CHAPTER III

RESEARCH METHODOLOGY

3.1 Preparation

3.1.1 Chemicals, equipment, and characterization

Table 3.1 Chemicals, Chemical formula

Chemicals	Chemical formula	Figure
Ball clay	$Al_2Si_2O_5(OH)_4$	 Figure 3.1
Ranong white clay	$Al_2Si_2O_5(OH)_4$	 Figure 3.2

Table 3.1 Chemicals, Chemical formula (Continued)

Chemicals	Chemical formula	Figure
Lampang white clay	$Al_2Si_2O_5(OH)_4$	 Figure 3.3
Sodium hydroxide	NaOH	 QReC Figure 3.4
Lithium hydroxide Monohydrate 99.5%	LiOH	 Loba Chemie Figure 3.5

3.1.2 Equipment

Table 3.2 Research Equipment

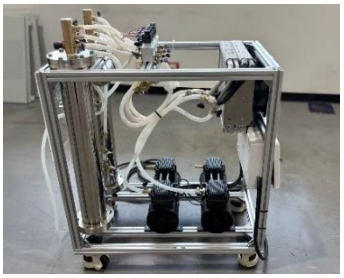
Equipment	Figure
Oxygen concentrator	 Figure 3.6
Oxygen gas detector	 Figure 3.7
Magnetic stirrer and Magnetic bar	 Figure 3.8

Table 3.2 Research Equipment (Continued)

Equipment	Figure
Furnace	 Figure 3.9
Oven	 Figure 3.10
Alumina crucible	 Figure 3.11

Table 3.2 Research Equipment (Continued)

Equipment	Figure
Mortar	 Figure 3.12
Spray dryer	 Figure 3.13
Deionization water	 Figure 3.14

Table 3.2 Research Equipment (Continued)

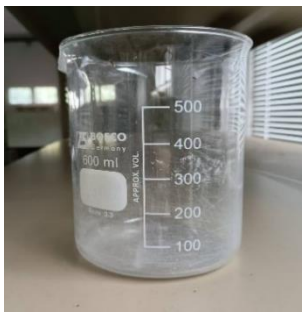
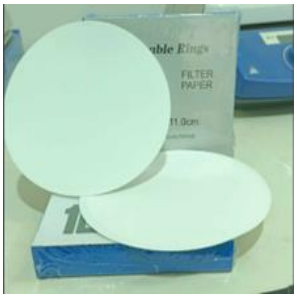
Equipment	Figure
Beaker	 Figure 3.15
Buchner Funnel	 Figure 3.16
Erlenmeyer Flask	 Figure 3.17

Table 3.2 Research Equipment (Continued)

Equipment	Figure
Filter paper	 Figure 3.18
litmus paper	 Figure 3.19

3.1.3 Characterization techniques

Table 3.3 Characterization techniques

Characterization techniques	Figure
X-ray Diffractometer: XRD (D2 PHASER)	 Figure 3.20
Scanning Electron Microscope/Energy Dispersive X-ray Spectroscopy: SEM-EDS (JEOL JSM-6010LV)	 Figure 3.21

Table 3.3 Characterization techniques (Continued)

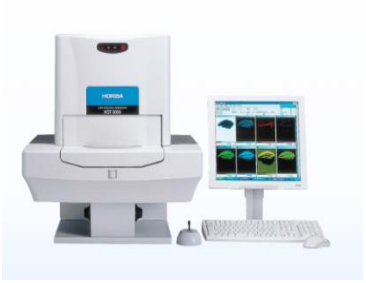
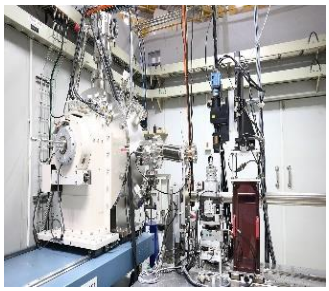
Characterization techniques	Figure
Thermogravimetric Analyzer: TGA (Mettler Toledo TGA/DSC1)	 Figure 3.22
BET Surface Area Pore Size and Pore Volume Distribution Analyzer micrometric (ASAP2020P)	 Figure 3.23
Energy Dispersive Spectrometer X-ray Fluorescence (ED-XRF) (Horiba/XGT-5200)	 Figure 3.24

