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Modification of Y-zeolite mesoporosity by ultrasonic-assisted technique as active catalysts for catalytic cracking of VGO

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Abstract

In this study, Pristine Y zeolite (CBV 400) of Si/Al (2.5) was treated to produce modified mesoporous zeolites. The process involved the aid of post-synthesis sequential dealumination in its conventional mode and ultrasonic-assisted desilication techniques. Both ethylene-diaminetetraacetic acid (EDTA) and oxalic acid (OX) were tested as chelating reagents in the dealumination step for 1, 3, and 6 h. The dealuminated samples of zeolite were treated sophisticatedly with sodium hydroxide NaOH in a water bath sonication at a frequency of 20 Hz and 65 ºC for different times of 5 and 15 min. Dealumination of the original zeolite by OX acid for 3h and desilication of the acid-treated sample with NaOH solution for 15 min has improved the mesoporosity by around 120% higher than the parent sample of V_{meso}=0.08 m^3/g. The modified sample has a mesopore volume (V_{meso}) of 0.2 m^3/g and an external surface area (S_{external}) of 89 m^2/g. The resulting mesoporous zeolite was examined for stability measurements via thermal treatment at 550 ºC for 3h, which reveals high stability. Parent and modified zeolite were tested as heterogeneous catalysts for the catalytic cracking reaction with VGO and 1g catalyst load with different temperatures (from 400 to 420 °C) and a reaction time of 480 minutes. The reaction was carried out using a batch reaction system, and a higher yield of gasoline (about 44.44 vol%) was obtained using the modified zeolite (UDSY15-OX3) at 420 °C reaction temperature, as opposed to the direct use of the non-modified catalyst, which gives out only 18.4 vol% gasoline yield. Thus, commercializing the OX-NaOH pathway with the assistance of ultrasonic energy can decrease the treatment duration and energy consumption, producing a thermally stable mesoporous material to catalyze the cracking reactions of heavy oils.

Keywords: Mesopore volume; Y zeolite; dealumination; Ultrasonic-assisted desilication; Modified Zeolite; VGO; catalytic cracking.

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1- Introduction

Today, crude petroleum is one of the most important fuels. It is also used to make smaller, more efficient fuels like gasoline, gas oil, and kerosene. The main problems in the crude petroleum sector are falling demand for low-quality fuels around the world and changing oil prices. For another thing, Earth's primitive oil supplies can't be replaced and won't last forever. Some sources say that almost half of the world's oil supplies are already being used [1, 2].To solve this problem, some people have been thinking about using different types of oil. In order to reach these goals, the petrochemical and processing industries now think about the heavy oil conversion process. [1, 3]. Thermal cracking [4] and catalytic cracking [5, 6] are a few of the several methods by which heavy hydrocarbons can be transformed into light fuels. Among these, thermal cracking faced problems like not being cost-effective and having to operate in demanding settings (high temperatures). One recent approach to addressing these problems has been considered catalytic cracking.Vacuum Gas Oil (VGO) is a significant intermediate product in refining, derived from the vacuum distillation of heavy crude residues. It serves as the primary raw material for catalytic cracking units, which decompose it into lighter, higher-value value such as gasoline products (C5-C12). Gasoline is the primary product of C5-C12) hydrocarbons,but an intermediate distillate fraction (light cycle oil (LCO), (C12-C20) hydrocarbons. Zeolite Y is practically utilized for catalyzing cracking reactions due to its significant specifications, such as substantial surface area, acidic activity, and ion exchange function [7]. The microporous structure of zeolites Y, particularly the low Si/Al ratio of around 2.5, limits the diffusion of species, inhibiting the activity of zeolites Y for catalyzing and reducing the conversion of feedstock. In addition, deposition of coke obstructs the micropores' active sites[8, 9]. Therefore, zeolite Y with modified surface properties to enhance the transfer of molecules via acid-active sites is essential in fluid catalytic cracking (FCC) technology to fractionate vacuum gasoil in the petroleum industry [10, 11]. Accordingly, modification of zeolite Y to allow better catalytic properties and a mesoporous skeleton with a hierarchical porous framework can effectively enhance the active sites for more accessibility, which is highly beneficial in the catalytic cracking reactions of heavy petroleum feed materials[12-14].

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Production of mesopores hierarchical Y-zeolites by post-synthesis dealumination-desilication processes has been confirmed as an efficient method that can be efficiently applied via acids and alkaline treatments [15-17]. The Y zeolites with a Si/Al molar ratio of around 2.5 are treated with dealumination to reduce Al contents to improve their thermal stability through catalyzing the high temperature reactions of about 500 – 550 °C [18]. In practice, the dealumination scenario eliminates the structural aluminium from zeolites using mineral and chelating acids such as hydrochloric acid, sulphuric acid, citric acid, ethylene-diamine-tetra-acetic (EDTA) acid, and oxalic (OX) acid [19-22]. In the desilication step, alkaline reagents, such as sodium hydroxide NaOH [21-24], are employed for reducing the Si species. This step becomes practical only after decreasing the Al concentration to obtain low Si/Al ratios[25-27]. The desilication part is critical to enhancing the mesoporosity and re-crystallinity of the modified zeolites [26, 28].

The post-synthesis treatment can be associated with energy-intensive means, including operation at relatively high temperatures rather than mechanical agitation. Synthesizing mesoporous zeolite via alternative energies, such as ultrasound, has been proposed instead of the typical hydrothermal protocol [25]. Ultrasound with frequencies of more than 20 kHz has been experimented with to enhance the synthesis of porous materials such as zeolites to enhance the transfer of molecules via acid-active site in a technique that is known as sonochemistry [29]. Ultrasound transmits through liquids, causing vibrations of molecules and resulting in the rapid formation and collapse of cavities or bubbles in a liquid medium. Such phenomena are responsible for many effects [30], including the aid of microscopic agitation that increases the Si mass transfer [28].

This work explored the potential of using ultrasound irradiation to intensify the alkaline treatment step in the sequential post-synthesis treatment of pristine zeolite Y, aiming at preparing hierarchical Y zeolites more efficiently than the conventional sequential method under hydrothermal conditions. The first-step chemical treatment of the pristine Y zeolite for dealumination was performed under hydrothermal conditions using EDTA and OX chelating agents at different treatment times. Then, the second step of alkaline treatment (of the dealuminated Y zeolites) was performed comparatively using the conventional hydrothermal treatment and ultrasound-assisted treatment in the NaOH solution at 65°C. The resulting Y zeolites are comprehensively characterised regarding their physical and chemical properties, such as crystallinity, porosity, Si/Al ratio, and acidity, as well as being assessed using the model cracking reactions, demonstrating the effectiveness of the developed ultrasonic treatment to improve the sequential post-synthesis treatment for making mesoporous Y zeolites.

2- Methodology

2.1. Material

Y-zeolite (CBV-400) of a 2.5 Si/Al ratio as shown on Table 1, was obtained from Zeolyst International (China). Acids of 99% EDTA ($C_{10}H_{16}N_2O_8$) and OX ($C_2H_2O_4$) were supplied by Sigma Aldrich with pH values of 1.44 and 2.50 measured at 10 g/L and 23 °C. The alkaline material of NaOH with a purity of 97% was also provided by Sigma Aldrich.

