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OPTIMIZATION OF n-HEPTANE ISOMERISATION USING NI DOPED ON ZSM5 CATALYST VIA RESPONSE SURFACE METHODOLOGY

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ABSTRACT
Ni-modified ZSM-5 catalysts with varying nickel contents were successfully synthesized via the impregnation method and evaluated for their catalytic activity in n-heptane isomerization. Characterization using XRD, SEM, BET, and FTIR confirmed that the structural integrity and crystallinity of the ZSM-5 framework were retained, while nickel incorporation effectively modified the acidity. BET results showed a slight decrease in surface area and pore volume with increasing nickel content. Catalytic performance was assessed in a reflux setup across a temperature range of 50–200 °C, reaction times of 20–180 minutes, and catalyst loadings from 0% to 15% Ni-ZSM-5. GC-MS analysis was used to determine product composition. Response surface methodology based on a Box-Behnken design was employed to optimize the process. The highest yield of isomerized n-heptane (84.52%) was achieved at 112 °C, 10% catalyst loading, and 180 minutes. Yields above 70% were generally obtained at higher catalyst loadings (10–14%), extended reaction times, and elevated temperatures. The results demonstrate the industrial potential of Ni-ZSM-5 as a cost-effective catalyst for octane number enhancement in gasoline production. Nickel offers a cheaper alternative to noble metals, and the moderate reaction conditions reduce operational energy demands. Additionally, the catalyst's thermal stability and ease of preparation by impregnation make it attractive for scalable applications in the petroleum refining and petrochemical sectors.

Keywords: n-heptane isomerization, Ni-ZSM-5 catalyst, Zeolite modification, Catalytic activity, Response surface methodology, Octane enhancement

INTRODUCTION

The current global environmental concerns have prompted regulations to reduce the level of aromatic compounds, particularly benzene and its derivatives in gasoline, isomerisation of n-alkanes is becoming a major alternative for enhancing octane number. A Series of solid acid catalysts comprising of Freidel crafts, zirconias, MoO3-based (MOB), chlorinated Al2O3, heteropoly acids and bifunctional zeolite-based catalysts have been studied in this regard (Galadima et al., 2009). The use of friedel crafts catalysts has been discontinued due to corrosion and disposal problems. MOB and heteropoly acids have good resistance to nitrogen and sulphur in a reaction stream but have poor thermal stability, are difficult to regenerate and with their mechanism of action is only partly resolved. Bifunctional catalysts with metallic and acidic functions are widely used in hydrocracking and hydroisomerization processes, and hydrocracking and hydroisomerization of hydrocarbons over bifunctional catalysts involve rather complex reactions, including (de)hydrogenation, hydrogenolysis, isomerization, aromatization, and cyclization of hydrocarbons (Lugstein et al., 1999 and Wei et al., 2020). On the one hand, the role of metallic components, like Pt, Pd, Ni, Co, Mo, and their sulfides, is to catalyze hydrogenation and dehydrogenation reactions of hydrocarbons. On the other hand, acidic components over bifunctional catalysts, like Y zeolite, ZSM-5, and zeolite-like solid acids, which possess Brønsted acid sites (BAS), can crack and isomerize olefinic intermediates into smaller olefins (Kuznetsov, 2003, Wei et al., 2020). Among the solid acid catalysts, due to its high thermal and hydrothermal stability, ZSM-5 is widely used for isomerization, alkylation, and aromatization of hydrocarbons in the petrochemical industry (Chen et al., 2007; Yamaguchi et al., 2014; Sun et al., 2020, Wei et al., 2020).

Noble metals are commonly employed in theoretical studies of active sites on hydroisomerization catalysts, but their practical application in industry is limited due to economic constraints. In the use of bifunctional catalysts, such as chlorinated alumina catalysts enabled by Pt, can lead to corrosion and pollution due to the need to introduce chlorine gas repeatedly (Tan et al, 2020). Research in this topic focuses on alternatives to transition metals as metal sites, which are often less expensive. Previous research has explored transition metal-based bifunctional catalysts as a cost-effective solution (Sriatun et al, 2019, Tan et al, 2020). Ni belongs to the same group (group VIII) as the noble metals Pt and Pd share numerous similarities in atomic structure. Furthermore, it has been thoroughly researched and even used in actual production. Ni is the most active non-noble metal for dehydrogenation in n-alkane hydroisomerization (Sriatun et al, 2019, Tan et al, 2020). Ni's poorer (de)hydrogenation activity compared to noble metals necessitates increasing the Ni loading amount during operation to compensate. Increasing Ni concentrations will not have an economic impact because the price of Ni is approximately one-thousandth that of Pt.

