



# Hydrothermal Synthesis of Na-P<sub>1</sub> Zeolite from Pumice to Enhance Moisture Content and Water Retention Capacity of Sandy Soil

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**Abstract.** In this study, pumice as a precursor from Semen Shoa, Minjar Shenkora Woreda was used in synthesis of Na-P<sub>1</sub> zeolite. Hydrothermal treatment was performed with sodium hydroxide at different ratio of pumice to NaOH pellets (1:1.2, 1:1.5 and 1:1.8) at varying temperatures of 60, 80 and 100 °C and reaction time of 70,100 and 130 min. Effect of pumice to NaOH ratio, reaction temperature and reaction time on Si/Al ratio of synthesized zeolite was studied. Optimal silica to alumina ratio of 2.5 was obtained at temperature of 100 °C, reaction time of 100 min and pumice to NaOH ratio of 1:1.5. Na-P<sub>1</sub> zeolite obtained at optimum operating conditions was used for other characterizations such as FTIR, XRD and surface area analysis and for the moisture testing & pressure plate extractor testing. The surface area for this zeolite was found to be 56.04 m<sup>2</sup>/g. The XRD patterns of the synthesized material after formation of zeolite Na-P<sub>1</sub> was observed through reflections on  $2\theta = 19.31^\circ$ ,  $23.44^\circ$ ,  $28.32^\circ$ ,  $32.43^\circ$ ,  $33.5^\circ$  and  $49.10^\circ$ . The synthesized zeolite showed the most prominent reflection peak at  $32.43^\circ$ . Effects of different dozes of the synthesized zeolite on sandy soil moisture content and water retention capacity were studied. As the dose of zeolite increased from 0 g/kg to 20 g/kg, the rate of moisture loss from sandy soil decreased. The results showed that application of zeolite to the sandy soil significantly increased the moisture content in the mixtures of soil-zeolite. The water holding capacity of the amended soil was tested using pressure plate extractor at 1, 3, 5 and 15 bars. At 0 bars, all soil samples were observed to have the same water content since at the beginning all soil samples had been moistened with water (70% by weight). Then at 1 bar sandy soil without zeolite had a moisture content about 12% whereas sandy soil with 10 g/kg had 15.65%. As the amount of zeolite added increased from 10 g/kg to 15 g/kg and to 20 g/kg, it was observed that adding zeolite on the sandy soil increases the water retention capacity of the soil.

**Keywords:** Pumice · Na-P<sub>1</sub> zeolites · Hydrothermal synthesis · Moisture content · Water holding capacity · Pressure plate extractor

## 1 Introduction

An understanding of the nature of soils in natural and human influence ecosystems is essential [1]. Soil water retention is a major soil hydraulic property that governs soil functioning in ecosystems and greatly affects soil management. Extensive research has shown that water retention is a complex function of soil structure and composition [2, 3]. Soil organic matter content and composition affect both soil structure and adsorption properties; therefore, water retention may be affected by changes in soil organic matter that occur because of both climate change and modifications of management practices. Thus, effects of organic matter on soil water retention should be understood and quantified [4].

Basically the soil particle size, the soil particle size distribution, and the structure of the soil determine the moisture characteristics (soil water relationships). Soil particles are basically composed of sands, silt, clays and organic matter [5]. Soil texture and structure greatly influence water infiltration, permeability, and water-holding capacity [6]. Soil texture refers to the composition of the soil in terms of the proportion of small, medium, and large particles (clay, silt, and sand, respectively) in a specific soil mass [7]. The contents of particle size classes (sand, 2.0–0.05 mm; silt, 0.05–0.002 mm; and clay, <0.002 mm) are presented according to the FAO/USDA classification system [2]. Soils with smaller particles (silt and clay) have a larger surface area than those with larger sand particles, and a large surface area allows a soil to hold more water [8]. Sand has large particles which take up a lot of physical space. Also, as sand particles do not bind water, a lot of water will drain out of the sand due to gravity before field capacity is reached. For these two reasons, sand has much lower maximum and minimum water content than a clay soil does. The low content of clay in sandy soils usually limits humus accumulation, nutrients, and water availability as well as buffering capacity, which is a reason why many of these soils become acidified [9].

Contrary to most other African soils, the majority of Ethiopian highlands soils remain relatively fertile at depth [10]. All over Ethiopia there are varied rates of soil degradation due to climate, soil types, biological conditions, and plant life and farming systems. The soil is not getting the nutrients that it needs to grow vegetation. Salinization and acidification is seriously affecting crop yield and agriculture productivity [11].

The best sandy soil amendments are ones that increase the ability of the sandy soil to retain water and increase the nutrients in the soil as well [12]. One of the measures considered highly effective, biologically justified and environmentally safe, especially on degraded and other soils having unfavorable productive traits for crop cultivation, is the use of zeolite mineral [13]. Zeolites can be successfully used in cultivating different crops such as cereals, forage crops, vegetables, vine and fruit crops [14]. Their use in crop production stems primarily from high nutrient-exchange capacities, which allow them to absorb and release plant nutrients and moisture without any change in the nature of the zeolite [15]. In addition, zeolite adds a permanent water reservoir, providing prolonged moisture holding power during dry periods and it promotes rapid re-wetting and improved lateral spread of water in the root zone during irrigation [16].

Taking into account problem of sandy soils, it was found an immediate concern and essential to treat sandy soil with synthetic zeolite in order to keep moisture content of

sandy for longer period. Therefore, this study, based on pot experiment, was aimed at synthesizing of Na-P<sub>1</sub> from pumice, locally available rock, and analyzing moisture content and testing water retention capacity of sandy soils.

