



Optimization of Temperature, Asbuton Feed Mass, and FeNi/TiO₂ Catalyst Mass for Asbuton Pyrolysis Using a Green Nanocatalyst

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Abstract

Asbuton (Buton asphalt) is an Indonesian natural resource containing bitumen with potential for conversion into alternative liquid fuel through pyrolysis. This study investigated the effects of pyrolysis temperature, Asbuton feed mass, and FeNi/TiO₂ green nanocatalyst mass on oil yield and determined the optimum operating conditions. Experiments were performed in a laboratory-scale pyrolysis reactor using a Central Composite Design (CCD), while the experimental data were analyzed using Response Surface Methodology (RSM). The process variables were temperature, Asbuton–acetone mixture mass, and FeNi/TiO₂ catalyst mass, with oil yield as the main response. The experimental oil yield ranged from 14.79% to 45.17%, with temperature identified as the most influential factor, followed by catalyst mass and Asbuton mass. The interactions between temperature and Asbuton mass and between Asbuton mass and catalyst mass were also statistically significant. The analysis indicated that the optimum conditions were a temperature of 360 °C, Asbuton–acetone mass of 30 g, and FeNi/TiO₂ catalyst mass of 1.6014 g, resulting in a predicted oil yield of 56.5786%. Experimental validation under these conditions produced an oil yield of 56.7706%, corresponding to a percentage error of only 0.34%. These results demonstrate that CCD and RSM can effectively determine suitable operating conditions and improve oil yield from Asbuton pyrolysis using FeNi/TiO₂ green nanocatalyst, supporting the development of Asbuton utilization as an alternative hydrocarbon resource.

Keywords: Asbuton, Optimization, Pyrolysis, FeNi/TiO₂, Response Surface Methodology.

1. Introduction

Asbuton (Buton asphalt) is one of Indonesia's natural resources originating from Buton Island, Southeast Sulawesi. Asbuton is a naturally occurring asphalt rock composed of minerals and bitumen and is known to have substantial reserves in Indonesia. However, its utilization has predominantly been limited to road construction applications, while its bitumen content has the potential to be further developed as an alternative hydrocarbon resource with added value. These findings provide new insights in the development of pyrolysis technology that is sustainable and economical and encourages a wiser use of natural resources through the use of bitumen and other complex organic materials (Parapat, Zamaludin, et al., 2024).



Figure 1. Asbuton

The bitumen contained in Asbuton consists of hydrocarbon compounds with relatively heavy structures and carbon chains, requiring conversion processes to produce lighter fractions. Pyrolysis is one of the thermochemical

conversion methods that can be carried out under oxygen-deficient or oxygen-free conditions. Through thermal treatment, complex hydrocarbon compounds can undergo decomposition and cracking, producing liquid products with potential applications as fuels or feedstocks for the petrochemical industry. Therefore, enhancing liquid product formation or oil yield is an important aspect in developing the utilization of Asbuton through the pyrolysis process (Saadah et al., 2020).

The formation of oil yield is influenced by various process conditions, particularly temperature and catalyst utilization. Temperature determines the extent of hydrocarbon decomposition in bitumen. At excessively low temperatures, decomposition may be incomplete, whereas excessively high temperatures can promote secondary cracking, causing some liquid hydrocarbons to further decompose into lighter compounds and potentially reducing the oil yield. In addition to temperature, the feedstock mass and catalyst mass should also be considered, as variations in these parameters can affect the contact ratio between hydrocarbons and the active sites of the catalyst.

The use of catalysts in pyrolysis can facilitate the cracking of heavy hydrocarbons by providing active sites for the reaction. Nanocatalysts typically exhibit higher surface area and increased surface-to-volume ratio than conventional catalysts (Parapat, Laksono, et al., 2024). In addition to surface area, the structure and morphology of nanoparticles can also influence their catalytic performance. Parapat et al. (2019) reported that nanostructures with a greater number of defects, kinks, steps, and edges can provide more active sites, thereby enhancing catalytic activity. One catalyst that has been investigated for Asbuton pyrolysis is FeNi/TiO₂, with Fe and Ni serving as the active components and TiO₂ as the support material. Research on pyrolysis using a FeNi/TiO₂ catalyst has previously been conducted by (Parapat et al., 2023) demonstrated that the use of a green nanocatalyst FeNi/TiO₂ in the mild pyrolysis process has the potential to produce liquid fuel with characteristics comparable to diesel fuel. The importance of synthesis conditions in determining nanocatalyst performance has also been demonstrated by Parapat et al. (2022), who optimized the green synthesis of supported Pt nanoparticles using factorial design, Central Composite Design (CCD), and Response Surface Methodology (RSM). Their results showed that the synthesis conditions influenced both the synthesis yield and catalytic activity of the resulting nanocatalyst.

