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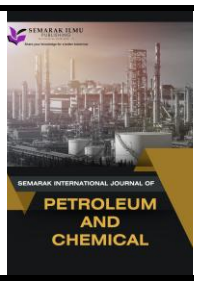

Adsorption of Dyes in Batik Liquid Waste using Strach Modified Fe_2O_3 and its Regeneration

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ABSTRACT

Research has been carried out regarding the use of biopolymers in the form of strach as a modification of Fe_2O_3 which is used to reduce the concentration of dyes in industrial wastewater. Fe_2O_3 (NF) nanoparticles and their modification with Strach (NF-Strach) were synthesized using bottom-up methods and co-precipitation techniques. NF and NF-Strach were successfully used as adsorbents to reduce liquid waste contaminated with dyes. The adsorption test showed that NF-S was able to adsorb better than NF in 10 ppm methylene blue dye, the resulting adsorption efficiency reached 98%. The regeneration test showed that both NF and NF-Strach could be reused even though the adsorption capacity decreased as the number of repeated contacts increased. The adsorption test on liquid waste from the textile industry resulted in a decreased adsorption efficiency when compared with contact with 10 ppm methylene blue.

Keywords:
Fe_2O_3; capping agent; adsorption; regeneration; liquid waste

1. Introduction

Water-soluble aromatic chemical molecules with complex structures are known as dyes [1]. In various industries, including the textile, paper, screen printing and culinary industries, dyes are often used. Wastewater contains between 10 – 15% of these dyes. Waste containing dyes can harm the ecosystem. This colored waste can disrupt the function of aquatic ecosystems by blocking the flow of sunlight and oxygen [2]. Dye waste can cause aesthetic problems, apart from that, dye waste also disrupts the living ecosystem there because it blocks sunlight from penetrating deeper into the water. Anaerobic organisms that produce foul-smelling compounds become more active when the activity of aquatic biota is disturbed, which reduces the oxygen supply in the water and promotes their growth [3]. In addition, non-degradable dye waste can enter the bodies of living creatures and is carcinogenic [4]. Wastewater treatment has been developed using various techniques, including adsorption processes, advanced oxidation, activated sludge systems, electrocoagulation, electrodegradation, adsorption, phytoremediation, artificial wetlands, and photodegradation [5,6].

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The adsorption method produces greater efficiency compared to other methods in reducing dye contamination [7]. Apart from that, the adsorption method has the potential to be further developed because the process is relatively simple and environmentally friendly [8,9]

The adsorption process occurs in two stages - gas-solid or liquid-solid, which is a mass transfer mechanism that occurs on the surface of the adsorbent pores [10]. The most commonly used adsorbent to remove different dyes is commercial activated carbon, but it is too expensive and difficult to regenerate [11]. Therefore, an adsorbent that has high stability is needed so that it can be regenerated and can be used optimally. In this research, an adsorbent in the form of a metal oxide, namely Fe_2O_3 (hematite), was used. Hematite is a form of iron oxide that is stable under environmental conditions with n-type semiconductor properties. Compared with other iron oxides, hematite nanoparticles have a high level of reactivity [12], are low cost and environmentally friendly [14], have good chemical stability [13], and are non-toxic [16], and is resistant to very high temperatures so it has characteristics as a superior pigment [15]. Hematite has antiferromagnetic magnetic properties at temperatures below 260K, but its magnetic properties are weakly ferromagnetic at ambient temperatures [16]. Therefore, hematite is used to reduce dye waste and can be used repeatedly with optimal results.

Hematite modification is carried out by adding *a* capping agent in the form of strach, which can increase the adsorption capacity more optimally. The use of strach as a modification of hematite is used because it does not require energy and chemicals that are less environmentally friendly [17,18]. In research conducted by Tatinting *et al.,* [19], the addition of capping in the form of PEG to Fe_2O_3 nanoparticles was able to improve adsorption performance.

