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Synthesis of Sugarcane Bagasse Based Zeolite and Optimization of Particle Size, Reagent Ratio, Contact Time, and Microwave Influence

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ABSTRACT: The persistent contribution of agricultural wastes to the total global solid waste underscores the urgent necessity for environmentally sustainable approaches to their management. This study explored the synthesis and optimization of zeolite derived from cost-effective sugarcane bagasse via microwave sintering, employing four key process variables: particle size (90-200 µm), reagent ratio (0.5-1), contact time (10-20 min.), and microwave power (400-700 W), through a full factorial design. The investigation revealed that these parameters exerted diverse influences on the porosity of the resultant zeolite. Optimal synthesis conditions were identified at particle size of 200 µm, reagent ratio of 0.5, contact time of 20 minutes, and power of 400 W. A linear model was developed for this process with indices such as R^2 (0.9976), Adjusted R^2 (0.9823), Predicted R^2 (0.8492), Adeq Precision (28.3118), Std. Dev. (0.5942) and C.V. % (0.9632) indicating the high accuracy of the model and its adequacy for predicting the porosity of the produced zeolite with good precision. Additionally, the enhanced porosity exhibited by the synthesized zeolite indicated heightened adsorptive capacity. The substantial adsorption potential of the optimized zeolite thus offers promising prospects for wastewater treatment, effectively addressing numerous environmental challenges and transforming waste into wealth.

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Agricultural wastes represent residual outputs from the production and processing of agricultural goods, potentially containing materials beneficial to humans but often deemed economically unviable due to the high costs associated with their collection, transportation, and processing for beneficial purposes. While estimates of agricultural waste generation are scarce, they are generally recognized as a significant component of total waste in developed nations (Obi *et al.*, 2016). Sugarcane bagasse, classified as an agricultural waste (Michel *et al.*, 2013), offers various potential applications. With its substantial cellulose and silicate content, it holds promise as an adsorbent material (Shah *et al.*, 2016). Moreover, its fibrous nature lends itself to applications in the textile and civil engineering sectors, albeit requiring specific treatments prior to use. Additionally, bagasse can reinforce composite materials, facilitating the creation of innovative materials (Loh *et al.*, 2013). The primary advantage of utilizing bagasse lies in its status as pure waste material. Even after minimal pretreatment, its incorporation into various applications yields economically viable products, many of which are fully or partially biodegradable—a crucial consideration in contemporary environmental discourse. Furthermore, properly treated bagasse fibers exhibit commendable

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mechanical properties (Mahmud and Anannya, 2021) and can be employed in the production of sustainable regenerated textile fibers (Costa *et al.*, 2013). Moreover, bagasse serves as a potential source for nanoparticle production, albeit at a higher cost compared to alternatives (Gilfillan *et al.,* 2012; Slavutsky and Bertuzzi, 2014). Zeolites constitute porous crystalline aluminosilicates comprised of silicon, aluminum, oxygen, alkali metals, and water molecules residing within the pores, alongside cations (Flanigen, 1991; Weitkamp and Puppe, 2013; Jha and Singh, 2016). Renowned for their exceptional attributes, including molecular sieving, high thermal stability, adsorption performance, and shape selectivity, zeolites find extensive application in catalysis, separation, ion exchange, and adsorption (Shyaa *et al.*, 2015; Grigorieva *et al.*, 2019; Kostyniuk *et al.*, 2019).

The catalytic and adsorption efficacy of zeolites is subject to numerous factors, such as the quantity and nature of exchangeable cations, Si/Al ratio, arrangement of elementary units, and thermal stability (Sivaguru and Lalitha, 2014; Balamurugan *et al.*, 2014). Given their remarkable properties, researchers have extensively explored zeolites for pollutant adsorption (Okolo *et al.*, 2000; Beutel *et al.*, 2001; Seo *et al.*, 2014; Ghiaci *et al.*, 2004; Flores *et al.*, 2021; Ma *et al.*, 2022). Utilizing full factorial design within the Design Expert software, modeling and optimizing process conditions were conducted. This methodology serves as a robust tool for refining zeolite synthesis via the microwave method and forecasting the adsorptive potential of the optimized zeolite. Such an approach holds promise for enhancing the efficiency and cost-effectiveness of zeolite production from sugarcane bagasse as an adsorbent, thereby making significant strides in water treatment and environmental remediation. The objective of this study was to synthesize and optimize zeolite derived from cost-effective sugarcane bagasse via microwave sintering, employing four key process variables: particle size (90-200 µm), reagent ratio (0.5-1), contact time (10-20 min.), and microwave power (400-700 W), through a full factorial design.

