Zeolite NaY From Coal Fly Ash with Ultrasonic Treatment: Synthesis and Characterization

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Abstract
Coal Fly Ash (CFA) is a waste material of the Hydrothermal Thermal Industrial field. Amorphous silica, commonly used as Coal Fly ash, was extracted from coal. The pure silica with a high specific surface area, high melting point and high porosity can be obtained from Fly ash. These properties make the ash a valuable raw material for many fields. The study of synthesized of zeolite from Coal Fly ash. The Coal Fly ash was calcined at temperature 800°C for eight hours using digital muffle furnace to produce pure silica. The Gel composition of synthesized of zeolite from Coal Fly ash was 4.6 Na2O:6 SiO2:Al2O3:144H2O. The gel solution was mixed at room temperature for 24 hours using stainless still autoclave. The characterization of zeolite like X-Ray Diffraction (XRD), FTIR, SEM etc.

Keyword: Coal Fly Ash, Ultrasonic Treatment, Zeolite NaY, Si/Al ratio, Sodium Aluminates, NaOH.

Introduction
The coal fly ash (CFA) generated by coal based thermal power plants has been increased at an upsetting rate throughout the world. Coal ash is the waste that is left after coal is combusted (burned). It includes fly ash (fine powdery particles that are carried up the smoke stack and captured by pollution control devices) as well as coarser materials that fall to the bottom of the furnace. Most coal ash comes from coal-fired electric power plants (Babcock and Wilcox, 1978) Depending on where the coal was mined, coal ash typically contains heavy metals including arsenic, lead, mercury, cadmium, chromium and selenium, as well as aluminum, antimony, barium, beryllium, boron, chlorine, cobalt, manganese, molybdenum, nickel, thallium, vanadium, and zinc.
The Environmental Protection Agency (EPA) has found that living next to a coal ash disposal site can increase your risk of cancer or other diseases (DiGioia and William. 1972). If you live near an unlined wet ash pond (surface impoundment) and you get your drinking water from a well, you may have as much as a 1 in 50 chance of getting cancer from drinking arsenic-contaminated water. If eaten, drunk or inhaled, these toxicants can cause cancer and nervous system impacts such as cognitive deficits, developmental delays and behavioral problems.

They can also cause heart damage, lung disease, respiratory distress, kidney disease, reproductive problems, gastrointestinal illness, birth defects, and impaired bone growth in children. The EPA estimates that 140 million tons of coal ash is generated annually (ACAA, 1997; FHAACAA, 1995; ASTM, 1994; Arsenic is one of the most common, and most dangerous, pollutants from coal ash. The EPA also found that living near ash ponds increases the risk of damage from cadmium, lead, and other toxic metals. More than a third is disposed in dry landfills, frequently at the power plant where the coal was burned. Coal ash may also be mixed with water and disposed in ponds; some are more like small lakes behind earthen walls (Cronstedt, 1756; Breck, 1974; Herreros, 1995). These wet surface impoundments account for about a fifth of coal ash disposal. That makes coal ash the second largest industrial waste stream.

The Chemical Composition of Fly ash from TPS as shown in the following table:

<table>
<thead>
<tr>
<th>Components</th>
<th>Wt.% Of fly ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₂O</td>
<td>2.14</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>30.03</td>
</tr>
<tr>
<td>SiO₂</td>
<td>55.20</td>
</tr>
<tr>
<td>CaO</td>
<td>0.87</td>
</tr>
<tr>
<td>K₂O</td>
<td>1.73</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>3.78</td>
</tr>
<tr>
<td>TiO₂</td>
<td>2.74</td>
</tr>
<tr>
<td>MgO</td>
<td>1.63</td>
</tr>
<tr>
<td>BaO</td>
<td>1.88</td>
</tr>
<tr>
<td>LOI</td>
<td>0.4</td>
</tr>
<tr>
<td>TOTAL</td>
<td>100.4</td>
</tr>
</tbody>
</table>

The synthesis of zeolite is prepared using a minimum excess of reactants by a method wherein required sodium hydroxide, silica, alumina and water reactants are combined in multi-stage procedure to obtain uniform fluid reaction slurry (Meier and Olson, 1992; Subhash Bhatia, 1990; Treacy and Higgins, 1996; Wang and Hongzhu, 1998). The procedure permits the efficient commercial production of high quality type zeolite and minimizes the formation of excess silicate containing by-product effluent.

