OPTIMIZATION OF THE UTILIZATION OF NAOH & HCl FOR AQUA-DEMINERALIZATION RESIN REGENERATION

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Abstract

The demineralization process of water using ion exchange resin is widely applied in the water treatment industry to produce pure water. However, the performance of ion exchange resin tends to decrease over time due to the accumulation of ions adsorbed on its surface. Therefore, resin regeneration is important to restore the resin's ability in the demineralization process. This study aims to optimize the regeneration method of aqua-demineralization resin using a combination of NaOH and HCl as regeneration materials. The variables tested include the concentration of NaOH and HCl solutions and the contact duration. The results of this study indicate that the combination of NaOH and HCl can increase the regeneration efficiency of aqua-demineralization resin, with optimal conditions at a concentration of 4% NaOH and 4% HCl, and regeneration duration of 30 minutes.

Keywords: Regeneration, Aqua-demineralization, Resin, NaOH, HCl, Water Treatment.

INTRODUCTION

Water is crucial for life, regardless of whether it is pure or has minerals. While mineral water is important for human intake, excessive mineral levels can pose health risks (Quattrini et al., 2016). The influence of minerals on industrial chemical processes is quite intricate, potentially leading to scale accumulation in heating operations like boilers and heat exchangers, decreasing yield and selectivity during reactions, among other impacts (Amin et al., 2024; Pezzuto et al., 2017). For both laboratories and the chemical process industry, water that is free from minerals is vital. Water can be deemed mineral-free if it complies with specified quality standards.

Aquademin is water that is free of ions or minerals. Demineralization of water is a process of removing the anion cations contained in it. Mineral content as an anion cation in water in macro form includes: Na^+, Ca^{2+}, Mg^{2+}, K^+, Fe^{3+}, Cl^-, $SO4^{-2}$, and CO_3^{-2} (Du Plooy & Pillay, 2015; El-Ghzizel et al., 2018). Demineralized water is produced from clean water containing ions, which undergoes a purification process that can be executed through methods involving heating or not. The approach to create demineralized water using heat is carried out via distillation (Gryta, 2018). Non-heating methods for producing demineralized water can be achieved through

gradual filtration, reverse osmosis membranes, adsorption, electrodialysis, ion exchange, or a combination of these techniques (Dammak et al., 2021; Gryta, 2018). In this research, demineralized water was generated through ion exchange operations. This process offers several benefits over other methods, such as reduced operational costs.

In the mineral-free water system, the ion exchange resin serves to eliminate undesirable impurity ions through an ion exchange reaction that occurs between the raw water and the resin, both carrying the same charge. The process for producing mineral-free water within pharmaceutical industry involves raw water passing through a series of ion exchange resin stages, which include a carbon tank (column), a 5μm filter, a cation exchange resin tank (column), an anion exchange resin tank (column), a mixed bed resin tank (column), and a 1μm filter.

The ion exchange resin has a limited capacity to capture impurity ions from the water, so after a certain duration, it becomes incapable of adsorbing these ions (referred to as saturated). Saturation occurs when both the cation exchange and anion exchange resins can no longer remove impurities from the water (Mangarengi et al., 2022; Nunes & Mulvaney, 2021; Ramzan et al., 2012). In the mineral-free water system at a pharmaceutical industry, saturation is defined by a conductivity level of ≥ 30 μS/cm at the outlet of the anion exchange resin column. Therefore, to sustain the efficiency of the mineral-free water system, it is necessary to regenerate the ion exchange resin. Regeneration involves using HCl solution for the cation exchange resin and NaOH solution for the anion exchange resin. By regenerating the ion exchange resin, it is anticipated that the resin's capacity to absorb impurities in raw water will be restored, ensuring that the quality of the water produced by the mineral-free water system meets the established specifications.

This study aims to optimize the regeneration method of aquademineralized resin using a combination of NaOH and HCl solutions. The focus of this study is to determine the effect of variations in concentration and regeneration duration on resin regeneration efficiency.

RESEARCH METHODS

This research was conducted to obtain a regeneration method that can be used effectively and efficiently and to obtain the optimal concentration of NaOH and HCl for the regeneration of demineralized aqua with the output of demineralized water meeting the requirements used.

Research Materials and Equipment

The materials used in the research are sodium hydroxide (NaOH; 98%), hydrochloric acid (HCl, 37%), and source water. In this study, an aqua-demineralization regeneration unit of PT SP was used, consisting of a carbon tube, 5 μm Filter, cation resin tube, anion resin tube, 1 μm filter, flow meter, conductivity meter reading sensor and aqua-demineralization storage tank.