Previous studies have reported nanoscale Fe and Ni structures, as illustrated in **Figure 2**. These nanoscale structures provide a relatively high surface-to-volume ratio, which can increase the availability of active surface sites for interactions with hydrocarbon molecules (Parapat et al., 2024). This characteristic is beneficial for catalytic pyrolysis because the active sites can facilitate the cracking of heavy hydrocarbon compounds into lighter products.

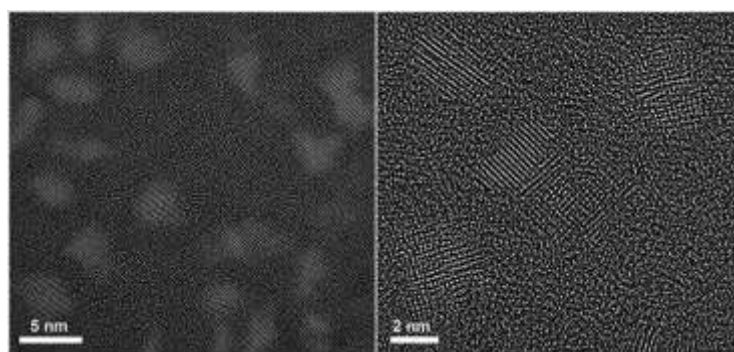


Figure 2. Nanoscale morphology of Fe and Ni nanoparticles. Adapted from Parapat et al. (2024)

The synergy between Fe and Ni improves hydrogenation activity while TiO₂ provides thermal stability and dispersion support (Parapat et al., 2025). The presence of the catalyst is expected to promote the cleavage of hydrocarbon bonds and enhance the formation of liquid fractions. Preliminary studies on Asbuton pyrolysis have also indicated that catalyst type and temperature can affect oil yield formation. Therefore, FeNi/TiO₂ was selected as the catalyst for further investigation.

However, the use of FeNi/TiO₂ cannot be separated from the operating conditions of the pyrolysis process. Variations in Asbuton mass affect the amount of hydrocarbons that must be converted, whereas variations in catalyst mass influence the availability of active sites for the cracking reactions. Therefore, increasing the feedstock or catalyst mass does not necessarily result in a higher oil yield if it is not accompanied by an appropriate pyrolysis

temperature and feedstock-to-catalyst ratio. Experimental data indicate that temperature, Asbuton mass, and catalyst mass are parameters associated with changes in oil yield. Thus, the effects of these three parameters need to be investigated simultaneously.

Based on these considerations, this study was conducted through the laboratory-scale pyrolysis of Asbuton using FeNi/TiO₂ as a catalyst. The pyrolysis temperature, Asbuton mass, and catalyst mass were varied to investigate their effects on the resulting oil yield, while other operating conditions were kept constant. The oil yield was determined based on the ratio of the mass of the liquid product obtained to the mass of the feedstock used. This approach was employed to evaluate the response of the liquid product to variations in each process parameter, as well as to investigate the interactions among the parameters.

Therefore, this study aims to analyze the effects of temperature, Asbuton mass, and FeNi/TiO₂ catalyst mass on the oil yield of Asbuton pyrolysis and to determine the experimental conditions that produce the highest oil yield. The results are expected to provide information on suitable operating conditions for enhancing liquid product formation from Asbuton and supporting its utilization as an alternative hydrocarbon feedstock.

2. Research Methodology

The pyrolysis process was divided into two main stages. The first stage involved the preparation of the Asbuton feedstock, while the second stage consisted of the pyrolysis of Asbuton rock to produce liquid fuel. In the first stage, the Asbuton rock was initially crushed using a mortar and pestle until a fine powder was obtained. The Asbuton powder was subsequently sieved using a 100-mesh sieve to obtain a uniform particle size. The Asbuton powder was then mixed with acetone as a solvent and subjected to sonication to reduce particle size and improve the homogeneity of the mixture through particle disruption.

