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Research Article

Green synthesis of a zeolite from natural extract of Sapindus as a template

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ABSTRACT

In recent decades, there has been considerable interest in creating hierarchical zeolites because of their enhanced catalytic qualities and ability to facilitate mass transfer. This study investigates the synthesis of ZSM-5 zeolites using a new natural surfactant extracted from the plant Sapindus mukorossi (Sapindaceae family), which contains Saponins known for their surface-active properties and are responsible for the surfactant's efficiency and considered a mesoporogen to create inter- or intracrystalline mesopores in the zeolite.
The last one is synthesized via the hydrothermal method by modifying the amounts of natural surfactant and contrasted with a conventional ZSM-5 as a reference. The synthesized materials are characterized using various analytical techniques, including X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), nitrogen adsorption isotherms, and thermogravimetric analysis (TGA). X-ray diffraction analysis confirmed the high crystallinity of all synthesized samples.
Moreover, Brunauer-Emmett-Teller surface area measurements revealed a significant increase in surface area to 468m^2/g compared to the conventional ZSM-5. Barrett-Joyner-Halenda pore size distribution showed a size above 5nm, indicating the existence of hierarchical mesoporosity. These findings were validated by enhanced adsorption properties, displaying a 90% removal rate of phenol. Green synthesis has seen the introduction of a new bio template, providing a simple use without the need for previous preparation and availability, being cost-effective, and most importantly, not harmful to the environment.

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INTRODUCTION

Zeolites are crystallized microporous materials made up of channels and cavities of regular dimensions comparable to the size of many organic molecules [1]. Their structure is based on a three-dimensional and regular sequence of TO_4 tetrahedral; the element T is generally Al^{+3} or Si^{+4} [2, 3]. The success of these materials in adsorption and especially in numerous essential catalytic processes [4] has given rise to countless patent publications and numerous books [2, 5]. The remarkable acidic properties of ZSM-5 zeolites and the selectivity of their porous structure have made them the most used catalysts in applications chemical and petrochemical [6, 7]. Another essential property of these zeolites is their great adaptability.

The size of their pores and their textures can be slightly modified, allowing the desired sieving of reactant or product molecules. The strength and density of the center's Acids of zeolites can be adjusted. Basic and redox sites can be created, which allows for numerous applications in organic synthesis and depollution [4, 8]. Classic zeolites are uniquely microporous materials with large crystals, generally on the micrometer scale [9, 10].

The wide range of micropores in the zeolite crystal can allow selectivity of well-known sizes and shapes during adsorption, separation, or catalytic reactions [11]. However, such a bulky crystal system may suffer from diffusional limitations [12]. It results in slow mass transport to and from the catalytic sites inside the micropores [13], which can increase the possibility of side or unwanted reactions. In addition, large molecules whose size is larger than the opening of the micropores cannot penetrate the zeolite network [14, 9]. Due to these steric and diffusional limitations, it is highly desirable to increase the zeolite's surface area, which is effectively accessible to molecules of different sizes [15, 12]. The creation of hierarchical zeolites is a skillful way to get around this restriction [16]. Recently, there has been a rapid development of new synthetic strategies for adapting the hierarchy of the zeolites' pore structure to optimize their characteristics [17].

It is important to note that different methods exist to introduce these so-called secondary or tertiary porosities. Generally speaking, they are classified into two families: the constructive introduction of mesoporosity is called bottom-up strategy, while the destructive ones are called top-down methods [15, 16]. The one is to use a secondary template in favor of making a mesopore agent for the creation of the mesoporosity intercrystalline [16]. A secondary, tertiary template is called a structure-directing agent (SDA), which is an organic compound consisting, of carbon, nitrogen, sulfur, or phosphorus elements [18]. SDA affects various characteristics of the zeolite. The main parameters are the size of particles and morphology [11] Efforts were made to promote green synthesis using cost-effective materials [16, 18].

Using the template is one of the methods for the synthesis of hierarchical zeolite. They play a crucial role in determining the structure and properties of the final material [19]. These templates can be broadly categorized into two types: hard templates and soft templates [18]. Robin J. White and all have reported that on the use of monolithic nitrogen-doped carbonaceous as a hard template, the creation of wide intra-crystalline mesopores in the zeolite crystals was made possible by impregnation with ZSM-5 precursor solutions, hydrothermal treatment, and template removal [20].

Soft templating is a recommended method over hard templating since it is more compatible with Zeolite precursors and provides an opportunity to adjust the pore size by changing the quantity of template [9]. In our study, we used Sapindus mukorossi, an eco-friendly natural surfactant, as a second template. A soft template that contains saponin as surface-active agent [21]. Sapindus is among the introduced plants in Algeria; it is a tree found in tropical and subtropical regions of Asia. That consists of 158 gen and 2230 species [22]. All Sapindus are soap trees; it is a small

Figure 1. Photograph of pericarp Sapindus mukorossi fruits (A), core and leaf (B).

tree with a short trunk, protruding rarely a dozen meters in height. Fruit extracts are used in several areas: A soap alternative surfactant for washing clothes, hair and skin sensitive (eczema), they are also used by Indian jewelers to restore shine to tarnished ornaments. To remove scalp lice, freckles, and heat: Treatment of burns, the fruit of S. mukorossi is used in the treatment of snake bites, stings of scorpion and dandruff [23].

