REPORT

FIELD INTERNSHIP

LITERATURE STUDY OF SIO₂ ION EXCHANGE PROCESS MECHANISM IN ION EXCHANGE RESIN



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FOREWORD

Assalamu'alaikum Wr. Wb.

Praise the author, pray to God Almighty so that the author can complete the field internship report with the title "Literature Study of SiO_2 Ion Exchange Process Mechanisms in Ion Exchange Resin". This report was written with the aim of meeting the course requirements at the Department of Chemistry, Diponegoro University, Semarang.

This report is the result of field internship activities that have been carried out at Pusat Reaktor Serba Guna (PRSG) - BATAN on January 18th, 2021 to March 2021. As long as the authors carry out the field internship and write this field internship report, the authors gain a lot of knowledge as well as experience at Pusat Reaktor Serba Guna (PRSG) - BATAN.

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- 9. Parents and families who have given a lot of prayers and always give encouragement to the author.
- 10. Close relatives and all parties who have helped to compile this report.

The author hopes that this practical work report can be useful and provide information for anyone who reads it, especially for students of the Faculty of Science and Mathematics, Diponegoro University, Semarang. However, the author realizes that the preparation and writing of this field internship report is still far from perfect and there are still many shortcomings. Therefore, all constructive criticism and suggestions from readers will always be expected and accepted with pleasure.

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Bekasi, 1 March 2021

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CHAPTER I

INTRODUCTION

1.1 Background

The G.A Siwabessy research reactor is one of BATAN's research reactors which has a power of 30 MW. The reactor is equipped with primary and secondary cooling water systems for heat transfer and exchange as well as a moderator. Cooling water for both the research reactor and the power reactor requires high quality water purity because the cooling water is directly related to the reactor components made of metal or metal alloys so that the corrosion reaction occurs (Lestiani, 2003).

Primary cooling water functions as a medium for transferring heat arising in the reactor core, as a moderator and as a radiation shield in the axial direction. The heat formed as a result of fission in the core will be taken up by the primary cooling system and transferred to the secondary cooling system through a heat exchanger and finally discharged into the atmosphere through the cooling tower. As a heat carrier medium in the primary cooling system, mineral-free water is used from the mineral-free water production system (GCA 01) with certain quality (Lestari & Utomo, 2015).

Water as a coolant will be directly related to the component material or the reactor structure, so there is a possibility that a chemical reaction will occur between the water and the component material or the reactor structure which can cause corrosion or scale. The presence of silica in cooling water can cause deposits on metal surfaces that contact the water flowing through the system which results in a decrease in the efficiency of heat transfer. One of the removal methods. Silica in pure water is a strong base anion exchange resin (Lestari & Utomo, 2015).

Chemistry is a science that is closely related to the analysis of water content in order to maintain Pusat Reaktor Serba Guna cooling water system so that the water quality is in accordance with specifications. As a form of output of implementing the field internship in a network, the authors conducted a literature study on water chemistry management entitled Literature Study of SiO₂ Ion Exchange Process Mechanisms in Ion Exchange Resin.

1.2 Formulation of The Problem

- 1.2.1 How is silica formed in water?
- 1.2.2 How is the ion exchange resin process mechanism in SiO_2 ion?
- 1.2.3 How does silica ion affect water?

1.3 Scope of Problem

- 1.3.1 Subject: water management system
- 1.3.2 Research object: silica content in cooling water at RSG-GAS
- 1.3.3 Implementation of practical work: this field internship is carried out online and in direct field visits

1.4 Research Purposes

The purpose of the chemistry students field internship emphasize the application of academic skills to chemical analysis problems that require solutions

- 1.4.1 General Instructional Objectives (GIO)
 - 1. Can apply the knowledge obtained from lectures on problems that occur in the field or the chemical industry.
 - 2. The realization of a link and match between theory and practice in the field.
 - The realization of good cooperation between industry/research centers with universities, especially the Department of Chemistry, Faculty of Science and Mathematics (FSM), Diponegoro University.
 - 4. Opening students insights in order to know and understand the application of their knowledge in the world of work in general and

to be able to absorb and associate with the world of work as a whole.

- 1.4.2 Specific Instructional Objectives (SIO)
 - 1. Introducing students to potential business aspects in the employment field, including the company's organizational structure, career paths, and company / agency management.
 - 2. Providing opportunities for students to socialize themselves in a real work environment both as employees and as independent workers, especially with regard to work discipline.
 - Adding insight into applied chemistry that is used in industrial environments in relation to the knowledge that has been studied in lectures.
 - 4. Knowing the primary and secondary cooling water instruments used in water purification.

CHAPTER II

GENERAL DESCRIPTION OF THE COMPANY

2.1 **Profile of the National Nuclear Energy Agency (BATAN)**

BATAN has a main task in accordance with Presidential Regulation Number 46 of 2013 concerning carrying out activities in the coordinator of research, development, and utilization of nuclear science and technology in accordance with the provisions of the legislation. Research, development, and utilization of nuclear science and technology in Indonesia is only directed for peaceful purposes and as much as possible for the welfare of the Indonesian people..

This commitment is firmly carried out by the Indonesian Government by ratifying the Treaty on the Prevention of the Spread of Nuclear Weapons with Law Number 8 of 1978 and ratifying the Treaty on Southeast Asian Areas Free of Nuclear Weapons by Law Number 9 of 1997. Then in accordance with Article 3 of Presidential Regulation Number 46 of 2013, in carrying out these duties BATAN carries out functions:

- 1. To study and compile national virtues in the coordinator of research, development, utilization of nuclear science and technology.
- 2. Coordination of functional activities in the implementation of BATAN duties.
- 3. To carry out research, development, utilization of nuclear science and technology.
- 4. Facilities and guidance for the activities of government agencies and other institutions of the coordinator of research, development, utilization of nuclear science and technology.
- 5. Implementing coaching and providing administrative support to all organizational units within BATAN.
- 6. Implementing the management of nuclear standardization and quality assurance.
- 7. Fostering education and training.
- 8. Supervision of the implementation of BATAN duties.

9. Submitting reports, suggestions and considerations to the coordinator of research, development, utilization of nuclear science and technology (Batan, 2020).

2.1.1 Badan Tenaga Nuklir Nasional (BATAN)

The State Committee has the task of investigating the possibility of radioactive fallout from nuclear weapons testing in the Pacific Ocean. The development and application of nuclear technology in Indonesia began with the establishment of the State Committee for Radioactivity Investigation in 1954.

By paying attention to the development of the utilization and utilization of atomic energy for the welfare of the community. So through Government Regulation No. 65 of 1958, on December 5, 1958, the Atomic Energy Council (LTA) was formed. Which was perfected to become the National Nuclear Energy Agency (BATAN) based on Law no. 31 of 1964 concerning Basic Provisions of Atomic Energy. Every December 5 th, which is a historic date for the development of nuclear technology in Indonesia and is designated as the anniversary of BATAN.

In subsequent developments, to further improve control of the coordinator of nuclear science and technology, in 1965 the operation of the first atomic reactor (Triga Mark II) was inaugurated in Bandung. Then on their knees several R&D facilities were built scattered in various research centers, including the Atomic Energy Research Center, Pasar Friday, Jakarta (1966). GAMA Atomic Energy Research Center, Yogyakarta (1967), and 30 MW Multipurpose Reactor (1987) along with supporting facilities, such as fuel fabrication and research, reactor safety testing, treatment of radioactive waste and other nuclear facilities. Meanwhile, with a paradigm shift in 1977, Law No. 10 concerning Nuclear Energy was enacted, which among them regulates the separation of the elements implementing nuclear energy utilization

activities (BATAN) from the nuclear power supervisory element (BAPETEN). (Batan, 2020).

