



Effect of Variation of Adsorbent and pH Doses on Boron Adsorption Using DMAPAA-co-DMAPAAQ Hydrogel



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Abstract

Boron is very dangerous for living things. The boron concentration allowed for drinking water is less than 1 mg/L. If not standard, boron causes nausea, lethargy, diarrhea, vomiting, dermatitis, and a risk of miscarriage in pregnant women. Thus, this research investigated the effect of variations in adsorbent dosage and pH on boron adsorption using the DMAPAA-co-DMAPAAQ hydrogel. The research began with the hydrogel synthesis process, which continued with the batch-mode sorption study. Based on research results, the higher the adsorbent dosage, the more boron is adsorbed. The highest removal percentage with an adsorbent amount of 0.5 g/L was 19.89% for pH 3, and for an adsorbent dosage of 2 g/L was 19.52% for pH 9. The highest percent removal was at pH 11. The DMAPAA-co-DMAPAAQ hydrogel adsorbent is shown to be environmentally friendly compared to commercial resins because the commercial resins are not biodegradable, making them difficult to recycle, causing more damage to the environment.

Keywords: Adsorbent; Adsorption; Boron; DMAPAA-co-DMAPAAQ

1. Introduction

Many worldwide issues related to a sustainable and equitable economy, life and health improvement, and hunger. In this case, water and energy are two crucial aspects supporting the resolution of these issues. However, some regions of the country still experience water shortages due to inadequate water management, environmental pollution, heavy metal contamination, insufficient infrastructure, and rapid economic growth [1]. Indonesia and Poland are two countries that experience difficulty accessing safe drinking water and affordable water. This happens because of water pollution, particularly boron, which is extremely difficult to remove from water [2]. Although it is categorized a micronutrient, boron is harmful to any living things [3]. The concentration of boron allowed in irrigation water differs depending on the plants the water is given to. In this case, boron concentrations allowed for blackberry and lemon orchards are less than 0.5 mg/L; boron for walnut, plum, pear, and apple should be less than 1 mg/L; boron for sunflower, potato, cotton, and tomato should be less than 2 mg/L; and boron for asparagus, palm, bean, and onion should be less than 4 mg/L [4][5]. Furthermore, high boron-polluted water and boron-containing vegetable consumption obtained that the contamination may cause various diseases, including nausea, lethargy, diarrhea, vomiting, dermatitis, intellectual and physical issues in children, and a risk of miscarriage in pregnant women [6][7]. As a result, the World Health Organization (WHO) suggests 2.4 mg/L, and the European Union (UE) suggests 1 mg/L of boron allowed in drinking water [8][9].

Boron has never been encountered in its elemental form. In contrast, boric acid ($B(OH)_3$ or H_3BO_3) and its salts (borates) or boro-silicate are forms of boron mostly found in nature [10]. In this case, boric acid is a very weak Lewis acid (pK_A of 9.2 at 298 K), which dissolves easily when it is present in the water, thus accepting electrons according to reaction rules (1):



When a specific solution has a pH of more than 9.2, the anionic form dominates, yet when the pH is less than 9.2, the neutral form overcomes. The pK_A value of boric acid is strongly affected by the strength of the solution ionic. In practice, when strong electrolytes are present, borate is more likely to form. When total boron concentrations do not exceed 0.025 M, the boron species are usually in H_3BO_3 form [10]. Boric acid is a weak electrolyte with a pK_a value

of approximately 9.2 at 25°C and is electrically neutral [11][12][10]. This value shows that when the pH is low (below 9), the boric acid presents, yet when the pH is high (above 9), the monoborate anion presents, and the solution is diluted.