The main constituents of Sapindusmukorossi fruits are saponins (10%-11.5%), sugars (10%), and mucilage. Sapindus saponins are a mixture of sapindosides [24, 25] .Saponins are a large family of structurally related substances composed of steroid or triterpenoid aglycones (sapogenin) linked to one or more oligosaccharide moieties by a glycosidic bond [26] A new anionic template is utilized for the first time in this extract of the *sapinduce mukorossi* as a second template for the synthesis of ZSM-5. These materials offer enhanced surface area, increased accessibility to active sites, and improved diffusion properties. Here are some key areas where hierarchical materials are making an impact in catalysis[27,28]. Different literature utilizes other substances as the secondary template we summarized in Table 1.

The development of hierarchically porous zeolite, including the synthesis methodologies, has been discussed in a number of papers [16]. Song et al. [17] created a hierarchical ZSM-5 zeolite by a one-step in-situ hydrothermal carbonization method with steam-assisted crystallization using sucrose as a mesoporous template precursor. Nevertheless, adding more water resulted in excellent crystallization but decreased mesoporosity.

Shi et al. [12] created hierarchical zeolites by altering the surface of chitosan and TPAOH as the meso- and microscale templates, respectively. Xue Ma and al. prepared a ZSM5 with cellulose aerogel as a green template and a coal gangue as a raw material at different times of crystallisation and temperature [34].

The present study focuses on the use of a novel green template extracted from a plant called Sapindus mukorossi known by washing nuts or reetha used as a mesoporogen template and tetrapropylamonium bromide (TPABr) to direct the crystallization of the zeolite ZSM5 type MFI. The surfactant used in the intended to generate mesopore intercrystallin within the zeolite to permit a transfer of balky molecule and increase its specific surface area, which will make it possible to improve these catalytic properties.

The template (saponin) employed in this research has a larger hydrodynamic size of micelle compared with different surfactants, triton X-100, cetyltrimethylammonium bromide (CTAB), and sodium dodecyl sulfate (SDS), as reported in the literature [35].

In fact, an aqua extract of this plant is prepared; methanol or ethanol was planned as a test to extract the saponins, however other literature suggests that water is the most efficient method. The extract obtained is used as a second template for the hydrothermal crystallization of the zeolite, keeping everything constant (the temperature of crystallization 160°C, time and the Si/Al ratio) and altering the amount of surfactant. The results show a good crystalinity for the modified zeolite and a larger surface area, reaching 468m2/g. A test is made to demonstrate the effectiveness of the modified zeolite is an adsorption of the phenol; it exhibited an attractive rate of reduction 90%.Optimizing the synthesis settings and even the extraction procedure can yield improved results, making the method straightforward and promising.

EXPERIMENT AND METHODS

Tetrapropylammonium bromide [$(CH_3CH_2CH_2)_4NBr$], tetraethyl orthosilicate ($C_8H_{20}O_4Si$; TEOS), aluminum isopropoxide ($C_9H_{21}AlO_3$; AIP), and sodium hydroxide (NaOH) were procured from Sigma-Aldrich and Merck and were used as received without further purification.

Table 1. Presentation of the studies done with a green surfactant

Surfactant added	Zeolite Type	Reference
chitin	ZSM-5	[29,27]
Gemini	ZSM-5	[30]
(sucrose, cellulose, and starch)	ZSM-5	[8]
Triton X-100	TS-1	[13]
Chitosan	ZSM-5	[12]
agricultural waste as solid template	ZSM-5	[31]
Triton X-100	ZSM-5	[2]
glucose	ZSM-5	[5]
HDSS	ZSM-5	[9]
CTAB	ZSM-5	[32]
choline	Y	[33]

The Extraction of Saponin from S. Mukorossi Fruit Pericarp Powder

The seedless pericarp (outer covering) of Reetha (Sapindus mukorossi) fruits was dried in an oven at 60°C for four days. Using a mixing grinder, the dried pericarp was reduced to a fine powder. Then, 50 g of dried powder was mixed with 500 ml of deionized water and stirred for 3 h using a magnetic stirrer. After settling of larger particles (10-15 min), the supernatant turned into filtered through Whatman N°1 filter paper and then centrifuged at 6,000 rpm for 45 min to remove any remaining suspended particles [36] .

Determining the critical micelle concentration (CMC) of a surfactant is crucial for understanding the surfactant's efficiency in the synthesis of the zeolite. Conductimetry measures are used to find CMC equivalent to 0.45 wt%, and the result is confirmed by different literature [37]. The determination of the CMC permits the selection of the surfactant concentration; a concentration below the CMC prevents micelle aggregation and, thus, mesopore development during the crystallization.