In the second stage, 75 g of the Asbuton–acetone mixture and 1.5 g of catalyst were weighed. The catalyst was added to the Asbuton–acetone mixture and stirred until a homogeneous mixture was obtained. The resulting mixture was then transferred into the pyrolysis reactor, which was tightly sealed. Prior to heating, the reactor was evacuated and purged with nitrogen gas to replace the air inside the reactor and establish an oxygen-deficient atmosphere. The reactor was subsequently heated to 75°C to remove the acetone, followed by an increase in temperature to 140°C to ensure that no residual acetone remained in the reactor. Afterward, the temperature was increased to the experimental temperature of 450°C. The pyrolysis process was continued until no further liquid product droplets were observed.

Following the pyrolysis process, the masses of the liquid oil and solid residue were measured. The density, viscosity, and calorific value of the resulting oil were also determined. The experimental results were subsequently recorded and used for data analysis based on the Central Composite Design (CCD). The schematic diagram of the experimental apparatus is presented in **Figure 3**.

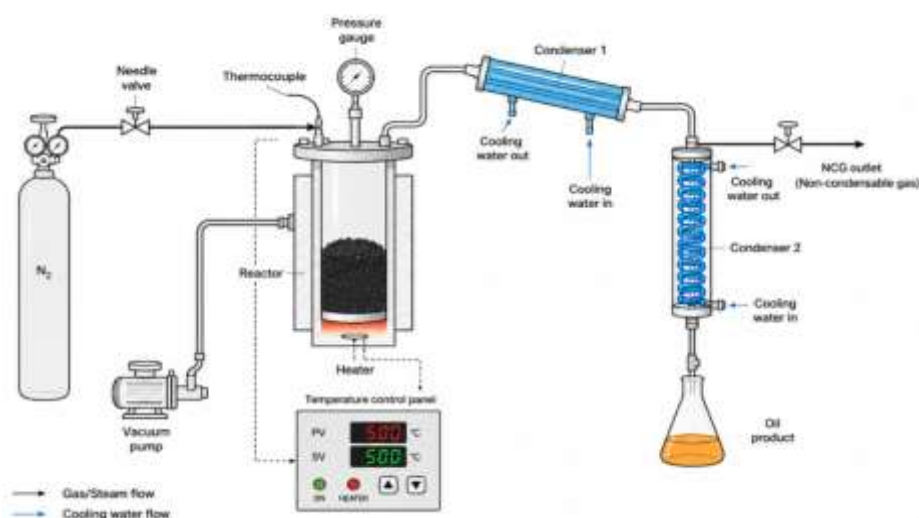


Figure 3. Schematic diagram of the Asbuton pyrolysis

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3. Results and discussion

3.1 Liquid Product and CCD Experimental Results

Pyrolysis is a thermal decomposition process of organic materials under oxygen-deficient or oxygen-free conditions, producing liquid, gaseous, and solid residue (char) products. In this study, Asbuton was subjected to pyrolysis using a FeNi/TiO₂ green nanocatalyst to promote the conversion of heavy hydrocarbon components into compounds with lower molecular weights. FeNi/TiO₂ consists of FeNi nanoparticles dispersed on a TiO₂ support, which can enhance the contact between the catalyst and the feedstock during the pyrolysis process (Parapat et al., 2023).

The variation in the characteristics of the liquid products was subsequently evaluated quantitatively based on the oil yield. The experiments were conducted by varying the pyrolysis temperature, the mass of the Asbuton–acetone mixture, and the mass of the FeNi/TiO₂ catalyst. The experimental data obtained were subsequently analyzed using the Central Composite Design (CCD) to determine the effects of the investigated factors on the oil yield and to identify the optimum process conditions. The experimental results are presented in **Table 1**.