Table 1. Chemical composition of parent hy zeolite

Component	MgO	Al_2O_3	SiO_2	P_2O_5	CaO	K_2O	Cl	SO_3
Wt.%	0.0034	22.13	60.2	0.607	0.1128	0.0012	0.01254	0.00050

2.2. Methods

2.2.1. Dealumination step

The zeolite substance first dealuminated EDTA and OX. The acids were prepared by dissolving 1.6 g of acid in 50 mL of distilled water to obtain a concentration of 0.11 M. 3.337 g of the zeolite was treated with a particular acid at 100 °C (373.15 K) for 1, 3, and 6 h. The mixture was cooled to about 25 °C and introduced to a centrifugal instrument at 4400 rpm. After that, the mixture was separated and splashed 5 times with deionized water.

The collected solid material was dehydrated at 110 °C. The resulting samples were then remarked as DAY-EDTA# and DAY-OX#, where the DA denotes the dealumination process, and the symbol # represents the time of treatments. The experimental setting of the dealumination step is portrayed in Fig. 1.

2.2.2. Desilication step

The DAY-EDTA# and DAY-OX# (acid-treated) samples were used for the alkaline treatments. These samples were mixed with the alkaline solution of 0.2M NaOH at 65°C under ultrasonic conditions. The ultrasound-assisted process was achieved by sonication of the mixture for 5 and 15 min via an ultrasonic bath (JP-040S, 50 Hz). The experimental setting of the ultrasonic-assisted desilication process is shown in Fig. 2.

The products were then split by centrifugation and washed 5 times with water. After that, the samples were subjected to a heating treatment overnight at 100°C to remove moisture and for further characterization stages. The resulting samples were remarked as UDSY-EDTA# and UDSY-OX#, where UDSY refers to the ultrasonically desilicated samples treated with NaOH in reaction times of (t) h. The dehydrated samples were then introduced to a high-temperature heating course for stability measurements, where the produced zeolites were calcined at 550 °C for 3h via an electric furnace.

Fig. 3 depicts the whole post-synthesis hydrothermal process of the conventional dealumination and ultrasonic-assisted desilication steps.

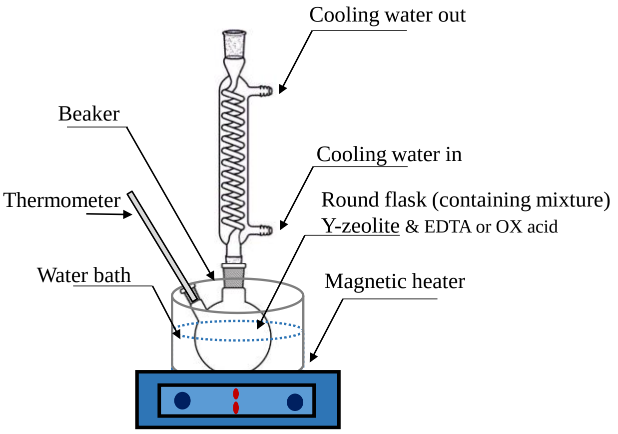

Fig. 1. Schematic diagram of dealumination step of Y-zeolite

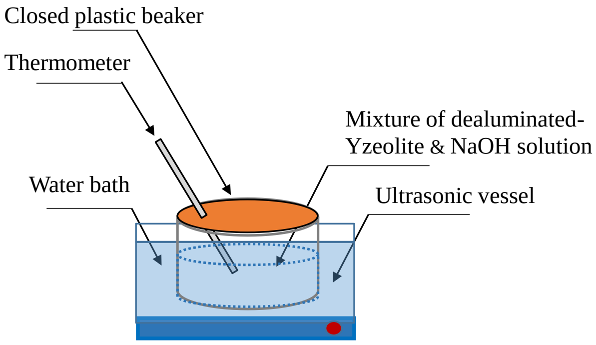

Fig. 2. Schematic diagram of ultrasonic-assisted desilication of Y-zeolite

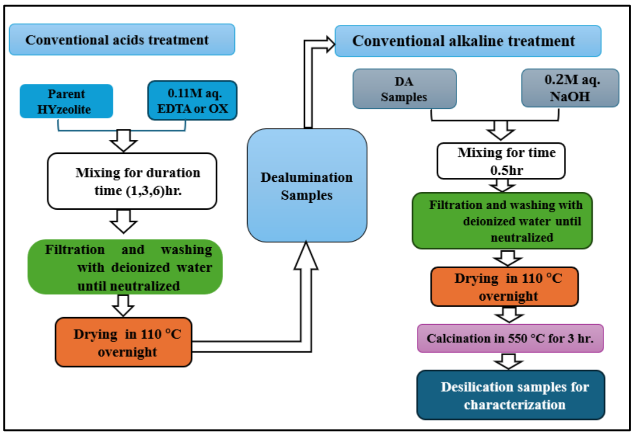

Fig. 3. Flowchart of the experimental setting of the conventional dealumination and ultrasonic-assisted desilication processes of Y-zeolite

2.2.3. Characterization

The samples were characterized by measuring the elemental content, surface area, pore sizes, and structural features. The surface area was measured by the Brunauer-Emmett-Teller (BET) technique. The pore size, N_2-physisorption isotherms, and surface area of the original, dealuminated, and desilicated zeolites were determined utilizing a highly accurate analyzer (BELSORP-mini II, BEL, Japan). The micropore volumes and external surface areas were estimated via the t-plot method. The mesopore volume was obtained from the subtraction of the total and the micropore sizes at $p/p^{\circ} = 0.95$. The micro- and mesopore sizes were also calculated in comparison with the total volume as percentages. The crystallographic features of samples were determined via X-ray diffraction (XRD), using the XRD-6000, Shimadzu, Japan diffractometer with Cu Kα radiation, 1.5406 Å wavelength, 2-theta window of 5° – 50°, step size of 2°/min, and voltage of 40 kV. The relative crystallinity (RC) of the produced zeolite samples was estimated in comparison with the parent sample (RC = 100%) based on the integrated area under the curve of the characteristic peaks by the equation below [30, 32].

$$\mathrm{RC} = \frac{\sum \text{integrated peak area of treated Y zeolite}}{\sum \text{integrated peak area of parent Y zeolite}} \times 100\% \qquad (1)$$

The samples' compositions, mainly Si and Al, were determined by the X-ray fluorescence apparatus (XRF Spectro-Xepos, Ametec, Germany). The results of the calcined were performed by using XRD in a 2θ extent of 10.5° – 40°. Collected liquid products were weighted and then analyzed by a distillation method using a petroleum fraction distillation apparatus ASTM-D86. The amounts of gasoline, kerosene, and gas oil were also weighted and quantified considering boiling point temperature ranges from initial boiling point (IBP) to end boiling point (EBP) =30°C to 175, 176 − 250, and 251 – 292 °C, respectively.