MATERIALS AND METHODS

Sampling and Pre-treatment: Sugarcane bagasse was procured from local markets in Kaduna State, North West Nigeria. It underwent a thorough washing process with distilled water to eliminate foreign debris, floating residues, and soluble impurities. Subsequently, it was dried at 100°C, ground in two stages, and meticulously homogenized. Sieving was performed to eliminate larger particles, and the sieved material was utilized for zeolite production.

Microwave Heating: Zeolites were synthesized using the microwave heating method described by Oluyinka *et al* (2020). Sugarcane bagasse sieved through a 200 µm mesh was suspended in a solution comprising 0.5 M NaOH and 1.5 M NaCl (in a ratio of 1:10, solid to liquid) within a microwave refluxing system equipped with a flat-bottom flask. The resultant mixture underwent irradiation at a frequency of 2450 MHz, corresponding to a wavelength of 12.2 cm, for a duration of 15 minutes. Following the treatment period, the resulting suspension underwent filtration, followed by repeated washing with distilled water to eliminate excess sodium hydroxide and sodium chloride. Subsequently, it was dried at 100°C in an oven. The synthesized zeolitic materials obtained were stored in clean, airtight plastic containers until required.

Optimization of Zeolite Production: A comprehensive approach utilizing a full factorial design with four factors at two levels was employed to optimize conditions for zeolite production through the Microwave Heating Method. These factors encompassed particle size (90 and 200 µm), reagent ratio (0.5 and 1), contact time (10 and 20 min), and Power (400 and 700 W). The selection of porosity as a pivotal indicator of adsorption efficacy was based on its significance, and its determination was executed employing the stochastic method elucidated by Matko (2004).

Statistical Analysis: The optimization process was facilitated through a full factorial design encompassing four factors and two levels. All experimental data underwent thorough analysis utilizing diverse statistical metrics such as ANOVA, R^2, and Standard deviation. Subsequently, the developed mathematical model was deployed for the construction of response surface plots, enabling the prediction of relationships between independent and dependent parameters. Notably, all statistical analyses were conducted utilizing the Stat-Ease Design Expert 13.0.

RESULTS AND DISCUSSION

Optimization of Zeolite Synthesis via Microwave Method: Optimization represents the strategic utilization of specific methodologies to ascertain the most efficient and effective solutions in addressing problems, designs, or processes. In this investigation, a comprehensive 2^4 full factorial design was employed to optimize the synthesis of zeolites derived from cost-effective sugarcane bagasse utilizing the microwave method. A total of sixteen experimental runs were obtained.

Adequacy of the Generated Model: The experimental design and response for the produced zeolite using microwave sintering method are presented in Table 1. The suitability of the generated model was assessed by plotting various analytical plots. A predicted data versus actual data plot for microwave method was constructed to determine the relationship between the predicted and actual values and to decide on the appropriateness of the model (Figure 1).

Table 1: Experimental Design and Responses for Zeolites Produced from Low-cost Sugarcane Bagasse using Microwave Sintering Method

		Factor 1	Factor 2	Factor 3	Factor 4	Response
Std	Run	A:Particle Size (μm)	B:Reagent Ratio	C:Contact Time (min)	D:Power (W)	Porosity (%)
9	1	90	0.5	10	700	62.2
12	2	200	1	10	700	67.9
5	3	90	0.5	20	400	65.4
13	4	90	0.5	20	700	63.6
2	5	200	0.5	10	400	64.5
10	6	200	0.5	10	700	56
8	7	200	1	20	400	59.6
4	8	200	1	10	400	58.7
1	9	90	0.5	10	400	52
15	10	90	1	20	700	65
14	11	200	0.5	20	700	55
3	12	90	1	10	400	65
16	13	200	1	20	700	60
6	14	200	0.5	20	400	66
7	15	90	1	20	400	61.7
11	16	90	1	10	700	64.5