## 2 Materials and Methods

### 2.1 Chemicals

NaOH was used to activate raw pumice before slurry preparation. Sulfuric acid (98%) was used for pH adjusting until a gel is formed. Analytical grade aluminium sulphate Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> and bentonite (Al<sub>2</sub>O<sub>3</sub>·4SiO<sub>2</sub>·2H<sub>2</sub>O) were used as a precursor for the standard solution preparation. Sodium hexametaphosphate was used as dispersing agent during soil texture analysis. Sandy soil was collected from Wollo farm land since it is the nearest when compared with others. And also clay, other type of soil that was collected from farm land (around Bahir dar) and used as control sample.

### 2.2 Instruments and Equipment

Electric furnace (Nabertherm B180, Germany) was used to activate the mixture of sodium hydroxide. Digital autoclave (Lx-B50L) which was available in environmental laboratory was used for crystallization of gel to get crystalline zeolite. All the above instruments were available in Bahir Dar Institute of Technology. After synthesis of zeolite different characterization techniques such as X-ray Diffraction, Induced Coupled Plasma-Optical Emission Spectroscopy Horiba Scientific and Fourier Transformed Infrared Radiation were used. Characterization of XRD and FTIR were conducted at Addis Ababa University, Faculty of Science whereas elemental analysis using ICP-EOS and surface area measurement using analytical methods (UV-VIS) were conducted at Bahir Dar Institute of Technology Research Grade Lab. Hydrometer (152H) was used for soil texture analysis. Pressure plate extractor (1500F215) was used to analyze the water-holding capacity of soil samples throughout the pressure range of interest. All soil testing materials and equipment were available at Bahir Dar Institute of Technology Soil Laboratory.

### 2.3 Experimental Procedure

#### Pumice Collection and Preparation

The main material used in this work was pumice grain and it was collected from Debrebirhan, Shenkora wereda, around Arerti town (coordinate 097°, 90' 36"N, 054°, 69'15"E) which is 150 km away from Addis Ababa. The pumice was dried at room temperature and ground using jaw crusher and sieved using 75 µm pore size. In order to determine the optimum amount of NaOH that can be used for zeolitization process, different ratio of pumice to NaOH were used. In this Na-P<sub>1</sub> zeolite synthesis pumice powder was mixed with different amount of NaOH (ratio of pumice to NaOH (g/g), 1:1.2, 1:1.5 and 1:1.8) [17] and activated at 600 °C in furnace.

### Activating Raw Pumice Using Sodium Hydroxide

Fixed amount of pumice (14 g) was weighed and mixed with different amount of NaOH (ratio of pumice to NaOH (g/g), 1:1.2, 1:1.5 and 1:1.8) to see the effect of sodium hydroxide on zeoliteization process. Hence, for the first ratio 16.8 g of NaOH pellet was mixed to the 14 g pumice powder (1:1.2), and put into the furnace using crucible for activation. Then the furnace was adjusted to 600 °C for 1 h. After withdrawal of the sample from furnace, it was again grounded to fine particles. Similarly for the 2<sup>nd</sup> ratio (1:1.5), 21 g of NaOH pellet was weighed and added to the 14 g pumice and put to the furnace with same activation temperature and duration. Similarly, the third ratio of pumice to sodium hydroxide (1:1.8) was also activated.

### The Formation of Aluminosilicate Gel as Zeolite Precursor

After preparation of the required pumice to sodium hydroxide ratio, the fused product was dissolved in appropriate amount of distilled water (with a ratio of fused product to volume of distilled water to be 1/4.9 g/ml) [18]. In this step, the slurry was heated on a magnetic stirrer hot plate under vigorous stirring at temperatures of 60, 80 and 100 °C and stirring time of 70, 100 and 130 min. The filtered solution was taken and adjusted its pH to be less than 10 by using H<sub>2</sub>SO<sub>4</sub>.

### Crystallization of Zeolite

After white amorphous gel formation, the resulting gel was then transferred into a beaker. The beaker was placed into autoclave and left for crystallization for 2 days at 180 °C under autogenous pressure. Then, the zeolite products were separated from the solution and recovered by filtration followed by washing the zeolite crystals with distilled water several times until pH of 9-10 of the filtrate was achieved. The zeolites products were then dried in an oven for 24 h at 110 °C. For calcinations, the dried powder was heated to 80 °C and kept at this temperature for 2 h. After that, the product was milled to convert it to a fine powder, which was sieved to a particles size  $\leq 75 \mu\text{m}$  for further analysis.

## 2.4 Determination of Surface Area of Na-P<sub>1</sub> Zeolite Using Methylene Blue Adsorption

The surface area of zeolite was determined by using methylene blue solution. It was done through three steps. The first step was determination of  $\lambda_{\text{max}}$  of absorption. This step was done by measurement of absorbance of the methylene blue solution 2.0 mg/L with ranges of wave length from 500–700 nm [19]. Secondly, using the maximum wave length, the calibration curve of methylene blue was done by measuring the absorbance of methylene blue in the variation of concentration (1, 2, 3, 4 and 5 mg/L). The data obtained was used to make the calibration curve. Then 3 g of Na-P<sub>1</sub> zeolite was added to 1000 ml methylene blue having 50 mg/L concentration. The amounts of methylene blue were contacted with the zeolite in variation of time (30, 40, 50, 60 and 70 min). Absorbance of the methylene blue remained in each contact time are used to substitute y value of the regression equation and therefore the x-values of the equation could be calculated. Amounts of adsorbed methylene blue were difference between the initial concentrations (50 mg/L) with amounts of remnant. The result of calculation was used to draw a curve: number of adsorbed methylene blue (x) per gram of adsorbent

