Zeolite has been synthesized from rice husk ash for use as a builder in surfactant in the detergency process. The use of this material as a builder aims to replace sodium tripolyphosphate (STPP) which is not environmentally friendly. In this research, zeolite was synthesized from sodium silicate derived from rice husk ash using NaOH with different concentration (1.0, 1.67; 3.33; 5.0, 6.67 M) and sodium aluminate by sol-gel method followed by hydrothermal process at 100 °C for 5 hours. The synthetic zeolite was then characterized using XRD. The results showed that the mineral formed was a mixture of sodalite, zeolite Na-A, zeolite-A and zeolite Na-Y. There was a shift in basal spacing to a shorter distance. Zeolite Na-Y gave higher detergency than other zeolites and STPP. Moreover, the higher the concentration of NaOH used the higher the detergency of the synthetic zeolite. In this research, the highest concentration of NaOH 6.67 M enabled to reach 94.313 % detergency.

Key word: Synthesized zeolite, builder

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Introduction

Detergent is a cleaning agent, consisting of a mixture of several chemical substances, i.e. surfactant as a surface active agent; builder that typically use a compound phosphate, citrate, acetate, or silicate (zeolite); filler and as additives such as fragrances, dyes, bleaches, and others. Substance builder in the detergent binding function of Ca and Mg cations in hard water, maintain pH, stabilize colloids, preventagglomeration, reduce production costs, buffer capacity, bleach compatibility, the mouth toxicity, eye irritation, effects on the environment and economic value (Yangxin et al., 2008).

Zeolites are hydrated aluminosilicate minerals with a three-dimensional structure is open so it is useful to address the mobility of toxic elements in a number of environmental applications (Wu et al, 2008). Zeolites have the ability to exchange cations (Belviso et al, 2009). Ions can be exchanged with similar ions, these capabilities are widely used in industry, one of them used in the detergent industry. The use of zeolites as detergent builder has previously been done by Hui et al., 2006 that the synthesis of zeolite 4A prepared from coal fly ash using the hydrothermal method. The results showed that the synthesis of zeolite 4A can eliminate calcium ions during the wash cycle and toxicological safety. Zeolite is able to replace the role of phosphate as forming a builder in detergents, because zeolites can prevent the formation of inorganic salts that dissolve poorly in water. (Hui et al., 2006)

Zeolite is able to replace the role of phosphate as a builder in detergents, because it can lead to deposits of phosphate increase in water or cause eutrophication (Udhoji et al, 2005). The main mineral zeolite framework is to form silicate and aluminate. Silicate taken from rice husk ash can reaches 86.9 to 97.80% (Yusof et al., 2010).

Synthetic zeolites can be prepared by using the sol-gel method followed hydrothermal, typical variation is a variation of components, hydrothermal temperature variations, and variations of hydrothermal time. Zeolite synthesis reaction is described in equation:

\[
\text{Na}_2\text{Al(OH)}_6 (aq) + \text{Na}_2\text{SiO}_3 (aq) + \text{NaOH} (aq) \rightarrow 25^\circ C \\
(\text{Na}_a \text{[AlO}_2]_b \text{[SiO}_2]_y \text{NaOH.H}_2\text{O}) \text{gel} \rightarrow 25^\circ C \text{to } 175^\circ C \\
\text{Na}_a \text{[AlO}_2]_b \text{[SiO}_2]_y \text{mH}_2\text{O Zeolite}
\]

Synthetic zeolites can be used as a builder is zeolite containing minerals that have many cations Na thus better able to reduce the hardness of the water that interfere with the process of detergency. Na cations in zeolites are cation balance Al, frame length changes
affect the peak position diffractogram. For example, the replacement of the Al-O bond (1.69 Å) with the shorter bond Si-O (1.61 Å) causes the unit cell to contract. This will reduce the distance d and the diffraction peaks shift towards higher 2θ (Hamdan, 1992:32-33).

Methodology

Rice husk ash (5 g) was dissolved into 50 mL of NaOH at various concentrations i.e., 1, 1.67, 3.33, 5, 6.67 M to obtain sodium silicate. NaOH (20 g) was dissolved in 100 mL of distilled water and added 8.5 g Al(OH)3 and heated with stirring until dissolved to obtain sodium aluminate. 20 mL of sodium aluminate added by 20 mL of sodium silicate then stirred using a magnetic stirrer for 2 hours at 300 rpm. Subsequently incorporated into the hydrothermal apparatus and heated at 100 °C for 5 hours in a tightly closed. Results were formed then filtered with Whatmann filter paper. The solid which formed was then washed with distilled filtrate to a pH of 10-11. Subsequently dried at 100 °C for 12 hours. Results were characterized and used as a detergent builder then compared by STPP builder. Zeolite synthesis coded by ZS1, ZS2, ZS3, ZS4 and ZS5 is a synthetic zeolite with the use of NaOH 1, 1.67, 3.33, 5, 6.67 M.

Detergency process is done by washing cloth (10x10 cm2) with washing solution. Formula washing solution consisting of sodium lauryl sulphate (SLS) 23%, synthetic zeolite 60% and filler to 100%, dissolved in distilled water. Sample code are D1: SLS + ZS1 + Filler D2: SLS + ZS2 + Filler D3: SLS + ZS3 + Filler D4: SLS + ZS4 + Filler D5: SLS + ZS5 + Filler D6: SLS + STPP + Filler D7: SLS + Filler.

