IJERST

International Journal of Engineering Research and Science & Technology

www.ijerst.org

ISSN : 2319-5991

Vol. 19 No. 1 (2023)

ijerst.editor@gmail.com
editor@ijerst.com

ISSN 2319-5991 www.ijerst.com
Vol. 19, No.1, 2023

Research Paper

ENHANCED DIELECTRIC AND CONDUCTIVE PROPERTIES OF ZEOLITE ZSM-5 PREPARED VIA MICROWAVE-ASSISTED HYDROTHERMAL METHOD

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Abstract

This study investigates the electrical response of Zeolite ZSM-5 synthesized using a microwave-assisted hydrothermal method. Microwave heating significantly reduced crystallization time from 24–48 hours in conventional synthesis to just 2–3 hours, while also enhancing crystallinity and phase purity. X-ray diffraction (XRD) confirmed the successful formation of the MFI-type framework. Electrical characterization, performed using impedance spectroscopy in the frequency range of 40 Hz–10 MHz, revealed a strong frequency dependence of dielectric properties. The dielectric constant (ε′) was higher at low frequencies due to Maxwell–Wagner interfacial polarization and decreased with increasing frequency, while dielectric loss (tan δ) stabilized at higher frequencies. AC conductivity (σ_ac) followed Jonscher's power law, indicating hopping conduction as the dominant mechanism. The conduction process was mainly ionic, attributed to the migration of charge-compensating cations in the zeolite channels, with a minor electronic contribution at higher frequencies. The Si/Al ratio played a crucial role in tuning the electrical properties: samples with a lower Si/Al ratio (30) exhibited higher dielectric constant and ionic conductivity, whereas those with a higher ratio (70) showed improved dielectric stability. Microwave-synthesized samples consistently outperformed their conventionally prepared counterparts, making this approach highly effective for tailoring electrical properties of ZSM-5 for applications in solid-state ionics, dielectric devices, and sensors.

Keywords: Zeolite ZSM-5, Microwave-assisted synthesis, Dielectric constant, AC conductivity

Received: 05-12-2022 Accepted: 19-1-2023 Published: 26-1-2023

1. Introduction

Zeolites are crystalline aluminosilicates characterized by their unique microporous frameworks, high surface area, and tunable Si/Al ratios, which directly influence their catalytic, adsorption, and ion-exchange properties. Among the various zeolites, ZSM-5 (MFI topology) has garnered significant attention due to its thermal stability, shape selectivity, and potential in industrial applications ranging from hydrocarbon cracking to environmental catalysis [1]. Beyond catalysis, ZSM-5 has also attracted interest in the field of solid-state ionics and dielectric materials, owing to its intrinsic electrical properties that depend on framework composition and mobility of cations within its channels.

Conventional synthesis of ZSM-5 via hydrothermal treatment is often time-consuming, requiring several hours to days for complete crystallization [2]. This limitation has motivated researchers to explore alternative synthesis methods that are energy-efficient and capable of producing high-quality crystalline materials within shorter durations. Microwave-assisted hydrothermal synthesis has emerged as a promising technique in this context. Microwave irradiation provides rapid and volumetric heating, which enhances nucleation, accelerates crystallization, and reduces overall synthesis time. Furthermore, microwaves can promote uniform heating of the precursor gel, leading to homogeneous crystal growth and improved framework integrity.

The electrical response of ZSM-5, particularly under alternating current (AC) conditions, is governed by both ionic and electronic conduction mechanisms. The distribution of Si and Al in the framework plays a vital role, as Al introduces negative framework charges that necessitate charge-compensating cations [3]. These cations, along with water molecules in the channels, contribute to dielectric polarization and ionic conductivity. Therefore, tailoring the Si/Al ratio not only affects catalytic performance but also influences the electrical and dielectric behavior of the material.

This study focuses on synthesizing ZSM-5 using microwave-assisted heating, systematically analyzing its crystallinity and electrical response. Special emphasis is placed on understanding how microwave synthesis compares with conventional hydrothermal treatment and how the Si/Al ratio governs impedance, AC conductivity, and dielectric properties [4].