(m) versus time of contact. The optimum amount of methylene blue adsorbed could be used to calculate the surface area of adsorbent by using equation [20]:

$$S_A = \frac{W_m * N * A}{M} \quad (1)$$

Where;  $W_m$  = Methylene blue (mg) adsorbed per gram of Na-P<sub>1</sub> zeolite

$N$  = Avogadro's number ( $6.022 * 10^{23}$  mol)

$A$  = surface area of one mole of methylene blue ( $197 * 10^{-20}$  m<sup>2</sup>/g)

$M$  = Mass of 1 mol methylene blue (320.5 g/mole)

## 2.5 Effect of Na-P<sub>1</sub> Zeolite on Sandy Soil Moisture Content and Water Retention Capacity

Sandy soil was collected from Meket Woreda (in Wollo) to 30 cm depth in an agricultural field (coordinate 11°, 12' 10"N, 38°, 56' 36"E) which is 130 km away from Bahir Dar. Clay soil was collected from farm land (around Bahir dar). After collection, the soil was put in oven at 80 °C for 2 h and allowed to pass through a 5 mm sieve before analysis. But before investigating the effect of zeolite onto sandy soil moisture content and water retention capacity, the soil has to be checked that it was sandy soil using hydrometer analysis.

### Effect of Na-P<sub>1</sub> Zeolite on Sandy Soil Moisture Content

In sandy soil treatments, 0, 10, 15 and 20 g/kg of Na-P<sub>1</sub> zeolite were used with duplication. For all treatments 1000 g of sandy soil was placed in a plastic container. And also 1000 g of clay soil without zeolite was taken as a control. Then, zeolite was added to the sandy soil and mixed well. Then all soil and zeolite mixtures were moistened with tap water to a water content of 40% (w/w). All treatments were then treated under identical conditions. The plastic containers were weighed every 2 h interval for 20 h to determine the soil moisture content using the following formula.

$$\text{Moisture content (\%)} = \frac{W_t}{m_i} \times 100 \quad (2)$$

Where:  $w_t = m_i - m_f$  = Mass of water with container after certain time interval (g)

$m_i$  = Mass of soil with container after moistened with water to 40% (w/w)

$m_f$  = Mass of soil with container after certain time interval (g)

### Effect of Na-P<sub>1</sub> Zeolite on Sandy Soil Water Holding Capacity

Sandy soil preparation for water holding capacity measurement was similar to that of moisture content measuring. For this 50 g of sandy soil was mixed with 10, 15 and 20 g zeolite and moistened with tap water to a water content of 70% by weight. Then the mixture was packed into brass rings. Thereafter, a pressure plate extractor was used to measure water retention at matric potentials of 1, 3, 5 and 15 bars. Water retention was measured in a constant room temperature to minimize changing temperature effects on soil water characteristics.

### *Measuring Moisture at Different Pressures*

Two 1 bar ceramic plates were submerged in water for 24 h to saturate. Then plates were placed on a workbench. The brass rings containing 50 g dried sandy soils with 0, 10, 15 and 20 g/kg and clay soil that were moistened with tap water to a water content of 70% by weight were placed on the plate. The triangular support was placed in the extractor vessel on the bottom. Then the plastic spaces should be placed between plates. In order to collect the extracted water, outflow tubes were connected. The extractor was closed and tightened, ensuring that the “O” ring is in place and all nuts were uniformly tightened. Then a pressure of 1.0 bar was applied. A beaker was placed to collect water from the outflow tubes. Pressure was maintained until no more water was being released for 24 h. Pressure from extractor was released. Finally the extractor was opened and the water collected in beaker was measured. A similar fashion was followed to measure water retention capacity of sandy soil at 3, 5 and 15 bars as it was followed for 1.0 bar. The only difference was the pressure supplied. The pressures applied were 3, 5 & 15 bars and after pressure was released from extractor the corresponding water collected in a beaker was measured.

## 3 Results and Discussions

### 3.1 Characterization of Pumice and Na-P<sub>1</sub> Zeolite

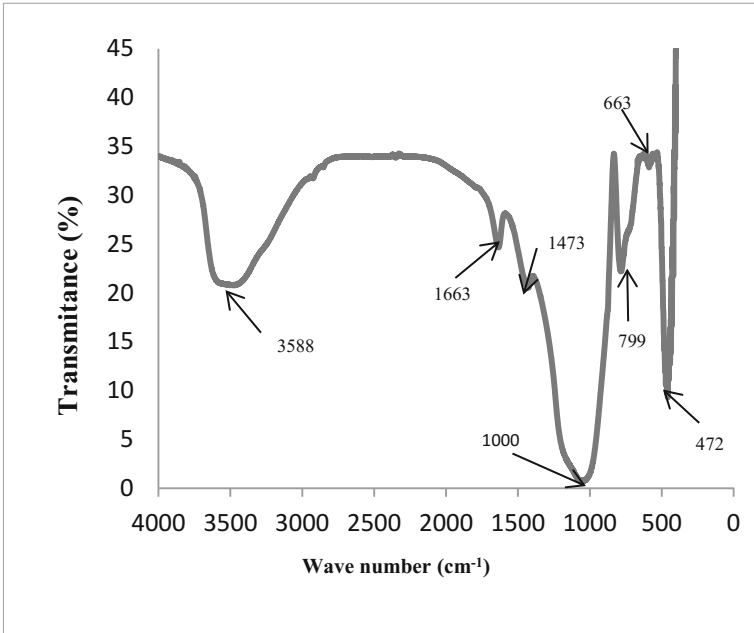
#### **Determination of Si to Al ratio of Na-P<sub>1</sub> Zeolite**