2.2.4. Catalysis (Ion Exchange, Calcined and crushing, and sieving)

Zeolites were selected, and ion exchange was employed to convert all samples from their sodium form to their hydrogen form prior to catalysis. The process involved three sequential treatments using a 0.1 M aqueous NH4NO3 solution, each comprising 2 g of Na from Y zeolite in 1 L of solution, maintained at room temperature for 8 hours. The samples were rinsed with deionized water, separated, and subsequently dried overnight at 110 °C. The ion-exchange process was conducted three times, including the post-treatment phase. To obtain H-form zeolites for catalysis [20], the ion-exchange products were calcined at 550 °C for 3 hours with a heating rate of 5 °C/min. Subsequently, all catalysts were pressed at 12 tons, crushed to 1.7 mm, and then sieved for evaluation in cracking reactions using VGO as the model compound. Fig. 4 illustrates the processes of catalyst pressing, crushing, and sieving.

2.2.5. Experiments with catalysts

Vacuum gas oil was cracked in a catalytic cracking unit (FBR) (e.g., Fig. 5) at 400 and 420°C. While the catalyst (1 g) stayed fixed, the weight hourly space velocity

(WHSV) was 12 h-1. Table 2 shows the VGO's salient features.

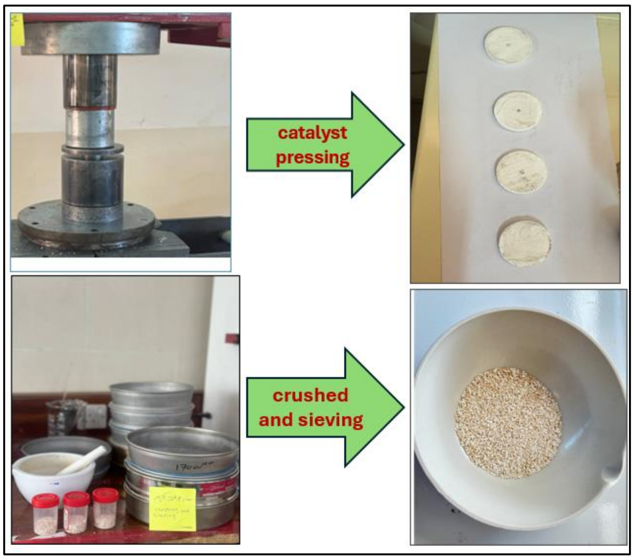

Fig. 4. processes of catalyst pressing, crushing, and sieving

Table 2. VGO properties

properties	value
Density @15 °C (gm/ml)	0.8836
Api	37.2-38.6
Viscosity @ 40 °C (cSt)	6.3
Water content ppm	98
Initial (BP) °C	180-206
Final (BP) °C	318-344
Color	1

Before the catalytic reaction, the catalyst bed was preheated at 350°C for 2 h, (N_2 as the inert carrier gas at 240 $mL.min^{-1}$) was introduced to initiate the cracking. Through the reaction and drying procedures, the product liquids were collected in corresponding glass receivers at the exit of the low-pressure separator, as shown in Fig. 5, which shows a photographic view of the experimental apparatus.

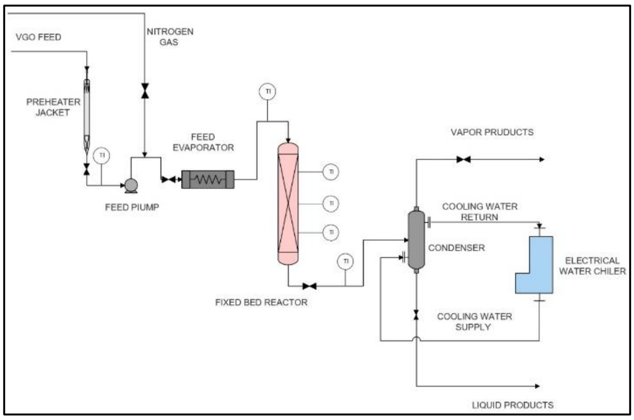

Fig. 5. Schematic diagram of the catalytic cracking unit

2.2.6. Product analysis

The collected liquid products were weighed and subsequently analyzed using a distillation method with a petroleum fraction distillation apparatus in accordance with ASTM-D86. The quantities of gasoline, kerosene, and gas oil were measured and quantified based on their boiling point temperature ranges from the initial boiling point (IBP)=30°C to 175, 176 − 250, and 251 – 292 °C, respectively.

3- Results and discussions

3.1. Porosity analysis

3.1.1. EDTA-NaOH Stage

Fig. 6 depicts the adsorption of N2 by using dealuminated-desilicated samples (UDSYt-EDTA#). The data are compared to the parent Y-zeolites sample. From Fig. 4, the results show the efficiency of the post-treatments after the acid and alkaline steps at treatment durations of 5 min (Fig. 6a) and 15 min (Fig. 6b) in the improvement of mesoporosity of the Y zeolite sample. The isotherm distribution of original Y zeolite material normally takes the Type-I shape of the microporous materials with very narrow split hysteresis loops at the region of P/P◦ = 0.4, indicating a deficiency of mesoporous volumes. After 5 min of treatment, these loops become slightly larger as displayed in Fig. 6a, with EDTA has dropped the absorbed N_2, reducing the overall adsorbed volume by around 55 cm^3/g (i.e., in the best case of 3h EDTA acid treatment).

This appears more recognizable in the sample UDSY5-EDTA3 (i.e., the 3 h acid treatment sample with EDTA, followed by 5 min alkaline treatment under ultrasound conditions). Nonetheless, the entire dealumination-desilication process. This decrease could be resulted from the strong effect of acid treatment that reduces the content of Al and some impurities, which affected the reduction of the accessible surface area. The reduction of the adsorbed N_2 in the treated zeolites in ranges lower than the original sample was also previously demonstrated by Zhang et al. [19]. Using different dealumination durations alters the Si/Al ratio between 4 and 7, with an increase of about 120% in average from the parent sample of Si/Al = 2.5. (This is the same for the sample of 3h dealumination with a Si/Al ratio of 5.5).

After 15 min of ultrasonic desilication (Fig. 6b), the N_2 adsorption of samples treated with EDTA and NaOH solution of all durations (i.e., UDSY15-EDTA#) is less than that of the original sample. The overall adsorbed volume has reduced by more than 95 cm^3/g (i.e., in the case of 3h EDTA acid treatment). The features of the adsorbed volume distribution of 15 min treated samples are like those of 5 min, except for the slightly smaller size of the hysteresis loops, especially after P/P◦ of 0.7 (clearly appears in the sample of 3 h acid treatment). The most important properties of specific total pore volume (V_{total}), mesopore volumes (V_{meso}), micropore volumes (V_{micro}), BET, and external surface area ($S_{external}$) with the values are presented in Table 3.

