

Coagulation-Flocculation Treatment for Naphthol Green Band Flour Wastewater

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To Cite this Article

Bukola M. ADESANMI, Yung-Tse HUNG and Howard H. PAUL, "Coagulation-Flocculation Treatment for Naphthol Green Band Flour Wastewater", *International Journal for Modern Trends in Science and Technology*, 6(12): 190-197, 2020.

Article Info

Received on 10-November-2020, Revised on 30-November-2020, Accepted on 04-December-2020, Published on 08-December-2020.

ABSTRACT

The interference of synthetic dye in the water bodies and environment poses a risk to both human and environmental health. Due to the recalcitrant nature of dye and presence of many other pollutants in industrial wastewater, efficient method of treatment of industrial effluent is required to address the lingering problem over the years. To address this major concern, experimental was carried out on synthetic dye and flour wastewater treatment by coagulation-flocculation while varying operating parameters (dosage, concentration, coagulant type etc.). The effectiveness of coagulation-flocculation process for the removal of Naphthol Green B in a mixture of dye wastewater and flour wastewater at different concentrations (50 ppm, 100 ppm, 150 ppm, 200 ppm) was investigated. Using 3 coagulant ($FeCl_3$, $FeSO_4$ and $Al_2(SO_4)_3$), color removal efficiency was also investigated. The effectiveness of the coagulation process was measured for transmittance and absorbance as indices using UV-Vis Spectrophotometer. Also, the total organic carbon (TOC) was measured. Transmittance and absorbance values of 99.6% and 0.001 respectively were achieved post treatment. Ferric Chloride and Aluminum Sulfate gave better results than Ferrous Sulfate which gave the poorest transmittance and absorbance values indicating reduced color removal efficiency. The results of this study revealed that coagulation process is an efficient preliminary treatment for appreciable suspended particles and color removal from dye wastewater. It also showed the impact of coagulant dosage, dye strength and combined wastewater samples on the removal efficiency and resulting effluent quality.

KEYWORDS: Dye, coagulation, flocculation, color removal, wastewater, transmittance, absorbance

INTRODUCTION

Industrialization is one of the essential factors for proper economic development, but the good management and disposal of industrial effluent is important for solid environmental health. Various industries including the food, plastics, paper, pharmaceutical, cosmetics, and textile use dye to color their product. Based on the wide use of dye, it is a content of effluent produced during various industrial processes. Textile production and yarn processing sector contribute the largest amount of hazardous chemical wastewater, accounting for

nearly 22% of the total volume of industrial effluent produced (Tang & Zaini, 2020). In the early days, natural dyes were used but with the advent of civilization, demand for large scale use of dye increased. The increase in demand brought about the introduction of synthetic dyes to balance demand and supply. There are more than 100,000 type of commercially synthetic dye produced annually to the tune of 7×10^5 metric tons (Abhiram et al, 2020). About 10-15% of this synthetic dye is lost within various processes of textile production" (Mohamed & Ahmed, 2017). Without the proper

investigation of the negative effect of the synthetic dye to the environment, rather focusing on the huge profit for industries, more companies produced and used the synthetic dye. Over the last decades, the use of synthetic dyes in various industrial processes has gained huge acceptance. This response has brought about an exponential increase in the discharge industrial effluents containing dye into the soil and water bodies (Mohamed & Ahmed, 2017). As a result, the environment has been polluted with dyes which are not easily degradable. The recalcitrant and carcinogenic nature of modern synthetic dyes has made the environmental regulations for their disposal stricter and more restrictive.

