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# Synthesis and characterization of nano zeolite-A with aid of sodium dodecyl sulfate (SDS) as particle size-controlling agent



OLLOIDS AN

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### G R A P H I C A L A B S T R A C T



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#### ABSTRACT

Synthesis of Nano zeolite-A has received considerable attention in the past decade and attracted profound attention as very essential commercial materials due to its nanoganic behavior., LTA Zeolites A (Linde Type A) are synthesized via low-temperature hydrothermal crystallization or microwave digestion methods in the presence of the 'organic templates'. Generally, Tetramethylammonium-hydroxide (TMAOH) has been used as a template however, non-recyclablity, numerous efforts have been hampered the synthesis of environmental friendly template. In this work, nano zeolite-A was synthesized via both hydrothermal and microwave methods. An anionic surfactant, Sodium dodecyl sulfate (SDS) was used for controlling the particle size and as a site directing agent (SDA). The effects of different crystallization conditions such as aging time, synthesis time and temperature on the changes in particle size, morphology were investigated. The synthesized nano zeolite A was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Energy-dispersive X-ray (EDX), Fourier-transform infrared spectroscopy (FTIR) techniques. The obtained SEM and XRD results showed that both methods have ability to produce pure LTA zeolites crystallites with a 300-500 nm range in size with a high degree of crystallinity. However, lower aging temperature may cause for formation of sodalite (SOD) phase. Compared to the hydrothermal method, microwave approach is effective because it requires less time and produced narrow particle size distribution. Therefore, surfactant based zeolite-A synthesis would be potentially important in the chemical industry due to its competitive advantages as a green approach and cost-effectiveness.

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# 1. Introduction

Zeolites are nanoporous, crystalline materials that have been used for many decades in applications ranging from catalysis over gas separation to ion exchange. Zeolite materials are found in nature (as minerals), and can be synthesized in the laboratory [1]. The history of artificial zeolites begins with 19<sup>th</sup> century after the Saint Claire's effort in 1962. First synthetic zeolite (zeolite A) was carried out using Na, Si, and Al at Linde's laboratories in Tonawanda, Linde synthesized zeolite X and Y, the structural analogues of the natural zeolite Faujasite [2,3].

Usually, Zeolites have 3-dimensional, open anion framework consisting of oxygen-sharing  $TO_4$  tetrahedra, where T stands usually for Si and/or Al. Their framework structure contains interconnected voids that are filled with adsorbed molecules or cations. The tetrahedra are linked together to form cages connected by pore openings of defined sizes and shapes. Generally, depending on the structural type, the pore sizes range from approximately 0.3–1 nm [3].

Zeolites have received considerable attention in many areas and have today turned into essential commercial materials [2]. Zeolites are most useful commercially because of their unique crystal structure, which allows them to act as molecular sieves selectively sorting molecules based on particle size [4,5].

The synthesis of nano-crystalline zeolites has received much attention in the past decade because those can have different properties than their micro scale counterparts. As usually, the reduction of particle size from the micro level to the nanometer scale can change several chemical and physical properties such as mass transfer resistance, heat transfer resistance in catalytic and sorption processes, thereby improving the catalytic selectivity and reducing coke formation in some petroleum reactions [6]. Moreover zeolite nano crystals can be used to fabricate zeolitic membranes and ordered porous materials [3,7]

The great attention was given to various self-assembled materials such as catalysis, hydrogels, and supercapacitors. Therefore, it has broad application prospects and expands new ideas for the research of gas sensor film materials and other self-assembled nanocomposites in the future [8]. Zeolites are usually synthesized via self-assemble, under hydrothermal conditions from reactive aluminosilicate gels in alkaline media at temperature between ambient to 200 °C and at autogenous pressures. The duration required for crystallization varies from a few hours to several days [9,10]. Here the initial system is usually an aluminosilicate hydrogel that evolves under combination of Mineralizing (OH<sup>-</sup>, F<sup>-</sup>) and structure directing agents (SDA).