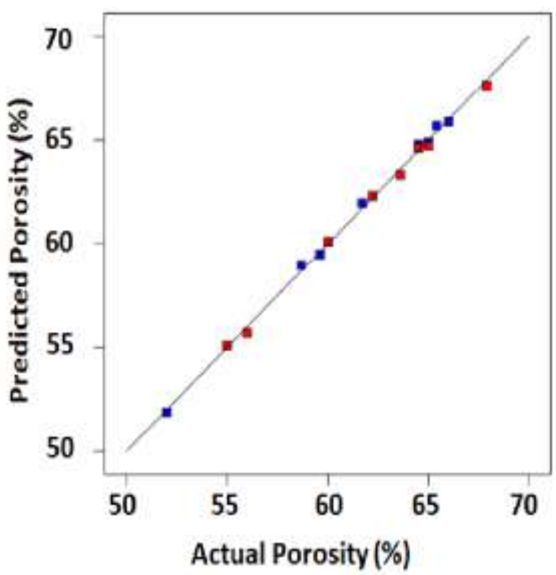

Fig 1: Actual value vs. predicted value plot of response from zeolites produced from Sugarcane Bagasse using microwave sintering method

Consequently, the linear model derived from the full factorial design was deemed optimal for elucidating the impacts of particle size, reagent ratio, contact time, and power on the analytical method (i.e., microwave heating method) employed for zeolite synthesis. The ultimate equations formulated in terms of coded factors for the microwave method employed for the study is presented below for comprehensive understanding:

Equation 1: Final Equation in Terms of Coded Factors for Zeolites Produced from Sugarcane Bagasse using Microwave Sintering Method

$$Y = + 61.6937 - 0.73125 * A + 1.10625 * B + 0.34375 * C - 0.51875 * AB - 1.15625 * AC - 1.31875 * AD - 1.56875 * BC + 1.46875 * BD - 1.21875 * CD + 0.63125 * ABC + 2.16875 * ABD + 0.59375 * BCD - 1.38125 * ABCD$$

Where Y = Response (% Porosity), A, B, C and D are the particle size (μm), reagent ratio, contact time (min.) and power (W) respectively.

Statistical Evaluation: The fit summary, encompassing the sequential model sum of squares and model summary statistics pertaining to the microwave sintering method employed for zeolite synthesis, is elaborated in Table 2). The significance and suitability of the developed model was evaluated via analysis of variance (ANOVA). Upon scrutiny of the sequential model sum of squares and F-value, the model was determined to be highly significant. Hence, the full factorial model was adjudged to be very appropriate for the selected analytical method (microwave sintering method). Furthermore, the outcomes revealed that both the interactive (2FI) and linear model exhibited R^2 values approaching unity (0.9976), signifying a high degree of correlation. Furthermore, the adjusted R^2 value (0.9823) and predicted R^2 values (0.8492) demonstrated reasonable concordance, with a disparity of less than 0.2.

Moreover, notably lower p-values (below 0.05) underscored the adequacy of the model.

Notably, the Model F-values, amounting to 65.13, underscored the significance of the model. This F-value suggests that the factors effectively expound upon the variation in data concerning its mean, thereby substantiating the legitimacy of potential factor effects. Furthermore, the conditions for achieving optimum response for zeolite production from sugarcane bagasse via the microwave sintering method are outlined in Table 3.

Table 2: ANOVA for Factorial Model of Zeolites Produced from Sugarcane Bagasse using Microwave Sintering Method.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	299.00	13	23.00	65.13	0.0152
A-Particle Size	8.56	1	8.56	24.23	0.0389
B-Reagent Ratio	19.58	1	19.58	55.45	0.0176
C-Contact Time	1.89	1	1.89	5.35	0.1467
AB	4.31	1	4.31	12.19	0.0731
AC	21.39	1	21.39	60.58	0.0161
AD	27.83	1	27.83	78.80	0.0125
BC	39.38	1	39.38	111.51	0.0088
BD	34.52	1	34.52	97.74	0.0101
CD	23.77	1	23.77	67.30	0.0145
ABC	6.38	1	6.38	18.05	0.0512
ABD	75.26	1	75.26	213.11	0.0047
BCD	5.64	1	5.64	15.97	0.0573
ABCD	30.53	1	30.53	86.44	0.0114
Residual	0.7062	2	0.3531		
Cor Total	299.71	15			