MATERIALS AND METHODS

Reagents

n-heptane, Nickel Chloride hexahydrate and ZSM-5 which was purchased from PIANGXIAN, CHINA

Preparation of the Catalysts

Ni on ZSM-5 catalysts was prepared using the conventional wet impregnation method as described by Sriatun et al. (2019). About 5 g of ZSM-5 was treated with 10% (w/v) aqueous nickel chloride.

The suspension was stirred for 2 hr at room temperature. Then, the solvent was evaporated at 60 °C. The material was

dried in an oven (110 °C, 24 hr), and calcined NiO/ZSM-5 in a furnace (550°C, 5 hr).

Evaluation of Catalytic Performance

The catalyst performance was carried out via isomerization of n-heptane using a reflux set-up, 25ml of n-heptane was transferred into a 100ml round-bottom flask and 0.1g of the catalyst was added and the mixture heated with a heating mantle. The evaluation experiments were carried out at temperature ranges of 50°C to 200°C with reaction time of 20 minutes to 180 minutes and catalyst loading of 0% Ni-ZSM5 to 15% Ni-ZSM5, the chemical composition of the isomerized n-heptane was determined using GC-MS analysis.

Table 1: Process Variables and their Levels to be used in the Box-Behnken Design

Independent Variables	Unit	Level of Variables	
		Low(-)	High(+)
Temperature	°C	50	200
Time	Min	20	180
Catalyst loading	%	0	15

GC-MS Analysis

GC-MS analysis was carried out at Multi-User Science Research Laboratory, Umaru Musa Yar'adua University, Katsina on Agilent Technologies GC 7890B, MSD 5977A. The injection volume was 1 µl and the inlet temperature was maintained at 300oC. The oven temperature was programmed initially at 50°C for 2 minutes and then maintained at 250°C at the rate of 8°C per minute and held at the temperature for 7 minutes, the total runs time was 30 minutes. The MS transfer line was maintained at 300°C, the source temperature was 230oC and maintained at maximum temperature of 250°C, the MS Quad at 150oC and maintained at maximum temperature of 200°C. The sample in cubicle was inserted onto GC sample holder and injected by sample injection unit of the machine. The spectrum of the separated compounds was compared with the database of the spectrum of known compounds saved in the NIST02 Reference Spectral Library (Aji et al., 2022).

RESULTS AND DISCUSSION

Characterization of the Catalyst

The catalysts (before and after doping) were characterized using a wide range of techniques. The functional groups of the Ni/ZSM-5 catalysts were analyzed using an FT-IR spectrometer in the wavenumber range from 400 to 4000 cm−1 using the transmittance mode. The morphology of the catalysts was characterized using a scanning electron microscope (SEM), and the elemental composition was determined using an X-Ray fluorescence spectrometer. And measurement of surface area, total pore volume, and mean pore radius was carried out using the method of Brunauer, Emmet, and Teller (BET) (Sriatun et al. 2019; Sun et al., 2020; and Wei et al., 2020).

Table 2: Absorption Bands of the Catalysts and Possible Functional Groups

Bond type	ZSM-5	Absorption bands (cm-1)	
		7.5%Ni-ZSM5	15%Ni-ZSM5
Al-OH-Si	3391	3382	3384
H-OH	1625	1625	1610
AlO4	1051	1051	1051
SiO4	792	790	790

The differences in the functional groups before and after doping of metals were observed. The FT-IR spectra of ZSM-5 zeolite show in the range of 400-4000 cm-1, the peaks at 682 cm-1, could be assigned to double a five-membered ring tetrahedral vibrations, and typically for the crystalline ZSM-5 zeolite, the peak at 792 cm-1and 1051 cm-1, are assigned to the internal vibrations of SiO4 and AlO4 tetrahedra and the small band near 1233 cm-1, is attributed to the asymmetric stretching vibrations of Si-O-Si. The band at 1625 cm-1is the H-OH bending vibrations of the adsorbed water molecules during the preparation of the catalyst, at 3391 cm-1corresponds to hydroxyl groups of solid catalyst, while there was no band at 3605 cm-1, which could correspond to characteristics of the protonated form of zeolite and its intensity that will correlate with the framework of aluminum (Fal) as observed by isak et al., (2015).

