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Synthesis and Evaluation of a Dual-Structured Ni-Nano Silica/CoMo-Al_2O_3 Catalyst for Enhanced Sulfur Removal from Kerosene via Advanced Hydrodesulfurization Techniques

Abdulrahman Abdulsttar Jber[1] , Aysar T. Jarullah[1] , Halla M. Hussein[2]
[1] Chemical Engineering Department, College of Engineering, Tikrit University, Tikrit, Iraq
2 Petroleum Research and Development Center, Ministry of Oil, Baghdad, Iraq.

Corresponding author: Aysar T. Jarullah
E-mail: (a.t.jarullah@tu.edu.iq)
Chemical Engineering Department, College of Engineering, Tikrit University, Tikrit, Iraq

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Abstract
This study examines the use of a new composite catalyst made of Ni-Nano silica coated on CoMo/Al_2O_3 to remove sulfur compounds from kerosene by hydrodesulfurization (HDS). The technological and environmental difficulties caused by sulfur in fuels, including its role in acid rain and catalyst poisoning, are the driving forces. The Ni/Nano-silica composite was created by synthesizing a nanosilica support using the sol-gel process and impregnating it with nickel nitrate. After that, this composite was physically combined with ground CoMo/Al_2O_3, and with bentonite that used as a binder to form it. The catalyst's mesoporous structure (10.85 nm) showed a uniform dispersion of active metals, and large surface area (198.13 m^2/g, compared with that used in industrial refinery) and all other tests, mainly XRD, FTIR, SEM-EDX, and BET investigations. A laboratory-scale hydrotreating unit with variable temperatures (315–471°C), hydrogen pressures (18–39 bar), and liquid hourly space velocities (1–4 h^{-1}) with constant H_2/Oil ratio at was used to test the catalyst. The findings showed that these factors had a significant impact on the efficiency of sulfur removal. Where at 471°C, 39 bar, and LHSV of 1 h^{-1}, the highest desulfurization performance of 99.47% was attained.. The work demonstrates that Ni-Nano silica-coated CoMo/Al_2O_3 is a successful method for deep desulfurization of kerosene, providing a viable path toward the production of environmentally acceptable ultra-low sulfur fuels.

keywords: Hydrodesulfurization (HDS), Ni-Nano Silica, CoMo/Al_2O_3 Catalyst, Sulfur Removal.

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1. INTRODUCTION

The presence of sulfur compounds in petroleum-derived fuels such as kerosene presents significant environmental and operational challenges. During combustion, these sulfur-containing compounds are oxidized to sulfur oxides, contributing to air pollution, acid rain, and public health hazards including respiratory illnesses and cardiovascular problems.[1] Additionally, sulfur compounds accelerate the corrosion of pipelines, refinery equipment, and engine parts, and they significantly poison and deactivate catalysts used in oil refining processes. In response, stringent global regulations have been established to reduce sulfur levels in transportation fuels to ultra-low thresholds [2].

Kerosene, in particular, plays a pivotal role in both domestic and industrial applications and is a critical fuel for jet aviation. However, it contains substantial concentrations of refractory sulfur species such as benzothiophenes and their alkylated derivatives [3] , which are notably resistant to conventional hydrodesulfurization (HDS) processes. This necessitates the development of more advanced catalytic systems capable of achieving deeper desulfurization under practical operating conditions[4].

Catalytic materials must be coated in order to increase their performance, stability, and lifespan under demanding operating settings. It maintains the catalyst's surface area high, shields against heat and chemical degradation, and helps prevents metal nanoparticle aggregation [5]. Additionally, coating provides constant active site distribution, minimizes catalyst deactivation, and increases metal–support interaction. Because of these advantages, coating is a significant strategy in current catalyst design. Coating is therefore crucial to attaining efficient and sustained catalytic activity[6].

The present work explores the HDS of sulfur compounds in kerosene utilizing a novel catalyst system containing Ni-nano silica coated on a $CoMo/Al_2O_3$ substrate. The $CoMo/Al_2O_3$ catalyst was taken straight from North Refineries Company as a newly launched catalyst. Prior to coating, the catalyst demonstrated a Brunauer–Emmett–Teller (BET) surface area of 200 m^2/g, which served as a significant benchmark for the modification procedure.

The nano-silica coating was applied using an impregnation process with the purpose of increasing catalyst stability, surface dispersion, and sulfur-removal activity. This coating serves a significant function by enhancing thermal stability, decreasing sintering, and limiting the agglomeration of nickel nanoparticles, which may lead to deactivation [7]. Additionally, nano-silica offers a large surface area matrix that permits a higher number of active hydrogenation sites, while nickel contributes to effective hydrogen transfer reactions [8]. The study stresses the synergy between the mechanical robustness and catalytic characteristics of the $CoMo/Al_2O_3$ support and the reactive, high-surface-area properties of the Ni/nano-silica coating [9]. The work involves the synthesis of nano-silica, preparation of Ni/SiO_2 and Ni/SiO_2-$CoMo/Al_2O_3$ composites, and the evaluation of their desulfurization ability under varied operating conditions (temperature, pressure, LHSV) [10].

Ultimately, the objective is to design an efficient, thermally stable catalyst that can achieve desulfurization of kerosene, therefore fulfilling international clean fuel standards and contributing to environmental sustainability.

2. MATERIALS AND METHODOLOGY

2.1. Materials

2.1.1 Oil Feedstock (Kerosene)

In this research, kerosene was selected as the primary hydrocarbon feedstock for experimental investigations. The sample, procured from the Baiji North Refinery operated by the North Refineries Company, exhibited a total sulfur concentration of 2651 ppm. The choice of kerosene is attributed to its widespread application as a household and industrial fuel, in addition to its vital role in aviation as jet fuel. The characterization and quality assessment of the kerosene sample as shown in Table 1 were carried out at the feedstock central laboratory within Baiji Refinery complex, located in Salah Aldeen province, Iraq.

Table 1: Specifications of Kerosene

No	Test Description	Unit	Results
1	Specific Gravity 15.6°C	Unitless (ratio)	0.7895
2	API	Degrees API (unitless)	48.69
3	Total Sulfur Content	ppm	2651
4	Kinematic Viscosity 40 °C	Centistokes (cSt)	1.174
5	Loss	% (percentage)	0.5

6	Total Distillation (T.D)	% (percentage)	99%
7	Residue (Ress)	% (percentage)	0.5
8	Initial Point (I.B)	°C	158
9	End Point (EP)	°C	246

2.1.2 Bentonite

The bentonite has employed for catalyst preparation supplied and characterized by the Iraqi Geological Survey authorities. This mineral resource has been extracted from the Basheera Valley area located in Al-Rutba-Iraq, and incorporated into the catalyst formulation as a binding agent.

2.1.3 Chemical Substances

In the preparation of nano-silica and catalyst synthesis, several key chemical substances were utilized. Sodium silicate (Na_2SiO_3), obtained from Sigma Aldrich with a purity of 47.53%, served as the primary precursor for silica formation. Hydrochloric acid (HCl), procured from Sigma-Aldrich with a high purity level of 98%, was employed as the acidifying agent essential for the precipitation of silica particles. Nickel nitrate, supplied by Himedia, India, with a purity of 97%, was used as the active metal source for catalyst impregnation. Additionally, distilled water sourced from the Phytochemistry Laboratory at the scientific research center (SRC) was used throughout the synthesis process for solution preparation, washing, and purification steps to ensure product quality and consistency.

2.2. Catalyst Preparation

2.2.1.Preparation of Nanosilica Support

The sol-gel method was employed to synthesize the nanosilica support intended for the development of the catalyst[11]. The process began by dissolving 140 grams of sodium silicate into 500 milliliters of distilled water, with continuous stirring on a heated magnetic stirrer for duration of two hours at room temperature. Following this, concentrated sulfuric acid (98%) was introduced slowly, drop by drop, while stirring persisted until the pH level dropped to 1, leading to the formation of a gel. This gel was then allowed to age for (24 hours) under ambient temperature conditions. Once aged, the gel was separated using Whatman No. 41 filter paper, assisted by a vacuum pump to expedite the filtration and washing process. Such rinsing step has repeated with water continued until the pH of the gel reached to 7. The resulting gel was subsequently dried in an oven at 110°C for a full day (24 hours). In the final step, the dried material was calcined in a muffle furnace at 400°C for 3 hours, yielding approximately 30 grams of nanosilica powder.