Table 1. Experimental Results of Asbuton Pyrolysis Based on the Central Composite Design (CCD)

Run	Factor			Response
	Asbuton + Acetone Mass (g)	Catalyst Mass (g)	Temperature (°C)	Oil Yield (%)
1	112.5	1.75	640	14.79
2	75	1.5	550	21.73
3	112.5	1.75	500	29.98
4	112.5	1.75	360	45.17
5	112.5	1.05	500	24.50
6	7.5	1.75	500	33.37
7	75	2	550	26.57
8	150	1.5	550	22.68
9	150	2	550	27.25
10	217.5	1.75	500	26.59
11	150	1.5	450	32.05
12	150	2	450	33.10
13	75	2	450	40.84
14	112.5	2.45	500	35.46
15	75	1.5	450	35.64

Based on **Table 1**, the oil yield obtained ranged from 14.79% to 45.17%, with the highest oil yield of 45.17% obtained at a temperature of 360°C, an Asbuton + acetone mass of 112.5 g, and a catalyst mass of 1.75 g. In contrast, the lowest oil yield of 14.79% was obtained at 640°C with the same Asbuton + acetone mass and catalyst mass. This trend indicates that increasing the temperature within the investigated range does not necessarily result in an increase in oil yield.

These results are consistent with the study by Liu et al. on Buton oil sand, which reported that the liquid yield increased with increasing temperature until reaching an optimum condition, followed by a slight decrease at higher temperatures, while gas production continued to increase (Liu et al., 2016). This behavior can be associated with the cracking mechanism of bitumen components. At an appropriate temperature, sufficient thermal energy is available to break the bonds of heavy hydrocarbons and produce compounds that can be condensed into oil. However, at excessively high temperatures, primary products may undergo secondary cracking into lighter hydrocarbons and gases, thereby reducing the fraction that can be condensed as oil. A review of heavy oil pyrolysis reported that the optimum temperature for oil formation generally falls within the range of 350–500°C, depending on the characteristics of the feedstock and process conditions (Ore & Adebisi, 2021).

The effect of catalyst mass can be observed at a temperature of 450°C. At an Asbuton + acetone mass of 75 g, increasing the catalyst mass from 1.5 to 2.0 g increased the oil yield from 35.64% to 40.84%. At an Asbuton + acetone mass of 150 g, the same increase in catalyst mass resulted in an increase in oil yield from 32.05% to 33.10%. These results indicate that the addition of FeNi/TiO₂ can enhance liquid product formation; however, the magnitude of the catalyst effect depends on the amount of feedstock being processed.

The effect of Asbuton + acetone mass was less pronounced than that of temperature. At 450°C with a catalyst mass of 1.5 g, increasing the feedstock mass from 75 to 150 g decreased the oil yield from 35.64% to 32.05%. Similarly, at a catalyst mass of 2.0 g, the oil yield decreased from 40.84% to 33.10%. This finding indicates that increasing the feedstock amount does not directly increase the oil yield, as the effectiveness of hydrocarbon conversion is also influenced by the availability of catalyst active sites relative to the amount of feedstock being processed.

3.2 Analysis of the Effects of Process Variables on Oil Yield

The experimental data presented in **Table 1** were subsequently analyzed using the Central Composite Design (CCD) method to determine the effects of each factor on the oil yield. The significance of the factors and their interactions is illustrated by the Pareto chart of standardized effects in **Figure 4**. The red line at a value of 2.23 represents the significance threshold at a 95% confidence level ($\alpha = 0.05$). Factors or interactions exceeding this threshold are considered to have a statistically significant effect on the oil yield.