Synthesis of ZSM-5

The synthesis of ZSM-5 zeolite conventionally was synthesized by mixing trimethylpentylammonium bromide (TPABr), tetraethyl orthosilicate (TEOS), and aluminum isopropoxide (Al (O-CH (CH_3)$_2$) to serve as the structure-directing agent (SDA) and the sources of silicon and aluminum, respectively. The molar ratio of the starting gel was 0.8 SiO_2:0.01 Al_2O_3:0.16 TPABr: 0.08 Na_2O:30H_2O, respectively. The synthetic procedure for the gel preparation is outlined as follows:

Solution A was prepared by mixing TEOS, H_2O, and TPABr and allowing them to mix for 1 hour. Solution B was prepared by combining aluminum isopropoxide, NaOH, and distilled water. Solution A was added dropwise to Solution B over 1 h under vigorous stirring.

Crystallization occurred in Teflon-coated stainless-steel autoclaves at 160°C for 3 days without stirring under autogenous pressure. Subsequently, the solid product was collected by filtration, washed several times with deionized water until a neutral pH was achieved, and then dried overnight at 110°C. Finally, the catalyst samples were calcined at 550 °C for 6 h to completely eliminate the organic template [11].

Synthesis with A Green Template ZSM-5

The modified structured ZSM-5 zeolite was synthesized by adding solution A (TEOS, H_2O, and TPABr) dropwise solution B (aluminum isopropoxide, NaOH, and distilled water) over 1 h under vigorous stirring. As a final point, a different volume of the surfactant solution was added and stirred at room temperature. The resulting solution was transferred to an autoclave and treated at 160°C for three days. The obtained product was washed with deionized water, centrifuged to separate the solid product, and washed again with deionized water until it was neutral. The solid product was then dried overnight at 110 °C and calcined in air at 550 °C for 6 h to remove organic components.

Different zeolites are synthesized with different amounts of the solution as follows: ZSM5 (a): ZSM5 conventional and ZSM5 (b, c, d, and e) are to add a volume of 1, 2, 5, and 10 ml, respectively. The volume of the surfactant is selected and compared with the total volume of the initial gel in such a way that the molar ratio SiO_2/concentration of the surfactant remains unchanged.

Characterization

X-ray diffraction (XRD) patterns were collected using a Rigaku smart lab diffractometer, employing CuKα radiation (l = 1.540593 Å) with a step size of 0.033°. The XRD system was operated at a voltage of 40 kV and a current of 40 mA. Fourier transform infrared (FTIR) spectra were proved at room temperature using a Bruker FT-IR spectrometer in the range of 400–4000 cm^{-1}.

N_2 adsorption-desorption experiments are performed at -196 °C using a Micromeritics Quantachrome instrument with the "ASIQwin" automated gas sorption data software. Before each experiment, the sample is outgassed at 300°C for 6 h under a vacuum of about 10−5 mbar. The BET surface area (S_{BET}) is determined via the BET equation. The microporous volume, the mesoporous volume, and the external surface area are calculated using the t-plot method. The total volume of pores (VT) is determined at P/P_0 0.99.

Microscopy Scanning electron (SEM) images were acquired using an Fei Quanta 650 instrument and a Zeiss Variable Pressure SEM. The water loss is obtained via thermogravimetric analysis using a TGA, TA Instruments Trios V4.2.1.36612.

RESULTS AND DISCUSSION

Characterization by X-Ray Powder

The XRD patterns of the surfactant-modified and conventional ZSM-5 samples are shown in Figure 2. All The synthesized samples showed peaks at 2θ = 7.94°, 8.88°, 9.14°, 13.25°, 14.83°, and 23.19°, associated with the (011), (020), (111), (002), (332), and (520) planes, which are characteristic of the crystalline nature of the MFI structure of the ZSM-5 zeolite. The zeolites showed a crystal structure with no amorphous phase.

The most important difference between the conventional and modified XRD pattern observed for all sets of samples is the relative intensity of the peaks 8° and 23° that

correspond to the (020) and (332) Miller plane. We may restate that the addition of this surfactant always causes a crystalline form. Each zeolite's relative crystallinity was determined using the total peak areas at 22.5-25.0° [8]. ***XRD= crystallinity = Σ peak area intensities of sample/Σ peak area intensities of reference***

