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Prof. Liudmyla Gryzun
STRA President, PhD & Post-Doctoral Degree in Pedagogical science
Full Professor of Simon Kuznets Kharkiv National University of Economics
Information Systems Department (Kharkiv, Ukraine)
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Prof. Bahaa Talaat Shawky, Microbial Chemistry Department, Genetic Engineering and Biotechnology Research Division, National Research Centre, Dokki, Giza, Egypt
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Preface:

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Acknowledgements

Our sincere thanks go to our outstanding supporters who made this great and interesting conference possible.
Some special thanks go to our outstanding Key-Note speakers, not only for their inspiring and highly interesting presentations but also for their input and contributions in the discussions and Q&A sessions during the conference:

**KEYNOTE SPEAKERS 2023**

**Topic: Flexible Approach to Strategic Planning for Modern Business Organization**

Dr. Rawani is Director of National Institute of Technology Raipur (India). He has received his M.Tech. in Industrial Engineering and Management from I.I.T. Kharagpur and Ph.D. in Strategic Management from I.I.T. Delhi. He has about 40 years of teaching/research and administrative experience of educational institutes. Dr. Rawani has published more than one hundred and fifty research papers in International/National Journals and International Conferences. He has produced nine Ph.D. under his supervision. He has delivered invited lectures in many countries including Switzerland, Singapore, Malaysia, China, Mauritius, Dubai, USA, Hong Kong, Thailand and Indonesia. He has served as reviewer for many International Journals and conferences. Dr. Rawani is conferred with fellowship award by Indian Institute of Industrial Engineering, Mumbai for outstanding contribution in promotion of Industrial Engineering Profession. In 2022, Dr. Rawani has been conferred with the ‘Education Leadership Award’ by World Education Congress, ‘Exemplary Leadership Award’ by APAC News Network, ‘Director of the year’ Award by Universal Mentors Association and ‘Shiksha Ratna Award’ by Reverie Language Technologies. He was also conferred with 'International Corporate Leadership Award in the field of Education' in 2015 at Bangkok and with ‘Pride of Asia International Award’ in 2014 by Economic Growth Society of India. He has also received ‘Rashtriya Gaurav Award’ in 2010 by India International Friendship Society, India.

![Dr. Rawani](image)

Prof A M Rawani
Director of National Institute of Technology Raipur, India

**Topic: Would Be Quality Management and Organizational Excellence**

Malini Nair is a Business Faculty at the Higher Colleges of Technology. She teaches classes in Quality, Auditing, Innovation & Entrepreneurship, Management and Leadership as well as Economics. She is interested in the use of interventions, technologies, and tools that facilitate group/team processes and lead to better task outcomes through Quality standards. She has developed and taught several courses related to Quality, HR, Innovation, Marketing and Economics to both MBA and undergraduate students. She is currently a member of the Program Advisory Committee for Quality at the University level. She is a Doctoral candidate pursuing her PhD in Business and Management. She has written several research papers and continues do so. She has received a SEED grant for one of her research papers. She is actively involved in Community projects and her forte is mentoring and encouraging her students to actively participate in these initiatives. Prior to her appointment as a faculty, she has an immense amount of industry experience especially in the retail sector in the UAE. She studied Economics Honors and attended the prestigious Birla Institute of Technology (BIT) in India where she obtained her Master’s degree in Business Administration. She went on to work as a Group HR Manager and continued to teach as she believes in sharing the knowledge gained through the industry. Her initiatives at the college level have been highly commended.

![Malini Nair](image)

Malini Nair
Co-Curriculum Chair for the Quality Program in the Business Division at Sharjah Women’s Campus, United Arab Emirates
Abstract: Conventional hydrothermal and alkaline fusion methods have been successfully used to produced Linde Type A Zeolite from Nigeria raw kaolin deposit. The synthesized zeolites were characterized by X-ray diffraction (XRD). The percent crystallinity was estimated from the peak area. The percent crystallinity of as-synthesized Zeolite A ranged from 22.52 to 30.70 % for samples synthesized by conventional hydrothermal method (AC) and from 28.74 to 43.35 % for those produced by alkaline fusion route (AF). The AF’s gave better results than AC’s samples. It was concluded that the fusion process aid in transformation of quartz and kaolinite into large amount of amorphous aluminosilicate (metakaolinite). In each case a replicated 23 two-level factorial design was used to study the influence of three different variables/ parameters (crystallization temperature, crystallization time and ageing time) on the quality of the synthesized zeolites (expressed in terms of CEC). The magnitude of the effect of these parameters and their interaction were also investigated using factorial analysis. The crystallization temperature is the most significant factor and the interaction between the ageing time and crystallization temperature is less significant on the CEC. In both cases the other variables were found to be statistically significant. The average CEC for samples AC and AF respectively ranged between 264.32 – 585.75 and 303.31 – 627.74 meq/100 g. Some of the calculated CEC values are quite high compared to the theoretical value of 548 meq/100 g for zeolite A which may be due to presence of some unidentified/undetected phases or impurities that has adsorption capacity. The synthesized zeolites showed high CEC as compared to other commercial zeolites and therefore can be used in the removal of heavy metals from aqueous phase.