**R^2 = 0.9976; Adjusted R^2 = 0.9823; Predicted R^2 = 0.8492; Adeq Precision = 28.3118; Std. Dev. = 0.5942; Mean = 61.69; C.V. % = 0.9632*

Table 3: Conditions for Obtaining Optimum Response in the Production of Zeolite from Sugarcane Bagasse via Microwave Sintering Method

Agricultural Waste	Particle Size (µm)	Reagent Ratio	Contact Time (min)	Power (W)	Porosity (%)	Desirability	
Sugarcane Bagasse	200	0.5	20	400	65.888	1.000	**Selected**

Conclusion: The synthesis and optimization of zeolite derived from low-cost sugarcane bagasse were meticulously conducted utilizing the microwave sintering method, employing four variables at two levels through a comprehensive full factorial design. Remarkably, optimal zeolite production was attained at specific parameter settings. It is noteworthy that the optimized zeolite exhibited high porosity, indicative of its high adsorptive capacity. This heightened adsorption potential positions the optimized zeolite as a viable solution for wastewater treatment.

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REFERENCE

Balamurugan, D; Udayasooriyan, C; Vinoth Kumar, K; Jayabalakrishnan, N (2014). Removal of hexavalent Chromium [Cr (VI)] from Spiked Soil Using Na Y (Nano Sodium) Zeolite Supported Zero Valent Iron Nanoparticles. *Environ. Ecol. Res*. 2 (8): 291-300. DOI: https://doi.org/10.13189/eer.2014.020802

Beutel, T; Peltre, MJ; Su, BL (2001). Interaction of phenol with NaX zeolite as studied by ^{1}H MAS NMR, ^{29}Si MAS NMR and ^{29}Si CP MAS NMR spectroscopy. *Colloids Surf. A: Physicochem. Eng. Asp.* 187: 319–325. DOI: https://doi.org/10.1016/S0927-7757(01)00647-1

Costa, SM; Mazzola, PG; Silva, JCAR; Pahl, R; Pessoa, A; Costa, SA (2013). Use of sugar cane straw as a source of cellulose for textile fiber production. *Ind. Crops Prod.* 42: 189–194. https://doi.org/10.1016/j.indcrop.2012.05.028

Flanigen, EM (1991). Chapter 2 Zeolites and molecular sieves an historical perspective. *Stud. Surf. Sci. Catal*. 58:13-34. DOI: https://doi.org/10.1016/S0167-2991(08)63599-5

Flores, CG; Schneider, H; Dornelles, SJ; Gomes, B; Marcilio, R; Melo, JP (2021). Synthesis of potassium zeolite from rice husk ash as a silicon source. *Clean. Eng. Technol.* 4 (100201): 1-7. DOI: https://doi.org/10.1016/j.clet.2021.100201

Ghiaci, M; Abbaspur, A; Kia, R; Seyedeyn-Azad, F (2004). Equilibrium isotherm studies for the sorption of benzene, toluene, and phenol onto organo-zeolites and as-synthesized MCM- 41. *Sep. Purif. Technol.* 40: 217– 229. DOI: https://doi.org/10.1016/j.seppur.2004.03.001

Gilfillan, WN; Nguyen, DMT; Sopade, PA; Doherty, WOS (2012). Preparation and characterisation of composites from starch and sugar cane fibre, *Ind. Crops Prod*. 40: 45–54. DOI: https://doi.org/10.1007/s10570-014-0277-4

Grigorieva, HG; Kostyleva, SA; Bubennov, SV; Bikbaeva, VR; Gataulin, AR; Filippova, NA; Khazipova, AN; Prosochkina, TR; Kutepov, BI; Nama, N (2019). A hierarchically zeolite Y for the N-heterocyclic compounds synthesis. *J. Saudi Chem. Soc*. 23: 452–460, DOI: https://doi.org/10.1016/j.jscs.2018.08.011

Jha, B; Singh, DN (2016). Basics of zeolites, in: *Fly Ash Zeolites*, Springer, 5–31.