Research Variables

The variables used in this study are comprised of fixed and independent variables. The fixed variables consisted of volume of cation resin tube (56.7 liters), anion resin tube volume (56.7 liters). Moreover, the independent variables are the concentration of 98% NaOH solution (1%, 2% and 3%), the concentration of HCl solution 37% (4%, 5% and 6%), the residence time (contact time) of the NaOH solution in the Anion resin tube (10, 20, and 30 minutes) as well as

the residence time (contact time) of the HCl solution in the Cation resin tube (10, 20, and 30 minutes).

Response of the Research

Water conductivity value was assessed to determine the conductivity value of water before and after regeneration using the ion exchange method. The pH value of water is used to determine the pH value of water before and after regeneration using the ion exchange method. Water meter is used to determine the volume produced from the regeneration of aqua-demineralization by using each variable used.
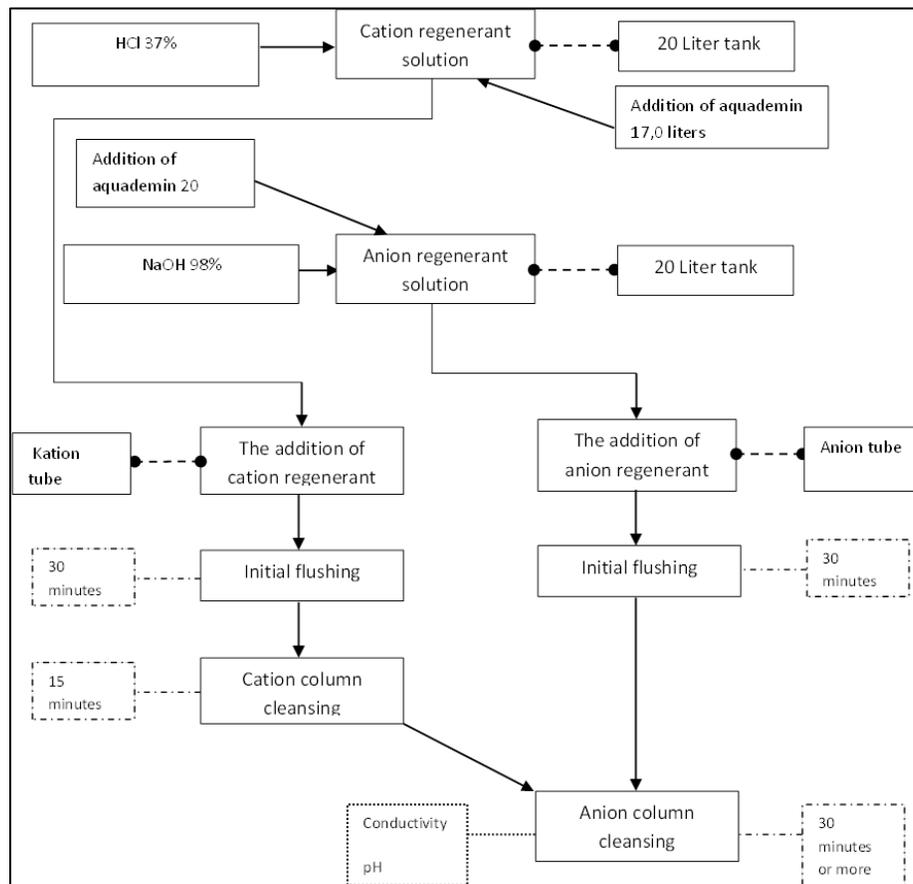

Figure 1. The Flow Chart of the Regeneration of the Aqua-demineralization Unit

Research Procedures

The regeneration process is carried out if routine inspections show that the resulting aqua-demineralization does not meet the requirements (Water conductivity > 30 μs/cm). During the regeneration process, the water hose and other tools used must be specifically for regeneration and not used for anything else. The HCl solution is injected into the HCl tank, and then the HCl tank hose is connected to the solution input tap on the cation tube. The same action is subjected to the NaOH solution which is feed into the anion tube. The input tap for the cation solution

and the input tap for the anion solution are both opened and subjected into the resins. The residence time (contact time) of the NaOH solution in the anion resin tube and the HCl solution in the cation resin tube are varied. After the regeneration process completed, the process is followed with the rinse and flushing process of both anion and cation resins (Figure 1). The output water than being collected and measured for its conductivity and pH values of the water, with the condition that the water conductivity is ≤ 30 µs/cm and the pH is 5 - 7.