Keywords: Zeolite, Alkaline fusion, Factorial design, Cation exchange capacity and Crystallinity.
earths elements, often called exchangeable cations in the structure of the zeolites which are easily exchanged when zeolites come in contact with solutions of “saturating” or “indexing” ions (Konstantinos, 1999). The commonly exchangeable ions are Ca2+, Mg2+, K+, Na+ and NH4+ and it is usually expressed as milliequivalents per gram (or per 100g) of material. The reaction between a zeolite and an ionic solution is illustrated as: N1(Z) + N2(S) → N2(Z) + N1(S) (1.1) N1 is the exchangeable cation in the zeolite Z and N2 is the saturating or index ion in solution S. According to the work of Mumpton (1999), CEC increase with the aluminum content of the zeolite because more extra framework cation is needed to balance the charge. The most widely used cation exchange process is by treating zeolites with aqueous solutions that contains the cation to be introduced. This is achieved by suspending the zeolites in the solution under the appropriate conditions (amount and concentration of the salt, temperature and pH) for ion exchange, followed by filtration and washing of the filter cake. Most common synthetic zeolites are types A, X, Y and ZSM-5. Due to their exceptional properties, both natural and synthetic zeolites are commercially used in adsorption, ion-exchange, as molecular sieve and as catalyst. Most natural zeolites are of lower Si/Al ratios, since structure-directing agents necessary for formation of siliceous zeolites are absent. Also, the catalytic activity of natural zeolites is limited by their inadequate supply, non-uniform pore size, impurities and low surface areas (Kovo, 2011). As a result of these shortcomings synthetic zeolites were developed mimicking the conditions of their natural counterparts but at lower temperature and shorter time (Auerbach, 2003; Kovo, 2011). Synthetic zeolites are generally synthesized by hydrothermal processes using commercial chemicals (sodium aluminate, aluminium hydroxide, silica gel, sodium metasilicate) as a source of silica and alumina which are quite expensive generally. The use of clay (kaolin) as a combined source of silica and alumina is economical because it reduces the cost of producing zeolite from expensive chemicals (Kovo, 2011). Previous work of Kovo, 2011 on the synthesis of zeolitic material from Ahoko kaolin was carried out by conventional hydrothermal method (which is based on dissolution of metakaolin with alkaline solutions, mainly NaOH followed by hydrothermal crystallization of the aluminosilicate gel) using one-factor-at-a-time approach which fails to detect interaction among variables. In this work, design of experiments (two-level factorial design) was used in the transformation of Ahoko kaolin into zeolite A by two different methods: (i) Conventional hydrothermal alkaline activation (ii) Alkaline fusion prior to hydrothermal treatment. The second method tends to dissolve more of the aluminosilicate and also aid in transformation of quartz and kaolinite into sodium silicate and metakaolinite (Rios, Williams, and Fullen 2008; Espejel-Ayala, Schouwenaars, Dura´n-Moreno, Ramı´rez-Zamora, 2013). Since zeolites vary with location and the optimal way to learn about zeolite synthesis is by examining a single zeolite synthesis from different perspectives (Auerbach, 2003).

2.0 Materials and Experimental Procedure: The raw material is kaolin which is sourced from Ahoko in Kogi state, Nigeria. Prior to zeolite production, the kaolin was refined/beneficiated to reduce the quartz content. This is followed by drying and pulverizing using porcelain mortar and pestle. The chemicals used are: sodium hydroxide pellets (97.5%), sodium hydroxide powder (99%), sodium metasilicate (95%), ammonium acetate (98%), ethanol (99%) and deionised water.

