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Research Article

Preparation of Low-cost Adsorbent Based on Mango Leaf (*Mangefira Indica L*) Biomass for Methylene Blue (MB) Adsorption

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Abstract: Preparation of leaf mango (*Mangefira Indica L*)-based adsorbent and its evaluation to reduce methylene blue (MB) concentration have been conducted. Two materials: ads-pristine and ads-CTAB which are referred as native leaf mango adsorbent and CTAB modified leaf mango have been prepared in this study. Cetyltrimethylammonium bromide (CTAB) is cationic surfactant usually used to modify and increase the surface area of the adsorbent for a specific purpose. The materials obtained were characterized by the Fourier Transform Infra-Red (FTIR) and the Scanning Electron Microscope (SEM) with general result, there were detected functional groups indicated the active sites on the adsorbent such as -OH, C-O, C=C. Ads-CTAB was specifically characterized with appearing of C-H aliphatic as the tail of CTAB molecule. SEM images show that both materials have roughness surface and irregular cavities. Additional of CTAB on the surface led the more regularly surface. The optimum pH was in basic region (8), while the optimum adsorbent mass and MB concentration are not clearly found due to the chart still increasing. However, the highest qe was found by 0.01 g of ads-pristine and ads-CTAB with the value 967.25 and 950.75 mg g^{-1} respectively. As the further evaluation, with the 0.5 adsorbent mass it may be applicated on the MB concentration at around 4000 mg L^{-1}. Kinetics model of this adsorption was followed pseudo second order reactions.

Keywords: Adsorbent, Adsorption, Methylene Blue, Mango Leaf Powder, CTAB

Introduction

Water pollution is still a strategic issue to be addressed. Water is the source of human life on this earth. In fact, the availability of clean water which is directly accessible by humans is being limited. Only one percent of the water can be accessed directly by living things, two percent is fresh water from melted glaciers at the north and south poles and the remaining nineties percent is salt water in the ocean [1]. Thus, ensuring the accessible water is completely clean and free from contaminants is very important. Unfortunately, increasing of human and industrial activity have a high potential to make the clean water to be more limited, with their disposal waste and pollution to the environment.

As a phenomenon, the use of dyes as the main material in the coloring process in giant industries such as textiles, paper, printing and tanning has produced waste in large amount [2],[3],[4]. Synthetic dyes are most widely used in large industries because of their wide range of colors, durability and low cost. However, behind these advantages, the use of synthetic dyes becomes a threat if released into the environment as pollution. Some studies report that up to 15% of the use of dyes in coloring process will be waste[5]. Some studies report that the use of dyes in industrial sectors reaches hundreds of thousands of tons per year, meaning that dye waste has the potential to reach tens to hundreds of thousands of tons every year [6]. Various literatures reveal that the wasted dyes can be harmful to human due to their genotoxic and mutagenic [7]. In high concentrations, the dyes can block the penetration of sunlight into aquatic systems, causing biological processes of living things to be disrupted [8]. In addition, the presence

of dyes substances can also increase the number of BOD and COD in the waters. In the environment, large quantities of the dyes are difficult to be naturally degraded unless being assisted by external treatment [9].

Adsorption is one of the leading methods of handling waste. It has been reported to be effective in reducing contaminants such as heavy metals, organic pollutants and dyes. Currently, adsorption technology has developed in the use of organic/biomass waste such as agricultural waste and forest products such as rice husks, wood powder, tree bark and foliage. In adsorption process, biomass or biosorbent interacts with pollutant molecules in certain interactions. The interaction is often known as biosorption. The use of biosorbents of biomass has several advantages in reducing pollutants such as inexpensive raw materials and preparations, simple process, able to be regenerated, selective for some heavy metals and safe for the environments [10].

Mango (*Mangefira Indica L*) trees are seasonal fruit trees that can grow in almost any region. The tree is shady with dense leaves. Its fruit became a commodity of inter-continental trade. In various regions in Indonesia, mango trees are easily found in front of the yard as fruit trees and shady trees. Even in urban areas, mango trees are still often found as fairy shady. Mango trees produce litter dominated by leaves every day. Mango leaf litter has no economic value. The use of mango leaves as biosorbents does not cause conflict for the utilization of others.