Wastewater is water discharged from the community, commercial properties, industry, agricultural processes after it has been used in a variety of applications and that contained contaminants that make it unsuitable for most uses without treatment. Industries particularly textile, food, paper, and pulp manufacturing are known to consume large volumes of water during their daily operations. They therefore contribute a pro-rata amount of wastewater as effluent that is characteristic of their raw material properties. Disposal of untreated wastewater leading to color pollution in the water bodies is a major problem. Although considerable research has been done to modify the dyeing process to achieve a better fixation of the dyestuffs onto the substrate (Sathiyamoorthy et al, 2012). Too often, carcinogenic compound produced by anaerobic degradation of dye end up in the food chain (Chen et al, 2018). Furthermore, highly colored wastewaters potentially block sunlight and oxygen from penetrating into the ocean. These two are essential for aquatic lives. Also, because of increasing environmental awareness and stricter regulations various treatment technologies for dye wastewater has been developed (Chen et al, 2018). Over the years, dyes from wastewater have been treated by physical, chemical, and biological methods, such as coagulation, adsorption, membrane filtration, chemical precipitation, advanced oxidation with light or electricity, and biological degradation with microorganisms [5]. Biochemical oxygen demand (BOD) and suspended solids (TSS) are effectively removed using these processes but are not fantastic alternatives for color removal from wastewater [Aziz et al, 2007 and BasavaRao&MaohanRao, 2006]. The two major methods for decolorization of wastewater are destruction of dye molecules (e.g., chemical

oxidation and bio-oxidation) and separation of dyes from water (e.g., coagulation/flocculation, sand filtration and membrane separation) (Zahrim&Hilal, 2013). Although the separation method such as adsorption is effective and widely accepted, the destruction methods that involve the dye change through chemical oxidation, photo-catalysis and biodegradation require large amount energy to breakdown light stable, oxidizable and microbial degradable dye molecules (Minke&Rott, 1999 and Booth et al, 2000). "To treat industrial wastewater containing dye, various biological, physical, and chemical process methods have been used but most of this treatment methods are usually very expensive and environmentally unsafe" (Nigam, et al, 2000 and, Rauf, et al, 2007). A good and popular example involves treatment using powdered activated carbon and activated bentonites (Pala, 2002 and Yavuz&Aydin, 2002). Even so, the use has been restricted with some dyes because of the huge quantity of sludge produced and the low efficiency (Pearce et al, 2003). A rapid and effective method of colour treatment involves using ozone, but some of the other methods employed do not give satisfactory results especially for certain dispersed dyes (Aksu, 2005). "Physical-chemical flocculation with metal hydroxides aided by polymer flocculants is another treatment method popularly used for colored effluents" (Bako, 2014). A method more practically accepted is the use of premixed polyelectrolyte complexes brought about by aqueous solutions of polycation and polyanions reacting together (Petzold. Et al, 1998) Through hydrophobic and electrostatic interaction forces, disperse dyes can effectively be bonded over large distances due to their size and structure by such complex particles (Buchhammer, et al, 2001) However, it is essential to apply other flocculation principles due to the incomparably small size of dye molecules and their aggregates in relation with such inorganic particles, which are sometimes uncharged. Many of the dye wastewater methods of treatment are not cheap and have low purification efficiency.

Coagulation treatment has been effective in removing color especially for wastewaters, containing dissolvable solids. As far back as the 19th century, coagulation has been known for effluent treatment through the utilization of a combination of lime with calcium chloride or magnesium (Muruganandam et al, 2007). In the sequence of treatment, coagulation and flocculation process is succeeded by sedimentation, filtration, and disinfection, in the

primary stage and followed by chlorination. This water treatment method is universally employed by industries before water distribution to the final consumers. In normal water treatment processes, various types of coagulants are been used for making the water suitable for consumer consumption (Matilainen et al, 2005)]. These coagulants can be classified as biological coagulants, inorganic coagulants, and synthetic polymers (binti&Nithyanandam 2013, Renault et al 2009). Coagulation is a process simple to operate, effective for wastewater treatment and involves low capital cost (Liang et al, 2014).

Based on this background, this study is carried out to evaluate the extent of color removal from textile and flour wastewater by coagulation-flocculation treatment. This article reports the methods, conditions, and result of the experiment.

Coagulation and flocculation

Coagulation treatment is a method involving the use of certain chemicals called coagulants, traditionally alum (aluminum sulfate $Al_2(SO_4)_3$), ferric chloride ($FeCl_3$), or ferrous sulfate ($FeSO_4$) to neutralize charges and form gelatinous large mass of particles which when settled can be trapped by filtration (Tzoupanos&Zouboulis., 2008) It is the application of chemical substance called coagulant to enable the agglomeration and settling of suspended and colloidal particles in wastewater. Due to the relatively low density of suspended and colloidal particles, they do not settle under gravity (Tzoupanos&Zouboulis., 2008). Also, because they have a negative charge repelling each other, cannot they be removed by physical processes such as sedimentation.