2.1 Experimental Design for Factorial Analysis: In order to reduce the total number of experiments, two level experimental designs with three factors was used. The experimental factors which were chosen from literature are ageing time (hr), crystallization time (hr) and crystallization temperature in (°C). The number of experiments is expressed as

\[ N = (2k) \times R \] (2.1)

Where: N= Number of experiments, k = Number of factors and R = Number of replica

<table>
<thead>
<tr>
<th>Table 2.1: Levels of Independent Variable for Zeolite A</th>
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<tr>
<td>Levels</td>
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<tr>
<td>Coding</td>
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<tr>
<td>Ageing Time (hr)</td>
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<tr>
<td>Crystallization time (hr)</td>
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Table 2.2: Factorial Design Matrix for Zeolite A Synthesis Using Conventional Hydrothermal Treatment (AC) and Alkaline Fusion (AF) Methods.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Ageing Time (hr)</th>
<th>Crystallization Time (hr)</th>
<th>Crystallization Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC1</td>
<td>AF1</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>AC2</td>
<td>AF2</td>
<td>12</td>
<td>3</td>
</tr>
<tr>
<td>AC3</td>
<td>AF3</td>
<td>3</td>
<td>6</td>
</tr>
<tr>
<td>AC4</td>
<td>AF4</td>
<td>12</td>
<td>6</td>
</tr>
<tr>
<td>AC5</td>
<td>AF5</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>AC6</td>
<td>AF6</td>
<td>12</td>
<td>3</td>
</tr>
<tr>
<td>AC7</td>
<td>AF7</td>
<td>3</td>
<td>6</td>
</tr>
<tr>
<td>AC8</td>
<td>AF8</td>
<td>12</td>
<td>6</td>
</tr>
</tbody>
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2.2 Synthesis of Zeolitic Materials: 2.2.1 Conventional Hydrothermal Alkaline Activation: The metakaolin (which served as a combined source of silica and alumina) was obtained by calcinations of refined kaolin in a muffle furnace at 600°C for 1 hour using a crucible. The production of Linde type A zeolite (LTA) was carried out from reaction mixture with molar composition 4.25Na2O:Al2O3:2SiO2:275.4H2O.

In order to produce zeolite A with the above molar composition, 2.434 g of NaOH pellets was dissolved in 35 g of distilled water in reaction beakers and subsequently 1.574 g of metakaolin was added under stirring condition. The aluminosilicate gel was aged at room temperature for different ageing time. Hydrothermal treatment of the aged mixture was carried out in an autoclave at varying crystallization time and temperature. At the end of which the mixture was filtered and washed with distilled water until pH of filtrate were about 8. The samples were subsequently dried in an oven at 80°C for 3 hours and characterized with aid of XRD.

2.2.2 Alkaline Fusion method: In this method, 6.2 g of kaolin (combined source of silica and alumina) was dry mixed with 7.44 g of NaOH powder (kaolin/NaOH = 1/1.2) for 30 minutes. The mixture was heated in a muffle furnace at 600°C for 1 hour. The fused product of kaolin and NaOH was ground and then 4.4 g of this was dissolved in 21.5 ml of distilled water under stirring (in ratio of 4.9 ml of water/g of fused product) under stirring condition to form the amorphous precursors followed by ageing at room temperature and hydrothermal treatment at different crystallization temperatures and times. At the end of the hydrothermal treatment, the sample was filtered, washed (to a pH of 9) and dried in an oven for 6 hours at 80°C. The products obtained were characterized by XRD. Rios et al. (2008) used this technique to synthesize zeolite with molar composition of 3.9Na2O:Al2O3:2SiO2:153.9H2O.

2.3 Determination of Cation Exchange Capacity: The cation exchange capacity (CEC) was determined using the ammonium acetate saturation method at room temperature under standard atmospheric pressure (1atm) using zeolite powders. About 150 mg of the <125 µm size fraction was accurately weighed from each sample and transferred to a mechanical shaker where 10 ml of 1N solution of sodium acetate (CH3COONa) was mixed for 5 minutes. The mixture was then centrifuged in order to obtain a clear supernatant solution which was decanted. This procedure was repeated another two times to ensure that all cations in the zeolite have been replaced with Na ion. The Na-laden zeolite was washed with 30 ml of 99% ethanol and shaken in a mechanical shaker for 5 min. The supernatant was removed, and the procedure was repeated one more time to ensure that the zeolite was clean and laden only with Na. The Na laden zeolite was then mixed with 1N ammonium acetate solution (CH3COONH4) buffered at pH 7 to replace the exchanged sodium ion with ammonium ion. The suspension was well shaken, left overnight and centrifuged. The clear liquid was decanted. The same procedure was repeated with CH3COONH4 five times adding fresh 10ml of CH3COONH4 to ensure that
all Na ions were replaced by NH4 ion. After each step, the supernatant of CH3COO- solution was decanted to a 100 ml volumetric flask which was diluted to 100 ml with ammonium acetate solution and the sodium ion concentration in this supernatant was determined by atomic absorption emission spectroscopy. The sodium binding capacity was then calculated from:

\[ q_e = \frac{(c_o - c_b) \times V}{MW \times m} \]  

(2.2)

Where \( q_e \) is the exchanged Na ions per weight of zeolite (mg/100 g) or CEC value, \( c_o \) is the Na concentration (mg/L), and \( c_b \) is the Na concentration in the blank (mg/L). Both \( c_o \) and \( c_b \) were measured by AAS. \( V \) is the volume of the aqueous phase (ml), equal to 100 ml in this experiment; \( m \) is the amount of zeolite (g), equal to 150mg and \( MW \) is the molecular weight of Na (g).