2.1.2 Vision and Mission of Batan

In carrying out its duties, Badan Tenaga Nuklir Nasional has a vision and mission as follows:

A. Vision

"Badan Tenaga Nuklir Nasional (BATAN) excels at the regional level, plays a role in accelerating welfare towards national independence."

- B. Mission
 - 1. Formulate a national nuclear science and technology policy and strategy.
 - 2. Developing reliable, sustainable and beneficial nuclear science and technology for the community.
 - 3. Strengthening the role of BATAN as a leader at the regional level, and actively participating internationally.
 - 4. Carry out excellent service for the use of nuclear science and technology for the sake of stakeholder satisfaction.
 - 5. Implementing nuclear science and technology dissemination by emphasizing the principles of benefit, safety and security.

2.1.3 Main Duties and Functions of BATAN

The main task of BATAN in accordance with Presidential Regulation Number 46 of 2013 is to carry out government duties in the coordinator of research, development and utilization of nuclear science and technology in accordance with the provisions of laws and regulations. Research, development and utilization of nuclear science and technology in Indonesia are only directed for peaceful purposes and as much as possible for the welfare of the Indonesian people.

This commitment is firmly carried out by the Government of Indonesia by ratifying the Treaty on the Prevention of the Spread of Nuclear Weapons with Law Number 8 of 1978, and ratifying the Treaty on Southeast Asian Areas Free of Nuclear Weapons by Law Number 9 of 1997. Then in accordance with Article 3 of the Regulation President Number 46 of 2013, in carrying out these duties BATAN carries out functions:

- 1. To study and formulate national policies in the coordinator of research, development and utilization of nuclear science and technology;
- Coordination of functional activities in the implementation of BATAN duties;
- To carry out research, development and utilization of nuclear science and technology;
- Facilitating and fostering the activities of government agencies and other institutions in the coordinator of research, development and utilization of nuclear science and technology;
- 5. Implementing coaching and providing administrative support to all organizational units within BATAN;
- 6. Implementing the management of nuclear standardization and quality assurance;
- 7. Fostering education and training;
- 8. Supervision of the implementation of BATAN duties; and
- 9. Submitting reports, suggestions and considerations to the coordinator of research, development and utilization of nuclear science and technology.

2.1.4 The Purpose of BATAN

The purpose of nuclear science and technology development is to provide real support in national development with the following roles:

- 1. Increase the yield of nuclear energy, isotope and radiation litbags and their utilization / utilization by the community in supporting national development programs.
- 2. Improve the performance of institutional management and strengthen innovation systems in order to support research, development and application of nuclear energy, isotopes and radiation.

2.1.5 Target BATAN

The targets for the development of nuclear science and technology to be achieved are:

- 1. Increasing the yield of enisora litbags in the form of superior food plant seeds, availability of basic infrastructure for nuclear power plant development, public understanding of nuclear technology, utilization of isotope technology applications and radiation for health.
- 2. Improving the performance of institutional management and strengthening the innovation system covering science and technology institutions, science and technology resources in order to support the utilization of research results, development and application of nuclear energy, isotopes and radiation in society.

2.1.6 The Principle of BATAN

All nuclear science and technology activities are carried out professionally for the purpose of safety and security, as well as environmental preservation.

2.1.7 Values of BATAN

All nuclear science and technology activities are based on the following values

- 1. Visionary, Innovative, Excellent, and Accountable.
- 2. Honesty, Discipline, Openness, Responsibility, Creative and Solidarity.

2.1.8 BATAN Organization



Figure 2. 1 Organizational Structure of the National Nuclear Energy Agency

2.1.9 Unit / Work Unit of BATAN in Serpong Nuclear Area -PUSPIPTEK

The BATAN area is divided into 5 nuclear areas, namely the South Jakarta Headquarters Nuclear Area, the Pasar Jumat Nuclear Area, the Serpong Nuclear Area, the Bandung Nuclear Area, dan the Yogyakarta Nuclear Area. The work units in the Serpong Nuclear Zone are:

- 1. Pusat Sains dan Teknologi Bahan Maju (PSTBM).
- 2. Pusat Teknologi Bahan Bakar Nuklir (PTBBN).
- 3. Pusat Teknologi dan Keselamatan Reaktor Nuklir (PTKRN).
- 4. Pusat Tekologi Limbah Radioaktif (PTLR).
- 5. Pusat Rekayasa Fasilitas Nuklir (PRFN).
- 6. Pusat Teknologi Radioisotop dan Radiofarmaka (PTRR).
- 7. Pusat Reaktor Serba Guna (PRSG).
- 8. Pusat Standarisasi Mutu Nuklir (PSMN).
- 9. Pusat Pendayagunaan Informatika dan Kawasan Sterategis Nuklir (PPIKSN).

2.2 Profile of Pusat Reaktor Serba Guna (PRSG)

Pusat Reaktor Serba Guna (PRSG) is one of the BATAN work units / work units located in the Serpong Nuclear Area. PRSG performs its duties under the coordination of the Deputy Coordinator for Nuclear Technology Utilization. The main task of PRSG is to carry out the formulation and control of technical policies, implementation and guidance, and guidance at Reaktor Serba Guna G.A Siwabessy Coordinator. PRSG is located at Kawasan Pusat Penelitian Ilmu Pengetahuan dan Teknologi - PUSPIPTEK (Serpong Nuclear Area) Building. 30/31, Serpong, South Tangerang City, Banten.

2.2.1 History of Pusat Reaktor Serba Guna (PRSG)

This nuclear reactor has been built since 1983 by Interatom International, a company from West Germany, known as Reaktor Serba Guna (RSG-GAS) located in Pusat Ilmu Pengetahuan dan Teknologi (PUSPIPTEK) Serpong, South Tangerang. The RSG-GAS reactor is one of the research reactors owned by the Indonesian nation which is managed and operated by PRSG-BATAN. This reactor functions as a research facility in various coordinators such as nuclear, health, advanced materials and nuclear fuel. After reaching the first critical point on March 27, 1987, the reactor was inaugurated by the President of the Republic of Indonesia II (General Besar TNI Purn. H. Muhammad Soeharto) on August 20, 1987.

After the reactor was inaugurated, in March 1992 reactor operation was achieved at full power 30 MW for the first time. With this power, this reactor is the largest nuclear research reactor in Southeast Asia and is the second largest research reactor in the world. The construction of RSG-GAS in the PUSPIPTEK Serpong area is accompanied by the construction of other supporting facilities, such as fuel research and fabrication facilities, radioactive management facilities. waste radioisotope and radiopharmaceutical production facilities and several other laboratory facilities that can support RSG-GAS operations. The name RSG-GAS itself comes from the name of the first Director General of BATAN, dr. Gerrit Agustinus Siwabessy (Batan, 2020).

2.2.2 Vision and Mission of PRSG

The vision and mission of PRSG in carrying out its duties are:

A. Vision

"BATAN excels at the Regional Level, plays a role in The Acceleration Of Welfare Towards National Independence".

- **B.** Mission
 - 1. Increasing the availability and reliability of RSG-GAS operations (operating on schedule at least 2700 hours per year and Unplanned Shutdown Reactor due to internal factors less than 5 times per year);

- 2. Guarantee the management of RSG-GAS in complying with the applicable nuclear regulations;
- 3. Ensuring the achievement of zero accidents in a sustainable manner, both from the aspects of safety, security and the environment;
- 4. Increase the capacity and capability of human resources in managing RSG-GAS;
- 5. Improve the types and quality of RSG-GAS irradiation services.