However, in the case of nano zeolite synthesis, the synthesis is often carried out under mild conditions. As an example, reactant mixture temperature should keep below 120 °C to control particle size within nano range. However in the case of nano-NaX zeolite synthesis conventional hydrothermal crystallization was performed at 60 °C for 4 days in a shaker with a rotation rate of 250 rpm [11]. This is because, law temperature conditions favors nucleation over crystal growth and leads to nano-crystals and minimize the Ostwald ripening [12,13]. Reaction times, aging time, molar ratio, pH, organic structure-directing agents are critical parameters of formation of nano zeolite. In this research, nano zeolite was synthesized using SDS as organic structure directing agent [14].

There are advantages and drawbacks of the organic-template assisted synthesis of nanozeolites. The advantages are (i) fine control of the particle size, (ii) well defined crystal morphology, (iii) high colloidal stability of the product, and (iv) tuneable surface reactivity. The drawbacks are (i) difficult scale up due to the use of large amounts of expensive and in some cases toxic organic templates, (ii) a low crystalline yield and (iii) the need for post synthesis treatments (purification and calcination). The main drawbacks however are their high cost, a consequence of low crystalline yields, time consuming and complex separation and purification processes. This bottleneck needs to be circumvented to open industrial prospects for nano-zeolites [15].

Synthesis of zeolite in surfactant template is one of the frequently

taken to a falcon tube. Then 3.43 g of Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O was measured separately as silica source. As alumina source 2.42 g, of NaAlO<sub>2</sub> was taken. Then 0.05 M, 25 ml of SDS solution was prepared. Finally, 5.34 g of NaOH was measured into a separate conical flask to minimize the melting and maintain weight accuracy. As a first step, NaOH pellets were dissolved in 50 ml of deionized water using an ice bath. Next, NaAlO<sub>2</sub> was added slowly into NaOH solution with vigorous stirring (250 rpm). Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O was added in to above mixture. Then whole mixture was kept at 50 °C for 48 h for proper ageing. The synthesized solution was transferred to a 50 ml Teflon sealed stainless-steel autoclave and placed in an oven at 60 °C for 4 days. The products were regained with centrifugation (7500 rpm), washed with deionized water until the pH value reached below 8, and dried at 40 °C for 24 h for further characterization (Sample ID: HT 1) [21,22].

#### 2.2. Microwave digestion synthesis

For the microwave-assisted synthesis, MARS 6 Microwave Digestion System was used. For the whole synthesis, previously mentioned seed solution was used. As previously mentioned, the total seed solution with a molar ratio of  $5.5 \text{ Na}_2\text{O}:1.0 \text{ Al}_2\text{O}_3:4.0 \text{ SiO}_2:190 \text{ H}_2\text{O}.0.2 \text{ SDS was}$  prepared and kept 2 days for aging at 50 °C [23].

Then microwave heating was applied with 900 W at 90 °C for 3 h. After completing the digestion the products were regained with centrifugation (7500 rpm), washed with deionized water until the pH value reached below 8, and then for further characterization dried at 40 °C for 24 h (Sample ID: MW-2)

#### 2.3. Characterization techniques

The final products were characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM), and energy-dispersive X-ray (EDX) and Fourier-transform infrared spectroscopy (FTIR) techniques. Crystallinity and purity of the synthesized zeolite phases were

used methods to control morphology and size. Ammonium compounds are mostly used as surfactant template, however most chemicals are environmentally harmful. For that sodium dodecyl sulphate (SDS) used as surfactant template [16]. It less harmful compared with ammonium surfactant. Presence of surfactant in the zeolite synthesis mixture is known to have profound effect on the zeolite crystallization. Influence of non-ionic, cationic and anionic surfactants has been reported. Variation of crystal size, morphology of the zeolites may effect by concentration and types of surfactants. Synthesis of zeolite has gained importance, particularly for tailor making the crystal size/ morphology which are very important aspects for zeolite to be used as catalyst [17]. One approach for control of crystal size/morphology of zeolite is through use of emulsifier during synthesis of zeolites [16,18–20].