3.0 Characterization of Zeolitic Materials: The nature of phase and percent purity (crystallinity) of as-synthesized zeolites were identified using an AXS Bruker advance-8 Diffractometer using CuKα (\( \lambda = 0.1541 \text{nm} \)) radiation at 40 kV and 40 mA. Using a scan speed of 0.04° in the 2θ range of 5-50°, the diffraction patterns of as-synthesized zeolites were obtained.

4.0 Results:

![Figure 4.1: XRD Pattern of Laboratory Synthesized Zeolite A (AC Samples)](image)

![Figure 4.2: XRD Pattern of Laboratory Synthesized Zeolite A (AF Samples)](image)

Figures 4.1 and 4.2 show the XRD pattern of the as-synthesized zeolite A using both conventional hydrothermal and alkaline fusion prior to hydrothermal treatment methods respectively. From the conventional hydrothermal method, the formation of zeolite A was observed through the peak diffraction at 2θ = 12.49, 17.69, 24.04, 35.83, 39.53, 43.61, 47.41 and 48.02 as reported by Traecy and Higgins (2001). The zig-zag peaks in the background indicates the presence of amorphous phase of metakaolinite. The peak at 2θ is 28.53 confirm the presence of sodalite. This is in accordance with Ostwald’s rule of successive reaction. From the analysis of the peak area, percent crystallinity was estimated. The percent crystallinity ranged from
22.52 - 30.70 with sample AC7 having the highest percent crystallinity. While the characteristic peak of zeolite A synthesized by alkaline fusion method was observed at 2θ between the range of 7.21, 10.19, 12.49, 20.47, 24.04 and 36.59. The peak due to quartz was observed to be present at 2θ = 26.6 (Treacy and Higgins, 2001) in both instances. The percent crystallinity ranged from 28.74 - 43.35 % with sample AF6 having the highest percent crystallinity. Although the zeolite peaks are weak, but they are quite consistent with the reference especially the AF samples. From the analysis of the results for the two methods of synthesis, except samples AF2 and AC2, samples synthesized through alkaline fusion route (AF) possess higher percent crystallinity than the samples synthesized by conventional method (AC), also the first diffraction peak appeared at 2θ = 7.21 and 12.49 for AF’s and AC’s samples respectively. This can be attributed to the fact that the fusion aid in transformation of quartz and kaolinite into large amount of sodium silicate amorphous aluminosilicate (metakaolinite), which indicates that the fusion process was very effective in extracting the si species in these mixture (Rios et. al., 2008 and Espejel-Ayala et al., 2013). The absence of peak diffraction at 2θ for some of the samples could be due to incomplete transformation of MTK or due to low crystallinity at that point. The full factorial design presented in Tables 2.2 and their uncoded values are shown in Figure 4.3.

![Cube Plot (fitted means) for AC](image1.png)

![Cube Plot (fitted means) for AF](image2.png)

**Figure 4.3. Cube Plots for Zeolite A CEC Response**

The cube plot was used to examine how the process variables affect the zeolite A CEC. The plots show the response surface plot of zeolite A CEC. The cube plot corner points represent a different factorial design and illustrate the average zeolite A CEC result for the synthesis based on the process variables level. Some of the calculated CEC values are quite high compared to the theoretical value of 548 meq/100 g for zeolites A which may be due to presence of some unidentified/undetected phases or impurities that has adsorption capacity. These values are higher than the reported values of San Cristobal (2010) who reported a CEC of 292.8 meq/100 g for zeolite A.

**4.1 Factorial Analysis Results for the Synthesized Zeolites:** The factorial analysis was carried out using Excel 2013 and Minitab release 17. For all the analysis, the t-distribution, coefficients, P-values and estimated effects for the experimental results were obtained. The sum of squares and the F-distribution were also determined. The 95% confidence level was used for the statistical calculations. The effect defined as the increase or decrease of the zeolite CEC when process factor was changed was also determined. A negative effect indicates a decrease in zeolite CEC as the process factor setting is increased. A positive effect designates an increase in zeolite CEC as the process factor setting is increased. The effect of the magnitude is used in ranking the influence of the factors on the experimental results. The regression equation coefficients were also acquired from the fit of the zeolite CEC results, if the P-value is <0.05, then the factor
is significant statistically at the chosen 95% confidence level.

