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# Analysis of nitrogen adsorption capability at various activation temperatures of Klaten natural zeolite

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**Abstract**

The Pressure Swing Adsorption (PSA) method operates by passing air through an adsorbent to produce concentrated oxygen gas. Zeolites are commonly utilized as adsorbents due to their ability to adsorb nitrogen from the surrounding air. Two types of zeolites commonly employed are natural and synthetic zeolites. While the utilization of natural zeolites as adsorbents in oxygen purification remains limited, their potential as an alternative adsorbent is worth exploring in this field. This study focused on developing physically activated Klaten natural zeolite as an adsorbent to enhance oxygen purity. Physical activation involved heating for 1.5 hours using an electric oven at four temperature variations (250ºC, 300ºC, 350ºC, and 400ºC). Additionally, four distinct flow rates were tested: 0.1; 0.5; 1.0; and 1.5 lpm. Oxygen purification testing revealed that higher activation temperatures led to greater increases in oxygen concentration. The highest increase of 2.45% was achieved at an activation temperature of 400ºC, while the lowest increase of 1.75% was observed at 250ºC with a flow rate of 0.1 lpm. With a 10-minute holding period, oxygen content during the adsorption process ranged from 1.35% to 2.45%, compared to 0.60%-0.75% without holding. Physical activation of zeolite from Klaten enhanced its nitrogen absorption capacity, indicating the potential of natural zeolite from Klaten for oxygen purification through optimized activation processes, possibly via chemical activation.

**Keywords:**
Flow rate, Klaten natural zeolite, oxygen concentration, physics activation, PSA.

## 1 Introduction

The World Health Organization (WHO) declared Covid-19 a worldwide pandemic in 2020. Hospitals are still treating patients who have moderate to severe symptoms despitethe WHO has lifted the state of emergency related to the COVID-19 epidemic. Patients with these symptoms need breathing assistance with focused oxygen to increase their recovery time. The need for oxygen for medical treatment has only increased since then. Employing convenient, portable, efficient, and user-friendly oxygen-purifier equipment can help alleviate uneven oxygen delivery, particularly in isolated regions. The development of oxygen-purifier equipment, especially adsorbent materials, needs to be carried out to obtain alternative technologies and materials for purifying oxygen from the air.

Various methods used in the oxygen purification process include pressure vacuum swing adsorption, vacuum swing adsorption, pressure swing adsorption, crygenic distillation, membrane separation, and electrolysis[1]. A popular technique for oxygen purification is Pressure Swing Adsorption (PSA). Through circulating air over an adsorbent media, this process produces concentrated oxygen as the output gas, making it well-suited for air separation [2]. Because ambient air includes 21% oxygen, 78% nitrogen, and 1% other gases, it has the potential to be converted into concentrated oxygen [3]. Effectiveness, safety, and versatility are among the benefits of the PSA method, an air separation technology that may be used for both small- and large-scale production [4]. Based on the results of a journal review, the PSA method is proven to increase medical oxygen concentrations by up to 95% [5]. The pressure and flow rate of the entering air impact how oxygen purification devices function. Research conducted by Satria et al. and Bahari et al. regarding the separation of oxygen from air using the PSA method shows the results can produce oxygen concentrations of 40-70% [6], [7].

Activated carbon, zeolite, and silica gel are a few adsorbent media used in the PSA method to purify oxygen. Adsorbents absorb water, nitrogen, and other gases from the air, raising oxygen concentration. Silica gel is used to adsorb water vapor, silica gel is usually used also to maintain humidity in food, medicine, luggage, shoes, and electronics. Air filtration operations can benefit from activated carbon since it absorbs materials such as CO, $CO_2$, and particulate matter [8]. Meanwhile, zeolite is commonly utilized in the PSA method and can adsorb nitrogen from surrounding air.