2. Materials and methods

2.1 Preparation of Adsorbents and Adsorbate

The adsorbate used in this research is an artificial dye in the form of methylene blue (MB) which has the chemical formula $C_{16}H_{18}ClN_3S$, its molar mass is 319.352 $gmol^{-1}$, its solubility in water is approximately 43.6 gL^{-1} at a temperature of 25◦C, and has a wavelength of 664 nm. 1000 ppm MB stock solution was prepared by dissolving 0.1 g of MB in 100 mL. Then dilution was carried out until the concentration was 10 ppm. The MB used in this research was produced by PT. Smart Lab Indonesia.

The adsorbent materials in this research were pure Fe_2O_3 and Fe_2O_3 which was modified by adding strach. Fe_2O_3 is made from $FeSO_4$. The $7H_2O$ dissolved in 20 mL of distilled water with continuous stirring for 15 minutes on a magnetic stirrer. 50 mL of ammonia solution (25% v/v) was added drop by drop while continuing to stir until the pH reached 11 and a precipitate formed. The results obtained were filtered, washed with distilled water until the pH was neutral and dried in the oven for 24 hours. Then, the precipitate was calcined at a temperature of 400 ^{o}C for 4 hours using a furnace to obtain Fe_2O_3 (NF) nanoparticles. Making strach-modified Fe_2O_3 was carried out using the same procedure as making NF with a mass of 5 grams of $FeSO_4$. The $7H_2O$ and 3 grams of strach to obtain strach-modified Fe_2O_3 nanoparticles (NF-Strach). In this research, three types of strach were used as Fe_2O_3 modifications, namely tapioca (Tp), cornstarch (Mz) and mocaf (Mf).

2.2 Adsorption Process on Methylene Blue

Contacting NF and NF-Strach on 10ppm MB using the adsorption method was carried out by adding 0.1g of NF to 100 mL of 10ppm MB solution in a 250 mL Erlenmeyer flask. Stir using a shaker at 180 rpm for 120 minutes. Then filter the solution using Whatman grade 3 filter paper. Take the filtrate and measure the absorbance using UV-Vis spectrophotometry at a wavelength of 664 nm. Perform the same procedure using NF-Strach. Contacting was also carried out with variations in MB concentration, namely 10, 20, 30, 40, and 50 ppm to determine the effect of concentration on adsorption capacity.

2.3 Adsorbent Regeneration

Regeneration is carried out by filtering the remaining contact with MB dye and collecting the sediment. The precipitate was calcined at a temperature of 400 °C for 4 hours using a furnace to obtain NF and NF-Strach regeneration results. Then re-contacting 10 ppm MB dye was carried out for 60 minutes. This stage is repeated until the NF and NF-Strach mass is used up.

2.4 Adsorption Process in Liquid Waste from The Batik Industry

Preparation of adsorbate in the form of liquid waste from the batik industry was carried out by adding 90 mL with 10 mL of 0.01 M NaOH solution until the pH reached 11. Adding 0.1 g of NF to the sample solution in a 250 mL Erlenmeyer flask. Stir using a shaker at 180 rpm for 120 minutes. Then filter the solution using Whatman grade 3 filter paper. Take the filtrate and look at the spectrum and measure the absorbance using UV-Vis spectrophotometry in the wavelength range of 200-800 nm. Perform the same procedure using NF-Strach.

3. Results and Discussion

3.1 Synthesis of NF and NF-Strach

NF and NF-Strach adsorbents were synthesized using the coprecipitation method from $FeSO_4 .7H_2O$. Ammonia solution (25% v/v) was added as a precipitating agent. The results obtained were filtered, washed with distilled water until the pH was neutral to remove any impurities and dried using an oven. Then, the precipitate was calcined at a temperature of 400 °C for 4 hours using a furnace to obtain pure Fe_2O_3 nanoparticles. The results of the NF and NF-Strach that have been synthesized are in the form of solid powder with a brick red color. This color corresponds to that of α- Fe_2O_3 [20]. In terms of texture, NF-Strach has a smoother texture compared to NF.

