

## Pineapple Leaves (*Ananas Comosus*) Ca-Alginate Immobilized as Adsorbent for Removal of Rhodamine B Dye

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### ABSTRACT

*Rhodamine B dye is one of the dye wastes from the textile dyeing industry. One way to deal with Rhodamine B in aquatic environments is by adsorption. The contribution of this study is to provide methods to remove Rhodamine B dye using Ca-alginate immobilized pineapple leaves. Ca-alginate immobilization of pineapple leaves was to improve adsorbent performance, mechanical strength, and adsorption capacity. Pineapple leave Ca-alginate immobilized adsorbents were characterized using Fourier Transform Infrared (FTIR), Scanning Electron Microscopy (SEM), and X-Ray Fluorescence (XRF) instruments. Some adsorption parameters, such as the effect of pH, contact time, mass of adsorbent, stirring speed, and concentration of Rhodamine B dye, were evaluated. The optimum adsorption conditions parameters were obtained at pH 3, contact time during 90 minutes, agitation speed of 150 rpm, the adsorbent mass of 0.1 gram, and Rhodamine B concentration of 80 mg/L with a maximum adsorption capacity of 8.964 mg/g.*

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## 1. Introduction

Synthetic dyes are preferred over natural dyes because synthetic dyes are cheaper, easy to use, bright in color, stable, and resistant to the environment. Synthetic dye is difficult to degrade due to dye complex synthetic compounds resistant to light, reaction chemistry, and biology. It makes dye waste challenging to remove from the body water. One of the dyes, Rhodamine, dyes are often used B. Synthetic dyes other than the textile industry can also come from the cosmetic, pharmaceutical, leather, photographic, and agricultural industries [1]. The use of synthetic dyes can have a pollution impact on the environment in the form of dye waste. Dye waste is one of the hazardous substances, the discharge of dye-containing wastewater into natural streams and rivers has a severe environmental problem. The colored waste is not only aesthetically pleasing but also blocks light from entering the waters, as a result, it can disrupt biological processes in water bodies [2]. This requires serious attention in reducing dye waste because its presence can have several negative impacts, including reducing light penetration in waters, blocking the photosynthesis of aquatic plants, and reducing water clarity for drinking and other purposes [3]. Another consequence is that it can cause allergies, dermatitis, skin irritation, cancer, and human mutations. It is, therefore, necessary to treat wastewater before it is discharged into a body of water. However, wastewater containing synthetic dyes is challenging to treat because the dyes have organic matter containing aromatic rings, are non-biodegradable, and stable in light, heat, and oxidizing agents because of their molecular structure and size [4], thus making dyes mutagenic and carcinogenic.

Rhodamine B (Rho B) dye is a water-soluble, non-biodegradable basic organic dye and chemically stable compound [5], [6]. Rho B compound contains an alkaline amino group with a benzene core,

and it is non-degraded naturally by microorganisms. The presence of Rho B dyes in water bodies caused severe environmental problems [7], [8], [9].

Several dye waste treatment methods include flocculation, coagulation, precipitation, biological treatment, catalytic oxidation, filtration, oxidation-reduction, electrolysis, membrane separation technology, adsorption, and many other ways [10], [11], [12]. Adsorption is one method that is widely used to remove contaminants [13], [14], [15]. Adsorption absorbs a substance (ion or molecule) on an adsorbent surface. This method can be utilizing of biomaterial waste as an adsorbent.

The pineapple leaves waste has yet to be optimally utilized. It contains chemical compounds such as cellulose, hemicellulose, lignin, pectin, and holocellulose. The functional group contains hydroxyl (-OH), which can be transferred to be adsorbent. The pineapple leaf contains about 70 - 80% cellulose, has tensile strength values are higher on pineapple leaf fiber cloth that is to the direction of feed [16], and it's can be used as an adsorbent for dye waste [13], [14], [17].