Two varieties of zeolite are utilized: synthetic and natural. During the oxygen purification process, both can adsorb nitrogen. The application of natural zeolites as adsorbents for oxygen purification remains relatively uncommon. Synthetic zeolites such as zeolite A, zeolite X, and zeolite Y are more commonly used in the process of separating oxygen from air[9]. Prior research by Abdel-Rahman [10] concentrated on oxygen purification with synthetic zeolites 5A and 13X, obtaining up to 95% oxygen concentrations. However, the potential of natural zeolites as an alternative adsorbent is worth developing in this field. Naturalzeolite is widely available, inexpensive, and abundant in Indonesia, especially in Klaten, Central Java. This type of zeolite is frequently used in soil and water for the binding, chemical stabilization, and separation of inorganic compounds, hazardous organic chemicals, and radioactive species from soil and water[11]. Natural zeolite has high adsorptive qualities that help remove impurities from extracted petroleum products like sulfur, nitrogen, and heavy metals [12]. In this study, natural zeolite may also adsorb other gases during oxygen purification; it is the adsorbent of choice [13]. Natural zeolite can absorb $H_2O$, $CO_2$, and nitrogen from the surrounding air [14]–[17]. Klaten natural zeolite has so far been focused as an adsorbent for water purification[18]–[20]. However, no one has utilized it as an adsorbent for oxygen purification. Research on developing the potential of Klaten natural zeolite as an oxygen-purifying adsorbent can make a significant contribution in the future, especially for the needs of Indonesia.

Research on an oxygen purification device utilizing natural zeolite from Klaten that has been activated through physics by heating it between 250ºC-400ºC is imperative, considering the arguments above. The purpose is to evaluate the oxygen-purification potential of physics activated Klaten natural zeolite. The best adsorption periods and purity levels for each zeolite activation temperature are found in the study using the PSA method with flow rate adjustments.

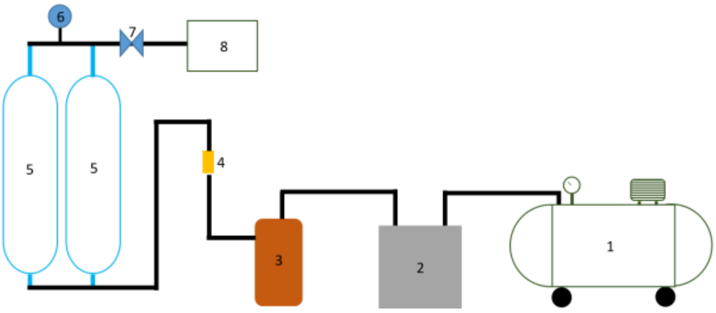
## 2 Materials and Methods

Raw natural zeolite from Klaten Regency in Central Java was the chosen material for this research (Fig. 1) and was used as an adsorbent. In order to attain a uniform size of between 30 and 50 mesh, the natural zeolite is crushed and ground before use. Natural zeolite isactivated through physics by heating for 1.5 hours using an electric oven at 4 temperatures (250ºC, 300ºC, 350ºC, and

400ºC). Aside from oxygen, physics activation aims to enhance the adsorption ability to capture $H_2O$ and other gases from the surrounding air.

Fig. 1. Klaten natural zeolite.

Fig. 2 shows the schematic of the oxygen purification apparatus used in this study. With an adsorption tank pressure of 5 bar, the oxygen purification system uses the Pressure Swing Adsorption (PSA) method of operation. Four different flow rates are used in the study: 0.1, 0.5, 1.0, and 1.5 lpm.




Descriptions:

| | |
|---|---|
| 1. Compressor | 5. PSA cylinder |
| 2. Air dryer | 6. Pressure gauge |
| 3. Water trap | 7. Valve |
| 4. Flow meter | 8. KE 25 sensor |

Fig. 2. Oxygen purification apparatus scheme.

Using a compressor, the evaluation procedure started with the absorption of ambient air. Subsequently, the compressor's valve was opened to allow outside air into the air dryer and water trap. These steps reduced the moisture content in the surrounding air. Afterward, the air with reduced moisture passed through a coarse air filter to remove any debris or dirt it may have carried. Following the initial filtering process, the air was directed towards the PSA cylinder, where the flow rate was adjusted to achieve the desired variation until the pressure gauge indicated 5 bar pressure. To optimize the adsorption process of nitrogen and other gases using physically activated natural zeolite, the air inside the PSA cylinder was held for 10 minutes. Once the airflow generated had decreased, the air injection procedure was repeated to push the remaining air into the PSA cylinder. The produced oxygen was then directed by opening the PSA valve, and its concentration was measured using a KE 25 sensor. Data on oxygen concentration were collected every minute over a period of 20 minutes.

The KE 25 sensor is a specialized galvanic cell-type oxygen sensor that provides an output voltage linear to the percent presence of oxygen in the atmosphere [21]. The sensor is widely used for medical, biotechnology, food industry, and safety applications. In the medical field this sensor is used to measure oxygen concentration and flow in ventilators [22], [23]. The KE 25 sensor can optimally detect oxygen at an air pressure of 811hPa-1216hPa with a temperature range of 5°C-40°C in a relative humidity of 10-90%RH without condensation. Its response time to reach 90% oxygen detection is only 14 seconds. The design diagram of the oxygen concentration measuring instrument is shown in Fig. 3.

