PYROLYSIS OF RUBBER SEED OIL USING ZEOLITE-A SYNTHESIZED FROM PUMICE SILICA AND FOOD GRADE ALUMINIUM FOIL AS CATALYST

(Skripsi)

By

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ABSTRACT

PYROLYSIS OF RUBBER SEED OIL USING ZEOLITE-A SYNTHESIZED FROM PUMICE SILICA AND FOOD GRADE ALUMINIUM FOIL AS CATALYST

By

Mohammad Rizky Arpan Zein

In this research, a pyrolysis study was conducted on a rubber seed oil using zeolite-A synthesized by hydrothermal method using food grade aluminium and amorphous silica extracted by sol-gel method using pumice. The extraction was performed using variations of NaOH concentration 2.5 M and 4 M. Characterization with XRD indicates that the silica extracted using NaOH concentration of 2.5 M is amorphous phase, while the silica extracted using NaOH concentration of 4 M is crystalline phase. Characterization using XRF indicates that the amorphous silica contains 81.41% SiO_2 and 10.29% Al_2O_3**.** Characterization using FTIR indicates the presence of siloxane bond (Si-O-Si) and silanol bond (Si-OH). The synthesized of zeolite-A was performed using variations of distilled water volume 150, 200, 250, 300 and 430 mL. Characterization using XRD indicates that only the synthesized using 430 mL distilled water contain zeolite-A. Before use, the zeolite-A were calcined at different temperatures of 600, 700 and 800 ℃. The first pyrolysis process was performed with and without silica sand as heat exchanger using 250 mL of rubber seed oil to produce BCO. Then, the BCO from first pyrolysis with silica sand was used to the second pyrolysis with zeolite-A as catalyst and 100 mL of BCO to produce upgraded fuel. The GC-MS analysis result show that the optimum zeolite-A catalyst was obtained at temperature of calcination of 600 ℃ with relative amount of biogasoline is 30.61%, while zeolite-A calcined at temperatures 700 and 800 ℃ has relative amount of biogasoline 22.27% and 29.36%.

Keywords: pyrolysis, pumice, amorphous silica, zeolite-A, rubber seed oil, BCO.

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Skripsi

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At

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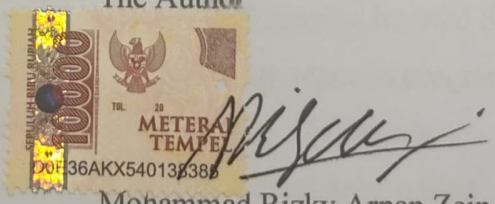
Truly declare that my final project report entitled **"Pyrolysis of Rubber Seed Oil Using Zeolite-A Synthesized from Pumice Silica and Food Grade Aluminium Foil as Catalyst"** is truly my own work, both the ideas, results, and analysis. Furthermore, I also have no objection if some or all of the data in this final project report are used by the lecturer or department for publication purposes, as long as my name is mentioned and there is an agreement prior to publication.

Bandar Lampung, 15 August 2023
The Author



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The author's full name is Mohammad Rizky Arpan Zein. The author was born in Samarinda, on September 7th 2001. The author is the first daughter of Mr. Mohammad Sukri and Mrs. Nurhasanah. The author lives in Long Ikis district, Paser, East Borneo.

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MOTTO

"Allah is kind, and he loves kindness in all matters."

(Bukhari 6528)

.

This scientific work is dedicated entirely to the people I love:

My beloved mother
Mrs. Nurhasanah

My beloved brothers and sisters
Ayu Rizky Amalia
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Muzein Nur Rahman Nihan
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The author realizes that in the process of working and writing this skripsi is inseparable from the difficulties and obstacles that the author faces. But it can all be passed thanks to the grace and pleasure of Allah SWT as well as help, direction, and guidance from various parties. Therefore, on this occasion as a form of respect, the author would like to thank you:

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The author realizes that in writing this skripsi there are still some imperfections, but the author hopes that this skripsi can be useful for the development of science in the future.

Bandar Lampung, 15 August 2023
The Author

Mohammad Rizky Arpan Zein

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I. INTRODUCTION

1.1 Background

Indonesia has abundant natural resources, among them is pumice (Hasanah et al., 2019). Pumice is spread in various regions in Indonesia such as Maluku (Gaus et al., 2019), Nusa Tenggara Barat (Ratnawati et al., 2020), Kawah Ijen (Lowenstern et al., 2018), and Lampung (Manurung et al., 2022). In Lampung Province, data related to pumice resources has not been widely published. However, based on a map of the potential of non-metallic mineral resources in Lampung Province, issued by the Directorate Inventory of Mineral Resources, there is potential resources of pumice in South Lampung, Kalianda district (Isnugroho et al., 2020). Pumice is a volcanic rock containing silica (SiO_2), alumina (Al_2O_3) as primary components with relative amount of 48% and 14.9%, respectively (Mourhly et al., 2015). Other studies revealed that silica of pumice is mostly amorphous, with the quantity may reach 71.89% (Azhar et al., 2020), suggesting that pumice is a potential source of amorphous silica.