Mango leaf is very familiar and abundant materials in the around us. With the simple preparation method, as the adsorbent preparation in general, it will become the low-cost adsorbent. In previous studies, mango leaf powder have been used for adsorption several types of heavy metals such as Pb(II)·Cu(II), Zn(II), Ni(II), Cr(VI), [1],[10],[11],[12],[13] dyes such as Rhodamine B [2],[3], Rose Bengal[4], Victazol Orange 3R[6], Acid yellow[14], Grey BL [15], Methylene Blue [16], Malachite Green [17]. Mango leaves have several functional groups that are mostly negatively charged, namely carbonyl, hydroxyl, alkoxy. The functional group is derived from compounds such as phenols, flavonoids, terpenoids, tannins, xanthones and gallic acid derivatives [18]. However, in previous studies, its not be found a comparison study of the pristine and modified version of mango leaf powder with surfactant in adsorption process, moreover in MB adsorption. To complete information in the adsorption study using mango leaf-based adsorbent, it was interesting to explore that comparison study. Thus, in this study, mango leaf powder (Ads-pristine) will be evaluated in MB adsorption in compared with its modified version with CTAB (Ads-CTAB). MB is a cationic dye with the center of the positive charge was on the sulfur (S) atom. Theoretically, positive charge of MB will interact with the negative charge of the adsorbent surface. Meanwhile, CTAB is a cationic surfactant with a positive charge centered on the Nitrogen (N) atom after the bromide ion leaves the molecular structure. The interesting thing to evaluate in this study is how the effect of CTAB (1%) on MB adsorption. Some of the parameters considered in this study, including pH, adsorbent mass and adsorbent concentration.

Methods

Materials

Mango leaf was collected from the region of Bantul (Yogyakarta, Indonesia), Methylene Blue (MB), *Cetyltrimethylammonium Bromide* (CTAB) was purchased from Sigma Aldrich, hydrochloric acid (HCl) and Sodium Hydroxide (NaOH) are used to set the pH solutions.

Adsorbent Preparation

Mango leaves are dried for a few days. The dried leaf was grinded and sieved as 80 mesh to get a homogeneous size. The powder was then washed with distilled water until the filtrate is colorless. Furthermore, the powder is dried in an oven with a temperature of 55^{o}C for 24 hours and labeled Ads-pristine**.** For further activation and washing process, a certain amount of powder is soaked in a NaOH solution at a ratio of 1:10 for several hours followed by stirring with magnetic stirrer. Mango leaf paste is then washed with distilled water until the filtrate is colorless and reaches a neutral pH and followed by re-drying.

In adsorbent modification with CTAB, dry powder is soaked with 1% CTAB with a ratio of 1:20 for 24 hours. Once separated, the wet powder is washed using distilled water to remove bromide ions on the adsorbent surface. The powder is dried in an oven and labeled Ads-CTAB.

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Evaluation of pH, adsorbent mass and concentration effect

In this work, some parameters such as influence of pH solutions, adsorbent mass and MB concentration were evaluated in batch technique. The influence of pH (8, 9, 10 and 11), adsorbent mass (0.1 – 0.7 gram) and MB concentration (25-200 ppm) were evaluated in 100 ml solution with shaker treatment for 60 minutes. This study was conducted at room temperature, which is at the same time to find out the effectiveness of MB adsorption with ads-pristine and ads-CTAB if done under normal conditions. Final concentrations were monitored using spectrophotometer UV-Vis at 664 nm of wavelength. To determine the percentage of removal and adsorption capacity (qe), we used the following equations:

$$\% \text{ removal} = \frac{(Co-Ce)}{Co} \text{ x } 100\%, \quad (1)$$

and

$$qe = \frac{(Co-Ce)}{m} \text{ x } V \quad (2)$$

where Co (mg L^{-1}) is initial concentration of MB, Ce (mg L^{-1}) is MB concentration at equilibrium condition.

Characterization and Analysis

Ads-pristine and Ads-CTAB materials are characterized by Fourir Transform Infra-Red (FTIR) (Nicolet Avatar 360 IR) at wave number range 500 – 4000 cm^{-1} to find out the functional group and Scanning Electron Microscope (SEM) Phenom Desktop ProXL is used to determine the morphology of each adsorbent. Methylene blue concentrations determined using UV-Vis spectrophotometer.