The results of elemental composition for major elements (Si and Al) in ICP-EOS were expressed in mg/l. The concentrations of all the analysis solutions were determined on the basis of the calibration points. The obtained result is in agreement with the Si/Al ratio of Na-P<sub>1</sub> zeolite reported elsewhere [21] which is in the range of 2 to 3. At low value of pumice to NaOH ratio, reaction temperature and reaction time the obtained Si to Al ratio was low. The gel obtained at these low operation conditions requires longer aging period and longer hydrothermal treatment to obtain very crystalline zeolite. Hence, at lower operating conditions the obtained zeolite cannot be selected as optimal Si/Al ratio. Therefore the optimum operating conditions at pumice to sodium hydroxide, reaction temperature and reaction time were 1:1.5, 100 °C and 100 min respectively. Na-P<sub>1</sub> zeolite obtained at these operating conditions was used for other characterizations such as FTIR, XRD and surface area analysis.

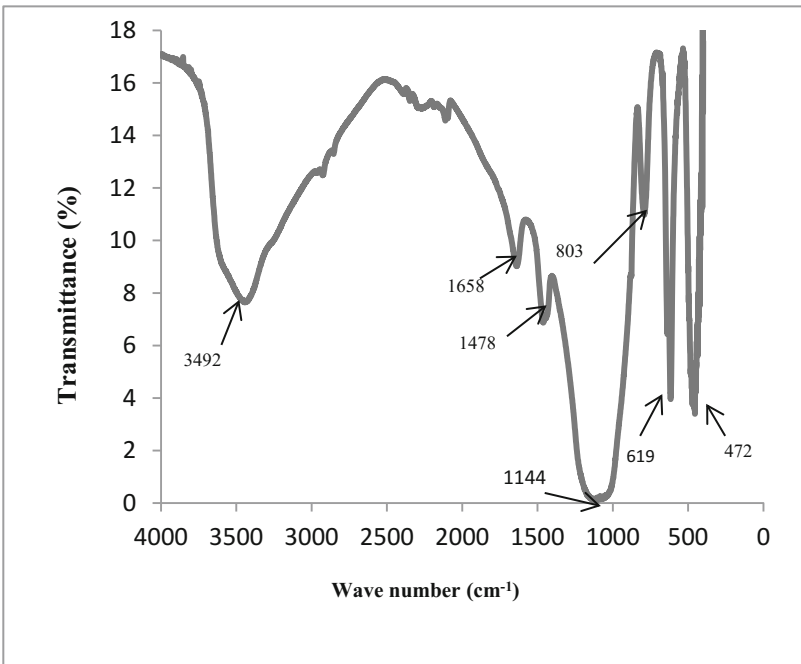
#### **FTIR Analysis of Pumice and Na-P<sub>1</sub> Zeolite**

Comparison of IR spectra of raw pumice and zeolite Na-P<sub>1</sub> is shown in Figs. 1 and 2 respectively. Pumice showed a band at 3588 cm<sup>-1</sup>, which is attributed to free OH stretching vibration. An absorption band at 1663 and 1473 cm<sup>-1</sup> is due to the bending vibration of adsorbed water molecule.

Vibration bands in a region of TO<sub>4</sub> tetrahedral units of pumice at about 1,000 cm<sup>-1</sup>, 799 cm<sup>-1</sup>, 663 cm<sup>-1</sup> and 472 cm<sup>-1</sup> are asymmetric stretching (internal vibration of (Si, Al)-O), symmetric stretching (internal vibration of (Si, Al)-O), double ring and T-O bending vibration (bending mode of Si-O-Al vibration) respectively. Na-P<sub>1</sub> zeolite showed Al-O-H vibration stretching bands around (3601–3355) cm<sup>-1</sup>. A strong



**Fig. 1.** FIR spectra of raw pumice



**Fig. 2.** IR spectra of Na-P<sub>1</sub> zeolite

characteristic structure sensitive bands due to the presence of attached water molecule indicates a water bending vibration at  $(1658, 1478) \text{ cm}^{-1}$  (see Fig. 2).

The vibration which are sensitive to the overall structure and joining of the individual tetrahedral in secondary structural unit as well as their existence in the large pore openings are of second types of vibrations. These were vibration bands in a region of  $\text{TO}_4$  tetrahedral (T = Si or Al) units of zeolite at about  $1144 \text{ cm}^{-1}$ ,  $803 \text{ cm}^{-1}$ ,  $619 \text{ cm}^{-1}$  and  $472 \text{ cm}^{-1}$ . The symmetric stretching vibrations of zeolite Na-P<sub>1</sub> framework structure of Si-O and Al-O were noticed at,  $803 \text{ cm}^{-1}$ . It was observed that, frequencies near  $1144 \text{ cm}^{-1}$  were ascribed to asymmetric stretching of bonds Si-O or Al-O. Na-P<sub>1</sub> zeolite exhibited the bending vibration at around  $430\text{--}580 \text{ cm}^{-1}$ , this bands are related to the deformation mode of the same bonds. The band at  $472 \text{ cm}^{-1}$  was assigned to be O-Si-O bending. The peak at  $619 \text{ cm}^{-1}$  indicates the double ring vibration which constitutes the structure of the zeolite. All the above vibration bands were consistent with the findings of Sudaporn [21, 22].