**4.1.1 Factorial Analysis Results Showing Effects of Crystallization Temperature, Crystallization Time and Ageing Time on Zeolite A CEC:** Tables 4.1 and 4.2 show the estimated effects and coefficients for zeolite – A CEC. The result of the estimated effects suggests that the model contains three main effects, which can be evaluated in the absence of significant interactions and three two-way interaction effects. The p-values for all three main effects are less than 0.05 (Crystallization temperature = 0.000, Crystallization time = 0.000 and ageing time = 0.000). Therefore, there is evidence of a significant effect. The p-value result from the Tables also indicates that there is significant interaction between crystallization temperature and crystallization time (0.000) and between crystallization time and ageing time as their terms have p-values less than 0.05 (α=0.05). The Tables also show that crystallization temperature has the greatest effect (177.56 and 176.56) on zeolite A CEC. In addition, the tables show that setting the crystallization temperature high produced higher zeolite A CEC than setting the crystallization temperature low. The interaction between crystallization temperature and crystallization time has the second greatest effect (-108.34 and -115.84) on zeolite A CEC. In addition, setting the crystallization temperature high produced higher zeolite A CEC than setting the crystallization temperature low. The negative sign shows the settings of the two process variables have antagonistic effect (need to be at opposite setting). The result of the main effect plots show that the crystallization temperature is set high and crystallization time is set low to produce zeolite A of high CEC. Crystallization time has the third greatest effect (-86.58 and -93.58) on zeolite A CEC. In addition, setting the crystallization time high produced lower zeolite A CEC than setting the crystallization time low. Ageing time has the fourth greatest effect (-23.09 and -17.09) on zeolite A CEC. In addition, higher ageing time produced lower zeolite A CEC than lower ageing time. The interaction between crystallization temperature and ageing time has the smallest effect (-1.23 and 3.27) on zeolite A CEC. The negative sign shows the settings of the two process variables have antagonistic effect (need to be at opposite setting). The result of the main effect plots show that the crystallization temperature is set high and crystallization time is set low to produce zeolite A of high CEC. Crystallization time has the third greatest effect (-86.58 and -93.58) on zeolite A CEC. In addition, setting the crystallization time high produced lower zeolite A CEC than setting the crystallization time low. Ageing time has the fourth greatest effect (-23.09 and -17.09) on zeolite A CEC. In addition, higher ageing time produced lower zeolite A CEC than lower ageing time. The interaction between crystallization temperature and ageing time has the smallest effect (-1.23 and 3.27) on zeolite A CEC. In addition, setting the interaction high produced lower zeolite A CEC than setting the interaction low. The values in brackets are for AC and AF respectively.

**Table 4.1: Estimated Effects and Coefficients for Zeolite A (Samples AC) CEC**

<table>
<thead>
<tr>
<th>Term</th>
<th>Effect</th>
<th>Coef</th>
<th>SE Coef</th>
<th>T-Value</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-1143.</td>
<td>2.56</td>
<td>145.35</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Crystallization Temperature</td>
<td>177.56</td>
<td>19.95</td>
<td>34.71</td>
<td>0.000</td>
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</tr>
<tr>
<td>Crystallization Time</td>
<td>-86.58</td>
<td>227.9</td>
<td>-16.93</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Ageing Time</td>
<td>-23.09</td>
<td>29.5</td>
<td>-4.51</td>
<td>0.002</td>
<td></td>
</tr>
<tr>
<td>Crystallization Temperature*Crystallization Time</td>
<td>-108.34</td>
<td>-3.102</td>
<td>2.56</td>
<td>-21.18</td>
<td>0.000</td>
</tr>
<tr>
<td>Crystallization Temperature*Ageing Time</td>
<td>-1.23</td>
<td>-0.426</td>
<td>2.56</td>
<td>-0.24</td>
<td>0.816</td>
</tr>
<tr>
<td>Crystallization Time*Ageing Time</td>
<td>12.44</td>
<td>-6.95</td>
<td>2.43</td>
<td>0.041</td>
<td></td>
</tr>
<tr>
<td>Crystallization Temperature<em>Crystallization Time</em>Ageing Time</td>
<td>18.76</td>
<td>0.0926</td>
<td>2.56</td>
<td>3.67</td>
<td>0.006</td>
</tr>
</tbody>
</table>

