 100\% \quad (6)$$

$$\text{Hydrocarbon selectivity (\%)} = \left(\frac{\text{desired hydrocarbon fraction}}{\text{total area of liquid product}} \right) \times 100\% \quad (7)$$

$$\text{Aromatic selectivity (\%)} = \left(\frac{\text{desired aromatic fraction}}{\text{total area of liquid product}} \right) \times 100\% \quad (8)$$

Table 1
Experimental runs using Box-Behken design (BBD).

Experiment No.	Parameters		
	Temperature ($^\circ\text{C}$)	Catalysts Loading (%)	Reaction Time (h)
1	360	1	2
2	400	1	2
3	360	5	2
4	400	5	2
5	360	3	1
6	400	3	1
7	360	3	3
8	400	1	3
9	380	5	1
10	380	3	1
11	380	1	3
12	380	5	3
13	380	3	2
14	380	3	2
15	380	3	2

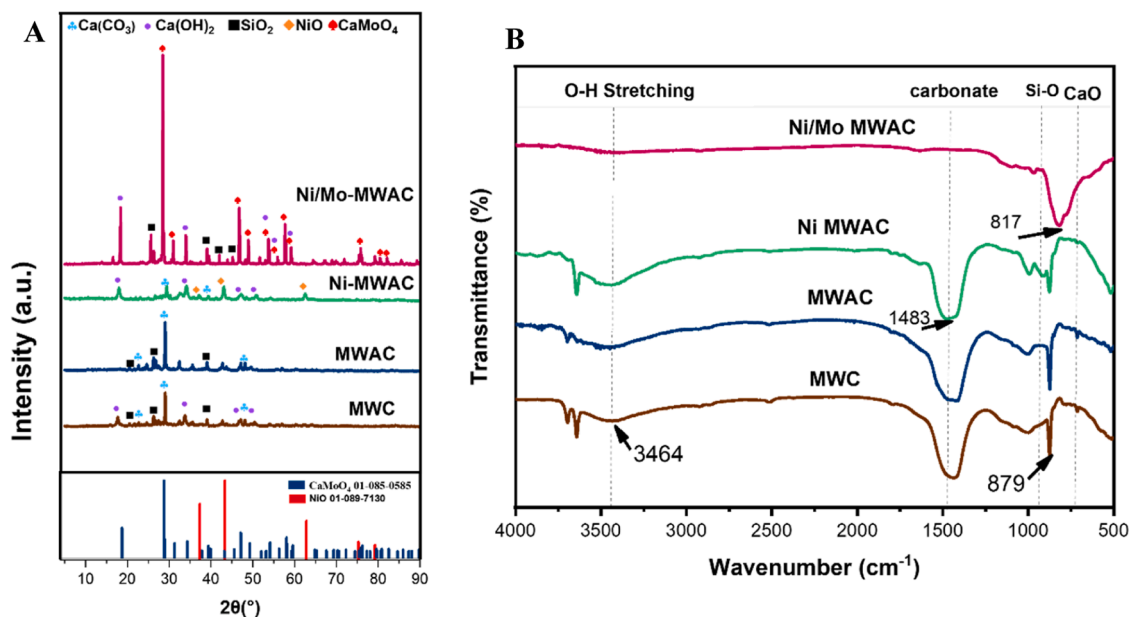


Fig. 2. XRD pattern for wide angle (A) FTIR spectra (B) of catalysts (MWC, MWAC, Ni-MWAC, Ni/Mo-MWAC).

2.6. Response methode surface (RSM)

Response Surface Methodology (RSM) was employed as a statistical and mathematical approach to design the experimental framework, optimize the reaction conditions, and evaluate the effects of independent variables on the desired response. In this study, RSM was applied to determine the optimum conditions for producing jet-fuel-range hydrocarbons through the catalytic pyrolysis/deoxygenation of waste lubricant oil (WLO). The experimental matrix was generated using Statistica software version 6.0 based on the Design of Experiments (DOE) approach and Box-Behnken Design (BBD). This optimization strategy was preceded by a preliminary assessment to establish the appropriate range of operating conditions. Three independent variables were investigated, namely reaction temperature (x_1), loading catalysts (x_2), and reaction time (x_3), with the objective of improving product yield, WLO conversion, and jet-fuel-range hydrocarbon selectivity. As shown in Table 1, a total of 15 experimental runs were performed in a randomized order according to the Box-Behnken Design (BBD).

The effects of the independent variables on the response were modeled using a second-order polynomial equation, as typically adopted in Response Surface Methodology (RSM) to capture linear, interaction, and quadratic effects (Singmai et al., 2026).

$$Y = \beta_0 + \sum_{i=1}^3 \beta_i x_i + \sum_{i=1}^3 \beta_{ii} x_i^2 + \sum_{i < j}^3 \beta_{ij} x_i x_j + \varepsilon \quad (9)$$

In this equation, Y represents the predicted freshwater production rate, β_0 denotes the intercept term, β_i represents the linear coefficients, β_{ii} the quadratic coefficients, β_{ij} the interaction coefficients, and ε the residual error.

The experimental data were statistically evaluated using analysis of variance (ANOVA) to determine the significance of the model terms and estimate the coefficients of the fitted polynomial equation. The coefficient of determination (R^2) was employed to assess the predictive quality and fitting performance of the model. Meanwhile, the F-value and p-value were used to confirm the statistical significance and adequacy of the regression model.

3. Results and discussion

3.1. Catalysts characterization

3.1.1. X-ray diffraction

The XRD patterns of the synthesized materials are shown in Fig. 2 (A). The MWC sample exhibits characteristic diffraction peaks of CaCO_3 at 2θ around 29.4° , 39.4° , 43.1° , and 47.5° , indicating that marble waste is mainly composed of calcite. Minor peaks corresponding to Ca(OH)_2 and SiO_2 are also observed. After activation (MWAC), the intensity of CaCO_3 peaks decreases, while new reflections appear at 2θ values of 32.2° , 37.3° , 53.8° , 64.1° , and 67.3° (JCPDS no file 01-085-0585), which correspond to CaO phases, suggesting partial decomposition of CaCO_3 during calcination (Mustafa et al., 2025; Murguía-ortiz et al., 2021).

Catalyst Ni-MWAC, additional peaks at 2θ of 37.2° , 43.3° , 62.9° , and 75.4° are attributed to NiO (JCPDS 01-089-7130), confirming the successful impregnation of Ni species (Murguía-ortiz et al., 2021). In the Ni/Mo-MWAC catalyst, new reflections at 2θ around $26-27^\circ$, 32° , and $47-58^\circ$ correspond to CaMoO_4 (JCPDS 01-085-0585), indicating the formation of Mo-containing phases (Kouider et al., 2024). The presence of NiO peaks suggests that Ni and Mo species coexist on the catalyst surface.

3.1.2. FTIR

FTIR results of the samples, as shown in Fig. 2(B) a spectra absorption band at $3400-3600 \text{ cm}^{-1}$, indicating the presence of hydrogen-bonded $-\text{OH}$ groups associated with adsorbed H_2O . The strong band at around 1450 cm^{-1} corresponds to the asymmetric stretching vibration (ν_3) of the carbonate group (CO_3^{2-}), while the bands at approximately 875 cm^{-1} and 712 cm^{-1} are assigned to the out-of-plane (ν_2) and in-plane (ν_4) bending vibrations of carbonate, respectively (Tamim et al., 2024). These bands are characteristic of calcium carbonate (CaCO_3), which can form due to the interaction of CaO with CO_2 from ambient air. As CaO is hygroscopic and highly reactive, the formation of Ca(OH)_2 may also occur (Macheli et al., 2026). Absorption bands below 600 cm^{-1} are attributed to Ca-O lattice vibrations, while the band at $1000-1100 \text{ cm}^{-1}$ indicates the presence of Si-O bonds. In the Ni/Mo-MWAC sample, the intensity of the Ca-related carbonate and Ca-O bands decreases, suggesting that free Ca species are no longer dominant. This decrease can be attributed to the interaction between Ca species and molybdate species, leading to the formation of CaMoO_4 . The appearance or

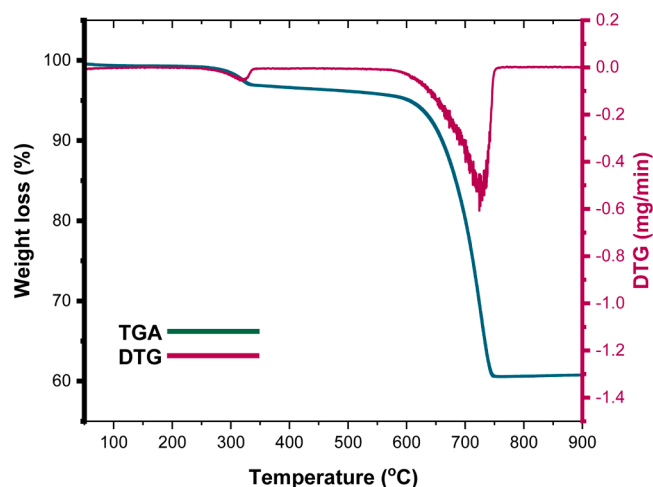


Fig. 3. TGA/DTG analysis of MWA uncalcinated catalysts.

strengthening of bands in the region below 900 cm^{-1} can therefore be associated with Mo–O vibrations in MoO_4^{2-} units, confirming the formation of a Ca–Mo–O environment rather than isolated CaO or CaCO_3 species, accompanied by the appearance of new bands below 800 cm^{-1} corresponding to Ni–O and Mo–O vibrations (Tamim et al., 2025).

3.1.3. Thermal analysis

The thermogravimetric (TGA) profile of MWA based on CaCO_3 before calcination shows the thermal decomposition process of the material. As shown in Fig. 3, A small mass loss of about 3% occurring at temperatures around 241–300 °C is attributed to the desorption of organic compounds from *Aloe vera* (Abu-thabit et al., 2026). The main mass loss occurs in the temperature range of approximately 650–780 °C, with a weight loss of about 37%, where the TGA curve exhibits a sharp decrease associated with the decarbonation of CaCO_3 into CaO accompanied by the release of CO_2 gas ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$) (Khosa and Zhao, 2019). At temperatures above approximately 780 °C, the mass of the material becomes constant, indicating that the decomposition process has been completed and the material has largely been converted into thermally stable CaO. Therefore, the application of a calcination

temperature above 800 °C is required to ensure the complete transformation of CaCO_3 into CaO.

3.1.4. Morphology and chemical composition

FESEM analysis of the MWA, MWC, MWAC, Ni-MWAC, and Ni/Mo-MWAC catalysts is presented in Fig. 4. The MWA sample, shown in Fig. 4 (A), exhibits a layered and agglomerated morphology with a relatively compact surface. After calcination or activation in MWC (Fig. 4B) (Hafriz et al., 2021) and the addition of aloe vera extract in MWAC (Fig. 4C), the surface structure becomes rougher and more irregular, with the formation of interparticle voids or pores (Strano et al., 2026). This morphological change indicates that the activation process can open the surface structure and enhance the porous texture of the marble waste-based material.