### Powder X-Ray Diffraction of Pumice and Na-P<sub>1</sub> Zeolite

As shown in Fig. 3, the XRD pattern of raw pumice mainly showed the presence of crystalline quartz and mullite phases. Besides some crystalline phases (quartz, mullite, hematite), pumice was primarily composed of amorphous material. A small amount of quartz was observed by a peak of low intensity at  $2\theta = 26.7^\circ$ .

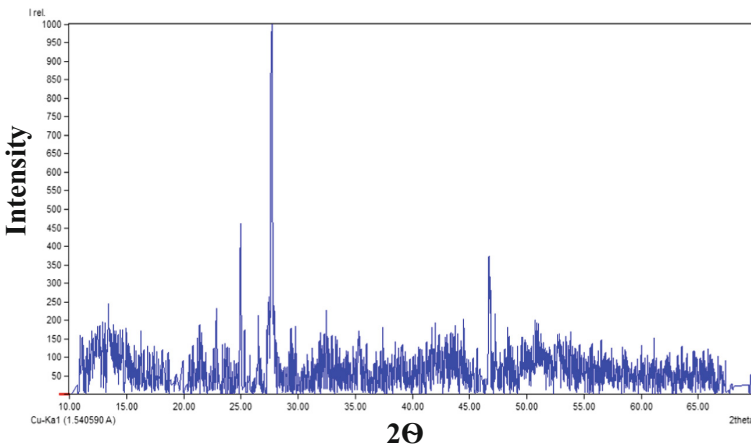


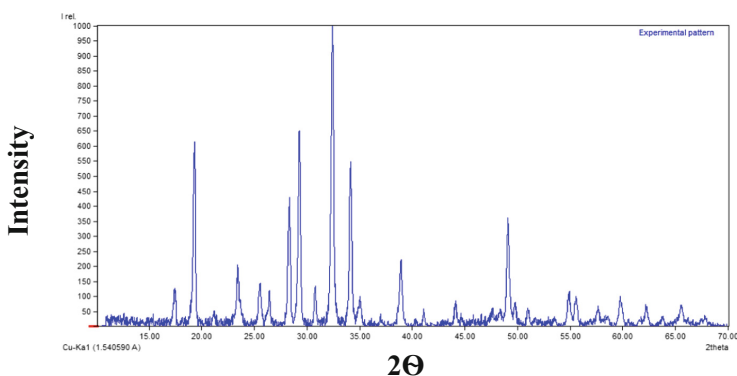
Fig. 3. X-ray diffraction (XRD) of pumice

In XRD patterns of pumice impurities such as mullite, hematite were observed through reflections on two theta values ( $2\theta = 12.48^\circ, 17.66^\circ, 21.89^\circ, 28.29^\circ$  and  $33.4^\circ$ ). This was consistent with the findings of Chigondo et al. which was observed to have reflections on two theta values ( $2\theta = 12.4^\circ, 17.7^\circ, 21.8^\circ, 28.2^\circ$  and  $33.5^\circ$ ) [23].

After pumice treatment, several sharp diffraction peaks of great intensity emerge confirming formation of zeolites (see Fig. 4). The findings of this study showed that XRD patterns of the synthesized product obtained after the zeolitization process from



pumice, the formation of zeolite Na-P<sub>1</sub> was observed through reflections on  $2\theta = 19.31^\circ, 23.44^\circ, 28.32^\circ, 32.43^\circ, 33.5^\circ$  and  $49.10^\circ$  which was consistent with data from other study by Hildebrandoa et al. [24]. According to Hildebrandoa et al., the formation of Na-P<sub>1</sub> zeolite was observed through reflections on  $2\theta = 12.4^\circ, 17.7^\circ, 21.8^\circ, 28.2^\circ$  and  $33.5^\circ$  [24]. A similar peak reflections result was also observed elsewhere (Rodrigues [25]) in which Na-P<sub>1</sub> zeolite was synthesized from coal fly ash. According to Rodrigues, the X-ray diffraction analysis showed the presence of Na-P<sub>1</sub> zeolite in the synthesized sample as evidenced from reflections near  $2\theta = 12.4^\circ, 17.8^\circ, 21.7^\circ, 28.2^\circ, 33.3^\circ, 35.76^\circ, 38.01^\circ, 42.20^\circ$  and  $44.18^\circ$  [25]. It can be said in general that, the material synthesized showed the most prominent reflection peak at  $32.43^\circ$  indicating highly crystalline of the material. While the peaks positioned in  $34.88^\circ, 41.09^\circ$  and  $47.39^\circ$  may be zeolite A or  $35.05^\circ, 38.94^\circ$  and  $44.12^\circ$  zeolite Y and this result was also consistent with finding presented by Rodrigues [25]. The result obtained in this study showed that almost complete transformation of pumice into Na-P<sub>1</sub> zeolite of high purity.



**Fig. 4.** X-Ray diffraction (XRD) of Na-P<sub>1</sub> Zeolite

#### **Determination of Surface Area of Na-P<sub>1</sub> Zeolite Using Methylene Blue Adsorption**

Based on the spectra, the  $\lambda_{\max}$  of the methylene blue was 660 nm. The linear regression equation was  $y = 0.144x - 0.052$  with  $R^2 = 0.9972$ . This equation would be used to measure the remains concentration of the methylene blue that was unable adsorbed by the zeolite matrices. The absorbance measurement of the remnant of 50 mg/L of 1000 ml methylene blue after adsorption with 3 g Na-P<sub>1</sub> zeolite with time was.

