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Experimental Studies on the Removal of Textile Dye of Naphthol Green B from Artificially Contaminated Water Using Purolite MB400 Ion Exchange Resin

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ABSTRACT

Experimental research was conducted in order to point out the issue of dye removal from textile wastewater. Dyes are among the most dangerous substances emitted into the environment from the textile industry. The aim of this study was to determine the adsorption efficiency of the removal of naphthol green B from artificially contaminated water using Purolite MB400 ion exchange resin. In experimental research was used 0.3-1.2 mm fraction of Purolite MB400. 6 samples were prepared for this analysis. The experiment of sorption was performed at the same pH level, which ranged from 6 to 7. In order to find out whether the changes of the pH values of the solution after the sorption process did not affect the sorption process itself, optical density measurements were performed at different pH values. pH values ranged from 6 to 8.5. The sorption process was performed for 1 hour. A calibration curve at 484 nm was generated before measurements using Photo colorimeter. The residual carbon content of the samples was determined using a carbon analyzer. After 60 min of the adsorption process, the adsorption efficiency of the dye ranged from 50.96 % to 98.30 %.

Keywords: Circular economy; naphthol green B; Purolite MB400; sorption

INTRODUCTION

Lithuania is rich in groundwater resources: according to the Lithuanian Geological Survey (2022), there are currently more than 1780 deep groundwater wells in the country. Due to the fact that the quality of this resource, according to monitoring data, is quite good, groundwater is used as drinking water in Lithuania. Unfortunately, drinking water quality is poor in most European cities. The chemical, agriculture, manufacturing or transport industries, which are constantly expanding, are the main industries that are affecting and polluting the air, soil or water bodies (Rahman 2021).

Over the last 20 years, global fiber production has almost doubled: from 58 million tons in 2000 up to 109 million tons in 2020 (Trade report 2021). According to Organization for Economic Co-operation and Development (2021), the textile industry is one of the most water consuming industries. However, as the consumed amount of water is increasing, quantity of wastewater also rises. Dye effluents contain high biological and chemical oxygen demand and they are very rich in organic and inorganic pollutants such as dyes, heavy metals (chromium, zinc, iron, lead, mercury, cobalt, copper, etc.), sulphates, chlorides, petroleum products and other polluting chemicals (Wei 2020). These pollutants unfavourably affect aquatic life. Dyes are key constituents of textile effluent. Dyes found in wastewater hinder the sunlight reaching to water and decrease photosynthetic activity, reduce transparency and disturb the ecosystems (Zhang 2021). Dyes that are found in the effluents of the textile industry, could cause significant damage to the environment and have long-term adverse effects on human health (Slama 2021). Dyes could affect some vital organs in humans, such as the brain, kidneys, liver or heart (Kishor 2021). Direct inhalation of these substances may cause respiratory distress, asthma, allergies, nausea or even skin and eye irritation or dermatitis (Slama 2021). Synthetic dyes are nonbiodegradable, toxic, mutagenic and carcinogenic (Ardila-Leal 2021).

Untreated or incompletely treated wastewater could be harmful for the environment. The textile wastewater contains multiple toxic materials; its color results from the discharge of several dyes (Samsami 2020). It is noticeable even in low concentrations (>1 mg/L), the average concentration of textile effluent dye is about 300 mg/L (Slama 2021). Discharge of dying effluents in the environment could be the primary cause of a significant decline in freshwater bodies. Textile wastewater should be treated before dumping the water into the rivers, reservoirs or other open water bodies. There are various methods used for wastewater treatment, for example, physical, chemical or biological. All of these methods have advantages and disadvantages. However, they are not universal and usually suitable for use only in cases where the concentration of dyes is low.

Textile wastewater contains highly variable dyes, that have structural varieties, including acidic, basic, reactive, azo, metal complex or diazo dyes. Naphthol green B is an iron complex belonging to the group of acid dyes. Dyes are available in liquid, powder, paste or granular form. Naphthol green B is a water-soluble dye with the molecular formula $C_{30}H_{15}FeN_3Na_3O_{15}S_3$ (Gunasundari 2020). This material is used to dye collagen as well as wool, nylon, paper and soap.

Adsorption has emerged as one of the most promising technique due to its versatility and simplicity (Sočo 2021). The advantages of adsorption process include low cost, simplicity of operation and the capability to treat dye effluent even at high concentrations (Ardila-Leal 2021).