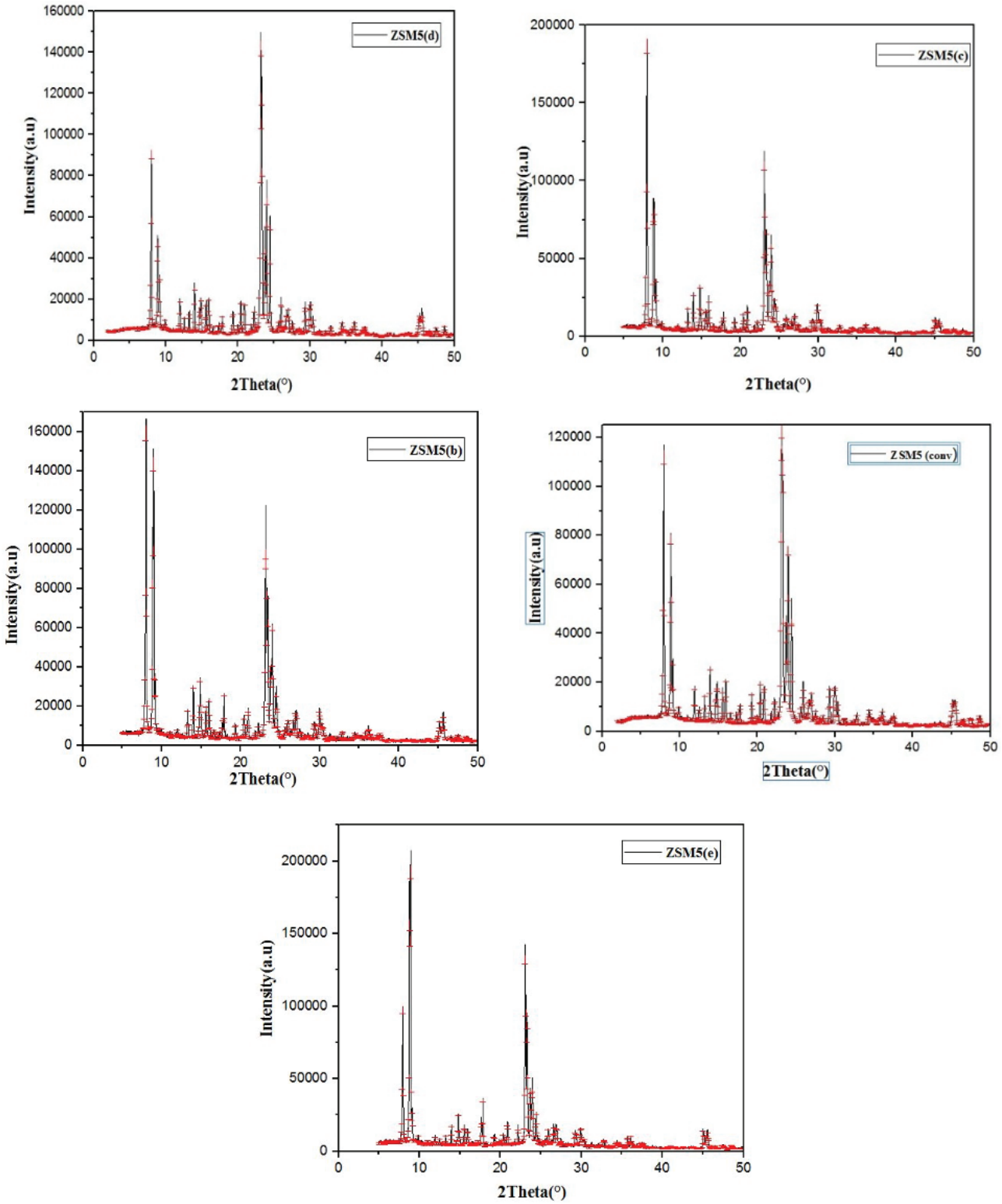

Figure 2. XRD patterns of the (a): ZSM-5, (b): ZSM-5(c): ZSM-5, (d): ZSM-5 and (e): ZSM-5.

We may infer that sample (d) shows improved crystallinity at 95%, whereas samples (b) and (c) show 85%, indicating that the addition of the surfactant creates mesopore at predetermined intervals. Although, as shown in Fig. 2. For the zeolite (ZSM5e), increasing the amount of a green template, the crystallinity decreased. Comparing with numerous studies, including one by Xue Ma, they all discovered a decrease in crystallinity when adding more of a green template (cellulose aerogel), and they suggested that this could be caused by the molecules in the channel clumping together due to the excessive addition [34]. A similar results found by Qingfeng Che when sucrose, cellulose, and starch were be used as a green template to synthesize a micro-mesoporous ZSM5 [8], and F. C. Drumm used chitin to prepare a mesostructured ZSM5 [29].

Characterizations by Fourier Transform Infrared Spectroscopy

The results obtained by FTIR spectra of the surfactant-modified ZSM-5 recorded from 400 to 4000 cm^{-1} range are presented in Fig.3. Absorption bands at 433 cm^{-1} correspond of (T–O bending) is attributed to the vibration of internal AlO_4 and SiO_4 tetrahedral units, 545 cm^{-1} is assigned to the 5-membered rings, 793 cm^{-1} has resulted from the external symmetrical stretching vibrations at 1059.11 cm^{-1}. Anti-symmetrical stretching vibrations of T-O correspond to siliceous materials. The band appears at 545 cm^{-1} corresponds to the double five-ring units present in pentasil zeolites and Si-O and Al-O skeletons, which are characteristic of the ZSM-5 with the MFI structure.

It is seen that all the samples present the main characteristic vibrations. The intensities of the picks related to the T-O binding are slightly decreased by increasing the quantity of the natural surfactant in the modified zeolite. This was consistent with earlier research findings [34]. The absence of a peak beyond 2000 cm^{-1}, confirms the complete calcination procedure and sufficient removal of template molecules from the zeolite structure. Moreover, Pan et al. [38] described the vibration band around 1090 cm^{-1} to the Si/Al ratio of zeolite.