2.2.2 Preparing Composite (Active metal Ni on Nano Silica)

To prepare the composite solution, precisely 4.6 grams of nickel nitrate hexahydrate ($Ni(NO_3)_2 \cdot 3H_2O$) were dissolved in 300 milliliters of distilled water. This solution was stirred vigorously using a magnetic stirrer for half an hour at ambient temperature until a fully saturated solution was formed.

Subsequently, 30 grams of nano-silica powder were carefully measured and transferred into a separate beaker. The previously prepared nickel solution was then added slowly onto the nano-silica while maintaining continuous stirring for a duration of three hours at room temperature. This ensured thorough impregnation of the active metal onto the support material. After mixing step, the container was covered and left undisturbed overnight to complete the impregnation process.

The resulting Ni/nano-silica composite was then subjected to a drying process in a furnace, maintained at 110°C for 12 hours. Following drying, the composite underwent calcination in a muffle furnace at 400°C for three hours to achieve the desired catalytic properties.

2.2.3 Formulation of a Composite Catalyst (Ni/Nano Silica–CoMo/Al_2O_3)

Once the composite support was ready, 70 grams of $CoMo/Al_2O_3$ were finely milled using an electric grinder. The ground material was then passed through a sieve with a mesh size of 0.25 micrometers to ensure uniform particle size. This sieved catalytic powder was thoroughly blended with the previously prepared Ni/nano-silica support.

To enhance the cohesion of the mixture, 10.5659 grams of bentonite were incorporated as a binding agent, and the entire blend was kneaded to achieve a homogenous mass. This mixture was then fed into a granulating machine at the petroleum research and development center (PRDC) laboratory (Ministry of Oil, Iraq), where it was shaped into slender, thread-like granules.

The formed catalyst strands were dried in two consecutive stages: first at 60°C for six hours, followed by another six hours at 120°C to ensure complete moisture removal. Finally, the catalyst was calcined at 400°C for three hours, resulting in the final catalyst product.

3. Hydrotreating Unit Description and Function

A laboratory-scale hydrotreating unit, supplied by VINCI Technologies, was employed to evaluate the hydrodesulfurization (HDS) performance of the synthesized catalyst $CoMo/Al_2O_3$ coated with Ni-Nano silica. This unit simulates the conditions of industrial hydrotreating reactors and is specifically designed for catalyst testing under high-pressure and high-temperature conditions.

The system consists of a vertical fixed-bed stainless steel reactor enclosed within an electric heating furnace, as shown in Figures 1. The reactor was charged with 25 grams of the prepared catalyst, while both ends of the bed were packed with inert ceramic balls to ensure uniform flow distribution and thermal insulation.

During operation, kerosene feedstock containing organosulfur compounds is introduced into the reactor using a high-precision dosing pump. Simultaneously, hydrogen gas with high purity (99.99%) is supplied at a controlled flow rate and pressure. The system is equipped with multiple sensors and controllers to regulate key parameters such as temperature, pressure, and liquid hourly space velocity (LHSV).

The hydrotreating reaction occurs under elevated temperatures (typically between 315°C and 471°C) and hydrogen pressures ranging from 18 to 39 bar, depending on the experimental run. Under these conditions, sulfur compounds in kerosene are catalytically hydrogenated into hydrogen sulfide (H_2S), which is then removed from the liquid product stream.

The coated $CoMo/Al_2O_3$-Ni-Nano silica catalyst plays a vital role in enhancing HDS efficiency by offering high surface area, improved dispersion of active metals, and a mesoporous structure that facilitates the diffusion of large hydrocarbon molecules. This setup provides valuable data for assessing catalyst activity, stability, and regeneration behavior on a small scale prior to industrial implementation.

The sulfur conversion after the HDS process is calculated according to Eq. (1)[12]:

$$X_{sulfur} = 1 - \frac{C_{sulfur\ out}}{C_{sulfur\ in}} \qquad (1)$$

where:

X_{sulfur} : is the sulfur conversion.

$C_{sulfur\ in}$: is the initial sulfur concentration.

$C_{sulfur\ out}$: is the sulfur concentration after the run.

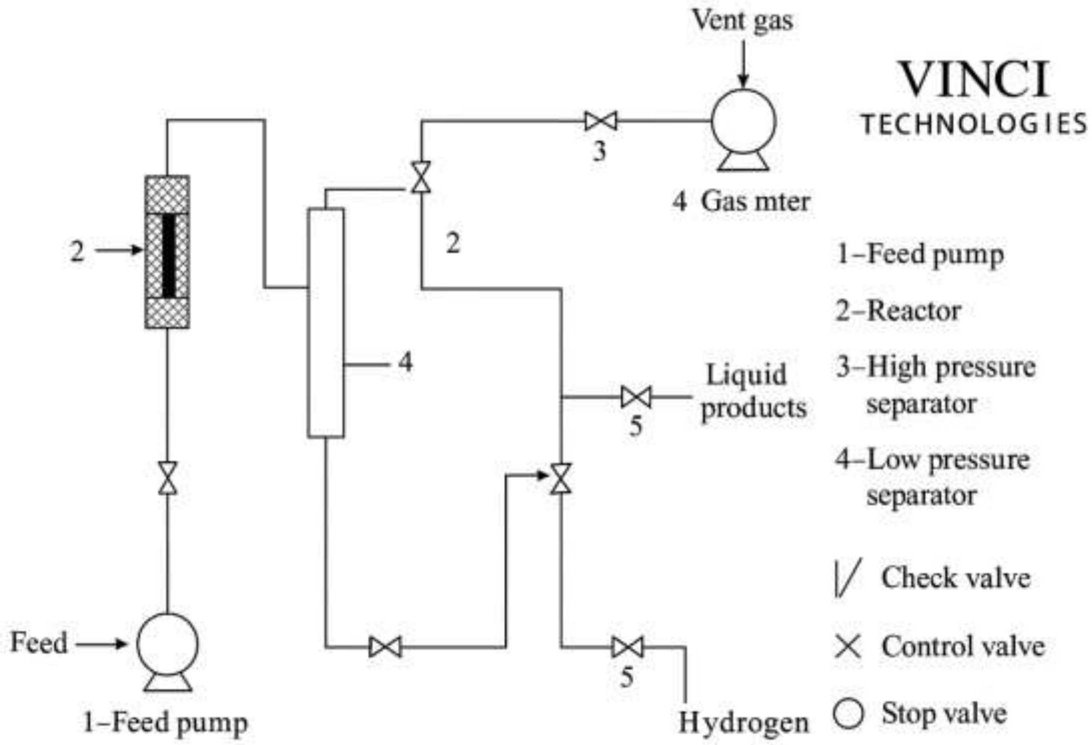


Figure 1: Schematic Diagram of The Hydrodesulphurization Unit

4. RESULT AND DISCUSSIONS

4.1 Catalyst Characterization

4.1.1 Diffraction of X-rays (XRD)

The existence of distinctive γ-Al_2O_3 peaks in the XRD pattern demonstrated that the alumina support had retained its crystalline structure during the catalyst production and coating procedure. as shown in the Figure 2 These peaks, which are usually seen at 2θ values at 37°, 45°, and 66°, are in line with earlier results on alumina-based supports that have been reported in the literature[10]. For the material to retain its mechanical and catalytic integrity during hydrodesulfurization, this support must remain stable when heated and treated chemically[13].

The diffractogram showed faint reflections from active metal oxide species including $CoMoO_4$ and NiO in addition to alumina, indicating that cobalt, molybdenum, and nickel were successfully incorporated into the catalyst framework. These species are recognized to be the main active phases in hydrodesulfurization processes, and their existence facilitates the catalyst's intended function[4, 14].

