Treatment of Combined Acid Black 48 and Coffee Wastewater by Low-Cost Adsorbents

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Removal of synthetic dyes from wastewater is essential both from the environmental and human health point of view. A small concentration of synthetic dyes can reduce water transparency and consequently influence photosynthesis and alter aquatic ecosystems. Acid black 48 is an Azo dye that falls under the category of synthetic dyes used in the textile industry. With dyes, coffee wastewater has high chemical oxygen demand (COD) that can affect dissolved oxygen (DO) in surface waters. A mixture of wastes in surface waters creates a need to investigate the efficiency of existing treatment methods and optimize them. Adsorption using activated carbon is a conventional method used to remove dyes and heavy metals from wastewater. Industries prefer efficient and economical treatment methods to meet challenging effluent standards regarding COD, BOD, and intensity of color. The adsorption process was optimized using low-cost adsorbents in the current study, including peanut hull and onion peel, to treat a binary mixture of acid black 48 and coffee wastewater. After adsorption, microfiltration was used to remove any suspended solids from the wastewater solution. The performance of combined treatment processes for the color removal of the binary mixture was analyzed and compared using transmittance and absorbance. Treatment efficiency of adsorption using low-cost adsorbents was compared with powdered activated carbon. Apart from absorbance and transmittance, non-purgeable organic carbon (NPOC) values were analyzed to determine organic carbon removal in the combined binary wastewater. Experimental results indicated that Langmuir isotherm was the best fit for a binary mixture with an optimum dosage of 1.2 g using onion peel. The regression coefficient value was 0.82, and the uptake was 58.13 mg of binary mixture per 1 g of onion peel. The effective pH for maximum uptake of acid black 48 using onion peel for adsorption
was 5.7. The increasing dosage of low-cost adsorbents adsorption improved in removing binary waste of dyes and coffee waste from wastewater. Adsorption using onion peel improved adsorbent performance up to 1.2 g dosage and steadily decreased beyond that. The adsorption capacity of onion peel was comparatively higher than the peanut hull based on the linear fit.

**Keywords:** dye wastewater, coffee wastewater, adsorption, microfiltration, acid black 48.

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**Introduction**

Dyes are used in commercial, pharmaceutical, textile, cosmetic, and food industries. Synthetic production of dyes has become very significant. Over 100,000 varieties are available commercially with an annual production of $7 \times 10^5$ metric tons (Fu and Viraraghavan, 2001; Robinson et al., 2001). Synthetic dyes use an enormous amount of surface water for processing and pre-treatment. Textile industries primarily use a large amount of water for washing, dyeing, and leaching purposes producing wastewater (Bharti et al., 2013; Imtiazuddin et al., 2012; Sultana et al., 2013). Discharge of dye wastewater from industries can pollute surface water streams. If the dye wastewater is not treated properly before releasing to the environment, the effluent from these industries can cause detrimental effect on quality of receiving waters in terms of pH, total suspended solids (TSS), total dissolved solids (TDS) and transmittance (Ahmed et al., 2012; Dey and Islam, 2015; Islam and Guha, 2013; Yaseen and Scholz, 2016).

Removing dye contaminants in wastewater using conventional wastewater treatment methods, including activated sludge and trickling filters, is not efficient. Although conventional wastewater treatment methods can be used as a pre-treatment step to remove significant organic content, they can not remove synthetic dyes from wastewater (Kornaros and Lyberatos, 2006) continuously and as a sequencing batch reactor (SBR). As untreated wastewater contains high concentration of TSS and TD, there is a high chance of environmental risk if untreated dye wastewater is released to surface water. As far as human health is concerned, reactive dyes create respiratory problems because of inhalation (Hassaan and Nemr, 2017). Inhaling dyes by humans is a potential risk in terms of health while showering. An increase in risk to both humans and the environment creates the need for treatment and makes the treatment process a priority to remove dyes from wastewater.