The methylene blue adsorbed by zeolite matrices was used to interpret the surface area of the zeolite. The result of calculation of number of adsorbed methylene blue (x) per gram of adsorbent (m) versus time of contact (30, 40, 50, 60 and 70 min) was shown in Fig. 5. Data reported in Fig. 5 indicate that the optimum time of contact was 60 min (at 15 mg of methylene blue per gram adsorbent (Na-P<sub>1</sub> zeolite)). The optimum amount of methylene blue adsorbed (15 mg/g) could be used to calculate the surface area of adsorbent by using Eq. 3.

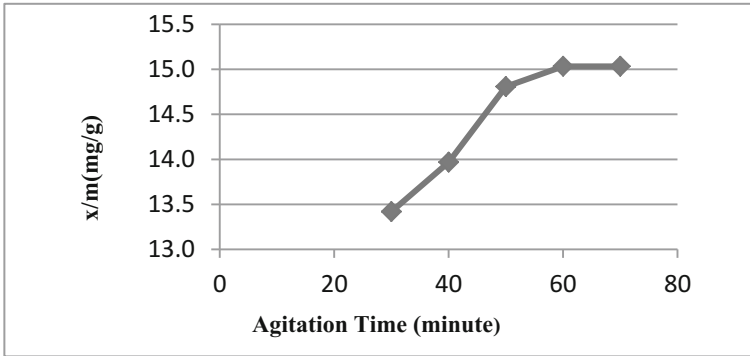


Fig. 5. Methylene blue adsorbed by Na-P<sub>1</sub> zeolite

$$S_A = \frac{W_m * N * A}{M} \tag{3}$$

W<sub>m</sub> = 15 mg/g or 15 mg of methylene blue per gram of adsorbent (Na-P<sub>1</sub> zeolite).

Where:

N = 6.022 \* 10<sup>23</sup> mol A = 197 \* 10<sup>-20</sup> m<sup>2</sup>/g; M = 320.5 g/mole

S<sub>A</sub> =  $\frac{15 * 10^{-3} * 6.022 * 10^{23} * 197 * 10^{-20}}{320.5} = 56.04 \text{ m}^2/\text{g}$ .

The surface area for the prepared zeolite material was found to be 56.04 m<sup>2</sup>/g. In other findings, the surface area of Na-P<sub>1</sub> was investigated using Brunauer-Emmett-Teller (BET), Surface area analyzer. Using BET, in the previous study by Dwivedi et al. (2016) the total surface area was found to be 60.36 m<sup>2</sup>/g. In another study, by Gitari et al. [26], total surface area for Na-P<sub>1</sub> zeolite was 67.63 m<sup>2</sup>/g.

### 3.2 Effects of Operating Conditions on Si/Al of Na-P<sub>1</sub> Zeolite

#### Effect of temperature on Si/Al ratio of Na-P<sub>1</sub> Zeolite

Increasing the reaction temperature leads to an increase in ratio of Si to Al (see Fig. 6).

This is because during zeolite synthesis, an increase in temperature results in an increase in silicon (Si) dissolution compared to aluminium (Al) dissolution from pumice [27]. Due to this Si/Al increases. But this increment continuous until no more silicon and aluminium present in the slurry. In this zeolite synthesis; 60, 80 and 100 °C and ratio of pumice to NaOH (1:1.2, 1:1.5, 1:1.8) were the factors. The outcomes showed that when the temperature increases from 60 °C to 80 °C and then to 100 °C, keeping other parameters constant (at reaction time of 70 min and ratio of pumice to NaOH 1:1.2), the ratios of Si to Al were in between 1.77 and 2.00. Similarly, for the same temperature variation and keeping other parametres constant (at reaction time of 100 min and ratio of pumice to NaOH of 1:1.2), the ratios of Si to Al were in between 1.80 and 2.18. This indicate that the increment of Si/Al ratios with variation of reaction temperature and at constant reaction time and ratio of pumice to sodium hydroxide is low (1.77–2.00) and (1.80–2.18). And even by increasing the reaction time but keeping the other the two parametres constant the Si/Al values were in range of 1.77 to 2.18

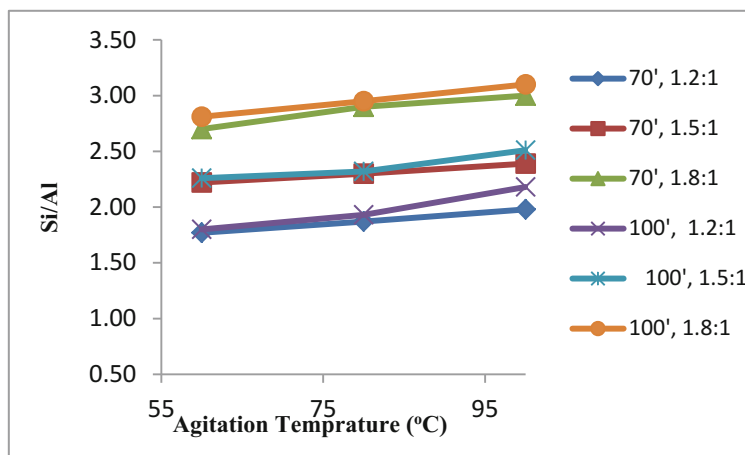


Fig. 6. Effect of temperature on Si/Al of Na-P<sub>1</sub> zeolite

which was also a small increment of Si/Al. This pointed out that varying reaction temperature and keeping the other two parameters constant, there was a small increase on the ratio of Si to Al. This implies that Si to Al ratio of zeolite obtained was highly affected by amount of sodium hydroxide when compared to effect of reaction temperature (Fig. 6).