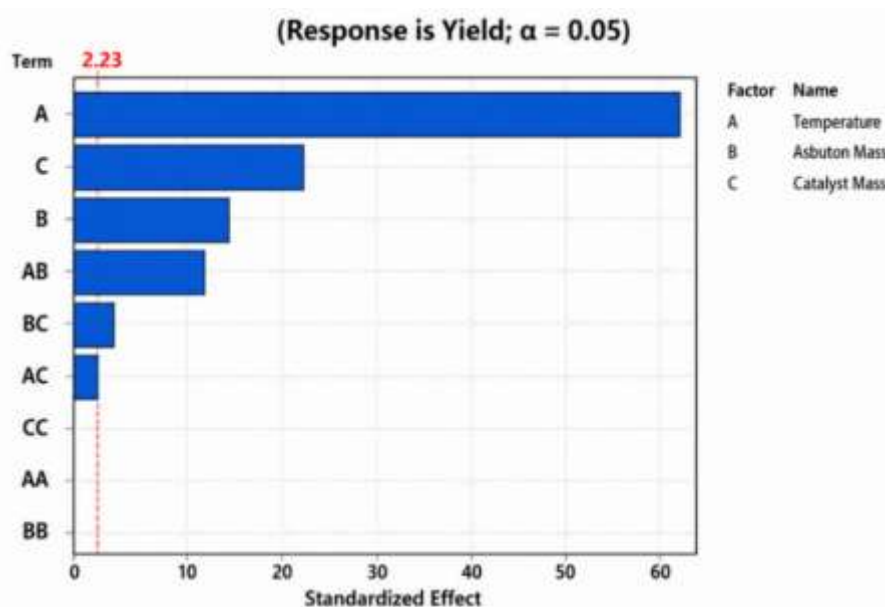


Figure 4. Pareto Chart of Standardized Effects on Oil Yield

Based on **Figure 4**, temperature (A) was the most dominant factor affecting the oil yield, with a standardized effect of approximately 61, followed by catalyst mass (C) at approximately 22 and Asbuton mass (B) at approximately 14. All three factors exceeded the significance threshold of 2.23, indicating that they had statistically significant effects on the oil yield. The dominant effect of temperature indicates that thermal conditions are the primary parameters governing hydrocarbon conversion and product distribution during pyrolysis. The strong influence of temperature is also associated with changes in reaction pathways at elevated temperatures. At an appropriate temperature, primary decomposition produces hydrocarbon vapors that can be condensed into oil. However, increasing the temperature may promote secondary cracking of these vapors, causing some liquid products to be further converted into lighter hydrocarbons and non-condensable gases. A recent study on oil shale pyrolysis reported that the oil yield tends to reach a maximum at intermediate temperatures and subsequently decreases at higher temperatures due to intensified secondary cracking, while gas formation increases (Zhao et al., 2025)

Catalyst mass (C) was the second most influential factor affecting the oil yield. The catalyst provides active sites that facilitate the cleavage of hydrocarbon bonds and influence product distribution. Li et al. reported that the

presence of a catalyst can alter the pyrolysis mechanism and product composition, with its effectiveness depending on the type and characteristics of the catalyst used (Li et al., 2021).

Asbuton mass (B) also had a significant effect on the oil yield. Changes in feedstock mass alter the ratio between the amount of feedstock and the available active sites of the catalyst. Therefore, increasing the Asbuton mass does not necessarily result in a higher oil yield when the catalyst mass and temperature are not maintained at appropriate conditions. This finding indicates that the process response is governed by the interaction between feedstock characteristics and reaction conditions rather than by the effect of a single factor (Ore & Adebiyi, 2021).

In addition to the main factors, the interaction between temperature and Asbuton mass (AB) had a standardized effect of approximately 12, exceeding the significance threshold. This indicates that the effect of temperature on the oil yield depends on the amount of Asbuton used. The interaction between Asbuton mass and catalyst mass (BC) was also significant, although its effect was less pronounced. In contrast, the interaction between temperature and catalyst mass (AC), as well as the quadratic terms AA, BB, and CC, were below the significance threshold. These results indicate that the model was primarily influenced by the linear effects of temperature, catalyst mass, and Asbuton mass, along with the AB and BC interactions.

3.3 Interaction Analysis of Process Variables on Oil Yield

After the factors affecting the oil yield were identified using the Pareto chart, the analysis was continued using an interaction plot to evaluate the effects of combinations of process variables on the oil yield. The interactions between variables can be assessed based on the pattern and slope of the lines at different factor levels. Non-parallel lines indicate the presence of an interaction between variables, while a greater difference in the slopes indicates a stronger interaction. The results of the interaction analysis between temperature, Asbuton mass, and catalyst mass on the oil yield are presented in **Figure 5**.