2.2.3 Main Duties and Functions of the PRSG

The main tasks and functions of the PRSG are in accordance with the Decree of the Head of Batan No. 14 of 2013 concerning the Organization and Work Procedure of Badan Tenaga Nuklir Nasional, namely:

A. The main tsks of PRSG

Carry out the formulation and control of technical policies, implementation and guidance, and guidance at the Coordinator of Multipurpose Reaktor Serba Guna G.A Siwabessy.

- B. The functions of PRSG
 - Implementation of Planning, Correspondence and Archives, Personnel, Finance, Equipment and Household Affairs, Scientific Documentation and Publication and Reporting;
 - 2. Implementation of Reactor Operations;
 - 3. Implementation of Reactor Maintenance;
 - 4. Monitoring of Work Safety and Reactor Operation;
 - 5. Implementation of the Quality Assurance Program;
 - 6. Implementation of Nuclear Security

2.2.4 Values of PRSG

The values espoused by Pusat Reaktor Serba Guna include:

1. Accountability

Ready to accept responsibility and carry out responsibilities properly as assigned.

2. Discipline

Act according to regulations, procedures, orderly, on time and on target while maintaining efficiency and effectiveness of time and budget.

3. Excellence

Have the attitude and desire to always try to achieve better results than others.

4. Integrity

Upholding and basing every attitude and action on moral principles and values, ethics, laws and regulations, including keeping away from the tendency of corruption, collusion and nepotism.

5. Collaboration

Prioritizing cooperation, developing networks with external parties and prioritizing team work to achieve better performance.

6. Competence

Emphasizes the quality of mastery and fulfillment of the qualifications of HR capabilities as needed.

7. Innovative

Increase creative efforts to find renewal in every R&D result.



2.2.5 Organizational Structure of PRSG

Figure 2. 2 Pusat Reaktor Serba Guna Organizational Structure

In carrying out its main duties and functions, PRSG has a structure that has main duties and functions, each of which aims to carry out PRSG activities in accordance with existing main tasks, functions, objectives and targets. The following are the main tasks and functions of the structure in PRSG:

A. Administration Section

The administrative division has the task of carrying out planning, correspondence and archiving, personnel, finance, equipment and household affairs, scientific documentation and publication and reporting. The functions of the administration section include:

- Implementation of planning, correspondence and archiving, staffing, administration of scientific activities, documentation and publication, and reporting;
- Implementation of financial affairs; and
- Implementation of household and equipment affairs

The administration section consists of:

Subcoordiantor for correspondence, personnel, and scientific documentation

The duties of this subsection are to carry out planning, correspondence and archiving, staffing, administration of scientific activities, documentation and publication, and reporting

2) Subbagian Keuangan;

The Finance Subdivision has the task of conducting financial affairs.

3) Equipment Sub-Section.

The Equipment Subdivision has the task of carrying out household and equipment affairs.

B. <u>Reactor Operations Coordinator</u>

The reactor operation coordinator is one of the technical coordinators whose main task is to carry out reactor operations and to carry out neutron irradiation services to the existing stakeholders. The reactor operation coordinator consists of:

1) Subcoordiantor of Nuclear Material Accounting

The task of the Subkoordiantorini is to carry out data collection (accounting) of the movement of fuel / uranium elements, carry

out calculations on the management of the RSG-GAS reactor core, and do everything related to safeguards.

- Sub-coordinator of Operational Planning This sub-coordinating office has the task of carrying out neutron irradiation services from stake holders, as well as planning the annual RSG-GAS reactor operating schedule.
- Sub-coordinator of Operations
 This sub-coordinating office has the task of operating the RSG-GAS reactor and other support systems.

C. <u>Reactor Maintenance Coordinator</u>

The reactor maintenance coordinator is one of the technical coordinators whose main task is to carry out maintenance and repair of the system structure and components of the RSG-GAS reactor and the support system for the RSG-GAS reactor. The reactor maintenance coordinator consists of:

1) Sub-Coordinator of Mechanical Systems

The task of this sub-coordinator is to carry out maintenance both in the form of maintenance and repairs to the existing mechanical systems in the reactor system and the reactor support system.

2) Sub-Coordinator of Electrical Systems

The task of this sub-coordinator is to carry out maintenance both in the form of maintenance and repairs to the electrical system in the reactor system and the reactor support system.

3) Sub-Coordinator of Instrumentation and Control

The task of this sub-coordinator is to carry out maintenance both in the form of maintenance and repair of the instrumentation and control systems in the reactor system and the reactor support system.

D. Work Safety and Operations Coordinator

The work safety and operation coordinator is one of the technical coordinators whose main task is to carry out monitoring and analysis related to work safety and the reliability of the support system in the operation of the RSG-GAS reactor. The work safety and operations coordinator consists of:

1) Sub-Coordinator of Work Safety and Radiation Protection

The task of this sub-coordinator is to provide assistance and analysis to workers in carrying out their duties in order to comply with existing SOPs, especially work related to radiation.

2) Sub-Coordinator of Operations Safety

This sub-coordinator has the task of monitoring and analyzing the reactor system and the support system for the gnat reactor to ensure that the reactor operation can run smoothly and safely.

E. Quality Assurance Unit

The Quality Assurance Unit has the task of developing, monitoring the implementation and internal audit of the quality management system at the Multipurpose Reactor Center.

F. Nuclear Security Unit

The Nuclear Security Unit has the duty to carry out guard and monitoring so that the safety of the reactor can be safe and under control.

2.2.6 Services of PRSG

The following are some of the services provided by Pusat Reaktor Serba Guna:

- 1) Neutron Irradiation Service: is a type of service for neutron irradiation applications (targets) at RSG-GAS.
- To paz Stone Irradiation Service: is a type of service for topaz irradiation applications at RSG-GAS.
- Mineral-Free Water Supply Service: is a type of service for requests for mineral-free water supply.
- Student Services / Practical Students and / or Research: is a service unit for students and students who wish to do Internships / Practical Field Work, Research and Final Projects at the Multipurpose Reactor Center.
- Visiting Services: is a service unit that provides easy access to information related to visits to Serba Guna Reactor and other Badan Tenaga Nuklir facilities.
- Public Information Service: is a type of service that provides easy access to information to the public/community.
- 7) Complaint Service: is a service unit that manages complaints in the form of input, criticism, suggestions and information from the public as material for evaluating and analyzing PRSG performance towards better governance of R&D institutions.
- 8) Whistle Blowing System (WBS) Service: is a service unit that manages reports on Korupsi, Kolusi, dan Nepotisme (KKN) actions whether accepted or rejected by oneself as recipients or rejectors or reporting actions taken by the Aparatur Sipil Negara (ASN) in charge at Pusat Reaktor Serba Guna BATAN.

CHAPTER III

LITERATUR REVIEW

3.1 Silica (SiO₂)

Silicon dioxide or silica is one of the most common chemical compounds. Pure silica comes in two forms, namely quartz and cristobalite. Silicon is always bonded tetrahedral to four oxygen atoms, but its bonds are quite ionic in nature. In cristobalite, the silicon atoms are placed in the same way as the carbon atoms in diamond with the oxygen atoms in the center of each pair. In quartz there is a helix to form enantiomorph crystals. Quartz and cristobalite are interchangeable when heated. This process is slow because it requires breaking and re-forming of bonds and its activation energy is high. Silica is relatively unreactive to Cl₂, H₂, acids and most metals at 25°C or higher, but can be attacked by F₂, HF aqua, alkaline hydroxides and carbonate melts. (Cotton & Wilkinson, 1989).