Nowadays, researchers are looking for adsorbents that are inexpensive, eco-friendly and abundantly available (Obulapuram 2021). Many different solid adsorbents vary in size, shape, surface area or porosity (Malatji 2021). The following materials could be used as sorbents as low-cost adsorbents to remove the color from dye-containing effluent: modified wheat residue, straw, peel and etc.

Purolite MB400 is a resin mixture which is used for direct purification of water. Only 0.3-1.2 mm fraction of Purolite MB400 was used in experimental research. Smaller particles were chosen because they have a higher specific surface area that should increase the adsorption efficiency values: the larger the surface area, the more intensive the sorption process (Yang 2021).

The aim of this study was to determine the adsorption efficiency of the removal of naphthol green B from artificially contaminated water using Purolite MB400 ion exchange resin.

METHODOLOGY

Naphthol green B that was used in the experimental research, was in powder form. Experimental sorption study was performed in a narrow range of pH values ranging from 6.0 to 7.0. The dye sorption process was studied for 60 min. by continuously stirring aqueous solutions with a magnetic stirrer.

The aim of this experimental research was to analyze the relationship between the adsorbent and adsorption capacity by changing the adsorbent dosage when the initial concentration of naphthol green B maintained at a high concentration of 100 mg/L. Theoretically, as the amount of introduced adsorbent into the solution is increased, corresponding increase in adsorption sites should be observed.

Table 1 provides information on the samples used in the experimental study. Sample No. 2 - blank sample - used for quality assurance of experimental study.

TABLE 1. Samples used in the experiment

Sample No.	Amount of Purolite MB400, g	Dye concentration, mg/L
1.	5	0
2.	0	0
3.	5	100
4.	10	100
5.	50	100
6.	100	100

Sample No. 1 had only 5 g of Purolite MB400 ion exchange resin, concentration of naphthol green B was 0 mg/L. This was done in order to compare the residual carbon concentration in the samples No. 1 and No. 3, because the amount of sorbent was the same.

Ion exchange resin is commonly used for preparation of deionized water. Purolite MB400 is a mixed resin for desalination. Granules of Purolite MB400 have large surface area. Based on other scientific work where it was mentioned that ion exchange resin could be used as sorbent for the removal of heavy metals, nitrites, nitrates or etc.

The sorption process was stopped after one hour by separating the Purolite MB400 ion exchange resin from the prepared aqueous solution of naphthol green B dye and filtering the solution through a "VWR Qualitative filter paper 413". The separated particles of Purolite MB400 ion exchange resin was not reused in the subsequent stages of the study.

The pH values of the aqueous solutions were measured before and after the sorption process. Prior to the experimental study, the "Mettler Toledo Multi seven" pH meter was calibrated using two buffer solutions with different pH values, of 4.0 and 7.0 pH. The pH of the solution was adjusted with 0.1 M aqueous HNO_3 and 0.1 M NaOH. The tests were performed at room temperature. The temperature of the prepared samples ranged from 21 to 23 °C.

The following laboratory equipment and chemical reagents were used for the experimental study:

1. chemical reagents - naphthol green B (p. a.); Purolite MB400 granules; deionized water; aqueous solutions of 0.1 M HNO_3 and 0.1 M NaOH.
2. laboratory equipment - 6 l glass vessel; "Radwag" analytical balances; "VWR Qualitative filter paper 413" filter paper; pH meter "Mettler Toledo Multi seven"; metal sieves, 50 ml volumetric flasks A class; 1, 2, 10 ml graduated pipettes, "Shimadzu UV-1800" Photo

colorimeter, glass cuvettes 4 cm, "Shimadzu TOC" carbon analyzer.

A "Shimadzu UV-1800" Photo colorimeter was used to determine the optical density of aqueous solutions. This device makes it possible to objectively assess the change in color intensity of a colored solution by measuring its optical density. 4 cm glass cuvettes were selected for measuring the optical density of aqueous solutions. Measurements were repeated 3 times and the mean value was calculated. Figure 1 represents a calibration curve of naphthol green B at 484 nm which was generated before the assay.