EXPERIMENTATION

Initially the collected collected CFA was washed with deionized water, number of times to remove the adhering materials i.e. carbon particlals, dust, mud etc and then dried at room temperature. to burn out lower degradable materials and removal of moisture. Finally, the CFA was calcined at 800°C for 5 hrs. in digital muffle furnace. After cooling, Coal Fly Ash (CFA) was screened through a BSS Tyler sieve of 80-mesh size to get uniform particles for our further investigations.

15gm of coal fly ash, 12gm of NaOH, 6.5gm of Sodium Aluminates, 300ml of H₂O, are mixed together and the mixture is stirred for 2hr then the homogeneous gel is formed. After formation of homogenous gel the ultrasonic treatment of time 30min., power 60, pulsation70. The gel aged for 24hrs .The hydrothermal treatment given at 80°C for 8hrs. The synthesized product was filtered and washed with distilled water until pH is neutral, and then it is dried at oven. This product was analyzed with X-Ray Diffraction (XRD).

The chemical composition of synthesis of zeolite NaY from CFA as

\[ 1\text{SiO}_2:0.63\text{Al}_2\text{O}_3:3.8\text{NaOH}:72.7\text{H}_2\text{O} \]

RESULTS AND DISCUSSION:

X-ray diffractograms of commercial zeolite is shown in Figure 1. It indicates that the sample is zeolite crystal, but has different crystallite percent. Percent of crystallinity and unit cell are calculated by following equation.
The characteristic peaks of CFA Zeolite NaY starts appearing after 80°C and the fully crystalline phase is obtained around 100°C (with 2θ= 2.12°, 3.78°, 4.4° and 5.8° values). This unusual shorter crystallization temperature may be due to higher reactivity of the source of silica extract derived from CFA.

This most crystalline sample in the synthesis system was treated as 100% crystalline.

**XRD of Zeolite NaY from CFA:**
It is surprisingly evident that the % crystallinity of CFA Zeolite prepared from no cost industrial waste such as coal fly ash is comparable to that synthesized from expensive and toxic chemical sources like tetra ethyl silicate, fumed silica, sodium silicate (Brett et al., 2003; Cundy CS., 2005; Trigueiro et al., 2002) etc.

The obtained values of % crystallinity were further plotted as a function of synthesis-temperature from which the gradient of crystallization has been evaluated. The percent conversion from amorphous to 100% crystalline product of CFA Zeolite NaY phase is shown in the Fig. 2. The kinetic curve describing the increase in the crystallinity of the crystals with the synthesis temperature (before and after calcination) is nearly "S" shaped that depends on rate of conversion. This type of sigmoidal nature of crystallization curve indicates two distinct stages, namely an induction period and a crystal growth period.

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% Crystallinity = \frac{\text{Sum of the peak height of Unknown material}}{\text{Sum of peak height of standard material}} \times 100

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![Figure 1. The XRD of Zeolite NaY from CFA](image1)

![Figure 2. Kinetics of crystallization](image2)

![Figure 3. SEM of Zeolite NaY from CFA](image3)
It is seen from the Fig.2. That up to 80°C the rate of conversion of amorphous to crystallization of CFA Zeolite NaY, phase was initially slow and then it increased sharply between 80°C to 100°C followed by subsequent slow down. Therefore, the rate of crystallization decreases as the process approaches to the completion indicated by constancy (100%) in percent crystallization.

The scanning electron micrographs (SEM) of the original and treated CFA are depicted in Fig.3. The morphology of the silica extracted from the Coal Fly Ash (CFA), shows agglomerated particles varying in sizes shapes confirming their tendency to cluster together with the uniform size in a range of 25-76 µm and shapes of the particles are like a silk cocoon with random arrangement.

The SEM photomicrograph reveals the siliceous nature of the ash, which is also confirmed by the presence of quartz in the XRD. Close examination of the SEM photomicrograph also suggests that CFA is highly porous. The porous nature of CFA and its honeycombed structure is responsible for its high specific surface area.

CONCLUSIONS

From the experimental result for synthesis zeolite using Coal Fly Ash, characterization analysis using X-Ray Diffraction (XRD), and optimize analysis using statistical software can conclude as follows

Coal Fly Ash is potential silica sources for produce synthesis zeolite. The temperature and time of synthesis are not affected of unit cells length, but affected of percent of crystallinity.

Temperature at 80°C and time at 8 hours are the best operation condition of synthesis zeolite, because the high percent crystallinity is obtained. The optimum condition for synthesis of zeolite from Coal Fly Ash is temperature at 80°C.

REFERENCES