#### 2. Materials and methodology

All the chemicals for nano zeolite synthesis used from Sigma-Aldrich and used as received unless otherwise mentions. Mainly following chemical regents were used. Sodium aluminate (NaAlO<sub>2</sub>) (As Alumina source), Sodium metasilicate (Na<sub>2</sub>SiO<sub>3</sub> ·5H<sub>2</sub>O) (As silica source), Sodium Hydroxide (NaOH) and Sodium dodecyl sulfate (SDS) (CH<sub>3</sub>(CH<sub>2</sub>)<sub>11</sub>OSO<sub>3</sub>Na). Deionized water was used in all cases.

The synthesis of Zeolite A via hydrothermal crystallization method

was performed as follows. The total seed solution (Sample HT 1) with

molar ratio of 5.5 Na2O:1.0 Al2O3:4.0 SiO2:190 H2O:0.2 SDS was pre-

pared by mixing the freshly prepared homogeneous aluminate, silicate

geneous aluminosilicate solution, initially 50 ml deionized water was

# 2.1. Conventional hydrothermal synthesis

evaluated by X-ray diffraction (XRD) using an X-ray diffractometer (Rigaku-Ultima IV) operating with a Cu-K radiation ( $\lambda = 1.54$  Å). FTIR measurements were performed by Nicolet iS 50 FT-IR spectrometer from Thermo Fisher Scientific. Those results were employed to confirm the bonding nature of crystalline structure. Scanning Electron Microscopic (SEM) imaging with Energy Dispersive X-ray (EDX) was done for all the samples by using spectroscopy (EEVO/LS 15 ZEISS). Morphological features and crystals size of the synthesized zeolite were examined with their elemental composition.

# 3. Results and discussion

# 3.1. Role of SDS

Anioic surfactant such as SDS, plays significant role during the zeolite synthesis [24]. During the synthesis, SDS plays mainly two different roles as emulsifier and also as a structure directing agent (SDA) [14,16,18]. SEM and XRD results revealed that effect of SDS for formation of LTA type zeolite. It facilitates to control particle size in nano range. Because law temperature conditions favor nucleation over crystal growth and leads to nano-crystals. Increasing the surfactant concentration in precursor suspensions induces a fast nucleation and the formation of smaller crystals with a narrow particle size distribution. In addition, it decreases agglomeration of the precursor and crystalline particles. A large amount of organic template also stabilizes zeolite nanoparticles in the crystalline suspensions even after postsynthesis treatment by the formation of a single organic layer increasing repulsive forces between the zeolite nano-crystals [15].

Surfactant minimizes the Ostwald ripening and control the particle size [13,25]. When the local concentration of the surfactant is high enough, it spontaneously assembles to form micelles within the zeolite crystals. As a result, the crystal structure rearranging around the micelles is driven by (i) the electrostatic interaction, and (ii) the hydrophobic effect responsible for the micelle formation [26]. Most importantly, a use of SDS is greener synthesis method compared with uses of quaternary ammonium salts [17,27]. Concentration of the SDS affects the particle size, size distribution and yield. When the SDS amount used increased, it reduces the particle size. However, Sample HT 2 shows completely different morphology by forming sodalite as major phase. This may be due to lower aging temperature prefer to form sodalite (SOD) rather than LTA zeolite [19,20,24,28].

#### 3.2. X-Ray diffraction studies

The XRD patterns of zeolite A synthesized in both hydrothermal, microwave are shown in Fig. 1. In this case, all the samples were found to have higher crystallinity as expected. Under the both methods,



Fig. 1. XRS pattern of sample (A) HT 1, (B) HT 2, (C) MW 1, (D)MW 2.