However, after doping of the metals on the parent zeolite, the synthesized catalyst shows no absorption band 3605 cm-1, which indicates the absence of an acidic hydroxyl group (Al-OH-Si) which has been replaced by NiO i.e the protonated form of zeolite was lost as a result of calcination, the band at 1051 cm-1, becomes a little bit narrow which corresponds to reduction of intensity and internal vibrations of SiO4 and AlO4 tetrahedra, 790 cm-1 tends to increase in the band areas. Doping of NiO on ZSM-5 had no significant influence on the framework of the parent ZSM-5 as observed by Zakari et al., (2012).

Elemental Compositions of the Catalyst

Table 2 shows the result of elemental composition, and relative concentrations of the metallic catalysts of NiO/ZSM-5 in which number of counts against Kev of each metal oxide and the support is presented. The bulk elemental composition for transition metal oxide (TMO)-doped with ZSM-5 catalyst was studied. The XRF results for NiO-doped ZSM-5 show varying concentrations of nickel oxide (NiO) across different samples. This increase in NiO concentration indicates successful doping of nickel into the ZSM-5 framework. The presence of other elements such as SiO2 and Al2O3 remains consistent, suggesting that the overall structure of ZSM-5 is maintained while nickel is incorporated Asikin-mijan et al., (2017).

Table 3: EDXRF Analysis of the Catalysts

Elemental composition	Relative concentration (%)		
	ZSM5	7.5%Ni-ZSM5	15%Ni-ZSM5
NiO	0.00441	15.406	27.064
ZnO	0.01088	0.00247	0.00285
Al2O3	10.677	9.212	7.630
SiO2	79.746	71.336	56.127
MgO	1.05	2.62	0

Surface Properties of the Catalysts

The surface area of the examined samples was determined using the Brunauer–Emmett–Teller (BET) equation, and the pore volume and pore diameter were obtained by using the Barrett–Joyner–Halenda (BJH) method with the N2 adsorption isotherm and the corresponding BET surface area, pore volume, and pore diameter are displayed in Table 4.10. The parent and NiO-modified ZSM-5 zeolites by the impregnation method have very similar N2 adsorption desorption isotherms. According to the International Union of Pure and Applied Chemistry (IUPAC) classification, the isotherms of those can be attributed to type IV, and the adsorption and desorption branches almost overlapped at relatively low pressure, indicating that the mesoporous structure and the microporous structure were preserved well after nickel modification by the impregnation method (wei et al., 2020). Compared to the parent ZSM-5 zeolite, incorporation of small amount of nickel by the impregnation method did not change the BET surface area, pore volume, and pore diameter of those materials (Table 3).

Table 4: Textural Properties of Catalysts

Catalyst	S_{BET} (m^2g^{-1})[a]	DP (nm)[b]	Vp (cm^3g^{-1})[c]	S_{Lang} (m^2g^{-1})[d]
zsm-5	374.13	2.173	0.203	516.17
ni-zsm5 7.5	328.9	2.47	0.2034	452.9
ni-zsm5 15	298.4	2.52	0.1879	411.1

a: BET surface area by Brunauer–Emmett–Teller method.
b: Mean pore diameter by BJH method.
c: Total pore volume (p/p0=0.990) (Vp) by BET plot.
d: Langmuir surface area by Langmuir plot.

Morphological Properties of the Catalyst

The SEM images provided in figure 1,2, and 3 are for different loadings of nickel (Ni) on ZSM-5 zeolite: 0%, 7.5%, and 15%.
The 7.5% sample shows more pronounced particle aggregation. The surface appears more textured and layered.
There are noticeable clusters, suggesting increased nickel loading affects particle distribution.
Moderate nickel loading may enhance certain catalytic reactions by providing more active sites, though excessive aggregation could reduce efficiency (Ali et al., 2024).
The 15% sample shows significant particle agglomeration, with larger and smoother particles dominating the image.
The surface is less textured, indicating possible sintering or excessive nickel deposition covering the ZSM-5 framework.
High nickel loading can lead to reduced catalytic activity due to blocked pores and decreased surface area (Ali et al., 2024).