Amorphous silica is soluble in alkaline solution, and for this reason, alkali extraction has been applied to extract amorphous silica from different sources, such as rice husk (Simanjuntak et al., 2021), sugarcane bagasse ash (Rilyanti et al., 2020), and sand (Silahooy, 2020). In this extraction method, silica dissolved in alkaline solution (silica sol) can be converted into silica gel by neutralization of the sol using acid. By drying of the gel, the solid silica is produced.

Another interesting feature of amorphous silica is wide use as raw material for production of various applied materials such as various type of zeolite (Prajaputra et al., 2019).

Synthetic zeolite is a class of aluminosilicate compounds with different compositions, primarily the Si/Al ratio. Many synthetic zeolites have been developed, such as zeolite-X (Febriyanti et al., 2021), zeolite-Y (Pangesti et al., 2021), and zeolite-A (Simanjuntak et al., 2021). Zeolite-A is one of great importance because of the small aperture of about 4 Å, and high molar ratio of aluminum to silicon (~1) (Covarrubias et al., 2005; Lei et al., 2016). Zeolite-A has potential to use as adsorbent, it is widely applied for heavy metal removal such as lead (Pb), cadmium (Cd), zinc (Zn), calcium (Ca) and copper (Cu) (Jangkorn et al., 2022). Zeolite-A also has potential as heterogeneous catalyst, such as for biomass pyrolysis (Simanjuntak et al., 2019) and biodiesel (Pandiangan et al., 2019).

In this study, zeolite-A was synthesized by hydrothermal method using amorphous silica from pumice and food grade aluminium foil as raw materials. Zeolite-A was then used as catalyst for catalytic upgrading of bio crude oil (BCO) produced from pyrolysis of rubber seed oil.

Pyrolysis is thermal cracking of large organic molecules at 400-550 °C into smaller molecules, in which one of them is liquid product generally known as bio crude oil (BCO). Pyrolysis is one of the methods of converting biomass into fuel, with several advantages such as simple process, the application of the resulted fuels for various applications, and the opportunity to improve the quality of the fuel (Mortensen et al., 2011; S. Wang et al., 2017). In pyrolysis, catalyst was used for lowering the pyrolysis temperature and chemical composition of BCO produced (Demiral and Şensöz, 2008; Simanjuntak et al., 2021).

In this study, rubber seed oil was selected considering its availability in Indonesia. In addition, rubber seed oil is non-edible vegetable oil and therefore no competition with food industry.

1.2 Research Objectives

The purpose of this study is:

1. Produce zeolite-A from pumice silica and aluminum foil by hydrothermal method.
2. Obtaining data on the characterization of synthesized zeolite.
3. Obtaining data on the composition of biogasoline resulting from the pyrolysis process of rubber seed oil with synthesized zeolite as catalyst.

1.3 Research Benefits

Apart from being an effort to enrichment of science, preparation of synthetic zeolite and biomass pyrolysis in particular, this research is also useful to increase economic value of pumice and seed oil for production renewable fuel.

II. LITERATURE REVIEW

2.1. Silica

Silica can be found as crystalline and amorphous material. Amorphous silica is more reactive than the crystalline one, and highly soluble in alkaline solvent. Due to this solubility, amorphous silica can be obtained by sol-gel method (Okoronkwo et al., 2013). This technique has been applied to obtain amorphous silica from various resources such as as rice husk (Simanjuntak et al., 2021), sugarcane bagasse ash (Rilyanti et al., 2020), sand (Silahooy, 2020), and pumice (Manurung et al., 2022).

Because of its reactivity, amorphous silica can be converted into different products, such as silica sols, silica gels, silica deposits, pyrogenic silica (Sapei et al., 2015) and synthetic zeolites including zeolite-A. In this study, zeolite-A was synthesized from amorphous silica extracted from pumice silica and food grade aluminium and the zeolite produced was then applied as catalyst for upgrading of BCO produced by pyrolysis of rubber seed oil.