The area between 20° and 30° 2θ showed a notable wide background hump, which is suggestive of an amorphous or poorly organized phase. This characteristic is explained by the presence of Ni-Nano silica, which does not create clear diffraction peaks because to its tiny particle size and disordered structure. This amorphous halo's appearance aligns with research on mesoporous and nanoscale silica materials Validating the effective application of nano-silica onto the catalyst surface [9, 15]. The non-crystalline characteristics of silica are viewed as beneficial since they augment the surface area of the catalyst and promote the distribution of active metals, leading to enhance the catalytic performance.

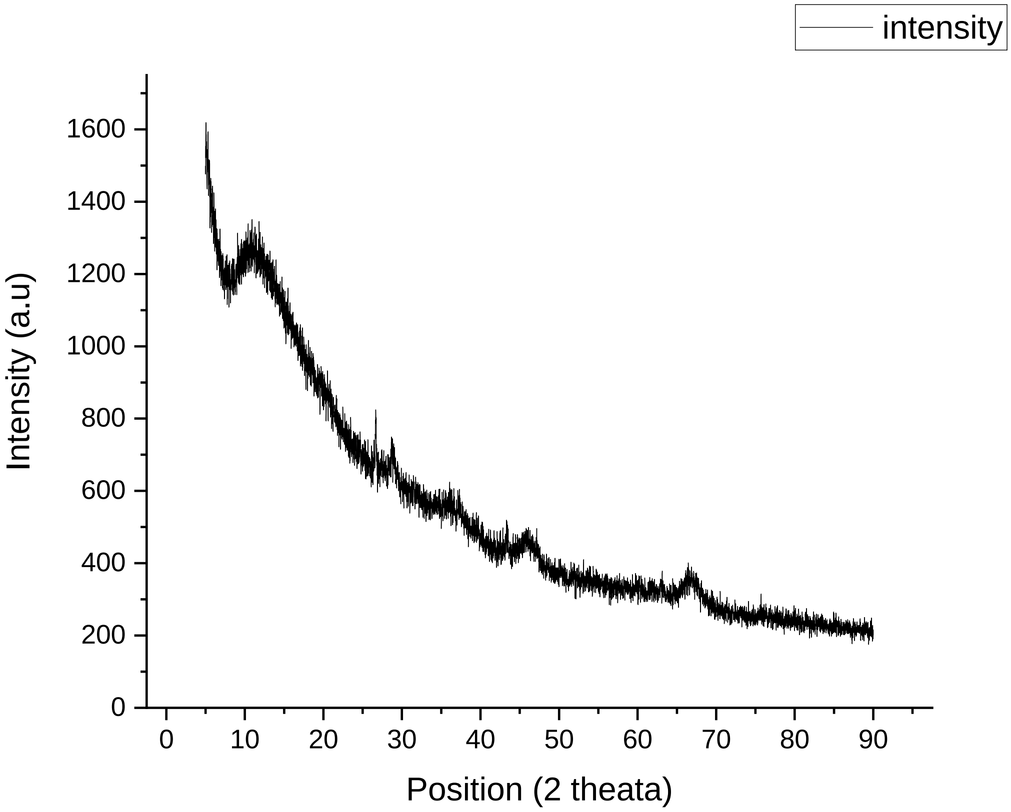


Figure 2: : The XRD of(Como/Al_2o_3 /Ni-Nano Silica) Catalyst

4.1.2 Infrared Fourier Transform (FTIR)

The FTIR spectrum displayed multiple distinct absorption bands. A wide band detected near 3400 cm^{-1} as shown in the Figure 3 is associated with the stretching vibrations of surface hydroxyl groups (–OH), suggesting the existence of adsorbed water or hydroxyl functionalities on the catalyst's surface. The band around 1630 cm^{-1} corresponds to the bending vibrations of molecular water (H–O–H), frequently observed in porous oxide materials[16].

Significant peaks observed in the range of 1000–1100 cm^{-1} were linked to the asymmetric stretching vibrations of Si–O–Si bonds, thereby affirming the integration of silica into the catalyst structure. The observation of bands in the range of 500–800 cm^{-1} is generally attributed to M–O (metal-oxygen) bonds. Such as Mo–O and Co–O, indicating the presence of cobalt and molybdenum oxides within the catalyst composition[17].

The observed spectral features validate the effective incorporation of silica, alumina, and metal oxides within the catalyst. Moreover, the identification of hydroxyl and water-related bands suggests significant surface hydroxylation, potentially facilitating the dispersion of active species and enhancing catalytic performance. The FTIR analysis provides essential evidence that underpins the structural and chemical functionality of the catalyst used in hydrodesulfurization applications.

