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Boosting catalytic performance in ZSM-5 (50) catalyst cracking: microwave and Taguchi method with nitrogen activation

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Abstract

Microwave power has a more significant effect on cracking with less energy consumption. The microwave power and the catalysis increase the racking rate and the conversion at lower temperatures. Accordingly, the generated hot spots in the catalyst offer a suitable condition for cracking (high rate and low temperature). The microwave technique was used to conduct the cracking of heavy naphtha. A set of experiments was done with microwave power (750–1250 W), preheating temperatures (150–250 ˚C), and space velocities (2–6 l/hr) with and without nitrogen injection. The nitrogen injection enhanced the conversion for all situations. The best result has a conversion of 47.37% with nitrogen injection at microwave power of 1250 W, a flow rate of 4 l/hr, and a preheating temperature of 250˚C. Generally, it was noted that the flow rate did not affect the conversion. Compared to conventional techniques, the microwave power increased both the reaction and conversion rates, allowing for work at lower temperatures that traditional methods cannot achieve. This study investigated how microwaves with catalysts affect residence time, energy savings, and conversion. The microwave technique experiment could be scaled up to become a mass production unit in any refinery.

Keywords: Microwave reactor; Catalytic cracking; Heavy naphtha cracking; Microwave heterogeneous catalysis.

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1- Introduction

Naphtha is the primary feedstock for cracking reactions; however, ethane, propane, butane, and gas oil can also be used as alternative feedstocks for this process [1, 2]. The ethylene production unit employs both gas and liquid cracking, employing ethane and naphtha as feedstocks [3]. The conversion of naphtha is directly influenced by its composition, which depends on its origin and location [4]. The origins of naphtha are defined by a significant composition of aromatics and olefins, which are among the many properties associated with molecule structure and chemical properties that define the substance's origins. [5]. The catalytic cracking of several types of hydrocarbons (including naphtha) using ZSM-5 zeolites was studied to produce aromatics and olefins [6]. The reaction takes place at significantly lower temperatures, ranging from 450 to 650 ˚C, in comparison to thermal cracking. Moreover, the production of ethylene and propylene by microwave is sufficiently high to be comparable to that of thermal cracking [7].

Zeolite is a porous material made of crystalline aluminosilicates, characterised by a large surface area and the capacity to adsorb molecules [8]. Zeolites are classified into two categories: synthetic and natural [9]. ZSM-5 is an appropriate catalyst for naphtha cracking. It is a synthetic form of zeolite [10]. The particle size, shape, and porosity of the catalyst significantly influence the hydrodynamics and reaction rate [11]. ZSM-5 is considered an efficient catalyst in the cracking process due to its unique structures, acidity, shape selectivity, and thermal and hydrothermal stability [12]. ZSM-5's qualities enable its application as a catalyst in hydrocarbon cracking. ZSM-5 is often synthesised from hydrogels by hydrothermal methods [13].

The fundamental interactions of microwaves with catalysts in naphtha cracking have been investigated. When comparing conventional heat sources with microwave energy, microwave energy has a great effect on many chemical reactions[14]. Microwave irradiation has a significant advantage, including a short reaction time, a low reaction temperature, a short start-up time, low-cost, accurate process control, and high-energy efficiency[15]. There are two distinct components to the microwave heating technique: electrical energy that is transformed into microwave energy and microwave energy converted to effective heat. [16].

Some researchers compared the amount of energy required for chemical reactions when using microwaves versus conventional methods and showed that the chemical activity of microwaves energy (10-1000) is times faster than that of conventional heating [17]. Microwave technology has grown in popularity and is generally regarded as a technological improvement in this field since it can increase the rate of many chemical reactions [18]. According to some research, the microwave's effectiveness could be related to the

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electromagnetic effect, which has been associated with the selective absorption of energy by polar molecules [19]. Because of its efficient energy transfer mechanisms, microwave power is quickly emerging as an essential resource for a number of industrial processes that involve direct heating of materials [14]. The primary objective is to determine the optimal conditions for the efficient and successful application of microwave technology in the cracking of heavy naphtha. Microwave power has been previously shown in several applications, including drying processes, sintering of ceramic components, metal processing, and plasma generation [20].

The utilisation of microwave power for naphtha cracking is favoured due to its ability to operate at lowered temperatures, produce greater conversion rates, and consume less energy, which is one of our conclusions on microwave-assisted cracking. Moreover, microwave-assisted reactions are generally more rapid and produce superior outcomes relative to conventional methods. The microwave-enhanced yields of the desired aromatic product reduced the presence of environmentally harmful compounds [21].