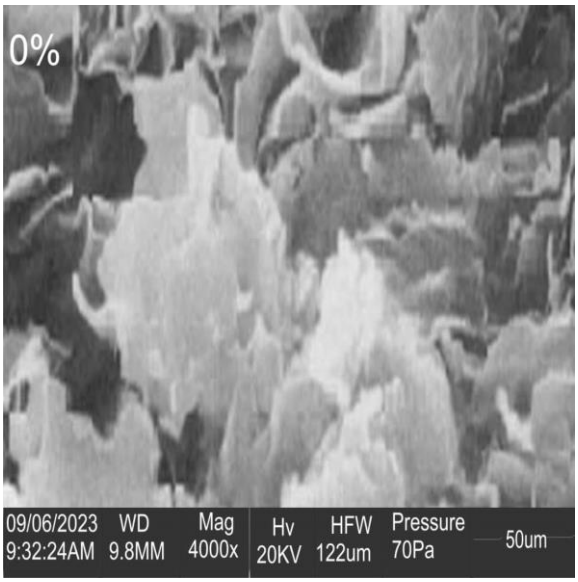

Figure 1: SEM images of 0% ZSM5

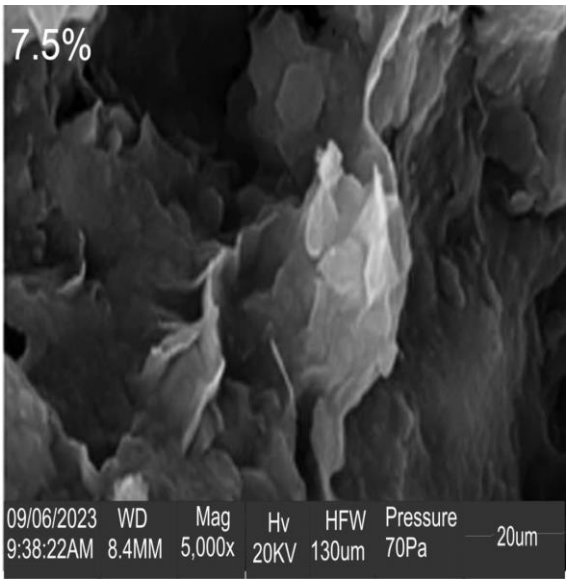

Figure 2: SEM images of 7.5% Ni-ZSM5

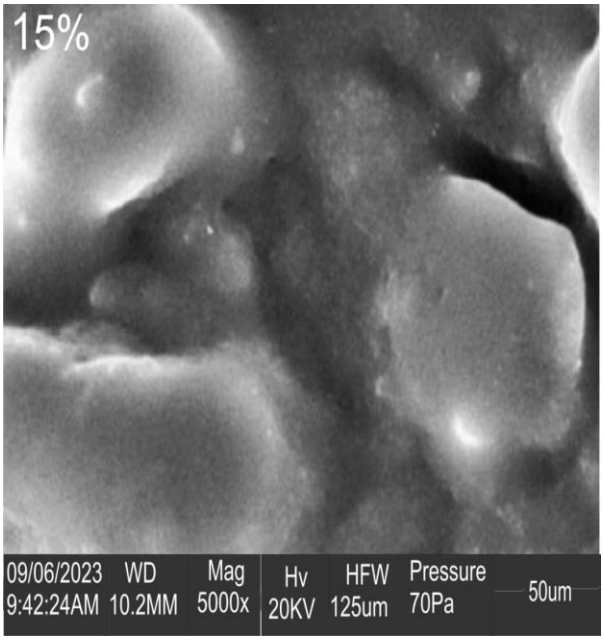

Figure 3: SEM images of 15% Ni-ZSM5

RESULTS AND DISCUSSION

Results of the Isomerization Experiments

The results of the isomerization experiments are shown in Table 5.