The Ni-MWAC sample (Fig. 4D), the surface becomes more granular, with small particles distributed on the support surface. This morphology suggests that Ni impregnation leads to the formation of metal oxide particles, mainly NiO. After Mo addition in the Ni/Mo-MWAC catalysts (Fig. 4E), the morphology changes into rounded or semi-spherical aggregates with larger particle sizes. This may be attributed to the formation of mixed oxide or molybdate phases during the impregnation and calcination processes. The EDX mapping results (Fig. 5) support these morphological observations by confirming the presence and distribution of Ca, O, Si, C, Ni, and Mo on the catalyst surface (Kouider et al., 2024).

HRTEM analysis of the Ni/Mo-MWAC sample shows in Fig. 6 the presence of dark particles dispersed on the support matrix. This dark contrast indicates the presence of metal or metal oxide species with higher electron density than the support. In the HRTEM image, a lattice spacing of 0.314 nm is observed, the observation of planes (112) corresponding to the tetragonal structure of CaMoO_4 (Almeida et al., 2025). This result indicates that Mo in the catalyst is associated with MoO_4^{2-} molybdate species, particularly in the form of CaMoO_4 or a Ca–Mo–O environment. The ring-like SAED/FFT pattern also indicates the polycrystalline nature of these particles.

3.1.5. N_2 adsorption-desorption

The N_2 adsorption-desorption isotherms of MWC, MWAC, and Ni/Mo-MWAC (Fig. 7) exhibit characteristics of type IV isotherms with a hysteresis loop, indicating the presence of mesoporous structures

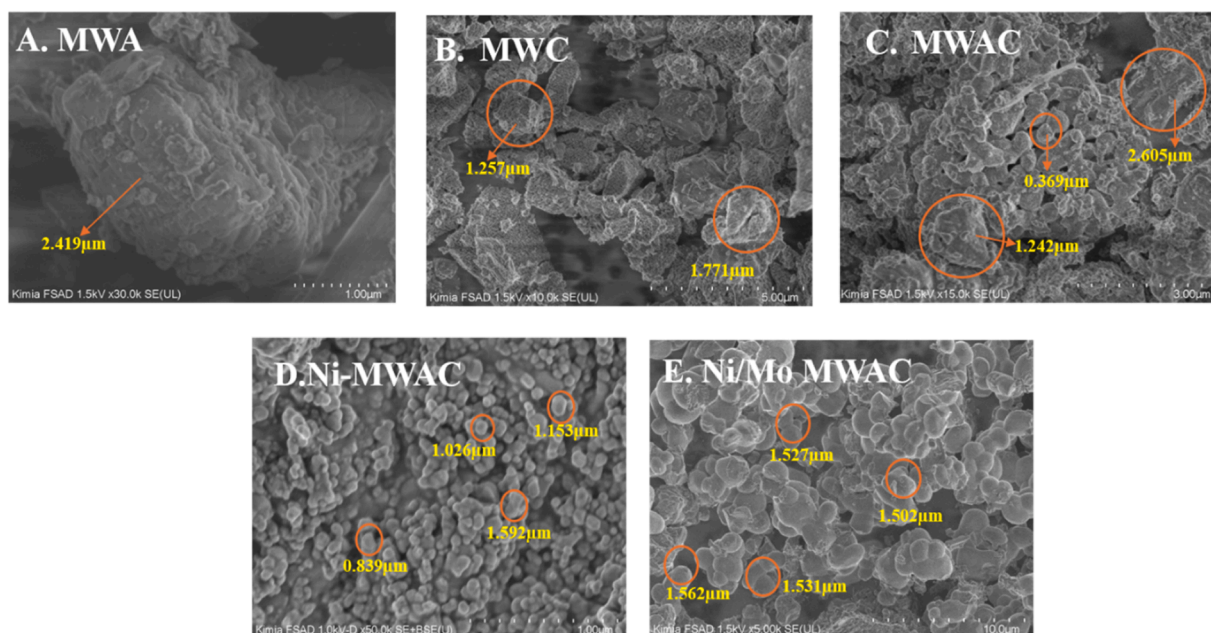


Fig. 4. FESEM images of MWA- uncalcinated (A); MWC (B); MWAC (C); Ni-MWAC(D); Ni/Mo-MWAC(E).

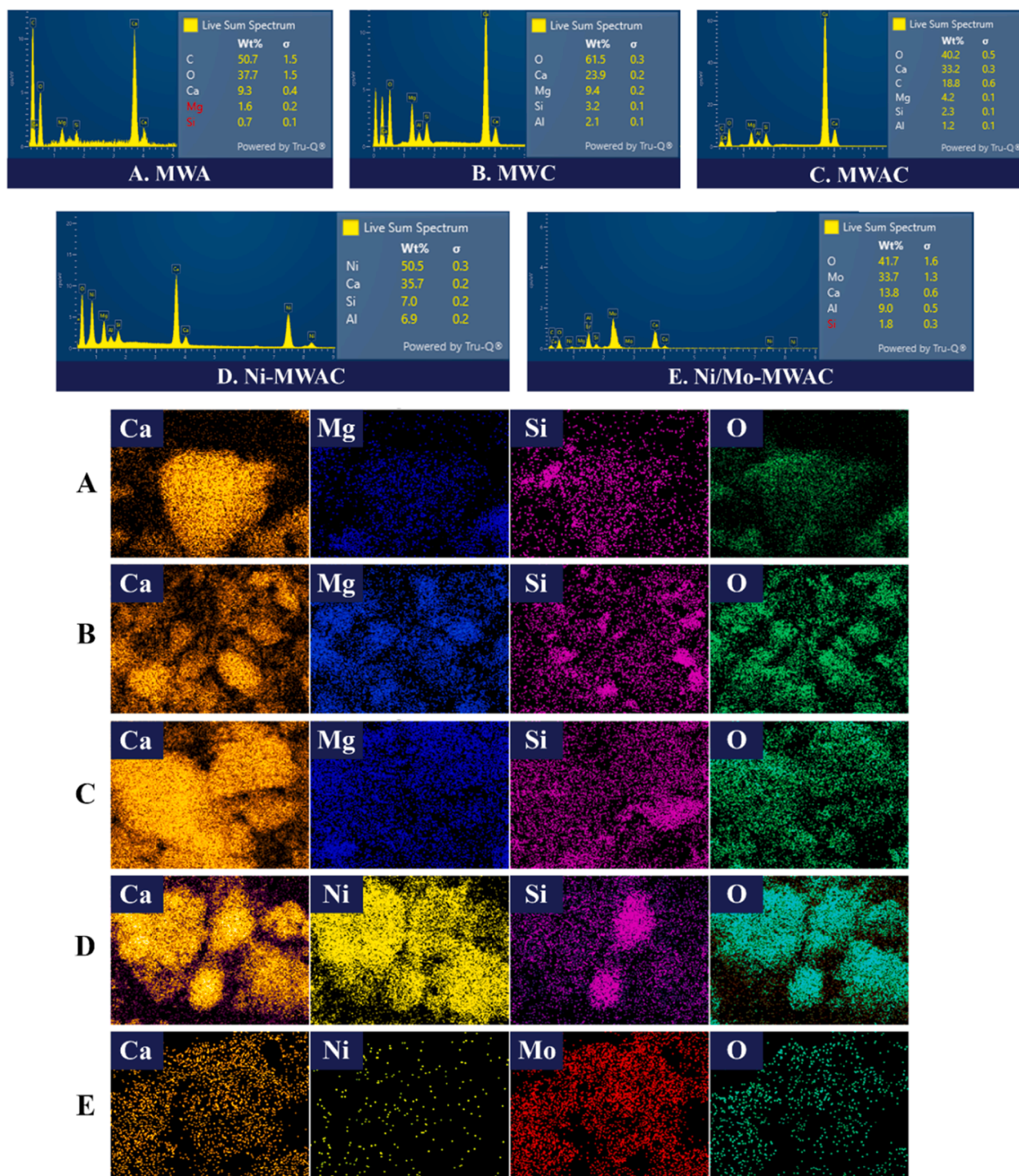


Fig. 5. EDX of MWA- uncalcinated (A); MWC (B); MWAC (C); Ni-MWAC(D); Ni/Mo-MWAC(E).

according to the IUPAC classification. The hysteresis loop suggests the formation of slit-shaped pores associated with aggregated particles and irregular pore structures (Tamim et al., 2025). Table 2 show the MWC sample shows a BET surface area (SBET) of $9.93 \text{ m}^2 \text{ g}^{-1}$ with a total pore volume of $0.0376 \text{ cm}^3 \text{ g}^{-1}$ and an average pore diameter of 28.12 nm. After activation, MWAC exhibits a slightly lower surface area ($8.2 \text{ m}^2 \text{ g}^{-1}$) but a larger average pore diameter (34.62 nm), suggesting structural rearrangement and pore development within the CaO matrix during the activation process (Ooi et al., 2021). The increase in pore diameter indicates that the activation step promotes the formation of

wider mesopores. After metal impregnation, the textural properties change significantly. Following Ni and Mo incorporation, the BET surface area increased markedly to $96.69 \text{ m}^2 \text{ g}^{-1}$, compared with $8.20 \text{ m}^2 \text{ g}^{-1}$ for MWAC and $6.85 \text{ m}^2 \text{ g}^{-1}$ for Ni-MWAC. The total pore volume also increased to $0.136 \text{ cm}^3 \text{ g}^{-1}$, while the average pore diameter reached 20.14 nm. However, the material remained predominantly non-porous, as indicated by its overall adsorption behavior. Therefore, the increase in surface area and pore volume is more reasonably attributed to surface roughening, particle rearrangement, interparticle void formation, or the development of cracks during impregnation and calcination, rather than

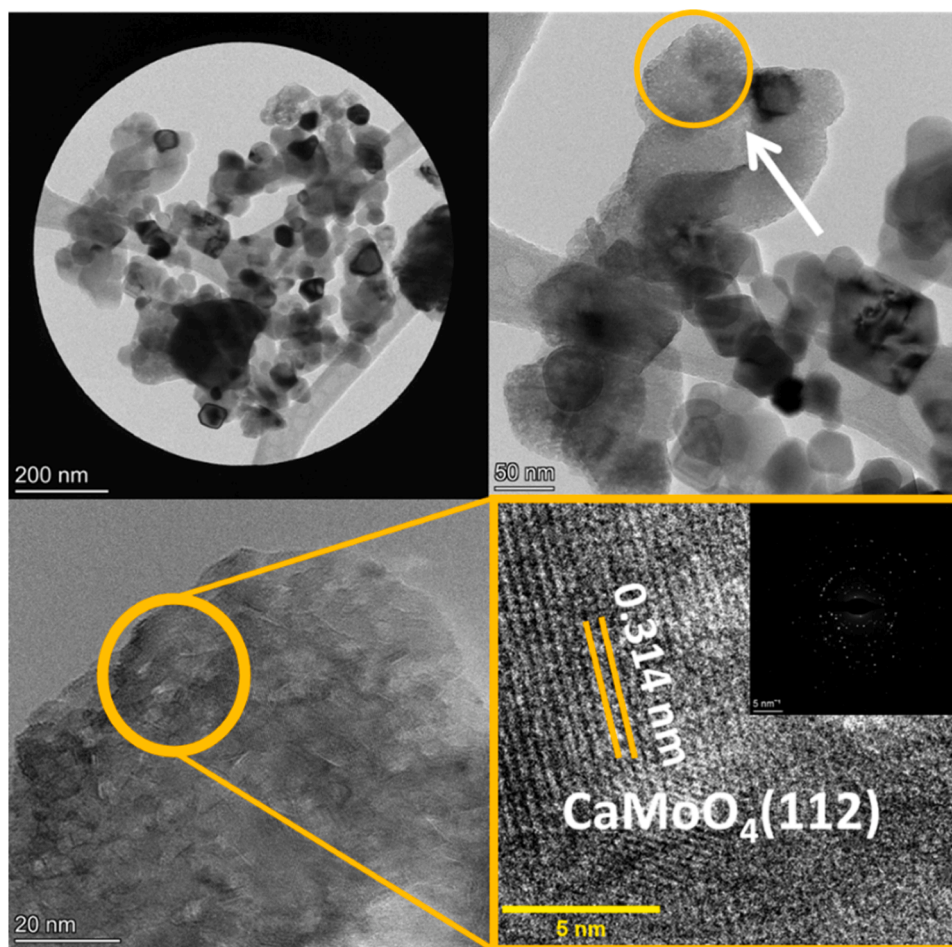


Fig. 6. HRTEM analysis of Ni/Mo-MWAC catalysts.

to the formation of a well-defined porous structure. These textural changes may improve the accessibility of WLO molecules to the Ni and Mo active sites.