Silica forms are some of the important crystal structures not only because silica is an abundant and useful substance, but because its structure (SiO_4) is the fundamental unit in most minerals. Silica crystals have two main characteristics, namely:

- 1. Each silicon atom is at the center of a tetrahedron consisting of four oxygen atoms.
- Each oxygen atom is in the middle between the two silicon atoms (Keenan, 1992).

3.2 Physical and Chemical Properties of Silica

3.2.1 Physical Properties

IUPAC name: Silicon dioxide

Other names: Quartz, Silica, Silicate oxide, Silicon (IV) oxide Molecular formula: SiO₂ Molar mass: 60.08 g mol⁻¹ Appearance: Transparent Crystal Density: 2,648 g cm⁻³. Melting point: 1600-1725°C Boiling point: 2230°C (Masramdhani, 2011).

3.2.2 Chemical Properties

Silica minerals have various chemical properties, including the following

a. Acid reaction

Silica is relatively unreactive to acids except for hydrofluoric acid and phosphoric acid.

 $SiO_{2(s)} + 4HF_{(aq)} \rightarrow SiF_{4(aq)} + 2H_2O_{(l)}$ (Svehla, 1985).

In excess acid the reaction is:

 $SiO_2 + 6HF \rightarrow H_2[SiF_6]_{(aq)} + 2H_2O_{(l)}$ (Svehla, 1985).

b. Base Reaction

Silica can react with bases, especially with strong bases, such as with alkaline hydroxides

 $SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_3 + H_2O$ (Svehla, 1985).

Commercially, silica is prepared by mixing a sodium silicate solution with a mineral acid. This reaction produces a concentrated dispersion which finally separates the particles from the hydrated silica, known as hydrosol silica or silicic acid which is then dried at 110°C to form silica gel. The reaction that occurs:

 $Na_2SiO_{3(aq)} + 2HCl_{(aq)} \rightarrow H_2SiO_{3(l)} + 2NaCl_{(aq)}$

 $H_2SiO_{3(s)} \rightarrow SiO_2.H_2O_{(s)}$

(Bakri, 2008).

3.3 Silica in Water

Silicic acid is found in natural water due to weathering of silicate minerals on earth. In general, the concentrations of silicic acid in groundwater and geothermal water range from 10 to 60 mg/L and 200 to 500 mg/L (as Si), respectively (Bai S. N., 2012). Silicic acid is mainly present as a monosilicic acid (H₄SiO₄) in groundwater at concentrations less than 60 mg/L (Bai, Han, & Ding, 2019).

Silica has a rather low solubility and tends to have little interaction with water molecules when dissolved in water. Silica is commonly found in water supplies in three different forms:

- 1. Silica monomer or dissolved silica which is reactive silica
- 2. Polymer silica or colloidal silica which is an unreactive silica produced from polymerization of silicic acid.
- 3. Granules or silica particulates (SiO₂)

(Lestari & Utomo, 2015)

3.4 Ion Exchanger Resin

Ion exchange resins are highly polemerized hydrocarbons that contain crosslinked bonds and groups containing exchangeable ions. Where the ion exchange reaction occurs in ions that have the same charge sign between the solution and the insoluble solid (resin) that comes into contact with the solution. The ion exchange process in resin is a reversible (back and forth) process and takes place stiochiometry (in an equivalent amount). Therefore, in the process of making mineral-free water, when all the cations or anions in the resin grain arrangement used have been used up, they are exchanged for cations or anions in water, so that the mass resin is no longer active by regenerating the resin (Lestari, Pujiarta, & Irwan, 2000).

3.4.1 Ion Exchange Principle

Ion exchange is a physico-chemical process. In this process the insoluble compound, in this case the resin, accepts certain positive or negative ions from the solution and releases other ions into the solution in the same equivalent amount. If the ion being exchanged is a cation, then the resin is called a cation exchange resin, and if the ion exchanged is an anion, then the resin is called anion exchange resin. Examples of cation exchange reactions and anion exchange resented in the reaction:

Cation exchange reaction:

 $2NaR_{(s)} + CaCl_{2(aq)} \rightarrow CaR_{(s)} + 2NaCl_{(aq)}$ (4.15)

Anion exchange reaction:

 $2RCl_{(s)} + Na_2SO_4 \rightarrow R_2SO_{4(s)} + 2NaCl_{(4.16)}$

Reaction (4.15) states that the solution containing $CaCl_2$ is treated with NaR cation-exchange resin, where R represents the resin. The stage where the ion exchange reaction occurs is called the service stage. If the resin has

exchanged all the Na⁺ ions it has, the ion exchange reaction will stop. At that time the resin is said to have reached the exhausted point, so it must be regenerated with a solution containing Na⁺ ions such as NaCl. The regeneration stage is the opposite of the service stage. The reaction that occurs in the regeneration stage is the opposite of the reaction (4.15). The cation exchange resin which exchanges the Na⁺ ions mentioned above is called the cation exchange resin with the Na cycle. The cation exchange resin with the H cycle will exchange H⁺ ions in the service and regeneration stages (Alfonsina, 2012).

3.4.2 Mekanisme Penukaran Ion

Ion exchange can be positioned as a unit of operation in chemical equilibrium. Ion exchange involves misplacing ions given by species from the exchange of insoluble material with ions of different species when the latter solution is brought up to contact / connect / mix. Ion exchange can be described in general terms of equilibrium:

 $B_1^+ + R^- B_2^+ \rightarrow B_2^+ + R^- B_1^+$

 $A_1^- + R^+ A_2^- \rightarrow A_2^- + R^+ A_1^-$

Where:

 $B1^+$, $B2^+$ = cations of 2 different species (types).

 $A1^{-}$, $A2^{-}$ = anions of 2 different species (types).

 \mathbf{R}^{-} , \mathbf{R}^{+} = Exchange of materials from cations and anions respectively.

(Pudjiastuti, 2008)

3.4.3 Types of Ion Exchange Resin

Based on the type of functional group used, ion exchange resins can be divided into four types, namely

1. Strong acid cation exchange resin.

Strong acid cation resins have hydrogen ions (\mathbb{R}^- . \mathbb{H}^+) in the presence of positively charged \mathbb{H}^+ ions, so this resin is often used to pick up positively charged ions. In operation, strong acid cation resistance can be operated under conditions (\mathbb{R}^- , \mathbb{H}^+), as well as in conditions \mathbb{R}^- , \mathbb{Na}^+ . the choice of which conditions will be operationalized affects the type of ion taken by the chemical to be produced and the chemical for reactivation (Montgomery, 1985).

2. Weak acid cation exchange resin

Weak acid cation resins are made of plastics or polymers which are reacted with the carbonic acid group (COOH⁻) thereby as a resin constituent. Weak acid cation resins required the presence of alkalinitis to remove hydrogen ions from the resin. In addition, the use of weak acid cation resins is only limited to conditions of water or wastewater that have a degree of acidity (pH) above 4 or 5. The regeneration process of weak acid cation resins can be carried out with a solution of hydrochloric acid (HCl) and sulfuric acid (H₂SO₄) (Montgomery, 1985).