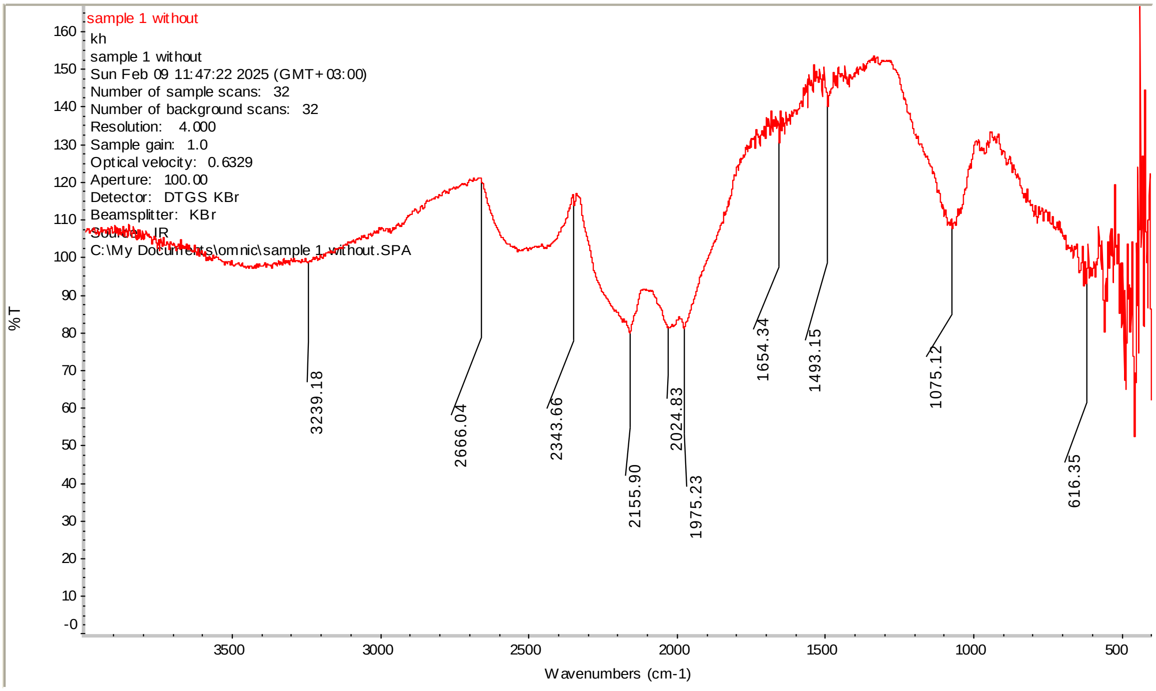


Figure 3: The FTIR of Composed Catalyst CoMo/Al_2O_3-Ni-Nano Silica

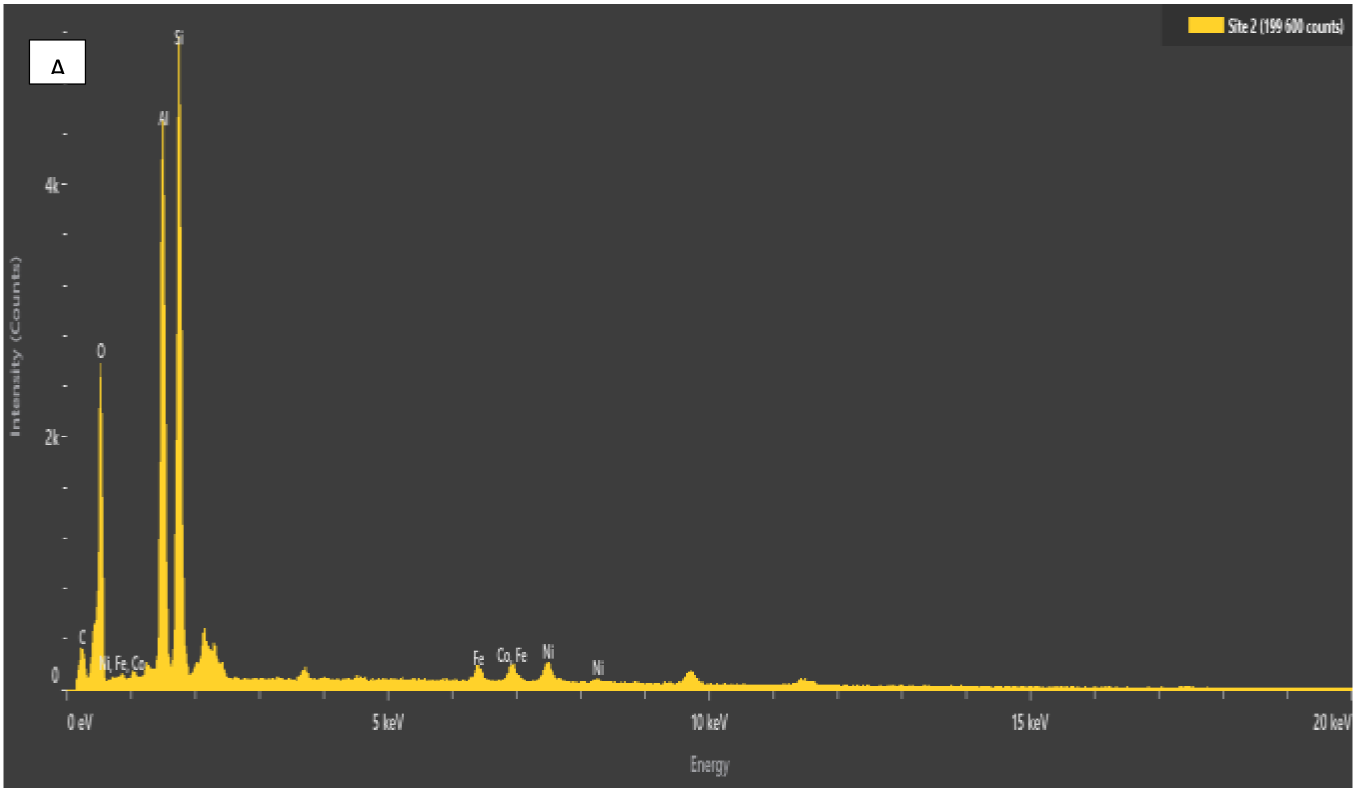
4.1.3 SEM-EDX Analyses

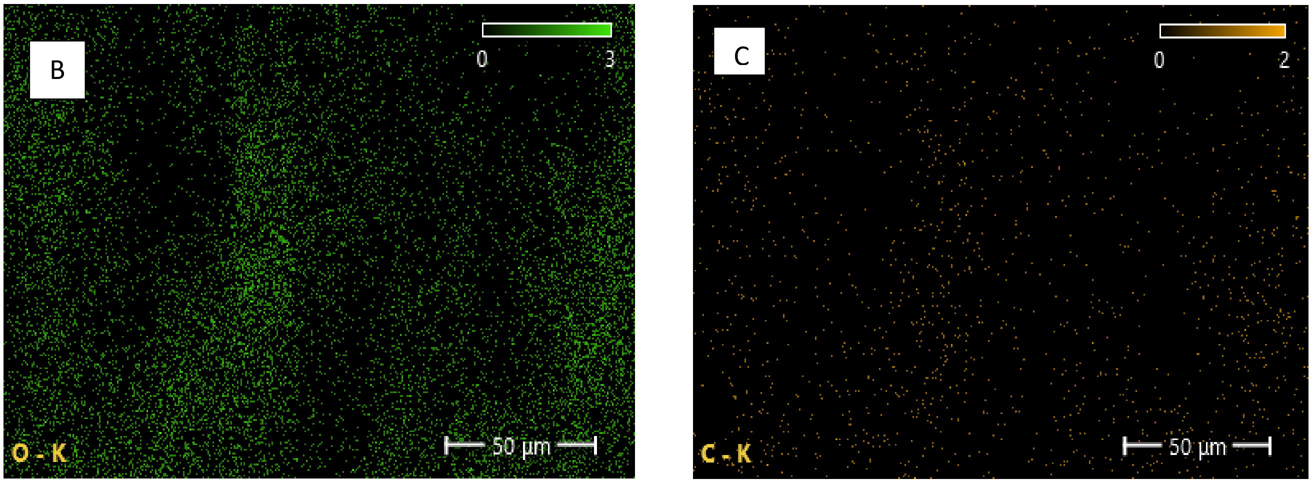
The surface morphology and elemental composition of the synthesized CoMo/Al_2O_3 catalyst coated with Ni-Nano silica were examined using Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDX). The analysis through SEM yields intricate images of the catalyst's surface topography, facilitating the observation of particle size, texture, and agglomeration behavior. EDX provides qualitative and semi-quantitative insights into the elemental distribution and relative abundance within the sample[18].

The SEM images (shown in Figure 4) displayed a rough, porous surface morphology featuring irregular particle shapes and indications of agglomeration, typical of metal oxide-supported catalysts. The presence of these textural features is advantageous, as they increase the surface area and improve the accessibility of active sites in catalytic reactions. The nano-silica coating manifested as a fine dispersion on the alumina surface, enhancing uniform particle distribution and possibly improving catalytic efficiency.

The EDX spectrum validated the existence of essential elements within the catalyst structure, such as aluminum (Al), oxygen (O), silicon (Si), cobalt (Co), molybdenum (Mo), and nickel (Ni). composite sample EDX results are shown in Figure 6. The identification of silicon and nickel offers compelling proof of the effective integration of Ni-Nano silica onto the support material. The notably elevated atomic percentages of aluminum and oxygen further confirm the prevalence of the Al_2O_3 support. The elemental mapping revealed a favorable dispersion of active metals, showing minimal clustering. This suggests that the preparation method successfully achieved a uniform distribution of the catalytic components[19].

