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Synthetic Wastewater Treatment using Agro-Based Adsorbents

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Abstract

This study was undertaken to determine the treatment a binary mixture of dye wastewater (containing Naphthol Green B) and the sugar industry wastewater for removal of color. The specific treatment in the current research consists of adsorption using low-cost adsorbents and microfiltration using Whatman-41 microfilters. Considerations of this treatment process are to take the samples using batch adsorption and avoid coagulation with further dilution. Numerous runs are made, with the ideal waste samples prepared in the laboratory. As a 1st step in the study, different dye concentrations are considered using different concentrations of sugar wastewater. Samples are treated with 3 different Agro-based low-cost adsorbents (orange peel, peanut hull, and Powdered Activated Carbon (PAC)). Transmittance values for Naphthol Green B after treatment with orange peel and peanut hull are 83.12 % and 76.98 % respectively. Peanut hull has the highest transmittance of 76.98 % with < 425 µm size. Orange peel contributes to the highest transmittance of 83.12 % with a 2 g dosage. The values of transmittance after treatment with PAC are taken as the datum for the comparison of adsorption performance after treatment using orange peel and peanut hull. Peanut hull has the highest Non-Purgeable Organic Carbon (NPOC) measurement of 37.86 mg/L when mixed with 600 ppm of sugar wastewater. Similarly, when mixed with 600 ppm of sugar wastewater, orange peel contributes to the NPOC value of 35.06 mg/L. These treated samples using low-cost adsorbents can be considered as pre-treated wastewater that can be sent to municipal wastewater treatment plants.

Keywords: Dye wastewater, Sugar wastewater, Adsorption, Absorbance, Transmittance, Microfiltration, Low-cost adsorbents.

Introduction

Sugar is the most essential substrate in the human diet. In this emerging world, the consumption of sugar is increasing in daily life. More than 115 countries are producing sugar in the world [1]. Since 1789, the United States of America (USA) government has continued to provide support for the domestic sugar industries [2]. Sugar cane and sugar beets are the 2 main sources from which sugar is produced. Sugar is used in candies, soft drinks, cakes, beverages, ice creams, and many other food products. The sugar industry requires massive amounts of water to produce sugar. Many types of pollution loads are released, in the form of solid, liquid, and gaseous states. Chemicals such as $Ca(OH)_2$, H_3PO_4, CO_2, SO_2, $NaOH$, Na_2CO_3, and HCL are used in the sugar industry to produce sugar and pre-treatment wastes [3].

The disposal of untreated wastewater from the sugar industry is a major environmental problem. This wastewater contains significant amounts of Total Dissolved Solids (TDS) and Total Suspended Solids (TSS). This water is not useful for irrigation purposes. If disposed on land, the rate of infiltration decreases due to the increase in TDS and TSS. The increase in TSS causes salt deposition, which leads to a decrease in soil porosity. Similarly, higher TDS contents are not favorable for crop growth [4]. This shows the typical environmental pollution caused by sugar wastewater.

Dyes are colored aromatic compounds used to impart color on different substrates. They may be organic or inorganic. In the USA, dye additives are used in textiles, food products, cosmetics, drugs, and medical devices [5]. Dyes are classified in several ways based on the source of materials, chromophore, nuclear structure, and industrial classification. Depending on the source of materials, they are classified into natural dyes and synthetic dyes. Depending on chromophore, they are classified into nitro and nitroso dyes, azo dyes, triaryl methane, anthraquinone dyes, and indigo dyes. Depending on the nuclear structure, they are classified into anionic and cationic dyes. According to industrial classification, they are classified into protein textile dyes, cellulose textile dyes, and synthetic textile dyes. Cellulose textile dyes are classified into direct dyes, vat dyes, basic dyes, and fiber reactive dyes. Protein textile dyes are again classified into acid dyes and mordant dyes. Synthetic textile dyes are again classified into disperse dyes and solvent dyes [5,6].