(a) (b) (c) (d)

Fig. 1. Fe_2O_3 from synthesis results and its modifications (a) NF (b) NFS-Mf (c) NFS-Mz (d) NFS-Tp

3.2 Adsorption on Methylene Blue (MB)

From the adsorption results on 10 ppm MB, it can be seen that when adsorbing 10 ppm MB by NF and its modifications, the adsorption efficiency is almost the same, namely around 98%. In NF, adsorption efficiency increases with increasing contact time with an optimum time of 120 minutes and an adsorption efficiency of 98.02%. In NFS-Mz, the optimum time was obtained at 90 minutes. Meanwhile, in NFS-Mf and NFS-Tp, the optimum time was obtained at 15 minutes with the highest adsorption efficiency in NFS-Tp at 98.46%. It can be concluded that the modification using Strach has a positive effect on adsorption performance as seen from the faster optimum time achieved.

Figure 2 shows that in general, as the interaction time increases, there is an increase in the adsorption capacity (qt) of MB dye by NF and its modification tends to be constant up to a certain time. Based on Figure 2, the absorption of MB dye by NF and its modification occurred very quickly in the first 15 minutes. After that, the adsorption capacity of MB dye increased slowly and no longer experienced a significant increase (constant). The ability of an adsorbent to adsorb is limited to a certain time because the saturated adsorbent surface is no longer able to absorb MB dye even though the contact time continues to be increased.

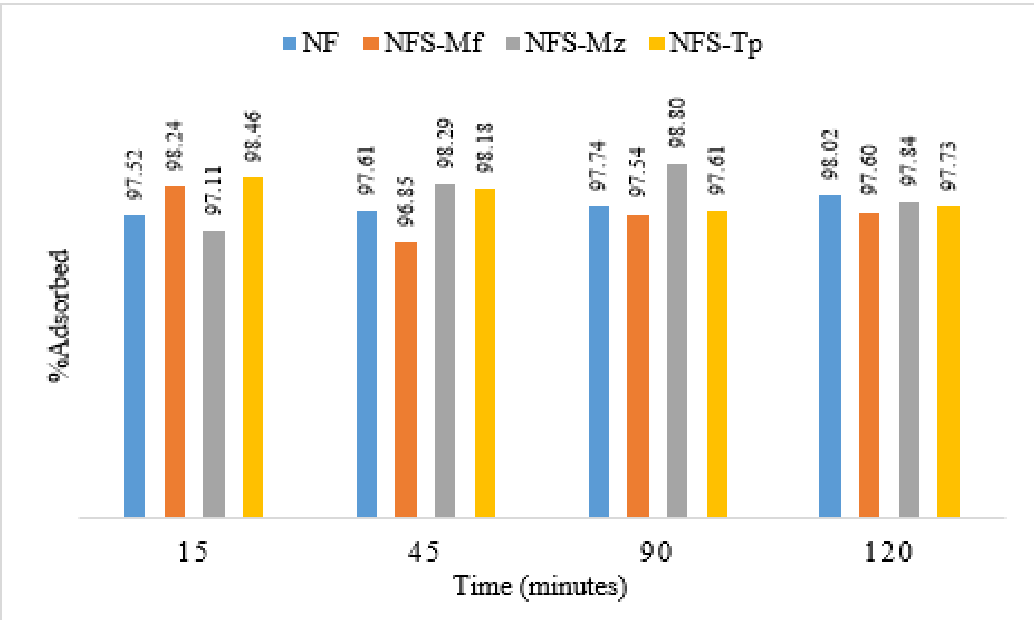
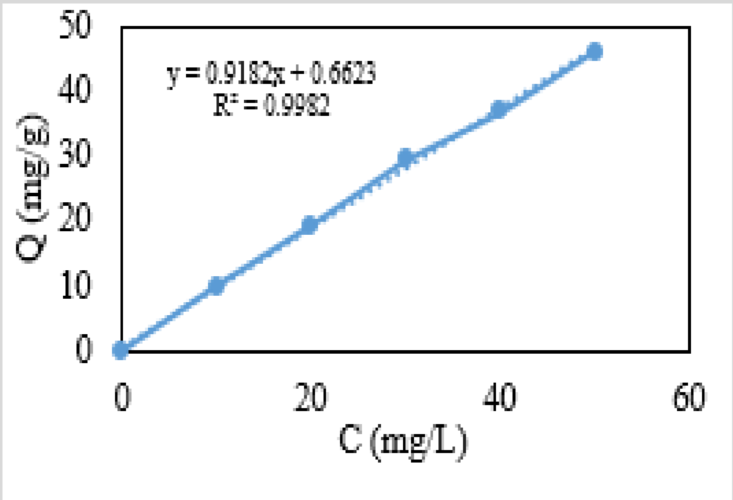