2.2. Zeolite-A

Zeolite-A has a molecular formula of $Na_2O.Al_2O_3.2SiO_2.4,5H_2O$. This zeolite is porous material composed of three-dimensional aluminosilicates that are perpendicular to each other in the x, y, and z planes to form eight-member oxygen ring with diameter of 4.2 Å, and free cavity with minimum diameter of 11.4 Å

(Rani et al., 2004). This zeolite is characterized by square-faced cubic structure surrounding the cavity, connected by eight sodalite cages (Pangan et al., 2021). Figure 1 is an illustration of structure of zeolite-A, specified as Linde zeolite A, according to International Zeolite Association (IZA) (Petrov and Michalev, 2012).

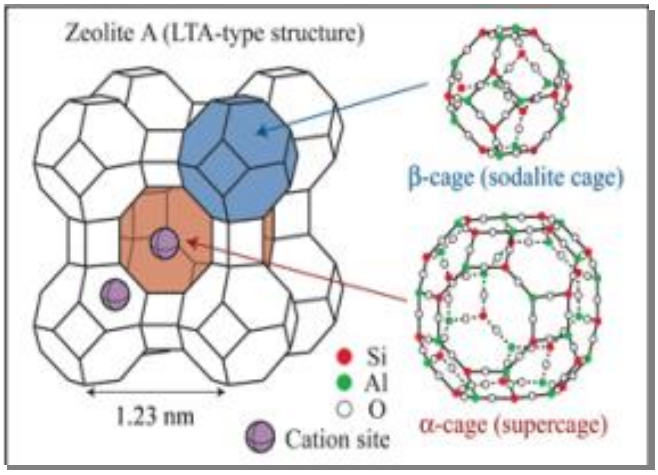

Figure 1. Features of the pores in Zeolite A (IZA code LTA).

One well known application of zeolite-A is as a catalyst for various reactions, such as transesterification of coconut oil to produce biodiesel (Pandiangan et al., 2017),co-pyrolysis of torrefied cassava root and palm oil for production of biogasoline (Febriyanti et al., 2021). Another application that has been reported is as an adsorbents for cationic dyes (Jamil et al., 2010). Good performance of zeolite-A has encouraged many studies for preparation of this zeolite. Various methods of zeolite-A synthesis that have been carried out previously such as hydrothermal method (Ji et al., 2020 andWulandari et al., 2019) and sol-gel technique (Georgiev et al., 2013). Various raw materials as a source of silica have been such as pumice (Prajaputra et al., 2019) and rice husk (Pandiangan et al., 2019). A as source of alumina, several raw materials have been used including aluminium metal (Pandiangan et al., 2017) and aluminium foil (Febriyanti et al., 2021).

2.3 Rubber Seed Oil

Rubber seed oil is non-edible vegetable oil and source of triglyceride biomass. Rubber seed oil can be extracted from rubber seed kernel by dry it and squeeze it. Rubber seed oil contains fatty acids such as palmitic acid, stearic acid, oleic acid, linoleic acid, linolenic acid and other acid. In food industry, rubber seed oil can't compete with the demands because it is a non-edible oil. Several study use it as raw material to produce biofuel (Chen et al., 2021).

2.4 Biomass Pyrolysis

2.4.1 Pyrolysis Reaction Mechanism

During the pyrolysis process, some reactions take places, i.e, dehydration, depolymerization, decarboxylation, and isomerization. The primary reactions include formation of char, depolymerization, and fragmentation. Formation of char happen by condensation of the benzene ring during the pyrolysis process, while the depolymerization by cracking of the bonds between the monomers (Zadeh et al., 2020). Figure 2 presents the overall pathways in the mechanism of pyrolysis.

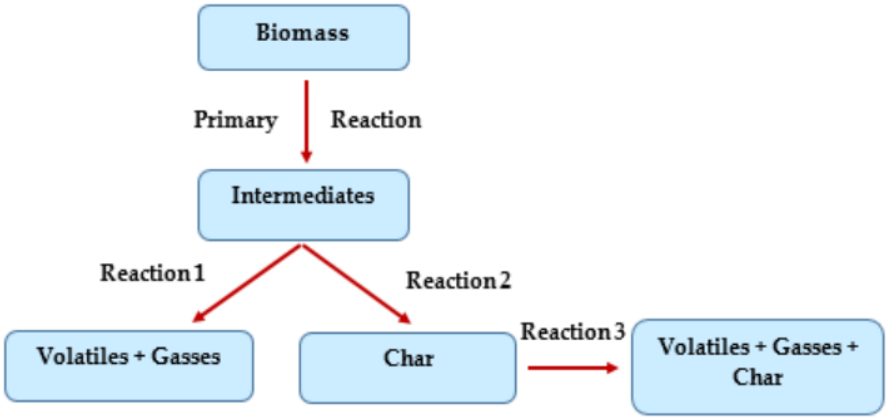

Figure 2. The overall pathway of pyrolysis mechanism (Zadeh et al., 2020).