Naphthol Green B comes under industrial dye. The C.I. (Color Index) number is 10020, with the C.I. name of acid green 1. It comes under the class of nitroso with acidic ionization. It is very soluble in aqueous solutions and is green in color. The structure of Naphthol Green B is shown in **Figure 1**. Its molecular formula is $(C_{10}H_5NO_5SNa)_3Fe$, with a formula weight of 878.79 g/mol [7].

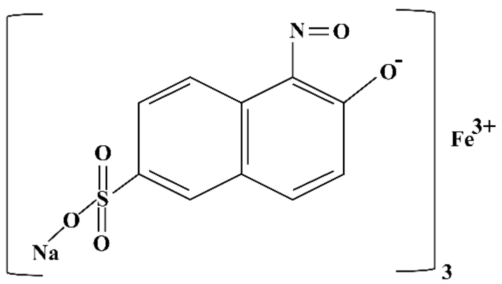


Figure 1 Structure of Naphthol Green B.

Research methodology

Experimental parameters

The spectrometer parameters of dye wastewater are transmittance and absorbance. For distilled water, the absorbance and transmittance values are 0 and 100 %, respectively. This implies that distilled water has a maximum transmittance capacity of ultraviolet radiation and a minimum absorbance capacity which is nearly zero. Due to this reason, it is considered as the standard measure to calibrate a spectrophotometer.

Transmittance

Transmittance (T) is a measurement of how much light passes through a substance. The higher the amount of light that passes through, the larger the transmittance. Transmittance is defined as the ratio of the intensity of incident light: the intensity of transmitted light is as given in Eq. (1). At times, this fraction may be represented as a percentage, where it is called the percentage transmittance (%T) [8].

$$T = \left(\frac{I}{\mathrm{I}}\right). \tag{1}$$

where the intensity of incident light is *I*. and the intensity of transmitted light is I.

Absorbance

The expression for Absorbance (A) is given in Eq. (2).

$$A = log_{10}(\frac{1}{T}). \tag{2}$$

Consequently, absorbance can also be given in terms of the percentage transmittance, as represented in Eq. (3).

$$A = 2 - log_{10}(\ \%T\ ). \tag{3}$$

According to Beer-Lambert's law, the absorbance of light, as it passes through a solution, is directly proportional to the path length of light through the material (l) and the concentration (c).

$$A = €\text{lc}. \tag{4}$$

where € is a constant called the molar absorptivity. This constant has a specific value for a given substance, provided the temperature of the substance and the wavelength of light passing through it are kept unchanged [8]. This is an extremely useful relationship that allows concentrations of unknown solutions to be found by measuring the absorbance of light through a sample.

Non-Purgeable Organic Carbon (NPOC)

This comprises all the carbon present after the sample has been acidified and purged with purified air to remove inorganic carbon. Note that volatile organics will be lost during the purging. It is common, but incorrect, for laboratories to measure NPOC, but report it as Total Organic Carbon (TOC). In the Shimadzu TOC-L machine for the addition method, the parameters Purgeable Organic Carbon (POC) and NPOC are measured. The TOC is then calculated. Measurement of the non-purgeable organic compounds is done after POC analysis using catalytic combustion at 680 ºC and subsequent determination of the resulting carbon dioxide using non-dispersive infrared sensor (NDIR) detection [9]. The TOC is calculated via the addition shown in Eq. (5).

$$\text{TOC} = \text{POC} + \text{NPOC}. \tag{5}$$

For the direct method or NPOC method, it is assumed that the sample does not contain any significant amounts of volatile or purgeable organic compounds. According to this assumption, the TOC is directly determined as NPOC. Acidification of the sample is done using a mineral acid (for instance HCL) to a pH < 2, whereby carbonates and hydrogen carbonates are completely converted to carbon dioxide. The carbon dioxide is removed from the sample solution via a spare gas. Direct NPOC measurement (like TC measurement) is done via oxidation to CO_2. Subsequent NDIR detection is conducted. The TOC corresponds to the NPOC, as is given in Eq. (6) [10].

$$\text{TOC} = \text{NPOC}. \tag{6}$$

Experimental materials

The materials used in this research work include: (a) Adsorbents: peanut hull, orange peel; (b) Reference Adsorbent: Powdered Activated Carbon (PAC); (c) Wastewater: dye wastewater containing Naphthol Green B, sugar wastewater.