The pineapple leaves adsorbent has the advantages of an easy preparation process, relatively low cost, and abundant natural availability. The adsorption process using an adsorbent in powder form has a weakness in separating the filtrate and adsorbent. Therefore it is necessary to improve the surface properties and performance of the adsorbent. Thus, to enhance the appearance of adsorbent pineapple leaves can be modified with alginate [18].

Alginate is one of the most commonly used biopolymers for immobilizing microorganisms, biomaterials, bacteria, or biomass. Alginate is an ammonium or alkali salt chain residues of  $\beta$ -D-manuronic acid and  $\alpha$ -L-guluronic acid linked to 1,4. Alginate becomes a negatively charged anionic polymer on acidification with the formation of carboxylate (R-COOH) groups. Alginate is an anionic co-polysaccharide consisting of  $\beta$ -1-4 D-mannuronate (M) and  $\alpha$ -L-guluronate (G) residues. Alginate is a natural polysaccharide that is commonly found in the cell walls of all species of brown algae (*Phaeophyceae*) and is non-toxic, non-biodegradable, and biocompatible [19], [20], [21]. Previous research has modified alginate as an adsorbent to remove dye waste such as Methylene blue, Rho B, and Methyl violet [22], [23]. The contribution of this study is to propose a modification of pineapple leaves by immobilized pineapple leaves (PL) in Ca - alginate polymer bead for adsorption of a textile waste dye such as Rho B. PL immobilized can improve adsorbent performance, increasing mechanical properties and stability of the adsorbent in aqueous media.

## 2. Research Methodology

### 2.1. Materials

The materials are Pineapple leaves, distilled water, HNO<sub>3</sub> p.a (Merck, Germany), HCl (Merck), NaOH (Merck), Rho B, NH<sub>4</sub>OH (Merck), NH<sub>4</sub>Cl (Merck), glacial CH<sub>3</sub>COOH (Merck), buffer solution pH: 2, 3, 4, 5, 6, Na-alginate, and CaCl<sub>2</sub>.

### 2.2. Procedures

#### 2.2.1. Preparation of Pineapple Leaves (PL)

Pineapple leaves were taken from Tangkit Village, Jambi, Indonesia. The leaves were washed with water until clean, cut into 1-2 cm sizes, and dried at room temperature for seven days. The leave was ground using a grinding mill and sieved to a particle size of 110  $\mu$ m. The powder was soaked in 0.1 M NaOH solution (1:10) for 2 hours, filtered, and repeatedly washed with distilled water until the pH was neutral. It was dried at 100°C for 4 hours, cooled and crushed with a mortar for homogenized particle size with uniform, and stored in a dark bottle.

#### 2.2.2. Preparation of Pineapple Leaf (PL) Immobilized Ca-alginate Bead

The procedure as in [24]: a 2% of alginate polymer solution is prepared by dissolving 2 gr of Na-alginate in 100 mL of deionized water, agitated with a magnetic stirrer at 100 rpm, at 50-60°C for 30 minutes, then added powder pineapple leaf as much as 2 g (1:1). The pineapple leaf-alginate mixture was prepared in the form of beads by dropped combination into a 0.2 M CaCl<sub>2</sub> solution by gravity using a syringe. The beads formed were hardened in a 2% CaCl<sub>2</sub> solution for 24 hours. The beads were washed with distilled water while stirring at 100 rpm for 30 minutes and repeated 3-5 times until the pH of the solution was neutral. The beads are dried at room temperature and stored in a desiccator.

### 2.2.3. The Characterization of Adsorbent

FTIR, SEM, and XRF characterized the PLA immobilized bead adsorbent. Fourier Transform Infrared Spectroscopy (FTIR) Type Unicam Mattson Mod 7000 FTIR was used to analyze the functional, X-ray fluorescence spectroscopy (XRF, PAN analytical Epsilon 3) was used to study of composition adsorbent and Scanning Electron Microscopy (SEM, Inpect F50) was used to analysis of the surface morphology of adsorbent.