Theoretically, particles in water are negatively (anionic) and are neutralized by the positive charges (cations) making the sample particles stick together to form larger particles of heavier density (flocs) by slow agitation which can easily be removed by filtration. Trivalent cations are more effective as coagulants than their divalent peer, meaning that the higher the number of cations present in the coagulant, the stronger it is. It is not uncommon to combine organic coagulant such as poly-DADMAC with other inorganic coagulants (alum, Fe_2SO_4 , $FeCl_3$, etc.) as a single coagulant in the treatment process for effective result. Both inorganic and organic coagulants have their advantage and disadvantage. Inorganic coagulants are limited in application because they are pH sensitive while Organic coagulants have complex structure which aid the coagulation

process and their sensitivity to pH insensitive is reduced. Flocculation is the method of stirring and agitation of treated sample to facilitate agglomeration and formation of particle into large masses filtered from solutions. This is the process in which the smaller particles merge to form bigger ones. The extent and rate of particles aggregation and breakup is dependent on applied velocity gradient and time of flocculation.

Dye wastewater quality parameters

Dye and Flour wastewater quality is described in spectrophotometry using two unique parameters namely, Transmittance and absorbance. Although a spectrophotometer can display both transmittance and absorbance of a sample, transmittance and absorbance values from spectrophotometry measurement are related by the equation (Buchhammer et al,2001):

$$A = \log_{10}(1/T)$$

To measure coloration in relation to amount of absorbed light at different wavelength, the Beer-Lambert Law applies:

$$A = \epsilon_{\lambda_{max}} LC$$

A - absorbance

T- transmittance

C – concentration (mg/l)

$\epsilon_{\lambda_{max}}$ - molar extinction of coefficient of the colored solution at maximum wavelegth

L- distance traveled by the light beam through the solution (cm)

Analysis of Total Organic Carbon

Total organic carbon measures the quantity of organic compounds present in water. Some of this organic contaminant in water do not affect the characteristic color of the water hence cannot be detected visually unlike dye. To detect and measure the quantity of this organic contaminant, TOC -L SHIMADZU was used by injection of samples through thermal catalytic oxidation at 680°C.

Total organic carbon can also be called NPOC (Non-purge-able organic carbon)and refers to organic carbon contained in a sample in the non-volatile state. This is because the sample is acidified and purged with carbon free air or nitrogen which helps to eliminate the inorganic carbon as carbon dioxide, oxidize and measure the remaining non-purge-able carbon. NPOC is calculated as

$$\text{NPOC (TOC)} = \text{TC} - \text{IC}$$

TC – total carbon in the sample

IC - total inorganic carbon

II. MATERIALS AND METHODOLOGY

The combined naphthol green b and flour wastewater was prepared in the laboratory using distilled water for standard solutions. Aluminum Sulfate, Anhydrous Ferric Chloride and Ferrous Sulfate were used as coagulants to investigate the color removal of naphthol green B dye by coagulation treatment. Equipment used to carry out the experiment included spectrophotometer (Carolina #65-3303), weighing balance (Mettler Toledo), open air platform shaker (Innova 2300) and TOC analyzer (Shimadzu 5050).

Naphthol Green B

The C.I. (Color Index) number is 10020 with C.I. name is acid green 1. It comes under the class of nitroso with acidic ionization and is very soluble in aqueous solution. Its molecular formula is $(\text{C}_{10}\text{H}_5\text{NO}_5\text{SNa})_3\text{Fe}$ with formula weight 878.79. This commercial dye is a lake, in which the mordant metal is ferric iron. It may undergo a photo redox reaction in analogy to numerous other Fe^{3+} complexes because it is an iron(III) compound. However, the iron appears to play no part in staining tissues. The unlocked dye is Naphthol green Y, C.I. 10005 i.e. that it is a sodium salt of Naphthol green Y. It attaches to tissues is through the sulphonic group. The dye is therefore an acid dye, but it is rarely used for staining tissues [17].