2- Materials

2.1. Feedstock

The Al-Najaf refinery produced heavy naphtha by atmospheric fractional distillation with an initial and final boiling point of (80˚C to 185˚C) and a specific gravity of (0.6846). The raw material for the catalyst cracking process is heavy naphtha. The heavy naphtha analysis carried out in the Basra oil company is shown in Table 1.

2.2. ZSM-5 catalyst

The spherical shape catalyst type ZSM-5 (50) with particles size between (2 – 3) mm and silicon to aluminum ratio is 50:1 mol. (Congyi City Meiqi Industry and Trade Co., China).

2.3. Nitrogen gas

The catalyst reactivation by nitrogen injection with flowrate (30 cm^3/min) started 15 minutes before the beginning of the experiment.

Table 1. PONA analysis of heavy naphtha

Composition	Wt.%
n-Paraffin	31.026
i-Paraffin	33.258
Naphthene	10.003
Aromatic	18.938
Olefine	1.169
Undefined	5.606

3- Experimental method

3.1. Catalytic cracking unit

The catalytic continuous flow reactor was utilised for heavy naphtha cracking. The parts of the process are a feedstock drum, feed pump, electrical preheater, microwave oven, QVF tubular reactor, cooling and condensation system, separator, gas flow meter, one-way valve, and electrical power cabinet, as shown in Fig. 1.

The feed stock is initially stored in the feed tank and subsequently circulated through the heating element to the reactor, which is positioned in the center of the microwave oven. The reactor's exiting product is directed to the condenser, which condenses a portion of it. The remaining portion is expelled into the atmosphere outside the laboratory. The condensate product was collected and subsequently analyzed using gas chromatography.

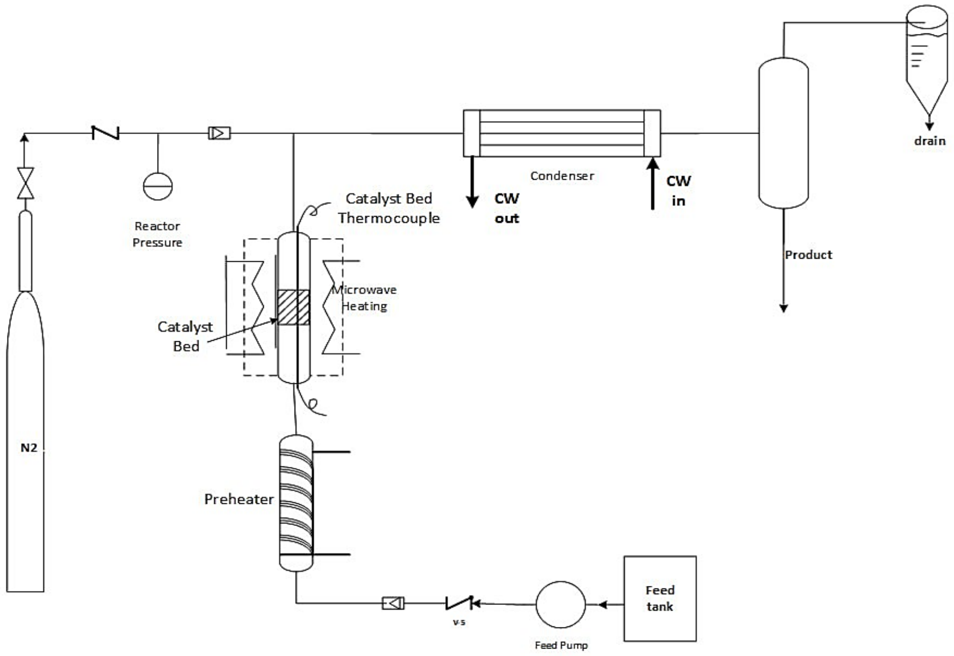


Fig. 1. The catalytic cracking unit process flow diagram

3.2. Reactor

Microwave effective zone is 240 mm made of QVF tube with 12.7 mm inner diameter. The height of the microwave reactor is divided into three parts (80 mm). The middle zone was packed by catalyst, and the top and bottom zones (inert particles) assist in improved distribution and avoided channelings.

4- Characterization of the ZSM-5 (50)

The evaluation of the catalyst needs to Various chemical, physical, and microstructural characteristics.