Adsorbents

The adsorbents used in this study are peel from oranges and the hull of peanuts: (i) peanut hull: The peanut hull is washed with water thoroughly with tap water. Then, they are oven-dried at 70 ºC for 24 h. Then, the dried peanut hull is ground with a Preethi blender and sieved into 4 assorted sizes. The retained matter on 4 sizes sieves is used in this research to treat the combined wastewater of Naphthol Green B and sugar wastewater. This helps us to determine the variation in adsorption capacity due to the change in the surface area. (ii) orange peel: Ripened oranges are collected, and the peel is taken out separately. The peel is then oven-dried at 70 ºC for 24 h. The dried orange peels are ground with a Preethi blender and sieved. The matter is passed through 425 μm. Single size is maintained, and the dosage is varied in the case of orange peel.

Reference adsorbent

The reference adsorbent used in this study is PAC. Its manufacturer is Darco, with its grade HDC. First, the experiment is conducted with PAC, and the results are tabulated. PAC values are used as reference values to compare the treatment capacity of the low-cost adsorbents (peanut hull and orange peel).

Wastewater

wastewater is prepared in the laboratory with Naphthol Green B and Sugar Powder. Mother samples are prepared with 1,000 ppm with both dye and sugar separately. Then, specific concentrations are fixed based on the trial test performed, resulting in noticeable variance in transmittance. Then, combined wastewater is prepared, with respective concentrations and in different combinations. One hundred mL is the fixed quantity of combined wastewater, with equal halves of dye and sugar wastewater. The molecular structure of Naphthol Green B is shown in **Figure 1**.

Experimental Methods

There are many methods to treat dye wastewater. There are many physio-chemical treatment methods, such as adsorption, coagulation, flocculation, biological treatment, electrochemical oxidation, etc. In this research, the authors mainly concentrate on the combined method of adsorption and microfiltration.

Adsorption

Adsorption is a surface phenomenon with a common mechanism for organic and inorganic pollutant removal. When a solution containing absorbable solute encounters a solid with a highly porous surface structure, liquid-solid intermolecular forces of attraction cause some of the solute molecules from the solution to be concentrated or deposited on the solid surface. The solute retained (on the solid surface) in adsorption processes is called an adsorbate, whereas the solid on which it is retained is called an adsorbent. This surface accumulation of adsorbate on adsorbent is called adsorption. This creation of an adsorbed phase having a composition different from that of the bulk fluid phase forms the basis of separation by adsorption technology [11].

Micro-filtration

Micro-filtration (or MF for short) is one of the pressure-driven membrane processes in the series micro-filtration, ultra-filtration (UF), nano-filtration (NF), and reverse osmosis (RO). The micro-filtration process uses a membrane- a simple permeable material- which, in the case of micro-filtration, only allows particles smaller than 0.1 microns to pass through it. The microfiltration membrane can consist of various materials like, for example, polysulfide, poly-vinyl-di-fluoride (PVDF), poly-ether-sulfone (PES), ZrO_2, and carbon. The pore size varies between 0.1 and 5 microns. Because the pores are large compared to other mentioned filtration techniques, the pressure needed to send the liquid through a micro-filter membrane is limited to 0.1 to 3 bar [12].

Approach

The experiments are performed by adding adsorbent to 100 mL of combined wastewater. The orange peel powder is added in 4 different weights of 0.5, 1, 1.5, and 2 g of powder passed through a 425 μm sieve. The 5 assorted sizes (powder retained on 3327 - 2380, 2380 - 2362, 2362 - 600, and 600 - 425 μm, < 425 μm) of peanut hull are added at a constant dosage of 2 g per 100 mL. A mechanical shaker is used to mix the adsorbents thoroughly at 100 rpm for 1 min and 30 rpm for 45 min. We allow them to undergo an adsorption process for 24 h and filter them with micro-filters (Whatman-41). The absorbance and transmittance values are tabulated before and after the treatment of combined wastewater using a spectrophotometer. The preparation of mother samples, wastewater samples, and treatment is conducted at room temperature (25 ± 2 ºC) unless otherwise stated. The adsorption capacity at equilibrium is calculated using the following Eq. (7):

$$q_e = \frac{(Co - Ce)\ x\ V}{W} \tag{7}$$

where,

Co is the initial concentration (ppm)
Ce is the equilibrium after treatment (ppm)
V is the volume of each sample considered (mL)
W is the weight of adsorbent added (g/L)

The details of the types of equipment used to perform the experiments are mentioned in **Table 1**.