Table 3.3 Characterization techniques (Continued)

Characterization techniques	Figure
Fourier Transform Infrared Raman Spectrophotometry (FT-IR-Raman/Vertex 70)	 Figure 3.25
X-ray Tomographic Microscopy (XTM)	 Figure 3.26

3.2 Experimental flow chart

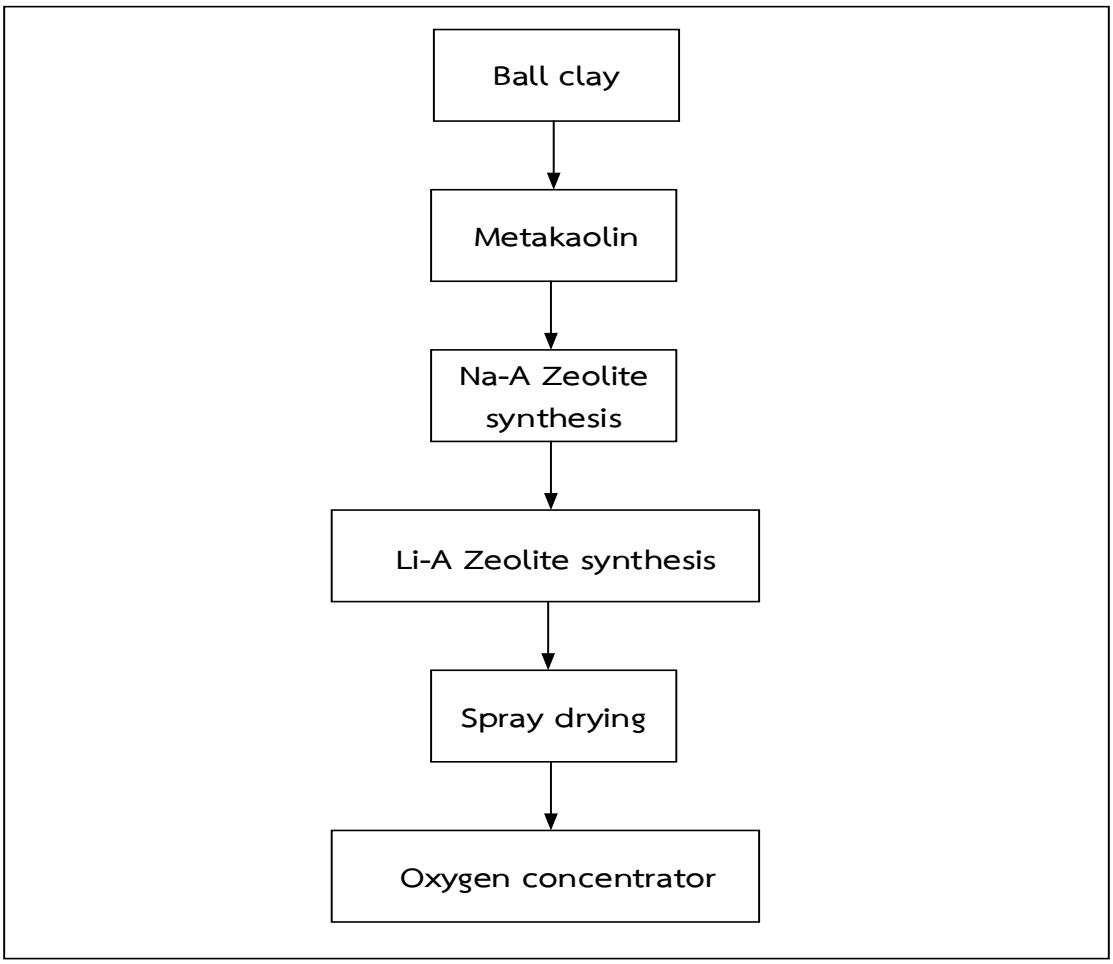


Figure 3.27 Experimental flow chart

The synthesis of Li-A zeolite from ball clay for medical oxygen production involves a sequential process beginning with the thermal pretreatment of the raw material. Initially, Ball clay is calcined to produce Metakaolin, which acts as the reactive aluminosilicate precursor. This Metakaolin is then subjected to a hydrothermal treatment with a sodium hydroxide solution to synthesize Na-A zeolite through a crystallization process. Following this, an essential ion-exchange step is performed,