### 2.2.4. Adsorption of Rho B

The adsorption experiment was carried out in a batch process: 20 mL of 10 mg/L Rho B solution was put in Erlenmeyer glass each, and 0.1 g of adsorbent was added, the shaker solution at 100 rpm for 15 minutes. Filtrate was filtered and analyzed of Rho B amount with a UV-Vis spectrophotometer at wavelength maximum. The studied adsorption parameters are the effect of pH (2-7), the mass adsorbent (0.1 - 0.5 g), contact time (15 - 150 minutes), and concentration of Rho B solution (10 -100 ppm).

The synthetic dye used in this research is Rho B which has a chemical structure of  $C_{28}H_{31}C_1N_2O_3$ . Rho B's molecular weight is 479.02 g/mol. The maximum wavelength ( $\lambda$  max) was obtained at 543 nm. A standard solution (1000 mg/L) was prepared by dissolving 1 gr of Rho B powder into 1 L of demineralized water in a volumetric flask. The batch adsorption study was performed for the removal of Rho B.

Adsorption capacity ( $Q_e$ ) was calculated according to equation 1.

$$Q_e (mg/g) = \frac{C_o - C_e}{m} \times V \quad (1)$$

Where  $C_o$  represents the initial concentration (mg/L),  $C_e$  is the equilibrium concentration (mg/L),  $V$  is solution volume (L), and  $m$  is the mass of adsorbent (g).

## 3. Results and Discussion

### 3.1. Characterization Pineapple Leaf (PL) Alginate Immobilized

Identification of PL and PL alginate immobilized functional group using FTIR is shown in Fig. 1.A and 1.B, respectively. In the PL FTIR spectrum, OH and NH showed at  $3268.56 \text{ cm}^{-1}$ , the O-H/N-H group. In PLA, the absorption peak occurs in the  $3700 - 3100 \text{ cm}^{-1}$  region due to stretching vibrations of O-H/N-H. Wave number  $2234.63 \text{ cm}^{-1}$  showed  $C \equiv C$  stretching vibration at  $2200-2260 \text{ cm}^{-1}$ . At peak  $1568 \text{ cm}^{-1}$  is carbonyl group  $C=O$ , peak  $1414.57 \text{ cm}^{-1}$  there is a deformed C-OH group from a carboxylic acid. At wave number  $1025.19 \text{ cm}^{-1}$ , there is a C-O carboxyl group stretching vibration. In the immobilized adsorbent, the C-OH group appears at wave number  $1414.57 \text{ cm}^{-1}$ , this is due to the deformation of the carboxylic acid.

**Table 1.** The Functional Groups in Adsorbent PL Immobilized Alginate

Functional group	Adsorbent spectra	
	PL ( $\text{cm}^{-1}$ )	PL alginate ( $\text{cm}^{-1}$ )
OH/NH	3326.26	3268.56
-CH	2908.08	2907.05
C=O	1637.55	1698.41
CH <sub>2</sub>	2088.86	2084.63
C-OH	1324.77	1414.57
C-O	1024.37	1025.19