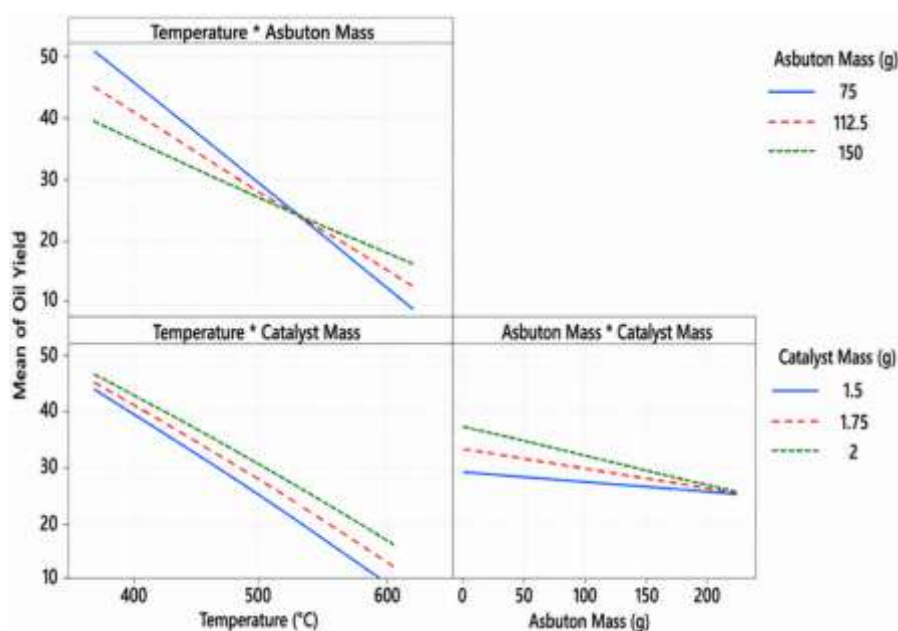


Figure 5. Interaction Plot of the Effects of Temperature, Asbuton Mass, and Catalyst Mass on Oil Yield

For the interaction between temperature and Asbuton mass, the oil yield showed a decreasing trend with increasing temperature across all Asbuton mass levels. The steepest decline was observed at an Asbuton mass of 75 g, whereas the line for 150 g was relatively less steep. This finding indicates that the effect of temperature on the oil yield depends on the amount of Asbuton used. This pattern is consistent with the Pareto chart results, in which the temperature–Asbuton mass interaction (AB) exceeded the significance threshold.

The interaction between temperature and catalyst mass also showed a decrease in oil yield with increasing temperature. The three lines corresponding to catalyst masses of 1.5, 1.75, and 2.0 g exhibited relatively parallel

patterns, indicating that the effect of temperature was more dominant than the interaction between temperature and catalyst mass. Previous studies on heavy oil pyrolysis have reported that temperature and catalyst-to-oil ratio are important parameters that jointly influence product yield and distribution. Increasing temperature can shift the reaction pathway from liquid product formation toward the formation of lighter products, while the catalyst can accelerate cracking reactions (Meng et al., 2005).

For the interaction between Asbuton mass and catalyst mass, increasing the Asbuton mass tended to decrease the oil yield. In contrast, at a catalyst mass of 2.0 g, the oil yield was relatively higher than those obtained at catalyst masses of 1.5 and 1.75 g. This condition indicates that increasing the catalyst mass can enhance the availability of active sites for the conversion of heavy hydrocarbon components. However, the effectiveness of the catalyst remains dependent on the amount of feedstock in contact with the catalyst, as changes in the catalyst-to-feedstock ratio can affect product distribution. This finding is consistent with a catalytic Asbuton pyrolysis study showing that temperature and the Asbuton-to-catalyst mass ratio influence conversion and liquid product yield (Saadah et al., 2020).

Interestingly, the patterns shown in **Figure 5** indicate that increasing the catalyst mass does not necessarily compensate for the effect of high temperature. At all three catalyst mass levels, the oil yield continued to decrease as the temperature increased. This finding suggests that, under the conditions investigated, the thermal effect was more dominant than the effect of increasing catalyst mass. At high temperatures, cracking can proceed more intensively, causing primary products that could potentially condense as oil to undergo further decomposition into lighter fractions. A similar mechanism has been reported in the catalytic pyrolysis of heavy oil, in which temperature and the catalyst-to-oil ratio influence the distribution between liquid products and lighter hydrocarbons (Meng et al., 2006).