4.1. X-ray diffraction analysis (XRD)

The diffraction lines (2θ and corresponding $d_2\theta$) observations in the XRD patterns of the zeolite ZSM-5 (50) is full agreement with the corresponding values for zeolite (Sodium Aluminum Silicate Hydrate), which has a chemical formula $((Na_2O)_{0.07} (Al_2O_3) (SiO_2)_{70.97} H_2O)$ and matched (JCPDS, Card No. 00-042-0120) as shown in Fig. 2.

4.2. Morphology analysis (SEM)

SEM micrographs of ZSM-5 (50) offer the details related to crystal forms, external surfaces, and purity of the phases. Clear variations were observed in magnification times of (10, 25, 50, and 200 kx) as shown in Fig. 3. There are two forms of crystals; one of them is hexagonal, and the other is rod-like, with an average diameter of 19.41 nm, as indicated in the deep focusing to aspect ratio. Aggregation, twinning, and sometimes intergrowth affected crystal size distribution phenomena. Single crystals were sensed by lower magnification.

4.3. Surface area and pore volume

Nitrogen physical adsorption using the BET method measures the specific surface area and total pore volume of ZSM-5 (50). Increasing the SSA) of zeolite ZSM-5 (50) is caused by increasing the combined total pore volume with a variation in SiO_2/Al_2O_3, as illustrated in SEM images. The highest SSA values give a higher cracking rate. Moreover, the zeolite ZSM-5 (50) samples show type IV isotherms with distinguishing hysteresis loops corresponding to mesoporous materials according to the IUPAC classification of adsorption isotherms, as shown in Fig. 4. The values of the surface area (SBET) and total pore volume are given in Table 2.

Higher p/p_0, as well as a hysteresis loop in type IV isotherms, increased adsorbed volume due to capillary condensation below the expected condensation pressure of the adsorbate. The low p/p_0 region in type IV isotherms has no effect on adsorbate volume, which indicates the absence of the micropores. The preformation of an adsorbed layer on the pore walls occurs by capillary condensation, which is formed by multilayer adsorption. Both processes occur simultaneously in the range of 0.3-1 p/p_0. The vertical asymptotic profile at high values of p/p_0 presented by isotherms, which is characteristic of macro and mesoporous with non-uniform size. Zeolites with smaller crystal sizes and higher surface areas exhibit better catalytic activity and selectivity. The presence of acidic sites within the pores contributes to their catalytic behavior. Moreover, the zeolite ZSM-5 (50) samples show type IV isotherms with distinguishing hysteresis loops corresponding to mesoporous materials according to the (IUPAC) classification of adsorption isotherms as shown in Fig. 5.