Table 5: Box-Behnken Design Process for Isomerization of n-Heptane and Yield Obtained

StdOrder	RunOrder	PtType	Blocks	TEMP	Time	CAT	% Yield
1	28	2	1	50	20	7.5	34.6
2	26	2	1	150	20	7.5	46.3
3	10	2	1	50	180	7.5	66.8
4	8	2	1	150	180	7.5	78.1
5	23	2	1	50	100	0	30.1
6	17	2	1	150	100	0	40
7	6	2	1	50	100	15	36.8
8	12	2	1	150	100	15	58.7
9	3	2	1	125	20	0	42.8
10	20	2	1	100	180	0	49.2
11	30	2	1	100	20	15	53
12	22	2	1	100	180	15	74.3
13	7	0	1	100	100	7.5	70.4
14	21	0	1	100	100	7.5	70.2
15	4	0	1	100	100	7.5	70.3
16	9	2	2	50	20	7.5	34.7
17	13	2	2	150	20	7.5	46.4
18	15	2	2	50	180	7.5	67
19	24	2	2	150	180	7.5	77.9
20	2	2	2	50	100	0	30.2
21	18	2	2	150	100	0	40.2
22	29	2	2	50	100	15	36.9
23	11	2	2	150	100	15	58.9
24	5	2	2	100	20	0	42.7
25	14	2	2	100	180	0	49.3
26	16	2	2	100	20	15	53.1
27	25	2	2	100	180	15	74.4
28	1	0	2	100	100	7.5	70.3
29	19	0	2	100	100	7.5	70.1
30	27	0	2	100	100	7.5	70.2

Analysis of Variance (ANOVA) for the Isomerized n-Heptane with Ni-ZMS-5

The result of the analysis of variance (ANOVA) Table 6 shows that the model P-value for the isomerized n-Heptane using Ni-ZSM-5 yield is significant. The significance of the model is confirmed by a relatively higher Fischer's 'F-statistics' value and a lower value of probability (P-value) (Chaudhary and Balomajumder, 2014).

The results of the analysis of variance (ANOVA) revealed that all linear interaction terms of the variables are statistically significant ($P < 0.05$, at $\alpha = 0.05$). It also shows that all the square terms are statistically significant except time*time which is statistically insignificant. The ANOVA result also indicates that, in 2-way interaction terms of the variables only Time*Catalyst loading is statistically significant ($P < 0.05$, at $\alpha = 0.05$), while the other terms i.e Temperature*Reaction time and temperature*Catalyst loading are statistically insignificant ($P > 0.05$, at $\alpha = 0.05$). The "lack of fit" (P-value = 0.000) implies that the data is statistically significant ($P < 0.05$, at $\alpha = 0.05$). The high correlation coefficient (R2 = 94.71%) shows that the model adequately accounts for the empirical relationship between the response (Yield) and the variables.

Table 6: Analysis of Variance (ANOVA) for the Isomerization of n-Heptane

Source	DF	Adj SS	Adj MS	F-value	P-value
Model	10	6822.04	682.20	34.03	0.000
Blocks	1	0.00	0.00	0.00	0.988
Linear	3	1652.47	550.82	27.48	0.000
Temperature, T (oC)	1	85.98	85.98	4.29	0.052
Time, t (min)	1	1259.28	1259.28	62.82	0.000
Catalyst load, c (%)	1	386.21	386.21	19.27	0.000
Square	3	2854.82	951.61	47.47	0.000
T*T	1	1310.61	1310.61	65.38	0.000
t*t	1	0.11	0.11	0.01	0.941
c*c	1	1688.68	1688.68	84.24	0.000
2-Way Interaction	3	170.94	56.98	2.84	0.065
T*t	1	1.32	1.32	0.07	0.801
T*c	1	61.53	61.53	3.07	0.096
t*c	1	116.33	116.33	5.80	0.026
Error	19	380.87	20.05		
Lack-of-Fit	15	380.83	25.39	2538.85	0.000
Pure Error	4	0.04	0.01		
Total	29	7202.91			

Key: DF= degree of freedom, Adj SS= adjusted sum of squares, Adj MS= adjusted mean squares, F-value= F-statistical values

The elimination of the insignificant terms resulted the new regression model Equation 1 with seven significant terms.