In recent decades, the remediation of boron-contaminated water has become more popular [13], leading to the development of various deboronation technologies such as membrane-based technology, adsorption [14][15][16], (electro) coagulation [4][17][18][19]. Reverse osmosis and membrane distillation [20][21], ultrafiltration, electrodialysis, and donan dialysis are examples of membrane-based technologies that remove boron through different techniques. Chemical precipitation and electrochemical precipitation are two types of coagulation. Chelating functional groups, layer double hydroxides (LDHs), and metal oxide/metal hydroxides are all used in the boron removal procedure. Adsorption membrane filtration (AMF) and polymer-enhanced ultrafiltration (PEUF) [10] are also included in the hybrid process. In this case, adsorption is one of the methods using boron-selective adsorbents to process solutions which contain boron at very low concentrations [16]. Because of its excellent efficiency in removing boron, ease of operation, and amazing regeneration capability, this method is considered the most effective technology to remove boron.

Traditional sorbents, including activated carbon [22][23], fly ash [24][25][26] and minerals [27] have been used to investigate boron adsorption performance. However, these sorbents have a limited boron adsorption capability because boron can rarely establish complexation on their surfaces [16]. This is the reason typical adsorbents are ineffective in removing boron from wastewater. In addition, chelation reaction has also been used to produce a complex ligand of adjacent hydroxyl groups, making it more effective for boron removal. In this reaction, hydroxyl group orientation is critical in maintaining the borate ester complex generated during separation. Regeneration can be done several times on the chelating functional groups without any significant loss of the adsorption capacity [10].

Chelating resins are structures supported by polymers whose surface is attached by certain effective boron. Numerous research projects have been carried out in studying polymers containing polyhydroxyl groups, including sorbitol [28], mannitol [29], and N-methyl-D-glucamine (NMDG) as boron adsorbents. Related to this, Ting et al. have developed a new adsorbent that can selectively adsorb boron through radiation-induced grafting of vinyl benzyl chloride onto nylon-6 fibers and then conducted functionalization using NMDG [30]. In addition, Jung et al. further conducted synthesis on a boron sorbent comprising of poly(glycidyl methacrylate) chains on porous polystyrene-based beads surface by employing atom transfer radical polymerization (ATRP) that has been previously controlled, followed by reaction with boron-selective NMDG. It is further important to note that in the case of this study, there is no direct relationship between NMDG grafting yield and the polymer surface employed. In addition, the pore volume dropped as the grafting chain became longer, leading to decreased boron adsorption [31]. Furthermore, Wang et al. also carried out a relevant study to remove boron by synthesizing poly(amic acid)(PAA) electrospun nanofiber membranes grafted with hyperbranched polyethylenimine (H-PEI) and glycidol to insert vicinal hydroxyl groups. In this study, the high-surface-area of PAA-g-PG nanofiber membrane could adsorb 5.68 mmol/g boron at most and 0.82 mmol/g boron from a 5 mg/L solution in 15 minutes, with 93.9% regeneration efficiency after 10 cycles [32].

Furthermore, polymers containing amino groups are especially well-known among the cationic polymers [33]. One of the examples is N,N-dimethylamino propylacrylamide (DMAPAAQ) which is a popular quaternary ammonium salt-type cationic monomer. In this case, the positively charged quaternary amino group of DMAPAAQ plays a role in maintaining the charge balance by attracting the counter anion Cl⁻ [34]. Hence, the exchange between Cl⁻ ions with them allows the absorbance of the important anions or the removal of harmful anions.

N-[3-(dimethylamino)propyl] acrylamide (DMAPAA) is another example of a cationic polymer. According to reaction (2), protonation occurs when the amino group swells in the water by a proton derived from the dissociation of water molecules [35].



In the case of dissociation of water molecules, the Donnan effect also occurs where hydroxyl ion is also formed and is retained in the hydrogel by ionic.

This study proposed copolymerization of the DMAPAA and DMAPAAQ, a novel easy boron removal approach employing cationic hydrogel with tertiary amino group. It is believed that boron may be recovered from the solution by swapping Cl⁻ in the DMAPAAQ and interacting with the amino group in the DMAPAA, which improves the gel's

adsorption sites and hence increases the hydrogel's boron ion adsorption abilities. Considering that the form of boron is pH-dependent, this is one of the most essential parameters in viewing adsorption performance. Therefore, the effect of variations in adsorbent dosage and pH on boron adsorption was investigated using the DMAPAA-co-DMAPAAQ hydrogel to determine the adsorption efficiency.