This band is associated with the asymmetric stretching mode of Si-O-T (T = Si or Al) bonds in the zeolite structure. This observation is consistent with the reported trend of decreasing Si/Al ratio with increasing surfactant concentration. Surfactants are known to interact with zeolite precursors and can influence the zeolite framework formation. In this case, the surfactant may be interfering with the incorporation of silicon atoms into the zeolite framework, leading to a decrease in the Si/Al ratio.

Textural Characterization Results

The N_2 adsorption-desorption isotherms obtained at -196°C are shown in figure 4. Figure 4 shows the adsorption/desorption isotherms of N_2 at 77 K and the pore size of the conventional and modified zeolites. The modified ZSM-5 exhibits an adsorption isotherm type IV and a

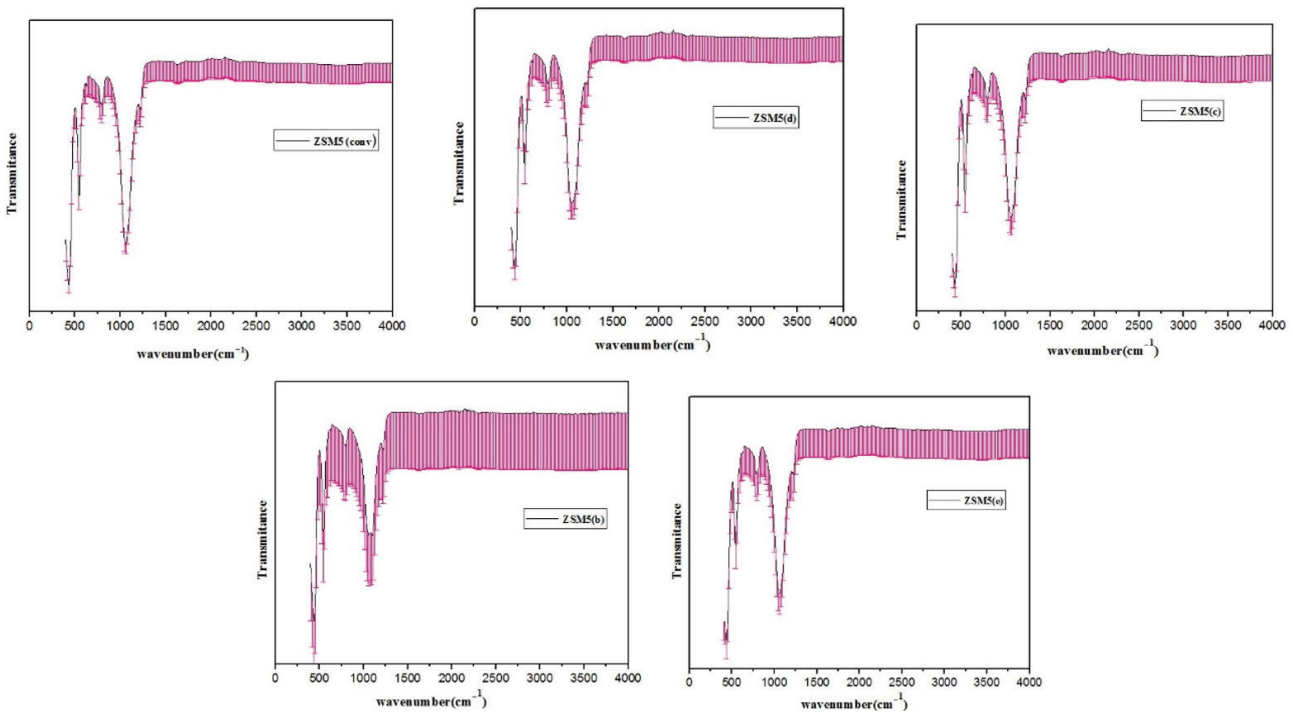

Figure 3. FT-IR spectra of different zeolites.

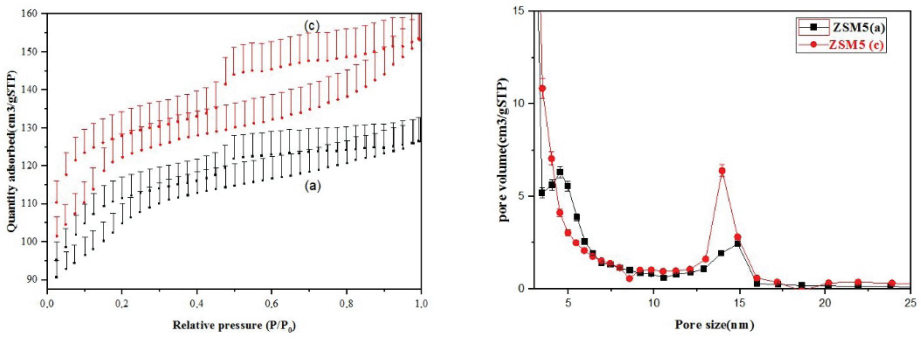

Figure 4. (a) N_2 adsorption-desorption isotherms (b) pore size distributions.