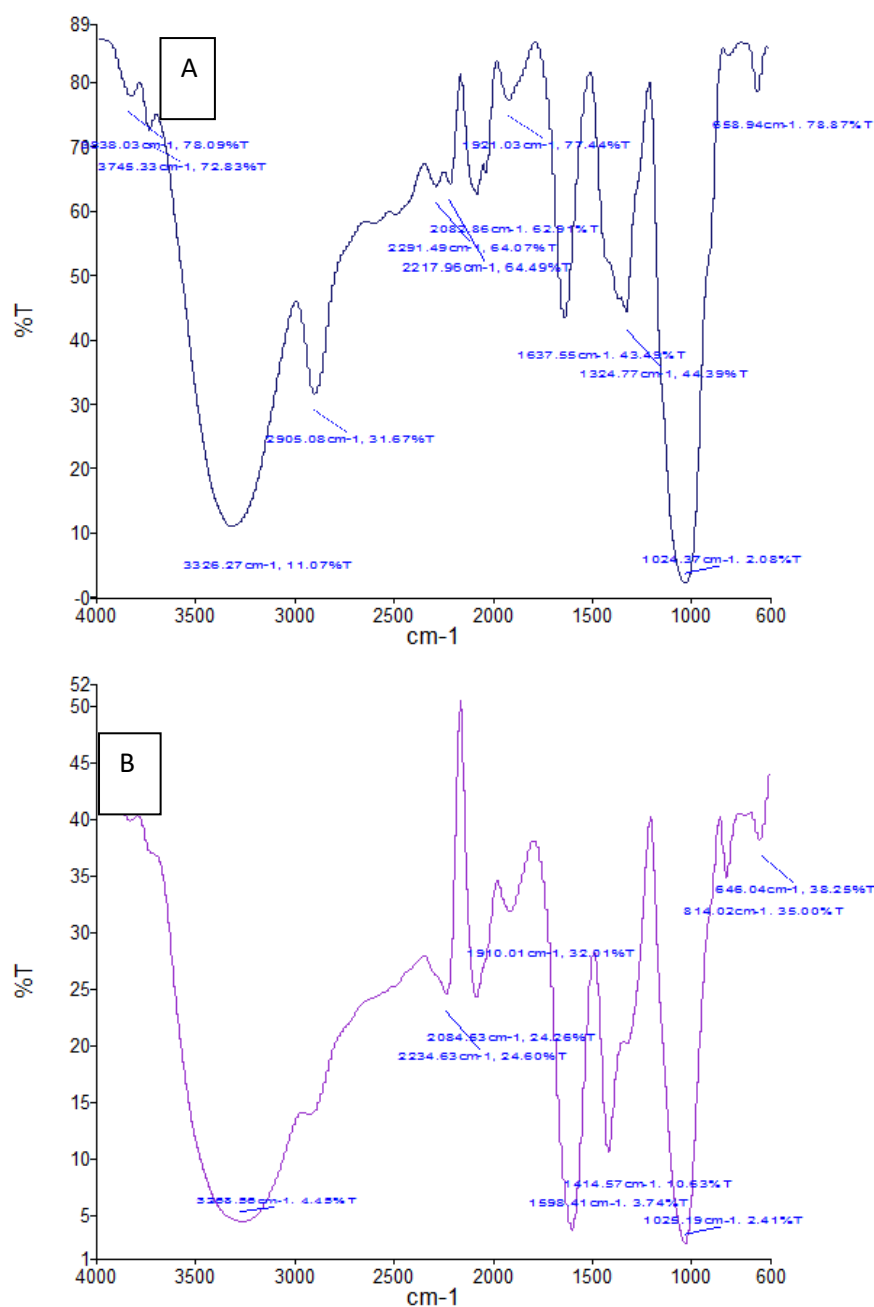
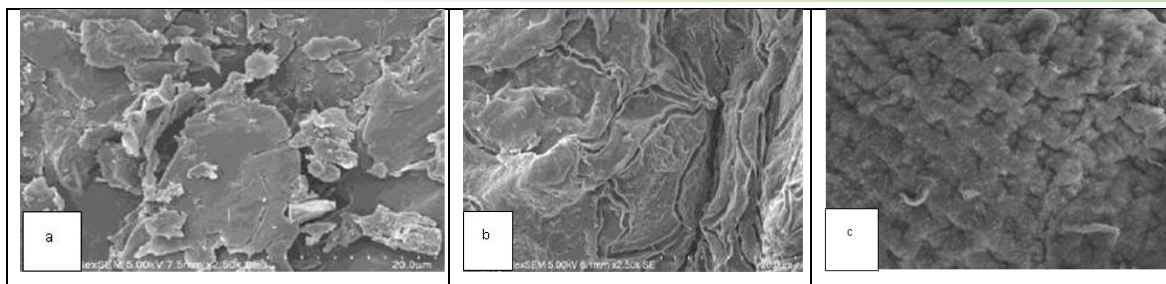


Fig. 1. FTIR spectra of Pineapple leaf (A) and Pineapple leaf alginate immobilized bead (B).