**Table 4.2: Estimated Effects and Coefficients for Zeolite A (Samples AF) CEC**

<table>
<thead>
<tr>
<th>Term</th>
<th>Effect</th>
<th>Coef</th>
<th>SE Coef</th>
<th>T-Value</th>
<th>P-Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>-1161.</td>
<td>1.56</td>
<td>262.56</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Crystallization Temperature</td>
<td>176.56</td>
<td>20.747</td>
<td>56.53</td>
<td>0.000</td>
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</tr>
<tr>
<td>Crystallization Time</td>
<td>-93.58</td>
<td>244.7</td>
<td>-29.96</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Ageing Time</td>
<td>-17.09</td>
<td>30.27</td>
<td>-5.47</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>Crystallization Temperature*Crystallization Time</td>
<td>-115.84</td>
<td>-3.343</td>
<td>1.56</td>
<td>-37.09</td>
<td>0.000</td>
</tr>
<tr>
<td>Crystallization Temperature*Ageing Time</td>
<td>3.27</td>
<td>-0.437</td>
<td>1.56</td>
<td>1.05</td>
<td>0.326</td>
</tr>
<tr>
<td>Crystallization Time*Ageing Time</td>
<td>14.94</td>
<td>-7.61</td>
<td>4.78</td>
<td>0.001</td>
<td></td>
</tr>
<tr>
<td>Crystallization Temperature<em>Crystallization Time</em>Ageing Time</td>
<td>20.76</td>
<td>0.1025</td>
<td>1.56</td>
<td>6.65</td>
<td>0.000</td>
</tr>
</tbody>
</table>

Pareto charts of the standardized effect for zeolite A CEC response in Figure 4.5 show that there are three significant effects (α = 0.05). These significant effects include crystallization temperature (A), crystallization time (B) and ageing time (C). In addition, it is observed that the Pareto plots show that the largest effect is crystallization temperature (A) because it extends the farthest. In 2011, Kovo concluded that
temperature is an important parameter that influences zeolite crystallization because a small change in heating temperature can cause instant transformation in the zeolite phase. The effect for the interaction between the crystallization temperature and ageing time (AC) is the smallest because it extends the least. The ageing time and crystallization temperature interaction is not significant at $\alpha = 0.05$ level in the estimated effects and coefficients in Tables 4.1 and 4.2.

**Figure 4.5.** Pareto Charts of the Standardized Effect for Zeolite A CEC Response

Main effect plots of the fitted means of Figure 4.6 indicate the following:

- **Crystallization temperature:** Crystallization temperature produced higher CEC at high temperature (100°C) than at low temperature (70°C) as the fitted mean increased from 280 to 460 and 320 to 490 for both AC and AF respectively.

- **Crystallization time:** Crystallization time produced higher zeolite A CEC at low crystallization time (3 hrs) than at high crystallization time (6 hrs) as the fitted mean decreased from 420 to 330 and 460 to 370 respectively for both AC and AF.

- **Ageing time:** Lower ageing time (3 hrs) produced zeolite A of higher CEC than higher ageing time (12 hrs) as the fitted mean decreased from 370 to 360 and 420 to 300 respectively in both cases. The earlier analysis obtained from $p$-values of Tables 4.1 and 4.2 show that the three main effects were significant at the 0.05 $\alpha$-level. By comparing the slopes of the lines on the Main Effect plots of Figure 4.6, the relative magnitude of the synthesis process factor effects were compared. These plots show that there seem to be a large difference in the magnitude of the effects with crystallization temperature being the largest (steepest slope) followed by crystallization time (steeper slope) and ageing time being the smallest.
Method, 3sm. Yti88e8e07thod, 3sm. 3ti88t.

Figure 4.6. Main Effect Plots for Zeolite A CEC Response
Surface plot of zeolite A CEC against crystallization time and crystallization temperature of Figure 4.7 shows how crystallization temperature and crystallization time are related to zeolite A CEC. To maximize zeolite A CEC, high setting of crystallization temperature of 100oC and low setting of crystallization time of 3 hrs, while holding ageing time at 7.5 hrs should be chosen.

Figure 4.7. Surface Plots of Zeolite A CEC against Crystallization Time and Temperature
The model Equations can be built up from estimated coefficients for zeolite A CEC of Tables 4.1 and 4.2.

For conventional hydrothermal method, Y_p=-1143.6+19.95x_1+227.9x_2+29.5x_3-3.102x_1 x_2-0.4261x_1 x_3-6.95x_2 x_3+0.0926x_1 x_2 x_3 (4.1) For alkaline fusion method, Y_p=-1161.6+20.747x_1+244.7x_2+30.27x_3-3.343x_1 x_2-0.4371x_1 x_3-7.61x_2 x_3+0.1025x_1 x_2 x_3 (4.2) Where x_1 = Crystallization temperature x_2 = Crystallization time and x_3 = Ageing time (The model equations are valid if x1, x2 and x3 ≠ 0)

Table 4.5: Statistical Parameters of the Model Correlating Zeolite A CEC to Crystallization temperature, Crystallization time, and Ageing time