2. Materials and Method

2.1. Materials

The materials employed in this research include N-[3-(dimethyl amino)propyl]acrylamide (DMAPAA) and N,N-dimethylamino propylacrylamide monomer (DMAPAAQ) that were kindly supplied by KJ Chemicals (Japan). Prior employed for removing polymerization inhibitors, this material was distilled. Furthermore, the other materials, which are N,N'-methylenebis(acrylamide) (MBAA) and Ammonium peroxodisulfate (APS) were purchased from Sigma-Aldrich. Meanwhile, N, N,N',N',-tetramethylethylenediamine (TEMED) was purchased from Nacalai Tesque (Kyoto, Japan). These materials were all reagent grades and employed in the research as received.

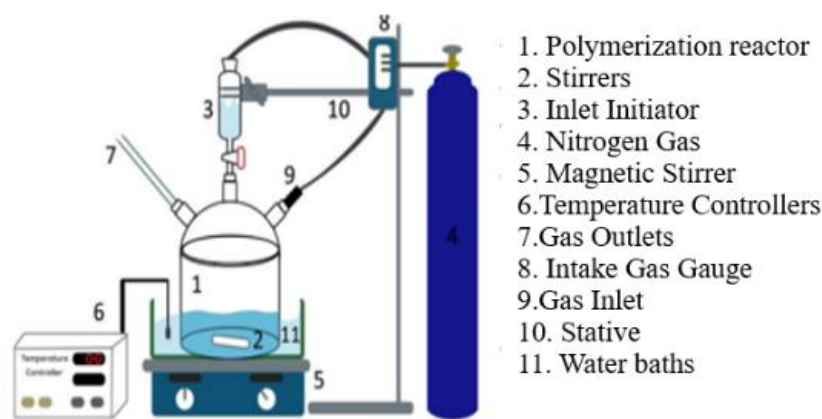


Figure 1. Series of Reactors for Polymerization

2.2. Method

2.2.1 Synthesis of Hydrogel

The main monomers employed in hydrogel synthesis are DMAPAA and DMAPAAQ. As a cross-linker, MBAA is used further. The accelerators and initiators used are TEMED and APS. To obtain a 100 mL solution, the monomers (DMAPAA and DMAPAAQ), MBAA, and TEMED are dissolved in deionized water. Then, make 20 mL of APS solution. The gel formed in these two solutions using nitrogen for 40 minutes. Next, the monomer, cross-linker, and accelerator are mixed, and the initiator solution is added and homogenized. Polymerization was carried out in a water bath at 25°C. The length of the hydrogel is reduced to match the diameter. Methanol was used to wash this hydrogel through a Soxhlet extraction procedure for 24 hours to remove the contaminants. After that, the hydrogel was dried at room temperature for 24 hours and dried using an oven at 50°C for one day.

2.2.2 Batch-mode Sorption Study

To obtain the optimum adsorption removal at different reaction times and temperatures, 40 mL of solution was mixed with 2 g/L of the hydrogel. Adsorption was carried out while continuously shaking. The levels of boron were further assessed both before and after the adsorption. Three separate measurements were used to compute the mean values for all the data collected, and part of the data also included the standard deviations. An equation was used to get the equilibrium adsorption capacity, Q (mg boron/g resin):

$$Q_e = \frac{v \times \left(\frac{C_0}{C}\right)}{M} \quad (3)$$

Information:

Co = concentration of boron in solution before adsorption (mg/L)

C = concentration of boron in solution after adsorption (mg/L)

V = volume of solution (L)

M = weight of adsorbent (g)

Equilibrium adsorption rates were measured at various pH values to determine the effect of solution pH on boron uptake. In each run, 0.08 g resin was added to 40 mL of a solution containing 70 mg/L of boron before the pH was adjusted, and boron concentrations were assessed 24 hours later at 25°C following continuous shaking.