3.1.6. XPS

XPS analysis was conducted to identify the oxidation states and elemental interactions in the Ni/Mo MWAC catalyst, which is mainly composed of Ca-O-Mo-Ni-C species (Fig. 8). In the O 1 s region, the peak at 530.0 eV can be assigned to lattice oxygen in the Ca-O-Mo framework of CaMoO_4 . This indicates the presence of MoO_4^{2-} molybdate units coordinated with Ca^{2+} , confirming the formation of CaMoO_4 species on the catalyst surface (Yong et al., 2024). The Ca 2p spectrum shows two peaks at 346.23 and 349.85 eV, corresponding to Ca $2p_{3/2}$ and Ca $2p_{1/2}$, respectively (Adaikalam et al., 2024). These peaks indicate the presence of Ca^{2+} species associated with CaO, Ca(OH)_2 , or Ca-O-Si interactions on the support surface. The C 1 s spectrum shows peaks at 282.5, 284.0, and 288.0 eV. The peak at 282.5 eV is assigned to Ca-C or Ca-interacting carbon species, while the main peak at 284.0 eV corresponds to C-C, C=C, and C-H from carbonaceous surface carbon. The peak at 288.0 eV is attributed to C=O groups from oxidized carbon species (Ben et al., 2025). The Si 2p peak at 101.75 eV confirms the presence of Si-O-Si bonds, suggesting that the silica framework was preserved after metal impregnation (Huang et al., 2024). The Ni 2p spectrum shows Ni $2p_{1/2}$ at 855 and Ni $2p_{3/2}$ at 872 eV, accompanied by satellite peaks at 860 and 880 eV, confirming the predominance of Ni^{2+} species related to the formation of NiO (Jovita et al., 2026). In the Mo 3d region, the peaks at 231.7 and 235.0 eV correspond to Mo $3d_{5/2}$ and Mo $3d_{3/2}$, respectively, indicating the presence of highly oxidized Mo^{6+} species in MoO_4^{2-} molybdate units (Yuan et al., 2026). These molybdate species may exist

as surface MoO_4^{2-} , Ca-Mo-O, or Ni-Mo-O environments.

3.1.7. Basicity catalyst analysis

The CO_2 -TPD profiles of MWC and MWAC as shown in Fig. 9, show desorption peaks in the high temperature region, indicating the presence of strong basic sites on the catalyst surface. The strong basicity is mainly attributed to surface O^{2-} species associated with CaO, which is formed from the thermal decomposition of CaCO_3 during calcination. The MWC catalyst shows a high-temperature CO_2 desorption peak at 735 °C, which is consistent with (Hafriz et al., 2024a) who reported a similar peak for calcined dolomite-based catalysts and assigned it to strong basic sites. MWAC exhibits a slightly higher desorption temperature at 723 °C. This shift suggests that the incorporation of Aloe vera extract before calcination modifies the surface environment and strengthens the interaction between CO_2 and the basic sites. The higher desorption temperature of MWAC indicates the formation of stronger basic sites, which may result from improved surface restructuring, better exposure of Ca-O species, or changes in the porous texture after decomposition of organic components from Aloe vera during calcination (Hafriz et al., 2021). In contrast, MWC showed a CO_2 desorption peak at 735 °C with a basicity of 4527.84 $\mu\text{mol g}^{-1}$, while Aloe vera modification increased the basicity of MWAC to 7182.17 $\mu\text{mol g}^{-1}$. Ni-MWAC exhibited two peaks at 687 and 849 °C with a total basicity of 6955.58 $\mu\text{mol g}^{-1}$, indicating that Ni incorporation slightly reduced the number of accessible basic sites while generating sites with different strengths. Ni/Mo-MWAC also showed two peaks at 714 and 815 °C, with a total basicity of 5750.29 $\mu\text{mol g}^{-1}$. Although lower than MWAC and Ni-MWAC, this value remained higher than MWC, suggesting that Mo addition modified the distribution of basic sites while preserving strong basicity (Tamim et al., 2024, 2025;

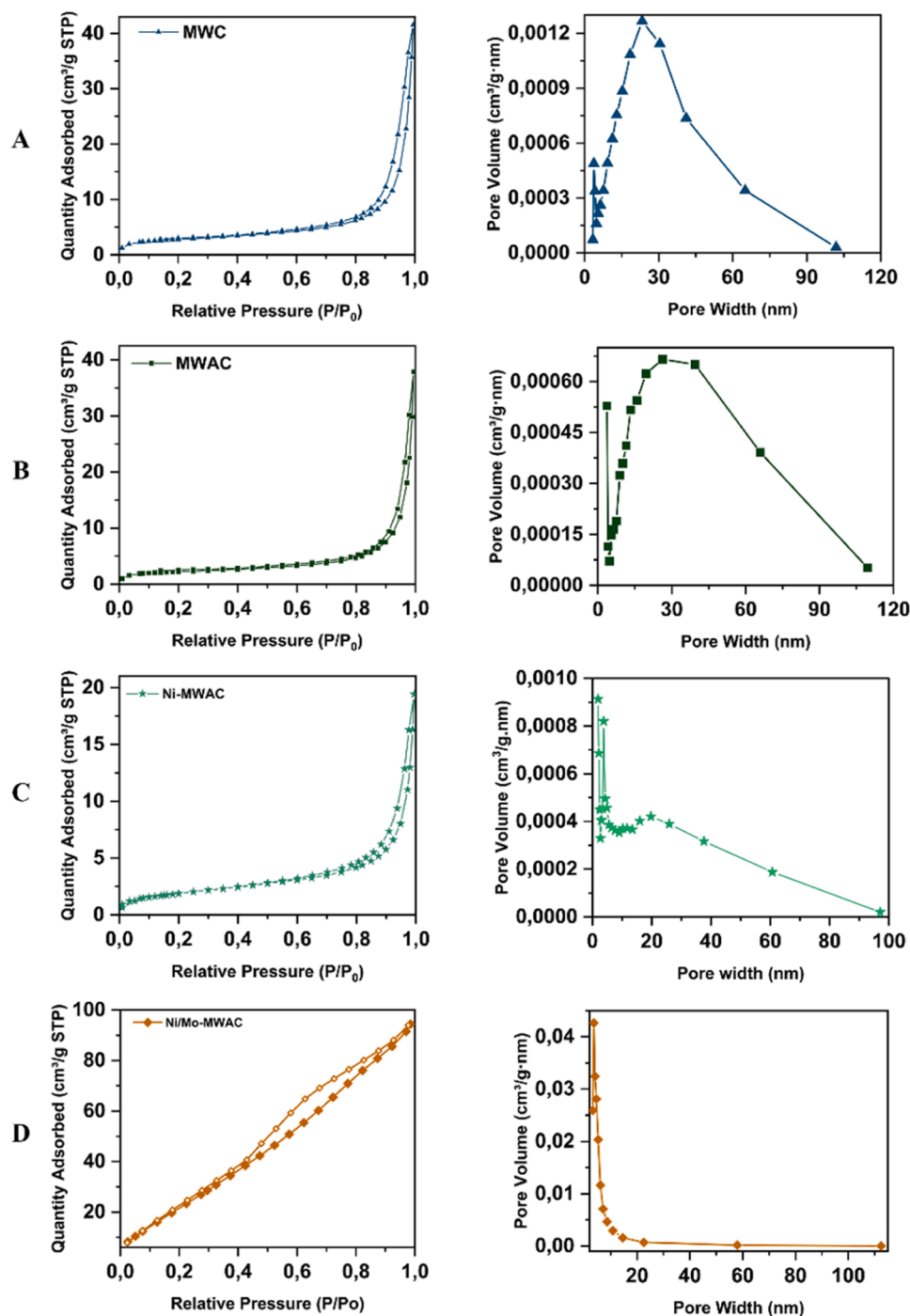


Fig. 7. Graph of pore distribution and Isotherm MWC (A); MWAC (B); Ni-MWAC(C); Ni/Mo-MWAC (D) catalyst.

Table 2

Textural properties of MWC, MWAC; Ni-MWAC; Ni/Mo-MWAC catalyst.

Catalyst	Surface Area (m ² /g) ^a		Pore Volume (cc/g) ^b			Average Pore width (nm) ^c	Basicity ^d	
	S _{BET}	S _{meso}	V _{meso}	V _{micro}	V _{tot}		Temperature (°C)	Amount (μmol/g)
MWC	9.93	9.01	0.0634	0.0011	0.0645	28.12	735	4527.84
MWAC	8.2	6.64	0.0574	0.0014	0.0588	34.62	723	7182.17
Ni MWAC	6.85	6.52	0.0298	-	0.0298	18.26	687	6323.02
Ni/Mo MWAC	96.69	66.60	0.136	-	0.136	20.14	849	632.56
							714	3655.72
							815	2094.57