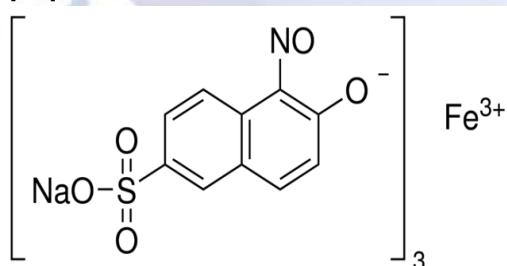


Figure 1 The Chemical Structure of Naphthol Green B (Li et al, 2017)

Flour wastewater

There are different type of flour including wheat flour, cassava flour, sweet potato flour and potato flour. Flours are important raw materials for different manufacturing and food processing industries. Flour fall under the starch class of food and starch wastewater generated from the

industries such as food, cosmetic, pharmaceutical, medicine, chemical, papermaking, textile, detergents are difficult to treat effectively (Fatah&Hawash, 2018). This is because they have high COD and starch is generally difficult to decompose by microorganism. For this experiment, wheat flour wastewater was prepared from flour bought from the grocery store.

Method

A weighing balance with the precision of ± 0.01 g is used for weighing the materials: dyes and flour waste. From the weighed materials, dye, and flour wastewater stock solution of 1L is prepared and stirred thoroughly with a glass stirrer. From the two mother samples of dye and flour wastewater, desired quantity of combined dye and flour wastewater is prepared. The combined dye and flour wastewater are treated with the coagulants and then the samples bottle is placed in a mechanical shaker and mixed vigorously for 1 minute at 100rev/min (fast shake) followed by 30 minutes of slow shaking at 30rev/min. The solution kept still to allow settle for 60min and so that the coalescence could precipitate. This the sedimentation phase.

The spectrometer warmed up for 30min upon turning on and then the wavelength set. The spectrometer was then calibrated by filling a sample vial with distilled water. The distilled water sample is used as reference because it has 0 absorbance and 100% transmittance (Madiraju et al, 2018). This followed by placing the treated sample in the spectrometer to be analyzed, transmittance and absorbance readings were taken, and supernatant was measured for TOC removal. The same procedure is repeated for different dye concentrations. Run protocol of combined Naphthol Green B and flour wastewater was developed based on literature as shown in Table 1.

Table 1. Run protocol of combined Naphthol Green B and flour wastewater treated with coagulants

Run Orde r	Dye Concentration(mg/l)	Flour Concentration(mg/l)	Coagulan t Dosage (mg/l)
1	50	0	0
2	50	100	0
3	50	200	0
4	50	300	0
5	50	400	0
6	50	0	30

7	50	100	30
8	50	200	30
9	50	300	30
10	50	400	30
11	50	0	60
12	50	100	60
13	50	200	60
14	50	300	60
15	50	400	60
16	50	0	90
17	50	100	90
18	50	200	90
19	50	300	90
20	50	400	90
21	50	0	120
22	50	100	120
23	50	200	120
24	50	300	120
25	50	400	120
26	50	0	150
27	50	100	150
28	50	200	150
29	50	300	150
30	50	400	150

III. RESULTS AND DISCUSSION

The present investigation was performed using conventional coagulants to evaluate their suitability for treatment of dye and flour effluent. To represent the performance of aluminum sulfate, anhydrous ferric chloride and ferrous sulfate coagulants on different dye and flour concentration, the results have been tabulated for effective comparison. Coagulant dosage of 30,60, 90,120 and 150 mg/l were introduced to dye wastewater of concentration 50, 100, 120 and 200 mg/l singly and in the presence of flour wastewater of concentration 100, 200,300 and 400 mg/l.

Influence of dye and flour concentration

The decolorization efficiency and appreciable turbidity removal for dye and flour wastewater is influenced by dye and flour concentration within the sample. The highest transmittance and lowest absorbance value were observed at low dye and low flour concentration between 50 ppm -100 ppm. Meanwhile, the lowest transmittance and highest absorbance value were seen at high dye and flour concentration between 150 ppm -200 ppm. Hence the overall trend observed was that the increase in dye and flour concentration decreases the transmittance and increases the absorbance translating to reduction in the effectiveness of the coagulation-flocculation treatment.