Table 2. The textural properties of different materials

Sample	SBET ($m^2 g^{-1}$)	Sext ($m^2 g^{-1}$)	Smicr (m^2/g)	Vmic ($cm^3 g^{-1}$)	Relative crystallinity %
$ZSM\text{-}5_{(a)}$	439	26.774	338	0.158	100
$ZSM\text{-}5_{(c)}$	468	38.820	399	0.178	80

hysteresis loop type H4, often found for aggregated zeolite crystals, according to IUPAC [36].

It can be mentioned that both modified samples exhibit type H4-type hysteresis loop at P/P_0 =0.45 adsorption peak. [39] The presence of a mesoporous structure (Sapindaceae) as a template can generate secondary intracrystalline or intercrystalline porosity. The use of Sapindus as a template facilitates the reassembly of dissolved material species, generating mesoporosity and maintaining the microporosity of the zeolite [40]. The ZSM-5 sample synthesized without surfactant ZSM-5(a) shows that the surface area is 439m^2/g and the modified ZSM-5(c) is 468m^2/g which is larger than the conventional one.

In that order, by comparison, it is discovered that the surface area increases when surfactant is added to the zeolite ZSM-5(e) with a greater amount of surfactant showing that the BET surface area is 318m^2/g. It means that increasing the amount of surfactant led to a decrease in the BET. The surface area decreased as the volume of surfactant increased, indicating an inverse rapport, and is consistent with previously published work [8]. A study with a smaller volume than we anticipate is interesting.

The shape of the isotherm can also be used to infer the presence of mesopores. For example, isotherms with a type IV H4 hysteresis loop are typically associated with hierarchical porous materials, which contain both micropores and mesopores [41]. All the isotherms exhibit steep N_2 adsorption below P/P_0=0.02, indicating the presence of micropores. Furthermore, the Barrett-Joyner-Halenda (BJH) pore-size distribution was found in the range of 5 nm, confirming the presence of micropores [42]. The peak pore size of the modified zeolite (14 nm) is larger than the conventional. BJH pore size distribution and SEM analysis further confirm the development of mesopore.

The surface area was calculated using the Brunauer–Emmett–Teller (BET) method using the p/p_0 ranging from 0.05 to 0.3

The surface micropore area was calculated from t-plot analyses.

V_{tot} is the single-point adsorption total pore volume at P/P_0 = 0.99.

Comparing with similar studies, such as synthesizing ZSM5 with other surfactant as in the case of the research Dongdong Xu and all [32] with CTAB, who provided a specific surface area BET=385m^2/g, Junjiang Jin and all synthesizing ZSM5 with chitin donated that surface area was 422m^2/g [12] and Shuting Du found a BET=413 m^2/g in the synthesis of TS-1 [13]. Qingfeng Chea and all [8] synthesize ZSM5 with green templates (sucrose, cellulose, and starch) to introduce additional mesopores and found the specific surface area does not exceed (468m^2/g). The strong point of the surfactant used in this study is not only the large specific surface area but also the simplicity of use; there is no need to modify or treat before employing.

Characterization by Scanning Electron Microscopic

The SEM images of zeolites conventional and modified ZSM-5 provided important information regarding the morphology of the zeolites.

The sample images of conventional and modified ZSM-5 (a), (c) respectively showed spherical morphology and aligns