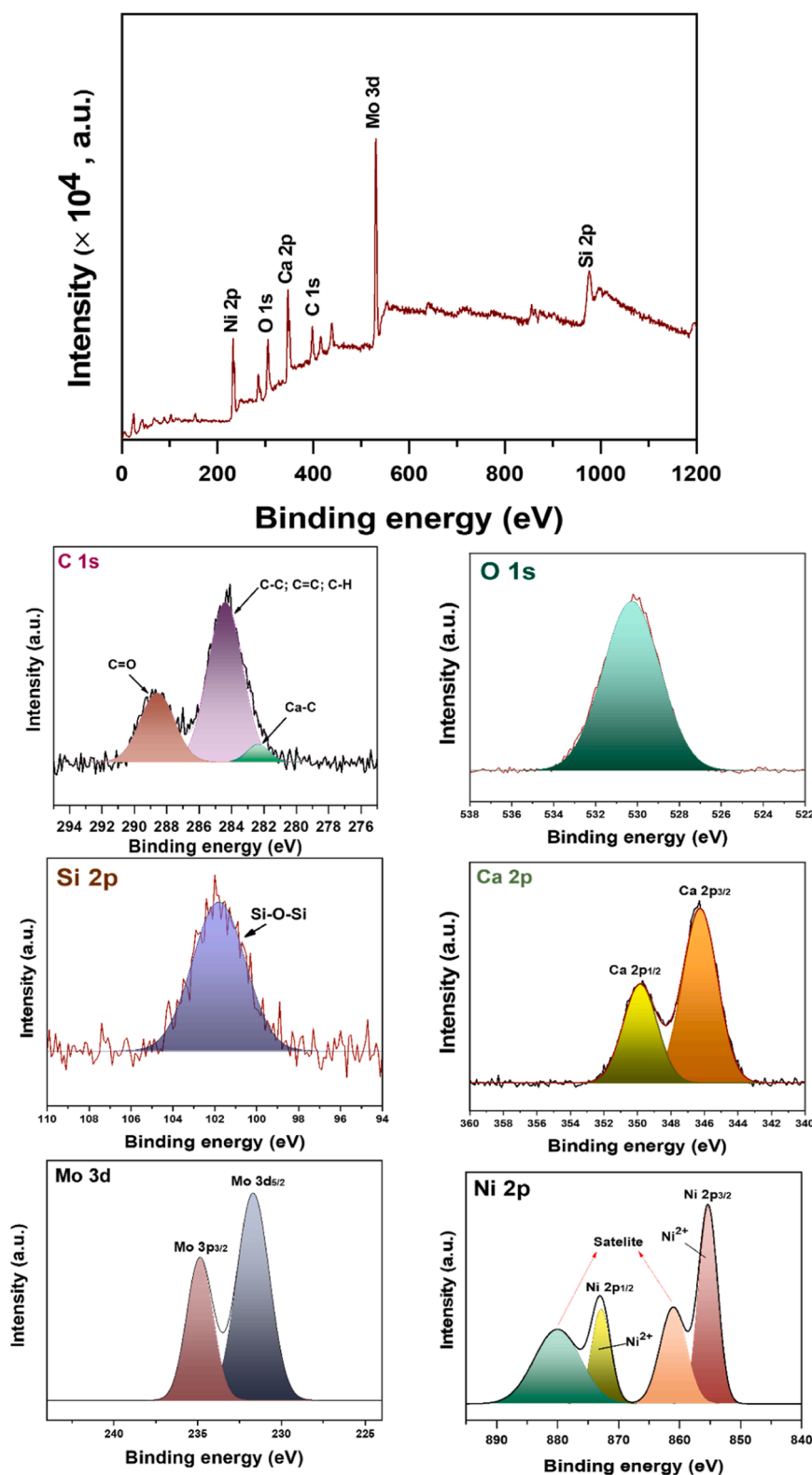


Fig. 8. XPS analysis of Ni/Mo-MWAC catalysts.

3.2. The catalytic activities study

3.2.1. Low temperature pyrolysis of waste lubricant oil (WLO)

The catalytic conversion of waste lubricant oil (WLO) over CaO-based catalysts shows clear improvements in both conversion and selectivity toward valuable liquid fuels. As shown in Fig. 10A, the blank

reaction resulted in a low conversion of 41.48%, whereas incorporation of metal-modified catalysts increased conversion to 67.03% (MWA), 66.53% (MWAC), 85.29% (Ni-MWAC), and 87.85% (Ni/Mo-MWAC). This trend confirms that the introduction of Ni and Ni/Mo active sites enhances the removal of heteroatoms and promotes cracking activity, consistent with recent findings on transition metal catalysts for efficient

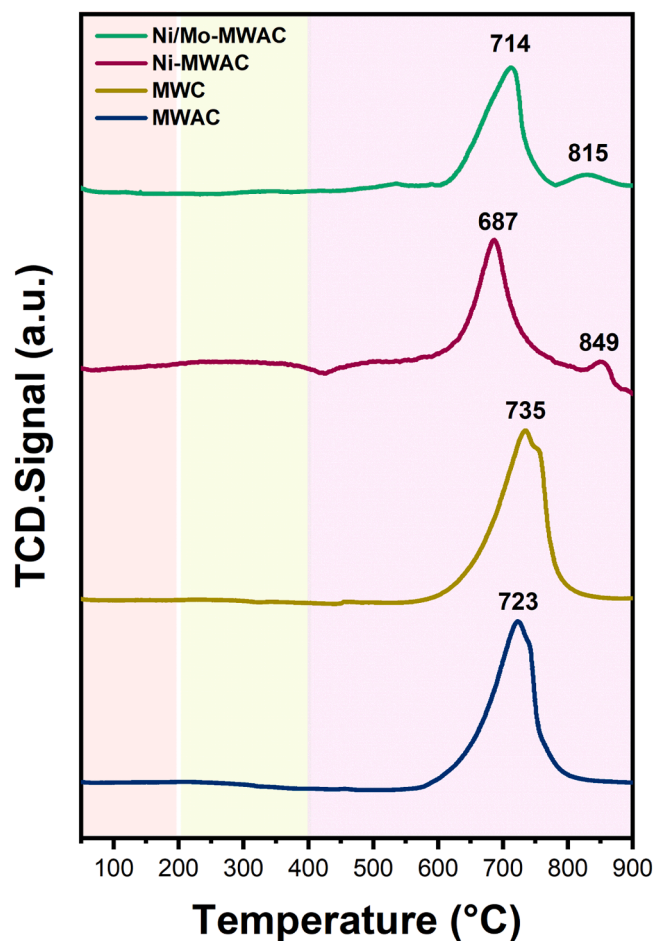


Fig. 9. TPD-CO₂ profiles for MWC and MWAC with different synthesis.

deoxygenation and hydrocarbon production from heavy feedstocks (Zhang et al., 2019).

Product phase distribution (Fig. 10B) shows that the blank reaction yields mostly gas (65.50%) and low liquid (14.60%). CaO base catalysts increase liquid yields (MWA: 26.54%, MWAC: 19.71%), and the addition of metals further increases liquid production. Ni/Mo-MWAC produces the highest liquid yield at 51.86%, with gas reduced to 25.70% and coke controlled at 22.44%. This demonstrates that Mo promotion improves Ni dispersion, allowing enhanced hydrogenation and controlled cracking that favors liquid fuel production and limits excessive gas formation. The addition of Mo increased the product yield, as observed for the Ni-MWAC catalyst from 31.21% to 51.86%, and simultaneously reduced the gas yield from 28.33% to 13.56%.

Liquid phase selectivity (Fig. 10C) shows that the blank feed is dominated by hydrocarbons (72.39%), with cyclic (14.78%) and oxygenate fractions. The basic oxide supports (MWAC) increase selectivity toward hydrocarbon and aromatic fractions by facilitating decarboxylation and decarbonylation pathways, a mechanism well established for basic CaO supports in upgrading heavy oils and bio oils (Hafiz et al., 2024b). Upon Ni incorporation, aromatic content rises to 15.30% for Ni-MWAC, while hydrocarbon content also increases, and oxygenates and cyclic species decrease. The addition of Mo as a promoter achieves a balanced hydrocarbon production of 76.78% while maintaining manageable aromatic and cyclic fractions, confirming that Mo improves Ni dispersion and increases the accessibility of active sites, reducing coke formation and enhancing stability (Swain and Patra, 2025).

Fig. 10(C) presents the results of low-temperature pyrolysis reactions for the blank, MWA, MWAC, Ni-MWAC, and Ni/Mo-MWAC samples. A

feedstock of 10 g of WLO was used, and the resulting masses of liquid, gas, coke, and unreacted feedstock are reported, with total conversion normalized to 100%. The Ni/Mo-MWAC catalyst produced the highest amount of liquid product at 5.22 g, whereas the sample without any catalyst showed 58.52% of the feedstock remaining unreacted. These results clearly demonstrate that the catalyst has a major influence on the conversion of WLO into jet-fuel.

Chain length distribution (Fig. 10D) is critical for jet-fuel suitability because conventional aviation kerosene falls in the C₈–C₁₆ range. The blank reaction produces mostly medium-chain hydrocarbons with limited distribution in the middle distillate range. Modification with CaO support (MWAC) shifts the distribution toward medium and long chains. Ni/Mo-MWAC produces a clear increase in C₁₃–C₁₆ (37.24%) and C₁₇–C₂₀ (15.27%) fractions, demonstrating that the catalyst effectively targets the jet-fuel relevant range (Tamim et al., 2024, 2025).

Basic CaO support promotes decarboxylation and decarbonylation of oxygenated intermediates, removing oxygen efficiently with minimal hydrogen consumption. Ni provides primary hydrogenation/dehydrogenation activity to cleave long-chain hydrocarbons into middle distillate components, and Mo enhances Ni dispersion while preventing sintering and coke deposition, increasing catalyst durability and selectivity toward jet-fuel range products (Widayat et al., 2024).

Table 3 presents the composition obtained from WLO as the feedstock. This study using waste lubricant oil from all type of motorcycle. Aromatic compounds and carboxylic acids dominate the content of the liquid reactant. Table 4 also provides the detailed composition of the liquid products obtained from the Ni/Mo-MWAC catalyst. Oxygenated compounds, such as carboxylic acids, alcohols, and cyclic species, were detected in the liquid product. However, the liquid is dominated by n-alkanes, including octadecane, pentadecane, and undecane, while the alkenes present include 1-hexadecene, 1-tridecene, and 1-decene.

3.2.2. Proposed deoxygenation mechanism of waste lubricant oil over Ni/Mo-MWAC catalysts

The low-temperature pyrolysis of waste lubricant oil (WLO) over Ni/Mo-MWAC proceeds through an integrated reaction network as illustrated in Fig. 11. WLO originates from a complex base-lubricant hydrocarbon matrix consisting mainly of long-chain paraffins, isoparaffins, naphthenic hydrocarbons, and alkyl aromatics, which undergo partial degradation during engine use. This results in WLO containing long-chain hydrocarbons, alkyl aromatics, naphthalene derivatives, and oxygenated degradation species (El-seesy et al., 2025).

The long-chain hydrocarbons, represented by heptadecane, octadecane, nonadecane, eicosane, and 2,6,10,14-tetramethylhexadecane, are mainly converted via catalytic cracking and β -scission to produce shorter hydrocarbons. Based on the GC–MS results, the main products are n-alkanes and 1-alkenes, including heptane, octane, nonane, decane, undecane, dodecane, pentadecane, heptadecane, octadecane, 1-nonene, 1-decene, 1-undecene, 1-tridecene, and 1-hexadecene. Oxygenated degradation species, such as hexadecanoic acid, octadecenoic acid, and octadecanoic acid, undergo deoxygenation through decarboxylation and decarbonylation, producing hydrocarbons like pentadecane, heptadecane, and 1-heptadecene with the evolution of CO₂, CO, and H₂O.

The catalytic performance of Ni/Mo-MWAC arises from the synergistic functions of CaO, Mo species, and NiO, as shown in the bottom “catalyst surface mechanism” row of the infographic. CaO basic sites, derived from calcined CaCO₃, facilitate adsorption and activation of acidic oxygenates, promoting decarboxylation (Hafiz et al., 2021). Mo species act as Ni dispersion and stabilization promoters, anchoring NiO nanoparticles on the support, preventing agglomeration, and ensuring a high density of active Ni sites (Kim et al., 2018). NiO contributes to hydrogen transfer, cracking, isomerization, and stabilization of radical or olefinic intermediates, favoring the formation of shorter, branched, and more stable hydrocarbons (Yuan et al., 2026).