Kostyniuk, A; Key, D; Mdleleni, M (2019). Effect of Fe-Mo promoters on HZSM-5 zeolite catalyst for 1-hexene aromatization, *J. Saudi Chem. Soc.* 23: 612–626. DOI: https://doi.org/10.1016/j. jscs.2018.11.001

Loh, YR; Sujan, D; Rahman, ME; Das, CA (2013). Sugarcane bagasse—the future composite material: a literature review. *Res. Cons. Recycl*. 75: 14–22. DOI: https://doi.org/10.1016/j.resconrec.2013.03.002

Ma, Z; Zhang, X; Lu, G; Guo, Y; Song, H; Cheng, F (2022). Hydrothermal synthesis of zeolitic material from circulating fluidized bed combustion fly ash for the highly efficient removal of lead from aqueous solution. *Chin. J. Chem. Eng.* 47: 193–205. DOI: https://doi.org/10.1016/j.cjche.2021.05.043

Mahmud, MA; Anannya, FR (2021). Sugarcane bagasse - A source of cellulosic fiber for diverse applications. *Heliyon* 7 (07771): 1-14. https://doi.org/10.1016/j.heliyon.2021.e07771

Matko, V (2004). Porosity determination by using two stochastic signals *Sens. Actuators A: Phys.* 112: 320-327. DOI: https://doi.org/10.1016/j.sna.2003.10.065

Michel, D; Bachelier, B; Drean, JY; Harzallah, O (2013). Preparation of cellulosic fibers from sugarcane for textile use. In: International Conference on Natural Fibers – Sustainable Materials for Advanced Applications 2013. Hindawi 2013 (651787): DOI: https://doi.org/10.1155/2013/651787

Obi, FO; Ugwuishiwu, BO; Nwakaire, JN (2016). Agricultural waste concept, generation, utilization and management. *Niger. J. Technol*. 35(4): 957-964.

Okolo, B; Park, C; Keane, MA (2000). Interaction of phenol and chlorophenols with activated carbon and synthetic zeolites in aqueous media. *J. Colloid Interface Sci*. 226: 308–317. DOI: https://doi.org/10.1006/jcis.2000.6796

Oluyinka, AO; Alpesh, VP; Bhavna, AS; Maryam, IB (2020). Microwave and fusion techniques for the synthesis of mesoporous zeolitic composite adsorbents from bagasse fly ash: sorption of *p*nitroaniline and nitrobenzene. *Appl. Water Sci.* 10 (236): 1-20. DOI: https://doi.org/10.1007/s13201-020-01327-8

Seo, SM; Park, M; Chung, DY; Lim, WT (2014). Preparation of excessively Ni^{2+}-exchanged zeolite Y (FAU, Si/Al= 1.70) and its single-crystal structure. *J. Porous Mater.* 21: 521–530. DOI: https://doi.org/10.1007/s10934-014-9799-2

Shah, BA; Pandya, DD; Shah, HA (2016). Impounding of ortho-chlorophenol by zeolitic materials adapted from bagasse fly ash: four factor three level Box-Behnken design modelling and optimization. *Arab. J. Sci. Eng.* 42: 241–260. DOI: https://doi.org/10.1007/s13369-016-2294-0

Shyaa, AA; Hasan, OA; Abbas, AM (2015). Synthesis and characterization of polyaniline/zeolite nanocomposite for the removal of chromium (VI) from aqueous solution. *J. Saudi Chem. Soc.* 19: 101–107. DOI: https://doi.org/10.1016/j. jscs.2012.01.001

Sivaguru, P; Lalitha, A (2014). Ceric ammonium nitrate supported HY-zeolite: an efficient catalyst for the synthesis of 1, 8-dioxooctahydroxanthenes. *Chin. Chem. Lett.* 25: 321–323. DOI: https://doi.org/10.1016/j.cclet.2013.11.043

Slavutsky, AM; Bertuzzi, MA (2014). Water barrier properties of starch films reinforced with cellulose nanocrystals obtained from sugarcane bagasse. *Carbohydr. Polym.* 110: 53–61. https://doi.org/10.1016/j.carbpol.2014.03.049

Weitkamp, J; Puppe, L (2013). Catalysis and Zeolites: *Fundamentals and Applications*, Springer Science & Business Media.