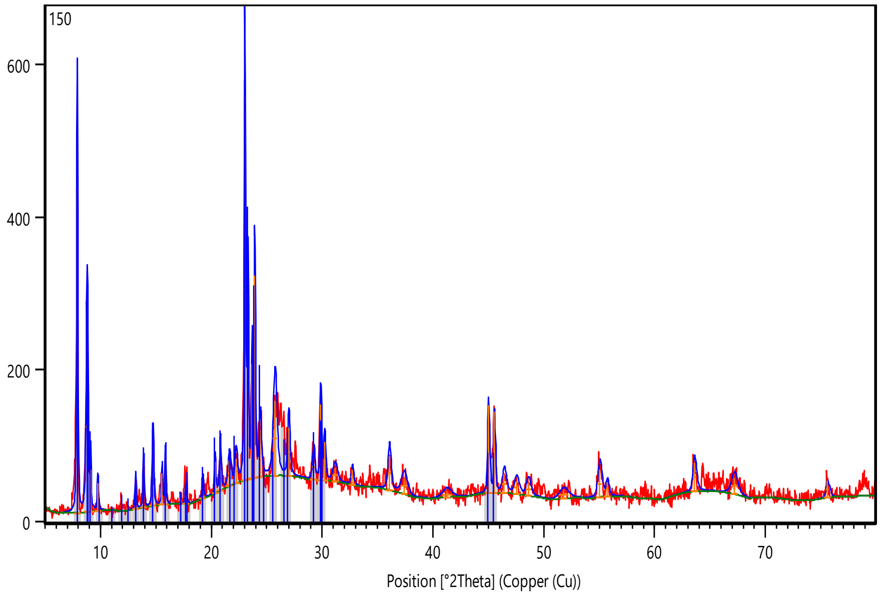
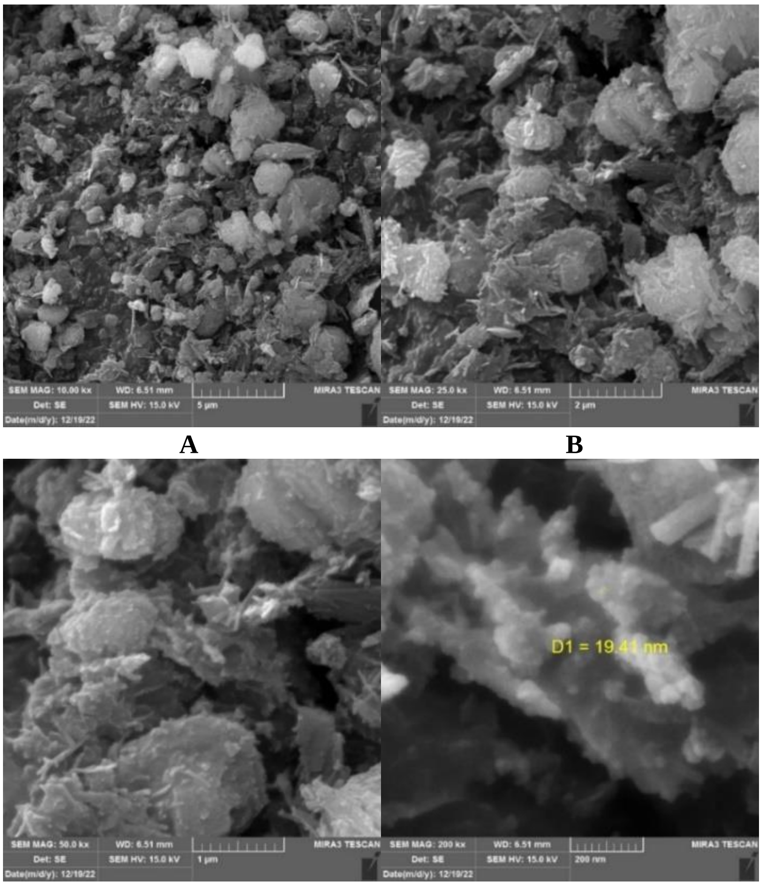


Fig. 2. The XRD patterns of the Zeolite ZSM-5(50) powders



A B

C D

Fig. 3. The micrographs of ZSM-5 (50) A: magnification times 10kx, B: magnification times 25kx, C: magnification times 50kx, and D: magnification times 200kx

Table 2. The Results of the Surface Area Test

Sample Code	SBET (m2/g)	Pore Volume (Vp) (cm^3/g)
50	220.49	0.2241

4.4. X-ray florescence analysis (XRF)

XRF technique analyzed the quantitative chemical compositions of the ZSM-5 (50) as listed in Table 3. SiO_2 (93.457%) is dominant in zeolites ZSM-5 (50), while Al_2O_3 (1.91%) is the second highest percentage. The morphology and characteristics of zeolites could be adopted from Si/Al ratio. The ratio of silicon to aluminium (Si/Al) has been calculated for zeolite, which is nearly (50).

5- Cracking of heavy naphtha

The heavy naphtha was analyzed by PIONA (paraffin, i-paraffin, olefin, naphthene, aromatic) according to the ASTM D5134 standard. A gas chromatograph (MS-GC manufactured by Agilent) was employed to evaluate the reaction conversion. The catalytic cracking of heavy naphtha was performed with different parameters (microwave power, preheating temperature, and flow rate) and SiO_2/Al_2O_3 ratio in ZSM-5 catalysts (50). The cracking of heavy naphtha compounds is mostly represented by the conversion of paraffins to aromatics and olefins. The catalytic cracking reactions were performed with and without nitrogen injection.

5.1. ZSM-5 (50) catalysts

The determination reaction conversion as Taguchi method response after 12-minute period for each set of variables (with and without nitrogen injection), were done Table 4.

The signal-to-noise ratios response for the conversion of paraffin has been affected by the interaction of variables (with and without nitrogen injection). Fig. 6 shows that the reaction conversion has the same behaviour for all control variables with and without nitrogen injection. The preheating temperature and microwave power have direct proportionality with reaction conversion, but the flow rate has inverse proportionality for the two sets.

In Table 5 and Table 6, the p value (without nitrogen injection) is more than 0.05 for the flow rate. The descending arrangement of control variables are (microwave power, preheating temperature, and flowrate, with p values 0.003, 0.032, and 0.592, respectively). The regression equation has an acceptable p value for the control variable with nitrogen injection) and arranged according to their effectiveness as follows (preheating temperature, microwave power and flowrate with p values, 0.002, 0.020, and 0.498, respectively).