where the the synthesized Na-A zeolite is treated with a lithium hydroxide solution to replace the sodium ions with lithium ions, yielding the final adsorbent, Li-A zeolite. Finally, the Li-A zeolite slurry is prepared for industrial application via spray drying, a technique that converts the slurry into free-flowing, spherical particles with controlled size and morphology. These optimized particles are then loaded into a pressure swing adsorption (PSA) system, forming the core component of the oxygen concentrator for efficient air separation by selectively adsorbing nitrogen.

3.3 Na-A zeolite synthesis

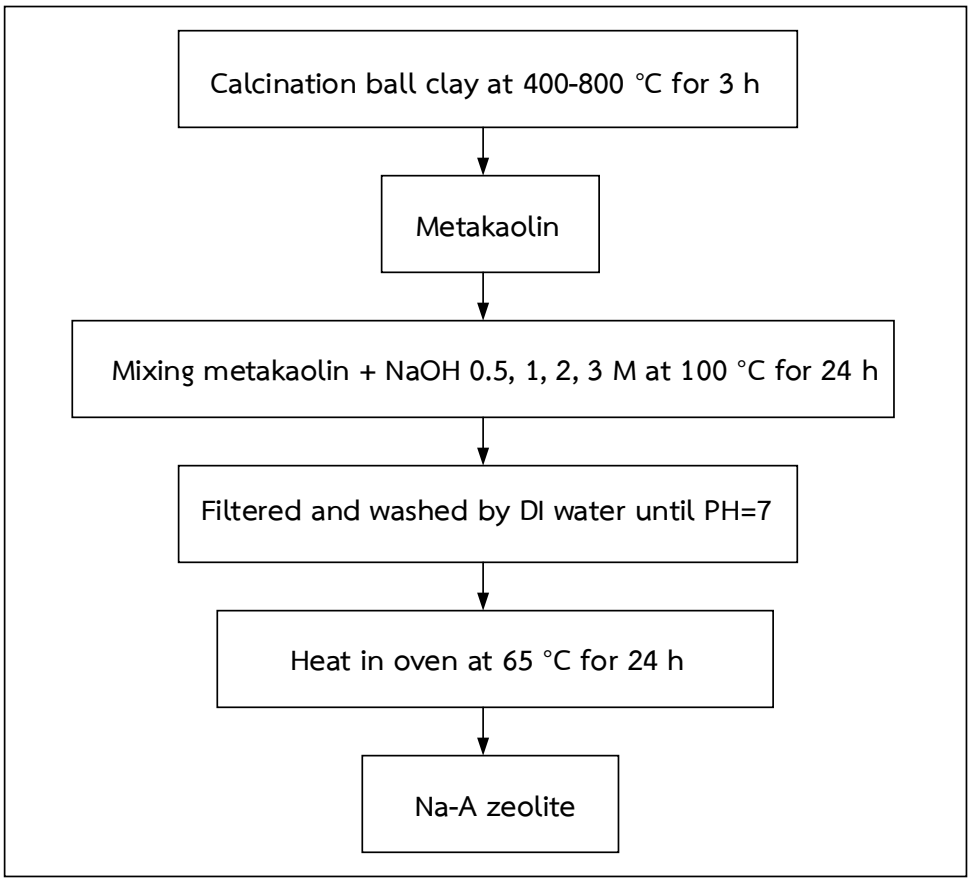


Figure 3.28 Flow chart for Na-A zeolite synthesis

The process for synthesizing Na-A zeolite from ball clay consisted of two fundamental steps: calcination and hydrothermal treatment. During calcination, the ball clay was heated at temperatures of 400, 500, 600, 700, and 800 °C for 3 h. This thermal treatment initiated a structural transformation