Result and Discussion

Fourier Transform Infra-Red (FTIR) Characterization

FTIR spectrum of ads-pristine and ads-CTAB are displayed at Figure 1 (A). Some characteristic peaks appear for both materials at around 3300 (O-H stretching for alcohol or carboxyl acid), 2910 cm^{-1} (C-H stretching of sp^3), 2850 cm^{-1} (C-H stretching of sp^3), 1609-1610 cm^{-1} (another peaks of O-H or unsaturation bond of C=C), 1450-1470 cm^{-1} (another peaks of C-H aliphatic compound), 1310-1316 cm^{-1} (C-O of primary alcohol), 1050 cm^{-1} (alkoxy C-O), 775 (C-H stretching of aromatic ring) and 443-455 cm^{-1} were considered as symmetric bending of SO_4 group [17][19]. These functional groups may be active sites in adsorbate binding [20]. Based on spectra in Figure 1 (A), modifications of mango leaf powder (ads-pristine) with CTAB do not produce new peaks. However, almost all peaks in ads-CTAB spectra appear sharper than ads-pristine, especially at around 2910 and 2850 cm^{-1} which are supposed to the fingerprint of the aliphatic C-H, from the tail of CTAB. Fingerprint peaks of aliphatic C-H also appear in the spectra after adsorption, this may indicate that the CTAB tail remains in the material, not leaching.

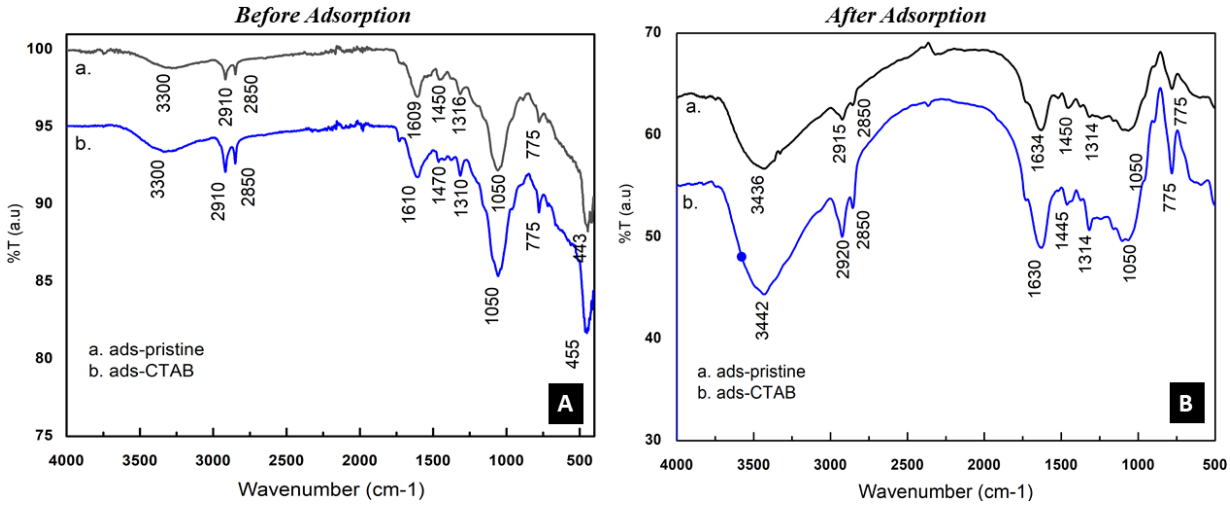

Figure 1. Spectra of adsorbent based-leaf mango powder: A) before MB adsorption, B) after MB adsorption.

Figure 1 also show the distinct pattern of the spectra before and after adsorption process. The spectrum observed after adsorption show broadening bands and intensities with no change positions. Such pattern was also obtained by Ainane et.al (2014), which mentioned the broadening bands and increasing intensities were due to the inclusion of MB on the biosorbent surface[21][22]. The effect of MB's presence on the surface also shifts the wavenumber at some spectral peaks, as also reported by Dhananasekaran et.al (2016)[23].