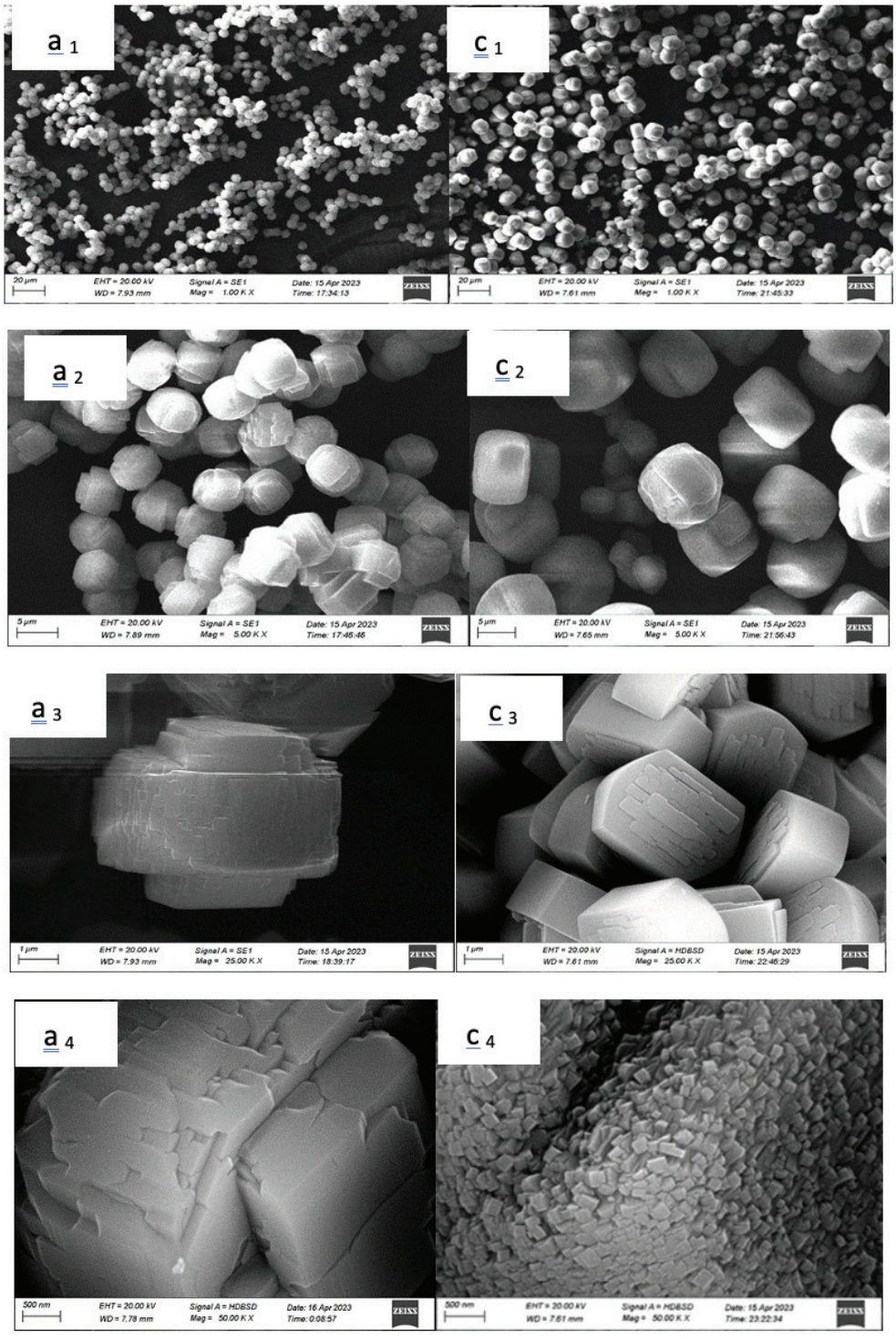

Figure 5. SEM micrographs of zeolitic materials (a) and (c) conventional and modified respectively.

with previously published research [9]. But their particle sizes and shapes were different. We can see that the morphology differs and the size of the pore is not homogen in modified zeolite. When surfactant is added, the morphology of the particles is composed of spherical particles with very rough surfaces. The micro-spherical aggregation of individual nanocrystals in modified zeolite suggests a hierarchical pore structure. This type of morphology provides a mesoporous structure. It is seen in the images that the templates influence the morphology and particle size of ZSM-5 samples.

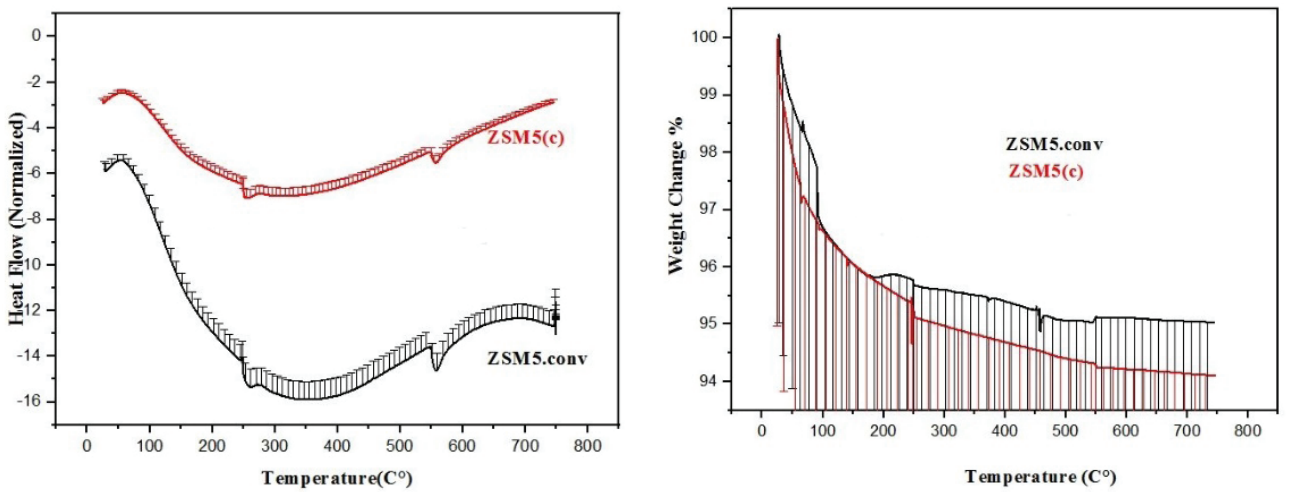

Figure 6. Thermogravimetric analysis (TGA-DSC) of zeolitic material.