For the adsorption isotherm investigation, 0.08 g of resin was in contact with 40 mL of boron solution for 24 hours while being continuously shaken at starting concentrations ranging from 20 mg-B/L to 100 mg-B/L. A comparison was then conducted between the experimental results and the Langmuir and Freundlich isotherm models. The following is the Langmuir equations:

$$\frac{C_e}{q_e} = \frac{C_e}{Q_m} + \frac{1}{KLQ_m} \quad (4)$$

Information:

q_e = adsorption quantity

C_e = equilibrium concentration of B at equilibrium

Q_m = maximum amount adsorbed (mg/g)

KL = Langmuir adsorption isotherm constant (L/mg)

The Langmuir separation factor, RL indicating the type of isotherm, is defined as follows:

$$RL = \frac{1}{1+KLC_0} \quad (5)$$

KL is the Langmuir constant, and C_0 is the initial boron concentration (mg/L). The Freundlich equation is illustrated as follows:

$$\text{Log } Q_e = \text{Log } KF + \left(\frac{1}{n}\right) \times \text{log } C_e \quad (6)$$

KF is the adsorption capacity (mg/g) (L/mg) $1/n$, and n is the adsorption intensity. Meanwhile, Q_e is the equilibrium adsorption capacity (mg/g), and C_e is the equilibrium concentration (mg/L).

3. Results and Discussion

3.1. Boron Adsorption

Boron is a type of weak Lewis acid with a pKA value of 9.2 at 25°C, so it does not have a single elemental form. As a result, boron dissolves easily when it meets water because it can accept its electrons [36]. In this study, absorption of boron in the form of boron acid was carried out. DMAPAA-co-DMAPAA-Q, as a cationic polymer, will exchange bonds with boron to bind it by following reaction:



According to Ateia et al., regeneration can be carried out by mixing the adsorbent in a 50 mL NaCl/methanol solution [37].

3.2. Effect of Adsorbent Dosage on % Boron Removal

The effect of adsorbent dosage on the percentage of boron removal is visualized in (Figure 2). Adsorption was carried out at pH 2-12, temperature 25°C for 24 h, with an initial boron concentration of 20 ppm, and various dosages of gels. Figure 2 shows that in all dosages, initially, the boron removal remains constant with increasing pH from 2-10 and significantly decreases with further increasing pH from 10-12. Moreover, adsorption carried out using a lower dosage of 2 g /L adsorbent has a higher boron removal rate than 0.5 g/L at the same pH range. Increasing the dosage makes many Cl^- ions or active ions exchange with the boron [38]. In addition, with a higher dosage, the contact area of the adsorbent surface becomes greater. Thus, the adsorbent has more side active to contact with the adsorbate. Because of this contact, ion substitution occurs at low pH with Cl^- . Meanwhile, the adsorbent tends to bind more strongly to Cl^- at neutral pH.