SEM Characterization

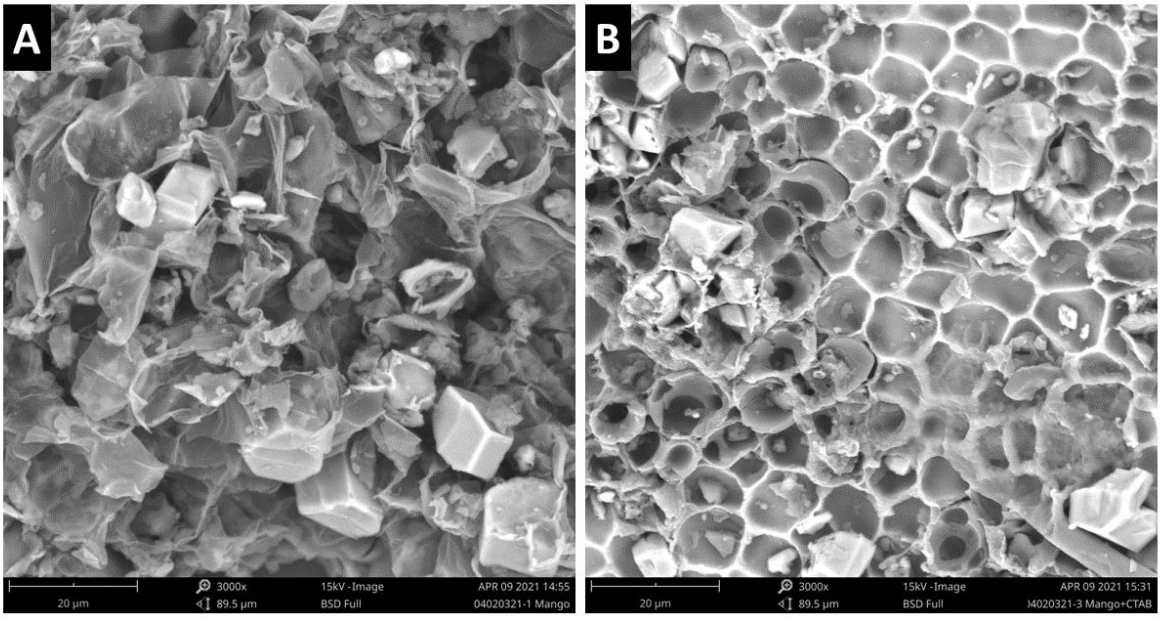

Figure 2. Micrograph of ads-pristine (A) and ads-CTAB

The picture of SEM with 3000x magnification (Figure 2) shows rough and irregular surface of ads-pristine. Some irregular cavities are also visible on the surface. The addition of CTAB to ads-pristine makes the surface more regular. Non-deep cavities are formed through the addition of surfactants. Although the cavity is not too deep, it is possible that the cavities can be occupied by MB.

Effect of pH

The effect of pH was important aspect in the adsorption process, it determines the surface charge of the adsorbents. The binding of adsorbent surface to adsorbate was influenced by the surface charge. In this study, The influence of pH at the range 8, 9, 10 and 11 with 100 mg L^{-1} MB solution was studied. The adsorption process was conducted using adsorbent mass of 0.5 g at room temperature. The use of PH at alkaline scale was referred to [24] [25] which found the highest qe of MB adsorption was exhibited at basic conditions. The presence of OH- ions in the solution can give a positively charged of MB a boost to the adsorbent surface. In the acidic PH that there are many H^+ ions, competition between positive ions will occur to reach the active site space. Based on the rationality, this study used PH on a alkaline scale.

Based on Figure 3, % removal of MB using ads-pristine decreased along increasing pH value: 95.6; 93.6; 92.9 and 91.2 % to 8, 9, 10 and 11 of pH solutions respectively. The value of qe also decrease from 19.12 to 18.24. The optimum PH obtained was at 8 for ads-pristine. While in MB adsorption with ads-CTAB, the value of % removal was obtained in average of 99%. MB was cationic surfactant, since the difference of charge, the positive charge of MB will interact with negative charge on the adsorbent surface, especially on the ads-pristine. The role of surfactant in the adsorption process actually depends on its concentrations. At certain concentration, surfactant will form micelles. Thus, the presence of any surfactant on adsorbent surface may not always give positive impact. This study has used 1% of CTAB (w/w) to modify the adsorbent. The result shows that % removal in all pH condition was in average 99%. The presence of CTAB (1% w/w) may be very dominant in interacting with MB molecule to ignore pH conditions.