### 3.2. Scanning Electron Microscopy Analysis

Fig. 2.a shows that the morphology of PL is flat, the distance between particles is very tight and has a small grain size, surface of morphology like as smooth due to the organic compounds present in PL. Fig. 2.b shows the formation of networks on PL, the surface morphology shape is rough and irregular due to the bond between cellulose and Ca-alginate. Fig. 2.c look like the surface morphology PL immobilized are uniform and flat, it was adsorbent surface has been filled by Rho B dye. This is indicated that Rho B dye has bounded with functional groups in PL. The morphology of PL alginate immobilized showed in Fig. 2.b. The SEM photograph with 1000x magnification, Fig. 2.a, offers an irregular adsorbent surface, this is the appropriate result of *Arenga pinnata* fruit peel alginate immobilized [25] the surface of the adsorbent is a distinctive structure and forms lumps with 1000x magnification.



**Fig. 2.** The SEM morphology of PL (a), PL before adsorption (b), and after adsorption Rho B (c).

### 3.3. Analysis of XRF (X-ray Fluorescence Spectrometry)

The XRF analysis showed in Table 2. The existing elements in Na-alginate are S, Cl, Ca, and Mg, while in PL, alginate immobilized elements are Al, Si, P, and Ca.

**Table 2.** Element of PL, PL Alginate Immobilized, and PL Immobilized-Rho-B.

Element	Concentration (%)			
	NA-Alginate	PL	PL Alginate	PL Alginate Rho-B
Mg	1.028	2.923	1.841	-
S	87.567	3.034	-	-
Cl	6.701	-	-	-
Na	0.449	-	-	-
Al	0.921	6.236	2.363	3.698
Ca	1.478	35.667	79.236	17.642
Si	-	24.58	5.743	4.77
P	-	8.037	2.664	11.667
K	-	3.579	0.178	-
O	-	-	-	25.5

All the tested samples had weak antibacterial activity (inhibition zone  $\leq 12$  mm) [29]. This result is in line with the research in [30], where the *Azadirachta indica* extract did not show an inhibition zone on the gram-negative bacteria *Escherichia coli* but had an impact on the gram-positive bacteria *Staphylococcus aureus*. Gram-negative bacteria can change cell walls, so antibiotic compounds cannot attach or produce enzymes to deactivate antibiotics. Although bacteria have no brain, they can process information. Just as bacteria search for food sources, they can move away from antibacterial compounds as a way to survive.

### 3.4. The Effect of pH

pH is one such parameter that affects the adsorption process. Determining optimum pH for adsorption capacity aims to determine Rho B's optimum pH color solution during adsorption. The pH result can change the solution's concentration of  $H^+$  and  $OH^-$  ions. On pH acidic ( $pH < 7$ ), the concentration of  $H^+$  ions is more significant than  $OH^-$  ions, and at alkaline pH ( $pH > 7$ ), the concentration of  $OH^-$  ions will be greater than  $H^+$ . Increasing the pH solution will increase the adsorption capacity of Rho B on the PL alginate immobilized bead and reach equilibrium at pH 3-4. At above pH four, decreased occur of adsorption capacity. This is because at a highly acidic pH of 2-3, there is competition between  $H^+$  ions and Rho B, which are cationic molecules that bond with PLA immobilized bead, which is electronegativity charged, and at pH above 4, there is a decreased in adsorption capacity, this is caused by increasing pH, the atmosphere of solution will be alkaline ( $OH^-$ ), and there is a repulsive force with the  $OH^-$  and  $COOH^-$  functional groups which it's electronegative on alginate bead. The effect of pH on the adsorption capacity of Rho B can be seen in Fig. 3.