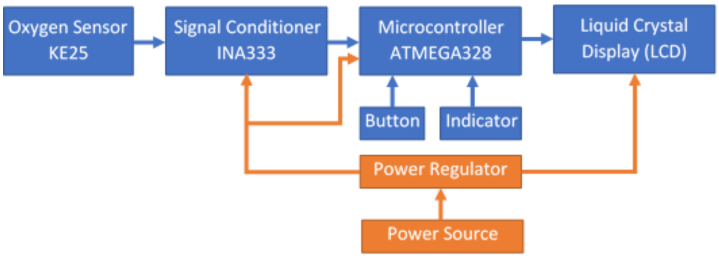



Fig. 3. Design diagram of the oxygen concentration measuring instrument.

## 3 Results and Discussion

In this investigation, the morphology of the zeolite was used to examine its properties. Scanning Electron Microscopy (SEM) examination was used to observe surface morphology. Fig. 4 shows the properties of natural zeolite from Klaten before and during physical activation.

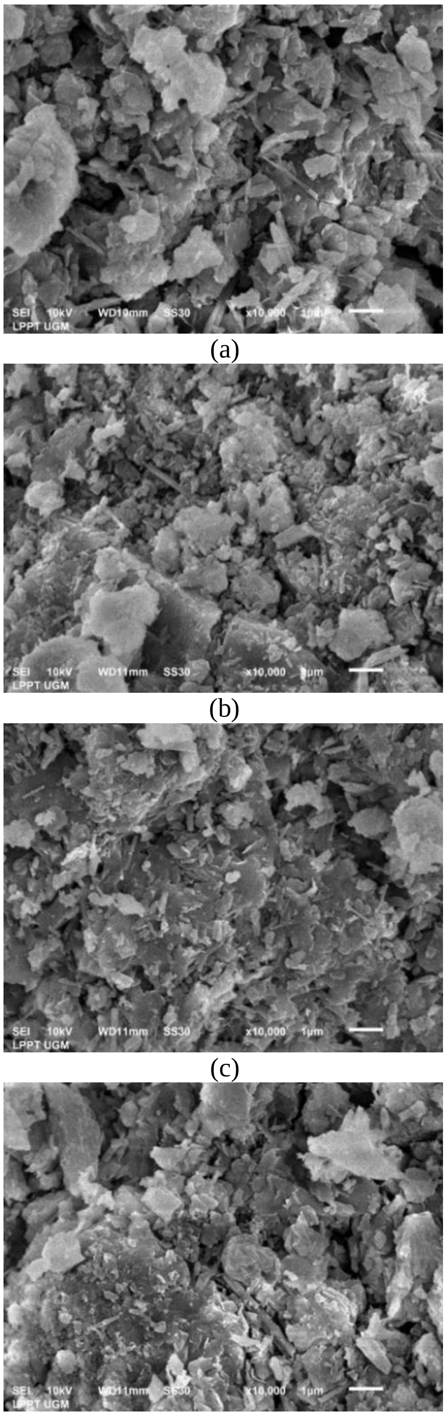



Fig. 4. The results of SEM testing on Klaten natural zeolite at various activation temperature variations (a) 250ºC (b) 300ºC (c) 350ºC (d) 400ºC.

The surface morphology of activated natural zeolite at 250ºC is shown in Fig. 4(a), where the surface is uneven and heterogeneous. The surface morphology of physics activated natural zeolite at temperatures of 300ºC and 450ºC is depicted in Fig. 4(b) and Fig. 3(c); both variations have surface morphological similarities. The zeolite surface, with a few small cavities, seems smoother at these temperatures. At 400ºC, the surface morphology of activated natural zeolite is shown in Fig. 4(d), which exhibits a more uniform, smooth surface with visible voids. It is assumed that the release of bound water content within the zeolite particles is the cause of this phenomenon. Krol and Jelen's [24] study into the effects of physical activation on zeolite A's structure, zeolite can undergo structural alterations due to heating between 25ºC-600ºC, which can remove water and bound water content. This indicates that heating may improve zeolite's surface uniformity and enlarge its pore structure [25].