Table 1 Details of the instruments used for the current study.

Name	Model	Application
Weighing Balance	OHAUS PA1502	An instrument used to measure sensitive weights.
Spectrophotometer	Carolina #65-3303	An instrument used to measure transmittance and absorbance at an adjustable wavelength with spectral bandpass.
Platform Shaker	Innova 2300 - (115V 60 CV AC)	An instrument that provides vibration to ensure proper mixing of samples.
TOC Analyzer	Shimadzu TOC-L	An instrument that adopts combustion catalytic oxidation method and measures TC, TOC, NPOC, etc.,
Fisher Oven	200 SERIES (Model 230F)	A conventional benchtop laboratory oven with accurate control of the temperature.

Run protocols

Tables 2 and **3** are the run protocols for this research study. **Table 2** represents the run protocols for high concentration dye treated with PAC and orange peel. **Table 3** represents the run protocols for high concentration dye treated with peanut hull. The parameters which are varied in this research consist of the concentration of dye wastewater and the concentration of sugar wastewater, type of adsorbent, and weight (dosage) of adsorbent.

Table 2 Run protocols for high concentration dye treated with PAC and orange peel.

Run order	Dye concentration (ppm)	Adsorbent weight (g)	Sugar wastewater concentration (ppm)
1	200	0.5	100
2	200	1	100
3	200	1.5	100
4	200	2	100
5	200	0.5	200
6	200	1	200

Run order	Dye concentration (ppm)	Adsorbent weight (g)	Sugar wastewater concentration (ppm)
7	200	1.5	200
8	200	2	200
9	200	0.5	300
10	200	1	300
11	200	1.5	300
12	200	2	300
13	200	0.5	400
14	200	1	400
15	200	1.5	400
16	200	2	400
17	200	0.5	500
18	200	1	500
19	200	1.5	500
20	200	2	500
21	200	0.5	600
22	200	1	600
23	200	1.5	600
24	200	2	600

Table 3 Run protocols for high concentration dye treated with peanut hull.

Run order	Dye concentration (ppm)	Size of the adsorbent (µm)	Sugar wastewater concentration (ppm)
1	200	3327 – 2380	100
2	200	2380 – 2362	100
3	200	2362 – 600	100
4	200	600 – 425	100
5	200	< 425	100
6	200	3327 – 2380	200
7	200	2380 – 2362	200
8	200	2362 – 600	200
9	200	600 – 425	200
10	200	< 425	200
11	200	3327 – 2380	300
12	200	2380 – 2362	300
13	200	2362 – 600	300
14	200	600 – 425	300
15	200	< 425	300
16	200	3327 – 2380	400
17	200	2380 – 2362	400
18	200	2362 – 600	400
19	200	600 – 425	400
20	200	< 425	400
21	200	3327 – 2380	500
22	200	2380 – 2362	500
23	200	2362 – 600	500
24	200	600 – 425	500

Run order	Dye concentration (ppm)	Size of the adsorbent (µm)	Sugar wastewater concentration (ppm)
25	200	< 425	500
26	200	3327 – 2380	600
27	200	2380 – 2362	600
28	200	2362 – 600	600
29	200	600 – 425	600
30	200	< 425	600

Results and discussion

The observations are compared, and the results are tabulated to interpret the performance and behavior of the low-cost adsorbents with 4 different dosages for PAC and orange peel powder (0.5, 1, 1.5, and 2 g) and peanut hull retained on 5 different sieve sizes of 3327 - 2380, 2380 - 2362, 2362 - 600, and 600 - 425 µm, < 425 µm with 4 different dosages of dyes of low, lower medium, upper-medium, and high, and 6 concentrations of sugar wastewater of 100, 200, 300, 400, 500, and 600 ppm. The effect of pH, dye concentration, contact time, and surface area are considered while conducting the experiments.