Some conventional wastewater treatment processes to treat dye wastewater are adsorption, coagulation, and electrochemical oxidation (Ge et al., 2012; Li et al., 2017; Lin and Peng, 1996; S. V. H. Madiraju et al., 2019; Ren et al., 2013). Adsorption is a preferred treatment method because of treatment efficiency, and simplicity. Dye manufacturing companies use the adsorption process to remove the color from wastewater. One of the significant variables that affect the dye wastewater treatment process is the adsorbent particle size (Ong et al., 2010). The adsorption process efficiency is closely related to the surface area and number of sorption sites in the adsorbent, which makes activated carbon an ideal adsorbent (Johnson et al., 2002; S. V. H. Madiraju et al., 2020). Adsorption as a treatment process is effective compared to all the other treatment processes. Still, the major disadvantage is the high investment involved in using virgin carbon and recycling cost in spent carbon usage (Alhashimi and Aktas, 2017) and its applications diversifying from its early uses as soil amendment, it is important to study the environmental impacts and economic performance of biochar in comparison to activated carbon in order to assess its value. The goal of the study was to assess, through a meta-analysis, the environmental and economic performance of biochar in comparison to activated carbon under an equivalent functional unit to adsorb heavy metals. More than 80 data points on adsorption capacity of biochar and activated carbon were identified through literature, which were statistically analyzed as part of the study. Biochar was found to have lower energy demand and global warming potential
impact than activated carbon, where average energy demands were calculated as 6.1MJ/kg and 97MJ/kg and average greenhouse gas emissions calculated as −0.9kg CO₂eq/kg and 6.6kg CO₂eq/kg for biochar and activated carbon, respectively. When adsorption of heavy metals were used as the functional unit during analysis, results indicate that there is typically an order of magnitude difference between the two materials, where biochar was found to have lower environmental impacts. The environmental impact resulting from long distance transportation of biochar would not overturn this conclusion. The adsorption cost of biochar was lower than activated carbon to remove chromium and zinc with a 95% confidence. Adsorption cost for lead and copper were found to be comparable, and therefore the specific type of biochar and its price could shift results both ways. There is evidence that biochar, if engineered correctly for the task, could be at least as effective as activated carbon and at a lower cost. Low-cost adsorbents from agricultural wastes were studied to reduce the cost involved in the sorption process to make the treatment process economical. Most of these low-cost adsorbents are readily available and are economical to use and dispose of (Weng and Pan, 2007) a waste produced from an edible oil manufacturer was investigated. Results showed that the adsorption increased with increasing MB concentration, temperature, and pH. The adsorption equilibrium data was well fitted by multilayer adsorption isotherm. The maximum adsorption capacities for MB ranged from 0.94×10⁻⁴ to 3.41×10⁻⁴mol/g between 5 and 45°C. Thermodynamic parameters suggest that the adsorption is spontaneous and endothermic. We proposed a modified double exponential equation accounting both with chemical and mathematical point of view to describe the adsorption kinetic data. The increases of mass transfer and adsorption capacity were mainly attributed to the interlayer of the SAC expanding at higher temperature. An activation energy of 13.5kcal/Kmol was determined suggesting that the adsorption involved a chemical reaction mechanism. The adsorption of a cationic dye (methylene blue. There are sufficient sorption sites in agricultural wastes that can reduce the expenses involved in using powdered activated carbon (Adegoke and Bello, 2015)these processes are effective and economic only in the case where the solute concentrations are relatively high. Most industries use dyes and pigments to color their products. The presence of dyes in effluents is a major concern due to its adverse effect on various forms of life. The discharge of dyes in the environment is a matter of concern for both toxicological and esthetical reasons. It is evident from a literature survey of about 283 recently published papers that low-cost adsorbents have demonstrated outstanding removal capabilities for dye removal and the optimal equilibrium time of various dyes with different charcoal adsorbents from agricultural residues is between 4 and 5h. Maximum adsorptions of acidic dyes were obtained from the solutions with pH 8–10. The challenges and future prospects are discussed to provide a better framework for a safer and cleaner environment.
Despite being efficient and economical, low-cost adsorbents have problems with disposal, similar to conventional adsorbents. Landfills might not be a feasible option if there are heavy metals and other toxic compounds that are adsorbed in the adsorption process (Bekbölet et al., 1996; Shon et al., 2005; Torretta et al., 2017). Cementitious materials, which include fly ash, provide potential physical encapsulation and chemical stability to toxic waste in landfills (Kosson et al., 2002; Okoronkwo et al., 2018). Using recycled products from the cement industry is a feasible option for the safe disposal of dye adsorbed materials (Huang et al., 2017; Limbachiya et al., 2012).