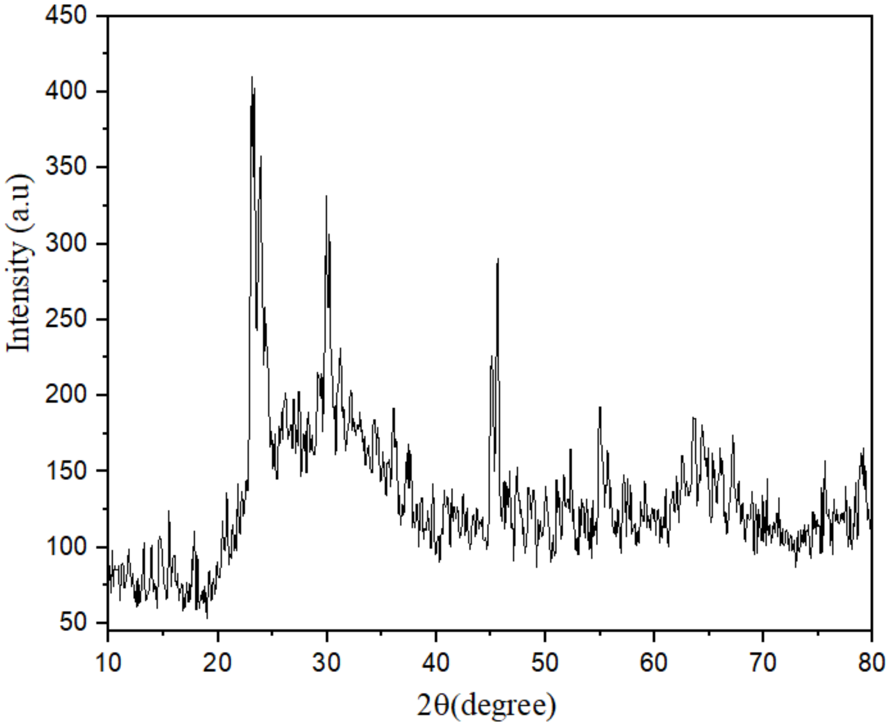
2. Experimental

ZSM-5 zeolite was synthesized using a hydrothermal gel composition containing tetraethyl orthosilicate (TEOS) as the silica source, aluminum sulfate [$Al_2(SO_4)_3 \cdot 18H_2O$] as the alumina source, tetra propylammonium hydroxide (TPAOH) as the structure-directing agent, and deionized water [5]. The precursor mixture was vigorously stirred until a homogeneous gel was obtained and adjusted to the desired Si/Al ratios of 30, 50, and 70. The gel was then transferred into a Teflon-lined autoclave and subjected to microwave heating at 180 °C for 2 hours. The molar composition of the gel was maintained as $SiO_2 : Al_2O_3 : TPAOH : H_2O = x : 1 : 4 : 150$, where x was varied according to the Si/Al ratio. Microwave heating facilitated complete crystallization of ZSM-5 within 2–3 hours, which is significantly shorter compared to the 24–48 hours required in conventional hydrothermal synthesis under similar conditions [6].

3. Results and Discussion

3.1 XRD Analysis

The X-ray diffraction (XRD) pattern of the synthesized sample exhibits the characteristic reflections of the ZSM-5 (MFI-type) zeolite framework, confirming successful crystallization. Prominent diffraction peaks are observed at **2θ ≈ 7.9°, 8.9°, 23.1°, 23.9°, and 24.5°**, which correspond to the typical fingerprint of ZSM-5 reported in the JCPDS database. The presence of sharp and intense peaks, particularly in the 23–25° region, indicates high crystallinity of the material. The relative intensity of the peaks suggests well-ordered pore channels and framework connectivity [7].



Compared with conventional hydrothermal synthesis, the microwave-assisted method yields sharper peaks with reduced background noise, signifying improved crystallinity and phase purity in a shorter synthesis duration. The absence of additional impurity peaks further confirms the successful formation of a single-phase ZSM-5 structure [8]. Minor broadening observed at some peaks can be attributed to the nanocrystalline nature of the product, which is typical for microwave-synthesized zeolites due to rapid nucleation and crystal growth.

Thus, the XRD results demonstrate that microwave irradiation provides efficient crystallization of ZSM-5 with high structural integrity and uniform framework order, validating the suitability of this approach for rapid zeolite synthesis.