Fig. 2. FTIR spectra of sample HT 1, HT 2, MW 1, MW 2.

except HT 2, the products show clear peaks at  $2\theta = 7.20^{\circ}$ ,  $10.19^{\circ}$ ,  $12.49^{\circ}$ ,  $16.14^{\circ}$ ,  $24.04^{\circ}$  and  $30.01^{\circ}$  which are characteristics peaks for Zeolite A / LTA framework and are in good arrangement with that of a LTA dehydrated phase [9,29]. Sample HT 2, sharp intense peak at 25.17°, shows the sodalite (SOD) phase in predominant [10,28]. Low aging temperature prefers to end up with sodalite phase [30].

Zeolite which synthesized hydrothermally have found with much sharper peaks, than the microwave method proved the hydrothermal approach has produce similar size distribution of zeolite A than alternative methods. Compared to MW 1, MW 2 samples, having sharp peaks in HT sample confirmed that synthesized zeolite in conventional approach is narrow size distribution than microwave approach [31].

#### 3.3. Fourier transformations infrared studies

The FT-IR spectrum of nano-sized A zeolite in the range of  $400-4000 \text{ cm}^{-1}$  is shown in Fig. 2 All the characteristic IR bands of the zeolite are observed under each compound.

As shown in figure, the absorption bands of the synthesized products at low temperature show the asymmetric and symmetric vibration bands for LTA zeolite in HT 1, MW 1, MW 2 and sodalite (SOD) in HT 2 in the region 1200 - 400 cm<sup>-1</sup>. Coincident with the spectra, including the asymmetric Al-O stretch located in the region of 1250-950 cm<sup>-1</sup>, and their symmetric Al-O stretch located in the region of 770-660 cm<sup>-1</sup>. The transmittance of the asymmetric stretch of the Si-O-Al in the SOD framework consists of a single band at 980 cm<sup>-1</sup> for HW 2. The bands in the region 650-500 cm<sup>-1</sup> are related to the presence of the double rings (D4R and D6R) in the framework structures of the zeolitic materials [32]. The bands in the region 500 - 420 cm<sup>-1</sup> are related to internal tetrahedron vibrations of Si-O and Al-O of the zeotypes. The region 800 - 400 cm<sup>-1</sup> can be considered as the fingerprint region for zeolite LTA and SOD. The typical bands of zeolite LTA representing the asymmetric Al-O (973–980 cm<sup>-1</sup>) and symmetric Al-O (673–690 cm<sup>-1</sup>) stretches, double rings  $(530 - 538 \text{ cm}^{-1})$  in the framework structures of the zeolitic materials were observed with a constant intensity. The FT-IR peak width is an indication of the level of the crystallinity of the synthesis products [30,33].

The peaks were given as 1003, 664, 557 and 466 cm<sup>-1</sup> for zeolite A. The broad band around 1650 cm<sup>-1</sup> is attributed to OH bending vibration of zeolitic water, the bands at 452 cm<sup>-1</sup> is due to internal vibrations of (Si, Al)O<sub>4</sub> tetrahedral of zeolite A, whereas the band 1008 cm<sup>-1</sup> is due to asymmetrical vibrations related to (Si, Al)O<sub>4</sub> tetrahedral



Fig. 3. SEM images of sample (A) HT 1, (B) HT 2, (C) MW 1, (D)MW 2.

of zeolite A [32,34]. Generally, the absorption bands within the range 420–500 cm<sup>-1</sup> are related to the T–OT– bending of vibration mode (T = Al, Si) and within the range 950–1250 cm<sup>-1</sup> are related to the T–OT– stretching vibration mode [30,32,33].

#### 3.4. Scanning Electron microscope studies

A SEM study was carried out to provide information about the particle morphology and crystal growth process. The SEM images of products crystallized at different conditions by hydrothermal crystallization and microwave digestion are presented in Fig. 3. It confirmed that all the samples were in higher crystallinity nature. The SEM reveals the presence of cubic crystals structure with sharp planer surface and well define edges as shown in figures [30,35].