Yield (%) = -10.89 + 0.6938 T + 0.0941 t+ 2.688 c - 0.002411 T2 - 0.0972 c2+ 0.00359 tc (1)

Regression Equation in Uncoded Units

Yield = -30.11 + 1.214 T + 0.0973 t + 4.449 c - 0.005377 T2 -0.2699c2+ 0.00614 t*c (2)

Where T = Temperature, t = Reaction time, c = Catalyst loading

The new regression model Equation 1 is better than the previous regression model , in terms of (Adjusted R2= 91.93 % compared to 91.88 %), (Predicted R2 = 87.01 % compared to 84.98%).

Effect of Catalyst on the Yield of Isomerized n- heptane Produced

The effect of Ni-ZSM5 on the percentage yield of isomerization of n-heptane was investigated. The isomerized n-heptane yield was observed by varying the Ni-ZSM5 catalyst concentration or loading between 0wt%, 7.5wt%, to 15wt%, the yield increases with increasing catalyst loading. In this current study, it was observed that, the isomerized n-heptane produced reached (77%) at (7.5wt %) concentration of Ni loaded on modified ZSM-5 at 150°C, 180 minutes. Veses et al., (2016), reported that, the liquid yield of isomerized n-heptane upgrading reached (79%) by (7.5wt %) concentration Ni-loaded on modified ZSM-5.

Effect of Reaction Time / Catalyst Loading on the Yield

It was revealed from the foregone discussion on the results of regression analysis that only catalyst loading and time are significant. Hence, the isomerisation yield was affected by time and catalyst loading. The plot shows a significant variation in yield with changes in catalyst load. Higher catalyst loads generally correlate with increased yields, particularly evident in the darker green areas representing yields above 70%. The highest yields are observed at longer reaction times, around 120 to 180 minutes, especially when combined with higher catalyst loads.

The optimal region for achieving yields greater than 70% appears at higher catalyst loads (around 10-14%) and longer reaction times (over 120 minutes). This suggests that both parameters need to be carefully controlled to maximize efficiency. Lower yields (less than 40%) are predominant at shorter reaction times and lower catalyst loads, indicating that insufficient catalyst or time leads to suboptimal conversion.

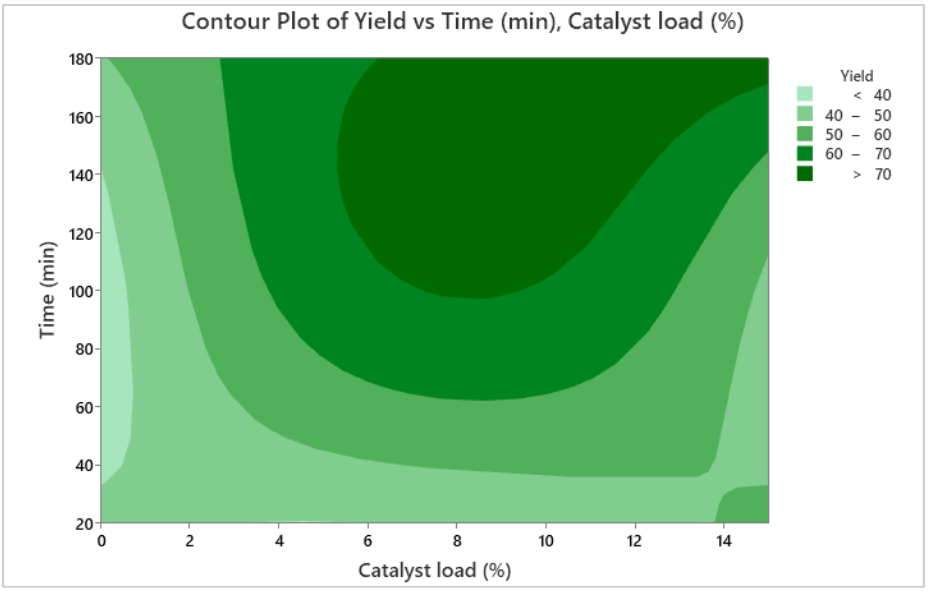

Figure 4: Effect of Catalyst Loading and Reaction Time

Effect of Temperature on the Yield Produced

A temperature range of 50oC - 200oC was used in this study as shown in table 4.11. Temperature has significant effect on the yield of isomerized n-heptane produced. The maximum yield of >80% was achieved at a temperature of 170oC, while the lowest yield obtained at 50oC was <40 %, the differences may be due to the other factors involved (i.e reaction time and catalyst loading). The highest and lowest yield of hydrocarbons at 160oC, was 70 % at 150 minutes and catalyst loading of 7.5 wt%, and 73.30 % at 170 minutes and catalyst loading of 15 wt%. This implies that, as the temperature increases from 50oC to 150oC, the yield obtained also increases. High temperature accelerates the isomerisation process by increasing the kinetic energy of the colliding molecules at faster rate. However, as the temperature increases from 150oC to 200oC, the yield obtained also increases from 70 % to 80 %.