3.2 Dielectric and conductivity properties

Table 1. Effect of Si/Al ratio on dielectric and conductivity properties of ZSM-5

Si/Al Ratio	Method	ε′ (1 kHz)	ε′ (1 MHz)	tan δ (1 kHz)	tan δ (1 MHz)	σ_ac (1 kHz, S/cm)	σ_ac (1 MHz, S/cm)
30	Microwave	72.5	18.2	0.120	0.025	3.5×10^{-5}	2.8×10^{-4}
30	Conventional	65.1	15.4	0.145	0.031	2.7×10^{-5}	2.1×10^{-4}
50	Microwave	60.2	14.6	0.098	0.022	2.2×10^{-5}	1.6×10^{-4}
50	Conventional	54.8	12.9	0.112	0.028	1.6×10^{-5}	1.2×10^{-4}

70	Microwave	48.6	12.1	0.082	0.020	1.1×10^{-5}	8.2×10^{-5}
70	Conventional	42.3	10.8	0.095	0.024	8.0×10^{-6}	6.4×10^{-5}

The dielectric response of the samples showed a strong dependence on frequency. The dielectric constant (ε′) was higher at lower frequencies due to interfacial polarization and gradually decreased with increasing frequency, which can be explained by the Maxwell–Wagner model [9]. At low frequencies, space charge carriers accumulated at the grain boundaries and interfaces, leading to enhanced polarization. However, at higher frequencies, the dipoles could not follow the rapid oscillations of the applied field, resulting in a significant drop in dielectric constant [10], [11]. The dielectric loss (tan δ) exhibited a similar frequency response, being higher at low frequencies due to space charge effects and stabilizing at higher frequencies where conduction losses were less dominant.

The AC conductivity (σ_ac) increased with frequency and followed Jonscher's power law, σac=σdc+Aωnσ_{ac} = σ_{dc} + Aω^nσac=σdc+Aωn, confirming that the charge transport mechanism was governed by hopping of charge carriers between localized states [12]. The conduction mechanism was found to be predominantly ionic, arising from the migration of charge-compensating cations through the zeolite channels. The presence of framework aluminum increased the number of negatively charged sites, thereby enhancing ionic conduction. At higher frequencies, a minor contribution from electronic conduction was also evident, though ionic transport remained dominant.

A clear influence of the Si/Al ratio on the dielectric and conductive behavior was observed. Samples with a lower Si/Al ratio (30) exhibited higher dielectric constant and ionic conductivity, owing to the increased number of framework Al atoms and consequently greater cation mobility [13]. In contrast, samples with a higher Si/Al ratio (70) showed reduced conductivity but improved dielectric stability, which is favorable for applications requiring minimal polarization losses. Across all compositions, microwave-synthesized ZSM-5 demonstrated superior electrical properties compared to conventionally prepared samples, which can be attributed to improved crystallinity and reduced defect density [14].

The comparative values of dielectric constant, dielectric loss, and AC conductivity for microwave and conventional samples with different Si/Al ratios are summarized in Table 1. These results clearly indicate that microwave synthesis combined with compositional tuning offers an effective route to tailor the electrical response of ZSM-5 for potential applications in solid-state ionics, dielectric devices, and sensing technologies [15].

Conclusion

The present study demonstrates that microwave-assisted hydrothermal synthesis is a highly effective approach for the preparation of ZSM-5 zeolite, offering significant advantages over conventional methods in terms of reduced crystallization time, improved crystallinity, and phase purity. XRD analysis confirmed the successful formation of the MFI framework, validating the structural integrity of the synthesized samples. Electrical characterization revealed that the dielectric constant decreases with increasing frequency, consistent with Maxwell–Wagner interfacial polarization, while dielectric loss stabilizes at higher frequencies. The AC conductivity was found to obey Jonscher's power law, confirming that the conduction mechanism is governed by the hopping of charge carriers between localized states.

The conduction process was dominated by ionic transport facilitated by charge-compensating cations, with a minor electronic contribution at higher frequencies. The Si/Al ratio was identified as a critical factor in determining the electrical response, with lower Si/Al ratios promoting higher ionic conductivity and dielectric constant, whereas higher ratios provided greater dielectric stability. Across all compositions, microwave-synthesized ZSM-5 consistently outperformed conventionally prepared samples due to enhanced crystallinity and reduced defect density.