Figure 4: SEM Images of Composite Catalyst




B
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3
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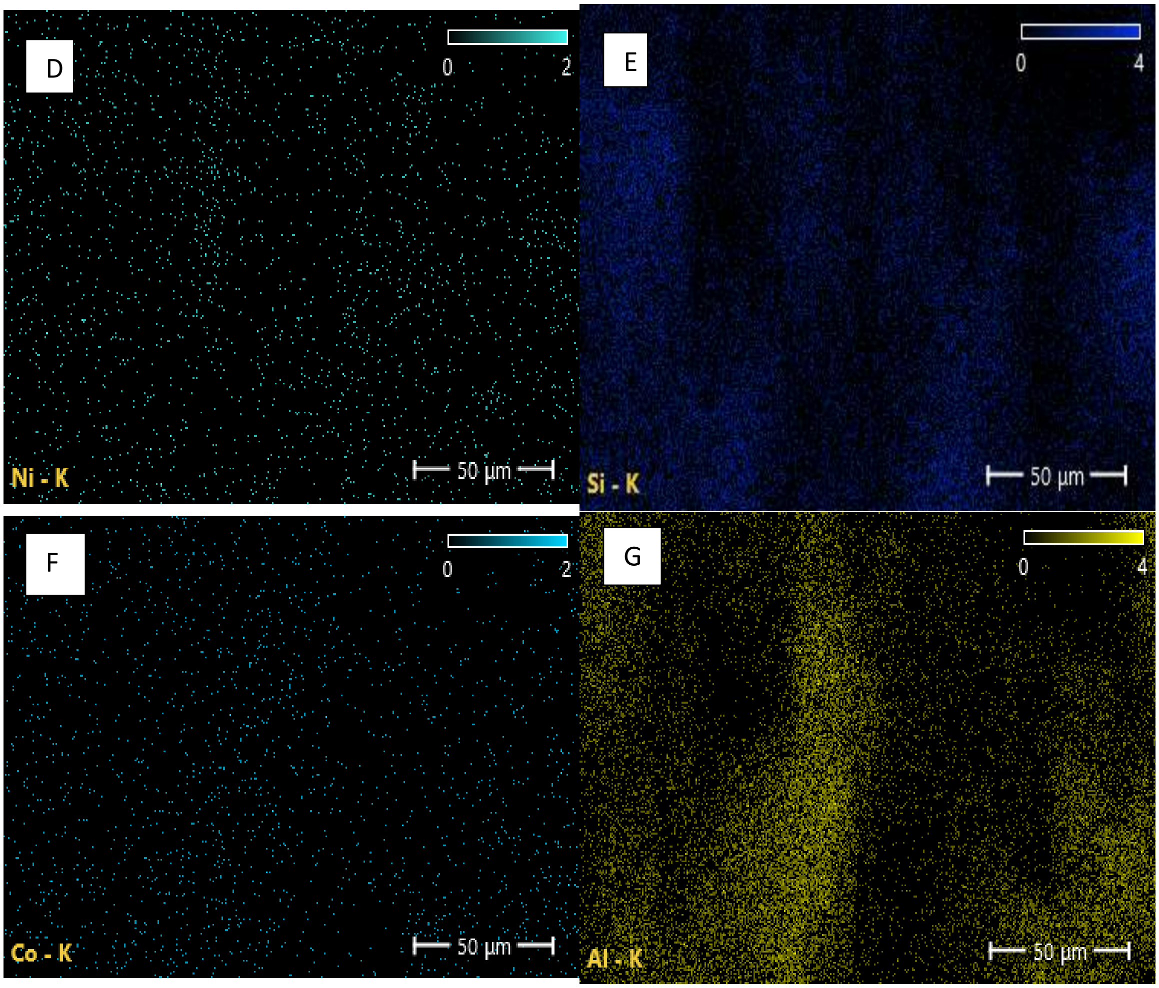
D
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Ni - K
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F
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Al - K
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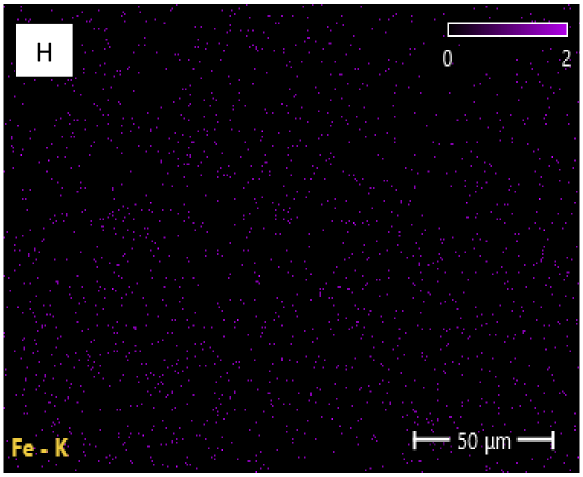

Figure 5: EDS Mapping and Elemental Analysis of CoMo/Al_2O_3 Catalyst Coated with Ni-Nano Silica. **(A)**: EDS Spectrum of the Catalyst Sample, **(B)**: Elemental Mapping of Oxygen (O-K), **(C)**: Elemental Mapping of Carbon (C-K), **(D)**: Elemental Mapping of Nickel (Ni-K), **(E)**: Elemental Mapping of Silicon (Si-K), **(F)**: Elemental Mapping of Cobalt (Co-K), **(G)**: Elemental Mapping of Aluminum (Al-K), **(H)**: Elemental Mapping of Iron (Fe-K).

4.1.4 Surface Area (Bet) Analyses

The BET surface area analysis was performed on the Ni-Nano silica-coated CoMo/Al_2O_3 catalyst using nitrogen adsorption. Prior to testing, the sample was degassed at 200°C for six hours to remove moisture and contaminants. As show in Table 2, The results showed a high specific surface area of 198.13 m^2/g and a total pore volume of 0.537 cm^3/g, indicating a well-developed porous structure. The average pore diameter, calculated as 10.85 nm, falls within the mesoporous range (2–50 nm), ideal for promoting the diffusion of large molecules during catalytic reactions such as hydrodesulfurization (HDS). These findings confirm that the catalyst possesses excellent textural properties, making it highly suitable for industrial HDS applications.

Table 2: Surface Area and Pore Volume in Formation for Catalyst

Property	**Catalyst (CoMo/AL_2O_3-Ni-Nano Silica)**
Surface area, (m^2/g)	198.13
pore volume, cm^3/g	0.537
Pore size, nm	10.85

4.2 The Effect of Parameters on Sulfur Removing:

The process of hydrodesulfurization (HDS), which removes sulfur from kerosene, is greatly influenced by a number of operational factors, including as temperature, pressure, and liquid hourly space velocity (LHSV).

4.2.1 Effect of Temperature on Sulfur Removal

The role of temperature is crucial in hydrodesulfurization (HDS) processes. The dataset comprising demonstrates a distinct and robust positive correlation between temperature and sulfur removal efficiency. The results were visually represented in Figures (6 - 9) at various Pressure, At low temperatures (e.g., 315°C), the performance for sulfur removal remains consistently inadequate, exhibiting values between 10% and 32%, regardless of the changes in pressures and LHSV values. At 425°C, sulfur removal typically surpasses 90%, while at 471°C, numerous experiments demonstrate removal efficiencies exceeding 95%, with maximum values reaching 99.47%.

This trend corresponds with the dynamic characteristics of HDS reactions, where elevated temperatures increase the rate of C–S bond cleavage, especially in resistant sulfur compounds such as dibenzothiophene (DBT) and its alkylated derivatives. Furthermore, elevated temperatures enhance hydrogenation by promoting the adsorption and activation of hydrogen on catalyst surfaces[4].

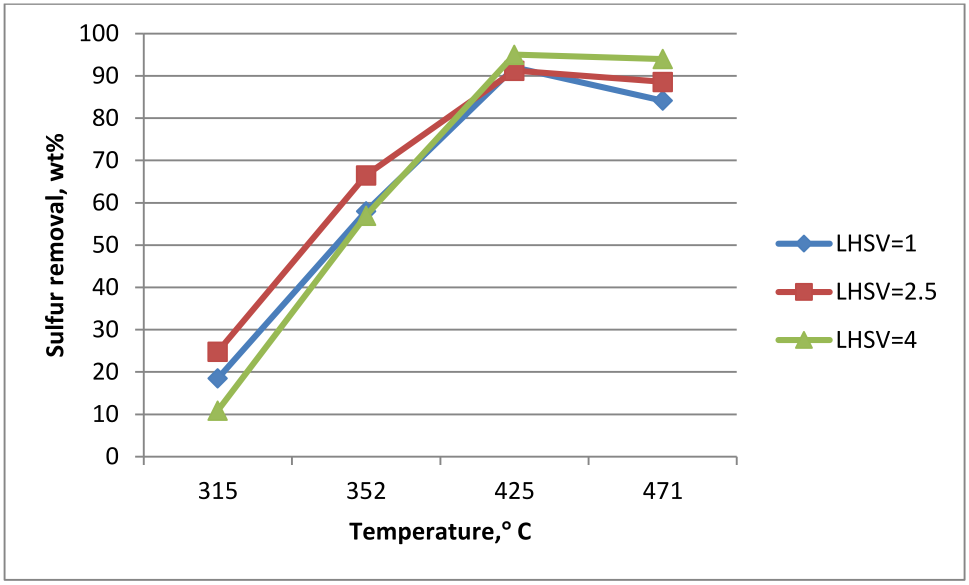

Figure 6: Effect of Temperature on the Sulfur Removal at P=18 bar.