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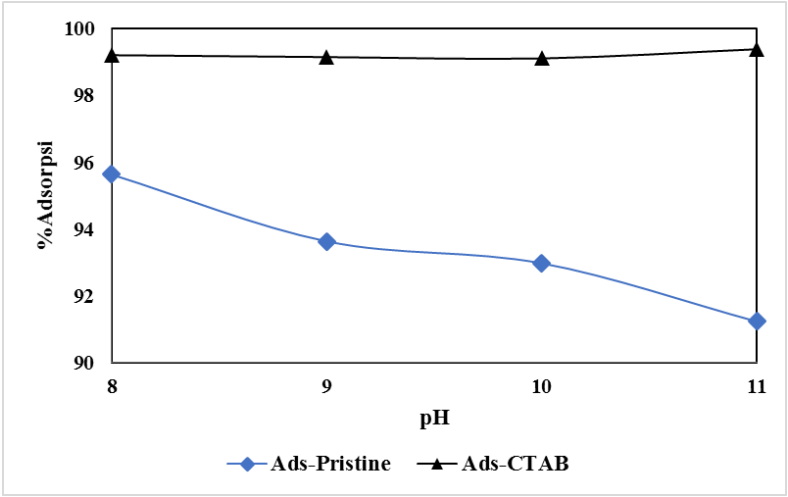

Figure 3. pH effect of MB adsorption on pristine adsorbent and CTAB modified adsorbent

Effect of adsorbent mass

The effect of adsorbent mass (0.01 – 0.7 g) on MB adsorption (100 mg.L^{-1}) was shown at Figure 4. The % removal increased from 96.72 to 98.93 % at ads-pristine use. The similar trend was also obtained in the using of ads-CTAB which show increasing of 95.07 to 99.38 %. The qe value decreased extremely for both materials. The highest qe values for both materials are 967.25 and 950.75 mg g^{-1}, which are obtained by 0.01 g of ads-pristine and ads-CTAB respectively. The lowest results were obtained at the highest adsorbent mass (0.7 g) of 14.13 and 14.19 mg g^{-1} for ads-pristine and ads-CTAB. The role of CTAB which is expected to increase the surface area of adsorbens is proven by the results obtained, namely the average %removal for all adsorbent mass points is higher compared to ads-pristine.

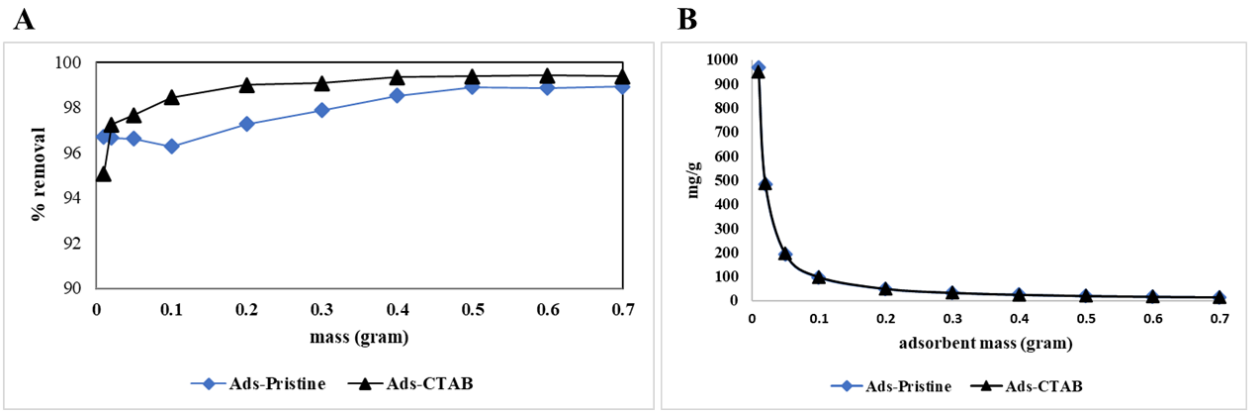

Figure 4. Mass effect of MB adsorption on pristine adsorbent and CTAB modified adsorbent

If the value of % removal and qe are combined, we can conclude that the use of small amount of adsorbent (0.01 g) has been effective in reducing more than 95% of 100 ppm MB for both adsorbent.