Fig. 2. Results of NF contacting and its modification of 10 ppm MB

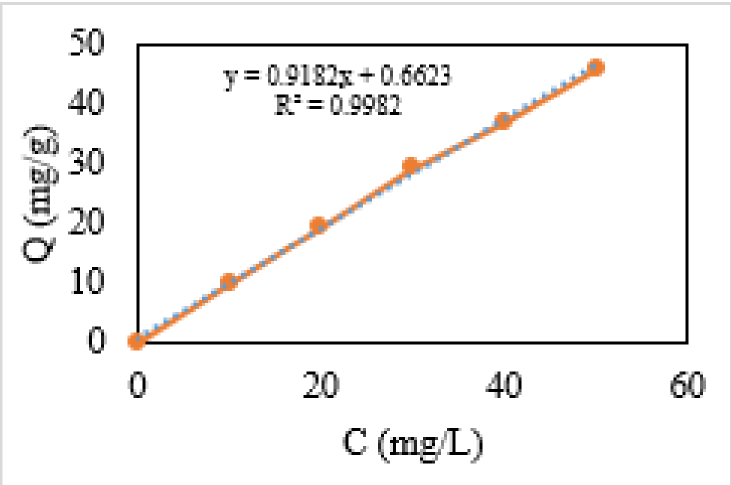
3.3 Effect of Initial Concentration on Adsorption Capacity

The initial concentration of MB dye is an important parameter to determine the capacity of the adsorbent to absorb adsorbate. This research was conducted to determine the optimum concentration of MB dye adsorbed by NF and its modifications. In the adsorption process, NF is reacted with MB dye solution at varying concentrations of 10-50 ppm with a contact time of 120 minutes.

The results of the analysis of the influence of the initial concentration of MB dye on adsorption capacity are shown in Figure 3. Figure 3 shows that the adsorption capacity of MB dye increases according to the increase in the concentration of MB dye used. When the initial concentration of MB dye was increased from 10 to 50 ppm, the adsorption capacity still increased to a concentration of 50 ppm with an adsorption capacity of 45.9994 mg/g for both NF and NF-Strach. The increase in the adsorption capacity of MB dye is due to the increase in the number of MB ions bound to the active groups of NF and NF-Strach so that the number of MB ions adsorbed is proportional to the number of active groups available on NF and its modifications. When the adsorbent active groups are saturated, the increase in adsorbate active groups will decrease, but if the adsorbent active groups are not yet saturated with adsorbate, then increasing the adsorbate concentration will increase the amount of adsorbed adsorbate [21].