Results and Discussion

Methods of X-Ray Diffraction is a qualitative and quantitative analysis methods to characterize or crystalline solids. The data that was obtained in the form of a distance between the fields, the intensity and angle 2 theta (θ) which is then matched with the X-ray diffraction pattern JCPDS-ICDD. Data JCPDS number 11-0401 (Sodalite), number 31-1269 (zeolite A/Na12Al12Si12O48), number 43-0168 (zeolite Na-Y/Na12Al12Si12O48(SiO2)12.25H2O), and number 38-0241 (zeolite Na-A/Na12Al12Si12O48(6H2O)). Diffractogram zeolite synthesis results in the fifth variation of the concentration of NaOH (1.0; 1.67; 3.33; 5.0; 6.67 M), giving the differences are not too significant, visible from the peaks that indicate sodalite and Na-Y that appears the peaks (2θ) of about 14°, 23°, 30° the use of NaOH 1.67; 3.33; 5.0; 6.67 M, whereas the use of 1.0 M NaOH to form zeolite A and zeolite Na-A, which appears the peaks (2θ) of about 7°, 25°, 35° is more dominant. XRD results of zeolite synthesis in the fifth variation of the concentration of NaOH, was not significant, the peaks that indicates sodalite, Na-Y, zeolite A and zeolite Na-A.

Diffractogram synthetic zeolite in the fifth variation of the concentration of NaOH (figure 1) affect to the crystallinity, indicating the increase in the concentration of NaOH resulted in higher crystallinity of the product and increasing the intensity of the reduced crystal phase and an amorphous phase. This indicates the formation of silica-alumina in the zeolite framework, especially sodalite (which is the main forming minerals in the zeolite).

Figure 1.X-ray Diffraction patterns of the prepared zeolite at (a) 1 M (b) 1.67 M (c) 3.33 M (d) 5.0 M (e) 6.67 M of NaOH solution.

Table 1. Basal spacing of each concentration variation of NaOH

<table>
<thead>
<tr>
<th>NO.</th>
<th>Sample</th>
<th>2θ (degree)</th>
<th>Basal Spacing (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>ZS1</td>
<td>7.330</td>
<td>12.0544</td>
</tr>
<tr>
<td>2.</td>
<td>ZS2</td>
<td>7.135</td>
<td>12.3752</td>
</tr>
<tr>
<td>3.</td>
<td>ZS3</td>
<td>14.095</td>
<td>6.2777</td>
</tr>
<tr>
<td>4.</td>
<td>ZS4</td>
<td>14.095</td>
<td>6.2777</td>
</tr>
<tr>
<td>5.</td>
<td>ZS5</td>
<td>13.960</td>
<td>6.3750</td>
</tr>
</tbody>
</table>

Table 1 explain that basal spacing tends to decrease with increasing concentration of NaOH, indicated the increase of Si-O bond is formed. This suggests that the higher the concentration of NaOH added for silicate recovery in rice husk ash so the higher the silicate be obtained.
### Table 2. Detergency of detergent formula

<table>
<thead>
<tr>
<th>No.</th>
<th>Sample</th>
<th>Detergency (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>D1</td>
<td>85.60</td>
</tr>
<tr>
<td>2.</td>
<td>D2</td>
<td>87.14</td>
</tr>
<tr>
<td>3.</td>
<td>D3</td>
<td>92.33</td>
</tr>
<tr>
<td>4.</td>
<td>D4</td>
<td>93.39</td>
</tr>
<tr>
<td>5.</td>
<td>D5</td>
<td>94.31</td>
</tr>
<tr>
<td>6.</td>
<td>D6</td>
<td>70.47</td>
</tr>
<tr>
<td>7.</td>
<td>D7</td>
<td>48.70</td>
</tr>
</tbody>
</table>

The composition of wash solution (detergent) consists of 23% sodium lauryl sulphate (SLS), 60% synthetic zeolite as builder and sodium sulphate as filler. Sodium Tripoly phosphate (STPP) is used as a control. As seen in Table 2, the increase of NaOH concentration for zeolite treatment increased detergency of SLS 94.31%. This suggests that the Z5S is more effective to bind cations interfering detergency process. Cation exchange occurs in Al neutralized by Na cations in zeolite so the more cations Na that can be exchanged, the greater detergency. Figure 1 shows that the zeolite (sample) containing zeolite-Y gives the highest detergency (94.31%), zeolite Na-Y has the molecular formula Na$_{16}$ (AlO$_2$)$_{16}$ (SiO$_2$)$_{25}$H$_2$O, more Na cations than zeolite Na-A (Na$_{12}$ (AlO$_2$)$_{12}$ (SiO$_2$)$_{25}$H$_2$O) and zeolite Na-A (Na$_{12}$Al$_3$Si$_{18}$O$_{75.1}$H$_2$O).

### Conclusions

The conclusion of this research is the synthesis of zeolites from rice husk consisting of zeolite A, zeolite Na-A for the use of 1 M NaOH and Na-Y zeolite, sodalite for the use of NaOH 1.67, 3.33, 5, 6.67 M. highest detergency represented by the formula D5 reaches 94.313% for the use of NaOH 6.67M.

### Acknowledgments

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### References


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### Presentation Discussion

**Q:**

What is the relationship between the zeolite with detergency?

**A:**

The process of detergency require surfactants and other ingredients such as Builder, bleach, deodorant, anti-rust agents and others. Builder is very important in the process of detergency, one of its functions is to bind cations such as Ca$^{2+}$ and Mg$^{2+}$ which would interfere with the work of surfactant. In general, Tripoli Sodium Phosphate (STPP) is used as a builder but STPP is not environmentally friendly and cause eutrophication in water, therefore zeolites (environmentally friendly)could be used as builder, because of zeolit was as a cation exchanger so Ca$^{2+}$ (in water) exchanged with Na$^+$ (in zeolite).

XRD of zeolite synthetics need to be determined, because to study what kinds of minerals contained in zeolite synthesis that can be used in detergency, further study the amount Na on the mineral of zeolite.