Effect of pH

pH is very important in the adsorption process, especially in dye adsorption. The rate of adsorption and the magnitude of electrostatic charges due to ionized dye molecules is controlled by the level of pH, preferably medium. pH is inversely proportional to anionic dye adsorption and directly proportional to cationic dye adsorption. The initial pH of the combined wastewater samples is in the range of 6 - 9.

Effect of dye concentration

The initial concentrations of dye taken in this research are considered based on trial and error methods. Trial samples are taken and tested with different concentrations. Suitable concentrations are selected to distinguish the change in transmittance (%). As the concentration increases, the transmittance value decreases.

Effect of contact time

During the experiments, the contact time of the combined wastewater samples with the adsorbent is 24 ± 1 h. The whole experiment for a set of samples is completed in not more than 48 h. The effect of oxidation of dye increases as the duration of the experiment increases.

Effect of surface area

Orange peel is considered in 4 dosages with the same sieve size (powder passed through 425 µm sieve). Peanut hull is added to the same dosage with 5 sieve sizes. The phenomenon observed after treatment is an increase in the rate of adsorption due to an increase in surface area, which has an impact on treatment capacity.

Transmittance (%)

The amount of light that can pass through a substance is called transmittance. Distilled water is considered a datum due to its 100 % (maximum) transmissivity. It can also be defined as the ratio of the intensity of incident light to transmitted light. It is measured in %. The transmittance, when treated with low-cost adsorbents and compared with PAC at optimum dosage and size, is plotted in **Figure 2**.

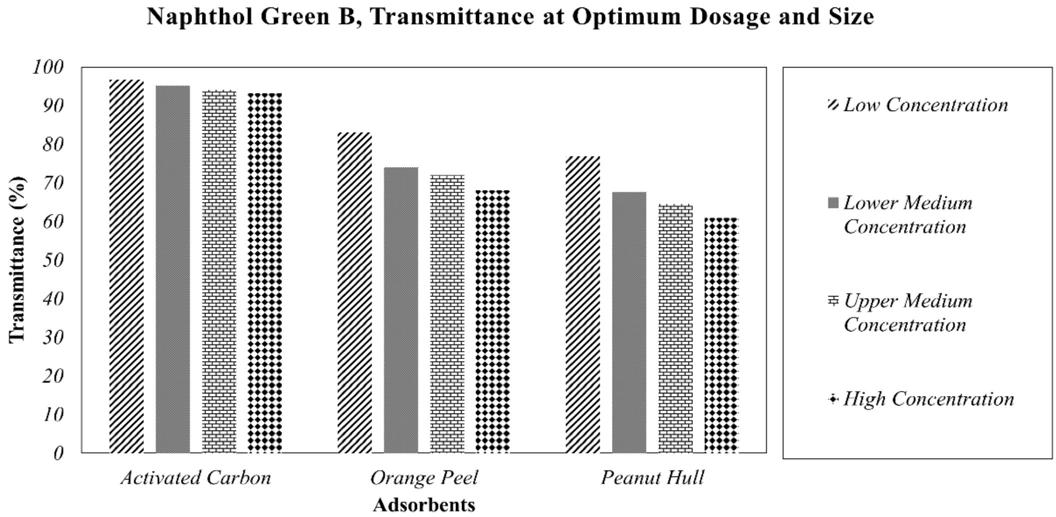


Figure 2 Transmittance of Naphthol Green B at optimum adsorbent size and dosage.

Non-Purgeable Organic Carbon (NPOC)

non-purgeable organic carbon is the organic carbon that remains after purging the acidified sample with gas. The NPOC for high concentration Naphthol Green B (200 ppm) with increasing sugar wastewater concentration (mg/L) is shown in **Table 4** as follows:

Table 4 Comparison of NPOC at high concentration Naphthol Green B.