<table>
<thead>
<tr>
<th>Statistical Parameters</th>
<th>AC</th>
<th>AF</th>
</tr>
</thead>
<tbody>
<tr>
<td>S</td>
<td>10.2303</td>
<td>6.2462</td>
</tr>
<tr>
<td>R^2</td>
<td>99.60%</td>
<td>99.86%</td>
</tr>
<tr>
<td>R^2 adjusted</td>
<td>99.25%</td>
<td>99.73%</td>
</tr>
<tr>
<td>R^2 predicted</td>
<td>98.39%</td>
<td>99.43%</td>
</tr>
</tbody>
</table>

Table 4.5 shows the model proportion of the response variability that is (R2), the predicted R2, which is the level of prediction of the future data by the model and the adjusted R2 useful for comparing models from the same data with different numbers of terms. The R2 value lies between 0 and 100%. The model predicts better when R2 value is closer to 100% (Doddapaneni, Tatineni, Potumarthi and Mangamoori, 2007). The sum of squares of the prediction errors (PRESS) for assessing model’s predictive ability from Table 4.5 is 10.2303 and 6.2462 for AC and AF samples respectively. Low PRESS value is an indication that the model fits the data as Montgomery (2005) reported that the model fits the data better when the PRESS is smaller.

5.1 Conclusions: Using both conventional hydrothermal and alkaline fusion methods, zeolite-A (NaA) have been successfully synthesized from Ahoko kaolin. It has also been demonstrated by this study the application of factorial analysis in determining the zeolite synthesis parameters that are having significant effect on zeolite CEC. For all the analysis the samples synthesized. From the percent crystallinity estimated from the
peak area, the AF’s gave better results than AC’s samples. Some of the calculated CEC values are quite high compared to the theoretical value of 548 meg/100 g which may be due to presence of some unidentified/undetected phases or impurities that has adsorption capacity. The synthesized zeolites showed high CEC as compared to other commercial zeolites and therefore can be used in the removal of heavy metals from aqueous phase. From the factorial analyses, the effects of crystallization temperature, crystallization time and ageing time and their interactions were investigated and for zeolite A (both AC and AF), the crystallization temperature is the most significant factor and the interaction between the ageing time and crystallization temperature is less significant on the CEC.


**Accelerating Renewable Energy Generation**

Mao Miyazato
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**Abstract:** Accelerating renewable energy generation is essential for the development of a sustainable society. Anaerobic digestion (AD) is a potential system for accelerating clean energy production from organic waste; however, the biogas production efficiency of AD is still poor and requires improvement. Recently, the addition of granular activated carbon (GAC) and the use of microbial electrochemical technology (MET) has reported that the efficiency of biogas production can be improved by promoting electron transfer on microbial reactions in the AD system. GAC facilitates direct interspecies electron transfer (DIET) in the AD system and stabilizes biomass decomposition. The strong conductivity, large surface area, and adsorption capability of GAC were supposed to work as an electron conductor for the DIET effect. However, no precise relationship between biogas production rate and GAC characterization has been investigated. The objective of this study is to investigate the effect of carbon-based conductive materials characterizations on biogas production rate, utilizing AC, GAC, and Biochar with different pore sizes and volumes variation. Activated Carbon showed the fastest biogas production rate and the findings revealed that the growth of both micro and macro pores was the main cause for the acceleration of biogas production efficiency. Although it cannot be certain, earlier research suggested that micropores contribute to adsorption and macropores contribute
to biofilm formation.

**Artificial Neural Network-Based Prediction Model of Properties of Sic Ceramics for the Optimization of Surface Treatment Parameters**

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Mechanical Engineering, Sun Moon University, Korea

**Abstract:** In this study, a novel alternative approach is proposed that is based on the artificial neural network (ANN) concept for predicting the surface properties of SiC ceramic that is subjected to ultrasonic nanocrystal surface modification (UNSM) treatment. In the UNSM treatment, not only the static load but also the dynamic load is exerted. The UNSM treatment is conducted by striking a surface up to 20K times per second with an attached ball to the horn in the range of 1K-100K per square millimeter. Moreover, a post-deposition development of procedures for the successful utilization of UNSM technology in manufacturing will be discussed. The advantage will be taken to provide an understanding of the hardening mechanisms and the microstructural evolution occurring in SiC ceramic by ultrasonic nanocrystal surface modification (UNSM) technology. Experimental measurement data were used in the ANN training process and validation. The trained model showed the capability of predicting the surface roughness and hardness accurately with a Pearson correlation value (R) of 0.984 and 0.997 for surface hardness and residual stress models when tested using the existing test dataset, respectively. It can be concluded that ANN as the alternative approach is a suitable method for accurately performing prediction for practical use in the absence of a mathematical model. Since the experimental result was used in the ANN model training process, the predicted result by the ANN model appears to agree with the experimental results of the UNSM treatment. Because of these demonstration results, the ANN-based prediction model can be used as a prediction tool to optimize the UNSM treatment parameters.