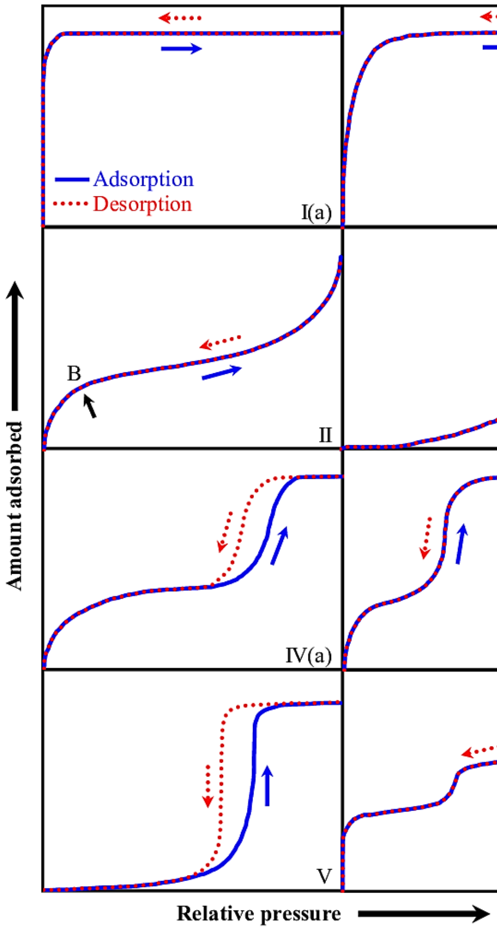


Fig. 4. Classification of adsorption/desorption isotherms of nitrogen at 77°K by the International Union of Pure and Applied Chemistry