The research on oxygen purification conducted using the Pressure Swing Adsorption (PSA) method with physically activated natural zeolite from Klaten as an adsorbent resulted in the data. Fig. 5 shows the oxygen concentrations generated during adsorption for various zeolite sample variations.

Fig. 5 shows that the oxygen concentration in ambient air before the purification process is 18.5%. The average peak oxygen concentration in the outflow from the PSA cylinder is 20.34%. The highest peak oxygen concentrations obtained for each zeolite variation are 20.35%, 20.60%, 20.65%, and 21.05% for activation temperatures of 250ºC, 300ºC, 350ºC, and 400ºC, respectively. Fig. 4 indicates that the oxygen concentration increases at t = 6 minutes, reaching its highest level at t = 10 minutes. The test data shows oxygen concentration increases after passing through the PSA cylinder. This phenomenon is presumed to occur because the air that initially exits has a lower oxygen content. Air with lower oxygen content is lighter and tends to be positioned at the top of the PSA cylinder, exiting first when the valve is opened. Research conducted by Al-Shawabkeh et al. [26] also indicates that the peak oxygen concentration is reached in the middle of the adsorption cycle and decreases at the end.

Furthermore, shown in Fig. 5, the oxygen concentration stabilizes at the end of the adsorption cycle. This happens because the injection process starts at minute 13. By minute 12, the PSA cylinder's airflow diminishes, requiring the injection procedure. Following minute 13, the oxygen concentration indicates the amount of oxygen in the air because of the adsorption process without the holding procedure. The PSA technique with holding procedure yields increases oxygen concentration from 1.35 percent to 2.5 percent. Conversely, the rise in oxygen concentration attained is about 0.60% to 0.75% if the adsorption procedure is carried out without holding.