3. Strong base anion exchange resin

Strong base anion resins are made of plastics or polymers which are reacted with amine or ammonium compound groups. Two types of strong alkaline resins that are often used in water or wastewater treatment are resins that have three methyl groups as follows:

$$CH_2$$

 $|$
 $(R - N - CH_2)^*$
 $|$
 CH_2

Another type of strong base anion resin is a resin that has an ethanol group placed on one of the methyl groups as follows:

Strong base anion resins are resins that are often used to pick up ions that are negatively charged. In operation this strong base anion resin can be operated under hydroxide conditions (R^+ .Cl⁻). If the strong base anion resin is operated under hydroxide conditions (R^+ .OH⁻), then this strong base anion resin can take up almost all types of negative ions and in the regeneration process it uses sodium hydroxide solution (NaOH), whereas if the strong base anion resin is operated under conditions (R^+ .Cl⁻), then the negative ions that can be taken, such as sulfate and nitrate, and in the regeneration process use a salt solution (NaCl) (Montgomery, 1985).

4. Weak base anion exchange resin

Weak base anion resins are used to remove acids such as hydrochloric acid (HCl) or sulfuric acid (H₂SO₄) so that the resin is known as acid adsorbers. The regeneration process of this weak base anion resin uses a solution of sodium hydroxide (NaOH), ammonium hydroxide (NH₄OH) or sodium carbonate (Na₂CO₃) (Montgomery, 1985). The important characteristics expected from an ion exchanger are large uptake (capacity), large selectivity, large exchange rates, resistance to temperature, resistance to ion exchangers that have been loaded easily, because ion exchange is a process very reversible (Bernasconi, Gerster, Stauble, & Scheiter, 1995).

3.4.4 Properties of Ion Exchange Resin

Resins as ion exchange media have certain properties and advantages. The properties of a good resin are as follows:

- 1. Resin has a strong cross-linking capacity which can remove a certain number of ions
- 2. Resin with a small particle size will be better, because it requires a large contact area
- 3. Resin has stability that can be used for a long time, not easily worn/damaged in regeneration (Prayoga, 2008).

3.5 Process Stage on Ion Exchange

3.5.1 Service Stage

The service stage is the stage where the ion exchange reaction occurs. The nature of the service stage is determined by the concentration of the removed ion over time, or the volume of product water produced. What matters in the service sector are capacity (theoretical and operating) and ion exchange load. The theoretical exchange capacity is defined as the theoretical number of ions that the resin union mass or volume of resin can exchange. Operating capacity is the actual resin capacity used for exchange reactions under certain conditions (Setiadi, 2007).

The ion exchange load is the weight of the ion removed during the service stage and is obtained from the product between the volume of water

treated during the service stage and the ion concentration removed (Setiadi, 2007). This service stage is carried out by draining the feed water from above.



Figure 3. 1 Demineralization Process

3.5.2 Backwash Stage

The backwash stage is carried out if the resin's ability is saturated or cannot exchange ions anymore. Washing using product water. The purpose of doing backwash as follows

- Breaking / separating the lumpy resin,
- Removal of fine particles trapped in the space between the resins,
- Removal of gas pockets or chambers in the bed, and
- Reshaping the resin layer to make it fluidized (Setiadi, 2007).

3.5.3 Regeneration Stage

The regeneration stage is the operation of replacing the adsorbed ion with the initial ion that was originally in the resin matrix and taking the capacity to the initial level or to the desired level. The regeneration solution must be able to produce a peak point (returns the regeneration time and the amount of solution used). If all systems can be returned to their original exchangeability, the equivalent of the ion replaced must be the same as the ion removed during the service stage. So theoretically, the amount of regeneration solution (in equivalent) must equal the number of ions (in the equivalent) removed (required for the theoitic regeneration solution). The regeneration operation so that the resin has its original capacity is very expensive, therefore regeneration is only done to produce a portion of the initial exchangeability.

The regeneration efficiency of the strong acid cation exchange resin regenerated with H_2 strong base anions regenerated with NaOH is between 20-50%, therefore the use of regeneration solutions is 2-5 times greater than the theoretical requirement. Regenersion operation is carried out by draining the regeneration solution from above, by injecting the regenerant for cations is HCl and for anions is NaOH. Regeneration process as follows:

- Backwash, which is multiplying clean water in the opposite direction through the cation or anion tank until the output water is clean.
- Doing a slow rinse, which is flowing water slowly to produce regenerants in resin.
- Fast rinse i.e. rinsing the unit at a faster rate to remove regenerant prior to operation (Setiyadi, 2014).

3.5.4 Rinse Stage

The rinsing stage is carried out to remove the remaining regeneration solution trapped by the resin, the rinsing is carried out using product water with down flow and is carried out in 2 levels, namely:

- 1. Low flow rate for removal of the regeneration solution, and
- 2. High flow rate to remove residual ions.

The low flow rate rinsing wastes are combined with the saline solution and disposed of, while the high flow rate rinsing wastes are stored and used as compound solvents for regeneration (Setiyadi, 2014).

3.6 Types of Silica Analysis Methods

Silicon does not occur freely in nature, but as free silica (SiO₂) in coarse crystals (quartz, rock crystals, amethyst, etc.) and microcrystals (flint, chert, jasper, etc.) Types of quartz, the main component of sand. and sandstone. Silicon is found in combination with other elements in silicates, which are represented by feldspar, hornblende, mica, asbestos, and other clay minerals. Silicates also occur in rocks, such as granite, basalt, and shale. Therefore, silicon is usually reported as silica (SiO₂) when rock, sediment, soil, and water are analyzed. The average silica abundance in various rock types is 7 to 80%, in typical soils 50 to 80%, and in surface and groundwater 14 mg/L. The common forms of aqueous silica are H₄SiO₄ and H₃SiO₄⁻. In the presence of magnesium, it can form scale deposits in boilers and steam turbines

3.6.1 Molybdosilicate Method

Principle: Ammonium molybdate at a pH of about 1.2 reacts with silica and any phosphate present to produce heteropoly acids. Oxalic acid is added to destroy molybdophosphoric acid, but not molybdosilicic acid. Although phosphates are known to be absent, addition of oxalic acid is highly desirable and is a mandatory step in this method and the blue heteropoly method. The intensity of the yellow color is proportional to the concentration of "molybdate-reactive" silica. In at least one of its forms, silica does not react with the molybdate although it is able to pass through the filter paper and is less cloudy. It is not known to what extent "unreactive" silica occurs in the waters. Terms such as "colloid", "crystalloidal", and "ionic" have been used

to distinguish various forms of silica, but these terms have not been verified. Silica "molybdate unreactive" can be converted into "reactive-molybdate" by heating or melting with lye Molybdate-reactive or unreactive does not imply reactivity, or lack of it, toward other reagents or processes.

The procedures in the molybdosilicate method include

- a. Color development: To 50.0 mL sample add in rapid succession 1.0 mL 1 1 HCl and 2.0 mL ammonium molybdate reagent. Mix by inverting at least six times and let stand for 5 to 10 min. Add 2.0 mL oxalic acid solution and mix thoroughly. Read color after 2 min but before 15 min, measuring time from addition of oxalic acid. Because the yellow color obeys Beer's law, measure photometrically or visually
- b. To detect the presence of molybdate-unreactive silica, digest sample with NaHCO₃ before color development. This digestion is not necessarily sufficient to convert all molybdateunreactive silica to the molybdate-reactive form. Complex silicates and higher silica polymers may require extended fusion with alkali at high temperatures or digestion under pressure for complete conversion. Omit digestion if all the silica is known to react with molybdate. Prepare a clear sample by filtration if necessary. Place 50.0 mL, or a smaller portion diluted to 50 mL, in a 100-mL platinum dish. Add 200 mg silica-free NaHCO₃ and digest on a steam bath for 1 h. Cool and add slowly, with stirring, 2.4 mL 1N H₂SO₄. Do not interrupt analysis but proceed at once with remaining steps. Transfer quantitatively to a 50-mL nessler tube and make up to mark with water. (Tall-form 50-mL nessler tubes are

convenient for mixing even if the solution subsequently is transferred to an absorption cell for photometric measurement.)