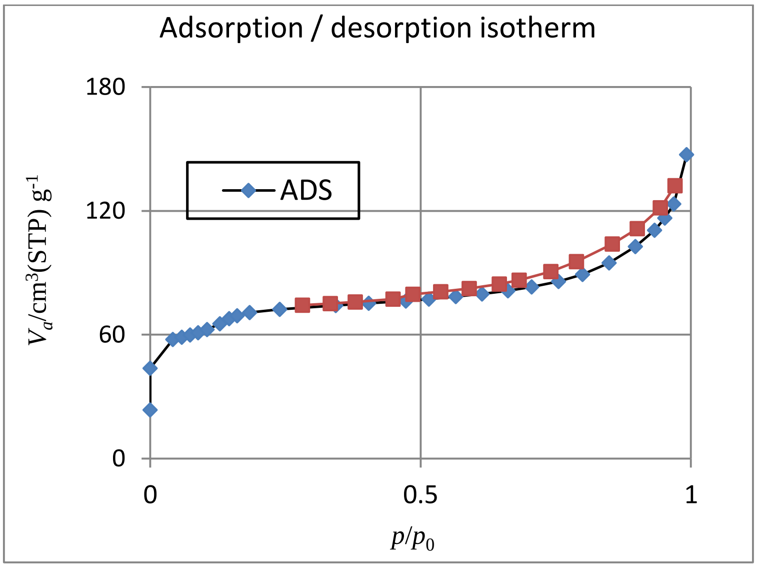


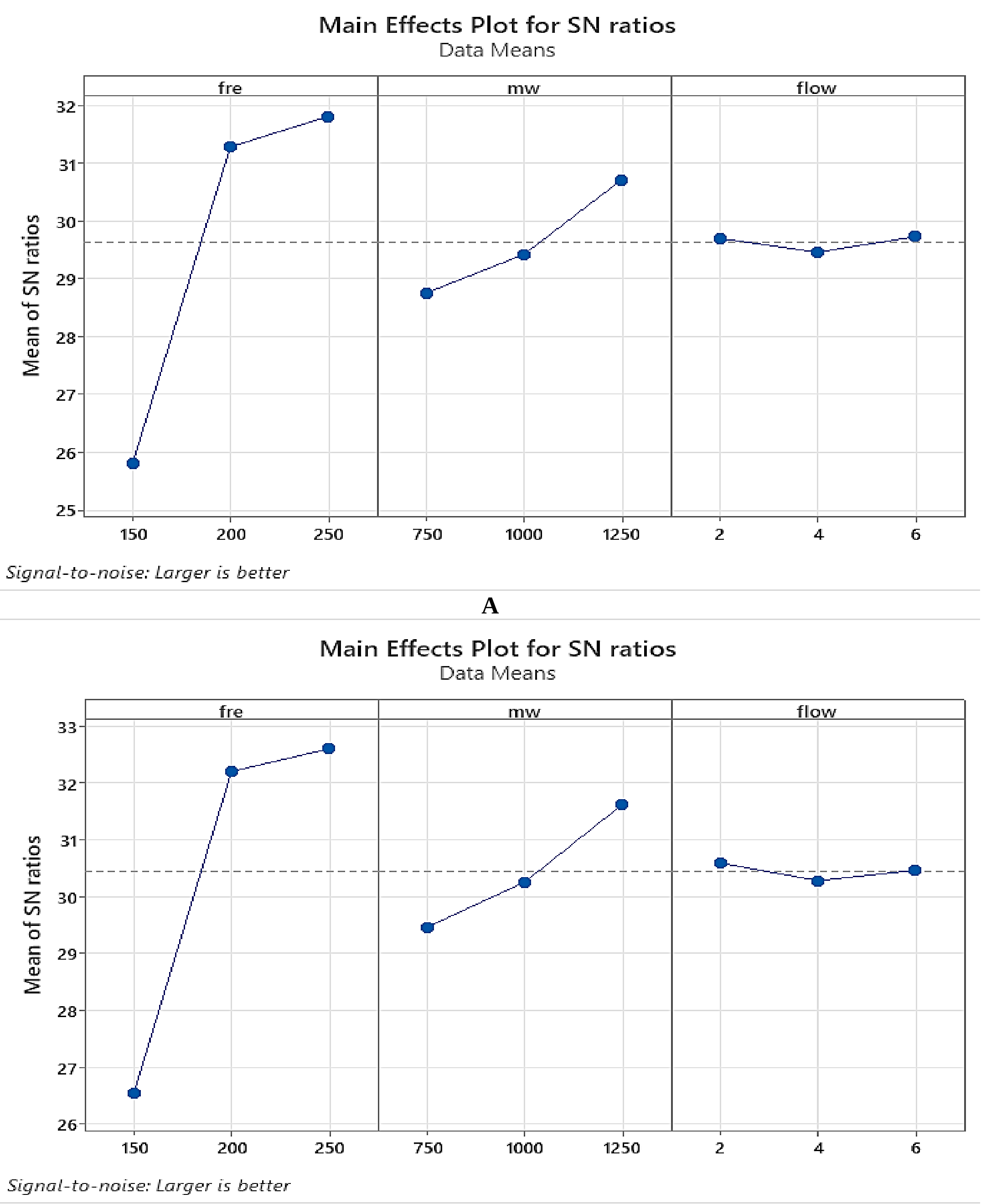
Fig. 5. Nitrogen adsorption/desorption isotherms at 77°K for ZSM-5 (50)

Table 3. The ZSM-5(50) XRF analysis results

SiO_2 %	**Al_2O_3 %**	**Na_2O %**	**K_2O %**	**CaO %**	**MgO %**	**Fe_2O_3 %**	**TiO_2 %**	**SO_3 %**	**MnO %**	**P_2O_5 %**	**Cl %**	**LOI %**
93.457	1.91	0.703	0.121	0.053	0.057	0.256	0.085	0.013	-	0.025	0.054	3.21

Table 4. Conversion of paraffin using Taguchi's OA for zeolite ZSM-5 (50)

Preheating	Microwave	Flowrate	Conversion without N_2 injection	Conversion with N_2 injection
150	750	2	17.605	19.090
150	1000	4	18.446	20.206
150	1250	6	22.910	24.876
200	750	4	33.288	36.437
200	1000	6	35.805	39.480
200	1250	2	41.349	46.954
250	750	6	35.177	37.804
250	1000	2	39.280	43.328
250	1250	4	42.793	47.373



A

B

Fig. 6. The signal-to-noise ratio for preheating, microwave power, and flow rate at zeolite ZSM-5 (50) A: without nitrogen injection, and B: with nitrogen injection

Table 5. Analysis of Variance for SN ratios at zeolite ZSM-5 (50)

		without nitrogen injection					**without nitrogen injection**				
Source	DF	Seq SS	Adj SS	Adj MS	F	P	Seq SS	Adj SS	Adj MS	F	P
Preheating	2	66.2678	66.2678	33.1339	341.25	0.003	68.7101	68.7101	34.3551	472.96	0.002
Mw	2	5.9465	5.9465	2.9733	30.62	0.032	7.1221	7.1221	3.5611	49.02	0.020
Flow	2	0.1341	0.1341	0.0670	0.69	0.592	0.1467	0.1467	0.0734	1.01	0.498
Residual Error	2	0.1942	0.1942	0.0971			0.1453	0.1453	0.0726		
Total	8	72.5426					76.1243				