3.4 Response Surface Analysis of Oil Yield

After the interactions among the process variables were analyzed using the interaction plot, the relationships were further visualized using the contour plot presented in **Figure 6**. The contour plot illustrates changes in the oil yield as a function of the combination of two factors, while the other factor is maintained at a specified condition. This approach can be used to identify the operating region that provides a higher oil yield response prior to optimization. The use of contour plots in RSM analysis has also been applied in pyrolysis processes to identify combinations of process variables that result in optimum oil yield (Sudalaimuthu et al., 2024).

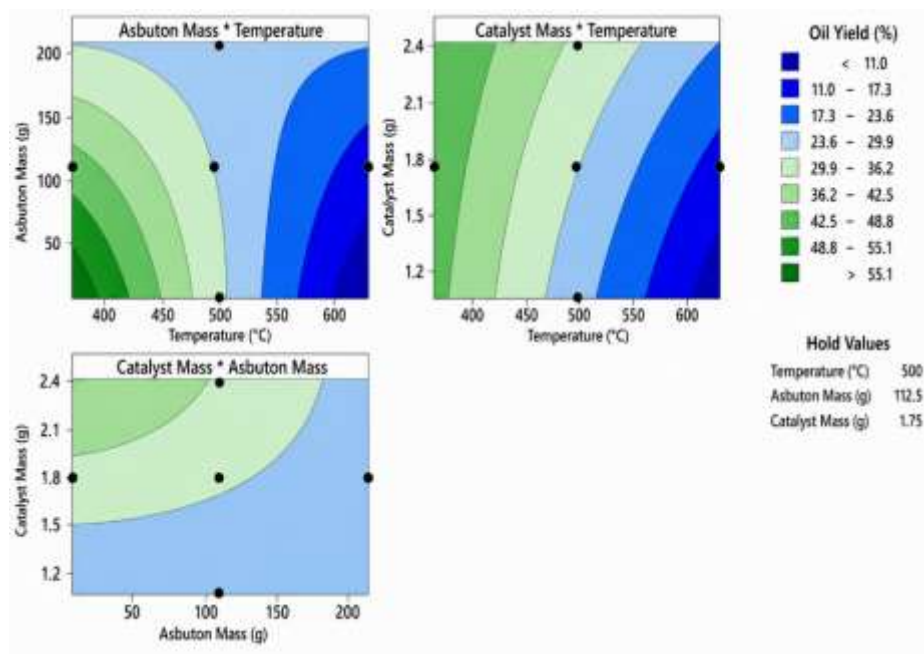


Figure 6. Contour Plot of the Interaction Effects of Variables on Oil Yield

For the relationship between Asbuton mass and temperature, the contour plot shows a color transition from green to blue as the temperature increases. The dark green region represents a higher oil yield, approximately 42.5–55.1%, whereas the blue region indicates a lower oil yield. The high-yield region is predominantly observed at temperatures of approximately 350–450°C and at relatively low to moderate Asbuton masses. This finding indicates that low to moderate temperatures are more favorable for liquid product formation than higher temperatures. This result is consistent with previous catalytic pyrolysis studies showing that increasing the temperature beyond the optimum condition can promote gas formation and consequently reduce the liquid yield (Sudalaimuthu et al., 2024).

For the relationship between catalyst mass and temperature, the contour plot indicates that the higher oil yield region occurs at low to moderate temperatures combined with relatively higher catalyst masses. Increasing the catalyst mass can enhance contact between the feedstock and the catalyst active sites, thereby facilitating the conversion of heavy hydrocarbon components. However, the effect of the catalyst remains dependent on temperature. Previous studies on heavy oil catalytic pyrolysis have reported that temperature and catalyst-to-oil ratio are important factors that jointly determine product distribution (Meng et al., 2005).

Meanwhile, the relationship between catalyst mass and Asbuton mass shows that the higher oil yield region is located at a combination of relatively high catalyst mass and low to moderate Asbuton mass. As the Asbuton mass increases, the high-yield region becomes increasingly limited. This condition can be attributed to changes in the feedstock-to-catalyst ratio. A higher catalyst mass provides a greater number of active sites; however, increasing the feedstock amount without a corresponding increase in catalyst mass can reduce the relative availability of active sites for the amount of feedstock that must be converted. The influence of the catalyst-to-feedstock ratio on product distribution has also been reported in studies of heavy oil catalytic pyrolysis (Meng et al., 2004).