Secondary reactions may further modify the product distribution. Olefinic intermediates can undergo hydrogen transfer to form n-alkanes

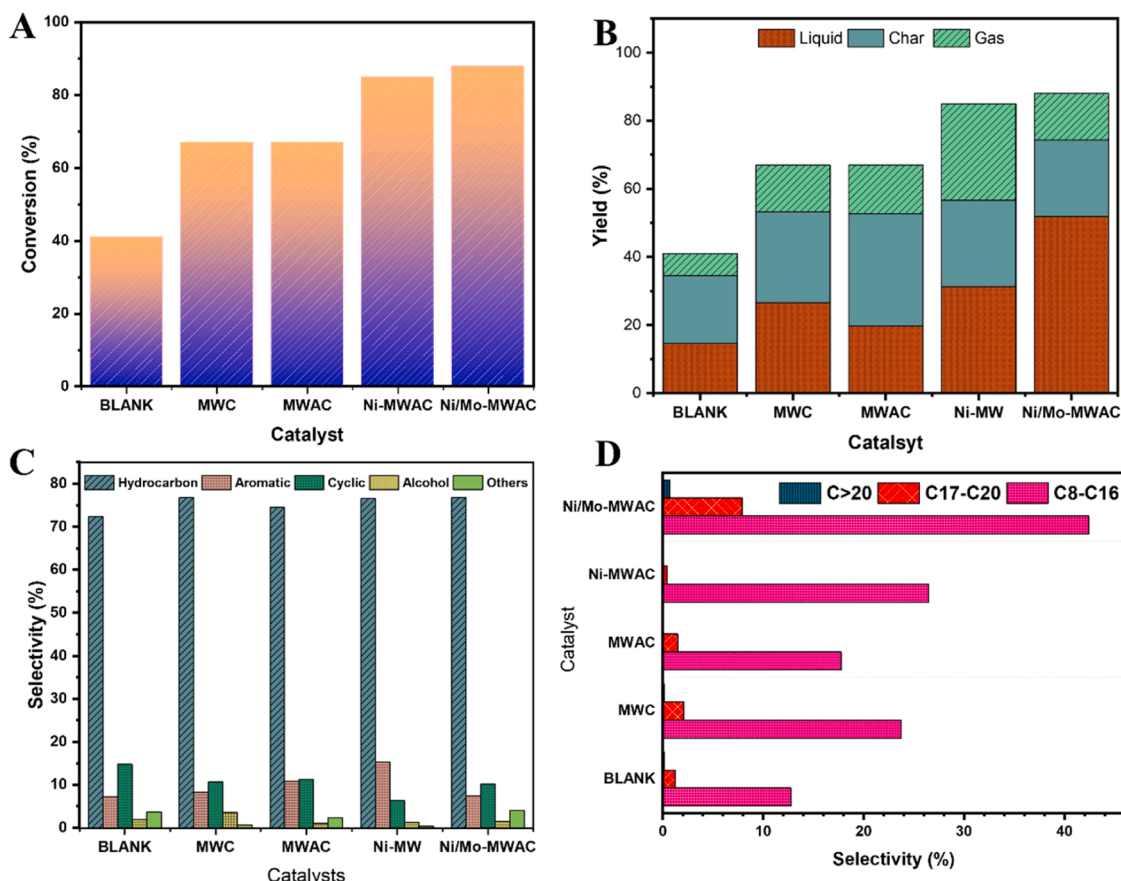


Fig. 10. Conversion (A); Selectivity product (B); product distribution (C); Carbon distribution (D) of waste lubricant oil over MWA, MWC, MWAC, Ni-MWAC, Ni/Mo-MWAC catalysts.

Table 3
Chemical composition of WLO.

Retention Time	Peak Area (%)	Compounds	Chemical Formula	Molar Mass (g/mol)
11.922	2.6	Benzene, 1-ethyl-3-methyl-	C ₉ H ₁₂	120
12.904	7.68	Benzene, 1,2,4-Trimethyl-	C ₉ H ₁₂	120
13.71	2.31	Benzene, 1-ethyl-3-methyl	C ₉ H ₁₂	120
14.671	1.47	Benzene, 1-ethyl-3,5-dimethyl	C ₁₀ H ₁₄	134
15.341	1.83	Benzene, 4-ethyl-1,2-dimethyl	C ₁₀ H ₁₄	134
16.095	1.35	Benzene, 1,2,3,4-tetramethyl	C ₁₀ H ₁₄	134
16.207	2.52	Benzene, 1,2,4,5-tetramethyl	C ₁₀ H ₁₄	134
17.759	2.8	Naphthalene	C ₁₀ H ₈	128
26.366	1.01	Octadecane	C ₁₈ H ₃₈	254
27.801	1.14	Heptadecane	C ₁₇ H ₃₆	240
29.165	2.75	Octadecane	C ₁₈ H ₃₈	254
29.57	14.24	Hexadecanoic acid	C ₁₇ H ₃₄ O ₂	270
30.47	8.11	Nonadecane	C ₁₉ H ₄₀	268
31.715	13.01	Eicosane	C ₂₀ H ₄₂	282
31.816	9.78	11-Octadecenoic acid	C ₁₉ H ₃₆ O ₂	296
31.885	7.6	10-Octadecenoic acid	C ₁₉ H ₃₆ O ₂	296
32.095	2.01	Octadecanoic acid	C ₁₉ H ₃₆ O ₂	296
32.184	3.03	Hexadecane, 2,6,10,14-tetramethyl	C ₂₀ H ₄₂	282
32.911	10.06	Heneicosane	C ₂₁ H ₄₄	296
34.122	4.7	Eicosane	C ₂₀ H ₄₂	282

or isomerization to produce branched hydrocarbons such as 4-methyldecane, 4-methylnonane, and 2,4-dimethylundecane. Cyclization reactions may also generate cyclic products, including methylcyclohexane, 4,4-dimethylcyclohexene, 1-methylcyclopentane, and cyclohexane. In addition, limited aromatization may contribute to the formation of alkylbenzenes and related aromatic compounds, such as 1,3,5-trimethylbenzene, 1,2,4-trimethylbenzene, 1,2,3,4-tetramethylbenzene, and 1-methylnaphthalene. Naphthalene is considered to originate mainly from the aromatic fraction already present in the WLO feed. Therefore, the proposed pathway in Fig. 10 should be regarded as a simplified interpretation based on the observed GC-MS product distribution rather than a direct identification of all intermediates.

3.2.3. Reusability of Ni/Mo-MWAC catalyst

The reusability of the Ni/Mo-MWAC catalyst was evaluated over three consecutive catalytic cycles, consisting of the initial run followed by three reuse without regeneration. After each reaction, the catalyst was separated from the reaction mixture, recovered, and reused under the same operating conditions. As shown in Fig. 12, the fresh Ni/Mo-MWAC catalyst produced 6.34 g of liquid product, corresponding to a liquid yield of 63.28 wt% and a WLO conversion of 83.45%. In the first reuse, 4.99 g of liquid product was obtained, representing a liquid yield of 57.45 wt%, while the WLO conversion reached 70.67%. In the second reuse, the catalyst produced 3.47 g of liquid product with a liquid yield of 41.23 wt% and a WLO conversion of 65.84%. During the third reuse, 3.00 g of liquid product was obtained, corresponding to a liquid yield of 37.65 wt%, while the WLO conversion was 56.71%. These results confirm that Ni/Mo-MWAC remained catalytically active over four consecutive reaction, although both liquid yield and WLO conversion gradually decreased upon repeated use.

Table 4

Chemical composition of WLO after deoxygenation using Ni/Mo MWAC catalyst.

Retention Time	Peak Area (%)	Compounds	Chemical Formula	Molar Mass (g/mol)
1.468	0.75	1-Butanol, 2-methyl	C ₅ H ₁₂ O	88
1.555	0.87	Oxirane, (2-methylbutyl)	C ₇ H ₁₄ O	114
1.607	1.29	Heptane	C ₇ H ₁₆	100
1.684	0.39	Cyclohexane, methyl	C ₇ H ₁₄	98
1.73	0.58	Cyclohexene, 4,4-dimethyl	C ₈ H ₁₄	110
1.761	0.61	Heptane, 2-methyl	C ₈ H ₁₈	114
1.8	0.69	Pentane, 2,2,4-trimethyl	C ₈ H ₁₈	114
1.835	0.99	2-Octene	C ₈ H ₁₆	112
1.866	1.05	Octane	C ₈ H ₁₈	114
2.067	0.37	Cyclopropane, 1,2-dimethyl-3-pentyl	C ₁₀ H ₂₀	140
2.134	0.73	Octane, 4-methyl	C ₉ H ₂₀	128
2.172	0.45	Octane, 3-methyl	C ₉ H ₂₀	128
2.275	0.87	1-Nonene	C ₉ H ₁₈	126
2.322	2.02	Nonane	C ₉ H ₂₀	128
2.535	1.18	Octane, 2,6-dimethyl	C ₁₀ H ₂₂	142
2.691	1.02	5-Undecene, 9-methyl	C ₁₂ H ₂₄	168
2.741	1.17	Nonane, 4-methyl	C ₁₀ H ₂₂	142
2.803	1.32	Benzene, 1,3,5-trimethyl	C ₉ H ₁₂	120
2.85	0.47	Benzene, 1,2,4-trimethyl	C ₉ H ₁₂	120
2.925	1.01	Cyclopropane, 1-methyl-2-(1-methylpentyl)	C ₁₀ H ₂₀	140
2.969	2.38	1-Decene	C ₁₀ H ₂₀	140
3.043	4.27	Decane	C ₁₀ H ₂₂	142
3.175	0.68	1-Decene, 3,4-dimethyl	C ₁₂ H ₂₄	168
3.335	0.49	Benzene, 1,2,4-trimethyl	C ₉ H ₁₂	120
3.404	0.55	2-Hexene, 3,4,4-trimethyl	C ₉ H ₁₈	126
3.58	1.02	Benzene, (1,2,2-trimethylpropyl)	C ₁₂ H ₁₈	162
3.647	1.23	Benzene, (1-ethyloctadecyl)	C ₂₆ H ₄₆	178
3.875	1.14	4-Decene, 5-methyl	C ₁₁ H ₂₂	154
3.941	2.69	1-Undecene	C ₁₁ H ₂₂	154
4.034	4.29	Undecane	C ₁₁ H ₂₄	154
4.303	0.4	1,3-Cyclopentadiene, 1,2,3,4-tetramethyl-5-methylene	C ₁₀ H ₁₄	134
4.356	0.85	Benzene, 1,2,3,4-tetramethyl	C ₁₀ H ₁₄	134
4.475	0.67	3-Undecene, 7-methyl	C ₁₂ H ₂₄	168
4.667	0.76	1-Octanol, 2-butyl	C ₁₂ H ₂₆ O	186
4.855	0.55	Undecane, 3-methyl	C ₁₂ H ₂₆	170
5.038	0.72	1-Undecene, 2-methyl	C ₁₂ H ₂₄	168
5.114	2.84	Cyclopropane, nonyl	C ₁₂ H ₂₄	168
5.175	1.2	Naphthalene	C ₁₀ H ₈	128
5.221	4.08	Dodecane	C ₁₂ H ₂₆	170
5.28	1.1	Benzaldehyde, 4-(1-methylethyl)	C ₁₀ H ₁₂ O	148
5.391	0.91	Tridecane, 6-methyl	C ₁₄ H ₃₀	198
5.671	0.51	Cyclopentane, 3-hexyl-1,1-dimethyl	C ₁₃ H ₂₆	182
5.77	1.07	Cyclopentane, 1-hexyl-3-methyl	C ₁₂ H ₂₄	168
5.906	1.18	Undecane, 2,4-dimethyl	C ₁₃ H ₂₈	184
5.968	0.57	Dodecane, 4-methyl	C ₁₃ H ₂₈	184
6.025	0.87	Dodecane, 2-methyl	C ₁₃ H ₂₈	184
6.117	0.96	Dodecane, 3-methyl	C ₁₃ H ₂₈	184
6.313	0.53	1-Heptene, 2,6-dimethyl	C ₉ H ₁₈	126
6.398	2.43	1-Tridecene	C ₁₃ H ₂₆	182
6.443	0.99	5-Undecene, 2-methyl	C ₁₂ H ₂₄	168
6.509	4.22	Heptadecane	C ₁₇ H ₃₆	240
6.573	1.06	Naphthalene, 1-methyl	C ₁₁ H ₁₀	142
7.115	0.4	Cyclohexane, hexyl	C ₁₂ H ₂₄	168
7.159	0.4	Tetradecane	C ₁₄ H ₃₀	198
7.325	0.49	Eicosane	C ₂₀ H ₄₂	282
7.42	0.5	Tridecane, 3-methyl	C ₁₄ H ₃₀	198
7.501	0.44	Dodecane, 2,7,10-trimethyl	C ₁₅ H ₃₂	210
7.621	0.86	Tridecane, 7-methylene	C ₁₄ H ₂₈	196
7.705	2.38	1-Tridecene	C ₁₃ H ₂₆	182
7.814	4.38	Heptadecane	C ₁₇ H ₃₆	240