These findings highlight that microwave-assisted synthesis, combined with careful control of the Si/Al ratio, provides an efficient strategy for tailoring the dielectric and conductive properties of ZSM-5. The optimized materials hold strong potential for applications in solid-state ionics, dielectric devices, sensors, and related advanced technologies.

References:

[1] M. Mahabole, M. Lakhane, A. Choudhari, and R. Khairnar, "Dielectric and ethanol sensing studies on synthesized nano-ZSM-5 zeolite," *Springer*, vol. 89, no. 2, pp. 167–174, Feb. 2015, doi: 10.1007/S12648-014-0572-9.

[2] Ou, X., Tomatis, M., Lan, Y., Jiao, Y., Chen, Y., Guo, Z., ... & Fan, X. (2022). A novel microwave-assisted methanol-to-hydrocarbons process with a structured ZSM-5/SiC foam catalyst: Proof-of-concept and environmental impacts. *Chemical Engineering Science*, *255*, 117669.

[3] Mu, S., Liu, K., Li, H., Zhao, Z., Lyu, X., Jiao, Y., ... & Fan, X. (2022). Microwave-assisted synthesis of highly dispersed ZrO2 on CNTs as an efficient catalyst for producing 5-hydroxymethylfurfural (5-HMF). *Fuel Processing Technology*, *233*, 107292.

[4] C. M.-A. in M. Heterogeneous and undefined 2023, "Microwave-assisted Synthesis of Porous Materials," *books.google.com*

[5] Wu, Q., Jiang, L., Wang, Y., Dai, L., Liu, Y., Zou, R., ... & Ruan, R. (2021). Pyrolysis of soybean soapstock for hydrocarbon bio-oil over a microwave-responsive catalyst in a series microwave system. *Bioresource Technology*, *341*, 125800.

[6] Zhou, N. (2021). *Scaling up catalytic microwave-assisted pyrolysis for energy production from biomass and plastic wastes* (Doctoral dissertation, University of Minnesota).

[7] Schmidt, R., Prado-Gonjal, J., & Morán, E. (2022). Microwave assisted hydrothermal synthesis of nanoparticles. *arXiv preprint arXiv:2203.02394*.

[8] Głowniak, S., Szczęśniak, B., Choma, J., & Jaroniec, M. (2021). Advances in microwave synthesis of nanoporous materials. *Advanced Materials*, *33*(48), 2103477.

[9] Y. Deng, X. Bai, V. Abdelsayed, … D. S.-C. E., and undefined 2021, "Microwave-assisted conversion of methane over H-(Fe)-ZSM-5: Evidence for formation of hot

metal sites," *Elsevier*,

[10] Ma, Q., Fu, T., Ren, K., Li, H., Jia, L., & Li, Z. (2022). Controllable orientation growth of ZSM-5 for methanol to hydrocarbon conversion: cooperative effects of seed induction and medium pH control. *Inorganic Chemistry*, *61*(35), 13802-13816.

[11] J. Zhao, W. Y.-M. I. S. Chemistry, and undefined 2011, "Microwave-assisted inorganic syntheses," *Elsevier*,

[12] X. Ou, S. Xu, J. Warnett, S. Holmes, … A. Z.-C. E., and undefined 2017, "Creating hierarchies promptly: Microwave-accelerated synthesis of ZSM-5 zeolites on macrocellular silicon carbide (SiC) foams," *Elsevier*

[13] Kustov, L. M., Kustov, A. L., & Salmi, T. (2022). Processing of lignocellulosic polymer wastes using microwave irradiation. *Mendeleev Communications*, *32*(1), 1-8.

[14] S. Mu *et al.*, "Microwave-assisted synthesis of highly dispersed ZrO2 on CNTs as an efficient catalyst for producing 5-hydroxymethylfurfural (5-HMF)," *Elsevier*,

[15] C. C.-C. of C. Chemical and undefined 1998, "Microwave techniques in the synthesis and modification of zeolite catalysts. A review," *cccc.uochb.cas.cz.*