within the ball clay, resulting in the formation of a metastable phase. Following calcination, the metastable phase samples underwent hydrothermal treatment with an aqueous NaOH solution to form Na-A zeolite. 20 g of ball clay was mixed with 100 mL of NaOH solutions at various concentrations (0.5, 1, 2, and 3 M) at 100 °C for 24 h. Subsequently, the samples were filtered and washed with deionized water until a neutral pH was achieved, thereby removing excess NaOH. Finally, they were dried in an oven at 65 °C for 24 h.

3.4 Li-A zeolite synthesis

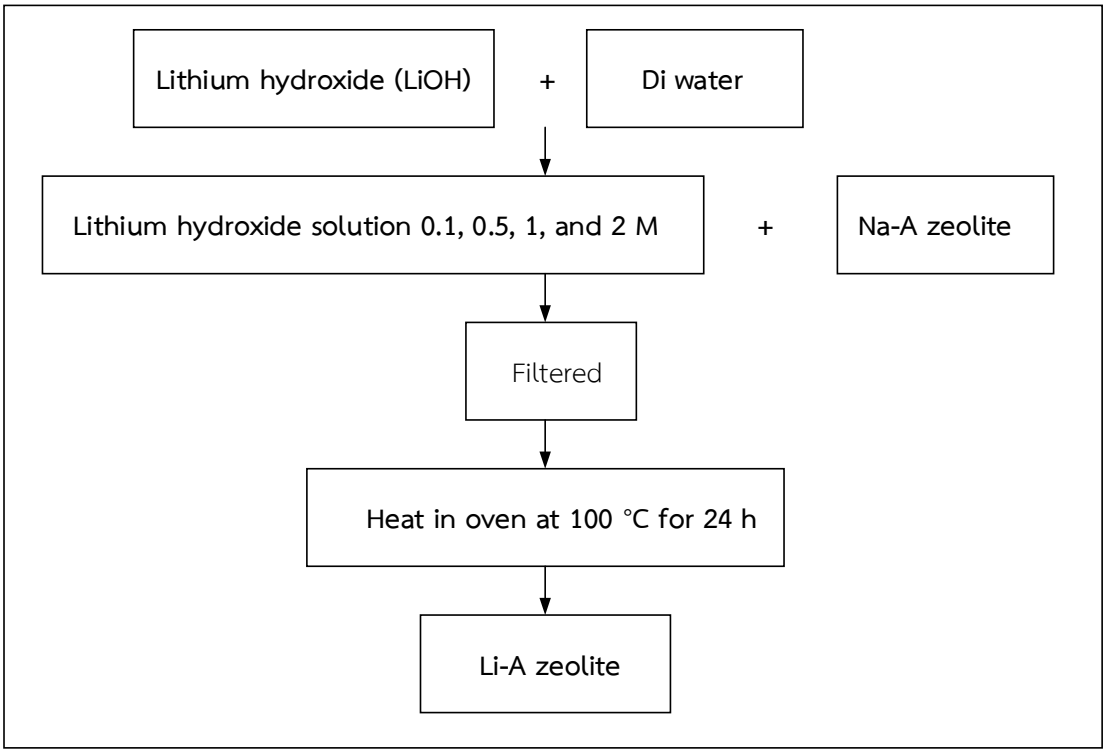


Figure 3.29 Flow chart for Li-A zeolite synthesis

In this experiment, varying amounts of LiOH powder were added to distilled water to prepare solutions with different concentrations of 0.1, 0.5, 1, and 2 M. Then, 50 g of Na-A zeolite was added to 1000 mL of LiOH solution at room temperature, with continuous stirring using a magnetic stirrer for 24 h. The samples were then filtered and dried overnight.