The desired meso-volume attains its highest magnitude of about 0.104 cm^3/g only after 3 h of acid and 5 min of ultrasonic alkaline treatments. Compared to the parent sample of 0.080 cm^3/g, the meso-volume increases by around 30%. The highest external surface area reached 66 cm^2/g at these conditions. A previous study reported that in the case of employing EDTA acid in the entire treatment of the Y zeolite with a Si/Al ratio of 2.4 using similar treatment conditions that were utilized in the current study, the obtained mesoporosity was in the extent of 0.1 – 0.14 m^3/g [31]. Such mesopore sizes are not effective in the catalytic cracking of heavy oils, and they are only applicable for treating wastewater to eliminate contaminants via adsorption techniques[32-33].

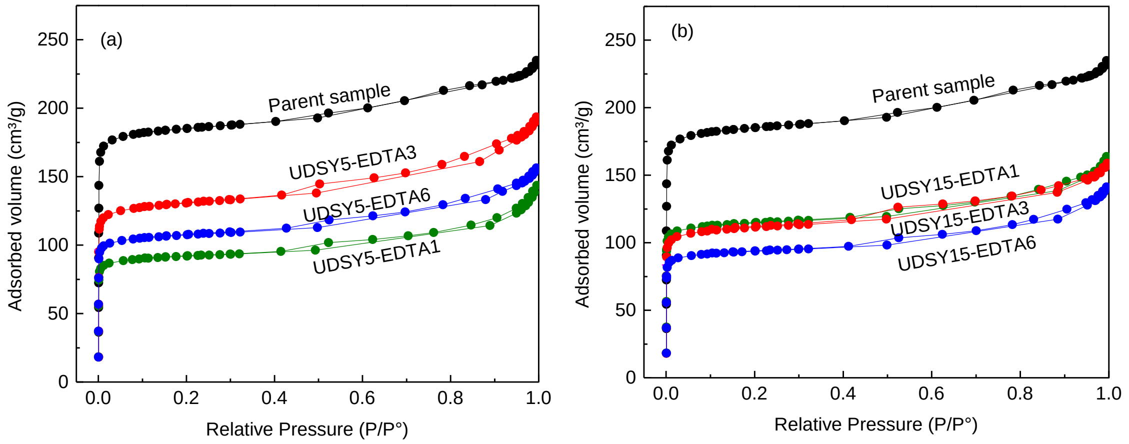

Fig. 6. N_2-physisorption isotherms of (a) UDSY5-EDTA# and (b) UDSY15-EDTA# samples for 1, 3, and 6h dealumination followed by 5 and 15 ultrasonic desilication treatment

Table 3. Porous properties of the parent Y zeolite and UDSYt-EDTA# samples

Samples	BET m^2/g	$S_{external}$$^{a)}$ m^2/g	$V_{total}$$^{(b)}$ cm^3/g	$V_{micro}$$^{(a)}$ %	$V_{meso}$$^{(c)}$ %	V_{meso} cm^3/g
Parent Y-zeolite	576	71	0.346	77	23	0.080
UDSY5-EDTA1	286	39	0.197	64	36	0.0881
UDSY5-EDTA3	409	66	0.279	62	38	0.104
UDSY5-EDTA6	335	50	0.225	64	36	0.082
UDSY15-EDTA1	356	46	0.233	68	32	0.080
UDSY15-EDTA3	347	53	0.230	65	35	0.080
UDSY15-EDTA6	284	40	0.201	63	37	0.080

(a) Estimated from t-plot technique.
(b) Obtained by single-point adsorption approach at p/p° = 0.95.
(c) Estimated from the cumulative pore volume.

3.1.2. OX-NaOH Stage

The results of treating the original Y-zeolite material with OX acid and NaOH via conventional hydrothermal dealumination at different durations and ultrasonic-assisted desilication at 5 and 15 min are displayed in Fig. 7.

From Fig. 7, the sequence processes of conventional dealumination and ultrasonic-assisted desilication of the parent sample with oxalic acid develop wider and extended hysteresis loops in the adsorbed volume curve. These loops appear after P/P° = 0.4 and broadened to the end of the relative pressure extent. In the case of the 5 min sonication process (Fig. 7a), the difference in the adsorbed volume reaches only around 28 cm^3/g (i.e., sample UDSY5-OX1). The appearance of extended and broad hysteresis loops indicates a considerable increase in mesoporosity, where it attains magnitudes of 0.16, 0.17, and 0.18 cm^3/g in dealumination times of 1, 6, and 3 min, respectively. By increasing the time of sonication within the desilication step (Fig. 7b), the adsorbed N_2 elevated to the values that are closer to the parent sample, especially at the highest P/P° values. The difference from the original sample reaches less than 6 cm^3/g (sample UDSY15-OX3). The mesoporosity values that vary with different treatment times of both dealumination and desilication processes are presented in Table 4. Just as mentioned above, the mesopore volume of the 5 min sonication-desilication samples varies between 0.160 and 0.180 cm^3/g, with an increase of about 85% more than the original sample. This increase further elevates to the range of 0.176 cm^3/g on average, and its highest value is around 0.198 cm^3/g at a treatment time of 3h dealumination and 15 min ultrasonic desilication. This exhibits a significant intensification of mesoporosity of the parent Y zeolite by about 147%.

For comparison, an experiment of using 3h delamination with OX acid and 15 min desilication with NaOH solution, without the aid of ultrasonic, was separately performed at the same experimental settings. The results showed that the BET, $S_{external}$, V_{total}, and V_{meso} values were 308 m^2/g, 66 m^2/g, 276 m^3/g, and 0.145 m^3/g. This reveals a superiority of using ultrasound-assisted technique over the conventional one. Compared with previous research, it was demonstrated that the scenario of oxalic acid-NaOH can efficiently modify the mesopore size of zeolite Y to the limits of higher than 1 cm^3/g based on the obtained Si/Al ratio, for instance, it can reach more than 2 - 3 m^3/g at Si/Al ratios of 6 – 12 [21]. In the current study, the Si/Al ratio was around 6, which will be discussed in the next sections.

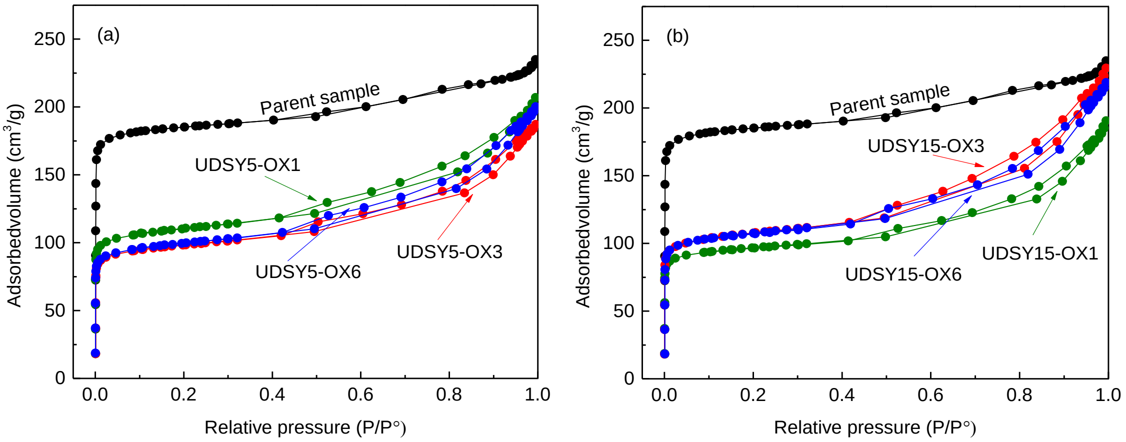

Fig. 7. N_2-physisorption isotherms of (a) UDAY5-OX#, and (b) UDSY15-OX# samples for 1, 3, and 6h dealumination followed by 5 and 15 min ultrasonic desilication treatment