In the pyrolysis mechanism, several main reactions or primary reactions occur that play a role in the thermal breakdown of biomass into intermediates and final products. This primary reaction involves breaking chemical bonds in complex compounds in biomass, producing smaller compounds.

Furthermore, intermediates formed during primary reactions can undergo a series of secondary reactions or subsequent reactions, which can involve condensation, polymerization, fragmentation, and other chemical reactions. This secondary reaction can produce various end products such as bio crude oil, biochar, and gases such as methane and hydrogen.

2.4.2 Factors Affecting Pyrolysis

Pyrolysis has many factors such as biomass composition, particle size, temperature, heating rate, vapor residence time and reaction atmosphere that affect the performance of pyrolysis. The composition of biomass feedstock should be taken into consideration when it aimed to produce higher yield of specific product as the physic-chemical characteristics of each component is different in various biomasses. Cellulose and hemicellulose are more preferable because

containing biomass for high bio-oil yield and lignin for the biochar (Feng and Lin, 2017; Akhtar and Amin, 2012 and Yang et al., 2006).

Particle size of biomass also has vital role in pyrolysis. It is reported that change on babool seed from below 1 mm influence product yield. The char yield increased from 18% to 25.8%, gas and liquid product yield decreases from 37% to 29% and 42.7% and 41.8%, respectively. It also reported that maximum bio-oil yield (~32%) is found at particle 0.4 mm. They suggest small particles are suitable for obtaining high bio-oil yield (Garg et al., 2016).

Bio oil yield also varies significantly with the pyrolysis temperature. To maximum the quality of the bio-oil it is important to consider the optimum temperature for pyrolysis. For example, maximum oil yields for pyrolysis of tamarind seeds occur at 400 °C is 44% and 45% (Kader et al., 2013) and safflower seed at 500 °C (Beis et al., 2002), respectively.

Pyrolysis of biomass can be categorized based on heating rate along with reaction temperature as slow, fast and flash. Slow pyrolysis is a pyrolysis method with a heating rate of less than (0.1-1.0 °C/sec), fast pyrolysis at temperatures ranging from 500 °C with a very high rate of temperature rise (10-200 °C/sec) and a very short time (1-2 seconds), flash pyrolysis uses higher heating with ambient temperature of 450-1000°C and very short gas residence time (< 1 second) (Feng & Lin, 2017 and Balat et al., 2009).

The yield of bio oil also differs with the vapor residence time. The low residence time favors the high yield of bio oil and high residence time produces high gas during the pyrolysis thus the oil yield reduces. Shorter residence time minimizes the secondary reaction which results high bio-oil formation (Dhyani & Bhaskar, 2017).

Pyrolysis of biomass is generally performed under inert atmosphere because devolatilization rate in deoxidizing atmosphere found more to that of oxidative degradation (Oladeji et al., 2015).

2.4.3 Application

The application of biomass pyrolysis has been carried out by several researchers such as the pyrolysis of rubber seed oil with cassava solid waste using a zeolite-A as a catalyst to produce liquid fuel with a hydrocarbon amount of 78% (Simanjuntak et al., 2019). Another application was pyrolysis of palm oil with a zeolite-A calcined at 700 °C and produced liquid fuel with a hydrocarbon content of 80% (Febriyanti et al., 2021). Co-pyrolysis of torrefied cassava root and palm oil using zeolite-A to produce biogasoline with a hydrocarbon content of 74% (Simanjuntak et al ., 2021).

2.5 Bio Crude Oil (BCO)

Bio Crude Oil (BCO) is a liquid product derived from the pyrolysis process of biomass, such as plant fibers, forest biomass, or agricultural waste. BCO bears resemblance to crude oil in terms of its physical properties and chemical composition. However, it is important to note that BCO is not petroleum but rather a product generated through thermal conversion of biomass.

The composition of BCO varies depending on the type of biomass used and the pyrolysis conditions. Generally, BCO consists of a mixture of hydrocarbons, oxygen, nitrogen, and a small amount of sulphur compounds. The specific chemical composition may differ between different biomass sources.

Here are some common physical properties associated with BCO:

1. Density: Ranging from 0.8 to 1.2 g/cm³, depending on the type of biomass utilized.
2. Calorific value: BCO exhibits varying calorific values, typically ranging from 30 to 45 MJ/kg.
3. Viscosity: BCO generally possesses higher viscosity compared to crude oil, with values ranging from 100 to 500 cP.