(a)



(b)

Fig. 3. Graph of the effect of initial MB concentration on adsorption capacity by adsorbent (a) NF (b) NF-Strach

In this research, it can be concluded that the active group of the adsorbent is not yet saturated because under conditions of adsorbate with a concentration of 50 ppm, the adsorption capacity continues to increase. This is in accordance with what was stated by Langmuir and Freundlich, that the amount of substance adsorbed will be proportional to the added pressure or concentration [21]. In addition, this may occur because the active side of the adsorbent is not yet saturated with adsorbate so that increasing the adsorbate concentration can increase the amount of dye adsorbed [22]. This is in accordance with research conducted by Ariyanto, *et al.,* [23] that the concentration of methyl orange dye that can be adsorbed by active carbon from ketapang shells increases with each additional concentration. The results of another study by Ngapa and Ika [24] stated that the adsorption capacity of natural zeolite for adsorbed methylene blue and methyl orange increased as the adsorbate concentration increased.

3.4 Adsorption Kinetics

Adsorption kinetics is used to determine the rate of adsorption that occurs on the adsorbent against the adsorbate and is influenced by time. The contact time required to reach adsorption equilibrium is used as a measure of the adsorption rate. In this research, adsorption rate testing was carried out by estimating the reaction order. Adsorption kinetics are determined using the most appropriate adsorption reaction kinetics equation approach by comparing the squared value of the correlation coefficient (R^2) or the linearity of the reaction kinetics used. The adsorption kinetics used in this research include pseudo first order and pseudo second order. A comparison of the R^2 values of the adsorption kinetics of crystal violet dye can be seen in Table 1. The adsorption kinetics data were obtained empirically using pseudo first order and pseudo second order models.

Table 1
Adsorption kinetics equation for MB dye on NF and NF-Strach

Kinetic Model	N.F Parameter			NF-Strach Parameter		
	R^2	k	R^2	k	R^2	k
pseudo first order	0.2137	0.0155	8.375175	0.3189	0.0229	9.215328
pseudo second order	1	2.1624	28105.87	1	1.159013	14852.55

From Table 1, it can be seen that the experimental data on MB dye adsorption kinetics using NF and NF-Strach adsorbents have good suitability to be explained using a pseudo 2nd order kinetic model. This can be considered based on the high linearity of the relationship between the variables used in the model where the correlation coefficient value R^2 is closer to 1 than the pseudo 1st order kinetic model. The R^2 value which is close to 1 indicates that the pseudo 2nd order kinetic graph is more linear than the pseudo 1st order kinetic graph. Pseudo 2nd order kinetics assumes that adsorption occurs between adsorbents with MB dye is chemical adsorption, namely the formation of chemical bonds between the adsorbate molecules and the surface of the adsorbent.

Chemical adsorption involves coordination bonds as a result of the shared use of electron pairs by the adsorbate and adsorbent [25]. It can be assumed that the NF and NF-Strach mechanisms in adsorbing dyes, namely metal ions from iron oxide, can form bonds with water molecules from the adsorption medium. The water layer formed can undergo an association-dissociation mechanism to form deprotonated OH groups. This negatively charged layer can absorb positively charged dyes. The O group from OH will bond with the S group from MB so that adsorption can occur. MB dye is a cationic dye which has a positive charge so that in this case it can be absorbed well by NF and NF-Strach adsorbents. These kinetic results are in line with research conducted by Mahardiani et al., [26] that the adsorption kinetics of methylene blue using nanofiber derived from palm fiber follows pseudo-order 2. In addition, research by Latupeirissa *et al.,* [27] obtained the results of the adsorption kinetics of methylene dye blue by activated carbon from candlenut shells following pseudo second order.

3.5 NF Regeneration and NF-Strach

The concentration of NF and NF-Strach adsorbents of 0.1 g in 100 mL of 10 ppm MB dye can be reused up to 4 times and still leaves a mass of 0.047 g in NF and 0.042 g in NF-Strach. From Table 2, it can be seen that the adsorption capacity of NF and its modifications decreases as the number of repeated contacts increases.