Table 4. Porous properties of the parent Y zeolite and UDSYt-OX# samples

Samples	BET m^2/g	$S_{external}$ m^2/g	V_{total} cm^3/g	V_{micro} %	V_{meso} %	V_{meso} cm^3/g
Parent Y-zeolite	576	71	0.346	77	23	0.080
UDSY5-OX1	344	86	0.295	45	55	0.160
UDSY5-OX3	323	94	0.297	40	60	0.180
UDSY5-OX6	313	84	0.288	41	59	0.170
UDSY15-OX1	293	66	0.265	46	54	0.144
UDSY15-OX3	336	89	0.326	39	61	0.198
UDSY15-OX6	341	85	0.315	41	59	0.185

3.2. XRD measurements

3.2.1. XRD analysis of the original Y-zeolite

The XRD results of the parent Y-zeolite are presented in Fig. 8. The crystallographic features of the parent Y-zeolite are typically identified by its highest peaks of the XRD diffraction at 2θ magnitudes of 6.2º, 15.7º, and 23.8º, which are related to planes (111), (313), and (533), respectively. The flat background of the XRD spectrum reflects good crystallinity of the parent sample without the existence of amorphous features. The data of XRD patterns was considered a reference for estimating the relative crystallinity (RC) of the modified samples after conventional and ultrasonic treatments of alumination with OX acid and desilication with NaOH for 5 and 15 min, respectively, as a better scenario (i.e., UDSY5-OX# and UDSY15-OX# samples).

3.2.2. XRD analysis of UDSY5-OX# and UDSY15-OX# samples

The XRD analysis for modification the parent Y zeolite by dealumination with OX acid and ultrasonic-assisted desilication with NaOH steps is displayed in Fig. 9.
The data was compared with the XRD pattern of the original Y-zeolite. From Fig. 9, the characteristic peaks still present in the base XRD spectrum (i.e., the original Y zeolite sample) are observed after 5 and 15 min of sonication. These peaks do not shift for all dealuminated samples. However, these peaks appear with lower intensities than the original sample, indicating decreasing crystallinity of the UDSYt-OX# samples. This could be due to the sequence loss of the framework Al and Si species. The XRD patterns of 5- and 15-min sonicated samples (Fig. 9a, b) seem quite similar, with the existence of an amorphous background at the 2θ window of about 15 – 35º due to leaching a high amount of Al and Si and/or constitution of amorphous silica phases. These outcomes were also demonstrated in a previous study using the sequential post-synthesis approach to improve the mesoporous properties of CBV300 zeolite[19]. The disappearance of new peaks reflects the nonexistence of intermediate or unstable crystalline phases. The literature showed that the OX acid has a highly acidic function with its conjugate bases, hydrogen oxalate (HC_2O^{-4}) and oxalate ($C_2O_2{}^{-4}$) that makes it a superior reducing material and an efficient chelating reagent for cations [34]. Furthermore, it has been reported that a high-concentration solution of oxalic acid can attack aluminium more vigorously than a dilute solution of sulfuric acid, which enables efficient removal of aluminium [35-36].

The effect of the conventional dealumination and ultrasonic desilication treatments on the crystallinity and Si/Al ratio under the applied conditions is shown in Table 5.

These properties of OX acid enabled removing a high amount of structural Al species, as shown in Table 4, represented by the high Si/Al ratio in the extent of 7 – 9 throughout the dealumination times. Due to the removal of high aluminum content, the crystallinity of the dealuminated samples is dropped to the range of 30 – 40%, reflecting an amorphousity of around 60%. Producing such amorphousity in the mid-step of dealumination is required to ensure the recrystallization of modified mesoporous zeolites.

The results in Table 5 show that the treated samples have lower Al concentration compared with the parent Y zeolite sample. This is presented by the increase in the Si/Al ratio. The Si/Al ratio of the samples with 5 min sonication treatment varies between 6.4 and 7, and this was associated with relative crystallinity values of 51 – 56% (i.e., the sample of USDY5-OX#). In the case of using 15 min desilication with sonication treatment after the dealumination step, the Si/Al ratio alters between 6 and 6.2 with relative crystallinities of 59 – 63%. The highest mesoporosity corresponds with a Si/Al ratio of 6 and a relative crystallinity of 63%, which is the best crystallinity that has been obtained over the tested modified Y zeolite samples. The elimination of aluminium species is more likely to enhance the amorphousity of the treated Y-zeolite to more than 60% in the mid-step of dealumination, which is necessary to enable the recrystallization of zeolite in mesoporous frameworks, Sequentially, the alkaline desilication process reduces the Si/Al ratio to the range of 6 – 7 because of eliminating the Si concentration by NaOH aids [37, 38].

The minimum Si/Al ratio of 6 resulted through the 3h of conventional dealumination stage and 15 min ultrasonic desilication step. This technique has enhanced the crystallinity to reach its highest value of 63% (UDSY15-OX3). Such findings of obtaining the best crystallinity of the modified Y zeolite via post-synthesis hydrothermal approach corresponds with producing the minimum Si/Al ratio after treatment.