Table 2. Dye Concentration Values for Naphthol Green B at 40mg/L Ferric Chloride

Dye Concentration(mg/L)	Absorbance	Transmittance (%)
0	0	100
50	0.164	68.0
100	0.287	51.5
150	0.393	40.5
200	1.141	7.2

Influence of coagulant dosage on dye and flour wastewater

Coagulant dosage was one of the most important parameters considered in determining the optimum conditions for dye and flour removal from the sample wastewater. Using five different dosage (30,60, 90,120, 150 mg/l) of the three coagulant, the study showed that increase in coagulant dosage resulted in increase of charge neutralization, settling, and fading of color in the sample solution. This was observable in the corresponding increase in transmittance values and decrease in absorbance values. An increase in coagulant dose improves color and turbidity removal and TOC removal between 18%-78% regardless of coagulant. This is illustrated in Table 3 and Figure 2.

Table 3. Influence of Ferric Chloride Dosage on 50mg/L Dye and 100mg/L Flour concentration

Dye Concentration(mg/L)	Flour Concentration(mg/L)	Coagulant Dose (mg/L)	Transmittance (%)
50	100	0	69.8
50	100	30	81.1
50	100	60	87.3
50	100	90	92.2
50	100	120	97.2
50	100	150	99.3

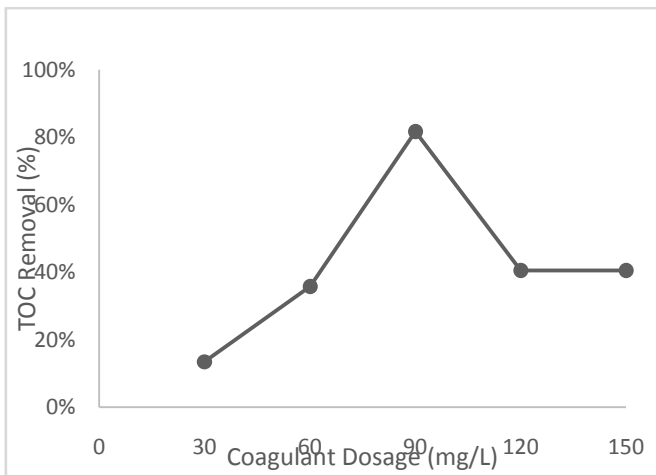


Figure 2 Influence of coagulant dosage on TOC removal for combined dye and flour wastewater at 100 ppm

Influence of coagulant type and valence

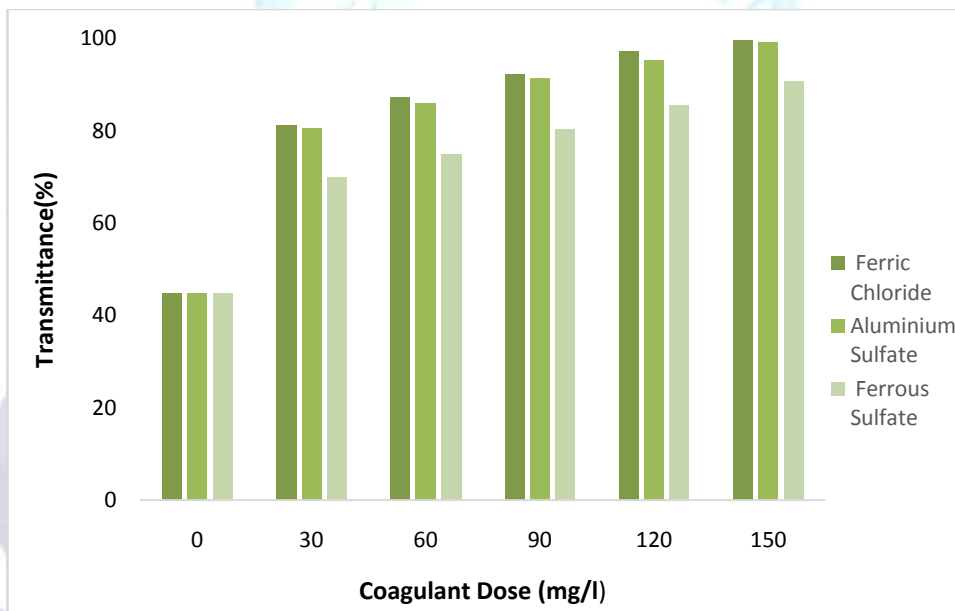


Figure 3. Naphthol Green B, 50 ppm and 100 ppm Flour Treated with Different Coagulants