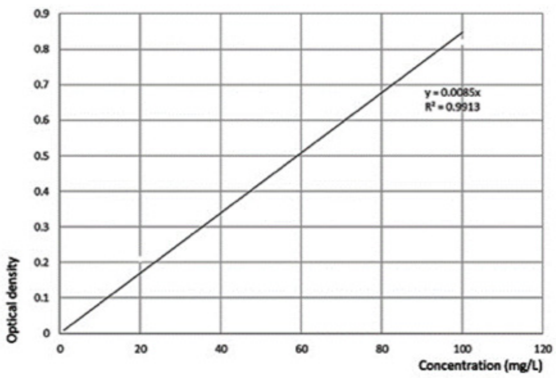

FIGURE 1. Calibration curve at 484 nm

Based on literature review, it was decided to use 484 nm wavelength curve. In the future the same experimental research could be done using 590 nm and 664 nm wavelength curves. 664 nm - maximum absorption wavelength.

The color of the naphthol green B solutions was not very intense, it was light green. The initial dye concentration in all samples was 100 mg/L. The molecular weight (M) of naphthol green B was 878 g/mol:

M ($C_{30}H_{15}FeN_3Na_3O_{15}S_3$) = 30 (C amount) + 15 (H amount) + (Fe amount) + 3 (N amount) + 3 (Na amount) + 15 (O amount) + 3 (S amount).

The amount of C in sample was 41.0 mg/L: W (C) = ((30 * 12) / 878) * 100 = 41.0 mg/L.

Based on the methodology, experimental study was performed to evaluate whether the naphthol green B used in the textile industry could be removed from aqueous effluents using sorbent made from Purolite MB400 ion exchange resin. The results of the study are presented in the graphs and tables in the next section.

RESULTS AND DISCUSSION

RESIDUAL CARBON AND NAPHTHOL GREEN B CONCENTRATIONS

The residual dissolved carbon content of the aqueous solution effluent after the sorption process was determined using a "Shimadzu TOC" carbon analyzer. It is a fast and reliable method for monitoring the concentration of organic matter dissolved in water using carbon as an indicator. After the sorption process, lower values of dissolved carbon content were recorded in samples No. 1 to No. 6 than in wastewater before the sorption process. The exact residual carbon values are given in Table 2.

TABLE 2. Carbon concentration in samples after sorption process

Sample No.	C, mg/L
1.	8.03
2.	1.16
3.	18.22
4.	12.30
5.	6.11
6.	7.73

As the concentration of naphthol green B in samples No. 1 and No. 2 was equal to 0, it was calculated the values of the naphthol green B concentration only in samples No. 3 to No. 6. Dye concentrations after sorption process are given in Table 3.

TABLE 3. Concentration of naphthol green B after sorption process

Sample No.	Dye concentration after sorption process, mg/L
3.	49.04
4.	33.87
5.	1.98
6.	1.70

MEASUREMENTS OF OPTICAL DENSITY

Optical density measurements of 100 mg/L naphthol green B solution were also performed by changing the pH values from 6.0 to 8.5. This was done in order to investigate whether the change in pH of the naphthol green B solution after sorption process did not significantly affect the sorption process or the optical density, which was chosen as an indicator to evaluate the dye concentration in water.

The color of dye and sorption process itself depends on pH values of aqueous solution.

The upper value range of pH, 8.5, was chosen based on the general requirements for wastewater discharged into the natural environment, presented in the Lithuanian Wastewater Management Regulation. Meanwhile, pH 6.0, the lowest pH value used in the study, corresponded to the pH value of naphthol green B dissolved in naturally deionized water, so it was decided not to perform measurements with aqueous solution values lower than pH 6.0. The measurement step chosen for the experimental study was 0.25. The obtained optical density values are given in Table 4.

TABLE 4. Dependence of the optical density of dyed wastewater on pH

Dye concentration, mg/L	pH	Optical density
100	6.00	0.767
	6.25	0.771
	6.50	0.775
	6.75	0.779
	7.00	0.781
	7.25	0.782
	7.50	0.784
	7.75	0.785
	8.00	0.786
	8.25	0.787
	8.50	0.788

Fluctuations in pH values from 6.0 to 8.5 were found to have no significant effect on the optical density values of the naphthol green B solution: optical density values ranged from 0.767 to 0.788. The change in optical density values was only 2.73 %. Changes in color intensity were observed with the eye: the color of the solution remained light green in the samples No. 3 and No. 4 throughout the experimental study. On the other hand, the color of the solution disappeared in the samples No. 5 and No. 6 (after sorption process aqueous solutions remained clear).