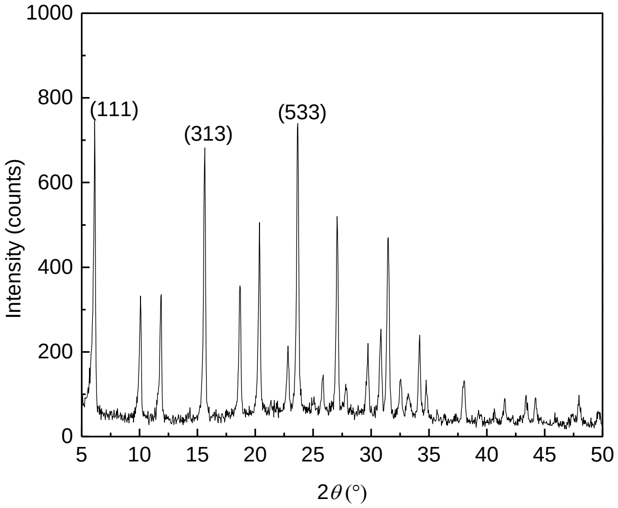

Fig. 8. XRD data of the original Y-zeolite sample

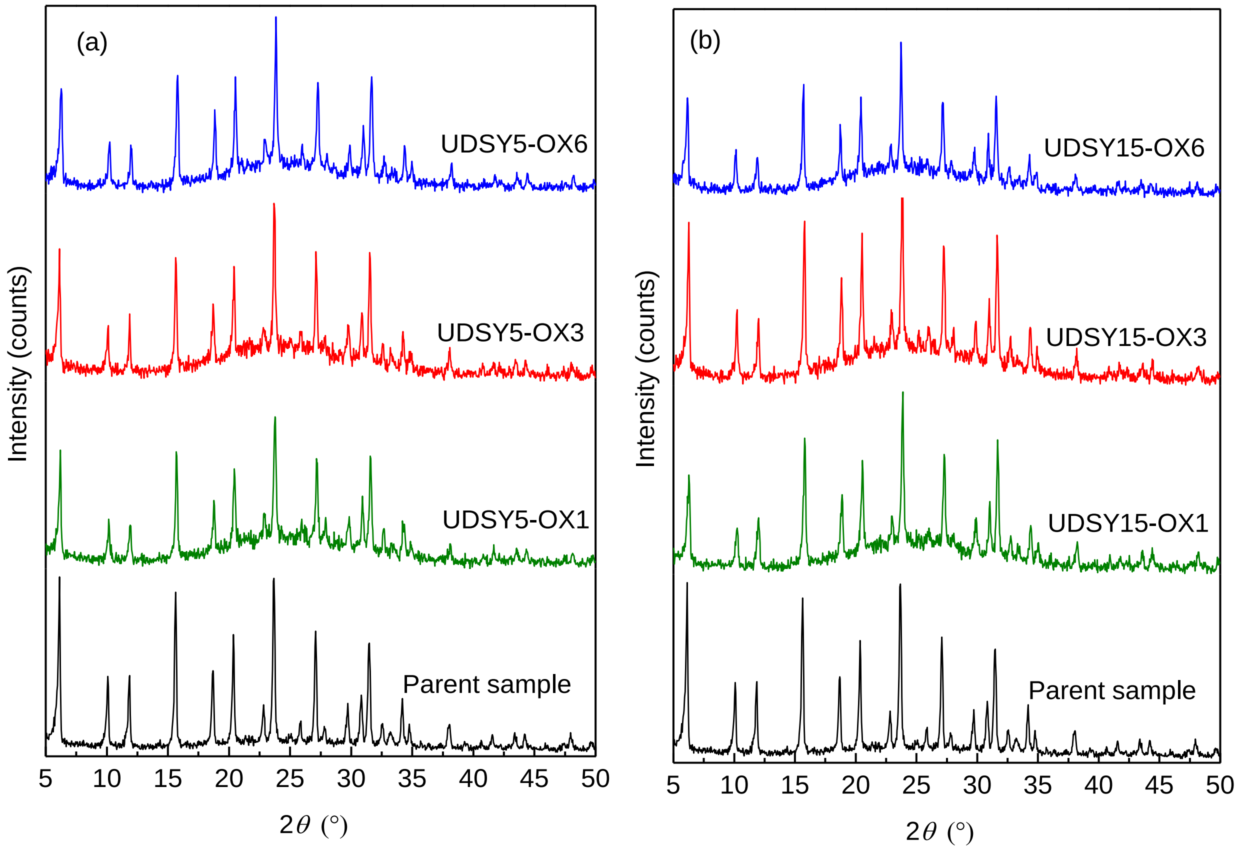

Fig. 9. XRD results of the original Y-zeolite and the modified samples after dealumination with OX acid at 1, 3, and 6 h and ultrasonic desilication with NaOH solution for (a) 5 min and (b) 15 min treatment time

Table 5. Si/Al ratio and relative crystallinity of UDSY5-OX# and UDSY15-OX# samples

Samples	**Si/Al molar ratio**	**Relative crystallinity RC (%)**
Parent Y-zeolite	2.5	100
DAY-OX1	7.2	30
DAY-OX3	9.0	43
DAY-OX6	9.0	36
UDSY5-OX1	6.4	52
UDSY5-OX3	7.0	51
UDSY5-OX6	6.5	56
UDSY15-OX1	6.1	59
UDSY15-OX3	6.0	63
UDSY15-OX6	6.2	60

4- Morphological analysis

The morphological measurements of the parent and treated samples (UDSY5-EDTA3 and USDY15-OX3) are shown in Fig. 10. Fig. 10 b illustrates that the grains of the UDSY5-EDTA3 sample Fig. 10 b have undergone alterations in shape and surface characteristics after hydrothermal treatment via EDTA dealumination, generating unstable hydroxyl sites that lead to framework breakdown during desilication. This is shown by the fractured morphology seen in SEM and the amorphous hump identified in XRD. Conversely, the selective extraction of aluminum by oxalic acid preserves significant crystallinity, enabling ultrasound-assisted desilication to create hierarchical mesopores without amorphization. This significantly enhances the stability of VGO cracking.

This may result from decreased crystallinity and amorphization effects. In the instance of the UDSY15-OX3 sample, the grain boundaries and particle morphology exhibited less damage Fig. 10 c compared to the UDSY5-EDTA3 grains. The alteration of crystallinity is also pertinent, since the relative crystallinity of the UDSY15-OX3 sample (subjected to hydrothermal dealumination and ultrasonic-assisted desilication) rose to around 63%. The alteration of the crystal structure of the UDSY15-OX3 species has rendered their morphological characteristics more akin to the original sample, as shown in Fig. 10 a.

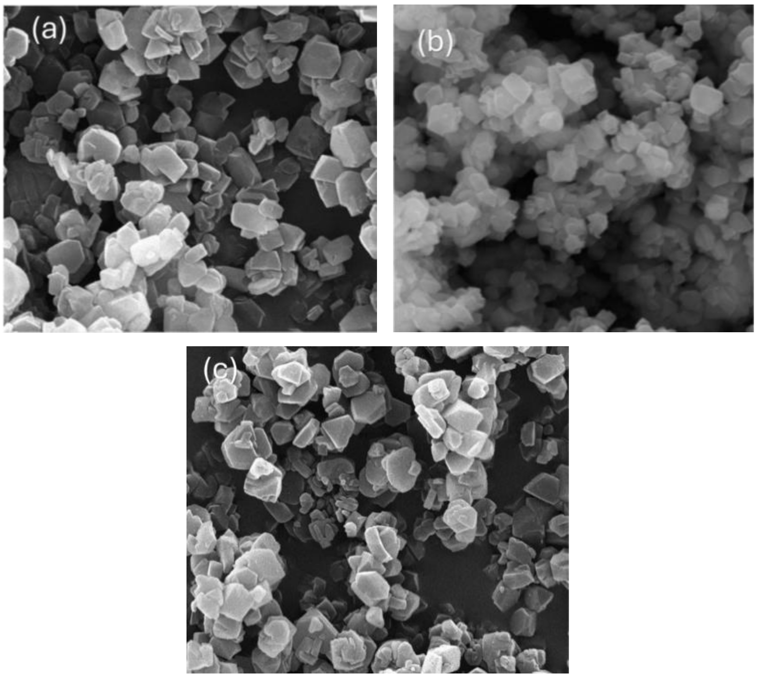

Fig. 10. Morphological Measurements of the Parent Sample (a), DSY-OX3 Sample (b), and UDSY15-OX3 Sample (c)

5- Thermal stability of modified Y zeolite

The stability of the best properties of the Y zeolite sample (UDSY15-OX3) was examined via a high-temperature heating course at 550 °C for 3 h and compared with the parent zeolite sample. The test was achieved by utilizing XRD measurements. The XRD spectrums of the modified samples of UDSY15-OX3 and the original zeolite sample before and after heating are shown in Fig. 11. No more symbols were employed to diagnose the calcined substances. Only the extension with the applied calcination temperature of 550 °C was used to refer to the calcined samples.