Sugar Wastewater Concentration (ppm)	Dye Concentration	NPOC (mg/L) for Naphthol Green B (200 ppm) using		
		Activated Carbon	Orange Peel	Peanut Hull
100	Low	9.76	10.62	12.47
100	Lower medium	8.62	10.91	12.83
100	Upper medium	8.37	11.28	13.42
100	High	8.57	11.74	13.91
200	Low	15.36	16.51	18.24
200	Lower medium	15.74	16.96	18.79
200	Upper medium	15.43	16.26	19.18
200	High	16.11	17.73	19.92
300	Low	18.29	19.13	23.47
300	Lower medium	17.98	19.43	23.69
300	Upper medium	17.66	20.27	24.84
300	High	16.42	20.76	26.91
400	Low	22.14	24.36	27.88
400	Lower medium	21.63	24.83	28.43
400	Upper medium	20.87	25.12	28.76
400	High	23.47	25.63	29.38
500	Low	27.63	29.26	32.01
500	Lower medium	26.42	29.17	32.78
500	Upper medium	23.21	30.24	33.11
500	High	26.87	30.75	33.54
600	Low	32.17	33.43	36.41
600	Lower medium	31.63	33.78	36.95
600	Upper medium	30.74	34.12	37.08
600	High	29.87	35.06	37.86

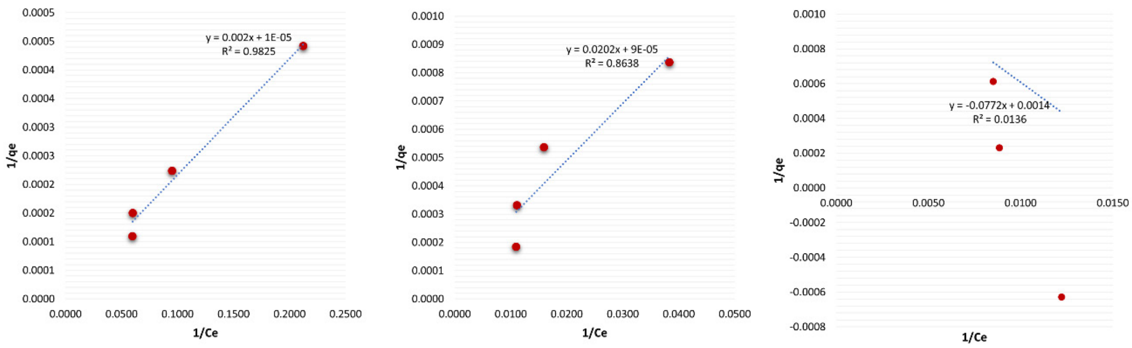
The above table represents the NPOC after treatment with 3 different adsorbents, which is used to describe the relationship between the NPOC and varying sugar wastewater concentrations with 3 different low-cost adsorbents (activated carbon, orange peel, and peanut hull) for a high Naphthol Green B concentration. We can observe that wastewater treated with peanut hull has the high NPOC value of 37.86 mg/L, with orange peel and activated carbon having NPOC values of 35.06 and 32.17 mg/L, respectively. We can also observe that the NPOC values increase with the increase in sugar wastewater concentration from 100 to 600 ppm.

Adsorption isotherms

The equilibrium studies are performed by adding 0.5, 1, 1.5, and 2 g of orange peel powder and 2 g of 5 assorted sizes of peanut hull to 100 mL of combined wastewater with 5 different concentrations of Naphthol Green B and 6 different concentrations of sugar wastewater.

The linearized Langmuir Isotherm model is written as Eq. (8):