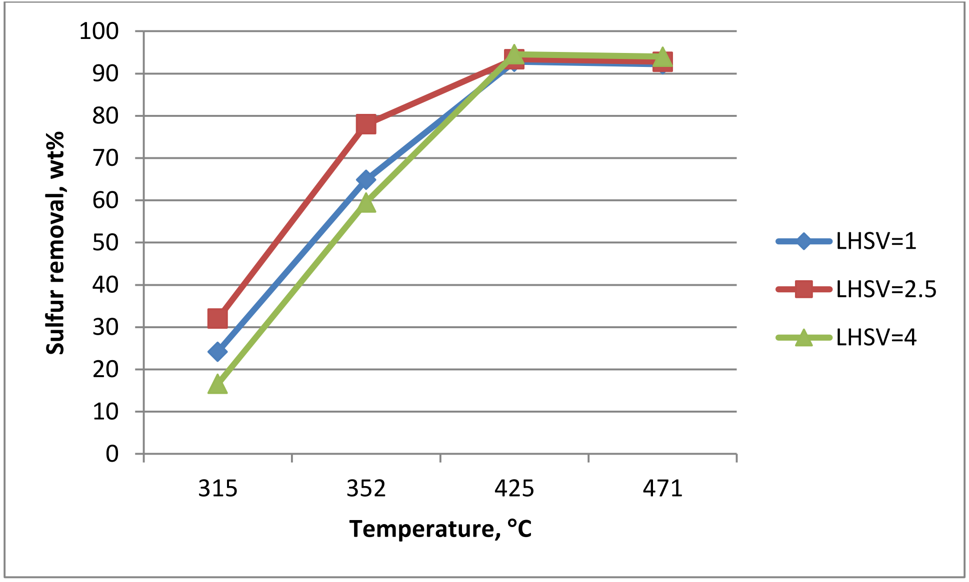

Figure 7: Effect of Temperature on The Sulfur Removal at P=26 bar.

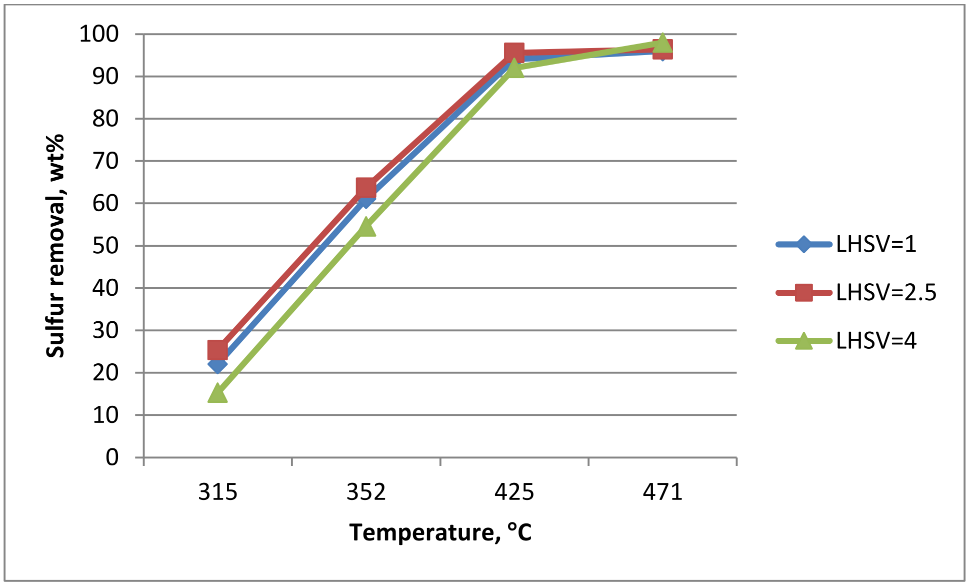

Figure 8: Effect of Temperature on The Sulfur Removal at P=34 bar.

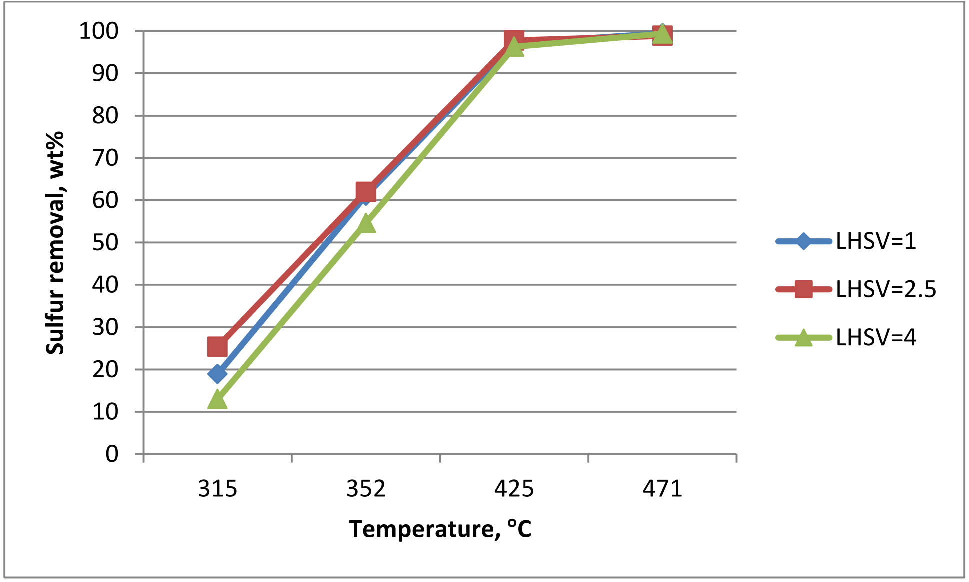

Figure 9: Effect of Temperature on The Sulfur Removal at P=39 bar.

5.2.2 Effect of Pressure on Sulfur Removal

The partial pressure of hydrogen plays a crucial role in HDS reactions by regulating the availability of dissociated hydrogen atoms. The results were visually represented in Figures (10 - 13) at various temperatures, a rise in pressure typically corresponds with enhanced sulfur removal. For example, under a constant temperature of 425°C and an LHSV of 1 hr^{-1}, the efficiency of sulfur removal increases from 92% at 18 bar to 97.2% at 39 bar. This indicates that increased hydrogen pressure improves the hydrogenation process, aiding in the conversion of aromatic sulfur compounds into removable thiols or hydrogen sulfide (H_2S).

Nevertheless, the dataset indicates that beyond 34–39 bar, the enhancement in sulfur removal appears to be minimal or may even reach a plateau. This outcome is anticipated, as once the surface hydrogenation capacity reaches saturation, further pressure increases offer minimal additional advantages and could impede diffusion[10].