4. Moisture content: BCO may contain a small amount of water, depending on processing and storage conditions.
5. Sulphur content: BCO can contain varying levels of sulphur compounds, which can affect its quality and combustion characteristics.

Limitations of BCO include stability and degradation. BCO can undergo degradation over time during storage, particularly if exposed to air or unfavorable environmental conditions. Therefore, careful handling and storage are necessary to maintain its quality. The biomass pyrolysis process can result in BCO contaminated with undesirable compounds, such as heavy metals or toxic organic compounds. This contamination can affect the quality and safety of BCO utilization. The availability of biomass as a feedstock for BCO production can be limiting, depending on geographical location, seasons, and sustainable biomass resource availability (Bridgwater, 2012; Ronsse et al., 2013).

As explained earlier, BCOs have undesirable properties such as being acidic, corrosive, containing high oxygenated compounds, and thermally unstable. Therefore, to improve its quality, unwanted components in BCO need to be converted into more useful components, one of which is by further BCO processing (upgrading) physically using hot steam filtration or chemically using catalysts in the pyrolysis process (Eddy et al., 2023).

2.6 Characterization

In this study, the zeolite was characterized using XRD, XRF, SEM, FT-IR, while characterization of BCO was conducted using GC-MS.

2.6.1 X-Ray Diffraction (XRD)

X-ray diffraction is a technique that studies crystal structure and atomic spacing. X-ray diffraction is based on monochromatic X-ray constructive interference and crystal samples. These X-rays are produced by cathode ray tubes, filtered to produce monochromatic radiation. The interaction of the beam with the sample produces constructive interference (and the diffraction ray) when conditions conform to Bragg's law (Equation 1):

$$n\lambda = 2dsin\theta \quad (1)$$

The symbol (n) is an integer, (λ) the wavelength of X-rays, (d) is the interplanar distance that produces diffraction, and (θ) is the angle of diffraction. This law connects the wavelength of electromagnetic radiation with the angle of diffraction and the distance of the lattice in a crystalline sample. The diffraction X-rays are then detected, processed, and counted. By scanning the sample through various 2θ angles, all possible directions of diffraction of the lattice should be achieved due to the random orientation of the material in the form of powder. The conversion of diffraction peaks to d-spacing allows the identification of compounds because each compound has a unique set of d-spacing. Typically, this is achieved by the comparison of d-spaces with the standard reference pattern of X-rays produced in the cathode ray tube by heating the filament to produce electrons, accelerating the electrons towards the target by applying voltage, and attacking the target material with electrons (Bunaciu et al., 2015).

The working principle of this tool is that when electrons have enough energy to release the inner shell electrons from the target material, an X-ray spectrum is produced. This spectrum consists of several components such as Kα and Kβ. Kα consists of Kα_1 and Kα_2. Kα_1 has a slightly shorter wavelength and is twice the intensity of Kα_2. The specific wavelength is characteristic of the target material (Cu, Fe, Mo, Cr). Filtration, with a foil or crystal monocracometer, is necessary to produce the monochromatic X-rays necessary for diffraction. Kα_1 and Kα_2 have wavelengths close enough that on average both are used. Copper is the most

common target material for single-crystal diffraction, with a Radiation CuKα D of 1.5418 A. These X-rays are arranged and directed to the sample. As the sample and detector are rotated, the intensity of the reflected X-ray is recorded. The detector records and processes these X-ray signals and is transmitted to output devices such as printers or computer monitors (Bunaciu et al., 2015). A typical example of XRD difractogram of zeolite-A is shown in Figure 3

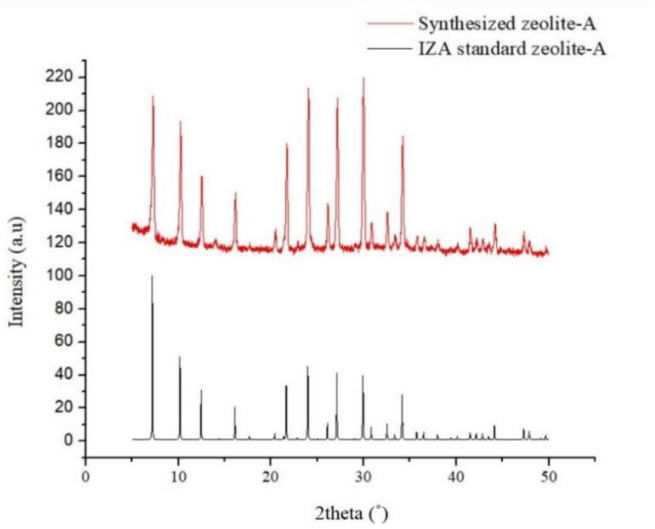

Figure 3. Diffractogram of synthesized zeolite-A and IZA standard zeolite-A (Pandiangan et al., 2023).