#### Effect of Pumice to Sodium Hydroxide Ratio on Si/Al of Na-P<sub>1</sub> Zeolite

Increasing amount of sodium hydroxide also results an increase in ratio of Si to Al. Sodium hydroxide fusion decomposes silicon and aluminum from pumice and also acts as a mineralising agent, which depolymerises the silicon (Si) feedstock and forces the reactants into solution [28] and hence an increase in silicon. When the ratio of sodium hydroxide to pumice increase from 1.2:1 to 1.8:1 but at reaction temperature of 60 °C and reaction time of 70 min, the ratios of Si to Al increase from 1.77 to the values of 2.71. Similarly when reaction temperature increased to 100 °C but keeping reaction time at 70 min the Si/Al values were in range of 1.98 to 3.01. When the reaction time increased to 100 min at temperature of 60 °C, the ratios of Si to Al is found to be in between 1.8 and 2.82 (see Fig. 6). This implies that higher amount of sodium hydroxide were more likely to increase Si/Al than higher temperatures and longer reaction time.

#### Effect of Agitation Time on Si/Al of Na-P<sub>1</sub> Zeolite

Similar to the other parameters, increasing the reaction time leads to an increase in ratio of Si to Al. However, similar to that of reaction temperature, the rate of increase in ratio of Si to Al was slow when it was compared to an increase due to amount of NaOH (see Fig. 6). When the reaction time increases from 70 to 100 and then to 130 min, keeping other parameters constant (at reaction temperature of 60 °C and ratio of pumice to sodium hydroxide oxide of 1:1.2), the ratios of Si to Al were in between 1.77 and 1.85. These values (Si/Al) are close to each other. This implies that varying reaction time and keeping the other two parameters constant, there was no a large increase in values of Si

to Al ratio (see Fig. 6). A high increment of Si to Al ratio of zeolite was obtained when amount of sodium hydroxide was increased in addition to varying reaction time.

### 3.3 Effect of Na-P<sub>1</sub> Zeolite Doze on Sandy Soil Moisture Content

From Fig. 7, it can be seen that as exposure time increased, all sandy soil treatment samples lost their moisture through time.

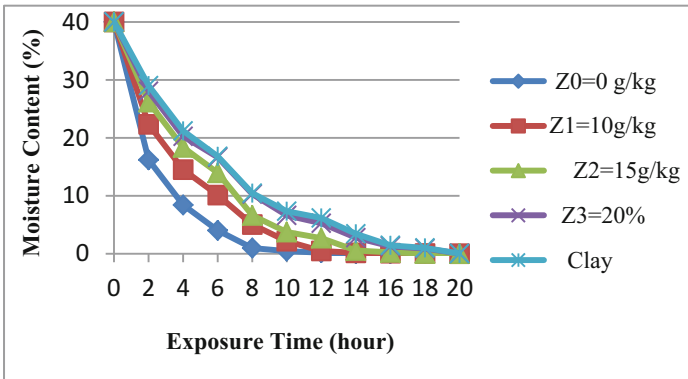


Fig. 7. Moisture content on sandy soil of particle size

However their rate of moisture loss was different depending on the amount of zeolite added to them. The results showed that as the dose of zeolite increased from 0 g/kg to 20 g/kg, the rate of moisture loss from sandy soil decreased. After 10 h, sandy soil without zeolite had mean moisture content of 0.45% and sandy soil with 10, 15 and 20 g/kg zeolite had mean moisture content of 2.04%, 3.58% and 6.48% respectively (see Fig. 7). The result obtained verified that greatest increase in moisture content in sandy soil was observed when 20 g/kg zeolite was used compared to the control other soil sample. In this study, effect of zeolite on sandy soil was in line with the findings of Tallai [29] that was aimed to compare effect of natural zeolite and bentonite on moisture content of sandy soil. The finding of Tallai stated that bentonite and zeolite have good water adsorption property [29]. It was, however, difficult to compare the findings of this paper with (Tallai [29]) since it used field experiment to test effect of zeolite on sandy soil whereas this paper was based on pot experiment. In this pot experiment, amount of zeolite was in g/kg in contrast the field experiment, zeolite was measured t/ha (amount of zeolite in ton per hectare). Due to this inconsistency of units, it was difficult to compare the findings of Tallai [29] quantitatively. Tallai [29] used natural zeolite to test moisture whereas this paper was based on Na-P<sub>1</sub> zeolite. Nevertheless, both this study and the finding of Tallai [29] showed that treatment of sandy soil with zeolite maintained higher soil water level than the control. According to Tallai, in small pot experiment the quantity of the available nutrients was increased by the two natural amendments. Since amount of nutrient in the soil depends on moisture content, this confirms that application of zeolite onto soil increases the moisture content.

Another research on the effect of clinoptilolite zeolite on typical agricultural soils of the U.S. Pacific Northwest was examined by Ippolito [30]. In this study, the effect of banding or fully mixing zeolite with N fertilizer on soil moisture content was investigated during a period of 6 weeks. The amount of zeolite added was measured in terms of Mg ha<sup>-1</sup> [30] where as in the present study, the amount of zeolite is measured in terms of g/kg and the duration was 20 h. Even though the units used to measure zeolite and the duration time is different for the two studies are different, there is an increment of moisture in sandy soil in both studies. According to Ippolito [30], mixing zeolite into a sand soil at 44.8 Mg ha<sup>-1</sup> increased soil moisture by 2.6% and 2.1% (by weight) as compared with a band application of 44.8 Mg ha<sup>-1</sup> or a control, respectively. In the present study, during 20 h, mean moisture content of the sandy soil for 20 g/kg was increased by 6% when compared to the control.