Thermogravimetric and Differential Scanning Calorimetry Analysis

The TGA/DSC results for both samples show similar weight loss profiles in the temperature ranges of 50–250 °C and 450–750 °C. This indicates that the organic template has been eliminated from the structure of both samples.

In the 50–250 °C temperature range, the loss is because of the loss of water molecules that are adsorbed onto the surface of the zeolite. The weight reduction in the 450–750 °C temperature range consists of the combustion of the remaining organic template. This first weight loss, directed by an endothermic peak appearing in the DSC curve, can be attributed to desorption of physically adsorbed water associated with samples. The second weight decreases between 450°C and 750°C, corresponding to the decomposition of the organic template. A TGA curve demonstrates that the weight loss of the modified ZSM-5 is higher than the conventional. It is certainly the amount of biosurfactant that was applied. Additionally, it should be mentioned that upon modification with a surfactant template, the ZSM-5 zeolite's thermal stability remains intact. This is confirmed by the phenol adsorption test. The stability of mesopores at high temperatures is crucial for expanding the application of zeolites in high-temperature catalytic reactions.

Adsorption of Phenol

The adsorption technique is typically regarded as the best, most efficient, least expensive, and most popular method for eliminating phenolic pollutants. Zeolites are an important class of hydrated aluminosilicates; the ability of these materials to regenerate while retaining their original characteristics is a crucial feature [43]. We investigated ZSM-5's adsorption performance of phenol. Experiments on batch adsorption were carried out in demineralized water. A 50ml aqueous solution containing 0.05g of zeolites was dosed with varying concentrations of phenol solution, following a 24-hour equilibrium period at room temperature (25±1°C).

The UV-visible UV-1800 Shimadzu Uv Spectrophotometer was employed to measure the absorbance of the solution before and after adsorption. The removal rate of the adsorbent was calculated according to: Removal rate: $R= (C_0-C_t)/C_0$. C_0 represents the initial concentration of phenol, and C_t represents the concentration of phenol solution at time t, unit: mg/L; R represents the removal rate, unit: %.

Compared with the conventional zeolite, phenol was successfully adsorbed on the modified one at the same equilibrium concentration range (0–100 mg L^{-1}).This test lets us know how well the modified zeolite adsorbs

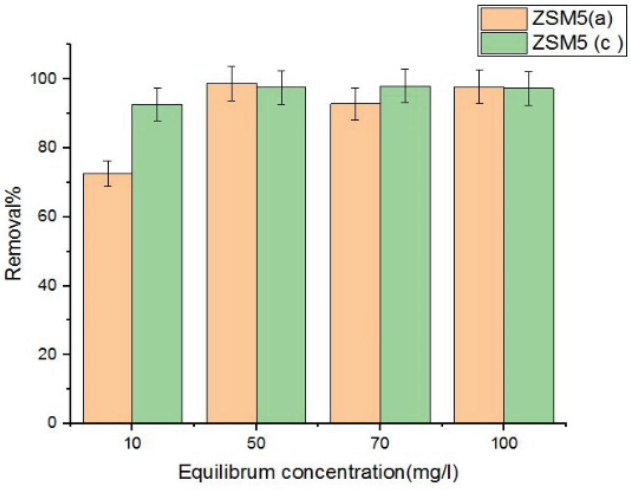

Figure 7. Removal efficiency for the phenol on ZSM5 conventional (a) and modified (c) at different concentration values.

substances. Although further application (catalytic activity) or carbon dioxide adsorption are highly intriguing. One possible explanation for the favorable adsorption on MFI zeolites is the pore size effect, which very roughly matches the molecular size of phenol molecules, which are 4.34 Å * 0.87 Å * 5.55 Å [44]. Too accurately target the right application, additional characterizations are required a priori, such as acidity, point of zero charge, and Si/Al ratio, which are essential.

CONCLUSION

We have successfully synthesized a hierarchical ZSM-5 using a natural extract from a plant called Sapindus mukorossi. Hierarchical zeolite was synthesized hydrothermally with tetrapropylammonium bromide and different amounts of a natural surfactant as a secondary template. The natural surfactant acted as a flexible template, directing the formation of mesopore in the ZSM5 zeolite. The crystallization degree, morphology, and textural properties of the ZSM-5 were affected by the amount of surfactant. The approach succeeded in enhancing the zeolite's textural characteristics in such a way that the specific surface area increased to 468m^2/g compared to the conventional and excellent crystallinity.

The phenol exhibited excellent adsorption on the modified zeolite and improved adsorption capabilities, showing a 90% phenol elimination rate. Adsorption of carbon dioxide is one of the other adsorption tests that may show promise. To improve the study's outcome, we intend to experiment with other synthesis factors, including temperature, crystallization time, and molar ratio silicon/aluminum. The technique is progressive in the field of green synthesis due to the fact that a new template is available offering managing simplicity without any prior treatment and availability and also not expensive and, above all, not dangerous for the environment. To the best of our information, this is the first time this biotemplate has been utilized.

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