3.5 Optimization and Validation of the Optimum Conditions

Based on the CCD analysis, optimization was performed using the desirability function with the objective of maximizing the oil yield. The optimization results presented in **Figure 7** show a composite desirability value of 1.000, with the optimum conditions obtained at a temperature of 360°C, an Asbuton + acetone mass of 30 g, and a catalyst mass of 1.6014 g. Under these conditions, the model predicted an oil yield of 56.5786%.

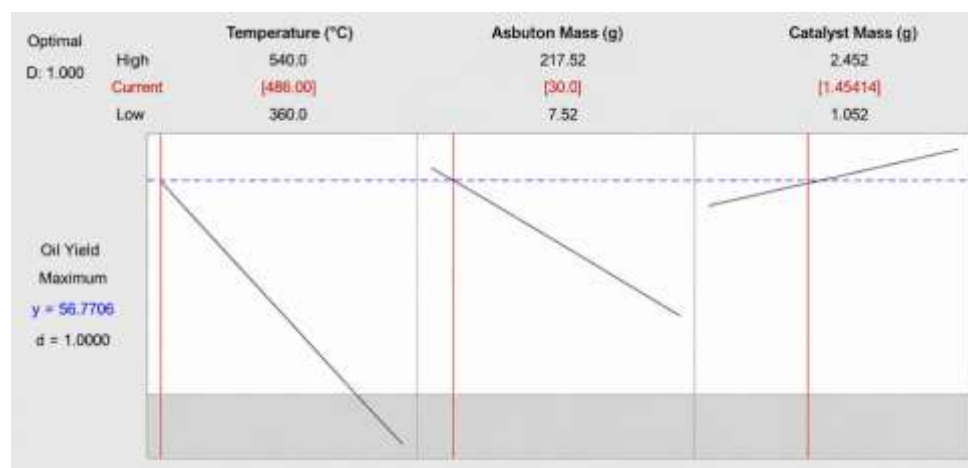


Figure 7. Optimization of Process Conditions

The optimum conditions obtained from the model were subsequently validated through an experimental run to evaluate the model's ability to predict the oil yield. The experimental validation under the optimum conditions resulted in an oil yield of 56.7706%, which was higher than the predicted value of 56.5786%. The difference between the experimental and predicted values was 0.1920 percentage points, corresponding to a percentage error of 0.34%. The relatively low error indicates very good agreement between the model prediction and the experimental result. These validation results demonstrate that the CCD model was able to adequately describe the relationship between the process variables and oil yield under the optimum conditions. A similar approach has been applied in pyrolysis optimization studies using RSM, in which the optimum conditions predicted by the model

were experimentally validated by comparing the experimental and predicted responses. Close agreement between the two values is commonly used as an indicator of the model's ability to predict the response under optimum conditions (Mishra & Meikap, 2023).

The experimental oil yield of 56.7706% also represents an improvement compared with the highest oil yield obtained in the initial experiments, which was 45.17%. Thus, CCD optimization increased the oil yield by approximately 11.60 percentage points, or approximately 25.68% relative to the highest oil yield obtained under the initial experimental conditions. This finding demonstrates that CCD optimization can be used not only to determine the effects of individual process factors but also to identify a combination of operating conditions that provides a higher response. The application of RSM/CCD for optimizing pyrolysis product yield and experimentally confirming the predicted optimum conditions has been widely reported in pyrolysis research (Onay & Koca, 2020).

4. Conclusion

Based on the results of this study, the pyrolysis of Asbuton using the FeNi/TiO₂ green nanocatalyst demonstrated that temperature, Asbuton mass, and catalyst mass significantly affected the oil yield. The CCD analysis showed that temperature was the most dominant factor, followed by catalyst mass and Asbuton mass, while the interactions between temperature and Asbuton mass and between Asbuton mass and catalyst mass were also significant. The optimum conditions were obtained at a temperature of 360°C, an Asbuton + acetone mass of 30 g, and a catalyst mass of 1.6014 g, with a predicted oil yield of 56.5786%. Experimental validation under these conditions resulted in an oil yield of 56.7706%, with an error of 0.34%, indicating good agreement between the predicted and experimental results.

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