It is obvious that hydrothermal crystallization has produced much larger particles about 500-850 nm range. This might be attributed to the mild temperature, which could help the system to be well distributed in conventional zeolite synthesis process [10,36].

The SEM images proved that zeolite-A crystals were morphologically similar and samples prepared by microwave approach are ultrafine with average size smaller than 350 nm. In addition, the uniformity of particle size distribution was higher than conventionally synthesized Zeolite A. This is due to supplying sufficient kinetic energy for SDS molecules. Comparing with hydrothermal method, microwave method, supply higher amount of energy. When the zeolite particles were formed, particles were covered by SDS and this process will prevent further growing of particles. Therefore, it has smaller particle size and narrow particle size distribution Table 1, 2.

According to the results obtained by EDX analysis, Na, Al, Si, O founded as major elements. Based on above elemental percentage Si/Al ratio was calculated (Table 3). It has been discovered the ratio of Si / Al for Zeolite-A is 1.03. According to this survey, that ratio is varying in

between 0.91-0.93.

# 4. Conclusions

The zeolite-A (LTA) nanoparticles were successfully synthesized by both conventional hydrothermal and microwave methods with sodium dodecyl sulfate as particle size controlling agent. The XRD results revealed that all synthesized samples have produced zeolite-A phase with high degree of crystallinity. The results showed that aging temperature above 40 °C, the pure zeolite-A phase was produced by hydrothermal method with higher crystallinity (sample HT 1). In addition, microwave method produced considerable amount of zeolite A within a short reaction time. Higher reaction temperature and SDS concentration produced smaller size in particles (MW 2). The XRD patterns revealed that increasing temperature from 100-110 °C resulted in higher degree of crystallinity with smaller particle size. Zeolite synthesized by microwave method had narrow particle size distribution with average size 300-350 nm in comparison with hydrothermal method. Further, the observed FTIR spectra confirmed the formation of expected zeolites with crystalline structures.

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#### Author contributions section

Ishara Kannangara: He was undergraduate final year student and this research was conducted as his final year project.

Yohan Jayawardhana: Conducted some experiments and guide the

#### Table 1

Different experimental conditions for Hydrothermal (HT) and microwave (MW) methods.

Sample	SDS Concentration /moldm <sup>-3</sup>	Aging Time /(days)	Aging Temperature /(°C)	Crystallization	
Name				Time	Temperature /(°C)
HT 1	0.05	2	50	4 days	60
HT 2	0.1	3	40	4 days	65
MW 1	0.05	2	50	3 hrs	90
MW 2	0.1	3	40	3 hrs	100

#### Table 2

Effect of method, aging time, temperature for zeolite A synthesis.

Sample	Aging Conditions		Synthesis conditions		D <sub>90</sub> /nm	Yield %
Manie	Time /(Days)	Temperature /(°C)	Time /(Hour)	Temperature / (°C)		
MW 1	2	50	3	100	388.8	55
MW 2 HT 1	2	40 50	3	60 67	675.7	100
HT 2	3	40	4	65	3624.6	-

#### Table 3

Summarized results of EDX analysis.

Sample Name	Atomic percentage%					Si /Al ratio
	Al	Si	0	Na		
MW 1 MW 2 HT 1 HT 2	7.33 7.04 8.31 8.18	6.86 6.46 7.76 8.67	74.55 75.24 72.52 75.05	11.19 11.26 11.41 8.10	0.93 0.91 0.93 1.06	

final year student.

Erandi Munasinghe: Conducted some experiments and guide the final year student.

Anuradha Rajapakse: Conducted some experiments

Athula Bandara: Contribute to prepare manuscript and co-supervisor

Rohan Weerasooriya: Contribute to prepare manuscript and co-supervisor.

Lakmal Jayarathn: Corresponding author and prepared manuscript. Principle Supervisor.

# **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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