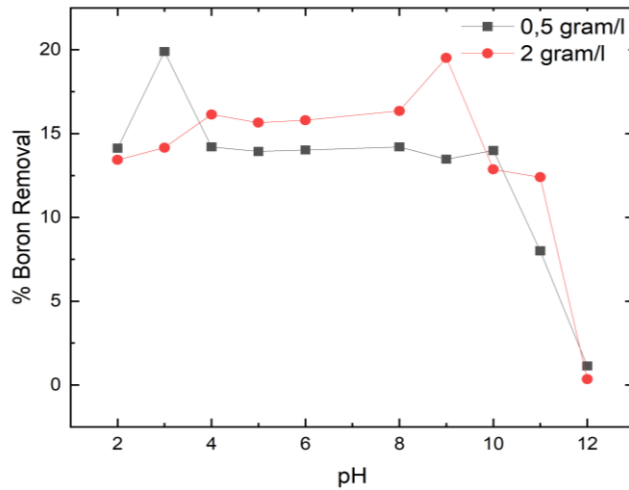


Figure 2. Effect of Dosage Adsorbent on % Boron Removal

3.3. Effect of pH on % Boron Removal

The effect of pH on the removal of boron with an initial concentration of 70 ppm is shown in (Figure 3). Boron adsorption that was carried out at pH 2-12 shows that the maximum % removal occurs at pH = 11. The increase in pH compared to removal shows an increasing trend. At a lower pH the boron removal shows an insignificant increase in boron removal, however at a higher pH after 8, the removal increases significantly. This result shows an opposite trend in comparison with an initial concentration of 20 ppm as shown previously in (Figure 2). This phenomenon also indicates that pH solution at a higher concentration can affect the number of active ions in the adsorbent.

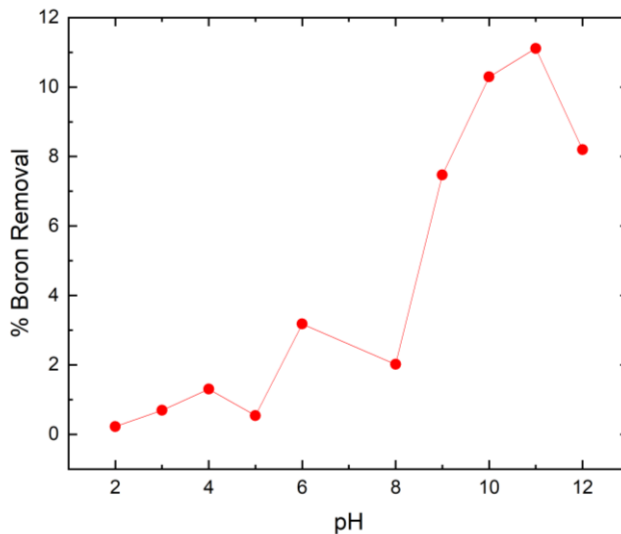


Figure 3. Effect of pH on % Removal of Boron

Song's research shows that as the pH increases, the percent removal increases and begins to reach stability at pH=3. The ineffectivity of adsorbent absorption at low pH can be caused by disruption of the selectivity of the adsorbent due to the presence of Cl ions [39]. When the hydrogen ion increases, the interaction between isopropyl and amino groups in the adsorbent increases, thus indicating the dissociation of boric acid [40] and boric acid dominates at low pH. In contrary, the increase in the percentage of boron removal at high pH is due to the dissociation of boron (borate ions $B(OH)_4^-$) dominate at alkali conditions or $pH > 9$, thus facilitating the process of binding boron ions with the quaternary amino group of DMAPAAQ is positively charged.

3.4. Effect of Difference in Initial Adsorbate Concentration on % Boron Removal

The effect of the initial concentration of the adsorbate on the percent removal of boron is shown in (Figure 4). The adsorption was carried out at initial concentrations of the adsorbate were 20 ppm and 70 ppm, at a pH range of 2-12, and the adsorbent of 2 g/L. Based on the figure below, for an initial concentration of 20 ppm, the maximum percent removal was obtained at pH 9 with a removal value close to 20%, and the minimum removal was obtained at pH 12. Moreover, for an initial concentration of 70 ppm, the maximum percent removal was obtained at pH 11 with a removal value close to 10%, and the minimum removal was obtained at lower pH from 2-5.