Effect of MB Concentration

The effect of initial MB concentration (25 to 200 ppm) was assessed using ads-pristine and ads-CTAB at room temperature. The adsorbent mass used in this assessment was 0.5 g. It was shown at Figure 5 that the increase of initial MB concentration resulted the % removal stay constant at around 95 – 98%. While, the total amount of MB adsorbed was increase with the maximum value of 49.29 and 49.7 mg g^{-1} at 200 ppm for ads-pristine and ads-CTAB respectively.

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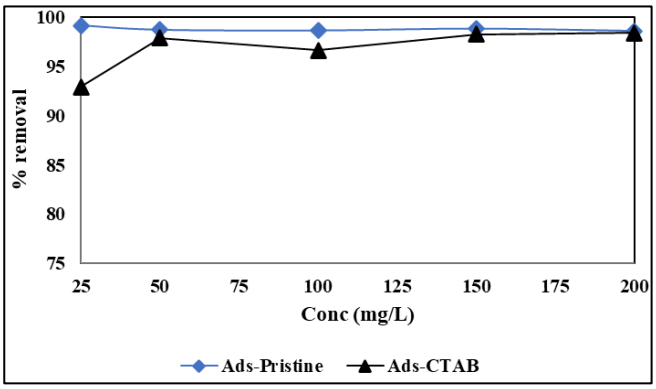

Figure 5. % Removal in MB adsorption using Ads-pristine and Ads-CTAB on various concentrations

Further, ads-CTAB has evaluated at higher concentrations with the result shown at Figure 6. The highest concentration used is 1000 mg L^{-1}. However, the % removal pattern tends to be little constant with average 98% for 25 to 1000 mg L^{-1}. The trend of qe value increased with the highest value is 245.85 for 1000 mg L^{-1}. This means that the active sites provided by ads-CTAB may be overly abundant compared to the number of MB in solution.

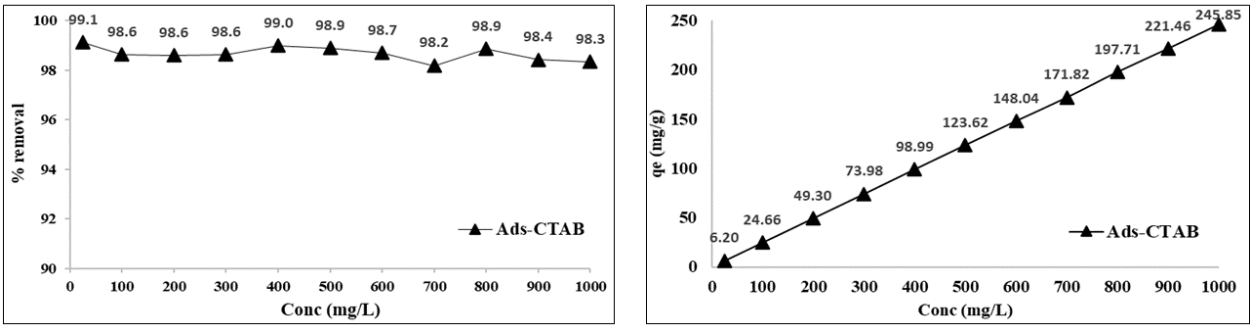

Figure 6. Result of MB adsorption using Ads-CTAB on various concentration: A) % removal, B) adsorption capacity (qe)

In this study, the highest qe value was obtained in the section of effect of adsorbent mass with the amount was 0.01 g which is has qe value of 950.57 mg g^{-1}. The qe value is 4x to be higher than the highest value at Figure 6 (245.85). With that comparison, 0.5 g of adsorbent mass may still have ability to adsorb MB with concentration of around 4000 mg L^{-1}.

Study of Adsorption Kinetic

In this study, the kinetic evaluation was conducted on MB solutions with 100 and 200 mg L^{-1} concentration for respective ads-pristine and ads-CTAB. The rate constant for this adsorption was evaluated with pseudo first order and pseudo second order. The linear equation was followed Lagergren (1898) model as shown at equation 3:

$$\log (qe - qt) = \log (qe) - K_1/2.303\ t \quad (3)$$

The pseudo second order of kinetic was assessed with Ho and Mckay (1998-1999) equation:

$$t/qt = 1/k_2qe^2 + (1/qe)t \quad (4)$$

Where, *qe* and *qt* were amounts of MB adsorbent on adsorbent surface at equilibrium time and at any time, *t* was the observed time (minutes), k_1 (l/min) was the rate constant of pseudo first order. The obtained parameters were shown at Table 1. The value of R^2 (1) in pseudo second orders (Figure 7) means that the adsorption process occurred in this study was follow the kinetic model of pseudo second order for each adsorbent at concentrations of 100 or 200 mg $^{L-1}$.