**Optimization Principles of Eddy Current Separator for Mixtures with Different Particle Sizes**

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Yuan Yia  
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Key Laboratory of Electromagnetic Processing of Materials (Ministry of Education), Northeastern University, Shenyang 110819, China

Amor Abdelkaderc  
Department of Design and Engineering, Faculty of Science & Technology, Bournemouth
Abstract: The study of the electrodynamic behavior of non-ferrous particles in time-varying magnetic fields is a promising area of research with wide applications, including recycling of non-ferrous metals, mechanical transmission, and space debris [1]. The key technology for recovering non-ferrous metals is eddy current separation (ECS), which utilizes the eddy current force and torque to separate non-ferrous metals. ECS has several advantages, such as low energy consumption, large processing capacity, and no secondary pollution, making it suitable for processing various mixtures like electronic scrap, auto shredder residue, aluminum scrap, and incineration bottom ash [2]. Improving the separation efficiency of mixtures with different particle sizes in ECS can create significant social and economic benefits. Our previous study [3] investigated the influence of particle size on separation efficiency by combining numerical simulations and separation experiments. A strong correlation between the eddy current force in simulations and the repulsion distance in experiments was found by Pearson correlation analysis, which confirmed the effectiveness of our simulation model. The interaction effects between particle size and material type, rotational speed, and magnetic pole arrangement were examined, which offered some optimization criteria for eddy current separators. The mechanism behind the effect of particle size on separation efficiency was discovered by analyzing eddy current and field gradient. The results showed that the magnitude and distribution heterogeneity of eddy current and magnetic field gradient increased with particle size in eddy current separation. We have further found that increasing the curvature of magnetic field lines within particles can also increase the eddy current force, providing an optimized method to improve the separation efficiency of fine particles. Based on the results of the above-mentioned studies, a more systematic and comprehensive set of optimization guidelines can be proposed for mixtures with different particle size ranges. The separation efficiency of fine particles could be improved by increasing the rotational speed, curvature of magnetic field lines, and electrical conductivity/density of materials, as well as utilizing the eddy current torque. When designing an ECS, the particle size range of the target mixture should be investigated in advance, and the suitable parameters for separating the mixture can be fixed accordingly. The results can guide the design and optimization of ECS, and also expand the application areas for ECS.

Keywords: Eddy current separation, Metal recovery, Numerical simulation, Particle size.

Facile Sol-Gel Preparation of High-Entropy Multielemental Electrocatalysts for Efficient Oxidation of Methanol and Urea

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Abstract: High-entropy multi-elemental (HEM) electrocatalysts present superior catalytic performance due to the efficient synergism of their components. HEM electrocatalysts are usually prepared through hydrothermal reactions or calcination, which could generate undesired heterogeneous structures that hinder the exploration of the structure–property relationship of these HEM electrocatalysts. Herein, we report a sol-gel method to synthesize homogeneous HEM electrocatalysts for electro-oxidation of methanol and urea (methanol oxidation reaction (MOR) and urea oxidation reaction (UOR)), through an acid-catalyzed gelation at room temperature. With Ni as the primary component for MOR and UOR, Co can reduce the overpotentials, while Fe can increase the catalytic activities and durability. Borate and phosphate can tune the charge distribution in active sites and speed up the reaction kinetics through fast proton transfer. Thus, the optimal Ni2Fe0.5Co0.5-BP HEM catalyst demonstrates superior catalytic activity together with good durability and great resistance to CO poisoning. In addition, a direct methanol fuel cell with Ni2Fe0.5Co0.5-BP electrode can not only provide power, but also produce formic acid with high yield and high Faradaic efficiency. This work presents a simple strategy to prepare high-performance HEM electrocatalysts for fuel cells and production of value-added chemicals.

Keywords: Catalytic Oxidation Reaction, High-Entropy Multi-Elemental Electrocatalysts, Sol-Gel, Formic Acid

Listeners

<table>
<thead>
<tr>
<th>Name</th>
<th>Institution</th>
<th>ERCICSTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amor Vincent Canono</td>
<td>Faculty of Computer Science, Union Christian College, Philippines</td>
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</tr>
<tr>
<td>Joseph Olatunde Ogunfemi</td>
<td>Managing Director, 4JS Power Engineering, Lagos, Nigeria</td>
<td>ERCICSTR2303078</td>
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</tbody>
</table>
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