2.6.2 X-ray fluorescence (XRF)

XRF is an analytical method for determining the chemical composition of all types of materials. The analyzed sample can be in solid, liquid, powder, filtrate or other form. This method is fast, accurate and does not damage the sample, generally only requires a minimum of sample preparation. The precision and reproducibility of XRF analysis is very high. Highly accurate results are possible when a good standard specimen is available, the measurement time depends on the number of elements to be determined and the required accuracy, as well as varies between a few seconds or 30 minutes. The analysis time after measurement is only a few seconds (Brouwer, 2010).

The working principle is as follows: There are three main interactions when X-rays come into contact with matter: Fluorescence, Compton scatter and Rayleigh scatter. If a beam of X-ray photons is directed to a slab of material in the fraction to be transmitted, a small part is absorbed producing fluorescent radiation and a small part is redistributed. Scattering can occur with a loss of energy or without loss of energy. The first scattering is known as the compton scatter and the second is Rayleigh. Fluorescence and scattering depend on the thickness (d), density (ρ) of the material composition and on the energy of the X-rays shown on Figure 4 (Brouwer, 2003). Other research reported, based on XRF results that Si/Al ratio of the zeolite-A remained constant and close to 1 (de Araújo et al., 2019).

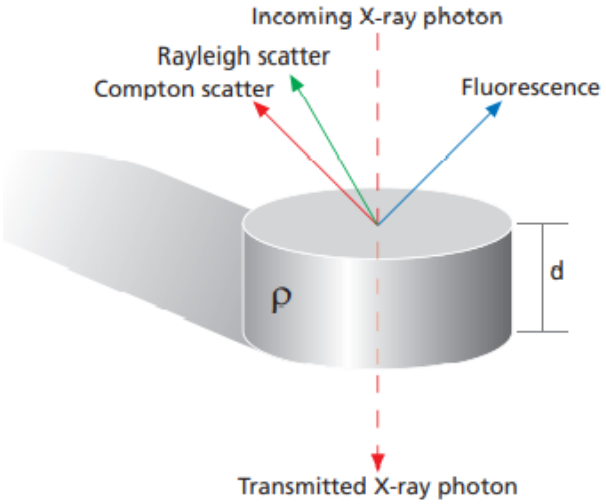

Figure 4. Interaction of contact X-rays with matter (Brouwer, 2010).

2.6.3 Scanning Electron Microscope (SEM)

SEM is a type of electron microscope that uses an electron beam to scan a sample. This technique is basically used to obtain a high-resolution picture of the surface features and allow conclusions about the distribution of different chemical elements in the sample (Goldstein, 2022). SEM works at high magnifications reaching 300,000x and even 1000,000 to produce very precise images of various

materials. Materials that can be used in SEM are organic and solid inorganic materials including metals and polymers (Abdullah & Mohammed, 2019).

SEM Working Principle as follows: The electron source is capable of emitting electrons accelerated by the applied voltage. The magnetic lens unites the flow of electrons into a focused beam, which then hits the surface of the sample in a smooth and appropriate place. The electron beam then scans the surface of the specimen in a rectangular raster. Users can increase the magnification by reducing the size of the scanned area on the specimen. The detector collects backscattered and secondary (SE) electrons. The corresponding signal is measured and the value is mapped as a brightness variation on the image display. Secondary electrons are more often used as read-out signals. They highlighted the topography of the sample surface, which is a bright area representing the edge while the dark area on a light microscope (Joy, 2019). A typical example of SEM micrograph of zeolite-A is shown in Figure 5

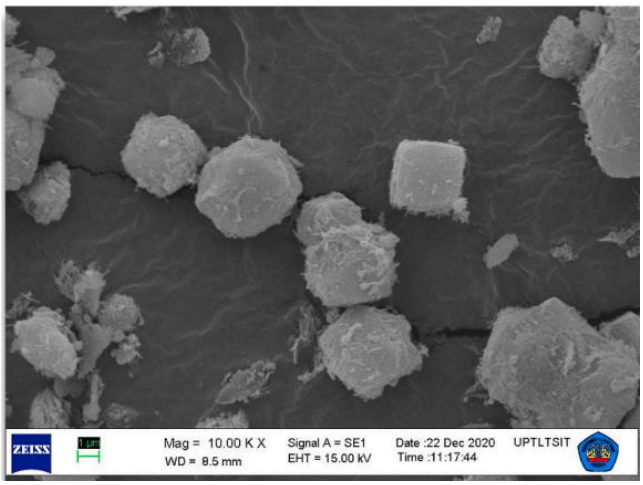

Figure 5. SEM of zeolite-A (Simanjuntak, 2021).