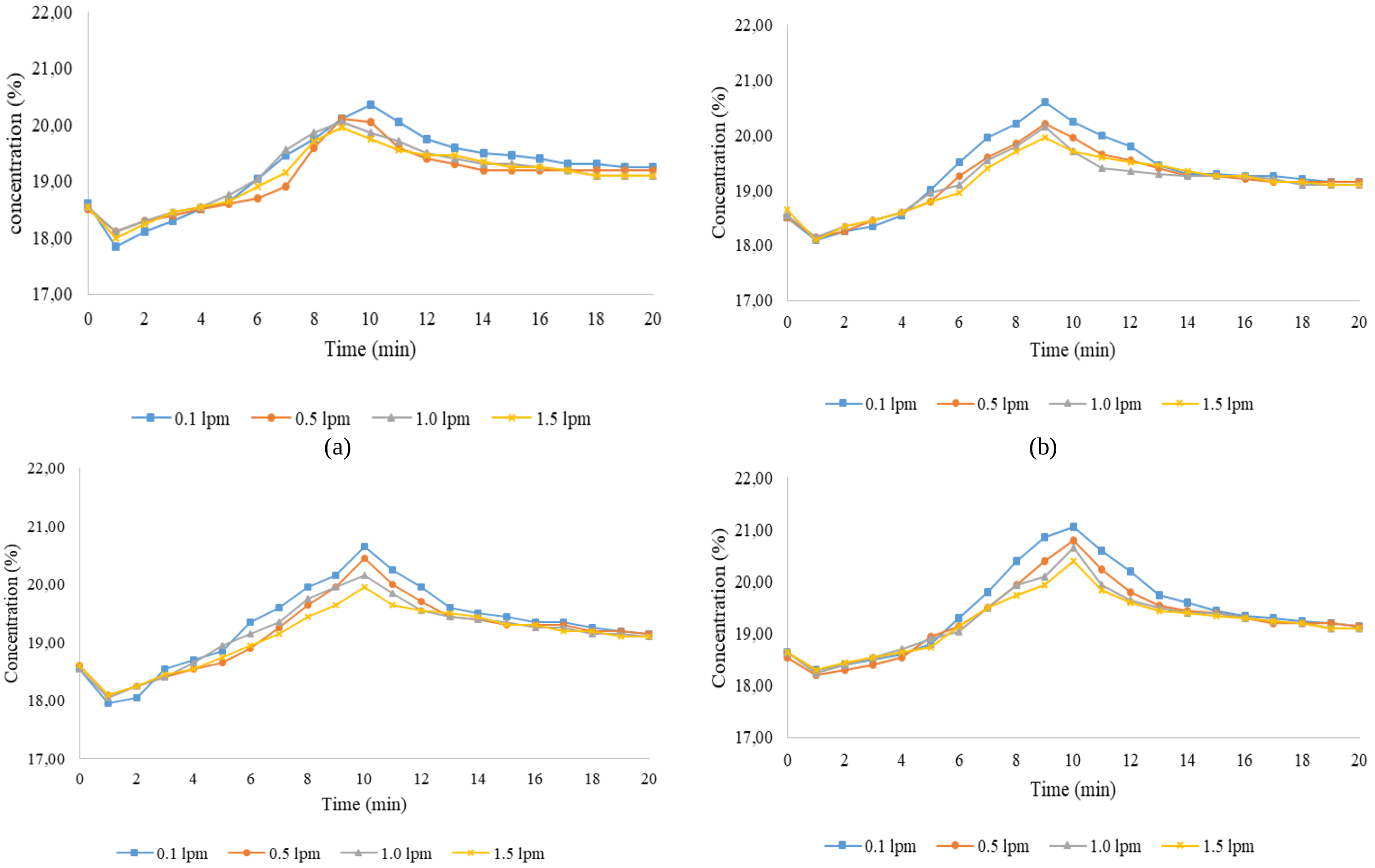



Fig. 5. Oxygen concentration in the PSA process with various zeolite activation temperature variations (a) 250ºC (b) 300ºC (c) 350ºC (d) 400ºC.

Table 1 shows the outcomes of evaluating the oxygen concentration using physics activated natural zeolite from Klaten and the PSA method. According to the test results, a flow rate of 0.1 ppm produced the most significant percentage rise in oxygen concentration. All four of the activation temperature changes that are used exhibit this effect.

Table 1 shows that oxygen concentration increases significantly at low flow rates. For activation temperatures of 250ºC, 300ºC, 350ºC, and 400ºC, respectively, the oxygen concentration rise is measured at 1.75%, 2.00%, 2.05%, and 2.45% at a flow rate of 0.1 lpm. This happens because nitrogen and other gases adsorb more optimally at lower airflow rates than oxygen. According to research by Irvan et al. [27] and Kottitum et al. [28], operating PSA devices at low flow rates also led to greater oxygen concentrations. This is explained by the prolonged contact time in the adsorption column between the gas and the adsorbent, which gives the gas molecules enough time to diffuse into the adsorbent's pores. The heating conditions of natural zeolite during the activation phase also have an impact on increasing oxygen concentration. An increase in oxygen concentration is proportional to higher activation temperatures. The heating procedure can improve the natural zeolite's adsorption capacity [16].

Table 1. The percentage increase in produced oxygen concentration.

| Flow rate | Temperature (ºC) | | | |
|---|---|---|---|---|
| (lpm) | 250 | 300 | 350 | 400 |
| 0.1 | 1.75 | 2.00 | 2.05 | 2.45 |
| 0.5 | 1.50 | 1.60 | 1.85 | 2.20 |
| 1.0 | 1.45 | 1.55 | 1.55 | 2.05 |
| 1.5 | 1.35 | 1.35 | 1.35 | 1.80 |

Although the results showed that the efficiency of oxygen purification using Klaten natural zeolite was not yet optimal, with oxygen concentration still low, an interesting finding from this study was the ability of Klaten natural zeolite to increase oxygen concentration. This shows significant potential in the use of Klaten natural zeolite as an adsorbent in the oxygen purification process. Therefore, although the current results have not reached the desired level, further research and development of oxygen purification devices, especially adsorbent materials, can be carried out to maximize the potential of Klaten natural zeolite in improving the quality of oxygen purification.

## 4 Conclusion

The following are the results from the testing of oxygen purification employing naturally occurring zeolite from Klaten that was physically activated using the Pressure Swing Adsorption (PSA) method:

1. Utilizing physically activated natural zeolite from Klaten at 400ºC yielded the highest increase in oxygen concentration, reaching 2.45%. At an activation temperature of 250ºC, a lower increase of 1.75% was observed. These findings were based on a flow rate of 0.1 lpm.
2. The PSA technique with holding increases the oxygen concentration by 1.35% to 2.45%. On the other hand, the rise in oxygen concentration that results from doing the adsorption process without the holding procedure is approximately 0.65% to 0.75%.
3. Drawing from the outcomes of this investigation, it is evident that physically activated natural zeolite exhibits potential as an adsorbent material. However, to enhance its adsorption efficacy, additional procedures such as chemical activation are deemed necessary.

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