### 3.5. The Effect of Contact Time

The determination of equilibrium time was found to be the minimum time required by a certain amount of Rho B dye adsorbed on the surface of the PL alginate immobilized adsorbent. The adsorbent is no longer able to absorb adsorbate. Therefore, to investigate the effect of contact time, the experiment was conducted at 15-100 minutes. The Rho B adsorption on PL alginate immobilized is shown in Fig. 4. Adsorption occurs rapidly in the first 30 minutes. Adsorption capacity increased with

increasing contact time and was optimal at 90 minutes with an adsorption capacity of 2.613 mg/g. The adsorption of dyes by PL alginate increases with increasing interaction time and will reach equilibrium at the optimum time. After passing through equilibrium, there was a decrease in adsorption capacity [20].

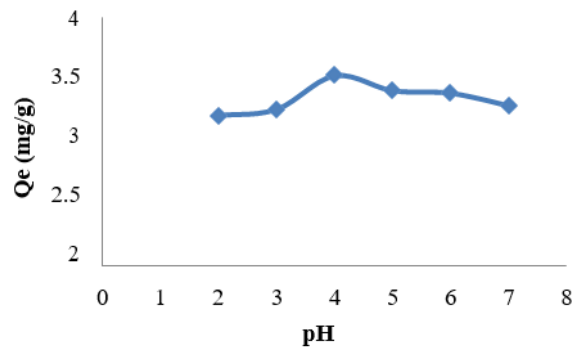


Fig. 3. The effect of pH on adsorption capacity Rho B.

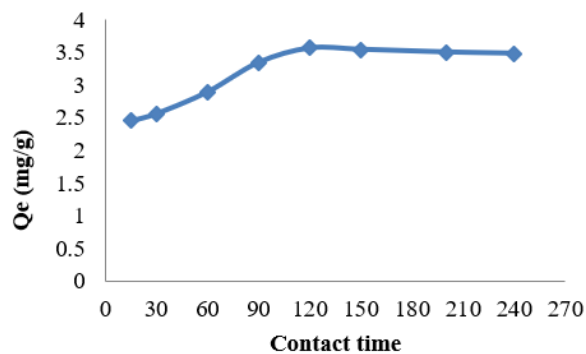


Fig. 4. The effect of contact time on adsorption capacity Rho B

### 3.6. The Effect of Stirring Speed

The effects of stirring speed on the adsorption of Rho B dye by PL can be seen in Fig. 5. Adsorption of Rho B increased with increasing stirring speed but decreased after passing the optimum condition. The optimum conditions were obtained at a rate of 150 rpm with an adsorption capacity is 2.379 mg/g. This is related to increasing the rate of stirring, which can increase the diffusion of dye to the surface of the adsorbent, it can grow to driving force of adsorbate to the surface of the adsorbent [3].

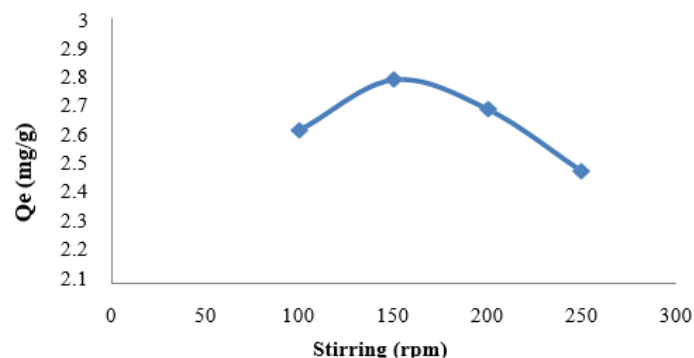


Fig. 5. The Effect of stirring speed on adsorption capacity Rho B.