Ridwan et al., (2020), also reported that, the yield of isomerized n-heptane increases with increase in temperature from 100oC to 200oC.

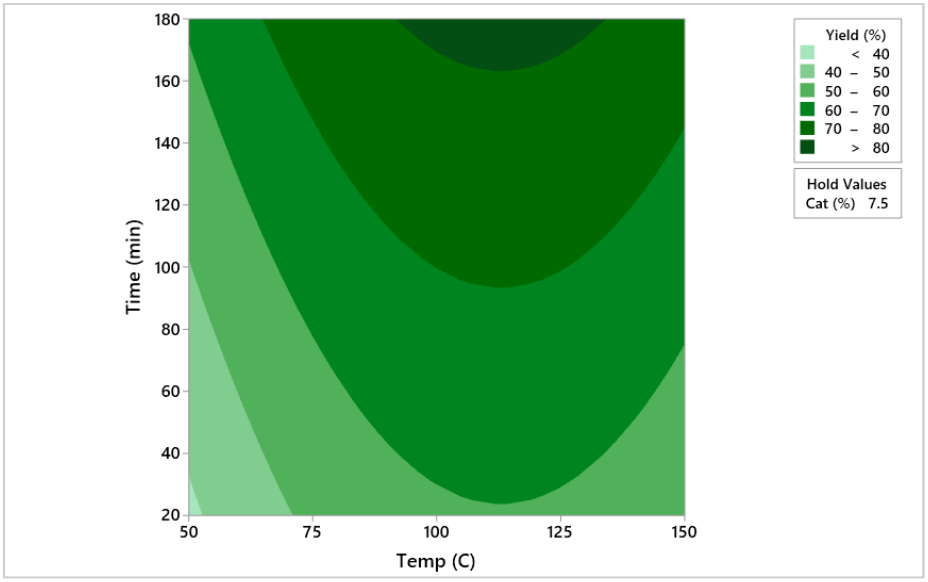

Figure 5: Effect of Temperature and Time

Response Optimization and Validation

The expected outcomes for optimal solutions attained through optimization are shown in Table 7. With optimal process conditions of temperature 112.62 0C, 143.350C and 81. 460C, time 180, 180 min and 180 min, catalyst loading of 10.30%, 10.40% and 10.60% in the validation process, the solutions predicted maximum isomerization yield of 84.52%, 79.53% and 79.18% with desirability of 0.380989, 0.181372, and 0.167280 for each of the three solutions. isomerization yield of 85.25%, 80.47% and 80.11% was obtained from validation of experiment carried out at the levels of the process variables predicted in solutions 1, 2 and 3. It was observed that the optimized predicted and experimental validated results for the three solutions were 84.52%, 79.53%, 79.18% and 85.25%, 80.47%, 80.11 % respectively, which indicate a relative deviation of 0.31 %, 0.19% and 0.48% respectively. The optimization model is reliable, which may make it more desirable for larger-scale of isomerization of n-heptane. The optimization plot is shown below

Table 7: Predicted Results of Optimization and Validation of the Isomerization

S/N	TEMP	TIME	CAT	% Yield		Composite Desirability
				PRED	EXPT	
1	112.62	180	10.30	84.52	85.25±0.100	0.380989
2	143.35	180	10.40	79.53	80.47±0.108	0.181372
3	81.46	180	10.60	79.18	80.11±0.040	0.167280