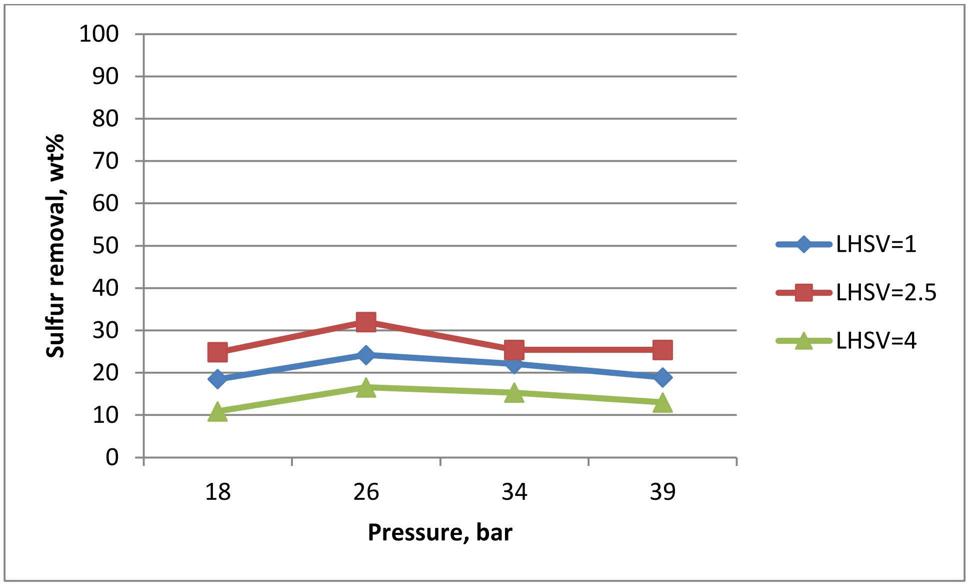

Figure 10: Effect of Pressure on The Sulfur Removal at T=315 °C

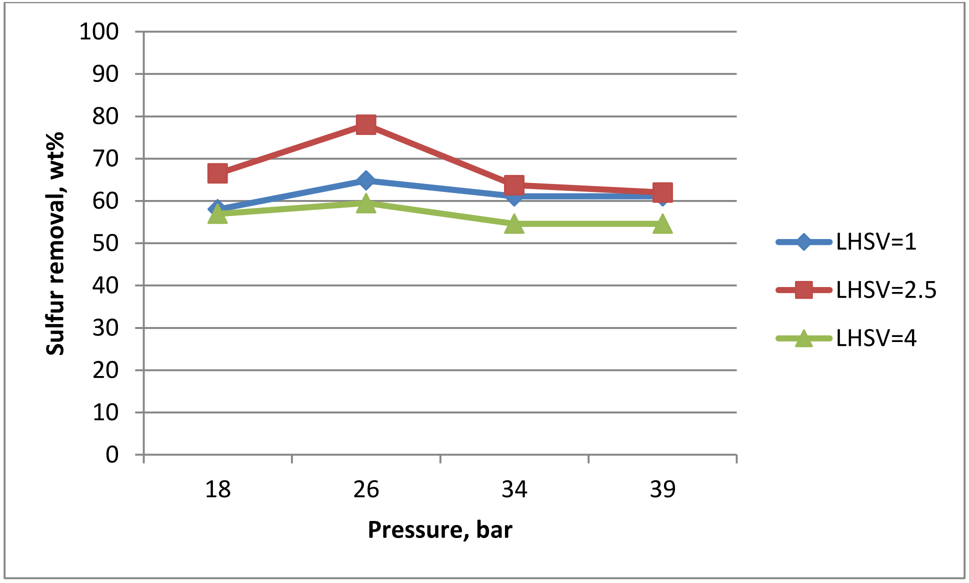

Figure 11: Effect of Pressure on The Sulfur Removal at T=352 °C

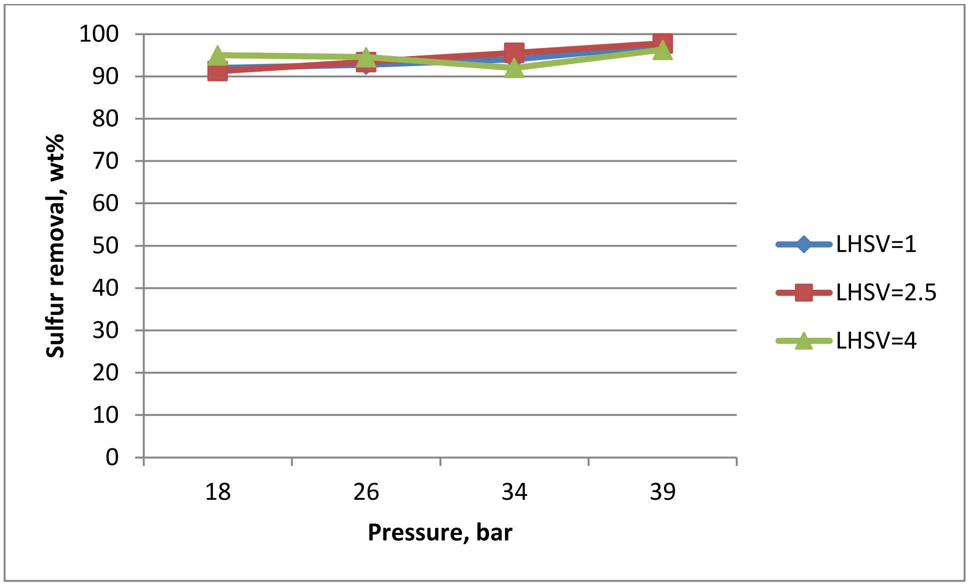

Figure 12: Effect of Pressure on The Sulfur Removal at T=425 °C

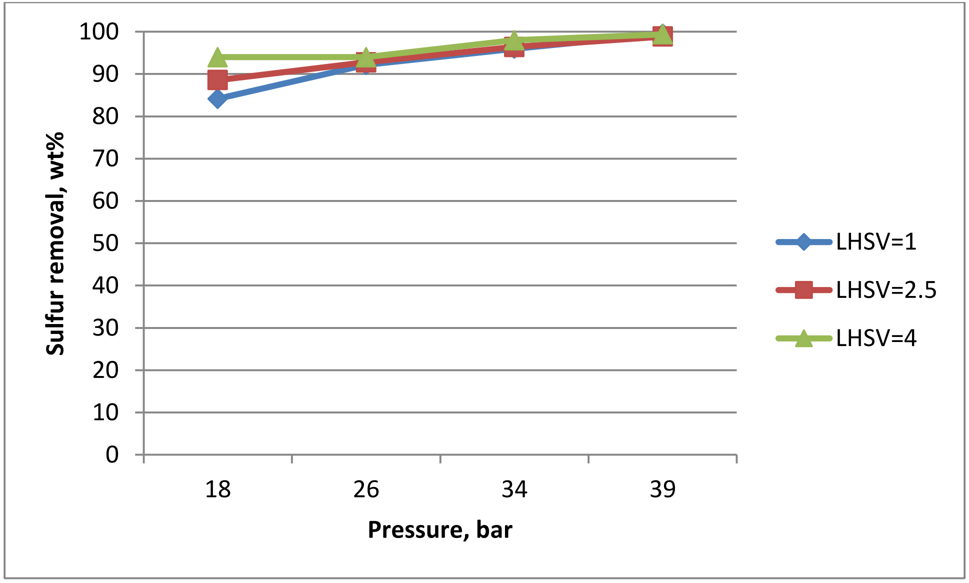

Figure 13: Effect of Pressure on The Sulfur Removal at T=471 °C

4.2.2 Effect of LHSV On Sulfur Removal

It is evident that the effectiveness of removing sulfur from kerosene by hydrodesulfurization (HDS) is significantly influenced by liquid hourly space velocity (LHSV). The results were visually represented in Figures (14 - 17) , The volumetric flow rate of the liquid feed in relation to the reactor's catalyst volume per hour (hr^{-1}) is denoted by LHSV. It directly regulates how long the feed and catalyst are in contact. Kerosene has a longer residence time over the catalyst bed with lower LHSV values (e.g., 1 hr^{-1}), which gives the sulfur compounds more opportunity to react with hydrogen on the catalyst's active sites. Higher desulfurization efficiency results from this prolonged contact, which improves the conversion of sulfur molecules into hydrogen sulfide (H_2S)[20].

Higher LHSV values, such as 4 hr^{-1}, on the other hand, shorten the duration that the feed and catalyst are in contact. Particularly for more complicated and refractory sulfur compounds like 4,6-dimethyldibenzothiophene, which need more time and hydrogenation steps, this might lead to an incomplete reaction. Usually, this results in less effective sulfur removal[21]. Nevertheless, catalytic activity is sufficiently increased at higher temperatures (e.g., ≥425°C) to sustain high desulfurization even at higher LHSV levels, indicating that the relationship between LHSV and temperature is not strictly linear.

Optimal performance is attained when LHSV is minimized, providing the catalyst ample opportunity to engage with the feed[22].