Table 4 (continued)

Retention Time	Peak Area (%)	Compounds	Chemical Formula	Molar Mass (g/mol)
7.862	0.64	Decane, 1,1'-oxybis	C ₂₀ H ₄₂ O	298
8.009	0.45	Dodecane, 2,6,10-trimethyl	C ₁₅ H ₃₂	212
8.476	1.33	Cyclohexane, (1-hexyltetradecyl)	C ₂₆ H ₅₂	244
8.551	0.79	Tetradecane, 4-methyl	C ₁₅ H ₃₂	212
8.615	0.73	Tricosane	C ₂₃ H ₄₈	324
8.715	0.49	Tetradecane, 3-methyl	C ₁₅ H ₃₂	212
8.909	0.66	cllopentane, 3-hexyl-1,1-dimethyl	C ₁₃ H ₂₆	182
8.992	1.64	3-Hexadecene	C ₁₆ H ₃₂	224
9.095	3.53	Pentadecane	C ₁₅ H ₃₂	212
9.67	0.66	Decane, 5-propyl	C ₁₃ H ₂₈	184
9.87	0.5	Tridecane, 2-methyl	C ₁₄ H ₃₀	198
10.155	0.4	Cyclopentane, 3-hexyl-1,1-dimethyl	C ₁₃ H ₂₆	182
10.233	0.97	1-Hexadecene	C ₁₆ H ₃₂	224
10.327	2.31	Pentadecane	C ₁₅ H ₃₂	212
10.903	0.6	Undecane, 2,5-dimethyl	C ₁₃ H ₂₈	184
11.508	1.54	Pentadecane	C ₁₅ H ₃₂	212
11.572	0.52	Hexadecane, 2,6,10,14-tetramethyl	C ₂₀ H ₄₂	282
12.634	0.83	Octadecane	C ₁₈ H ₃₈	254
13.711	0.39	Octadecane	C ₁₈ H ₃₈	254
14.74	0.2	Octadecane	C ₁₈ H ₃₈	254
15.726	0.09	Nonadecane	C ₁₉ H ₄₀	268

3.3. Analysis of coke

The structural characteristics of the spent Ni/Mo-MWAC catalyst were evaluated using XRD and FTIR analysis. The XRD pattern of the spent catalyst (Fig. 13A) retained the main reflections assigned to CaMoO₄ and NiO, indicating that the principal crystalline phases were largely preserved after the catalytic reaction (Mustafa et al., 2025; Murguía-ortiz et al., 2021). Peak intensity changed were observed compared with the fresh catalyst, which may be associated with surface coverage by carbonaceous deposits, hydration or carbonation of Ca-containing species, and changes in crystallinity during the reaction. Because coke is generally amorphous, its deposition cannot be conclusively identified from XRD alone (Kouider et al., 2024).

The FTIR spectrum (Fig. 13B) of the spent catalyst exhibited a broad absorption band around 3408 cm⁻¹, attributed to O–H stretching vibrations of adsorbed water or surface hydroxyl groups. The band at approximately 2924 cm⁻¹ corresponds to aliphatic C–H stretching vibrations, indicating the presence of residual hydrocarbon-derived species on the catalyst surface. The absorption band around 1458 cm⁻¹ is mainly associated with carbonate species, which may form through the interaction of CaO with CO₂ and oxygen-containing reaction products. Additional bands at approximately 1078 and 813 cm⁻¹ are related to vibrations of carbonate and metal-oxygen-containing species (Tamim et al., 2024). The presence of aliphatic C–H species on the spent catalyst supports the occurrence of hydrocarbon-derived surface deposition, which may contribute to active-site coverage and the progressive decrease in catalytic performance during reuse.

The EDX mapping of the spent catalyst after reaction was used to analyze the distribution of elements on the catalyst, as shown in Fig. 14. The spectrum confirmed the presence of carbon, oxygen, molybdenum, calcium, magnesium, nickel, and silicon. Carbon was the dominant element (60.4%), indicating the presence of coke formation and deposition on the catalyst surface during the pyrolysis reaction (Tamim et al., 2024). In contrast, molybdenum and nickel were still detected after the reaction, indicating that the active metals remained on the catalyst surface. However, the relative surface Mo content decreased by approximately 73% compared with that of the fresh catalyst, suggesting a substantial loss of exposed Mo species. This reduction may be

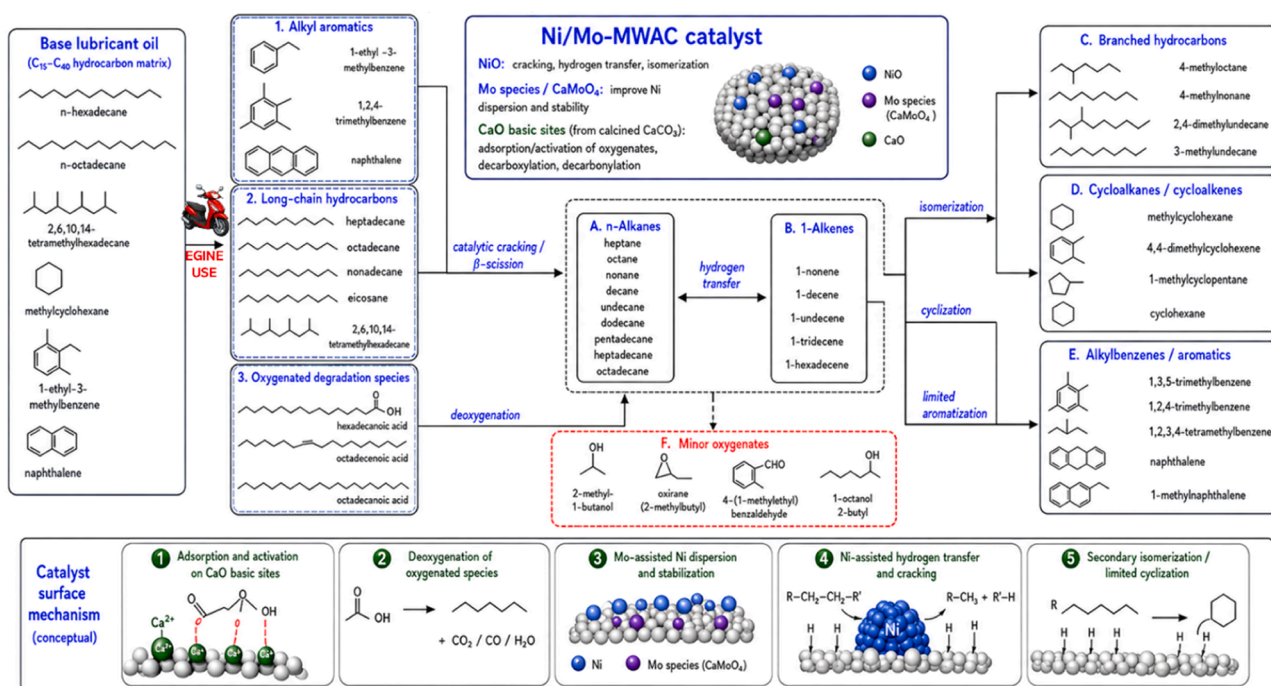


Fig. 11. Proposed mechanism for conversion waste lubricant oil for jet-fuel product.

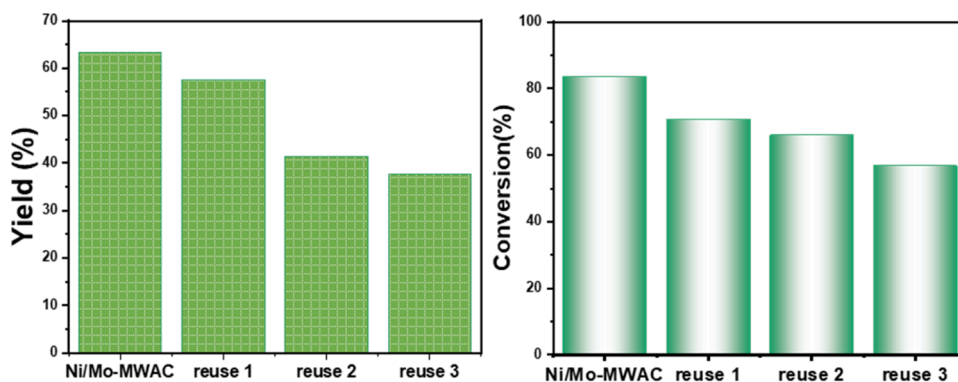


Fig. 12. Yield and conversion reusability result of Ni/Mo-MWAC catalyst.

attributed to partial metal leaching during the pyrolysis process (Alexey Kurlov et al., 2025)

3.4. Response surface methodology

Response Surface Methodology (RSM) was applied to evaluate the influence of key operating parameters on the reaction performance. Table 5 presents the 15-run RSM experimental matrix and the corresponding reaction results for WLO upgrading over the Ni/Mo-MWAC catalyst. The process variables, including temperature, catalyst loading, and reaction time, were systematically varied to evaluate their effects on the catalytic performance and product distribution. The following is the polynomial equation used in RSM data processing. Eq. (10) means where Y is the predicted liquid yield (%), A is the coded reaction temperature, B is the coded catalyst loading, and C is the coded reaction time. The coefficients β_0 , β_i , β_{ii} , and β_{ij} represent the intercept, linear, quadratic, and interaction coefficients, respectively, while e is the residual error.

$$Y = 27.03 + 1.32A - 0.5043B + 0.6992C + 1.23AB + 20.01AC + 3.41BC - 2.33A^2 - 19.02B^2 + 16.19C^2 \quad (10)$$

The response surface and contour plots in Fig. 15 illustrate the combined effects of temperature with catalyst loading and reaction time on the predicted liquid yield. In both interactions, liquid yield increased as the temperature approached approximately 380 °C, followed by a decline at higher temperatures, confirming a pronounced non-linear temperature effect (Rehman et al., 2026). The temperature-catalyst loading interaction, the highest predicted liquid yield was obtained at an intermediate temperature and relatively low-to-moderate catalyst loading, whereas excessive catalyst loading reduced the response. This behavior may be associated with intensified secondary cracking or further conversion of liquid products into lighter compounds at higher catalyst amounts. A similar curved profile was observed for the temperature-reaction time interaction, where the predicted yield was highest at approximately 380 °C and shorter-to-moderate reaction times, while prolonged reaction time decreased the liquid yield.