From Fig. 11, the original samples show a similarity in the XRD data even after the thermal treatment at 550 °C. The peak is the same with no shifting in their positions. This indicates that the original Y-zeolite is extremely stable and does not influence by the thermal treatment. In the case of the modified samples of UDSY15-OX3 and its calcined form, the data reflect good stability after heating at high temperatures. The background of the XRD spectrum appears smoother, and peaks are slightly sharper, implying a slight change in the crystallinity.

The full width at half maximum (FWHM) was calculated for further analysis for more understanding the structural changes of the samples before and after the calcination step. The characteristic peak (the highest intensity peak at 2θ = 23.96º) was selected for these calculations. The relative crystallinity and the mesoporesity were also determined for better explanation purposes. The resulting data is presented in Table 6.

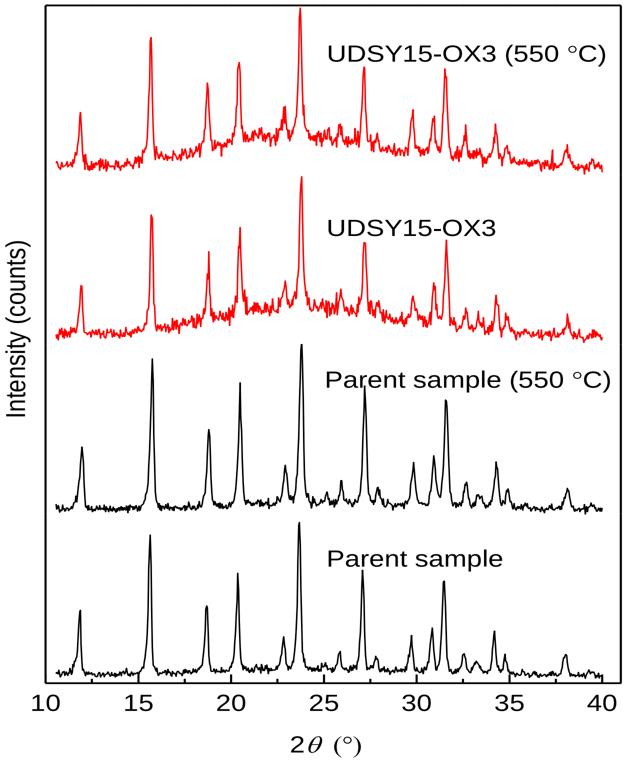

Fig. 11. Thermal stability of the original and modified (UDSY15-OX3) zeolites before and after calcination at 550 °C for 3 h

Table 6. Comparison of crystallinity, mesoporesity, and FWMH of the DSY-EDTA3, DSY-OX3, and the parent sample with and without calcination

Samples	Mesoporsity cm^3/g	Relative crystallinity RC (%)	FWMH
Parent Y-zeolite	0.080	100	NE
Parent Y-zeolite (550 ºC)	0.080	100	NE
UDSY15-OX3	0.198	63	0.2250
UDSY15-OX3 (550 ºC)	0.205	65	0.2014

NE: Not estimated

The estimated FWHM of the highest peak for the UDSY15-OX3 sample at 23.65º is 0.2250. After calcination at 550 °C for 2 h, this value has dropped to 0.2014. This increase could be due to a modification in the crystallite size. On the other hand, the mesoporosity and crystallinity of these two samples have slightly changed. The mesopore size of the UDSY15-OX3 sample is around 0.198, while after calcination it becomes 0.205 cm^3/g. Similarly, the relative crystallinity of the UDSY15-OX3 sample is 63% and it increased to around 65%.

Using the applied heating course of around 550 °C, the crystal and amorphous portions of the UDSY15-OX3 samples show good stability, suggesting them as efficient catalysts for stimulating cracking reactions that take place in the FCC unit.

6- Catalytic cracking experiments procedure (parent and UDSY15-OX3)

The tests for catalytic cracking of vacuum gas oil were conducted at the chemical engineering department postgraduate Research lap in a built catalytic cracking Unit using a (FBR) (I.D. = 12 mm) (length 620mm) at various temperatures (400 and ٤20°C) with a constant flow rate 0.2236ml/min and 1g of zeolite diluted with glass beads Table 7 represents the data on conversion.

Table 7. Conversion of the vacuum gasoil to light cuts by using the modified zeolites (parent and UDSY15-OX3)

Temperature (°C)	Catalyst Type	Total conversion (%)	Gasoline Yield (vol.%)	Kerosene Yield (vol.%)	Gas Oil Yield (vol.%)
420°C	Parent zeolite	45.353	18.4	18.4	12.5
	Modified zeolite (UDSY15-OX3)	91.99	44.44	44.45	11.11
400°C	Parent zeolite	46.824	9.9	27.3	13.7
	Modified zeolite (UDSY15-OX3)	91.995	37.5	43.75	18.75

The results in Table 7 clearly show that the treated zeolite with OX acid and NaOH via dealumination and desilication processes in ultrasonic scenario one enhanced the conversion of the VGO to the targeted light ends. For example, at 420 °C, the use of the (UDSY15-OX3) sample results in producing 44.44 vol. % of gasoline with an increase from the parent sample of more than 144%. This reveals the effectiveness of the ultrasonic route for increasing the ability of zeolites to catalyze the cracking reactions of VGO. The selectivity distribution of the parent and UDSY15-OX3 samples to produce the highest fuel value of gasoline at the two experimental temperatures is displayed in the Figure. 12. The Data in Fig. 12 shows that the UDSY15-OX3 sample has the highest selectivity to produce gasoline from VGO, especially at elevated temperatures (i.e., above 400 °C).