RESULTS AND DISCUSSION

The results of the data obtained to determine the effectiveness and efficiency of the regeneration method in the PT SP water demineralization system are presented in the Table 1-4. In the demineralized water production process at PT SP, softwater is first pumped through an activated carbon filter column to remove chlorine and remove foreign color, taste, and odor. It is collected in a filtered water tank and then passed through a cation exchange resin column and anion exchange resin column. The conductivity values of the softwater at the input of the cation exchange resin and anion exchange resin are not measured. The conductivity values of the output of the cation exchange resin and anion exchange resin are controlled at a limit of ≤ 30 µs/cm.

Aquademin regeneration is carried out before the testing process on anion and cation resins. Regeneration for anion resins uses NaOH because the anion resin used is a weak base anion exchange resin so it usually uses high concentration sodium hydroxide (for example, 4-5% NaOH solution (weight per volume, w/v)) at a flow rate (Goreng et al, 2016), while regeneration of cation resins uses 35% HCl. According to (Prayitno and Sardjono, 2002), regeneration using HCl is more effective, namely 70-85% when compared to H_2SO_4.

Table 1. The pH and conductivity of the water obtained from the regeneration at different concentration of HCl with NaOH concentration of 2%

Concentration of the HCl with 2% of NaOH	pH		Conductivity (µs/cm)		Water flow rate (m^3)		
	Initial	Final	Initial	Final	Initial	Final	Delta
HCl 4%	6,64	7,22	32,3	9,8	16340,99	16343,51	2,51
HCl 5%	6,43	7,85	34,1	6,8	16343,51	16345,94	2,42
HCl 6%	4,63	7,82	33,9	12,3	16345,94	16348,82	2,88

Based on the analysis data, the concentration of NaOH and HCl solutions can affect the regeneration process. Therefore, research was conducted on concentrations with several variables to determine the optimal concentration for the aquademin regeneration process.

The results of the regeneration process with the first variable, namely a fixed NaOH concentration of 2% and HCl concentrations of 4%, 5%, and 6%. Based on the results of the

analysis and observations listed in Table 1, the water discharge produced is the least for the variable of 2% NaOH with 5% HCl, namely 2,426m³, while the water discharge produced was the highest for the variable 2% NaOH with 6% HCl, namely 2,884 m³. Based on these results, the water discharge produced with a conductivity limit of ≤ 30μs/cm, the difference is not too significant.

Table 2. The pH and conductivity of the water obtained from the regeneration at different concentration of HCl with NaOH concentration of 3%

Concentration of the HCl with 3 % of NaOH	pH		Conductivity (μs/cm)		Water flow rate (m³)		
	Initial	Final	Initial	Final	Initial	Final	Delta
HCl 4%	5,05	6,61	32,0	9,5	16353,124	16356,17	3,054
HCl 5%	4,63	7,20	31,2	10,1	16356,178	16359,38	3,210
HCl 6%	5,15	6,76	32,2	9,1	16359,388	16363,00	3,620

The results of the regeneration process with the second variable, namely a fixed NaOH concentration of 3% and HCl concentrations of 4%, 5%, and 6% was tabulated on Table 2. Based on the results of the analysis and observations listed in Table 4.2, the water discharge produced was the least for the variable NaOH 3% with HCl 4%, namely3,054 m3,while the water discharge produced was the highest for the variable NaOH 3% with HCl 6%, namely3,620 m3. Based on these results, the water discharge produced with a conductivity limit of ≤ 30μs/cm, the difference is not too significant.

Table 3. The pH and conductivity of the water obtained from the regeneration at different concentration of HCl with NaOH concentration of 4%

Concentration of the HCl with 4 % of NaOH	pH		Conductivity (μs/cm)		Water flow rate (m³)		
	Initial	Final	Initial	Final	Initial	Final	Delta
HCl 4%	5,16	7,09	32,1	9,2	16363,008	16366,71	3,709
HCl 5%	5,71	7,65	32,6	8,5	16366,717	16370,23	3,522
HCl 6%	6,15	7,86	33,2	10,1	16370,239	16373,65	3,420

The results of the regeneration process with the fixed NaOH concentration of 4% and HCl concentrations of 4%, 5%, and 6% was tabulated on Table 3. Based on the results of the analysis

and observations listed in table 4.3, the water discharge produced was the least for the variable of 4% NaOH with 6% HCl, namely3,420 m3,while the water discharge produced was the highest for the variable 4% NaOH with 4% HCl, namely 3,709 m³. Table 3 shows that according to (Goreng et., al., 2016) regeneration for anion resin uses NaOH because the anion resin used is a weak base anion exchange resin so that it usually uses sodium hydroxide with a NaOH solution concentration of 4-5% (weight per volume, w/v). So that the variable of 4% NaOH with 4% HCl is the variable that produces the most water discharge while maintaining conductivity ≤ 30μs/cm, namely with a water discharge of 3,709 m³.