2.6.4 Fourier Transform Infrared (FT-IR)

FT-IR is a spectrophotometer used to identify functional groups and types of bonds on a molecule. For silica, this technique is used to identify the functional

groups of siloxane (Si-O-Si) and silanol (Si-OH). The siloxane absorption band in amorphous silica appears in wavenumber range of 1000 to 1250 cm^{-1}, this absorption indicates the presence of silicon-oxygen (Si-O) bonds in amorphous silica structure. The silanol absorption band appears in wavenumber range of 3200 to 3600 cm^{-1}, this absorption indicate the presence of hydrogen bonding between silanol groups on the amorphous silica surface (Meng et al., 2018).

Zeolite has a functional group that can be identified by FTIR, the absorption band widens with strong intensity in the area 1095-1092 cm^{-1} shows the vibrational characteristics of the siloxane group (Si–O–Si), the absorption band of about 494-420 cm^{-1} indicates the presence of a Si–O–Al group. The absorption band of Al–O vibration can be observed in the area of 480-470 cm^{-1}. The absorption band around the area 3400-3300 cm^{-1} indicates the presence of OH- functional groups in the water molecule (Platon and Thomson, 2003).

2.6.5 Gas Chromatrography-Mass Spectrofotometry (GC-MS)

GC-MS is an analytical technique that combines the properties of gas-liquid chromatography separation with mass spectrometric detection features to identify different substances in the sample being tested. GC requires analytes to have significant vapor pressures between 30 and 300 °C.

The working principle of GC-MS is that the sample mixture is injected, evaporated and flowed into a column thermally controlled by an inert gas. Sample compounds can interact with the stationary phase through various intermolecular forces such as van der Waals forces and dipole-dipole interactions. Some compounds tend to interact more strongly due to their polarity, thus generating a higher concentration in the stationary phase compared to the moving phase. As a result, these compounds are strongly retained in the column and have a longer retention time (RT) compared to weaker interaction compounds with the stationary phase. Over time, with a continuous flow of inert gases and thermally

controlled columns, variations in the partition coefficients of compounds result in the separation of compounds in the mixture. The separate compound then elute from the column and is detected (Jackie et al., 2020).

III. RESEARCH METHOD

3.1 Place and Time

This research was conducted at the Inorganic/Physical Laboratory of the Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Lampung. XRD analysis was carried out at ITS Surabaya, SEM analysis was carried out at UPT LTSIT University of Lampung, FTIR analysis was carried out at Dipenogoro University, XRF analysis was carried out at Padang State University, and GC-MS analysis was carried out at Gadjah Mada University Yogyakarta and Bandung Health Polytechnic. This Research was carried out from October 2022 to April 2023.

3.2 Tools and Materials

3.2.1 Tools

Tools used in this study include hotplate stirrer, magnetic stirrer, analytical balance, knife, Jisico J-300S oven, magnetic stirrer, lock and lock, drip pipette, glassware, spatula, pyrolysis device, polypropylene bottle, teflon, porcelain glass, statif and clamps, split funnel, 200 and 300 mesh sieves, mortar and pestle, and autoclave. Characterization analysis using X-Ray Diffraction (XRD) PANAlytical type XPert MPD diffractometer, X-Ray Fluorescense (XRF) PANAlytical Epsilon 3, Fourier Transform Infra-Red (FTIR) PerkinElmer Spectrum IR Version 10.6.1,

Scanning Electron Microscope (SEM) type ZEISS EVO MA 10, Gas Chromatography-Mass Spectrophotometry (GC-MS) type QP2010S SHIMADZU and GC-MS Agilent Technologies 7890A.

3.2.2 Materials

The materials used in this study were rubber seeds, pumice, 3 M HNO_3 solution, 2.5 M NaOH solution, 4 M NaOH solution, 1 M HCl solution, distilled water, filter paper, food grade aluminium foil, TBA paper 0,8 mm and universal indicator pH.

3.3 Research Procedure

3.3.1 Pumice Preparations

In this study, the pumice taken came from the coast of Suak, Kalianda, South Lampung. The first step taken is the preparation of pumice. Pumice is cleaned of impurities by soaking with water, then repeatedly washed using water and brushed on several parts to remove impurities that are still attached to the surface of the pumice. Then the pumice that has been cleaned of impurities is dried in the sun for 12 hours. And it is re-dried using an oven at 100 °C for 10 hours. The dried pumice is mashed with mortar and pestle and then sifted with a 150 mm mesh and then 500 g of pumice powder is purified by adding 1 L of 1 M HCl solution for 24 hours, then filtered and washed with distilled water. Then the pumice that has been purified is dried using an oven at 100 °C for 6 hours.