Table 2
NF and NF-Strach regeneration results

Adsorbent	Q (mg/g) 5th contact				Adsorption Efficiency (%) 5th contact				mass (g) after the 3rd contact			
	1	2	3	4	1	2	3	4	1	2	3	4
N.F	9.6355	9.7633	9.6543	9.2237	96.3546	97.6330	96.5426	92.2373	0.078	0.066	0.056	0.047
NF-Strach	9.7257	9.4982	9.6486	8.9079	97.2570	94.9821	96.4862	89.0788	0.07	0.066	0.057	0.042

This regeneration test aims to evaluate and check the performance of NF and NF-Strach regarding its stability and ability to be reused in adsorbing dyes. His research is very important to explore the application of using NF and NF-Strach adsorbents to reduce dye waste. No obvious decrease was found in the adsorption percent of MB by NF and NF-Strach even after three adsorption–desorption experiments (Table 2). However, the absorption capacity decreases gradually as the recycling time increases. The results show that the NF and NF-Strach adsorbents are feasible to use, cost-effective and have the potential to be reused over time for the removal of MB from wastewater.

3.4 Adsorption on Batik Industry Liquid Waste

In this research, contacting NF and NF-Strach was carried out on textile wastewater from the batik industry in the Laweyan area, Surakarta. The constituent dyes in this waste are unknown so UV vis spectrophotometric testing was carried out at several wavelengths, namely 664 nm (methylene blue), 464 nm (methyl orange), and 557 nm (rhodamine B). Methylene blue and rhodamine b dyes are cationic dyes, while methyl orange is an anionic dye.

NF and NF-Strach adsorbents were contacted with textile wastewater from the batik industry. Contact results at wavelengths of 664 nm, 464 nm and 557 nm showed fluctuating results *for* both NF and NF-Stach adsorbents. At a wavelength of 664 nm, NF obtained the best results at 30 minutes of contact with an adsorption efficiency of 57.76%, while NF-Strach obtained a greater adsorption efficiency of 74.37% at 120 minutes. At a wavelength of 464 nm, it was obtained. The best results were at 30 minutes of contact with an adsorption efficiency of 50.03%, while NF-Strach obtained a greater adsorption efficiency, namely 66.77% at 120 minutes. Meanwhile, at a wavelength of 557 nm, the best results were obtained at 30 minutes of contact. with an adsorption efficiency of 50.87%, while NF-Strach obtained a greater adsorption efficiency, namely 65.50% at 120 minutes. It can be concluded that the modification using Strach had a positive effect on the adsorption performance seen from the greater %adsorbed shown in Figure 4.

Ion from iron oxide will form a deprotonated OH group. This negatively charged layer will absorb the dye. In this industrial liquid waste, it is not known what the dye components are, but it can be ascertained that this waste does not only consist of one dye, so there will be competition between the existing dyes to bind with the OH groups of the NF and NF-Strach adsorbents. Therefore, when compared with the contact results at MB 10 ppm, the adsorption efficiency resulting from contacting this industrial liquid waste has decreased.