Table 6. Response for Signal-to-Noise Ratios at zeolite ZSM-5 (50)

	without nitrogen injection			**without nitrogen injection**		
Level	**preheating**	**Mw**	**flow**	**Preheating**	**mw**	**flow**
1	25.81	28.76	29.71	26.55	29.47	30.60
2	31.28	29.43	29.46	32.20	30.26	30.28
3	31.81	30.72	29.73	32.60	31.62	30.46
Delta	6.00	1.96	0.27	6.05	2.15	0.31
Rank	1	2	3	1	2	3

The reaction conversion with control variables (without nitrogen injection) formula has a better interaction effect than single variables. R^2 value equals 98.93% means good fitting. ANOVA analysis formulates an equation of reaction conversion with a control variable (Eq. 1).

$$Conv.\% = -18.71 - 0.01\mathrm{B} - 4.2\mathrm{e}^{-3}A + 17.34C + 2.74\mathrm{e}^{-4}\,AB - 0.031\mathrm{B}C - 0.0107AC \tag{1}$$

The set of the experiments with nitrogen injection composed Eq. 2 between the control variable and conversion. The experimental data have an R^2 value equal to 99.24%.

$$Conv.\% = -21.61 - 0.038\mathrm{B} - 3.92\mathrm{e}^{-3}A + 20.84C + 3.3\mathrm{e}^{-4}\,AB - 0.036\mathrm{BC} - 0.013AC \tag{2}$$

It was found that the reaction conversion directly proportional to the microwave power and preheating temperature while the flowrate change has no effect (almost equal) with and without nitrogen injection as shown in Fig. 7. Within the studied range of preheating temperature, the reaction conversion increased with increasing preheating temperature with and without nitrogen injection. All the results with nitrogen injection are better than those without nitrogen injection.

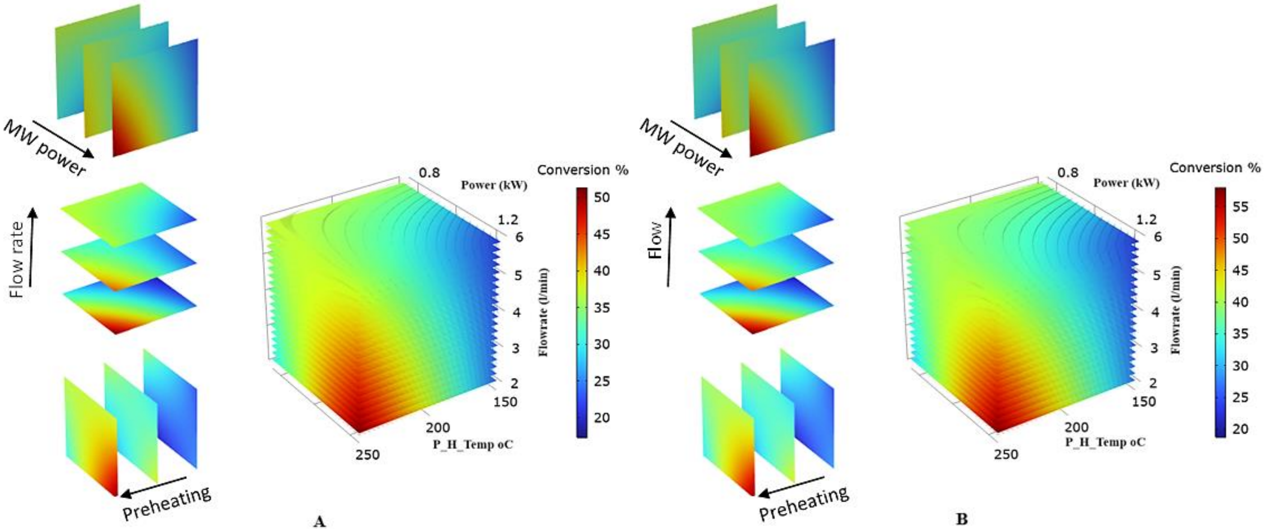


Fig. 7. 3D representation and the projections of a regression equation A: (1), B: (2)

At constant microwave power and preheating temperature, the reaction conversion decreased with increasing of the flowrate while it is almost equal due to interaction of the three variables in two sets (with and without nitrogen injection) The microwave power always increased the reaction conversion in two sets in the full range. The best reaction conversion (43.3%) was found at (1000 W) microwave power, (2 l/hr) flowrate, and (250 ˚C) preheating temperature with nitrogen injection.