Influence of dye strength on combined wastewater

Applying aluminum sulfate at low dye concentration (50 mg/l) and low flour concentration (100 mg/l) which represent the low strength sample, the best values for absorbance and transmittance of 0.010 and 95.3% was observed, respectively. This indicated that the coagulants were able to effect charge neutralization and subsequent settling relatively easily because of the combined low dye strength. The dilution effect of the low strength flour wastewater also contributed to the increased transmittance and reduced absorbance values accordingly.

From the results, it was observed that the type of coagulant has an impact on color and turbidity removal. Although all three coagulants provided a high color removal efficiency, ferric chloride was the most effective coagulant in decolorizing Naphthol Green B dye and achieving appreciable turbidity removal. The best color and turbidity removal were observed with ferric chloride at a dosage of 150 mg/l with transmittance of 99.6%. Also, a good color removal with Aluminum sulphate coagulant with transmittance value of 99.2% at a dosage of 150 mg/l was observed. Ferrous sulphate had the least impact on sample wastewater with a transmittance value of 90.7%. The resulting transmittance value and effectiveness of the coagulants is connected to reduced potency of +2 valence, exhibited by Ferrous Sulfate as compared to +3 exhibited by Ferric Chloride and Aluminum Sulfate.

The medium dye concentration (100 mg/l) and medium flour concentration (200 mg/l) which represent the medium strength sample gave comparatively lower transmittance and corresponding higher absorbance values than the low strength sample. Transmittance and absorbance values were seen to be 90.1% and 0.048, respectively. The least values of 78.9% and 0.067 were observed for high strength sample (high dye concentration-high flour concentration) as summarized in **Table 4**.

Table 4. Effect of Aluminum sulfate dosage on 100mg/L Naphthol Green B

Dye concentration (mg/L)	Flour wastewater Concentration (mg/L)	Al ₂ (SO ₄) ₃ Dosage (mg/L)	Transmittance (%)	Strength
50	0	120	92.6	Low
100	0	120	88.1	Medium
150	0	120	74.8	High
50	100	120	95.3	Low
100	200	120	90.1	Medium
150	100	120	78.9	High

IV. CONCLUSION

Synthetic dye and flour wastewater were successfully prepared using distilled water for dilution and treated by coagulation/flocculation process. The study has shown that dye and flour concentration, coagulant type and dosage and strength of combined wastewater contributed to the dye and turbidity removal from effluent sample. It was observed that increase in dye concentration causes a decrease in color removal efficiency. This is because the 50mg/l dye wastewater sample had higher transmittance values compared to 200mg/l dye wastewater sample. The flour wastewater sample also exhibited the same trend. Also, an increase in coagulant dosage lead to increase dye and turbidity removal across all the dye and flour concentration. It was also observed that Coagulants with a higher valence of +3 (FeCl₃ and Al₂(SO₄)₃) had more potent effect on wastewater sample than those with lower valence of +2 (FeSO₄). Moreover, adilution effect was observed during treatment of combined dye and flour wastewater depending on the sample concentration. Finally from the transmittance value obtained at optimum condition (50ppm dye concentration, 150 mg/l FeCl₃ dose), it can be concluded that the coagulation/flocculation treatment acts as a proper primary treatment process for suspended particles, organics, and color removal. Treated naphthol Green B and flour wastewater showed excellent transmittance and absorbance values and can be reused for processing or production in the textile and food industry. The coagulation-flocculation process can be used as a preliminary treatment while completing the treatment process with more sophisticated

technologies like reverse osmosis (RO) to reduce energy and operational cost.

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