The quadratic ANOVA results presented in Table 6 indicate that the model was statistically, with an F-value of 12.60 and a p-value of 0.0061. Among the model terms, the interaction between temperature and reaction time (AC), the quadratic catalyst loading term (B^2), and the quadratic reaction time term (C^2) affected liquid yield, with p-values of 0.0012, 0.0018, and 0.0037, respectively. In contrast, the individual

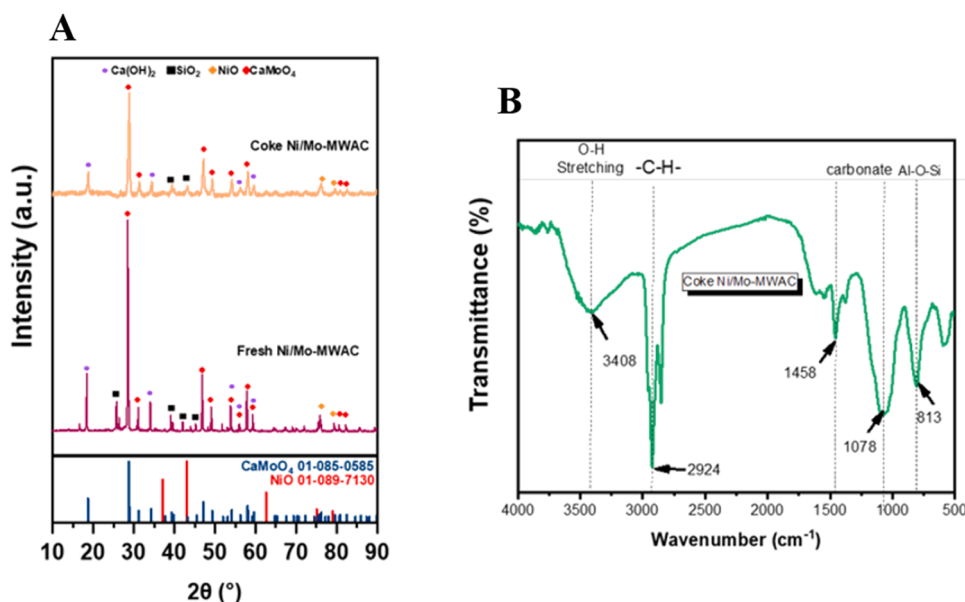


Fig. 13. XRD result (a); FTIR result (B) coke Ni/Mo-MWAC.

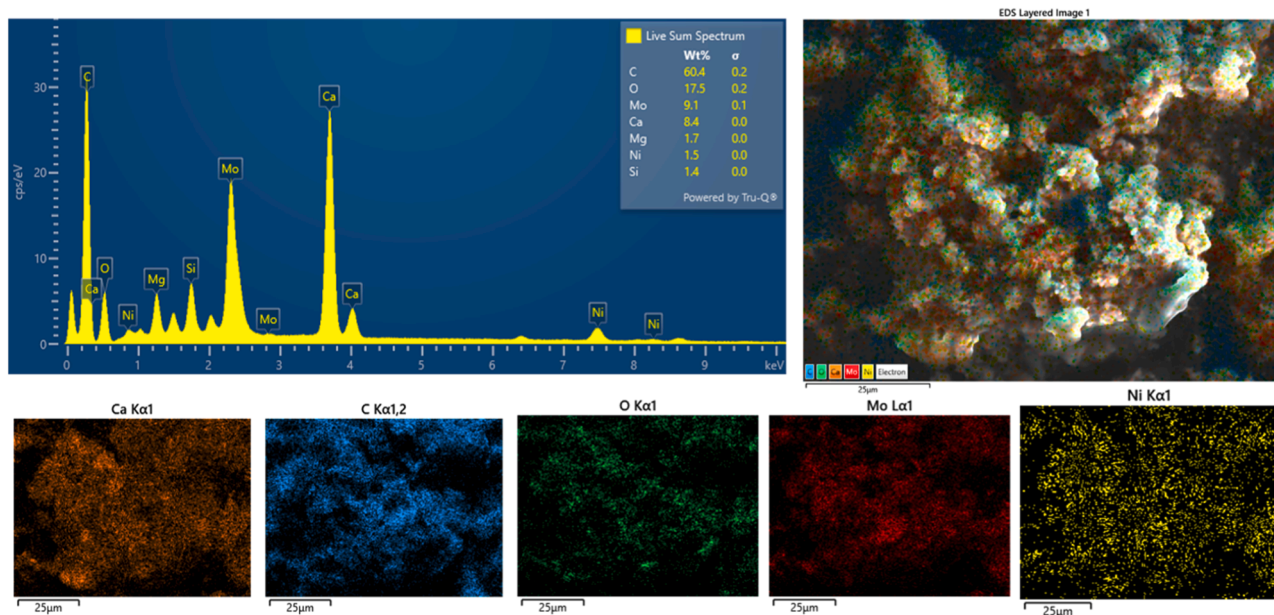


Fig. 14. EDS of spent catalyst.

linear effects of temperature, catalyst loading, and reaction time, as well as the AB and BC interaction terms and the quadratic temperature term, were not statistically significant at the 95% confidence level. The lack-of-fit test gave a p-value of 0.0503, which is slightly above 0.05, indicating that the lack of fit was not statistically significant, although the result was close to the significance threshold. The individual linear and quadratic effect analysis (Table 7) showed that none of the isolated factor terms reached statistical significance. This indicates that the predictive capability of the model was primarily associated with interaction and curvature effects rather than the individual main effects.

The ANOVA results in Table 7 indicate that none of the linear or quadratic terms affected liquid yield at the 95% confidence level ($p > 0.05$). The linear effect of catalyst loading showed the largest contribution ($F = 3.2143$, $p = 0.1108$), followed by reaction time ($F = 2.6624$, $p = 0.1414$), whereas the linear effect of temperature was the weakest ($F = 0.0146$, $p = 0.9068$). Thus, catalyst loading and reaction time had

relatively greater influences on liquid yield, although their effects were not statistically significant within the studied range (Sulaswatty et al., 2026).

The regression analysis in Table 8 shows that none of the linear or quadratic terms affected liquid yield at the 95% confidence level ($p > 0.05$). The linear and quadratic temperature terms showed comparable effects, with p-values of 0.3705 and 0.3694, respectively (Rehman et al., 2026). The effects of catalyst loading and reaction time were also statistically insignificant. Moreover, all 95% confidence intervals crossed zero, confirming that the estimated regression coefficients were not significantly different from zero within the studied ranges (Singmai et al., 2026).

The observed versus predicted plot in Fig. 16(A) shows that the predicted liquid yields generally follow the experimental trend, although noticeable deviations between the observed and predicted values are present. This indicates that the quadratic model provides only

Table 5

Experimental matrix of 15-run RSM using BBD.

Experiment	Temperature (°C)	Catalysts loading (%)	Reaction Time (h)	Yield liquid (%)
RSM-1	360	1	2	6.81
RSM-2	400	1	2	23.55
RSM-3	360	5	2	5.22
RSM-4	400	5	2	36.61
RSM-5	360	3	1	3.69
RSM-6	400	3	1	32.77
RSM-7	360	3	3	7.22
RSM-8	400	1	3	32.25
RSM-9	380	5	1	53.35
RSM-10	380	3	1	17.76
RSM-11	380	1	3	68.44
RSM-12	380	5	3	23.55
RSM-13	380	3	2	25.68
RSM-14	380	3	2	29.03
RSM-15	380	3	2	26.36

a moderate representation of the experimental response, which is consistent with the relatively low coefficient of determination ($R^2 = 0.4845$). The dispersion of several data points around the regression line suggests that experimental variability remains relatively high under the investigated operating conditions. The Pareto chart of standardized effects in Fig. 16(B) indicates that none of the investigated factors exceeded the statistical significance threshold ($p = 0.05$). Among the evaluated variables, the linear effect of catalyst loading exhibited the largest standardized effect ($|t| = 1.7928$), followed by the linear effect of reaction time ($|t| = 1.6317$). The quadratic effect of temperature showed a relatively smaller contribution ($|t| = 0.9511$), while the quadratic effects of catalyst loading and reaction time produced even weaker influences. The linear effect of temperature had the smallest standardized

Table 6

ANOVA for Quadratic model of temperature, catalyst loading, and reaction time on yield liquid in the low-temperature pyrolysis of WLO.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	4184.01	9	464.89	12.60	0.0061
A-Temperature	13.96	1	13.96	0.3784	0.5654
B-Loading Catalyst	2.03	1	2.03	0.0551	0.8237
C-Reaction Time	3.91	1	3.91	0.1060	0.7579
AB	6.02	1	6.02	0.1631	0.7030
AC	1601.27	1	1601.27	43.40	0.0012
BC	46.57	1	46.57	1.26	0.3123
A ²	20.03	1	20.03	0.5428	0.4944
B ²	1336.03	1	1336.03	36.21	0.0018
C ²	967.64	1	967.64	26.22	0.0037
Residual	184.50	5	36.90		
Lack of Fit	178.26	3	59.42	19.05	0.0503
Pure Error	6.24	2	3.12		
Cor Total	4368.51	14			

Table 7

ANOVA results for the effects of temperature, catalyst loading, and reaction time on yield liquid in the low-temperature pyrolysis of WLO.