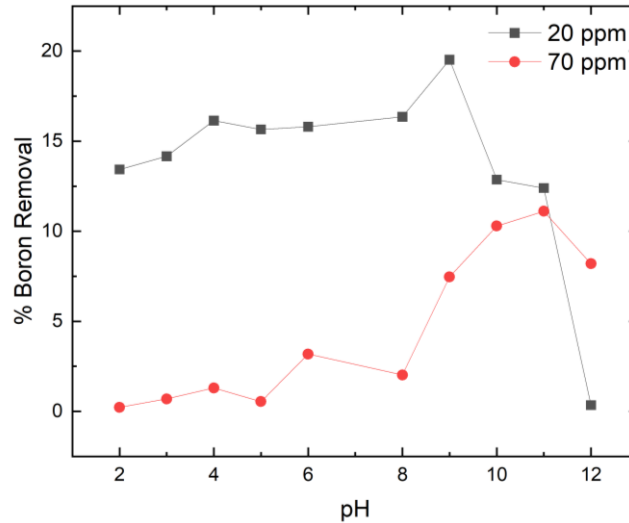


Figure 4. Effect of Adsorbent Initial Concentration of 20 ppm and 70 ppm on % Boron Removal

The effect of initial concentration of the adsorbate on the percent boron removal shown in (Figure 4) indicates an increase and decrease in the % removal value at each pH. However, the adsorption capacity has increased due to the surface's saturation and the adsorbent's active site [41]. Then, the increase and decrease in the percent removal value are also caused by the initial concentration of the adsorbate, which increases after that; the adsorption efficiency decreases because the capacity of the adsorbent is maximum to absorb ions. Moreover, the higher boron removal [42].

3.5. Comparison of DMAPAA-co-DMAPAAQ Hydrogel Adsorbents with Commercial Resins

The comparison of boron removal between DMAPAA-co-DMAPAAQ adsorbents and commercial resin such as DIAION PA312, DIAION WA21J, and DIAION CR11 is shown in (Figure 5). Adsorption was carried out at a dosage of 2 g/L adsorbent with an initial concentration of 70 ppm and at different pH values from 2-12. Based on the graph below, the maximum percent removal is on WA21J resin, reaching almost 27%, and the minimum boron removal is on DMAPAA-co-DMAPAAQ adsorbent.

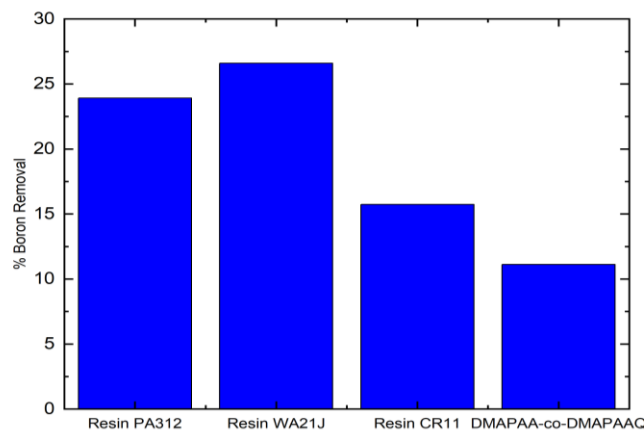


Figure 5. Comparison of DMAPAA-co-DMAPAAQ Adsorbents with Commercial Resins

Al Kakoun's research reported that commercial resins are not biodegradable, making them difficult to recycle, causing more environmental damage [43]. Although the percent removal of commercial resin is almost twice when compared to the synthetic DMAPAA-co-DMAPAAQ adsorbent, it is not environmentally friendly compared to this adsorbent. Meanwhile, the condition of DMAPAA-co-DMAPAAQ can be adjusted easily depending on environmental conditions during adsorption in real applications both at low and high pH. Therefore, this gel is concluded to be ecologically.

4. Conclusions

Based on research on the effect of variations in adsorbent dosages and pH on boron adsorption using DMAPAA hydrogel, several conclusions can be drawn: the higher the adsorbent dosage, the more boron is adsorbed. At an adsorbent dosage of 0.5 g/L, the highest removal percentage was obtained is 19.89% at pH 3, and for an adsorbent dosage of 2 g/L, the highest removal percentage was obtained is 19.52% at pH 9. Experiments varying the pH of the adsorbent on the percent removal showed various results and obtained the highest percent removal at pH 11. The DMAPAA-co-DMAPAAQ hydrogel adsorbent is shown environmentally friendly compared to commercial resins.

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