The results of the three catalysts give reaction conversion with nitrogen injection greater that without nitrogen injection due to reactivation of catalyst by nitrogen and change the hydrodynamic which increased the velocity. Because the nitrogen increases the contact time and number of molecules with catalysts, Catalytic cracking of heavy naphtha with nitrogen injection also has advantages for preventing deactivation due to de-alumination of zeolites that increase catalytic stability.

In the comparison of energy consumption between the microwave catalytic cracking and traditional methods, it was found that the catalytic cracking reactor can save 50-60 % compared to the traditional catalytic cracking reactor. The catalytic cracking of heavy naphtha proceeds at higher than 450 to 650 ˚C in traditional methods, while it needs 150-250 ˚C in microwave catalytic cracking, so the microwave catalytic cracking is preferred as superior to the catalytic cracking in terms of total energy consumption.

6- Conclusion

The synergistic effect of microwave power with catalysis assists the process in producing the required products in a shorter time and with less energy. Microwave energy creates hot spots in the catalyst's structure, greatly benefiting low-temperature cracking by reducing energy consumption and extending equipment life. The rapid heating of the microwave process increases its efficiency by increasing the temperature rapidly, which accelerates the cracking reaction. In addition, the creation of localized hot spots in the material ensures that the cracking reaction occurs efficiently and uniformly. The creation of the "hot spots" results from the absorption of microwave energy by specific material components.

Nitrogen injection was found to increase reaction conversion in all catalysts. This is due to the role of nitrogen, which modifies the surface properties of the catalyst and creates favorable conditions for the cracking reaction. The best result has a conversion of (47.37%) with nitrogen injection at a microwave power of (1250 W), a flow rate of (4 l/hr) and preheating temperature of 250 ˚C). Generally, it was noted that the flow rate did not affect the conversion. Compared to conventional techniques, microwave power increases the reaction conversion rates, allowing for work at lower temperatures that traditional methods cannot achieve. This study investigated how microwaves with catalysts affect residence time, energy savings, and conversion.

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تعزيز الأداء التحفيزي في التكسير الحراري بواسطة المحفز ZSM-5 (50) باستخدام الميكروويف وتاكوشي مع التنشيط بالنيتروجين

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الخلاصة

طاقة الميكروويف تمتلك تأثير عجيب مع استهلاك أقل للطاقة عند استخدامها بالتكسير الحراري .تعمل طاقة الميكروويف والمحفز على زيادة معدل التكسير والتحويل عند درجة حرارة منخفضة .وعليه، فإن البقع الساخنة الناتجة على الحدود البلورية للمحفز توفر ظروفًا مناسبة للتكسير (معدل تكسير مرتفع ودرجة حرارة منخفضة) . تم إجراء التكسير الحراري للنفتا الثقيلة بتقنية الميكروويف .وتم إجراء مجموعة من التجارب بقوة الميكروويف (٧٥٠-١٢٥٠) واط ودرجات حرارة التسخين المسبق (١٥٠-٢٥٠ درجة مئوية) وسرعات (٢ -٦ لتر/ ساعة) مع وبدون حقن النيتروجين .كانت أفضل نتيجة تحويل (٤٣,٣٧) مع حقن النيتروجين. بقوة الميكروويف (١٢٥٠) واط ودرجات حرارة التسخين المسبق (٢٥٠ درجة مئوية) وسرعات (٤ لتر/ ساعة). بصورة عامة نلاحظ ان سرعة الجريان لا تؤثر على معدل التحويل. وبالمقارنة بالتقنيات التقليدية، فإن طاقة الميكروويف تزيد من معدل التفاعل ومعدل التحويل، مما يسمح بالعمل في درجات حرارة أقل لا تستطيع الطرق التقليدية تحقيقها . وقد بحثت هذه الدراسة في كيفية تأثير الموجات الدقيقة مع المحفزات على وقت المكوث وتوفير الطاقة ومعدل التحويل. ويمكن توسيع نطاق تقنية الميكروويف لتصبح وحدة إنتاج ضخمة في أي مصفى.

الكلمات الدالة: مفاعل الموجات الدقيقة، التكسير الحراري التحفيزي، تكسير النافثا الثقيلة، مفاعل التكسير، الموجات الدقيقة والتحفيز غير المتجانس.