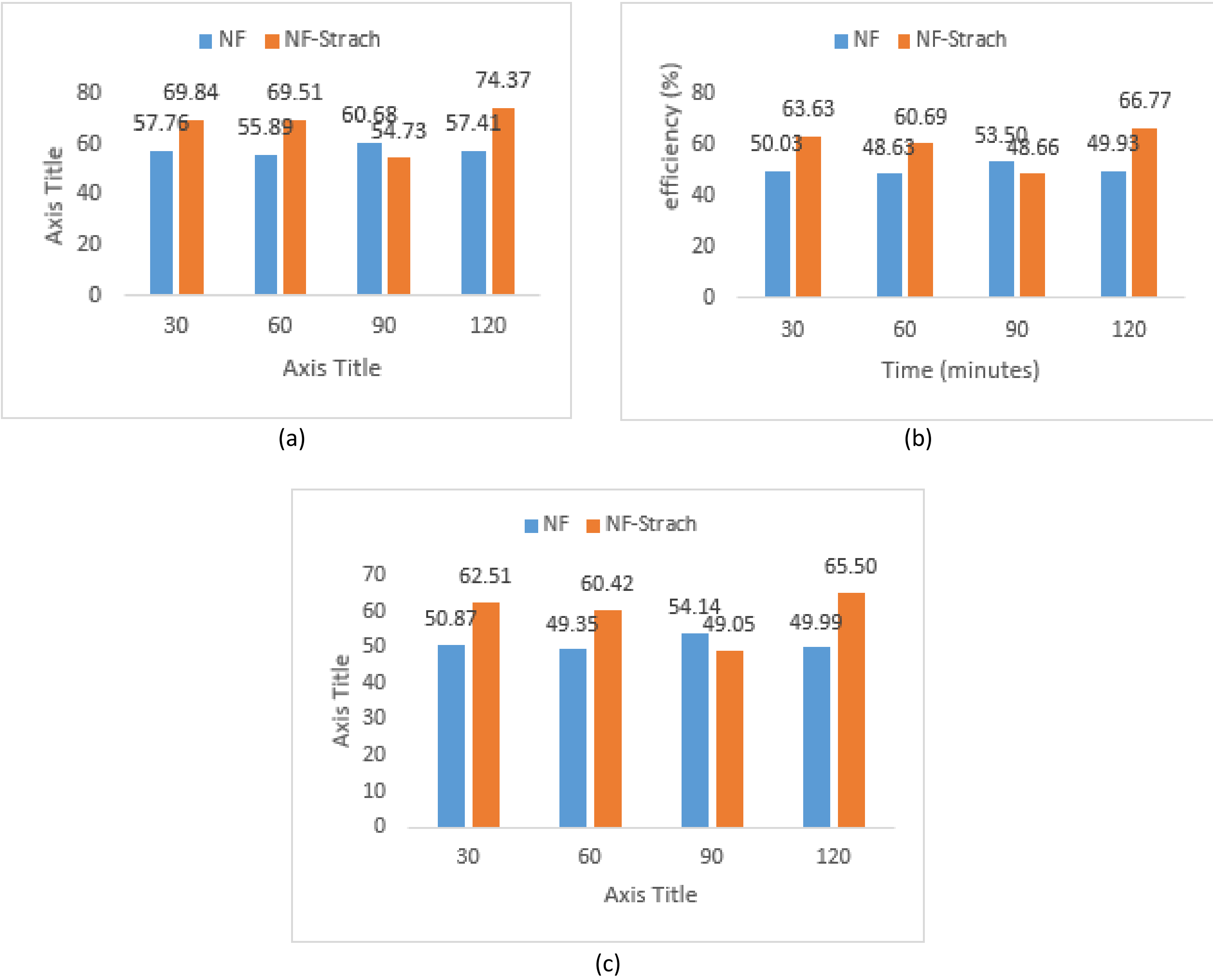


Fig. 4. Results of NF contacting and its modification of Laweyan batik industrial waste at wavelengths of (a) 664 nm, (b) 464 nm, and (c) 557 nm

4. Conclusion

Fe_2O_3 nanoparticles and their modification with *strach* have been successfully used as adsorbents to reduce liquid waste contaminated with dyes. Adsorption performance of NF-S better when compared to NF on 10 ppm MB dye. The adsorption capacity of MB increased with increasing concentration up to 50 ppm with the largest adsorption capacity of 45.9994 mg/g for both NF and NF-Strach. The adsorption kinetics that occurred were in accordance with the 2nd order pseudo model which assumed that degradation occurs chemically. Regeneration testing shows that the NF and NF-Strach adsorbents are suitable for use and have the potential to be reused to remove MB from wastewater. When contacting liquid waste from the batik industry, the results of NF-S adsorption performance were obtained better when compared to NF, but when compared with the contact results at MB 10 ppm, the adsorption efficiency produced by contacting this industrial liquid waste has decreased.

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