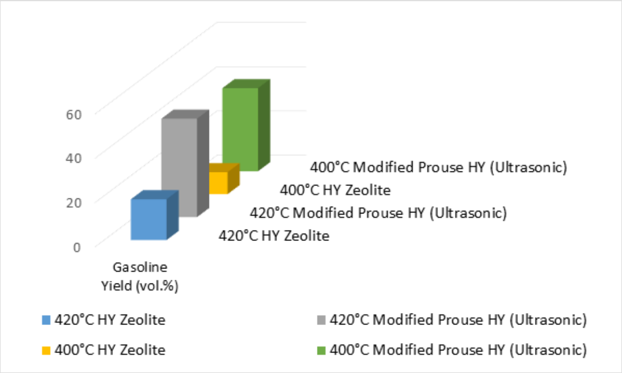

Fig. 12. Selectivity distribution of parent and UDSY15-OX3 samples to produce gasoline from VGO via catalytic cracking at 400 and 420 °C

7- Conclusion

The current work, conventional dealumination by acid and ultrasonic-assisted desilication by alkaline treatments, was achieved to improve the mesoporesity of CBV-400 Y-zeolite with a Si/Al ratio =2.5 for catalyzing the reactions of the FCC unit. A concentration of 0.11 M of EDTA and OX acids was conventionally evaluated to eliminate the framework Al of the parent Y zeolite for 1, 3, and 6 h. Next, the desilication step was accomplished by using a 0.2 M NaOH solution with the aid of ultrasonic energy. The scenario of EDTA-NaOH showed low efficiency in producing mesoporous zeolite, where the highest mesopore volume was 0.104 cm^3/g after 3 h dealumination and 5 min ultrasonic desilication. However, the scenario of OX-NaOH enhanced the mesopore size to around 0.2 cm^3/g after 3 h of dealumination and 15 min of desilication processes. This mesoporosity corresponded with a 47% crystallinity and a Si/Al ratio of 6. The obtained sample of UDSY15-OX3 exhibited high stability upon heating at 550 °C (the maximum temperature range of the FCC unit). The findings of the present study underline that treating Y zeolite of high Si/Al ratio by the sequential post-synthesis of conventional dealumination and ultrasonic desilication treatments is practical for mesoporosity modification. In catalytic cracking operations, the hierarchical mesoporosity of UDSY15-OX3 functioned more effectively as a catalyst than HY-zeolite. Under uniform conditions, VGO conversion via UDSY15-OX3 surpassed that of HY-zeolite. The catalyst exhibiting greater mesoporosity demonstrated superior activity compared to the microporous catalyst (HY-zeolite). This resulted in an improved distribution of large molecules in the feedstock (VGO), facilitating access to active sites and augmenting the number of acid sites that accelerated the cracking process. The catalytic findings indicate that an optimal liquid product yield was achieved by improved product distribution, yielding about 44.44 vol.% gasoline and 44.45 vol.% kerosene. UDSY15-OX3 exhibited more intracrystalline mesoporosity compared to the conventional catalyst HY-zeolite. This applied to both gasoline and kerosene. This indicates that mesoporosity is crucial in the cracking processes catalysed by Y zeolite (UDSY15-OX3). Enhanced mobility to and from the catalyst structure resulted in an increased yield of mid-distillates.

Nomenclatures

Abbrev.	**Description**
EDTA	Ethylene-diamine-tetra-acetic acid
OX	Oxalic acid
V_{meso}	Mesopore volume
V_{total}	Total pore volume
V_{micro}	Micropore volume
$S_{external}$	External surface area
FCC	Fluid catalytic cracking
BET	Brunauer-Emmett-Teller
CBV-400	Applied zeolite 2.5 Si/Al ratio
DAY-EDTA#	Dealuminated Y-zeolite by EDTA acid at a specific time (#)
DAY-OX#	Dealuminated Y-zeolite by OX acid at any specific time (#)
DSY-EDTA#	Desilicated Y-zeolite after dealumination with EDTA acid at a specific time (#)
DSY-OX#	Desilicated Y-zeolite after dealumination with OX acid at a specific time (#)
UDSYt-OX#	Ultrasonic desilicated Y-zeolite at time (t) after dealumination with OX acid at a specific time (#)
Si/Al	Silicon to aluminium molar ratio
RC	Relative crystallinity

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تعديل المسامية المتوسطة لـ Y-Zeolite باستخدام تقنية الموجات فوق الصوتية كمحفزات نشطة لعملية التكسير التحفيزي لزيت الغاز المفرغ

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الخلاصة

في هذه الدراسة، تمت معالجة الزيوليت البكر Y (CBV 400) من Si/Al=2.5 لإنتاج الزيوليت متوسط المسامية المعدلة. تضمنت العملية مساعدة من تسلسل ما بعد التصنيع، وإزالة الألومنيوم في وضعه التقليدي وتقنيات إزالة السيليكون بمساعدة الموجات فوق الصوتية. تم اختبار كل من حمض الإيثيلين ديامين تترا أسيتيك (EDTA) وحمض الأكساليك (OX) كواشف مخلبية في خطوة إزالة الألومنيوم لمدة ١ و٣ و ٦ ساعات. تمت معالجة العينات المنزوعة من الزيوليت بشكل متطور باستخدام هيدروكسيد الصوديوم بمساعدة الطاقة فوق الصوتية في حمام مائي بتردد ٢٠ هرتز و ٦٥ درجة مئوية في أوقات مختلفة ٥ و ١٥ دقيقة. أدى إزالة الزيوليت الأصلي بواسطة حمض OX لمدة ٣ ساعات وإزالة السيليكونات من العينة المعالجة بالحمض بمحلول هيدروكسيد الصوديوم لمدة ١٥ دقيقة إلى تحسين المسامية المتوسطة بحوالي ١٢٠% أعلى من العينة الأم لـ Vmeso= $0.08\ m^3/g$. يبلغ حجم العينة المعدلة mesopore (Vmeso) $0.2\ m^3/g$ ومساحة سطح خارجية (Sexternal) تبلغ $89\ m^2/g$. تم فحص الزيوليت متوسط المسامية الناتج لقياسات الثبات الحراري عن طريق المعالجة الحرارية عند ٥٥٠ درجة مئوية لمدة ٣ ساعات مما يكشف عن ثبات عال. تم اختبار الزيوليت الأصلي والمعدل كمحفزات غير متجانسة لتفاعل التكسير التحفيزي مع VGO وحمل محفز 1g بدرجات حرارة مختلفة من ٤٠٠ إلى ٤٢٠ درجة مئوية ووقت التفاعل ٤٨٠ دقيقة. تم تنفيذ التفاعل باستخدام نظام تفاعل دفعي، وتم الحصول على عائد أعلى من البنزين حوالي٤٤,٤٤% باستخدام الزيوليت المعدل (UDSY15–OX3) عند درجة حرارة تفاعل ٤٢٠ درجة مئوية، على عكس الاستخدام المباشر للمحفز غير المعدل الذي يعطي فقط ١٨,٤٪ من إنتاجية البنزين. وبالتالي، فإن تسويق مسار OX–NaOH بمساعدة الطاقة فوق الصوتية يمكن أن يقلل من مدة المعالجة واستهلاك الطاقة، مما ينتج عنه مسامية مستقرة حراريا لتحفيز تفاعلات تكسير الزيوت الثقيلة.

الكلمات الدالة: حجم الميزوبور، الزيوليت Y، إزالة الألومنيوم، إزالة السيلانات بمساعدة الموجات فوق الصوتية، الزيوليت المعدل، زيت الغاز الفراغي، التكسير التحفيزي.