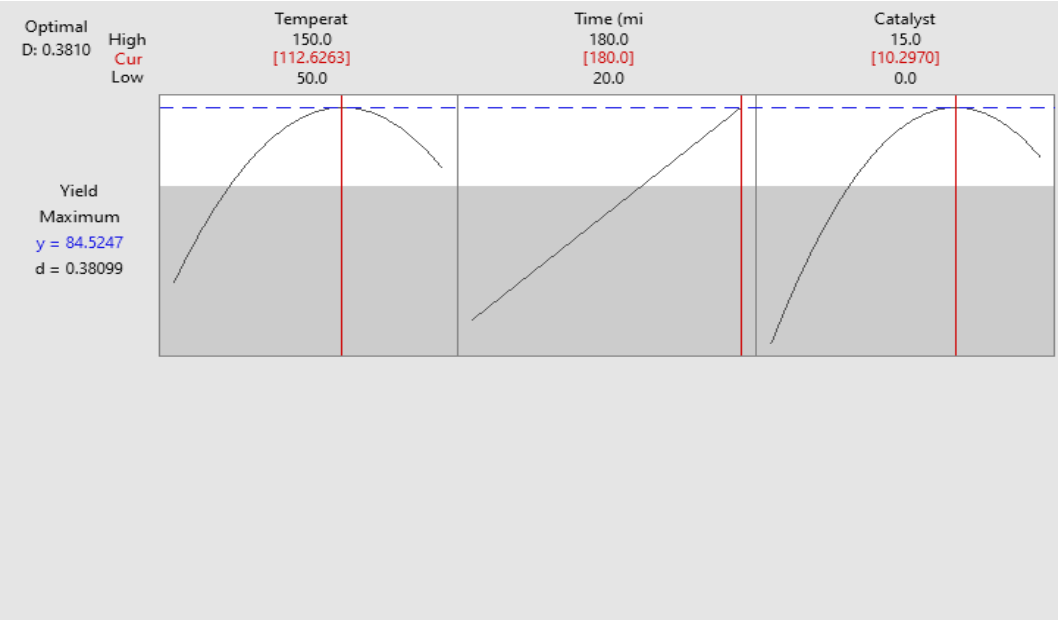

Figure 6: Optimization Plot

CONCLUSION

The study shows the isomerization of n-heptane to iso-heptane using NiO on ZSM-5 catalyst, by varying reaction temperature, reaction time and catalyst loading. The following conclusions were drawn from the studies:

i. Ni-modified ZSM-5 zeolites with different nickel contents were prepared.
ii. The XRD and SEM analyses showed that ZSM-5 zeolites were preserved well after the introduction of a small amount of nickel.
iii. BET analysis showed that the textural properties of Ni-modified ZSM-5 zeolites, especially for the microporous surface area, were hardly affected, indicating that those micropores were also not blocked by nickel. The FTIR characterization showed that the total amount of acid sites, especially for the strong acid sites, decreased by the introduction of nickel to ZSM-5 zeolites.
iv. Isomerization of n-heptane was successful based on the GC-MS result.
v. The optimal region for achieving yields greater than 70% appears at higher catalyst loads (around 10-14%), longer reaction times (over 120 minutes) and higher temperatures (over 150°C) . This suggests that both parameters need to be carefully controlled to maximize efficiency.
vi. Optimization of process parameters for isomerization of n-heptane was determined by response surface based on Box-Behnkhen design. The optimum yield of the isomerized n-heptane (84.52%) was achieved at the temperature, catalyst and time of 112°C, 10% and 180 minutes respectively.
vii. Other side reactions such as oxygenation, cyclization and oligomerization must have taken place based on the available products.
viii. These findings underscore the industrial relevance of Ni-ZSM5 as a viable and cost-effective alternative to noble metal-based catalysts for improving fuel quality through octane number enhancement.
ix. The use of an inexpensive transition metal(nickel), combined with low energy requirements and scalable synthesis via impregnation, makes the system highly attractive for commercial applications in petroleum refining sector.

Overall, the research highlights the potential of optimizing process variables to achieve high conversion efficiency while maintaining economic and environmental viability.

RECOMMENDATIONS

i. The catalysts should be more comprehensively characterized to understand its acidic and metal sites, which influence isomerization and other reactions.
ii. The sources of oxygenated compounds and long-chain hydrocarbons observed in the GCMS results should be investigated to improve process purity.
iii. Additional structural elucidation techniques (e.g., NMR) should be used to confirm structural isomers and quantify branching degree.

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