### 3.4 Effect of Doze of Na-P<sub>1</sub> Zeolite on Sandy Soil Water Retention Capacity

Effect of increasing zeolite dosage on water retention capacity of sandy soil was shown in Fig. 8. It can be seen that, using pressure plate extractor and applying 1, 3, 5 and 15 bars, as pressure increased, the water remained in the soil decreased.

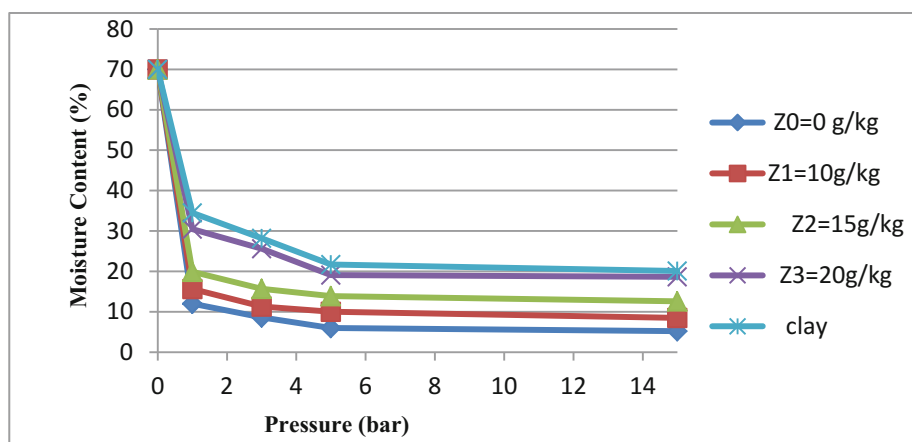


Fig. 8. Water retention capacity of sandy soil with zeolite at different bar

This implies that the water that was extracted from the soil increased as pressure increases. However, by adding zeolite onto sandy soil the water that can be retained in the soil could be increased. At 0 bar, all soil samples were observed to have the same water content. Then at 1 bar sandy soil without zeolite had a moisture content about 12% whereas sandy soil with 10 g/kg had 15.65%. As the amount of zeolite added increased from 10 g/kg to 15 g/kg and to 20 g/kg, moisture content in the soil increased from 15.65% to 19.90% and to 30.47% respectively. Again at 1 bar sandy soil with zeolite 20 g/kg had moisture content 30.47% which had no significant difference with clay soil (34.5%).

## 4 Conclusion and Recommendations

### 4.1 Conclusion

In summary, pure zeolite Na-P<sub>1</sub> has been successfully prepared from pumice. The silica to aluminium ratio of Na-P<sub>1</sub> zeolite obtained at reaction temperature of 100 °C, reaction time of 100 min and ratio of pumice to NaOH of 1:1.5 was 2.5. The FTIR analysis of pumice and Na-P<sub>1</sub> zeolite indicate the four vibration bands were found to be in ranges of expected vibrational wave number (cm<sup>-1</sup>). During pumice treatment there was disappearance of some mullite and quartz peaks as well as emergence of new peaks. The formation of Na-P<sub>1</sub> zeolite was observed through reflections on  $2\theta = 19.31^\circ, 23.44^\circ, 28.32^\circ, 32.43^\circ, 33.5^\circ$  and  $49.10^\circ$ . The surface area of Na-P<sub>1</sub> zeolite was 56.04 m<sup>2</sup>/g.

After synthesis and characterization, Na-P<sub>1</sub> zeolite was applied on to sandy soil to investigate moisture content and water retention capacity. Rate of moisture loss from soil samples decreased from soil samples due to the presence of zeolite. During water retention testing, applying 1 bar to the packed sandy soil without zeolite, moisture content about 12% was obtained whereas sandy soil with 10 g/kg had 15.65%. As zeolite dose increased from 10 g/kg to 15 g/kg and to 20 g/kg, moisture content in the soil increased from 15.65% to 19.90% and to 30.47% respectively. In general, as pressure applied increased, the extracted water that comes out of the soil increased. However, by adding zeolite to the sandy soil, the water that can be retained in the soil could be enhanced.

### 4.2 Recommendations

Based on the present study, pumice is suitable as raw materials for the synthesis of pure Na-P<sub>1</sub> type zeolite using NaOH as activating agent and an alkaline fusion step followed by hydrothermal treatment. Synthesis of Na-P<sub>1</sub> zeolite would require step-by-step optimization of the synthesis conditions in addition to reaction temperature, time and ratio of pumice to NaOH since Na-P<sub>1</sub> zeolite is sensitive and unstable so that it transforms into another zeolite (analcime). Further optimization of the synthesis conditions and adjustment of the Si/Al molar ratio of the synthesis gel by adding either SiO<sub>2</sub> or Al<sub>2</sub>O<sub>3</sub> from a supplementary source may be necessary if a pure phase of Na-P<sub>1</sub> zeolite targeted.

Besides small-pot experiment was used to investigate the effect of doze of Na-P<sub>1</sub> zeolite on moisture content of sandy soil. Field experiment has to be conducted to further explain the heterogeneity of the soil as well as to consider the actual environmental condition including the evaporation and transpiration rate of the soil and the plant.

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