### 3.7. The Effect of Mass of Adsorbent

The adsorbent mass plays a vital role in the adsorption process. The mass of adsorbent is related to the availability of active sites on the surface adsorbent to adsorption Rho B dye molecules through pores or site functional groups. The effect of adsorbent mass on adsorption capacity states that the

adsorption of dye per unit mass adsorbent will decrease with increasing adsorbent mass [22]. The increase in the amount of adsorbed dye with increasing doses can be associated with an increase in the number of more adsorbent surface active sites. However, if the adsorption capacity is expressed in mg adsorbed per gram of material, the capacity decreases with increasing doses of PL. This may be caused by over-lapping or aggregation of adsorption sites resulting in subsidence of the total surface area of the adsorbent available for dye and enhancement diffusion path length. The effect of doses on adsorption capacity can be seen in Fig. 6. Adsorption capacity decreases with increasing mass because, according to equation 1, if the adsorption capacity is expressed in mg adsorbed per gram of material, adsorption capacity decreases with increasing doses of PL.

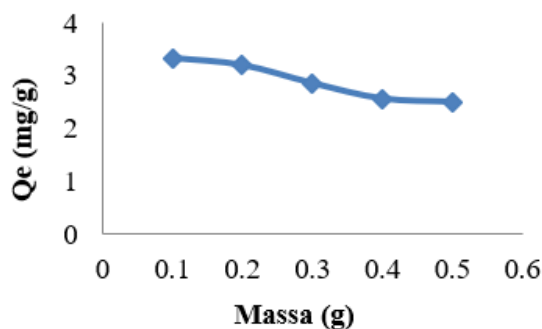


Fig. 6. Effect of mass on adsorption capacity

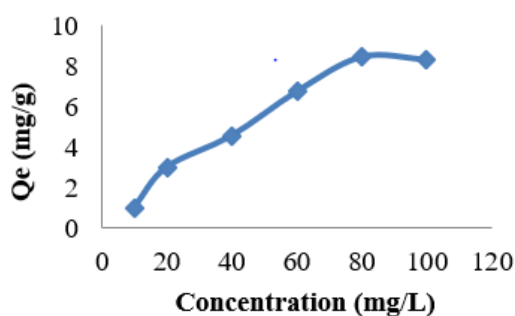


Fig. 7. Effect of concentration on adsorption capacity Rho B

### 3.8. The effect of concentration on adsorption capacity Rho B

The dye concentration is one of the adsorption parameters to study adsorption equilibrium. The amount of dye adsorbed is highly dependent on the initial dye concentration. From Fig. 7., an increase in the amount of Rho B molecule adsorbed on PL can be seen. The amount of dye adsorbed increases with increasing concentration of the solution. The adsorption capacity increased at a concentration of Rho B solution of 10-80 mg/L. The adsorption capacity increased because of many interactions between active sites in the PL alginate immobilized adsorbent and Rho B. In PLA, carboxyl and hydroxyl are active sites, which are electronegative and interact with Rho B is cationic, causing an increase in adsorption capacity. Fig. 7. shows that the maximum adsorption of dyes Rho B at a concentration of 80 mg/L with an adsorption capacity is 8.93 mg/g. the adsorbent surface has been filled by Rho B and reaches an equilibrium state. The adsorption of Rho B on activated carbon Gunitir plant bark occurred at a concentration of 120 mg/L with adsorption capacity is 2.3081 [17].

## 4. Conclusion

PL alginate immobilized bead adsorbent has been used for the adsorption of Rho B. The adsorption is influenced by several parameters such as pH, contact time, stirring speed, adsorbent mass, and concentration of Rho B solution. Adsorption of Rho B occurs at the following conditions: pH 4, contact time 120 minutes, mass adsorbent was 0.1 g, stirring speed was 150 minutes, and concentration of Rho B solution was 25 mg/L with a maximum adsorption capacity is 8.93 mg/g. The functional groups involved in the adsorption process are carbonyl, carboxyl, and hydroxyl, as well as other functional groups found on the surface of PL alginate, immobilized adsorbent.

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