Factor	Sum of Square	df	Mean Square	F	P
Temperature (L)	18.00	1	18.000	0.0146	0.9068
Temperature (Q)	1114.67	1	1114.673	0.9047	0.3694
Catalyst Loading (L)	3960.50	1	3960.500	3.2143	0.1108
Catalyst Loading (Q)	872.83	1	872.827	0.7084	0.4244
Reaction Time (L)	3280.50	1	3280.500	2.6624	0.1414
Reaction Time (Q)	290.83	1	290.827	0.2360	0.6401
Error	9857.25	8	1232.156	–	–
Total	19,122.40	14	–	–	–

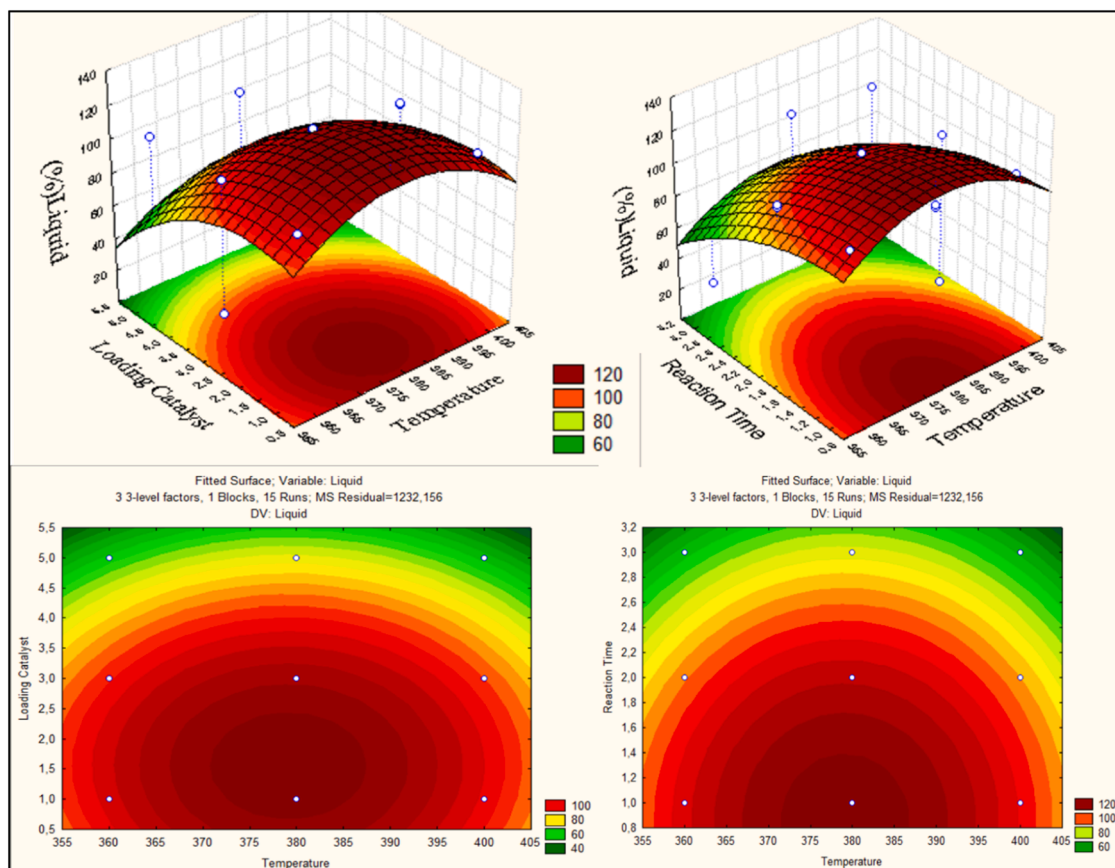
**Fig. 15.** Fitted surface of yield liquid.

Table 8
RSM coefficients and significance.

Factor	Std. Err	T-value	p-value	−95% Cnf. Limit	+95% Cnf. Limit
Intercept	6596.87	−0.9291	0.3800	−21,341.5	9083.33
Temperature (L)	34.71	0.9488	0.3705	−47.1	112.99
Temperature (Q)	0.046	−0.9511	0.3694	−0.10	0.062
Catalyst Loading (L)	28.10	0.4249	0.6821	−52.9	76.73
Catalyst Loading (Q)	4.57	−0.8416	0.4244	−14.4	6.69
Reaction Time (L)	74.12	0.2058	0.8421	−155.7	186.17

effect ($|t| = 0.1209$). These results indicate that, although catalyst loading and reaction time exerted relatively greater influence on liquid yield than the other variables, none of the investigated factors significantly affected the response within the studied operating range (Salimian et al., 2026).

The predicted-value and desirability profiles shown in Fig. 17 indicate that the response is governed by the combined effects of temperature, catalyst loading, and reaction time. The predicted liquid yield increases as the temperature rises from 360 to approximately 380 °C, after which it decreases at higher temperatures, indicating a curved temperature effect and an optimum near 380 °C. Increasing catalyst loading does not continuously improve the response; the predicted yield remains relatively high at low-to-moderate catalyst loadings and gradually decreases at higher loadings (Rehman et al., 2026). A similar trend is observed for reaction time, where prolonged reaction beyond the

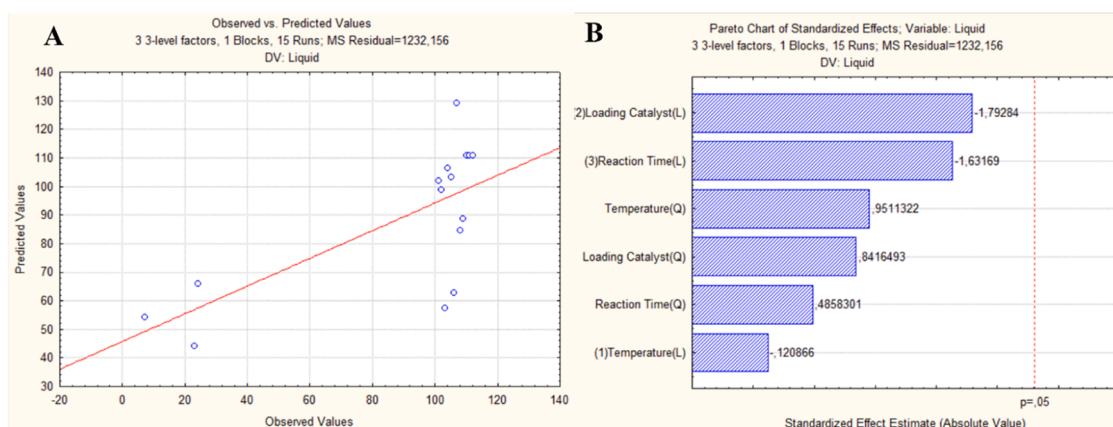


Fig. 16. Observed-predicted value(A); Pareto Chart (B) variable yield liquid.

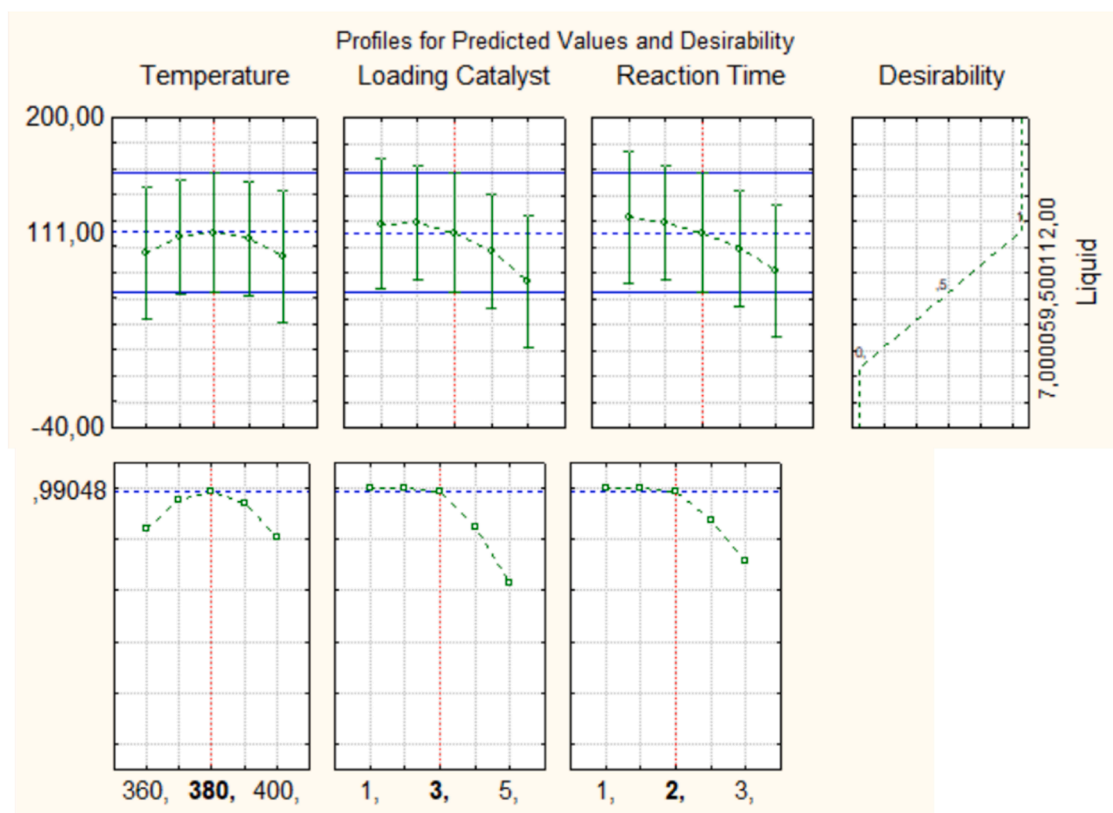


Fig. 17. Predicted values and desirability of yield liquid.

selected region reduces the predicted liquid yield, possibly because of secondary cracking and the conversion of liquid products into lighter compounds. The optimization profile identifies approximately 380 °C, 3% catalyst loading, and 2 h reaction time as the selected operating conditions, with an overall desirability of about 0.9905. These results indicate that the optimum liquid yield is obtained through balanced operating conditions rather than by maximizing catalyst loading or reaction time individually (Buasri et al., 2024).

4. Conclusion

The NiMo/MWAC catalyst derived from Aloe vera-modified marble waste showed the best performance in WLO upgrading, achieving 87.85% conversion and a maximum liquid yield of 51.86 wt%. The Aloe vera-assisted hydrothermal treatment transformed the irregular MWC aggregates into more spherical and relatively uniform particles, facilitating the distribution of Ni and Mo species on the Ca-based support. Characterization confirmed the formation of CaO, NiO, and CaMoO₄ phases. The improved activity was associated with CaO basic sites, the cracking and hydrogen-transfer functions of NiO, and the role of Mo in dispersing and stabilizing Ni species. The liquid product was dominated by hydrocarbons, with a substantial C₈–C₁₆ fraction. RSM analysis identified reaction temperature as the most influential variable, while the interaction between catalyst loading and reaction time also significantly affected liquid yield. The NiMo/MWAC catalyst offers a promising approach for converting marble waste and WLO into hydrocarbon-rich liquid products.

CRediT authorship contribution statement

Trias Alzatory Eryyada: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Anjas Bimanyu:** Visualization, Investigation, Formal analysis, Data curation. **Ingelia Yuan Fernanda:** Validation, Investigation, Data curation. **Stella Jovita:** Validation, Investigation, Data curation. **Khawiyatur Riv'ah Agustina:** Validation, Investigation, Data curation. **Brilliant Umara le Monde:** Writing – review & editing, Validation, Resources. **Reva Edra Nugraha:** Validation, Formal analysis. **Holilah Holilah:** Validation, Methodology. **Nurul Asikin-Mijan:** Writing – review & editing, Validation, Supervision, Methodology. **Yun Hin Taufiq-Yap:** Writing – original draft, Validation, Supervision, Resources. **Didi Dwi Anggoro:** Writing – review & editing, Supervision, Resources. **Didik Prasetyoko:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. **Eko Santoso:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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