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Table 1. Kinetic parameters of MB adsorption on ads-pristine and ads-CTAB

Pseudo First orde				Pseudo second orde			
Ads-Pristine (100 ppm)		**Ads-CTAB (100 ppm)**		**Ads-Pristine (100 ppm)**		**Ads-CTAB (100 ppm)**	
R^2	= 0.1533	R^2	= 0,064	R^2	= 1	R^2	= 1
k	= -0.0126	k	= -0.0075	k	= -0.9847	k	= 0.6135
qe	= 0.3036	qe	= 0.2704	qe	= 19.762	qe	= 24.57
Ads-Pristine (200 ppm)		**Ads-CTAB (200 ppm)**		**Ads-Pristine (200 ppm)**		**Ads-CTAB (200 ppm)**	
R^2	= 0.0314	R^2	= 0.4003	R^2	= 1	R^2	= 1
k	= 0.0069	k	= 0.0119	k	= 1.6002	k	= 0.0816
qe	= 0.6911	qe	= 1.5631	qe	= 39.52	qe	= 49.504

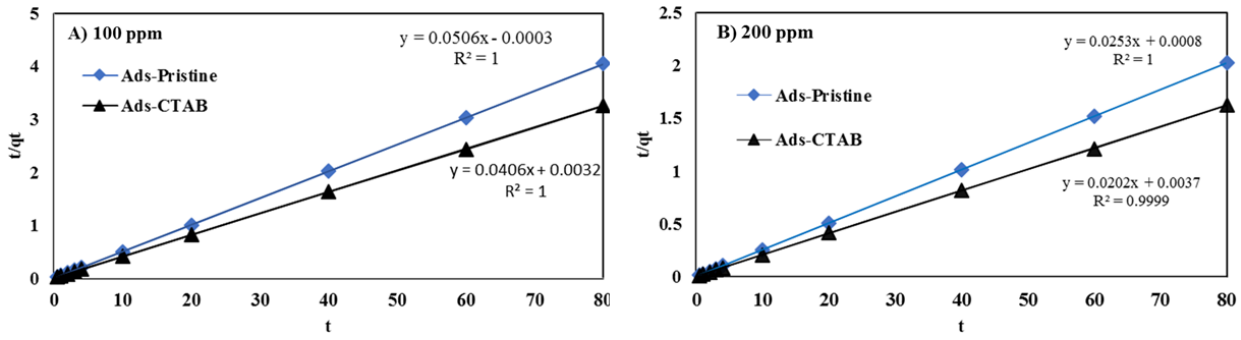

Figure 7. Pseudo second order plot in adsorption 100 ppm and 200 ppm of MB

Even though the determination of free adsorption energy not evaluated in this study, however another studies on MB adsorption with natural adsorbent namely rejected tea [26]and hen feather [27], explained that there is a correlation between adsorption kinetic results with the types of sorption. As a short conclusion, pseudo-order kinetic models can be an indication that adsorption is chemisorption.

Conclusion

Modification of leaf mango-based adsorbent with CTAB surfactant (1%) has successfully conducted. FTIR spectra confirm some fingerprints of ads-pristine and ads-CTAB. SEM images confirm that the existence of CTAB on the adsorbent led the surface to be more regular. Some regular cavities also found after addition of CTAB. The highest qe value of the adsorption process was found at adsorbent mass of 0.01 g with the value of 967.25 and 950.75 $mg.g^{-1}$ respectively for ads-pristine and ads-CTAB. The % removal indicated by ads-pristine and ads-CTAB at 25-200 $mg.L^{-1}$ was near to 100%. On the evaluation at the higher concentrations (till 1000 $mg.L^{-1}$), ads-CTAB still shows excellent performance. Kinetic adsorption model in this study was obeys pseudo second order for ads-pristine and ads-CTAB.

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