3.5 Spray Drying

The Li-A zeolite slurry was prepared by mixing 50 g of Li-A zeolite with 100 mL of deionized water as a solvent. The Li-A zeolite was thoroughly mixed with a magnetic stirrer and then ball-milled for 24 h. The spray drying process was performed using a spray dryer operated with a maximum inlet temperature of 220 °C, an air flow rate of 35 m^3/h, slurry feed rate of 15 ml/min, and spraying air flow rate of 357 L/h. The Li-A zeolite slurry was continuously mixed during the spray drying using a magnetic stirrer. Moreover, fluid nozzles with diameters of 450 μm were used in this study.

3.6 The efficiency of Li-A zeolite synthesized as a molecular sieve in a Pressure swing adsorption (PSA) oxygen concentrator

Medical oxygen concentrators are devices that generate high-purity oxygen by selectively eliminating nitrogen from the air. Natural zeolites are effective adsorbents in these devices thanks to their unique physical and chemical properties. The effectiveness of nitrogen absorption in natural zeolites is closely associated with their specific attributes. A significant factor that impacts nitrogen-selective absorption is the distribution of pore sizes within the zeolites. By customizing the uniformity of these pore sizes, zeolites can be aligned with the dimensions of nitrogen molecules, enabling efficient adsorption while excluding other gases. Thus, refining the pore-size distribution is essential for selective nitrogen capture. Moreover, the ion-exchange

capacity of natural zeolites is crucial to their function in medical oxygen concentrators. Zeolites can exchange ions with their environment, thereby enhancing their nitrogen adsorption capacity. By replacing specific ions in the zeolite with others that have a greater affinity for nitrogen, the material can become more efficient at capturing nitrogen gas. In addition to these factors, the surface area and porosity of natural zeolites are significant for their nitrogen-selective absorption performance. A greater surface area and increased porosity provide additional sites for nitrogen molecules to adhere to the zeolite, improving both the capacity and selectivity of the adsorbent. In conclusion, natural zeolites are highly valuable adsorbents for medical oxygen concentrators due to their pore size distribution, ion-exchange capacity, surface area, and porosity. Optimizing these characteristics can improve nitrogen-selective absorption efficiency. By understanding these properties, researchers and engineers can develop more effective medical oxygen concentrators using natural zeolites. Valuable insights can be gained from research aimed at developing more efficient oxygen-selective adsorbents and optimizing related methods for medical oxygen concentrators. Initial studies recognized that the oxygen-selective trait of various zeolites was due to the lower quadrupole moment of O_2 compared to N_2. In parallel with process research conducted by engineers, chemists, and materials scientists focused on adsorbents, a comparison of the numerous properties of well-characterized synthetic and natural zeolites can offer substantial learning opportunities. Collaborating, materials scientists and process engineers can likely achieve greater success than working independently in developing cost-effective, high-quality oxygen concentrators. However, selecting the appropriate natural zeolite for oxygen separation can pose challenges, and cost and quality considerations must always be kept in mind. Li-A zeolite synthesized from ball clay is employed in this study to capture N_2 in molecular sieve beds with a diameter of 98.20 mm and 649 mm. The Li-A zeolite prepared samples with several grain sizes (450 μm, 550 μm, and 650 μm) were prepared in the laboratory.

The compressed air moves to the sieve bed filters, which play a crucial role in removing nitrogen from the air. Inside the sieve bed, a material called zeolite, shaped like a six-sided microscopic cube with holes on each side, is responsible for this nitrogen removal.

There are two sieve beds located in the concentrator. After the air is first compressed, it is directed into the first sieve bed, where oxygen is separated and sent to the product tank. As a result, the first sieve bed becomes filled with nitrogen. In addition, the gas flow is switched, and the compressed air is directed to the second sieve bed. The compressor associated with the first sieve bed is vented to the outside, while the air from the product tank is returned to the first sieve bed.

As pressure drops in the first sieve bed and oxygen concentration decreases, the zeolite releases the trapped nitrogen. The released nitrogen recombines with the oxygen and is vented back into the room as regular air. The air is then compressed again and sent to the second sieve bed, where oxygen is once more separated and directed to the product tank. This entire cycle repeats itself every few seconds, starting again with the first sieve bed.