ADSORPTION CAPACITY AND ADSORPTION EFFICIENCY

Study on the removal of the naphthol green B from artificially contaminated water using Purolite MB400 ion exchange resin showed that the adsorption capacity of the 50 g and 100 g granules of sorbent differed minimally, ranging from 9.80 mg/g to 9.83 mg/g. Meanwhile, the adsorption capacity values of the granules in the presence of small amounts ranged from 5.09 mg/g to 6.61 mg/g. The values of concentration of naphthol green B after sorption, adsorption capacity and adsorption efficiency are given in Table 5.

TABLE 5. Dependence of adsorption capacity on sorbent content

Sample No.	Dye concentration after sorption process, mg/L	Adsorption capacity, mg/g	Adsorption efficiency, %
3.	49.04	5.09	50.96
4.	33.87	6.61	66.13
5.	1.98	9.80	98.02
6.	1.70	9.83	98.30

Although a significant difference in the adsorption capacity values was observed when comparing the 5 g and 10 g granules samples with the 50 g and 100 g granules samples, the difference between samples No. 5 and No. 6 was relatively small: the adsorption value for 100 g granules increased only by 0.28 %.

It could be speculated that increasing the amount of granules could increase the adsorption capacity if the sorption time would be longer than 60 minutes or fraction would be smaller than 0.3-1.2 mm.

An experimental study was carried out to determine whether Purolite MB400 ion exchange resin granules can be used to remove naphthol green B, a dye used in the textile industry, from wastewater.

Based on the results of experimental study, it could be assumed that sorbent of Purolite MB400 ion exchange resin is suitable for removal of naphthol green B from wastewater under the experimental conditions described earlier when the efficiency of the sorption process is controlled by dissolved carbon content. In the future, the experimental study can be repeated by changing the method of sorbent preparation – granules could be washed with deionized water several times before the following instance of sorption process.

It has been observed that the residual carbon content in aqueous solutions decreases with increasing sorbent of Purolite MB400 content. In sample No. 5, which has 50 g ion exchange resin, the residual carbon concentration after sorption process was only 6.11 mg/L. Malatji and others also pointed out that the bigger concentration of sorbent is used, the higher adsorption capacity could be reached.

During this experiment two methods were used in order to determine the results: residual carbon concentration was measured using carbon analyzer “Shimadzu TOC”; residual concentration of naphthol green B was determined using calibration curve at 484 nm wavelength. It is very important to ensure the accuracy of the measurements. Different methods allow to determine different parameters.

The experiments were conducted for the study of effects against the changes in pH. Obulapuram and others found out that among all, the optimal removal of dye was found out at a pH of 4. Although this experiment was done in a narrow range of pH values ranging from 6.0 to 7.0, it can be stated that the change in the pH of the solution did not significantly affect the sorption result.

Residual concentrations of the naphthol green B were calculated from the optical density values measured with a "Shimadzu UV-1800" Photo colorimeter. Values were converted to the naphthol green B concentration in water based on a calibration curve.

In all cases, after the sorption process the concentration of naphthol green B was lower than 100 mg/L: the lowest residual dye concentration was found in a sample containing 100 g of sorbent made from Purolite MB400 ion exchange resin granules. The volume of samples prepared for the experimental study of sorption process was 500 mL. The calculated adsorption efficiency for samples No. 3 t0o No. 6 ranged from 50.96 % to 98.30 %.

It is expected that the sorbent of Purolite MB400 granules can be reused several times: the washed and dried granules could be reused at least a few more times. The granules of Purolite MB400 can be washed not only with deionized water, but with other substances such as sodium chloride.

CONCLUSION

Purolite MB400 ion exchange resin may be used for the removal of the naphthol green B from artificially contaminated water. After 60 min of the adsorption process, the adsorption efficiency of the dye ranged from 50.96 % to 98.30 %. The lowest residual concentration of the naphthol green B was recorded in sample with 100 g of Purolite MB400 ion exchange resin granules. Adsorption capacity, of naphthol green B, values ranged from 5.09 mg/g to 9.83 mg/g. As the pH value increased from 6.0 to 8.5, only a slight increase in the optical density of the naphthol green B solution was observed, therefore it can be stated that the change in the pH of the solution after the sorption process did not significantly affect the sorption result and optical density of investigated solution.

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DECLARATION OF COMPETING INTEREST

None

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