- c. Preparation of standards: If NaHCO₃ pretreatment is used, add to the standards (approximately 45 mL total volume) 200 mg NaHCO₃ and 2.4 mL 1N H₂SO₄, to compensate both for the slight amount of silica introduced by the reagents and for the effect of the salt on color intensity. Dilute to 50.0 mL.
- d. Correction for color or turbidity: Prepare a special blank for every sample that needs such correction. Carry two identical portions of each such sample through the procedure, including NaHCO₃ treatment if this is used. To one portion, add all reagents as directed in ¶ a above. To the other portion, add HCl and oxalic acid but no molybdate. Adjust photometer to zero absorbance with the blank containing no molybdate before reading absorbance of molybdate-treated sample.
- e. Photometric measurement: Prepare a calibration curve from a series of approximately six standards to cover the optimum ranges cited in Table 4500-SiO₂:I. Follow direction of ¶ a above on suitable portions of standard silica solution diluted to 50.0 mL in nessler tubes. Set photometer at zero absorbance with water and read all standards, including a reagent blank, against water. Plot micrograms silica in the final (55 mL) developed solution against photometer readings. Run a reagent blank and at least one standard with each group of samples to confirm that the calibration curve previously established has not shifted

| | | $\frac{4500\text{-}\mathrm{SiO}_2\text{-}\mathrm{D}}{\text{Silica in 55 mL Final Volume}}$ | | | |
|---------------------|---------------------------------------|--|----------------------|--|--|
| | 4500-SiO ₂ .C | | | | |
| Light Path cm | Silica in 55 mL Final Volume µg | 650 nm Wavelength | 815 nm Wavelength | | |
| 1 | 200-1300 | 40-300 | 20-100 | | |
| 2 | 100-700 | 20-150 | 10-50 | | |
| 5 | 40-250 | 7-50 | 4-20 | | |
| 10 | 20-130 | 4-30 | 2-10 | | |

| TABLE 4500-SiO ₂ .I. | SELECTION | OF LIGHT | Ратн | LENGTH | FOR | VARIOUS |
|---------------------------------|-----------|----------|------|--------|-----|---------|
| | Course Co | | | | | |

 Table 3. 1 Selection of Light Path Length For Various Silica

 Concentration

f. Visual comparison: Make a set of permanent artificial color standards, using K_2CrO_4 and borax solutions. Mix liquid volumes specified in Table 4500-SiO₂:II and place them in wellstoppered, appropriately labeled 50-mL nessler tubes. Verify correctness of these permanent artificial standards by comparing them visually against standards prepared by analyzing portions of the standard silica solution. Use permanent artificial color standards only for visual comparison.

| TABLE 4500-SIO ₂ :II. PREPARATION OF PERMANENT COLOR STANDARDS FOR VISUAL DETERMINATION OF SILICA | | | | | | |
|---|---|-------------------------|-------------|--|--|--|
| Values in Silica µg | Potassium Chromate Solution mL | Borax Solution mL | Water mL | | | |
| 0 | 0.0 | 25 | 30 | | | |
| 100 | 1.0 | 25 | 29 | | | |
| 200 | 2.0 | 25 | 28 | | | |
| 400 | 4.0 | 25 | 26 | | | |
| 500 | 5.0 | 25 | 25 | | | |
| 750 | 7.5 | 25 | 22 | | | |
| 1000 | 10.0 | 25 | 20 | | | |

 Table 3. 2 Preparation of Permanent Color Standards For Visual

 Determination of Silica

3.6.2 Heteropoly Blue Method

Principle: The principles outlined in the molybdosilicate method also apply to this method. Yellow molybdosilicic acid is reduced with aminonaphtholsulfonic acid to heteropoly blue. The blue color is more intense than the yellow color of the molybdosilicate method giving increased sensitivity.

The procedures for the blue heteropoly method include:

- a. Color development: Continue as in the first molybdosilicate method procedure up to and including the words, "Add 2.0 mL oxalic acid solution and mix thoroughly." Measuring time from the moment of adding oxalic acid, wait at least 2 min but not more than 15 min, add 2.0 mL reducing agent, and mix thoroughly. After 5 min, measure blue color photometrically or visually. If NaHCO₃ pretreatment is used, follow the second molybdosilicate method.
- b. Photometric measurement: Prepare a calibration curve of a series of approximately six standards to cover the optimal range shown in Table 4500-SiO₂:I. Perform the steps described above on the appropriate portion of the standard silica solution diluted to 50.0 mL in a tube. nessler; pretreat standards if NaHCO₃ digestion is used (see second molybdosilicate method procedure). Adjust the photometer to zero absorbance with distilled water and read all standards, including reagent blanks, for distilled water. If it is necessary to correct for color or cloudiness in the sample, see the fourth molybdosilicate method procedure. To the special blank add HCl and oxalic acid, but no molybdate or reducing agent. Plot micrograms

silica in the final 55 mL developed solution against absorbance. Run a reagent blank and at least one standard with each group of samples to check the calibration curve.

c. Visual comparison: Prepare a series of not less than 12 standards, covering the range 0 to 120 g SiO₂, by placing the calculated volumes of standard silica solution in 50-mL nessler tubes, diluting to mark with distilled water, and developing color as described in ¶ a above (APHA, 2017).

3.6.3 Automated Method for Molybdate-Reactive Silica

Principle: This method is an adaptation of the blue heteropoly method using a continuous flow analysis instrument. The procedure of this method is to adjust the manifold as shown in Figure 4500-SiO2: 1 and follow the general procedure described by the manufacturer. Find the absorbance at 660 nm (APHA, 2017).



Figure 4500-SiO₂:1. Silica manifold.

Figure 3. 2 Silica Manifold

CHAPTER IV

METHODOLOGY

4.1 **Tools and Materials**

4.1.1 Tools

- 1. Beaker Glass
- 2. Nessler tube
- 3. UV-Vis spectrophotometer
- 4. Cuvette
- 5. Timer
- 6. Stirrer

4.1.2 Materials

- 1. Acid reagent
- 2. Citric acid reagent
- 3. Molybdate reagent
- 4. Distilled water
- 5. Reactor cooling water

4.2 **Procedures**

4.2.1 Sample Preparation

Prepare a sample solution in a beaker. First, prepare three beaker glasses, fill each beaker with 1 mL of sample solution. Next, add 2 mL of molybdate reagent to each beaker. Next, do some shaking. Then, add 2 mL of reagent acid and stir with a stirrer for 10 minutes. At last, add the citric acid reagent and stir again with a stirrer for 2 minutes

4.2.2 Measurement of Samples with a UV-Vis Spectrophotometer

Insert the blank into the cuvette and take the absorbance measurement of the blank. Then, put the solution into the cuvette and enter the cuvette into the UV-Vis spectrophotometer. The absorbance measurement was carried out three times (triplo).

CHAPTER V

RESULT AND DISCUSSION

Field Internship that was carried out on February 18 - March 31, 2021 focused on the Study of the Mechanism of the SiO₂ Ion Exchange Process in Ion Exchange Resin. In this case, SiO₂ ions are present in the reactor cooling water.