$$\frac{1}{qe} = \frac{1}{qmka} * \frac{1}{Ce} + \frac{1}{qm} \tag{8}$$



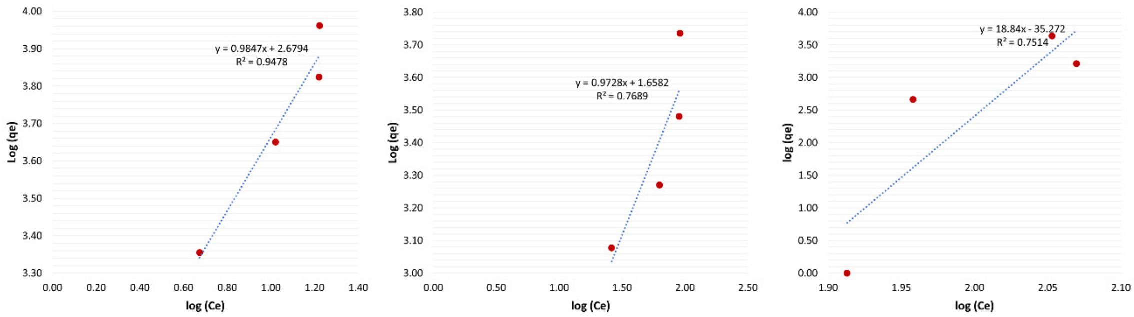
(a) (b) (c)

Figure 3 Langmuir isotherm model of Naphthol Green B adsorption on PAC - (a), on Orange peel - (b), on peanut hull - (c).

The linearized Freundlich Isotherm model is written as Eq. (9):

$$q = k * C^{1/n}. \tag{9}$$

The results of linearized Langmuir Isotherms and linearized Freundlich Isotherms of Naphthol Green B when treated with low-cost adsorbents are presented in **Figure 4**.



(a) (b) (c)

Figure 4 Freundlich isotherm model of Naphthol Green B adsorption on PAC - (a), on orange peel - (b), on peanut hull - (c).

The Langmuir isotherm model has the best fit for Naphthol Green B at the optimum dosage of low-cost adsorbents with the linear equation: y = 0.002x + 0.00001 and coefficient of determination R^2 = 0.9825.

The study shows the transmittance after treatment with 3 different adsorbents, which is used to describe the relationship between the transmittance and varying dye (Naphthol Green B) concentration with 3 different low-cost adsorbents (activated carbon, orange peel, peanut hull) for the various wastewater samples. The results indicate that the transmittance decreases from 96.72 to 93.17 % with the increase in dye concentration from low to high when treated with activated carbon. Similarly, it is observed that the transmittance decreases from 83.12 to 67.98 and 76.98 to 60.94 % with the increase in dye concentration from low to high when treated with orange peel and peanut hull, respectively. From the change in concentration of sugar wastewater, there is a significant change is observed in the NPOC values. This shows that the pollutants from sugar wastewater are directly proportional to the NPOC values. The conclusions drawn from the study are discussed in the next section.

Conclusions

Based on the experimental results, it is concluded that orange peel can treat Naphthol Green B, a basic dye in a sugar solution to achieve maximum transmittance (%) up to 83 % and, similarly, peanut hull up to 76 %. Peanut hull has the highest transmittance of 76.98 % with < 425 µm size. Orange peel has the highest transmittance of 83.12 % with a 2 g dosage. The values of transmittance after treatment with PAC are taken as the datum for the comparison of values after the treatment with orange peel and peanut hull. Peanut hull has the highest NPOC value of 37.86 mg/L when mixed with 600 ppm of sugar wastewater. Orange peel has an NPOC value of 35.06 mg/L when mixed with 600 ppm of sugar wastewater. These 2 are suggestable low-cost adsorbents to use with Naphthol Green B for color removal. However, the adjustment of pH is neglected since the considered samples are already in a medium-range (6 - 9) pH. The NPOC values are high due to the impact of sugar solution. The Langmuir and Freundlich isotherm models represent the equilibrium adsorption of Naphthol Green B on orange peel and peanut hull. The Langmuir isotherm model has the best fit for Naphthol Green B at the optimum dosage of low-cost adsorbents. These treated samples use low-cost adsorbents and can be discharged into surface water through municipal sewage. This research provides insight into dealing with complex wastes. The recommendations for future research include: (i) Adsorption must be studied with different adsorbents and more binary combinations of wastewater effluents from point and non-point sources. (ii) Conducting experiments with different dyes will help to identify the range of treatment capacity of low-cost adsorbents. (iii) Recommended research on micro-filters with various pore sizes can give the optimum filtration capacity for different dyes.

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