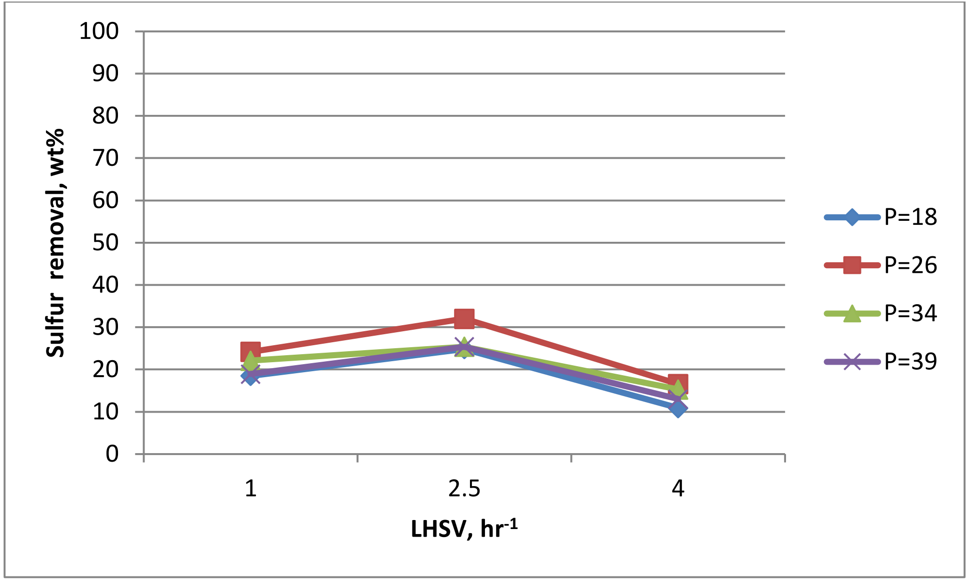

Figure 14: Effect of LHSV on Sulfur Removal at T=315 °C

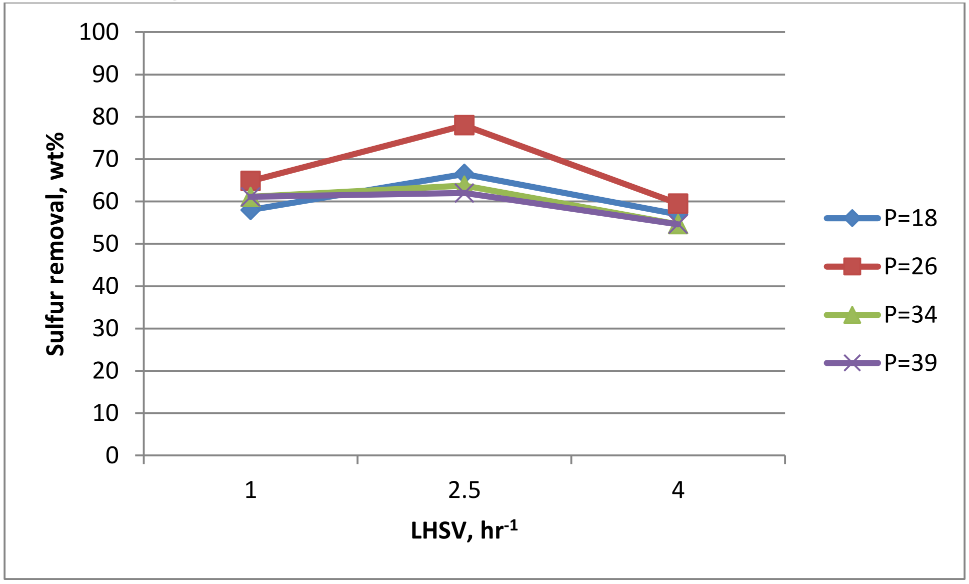

Figure 15: Effect of LHSV on Sulfur Removal at T=352 °C

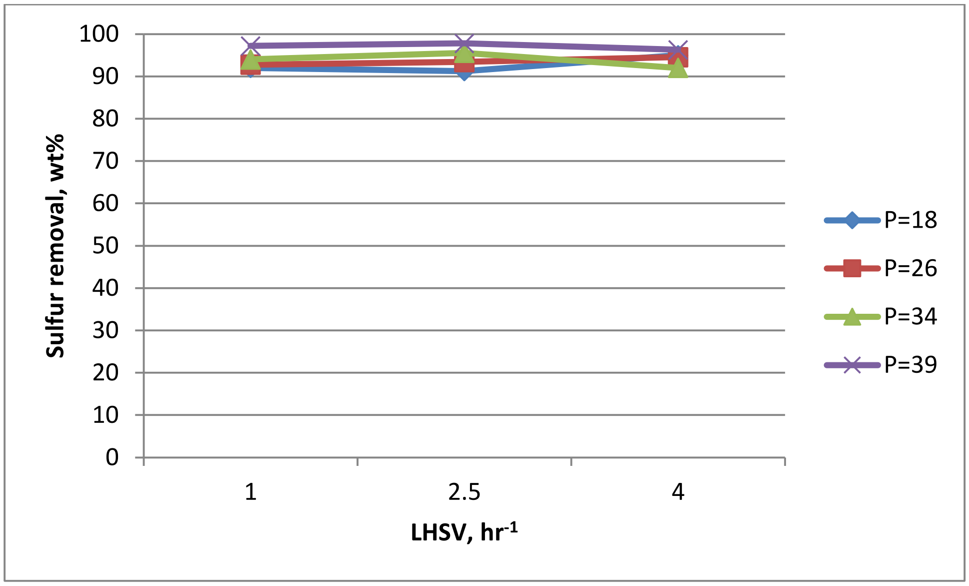

Figure 16: Effect of LHSV on Sulfur Removal at T=425 °C

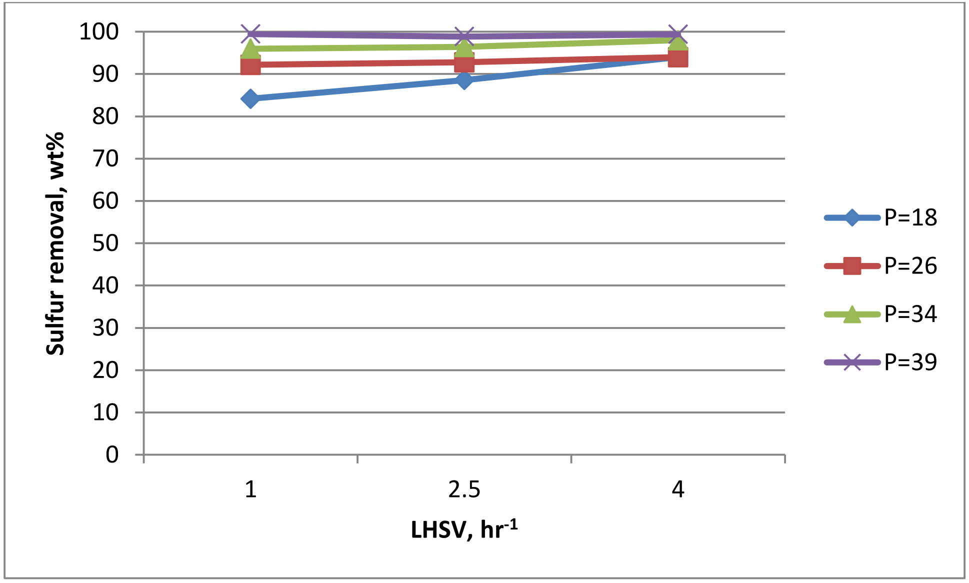

Figure 17: Effect of LHSV on Sulfur Removal at T=471 °C

6. Conclusions

For the hydrodesulfurization (HDS) of kerosene, this work effectively illustrated the creation, characterization and performance assessment of a new composite catalyst made of Ni-Nano silica coated on CoMo/Al_2O_3. XRD, FTIR, SEM-EDX, and BET studies verified that the catalyst synthesis, which included sol-gel, impregnation, and mechanical mixing processes, produced a material with a large surface area (198.13 m^2/g), a mesoporous structure (10.85 nm), and well-dispersed active metals.

Under various operating circumstances, such as temperatures between 315°C and 471°C, hydrogen pressures between 18 and 39 bar, and LHSV values between 1 and 4 h^{-1}, the catalyst was evaluated in a laboratory-scale fixed-bed hydrotreating machine. The findings showed that all three factors had a significant impact on the efficiency of sulfur removal. At 471°C, 39 bar, and 1 h^{-1} LHSV, the catalyst achieved an optimal desulfurization performance of 99.47% sulfur removal, demonstrating its strong activity under extreme HDS conditions.

Furthermore, the research demonstrated that although lowering LHSV increases contact time and

conversion efficiency, raising temperature and pressure typically improves the hydrogenation of refractory sulfur species. The new catalyst's performance is in line with findings documented in the literature and compares well with traditional hydrotreating catalysts.
All things considered, the results demonstrate how well Ni-Nano silica–CoMo/Al_2O_3 works as a scalable and promising catalyst for creating ultra-low sulfur kerosene, assisting with upcoming commercial applications meant to satisfy strict environmental standards.

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