5.1 Silica in Water

Silica exists in crystalline or amorphous form (Sheikholeslami R B. J., 2002). Amorphous silica is further classified as dissolved, polymeric, colloid, and particulate. Silica in natural water comes from dissolving rocks and minerals (Equation 1). The main dissolving reaction is hydrolysis of the Si-O-Si bonds, resulting in the production of silicic acid (H4SiO4).

 $x.SiO_2(s) + 2H_2O \rightleftharpoons (x-1) SiO_2 + H_4SiO_4$ (Equation 1)

The dissolved silicic acid is also referred to as reactive silica. The term 'reactive' means that the silanol group (Si-O-H) reacts with ammonium molybdate ((NH₄)₂MoO₄) to make a colored complex in quantitative determinations (Ning, 2003). Silicic acid is a weak tetravalent acid with an acid dissociation constant (pKa) of 9.9 (Ning, 2003). Silicic acid will remain in the monomeric state as long as the concentration is less than about 2 mM (Sheikholeslami R T. S., 1999). When the concentration of silicic acid reaches a certain point, it begins to polymerize polymeric silica, colloids and particulates. The common forms of aqueous silica are H₄SiO₄ and H₃SiO₄⁻ (APHA, 2017). The rate of silicic acid polymerization is strongly pH-dependent. Briefly, it is fast in neutral and slightly alkaline solutions, and drops to a minimum at pH above 9.5 and below 6.5, respectively (Sheikholeslami R A.-M. I., 2001).

The reactivity of polymeric silica is lower than that of silicic acid due to less silanol groups available. Polymeric silica with low molecular weight is considered to be unstable, and to have only a transient existence. Highly polymerized silica with a diameter of 5 nm or more are generally referred to as colloidal silica. In practice, the colloidal silica value is determined by the difference between the total and dissolved silica (Ning, 2003). Colloidal silica is nonreactive and not necessarily removable by traditional filtration because of its extremely small size. Finally, particulate silica is usually defined as having a size larger than 1 micron (e.g clays, silts and sand) (Park, Yeon, & Park, 2020).

5.2 Measurement of Silica with a UV-Vis Spectrophotometer

Silica measurement is done by determining the correlation and coefficient of determination for the calibration curve of the SiO₂ standard solution. Series of standard solutions prepared from the silicate stock solution 10 mg/L by adding 0; 5; 10; 20; 30 and 40 mL in 250 mL volumetric flask. Then added with 5 mL ammonium molybdate 5% and aged 4 minutes. Then added 5 mL oxalic acid 5% and allowed to stand 2 minutes. Furthermore, added with 5 mL ascorbic acid 4% and distilled water to the mark. Solution was homogenized and the absorbance was measured by UV-Vis spectrophotometry at 815 nm (Istiningrum, Permatasari, & Iryanto, 2014).

Precision test was carried out by the same procedure to the linearity test by replacing the standard solution to 10 mL industrial water sample. Absorbance measurements performed seven times. Accuracy test procedure used was the same as precision test procedures but 10 mL of the standard solution silicate 10 mg/L added as a spike. Absorbance measurements were also carried out seven times. The absorbance of the reagent blank were measured seven times by the same procedure with precision test but without samples water for determining the detection limit (Istiningrum, Permatasari, & Iryanto, 2014).



Figure 5. 1 SiO2 Standard Solution Calibration Curve(Istiningrum, Permatasari, & Iryanto, 2014).

The results of the measurement of silica using a UV-Vis spectrophotometer showed the coefficient of determination at R^2 = 0.998 and the resulting line equation was y = 0.794x + 0.005. This suggests that there is a close correlation and good linearity between the SiO₂ concentration and its absorbance.

5.3 Results of Analysis at the Multipurpose Reactor Center

Molybdosilicate method is used to analyze silica in water. Prepare a sample solution in a beaker. First, prepare three beaker glasses, fill each beaker with 10 mL of the sample solution. Second, react the molybdate reagent into each beaker. Third, do shaking to make the solution homogeneous. Then, add 2 mL of reagent acid to produce molybdosilicic acid to form complex ions by reducing silicates and stirring with a stirrer for 10 minutes. At last, add the citric acid reagent as a reducing agent and stir again with a stirrer for 2 minutes. The resulting yellow color in solution, the intensity of the yellow color is proportional to the concentration of "molybdate-reactive" silica.

Take measurements of the blank into the cuvette and take the absorbance measurement of the blank. Then, put the solution into the cuvette and enter the cuvette into the UV-Vis spectrophotometer with a wavelength of 452 nm.



Figure 5. 2 DR 2800 Spectrophotometer

Based on Figure 5. 2 that the test results using a UV-Vis spectrophotometer produced a concentration of 23.3 mg/L SiO_2 .

5.4 Mechanism of SiO₂ Ion Exchange Process with Ion Exchange Resin

Ion exchange technology can be used to remove unwanted elements by means of ion exchange which has the same charge sign between water as the raw material and the ion exchange resin in its path. In cation exchange resin, the cations that are bound to the resin will be replaced by cations in the solution that is passed. Likewise in anion exchange resin, the anions that are bound to the resin will be replaced by anions in the solution that is passed (Lestari & Utomo, 2015).

In general, ion exchange resin that contain cation and can be exchanged is called cation resin, while ion exchange resin that contain anion and can be exchanged is called anion resin. Strong acid cation exchange resins operate on the H cycle while the strong base anion exchange resins operate on the OH cycle. Hydrogen cycle strong acid cation exchange resin will convert dissolved salts to acids and strong base anion exchange resin will remove these acids, including silicic acid and carbonic acid. In the water purification process, silica removal aims to reduce the concentration of silica content so that deposits do not occur which can form a scale on the metal surface that contacts the water flowing through the system. The silicate is removed in the demineralization process by means of a strong base anion exchanger in the hydroxide mode (Lestari & Utomo, 2015).

The types of resin used in the Multi-Purpose Reactor Center are Lewatit® MonoPlus S 108 and Lewatit® MonoPlus M 500. Lewatit® MonoPlus S 108 is a strong acid gel type cation exchange resin in the form of Na with uniform sized granules (monodisperse) based on the styrene-divinylbenzene copolymer. Due to a special manufacturing process, this type of resin is highly resistant to chemical, osmotic, and mechanical stress. Meanwhile, Lewatit® MonoPlus M 500 is a highly alkaline gelular anion exchange resin with monodisperse granules based on styrene-divinyl benzene copolymer. Monodispersed beads are chemically and osmotically very stable. The optimized kinetics lead to increased operating capacity compared to ion-exchange resins with a heterodisperse bead size distribution.

The $(SiO_2)_n$ amorphous silica model is not applicable to water treatment because "SiO₂" depolymerizes in water, then hydrolyzes to form silicic acid (Meyers, 1999). The common forms of aqueous silica are H₄SiO₄ and H₃SiO₄⁻ (APHA, 2017). Silicic acid has an acid dissociation constant (Ka) of $2x10^{-10}$. At pH values greater than 10, silica is present as silicate ion, and is quite soluble (Meyers, 1999). Therefore, a strong base anion exchange resin in the form of hydroxide can be applied to remove dissolved silica.



Figure 5. 3 Molecular Structure of Alkyl Trimethyl Ammonium Chloride (Kuzma & Kuper, 2003).

The resin type Lewatit® MonoPlus M 500 has a styrene-divinylbenzenecopolymer chemical structure with a trialkyl ammonium group in the form of chloride in Figure 5. 3. This resin is a type I strong base anion exchange resin because it has a trialkyl ammonium group.



Figure 5. 4 A Small Fraction of A Polystyrene Chain (Naturwissenschaften, 2019).