Based on the results of the regeneration process using variable NaOH with variable HCl on anion and cation resins, the various treatments showing effective results. The decrease in conductivity to water occurs because the solution has been replaced by hydrogen and hydroxide ions from anion and cation resins (Pismenskaya et al., 2020; Ramzan et al., 2012), so that in the results of the regeneration process with various variables there are results of decreasing and increasing water discharge, this is because the resin is saturated so it needs to be regenerated and several factors that affect ion exchange in the aqua-demineralization system, including pH, water flow rate, dissolved ion concentration, height of ion exchange media and temperature. Ion exchangers that decompose ionogenic groups are pH-independent; some are strongly influenced by pH, depending on the strength of the acid or base.

Phenolic or carboxylic acid OH groups do not decompose at low pH, so their exchange capacity is optimal only at alkaline solutions. The effective pH range for ion exchangers for strong acid cations is 0–14. Flow rate affects the ion exchange process. The faster the flow rate set in the ion exchange process, the lower the concentration of ions that can be exchanged. This is due to the shorter residence time and contact time between the seawater and the resin. The higher the concentration of ions to be exchanged, the slower the ion exchange reaction will occur, and the lower the concentration of ions to be exchanged (Cho et al., 2019; Nagy et al., 2016). This is because the resin has a limited ion capacity. The higher the ion exchange media contained in the exchange column, the more ion concentration will be exchanged. This is because the higher the resin, the greater the amount of resin. Ion exchange is affected by temperature, but in practice, increasing the temperature is not sufficient to increase the process rate. High-temperature operation is only useful if the original solution is at that temperature or if the solution is too viscous at room temperature.

Table 4. The pH and conductivity of the water obtained from the regeneration at different contact time

Contact time (minutes)	pH		Conductivity (μs/cm)		Water flow rate (m³)		
	Initial	Final	Initial	Final	Initial	Final	Delta
10	5,23	7,25	32,3	8,2	16379,65	16382,08	2,428
20	5,16	7,34	31,6	9,1	16382,08	16385,55	3,463

| 30 | 5,14 | 7,15 | 32,1 | 8,7 | 16385,55 | 16389,96 | 4,412 |

Based on the analysis data in Table 4, according to the contact time of the regeneration solution to the resin, the longer the contact time, the more perfect the regeneration process will be. The process of the contact time of the regeneration solution to the resin, the water discharge produced is the least at a contact time of 10 minutes, namely 2.428 m^3, while the water discharge produced is the most at a contact time of 30 minutes, namely 4.412 m^3.

Hence, based on Table 4, the contact time of NaOH solution with anion resin and HCl solution with cation resin that produces the most water discharge is a contact time of 30 minutes. The contact time carried out in this study takes into account the production process of liquid preparations that continuously require aquademin to be reprocessed into RO water, so a contact time of 30 minutes is considered an effective contact time for the regeneration of anion and cation resins.

The contact time of the NaOH solution with the anion resin and the HCl solution with the cation resin was carried out because the process of inserting the solution was carried out by flowing it into each resin tube and without stirring, so it had to be left to stand (contact time) first before rinsing so that the NaOH solution and HCl solution were completely absorbed by each resin.

CONCLUSION

The research results show that the concentration of the regeneration solution in the demineralized water treatment process is 4% HCl for cation resin and 4% NaOH for anion resin, the optimal contact time in the regeneration process is 20-30 minutes, and the flow of the regeneration solution should not be too fast so that the regeneration process produces optimal results.

It can be suggested also for the the pharmaceutical industry, PT SP, that it is necessary to update the cation resin because the resin has experienced saturation. It is necessary to carry out routine calibration of the conductivity meter on the control indicator, to minimize product results that are outside the standard, It is necessary to carry out periodic work procedure updates, monitoring the use of resin and chemical solutions for resin regeneration.

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