3.3.2 Pumice Amorphous Silica Extraction

A total of 10 g of powder was refluxed in 150 mL of NaOH with a concentration of 2.5 M and 4 M with a stirring speed of 300 rpm. The formed sodium silicate is filtered and the filtrate is taken. Then the filtrate is titrated using a 3 M HNO_3 solution of 10 mL to neutralize the filtrate and produce white silica gel. Silica gel is let stand for 4 hours, then washed with hot distilled water, and dried in oven at 100 °C for 6 hours. The results of the silica are mashed and sifted with a 200 mm mesh.

3.3.3 Characterization of Pumice Silica

Pumice silica characterization will be performed using XRD to analyzing the type of silica is amorphous or crystaline, XRF to determine the content of silica and FT-IR to analyze the existing groups on silica.

3.3.4 Zeolite-A Synthesis by Hydrothermal Method

Zeolite-A was synthesized by developing methods and compositions in previous studies (Herliana et al., 2021). Preparation was carried out by preparing 20 g of pumice silica dissolved with NaOH solution (22.32 g in 150, 200, 250, 300 and 400 mL of distilled water) with a stirring speed of 500 rpm and heated at 70 ºC for 3 hours. The sodium silicate solution obtained was then cooled and filtered using filter paper. The filtrate obtained was aged for 24 hours. Then 13.31 g of food grade aluminum foil was dissolved in 150 mL of NaOH solution by stirring at 500 rpm for 3 hours to form sodium aluminate which was then added to the sodium silicate that had been aged.

The resulting mixture was put into an autoclave and aged for 24 hours. Then the mixture was crystallized in an oven at 100 ºC for 72 hours. The gel obtained was filtered, washed with distilled water to pH of 7-8, then dried in an oven at 100 ºC for 24 hours. The zeolite solid has been dried was grinding and calcined at a temperature of 600, 700, 800, 900 ºC for 6 hours.

3.3.5 Characterization of Zeolite-A Catalysts

Zeolite characterization will be performed using XRD to analyzing the effect of pumice silica on the crystallographic structure of zeolite samples, SEM analysis is used to determine the morphology on the surface of zeolite samples and XRF to determine the elements contained in zeolite samples.

3.3.6 Extraction of Rubber Seed Oil

The steps taken to extract rubber seed oil are first peeled and separated from the outer shell and then separated from the seed shell, then dried in the sun until dry. Before pressing to obtain rubber seed oil, dried rubber seeds are put in the oven for 20 minutes at a temperature of 100 ºC to remove the moisture content that is still remaining in the rubber seeds. The removal of oil from rubber seeds is carried out using a press. Then the result obtained is filtered to separate the oil and its pulp. Furthermore, rubber seed oil is ready to be used for the pyrolysis process.

3.3.7 Zeolite-A Activity Test

3.3.7.1 Pyrolysis Test

The way to obtain biogasoline pyrolysis results, the first step is that as much as 250 mL of rubber seed oil is put into the pyrolysis reactor, the pyrolysis process is carried out using 12.5 g of silica sand and without using silica sand. The steam formed is flowed from the reactor and then cooled in the condenser to produce a distillate, known as bio crude oil (BCO). The BCO result of 100 mL was mixed with 5 g of zeolite-A catalyst and further pyrolysis was carried out to obtain biogasoline.

3.3.8 Characterization of Pyrolysis Products

The characterization of biogasoline to identify the constituent components of the sample was carried out using GC-MS.

V. CONCLUSION AND SUGGESTION

5.1 Conclusion

Zeolite-a synthesis using pumice silica and food grade aluminum foil by hydrothermal method was successfully carried out at a NaOH solution concentration of 1.3 M. The results of XRD analysis show that zeolite-A calcined at temperatures of 600 and 700 °C has conformity with Zeolite-A IZA standards. The results of catalytic activities test show that zeolite-A calcined at temperature of 600 °C has the best catalytic activity with percentage of biogasline and hydrocarbon is around 30.61% and 77.51%.

5.2 Suggestion

In order for the purity of zeolite A obtained to be higher and for the sustainability of this study it is recommended to study the factors affecting zeolite A synthesis such as the composition of the starting material, reaction temperature, reaction time, Si/Al ratio and pH.

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