Figure 5. 5 Schematic Presentation of The General Structure of An Ion Exchange Resin Cross Linked With Divinyl Benzene (DVB) Adapted Fromin Cross Linked With Divinyl Benzene (DVB) (Bilandi & Mishra, 2014).



Figure 5. 6 Schematic Presentation of The General Structure of An Ion Exchange Resin (Srikanth, Rao, & MU., 2010).



Figure 5. 7 Typical Polystyrene Anion Exchange Resin (Cl⁻) (Ali, Rahman, & Alam, 2013).

In Figure 5. 4 until Figure 5. 7 describe that the polymer backbone is usually made of polystyrene, which is cross-linked to a divinyl benzene (DVB) with side chains of ionic functional groups generating the pores between cross-linked chains. The

interlinked chains of the resins give the polymer more stability and a tri-dimensional structure. The higher the amount of cross linking, the more rigid is the polymer structure. The cross links are evenly distributed along the matrix of the ion exchange resins (Bilandi & Mishra, 2014).



Figure 5. 8 Strong-Base Anion Exchanger In The Chloride Form (Im, 2015).



Figure 5. 9 Structure of The Anion Exchange Resin (Lee, Lee, Jung, Chen, & Lim, 2018).

Strong base anion resins have a quaternary ammonium group (NH4⁺) in their structure in the form of Cl⁻ as described in Figure 5. 8. Based on Figure 5. 9 that resins in the form of polymer chains that bind permanently, anion exchange resins have positive bonds and vice versa. Fixed coion binding is an ion that can and will be exchanged, so that if there is a mobile counterion, it will enter the resin and will be placed in the ion in the fixed coion. The mobile counterion is left in the resin and the ions present in the fixed coion are pushed out by the flow as ions to be exchanged or attached. This is in accordance with the law of equilibrium where the exchange is complete until the ions available in the state remain replaced by the mobile counterion.

Two basic components of an ion exchange resin are a cross-linked polymer matrix and charged functional groups. The exchanger resins have charged functional groups attached to this matrix by covalent bonding (Im, 2015). These mobile counter ions are attached to each of the charged functional group by electrostatic attraction to remain electroneutrality at all times not only within the exchange material, but also in the aqueous solution. This electroneutrality can be maintained because ions are exchanged on an equivalence basis, so they do not degrade or change form during the resin usage (Wachinski, 2005).



Figure 5. 10 Reaction of Resin with Silica Ions (Meyers, 2004).

Silica acts as a very weak acid, so the removal of silica is carried out specifically from a strong alkaline anion exchange resin with a high pH in the form of chloride. When the pH is relatively low, silica is non-ionic and when the pH is relatively high, silica becomes ionized. In fact, silica it almost always present in both non-ionic and ionized forms; however, the ratio of un-ionized to ionized silica increases with decreasing pH (Koji Yamanaka, 2019). The exchange reaction for silica removal is shown in Figure 5. 10.

Binding of chemical elements using ion exchange resins has been carried out to purify water to become mineral-free water in demin I water, demin II water, raw water, cooling water, and pump outlets (AP001, AP002, AP003). One of the uses for strong base anion exchange resins is to bind SiO_2 ions. This is done to reduce the content of silica ions in the water.

| NO | Date | Parameter | Der | nin | Raw Water | Vater Cooling Water | Outlet Pump | | |
|----|------------|-----------|------|------|-----------|---------------------|-------------|-------|-------|
| | | | Ι | П | | | AP001 | AP001 | AP003 |
| 1 | 30/12/2020 | Silica | 26,7 | 26,4 | 21,8 | 235,5 | 235 | 167,5 | |
| 2 | 10/01/2021 | Silica | | | 22,6 | 236,5 | 175 | 235,5 | |
| 3 | 23/02/2021 | Silica | | | 23,2 | 187 | 173,5 | | 182,5 |
| 4 | 08/03/2021 | Silica | 42,7 | 42,4 | 22,3 | 233 | 231,5 | | 227,5 |

Table 5. 1 Silica (SiO₂) Measurement Data in Water

From the Table 5. 1 of silica (SiO₂) measurement data in water, it can be seen that the strong base anion exchange resin on silica (SiO₂) did not have a significant decrease. In demin I water there was an increase of 28,7 mg/L to 42,7 mg/L and in demin II water there was an increase in silica of 26,4 mg/L to 42,4 mg/L. Changes in the amount of silica in raw water (21,8 mg/L; 22,6 mg/L; 23,2 mg/L; 22,3 mg/L), cooling water (233,0 mg/L; 187,00 mg/L; 236,50 mg/L; 235,50 mg/L), AP001 outlet pump (235,00 mg/L; 175,00 mg/L; 173,50 mg/L; 231,50 mg/L), AP002 outlet pump (235, 50 mg/L dam 167,50 mg/L), and AP003 outlet pump (182,50 mg/L and 227,50 mg/L) were also insignificant.

The ion exchange resin can be modified to reduce the silica content. One modification is the gallic acid type resin (GA type resin) made by the grafting method. GA type resins absorb monosilicate acid, silicate ion, and polymer silicic acid. The absorption capacity of the GA type resin is 30-40 greater than that of the OH-type resin and the adsorption time is short. The removal efficiency of silicic acid is almost unaffected by pH and common anions when the common anion and silicic acid content

are similar, proving that the GA-type resin shows excellent performance for the selective adsorption of silicic acid. The adsorption mechanism of silicic acid in GA type resins involves a dehydration reaction of hydroxyl groups in silicic acid and gallic acid. GA type resins can be regenerated efficiently and reused after treatment with HCl solution (Bai, Han, & Ding, 2019).

The strong-base anion-exchange resin (10 g) was added into the gallic acid solution (500 mL, 10 g/L, pH 7) and was stirred for 24 h. The resins that bound gallic acid were filtered with a qualitative paper filter, rinsed with water, and air dried at ambient temperature for 3 days and stored in a dryer. The concentration of gallic acid in the filtrate was determined by UV-Vis spectrophotometer under 260 nm. Then the amount of gallic acid on the resin was calculated by the difference in the gallic acid concentration between initial solution and the filtrate and was estimated to be 725.3 mg/g (dry). (Bai, Han, & Ding, 2019).



Figure 5. 11 Reaction of the Resin with Silicic Acid (Bai, Han, & Ding, 2019).

The surface of the grafted resin has outward facing hydroxyl groups that can interact with silicic acid molecules to form gallate–silicate complexes and attach silicic acid to the resin. It is shown in Figure 5. 11

CHAPTER VI

CLOSING

From the results of the treatment at the Multipurpose Reactor Center and the results of literature studies, it was found that various forms of silicic acid in water, among others; silica monomers, silica polymers, and silica granules or particulates. The various forms of silicic acid in water depend on the pH concentration. Silica which has low solubility and tends to have little interaction with water molecules. The reaction mechanism of silica in water occurs as follows

$$x.SiO_2(s) + 2H_2O \rightleftharpoons (x-1)SiO_2 + H_4SiO_4$$

The effect of silica in reactor water can cause scale on metal surfaces that contact the water flowing through the system. With this, the resin is used to capture the silica

$$RX-Cl + H_4SiO_4 \rightarrow RX-H_3SiO_4 + H^+ + Cl^-$$

However, the bonding of silica in water does not occur properly. To improve the quality of the good resin, resin modification can be done, namely with gallic acid type resin because this resin can bind 30 - 40 greater silica acid in a short time compared to OH-type resins.

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ATTACHMENT

- Secondary cooling tower



Water reservoir

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- Primary cooling water