In this study, low-cost adsorbents were used to remove acid black 48 combined with coffee wastewater. Some low-cost adsorbents involved in the treatment process were peanut hull and onion peel. Peanut is an important food crop and edible oil source in the world. Major countries, including China, India, and the USA, produce and export peanuts (Z. Chen et al., 2017; Fletcher and Shi, 2016). Similarly, onions are primarily consumed in China and India (C. Chen et al., 2008; Gummagolmath, 2012). The abundance of peanuts and onions in the world market makes them potential low-cost adsorbents that can remove contaminants from wastewater. In this study, both peanut hull and onion peel were used to remove the binary mixture of acid black 48 with coffee waste in aqueous solution. Batch adsorption sampling was used to analyze the initial and final concentrations in wastewater (Din et al., 2009; Hameed and Ahmad, 2009; Luengo et al., 2006; S. V. H. Madiraju et al., 2018a). Equilibrium studies were performed to find the final concentration of combined dye and coffee wastewater using Beer–Lambert’s law (Andorn and Bar-Eli, 1971). The dynamic equilibrium of combined acid black 48 and coffee wastewater was studied using adsorption isotherms. Furthermore, the amount of organic carbon removed from the wastewater was assessed using NPOC (Augugliaro et al., 2004; Kirkels et al., 2014).
A spectrophotometer was operated within a wavelength of units in nm. Absorbance and transmittance of the wastewater samples were obtained from the Carolina UV-VIS spectrophotometer with an absorbance accuracy check of ±2% at 1 Å. It accepts test tubes or square cuvette with size 10 mm. The wavelength range of the spectrophotometer was 335–1000 nm, with a spectral bandpass of 20 nm. Wavelength accuracy and wavelength repeatability is ±2 nm and ±1 nm, respectively.

**Platform shaker (Innova 2300)**

Vibration to the platform was provided by a triple-ec- centric counterbalance drive inserted in a cast iron housing. Shaking range is between 25–500 rpm with 2.5 cm orbit. Shaking speed can be increased or decreased by increments or decrements of 1 rpm. Platform size was 76 x 46 cm and had a timer from 0.1 to 0.99 h with both audio and visual alarm systems.

**TOC analyzer (Shimadzu TOC-L)**

TOC-L analyzer used can combust the wastewater sample at 680°C by catalytic combustion. It can analyze samples with a detection limit of 4 µg/L. This high sensitivity of detection enables this equipment to analyze both low-molecular-weight and hard-to-de- compose macromolecular organic compounds. It can measure total carbon (TC), inorganic carbon (IC), total organic carbon (TOC), non-purgeable organic carbon (NPOC), purgeable organic carbon (POC), and total nitrogen (TN).

**Methods**

**Binary dye and coffee wastewater**

Stock samples were prepared by weighing 1 g acid black 48 on a weighing scale (OHAUS PA1502) with ±0.01 g precision. A stock solution of dye wastewater was prepared by adding 1 g of dye to the 1000 mL of water. After preparation, it was stirred with a glass stirrer such that the dye mixes thoroughly. All the stock solution samples were covered with aluminum foil to prevent oxidation. Later, the required dye wastewater concentrations were prepared by dilution. Similarly, a coffee wastewater stock solution was prepared by weighing and mixing 1 g of ground coffee powder to 1000 mL of water. Later, the diluted
coffee wastewater was mixed with dye wastewater to create a binary mixture of combined dye and coffee wastewater.

**Batch experiments and techniques**

Adsorption investigations were carried out with 50 mL volume and 50, 100, and 200 mg/L concentration of dye solution in centrifuge bottles. Run protocol for dye, coffee, and adsorbent concentration was developed based on trial and error, as shown in Table 1. As different concentrations of dye wastewater were prepared at 50, 100 and 200 mg/L, they were designated as low, medium, and high concentrations for ease of reference (S. V. H. Madiraju et al., 2018).

| Run order | Dye concentration (Medium) (mg/L) | Adsorbent concentration (g) | Coffee concentration (mg/L) |
|-----------|---------------------------------|----------------------------|---------------------------|
| 21        | 100                             | 1.2                        | 150                       |
| 22        | 100                             | 1.2                        | 200                       |
| 23        | 100                             | 1.2                        | 250                       |
| 24        | 100                             | 1.2                        | 300                       |
| 25        | 100                             | 1.6                        | 0                         |
| 26        | 100                             | 1.6                        | 100                       |
| 27        | 100                             | 1.6                        | 150                       |
| 28        | 100                             | 1.6                        | 200                       |
| 29        | 100                             | 1.6                        | 250                       |
| 30        | 100                             | 1.6                        | 300                       |
| 31        | 100                             | 2                          | 0                         |
| 32        | 100                             | 2                          | 100                       |
| 33        | 100                             | 2                          | 150                       |
| 34        | 100                             | 2                          | 200                       |
| 35        | 100                             | 2                          | 250                       |
| 36        | 100                             | 2                          | 300                       |

From the coffee stock solution, batch samples were prepared using dilution including 100, 150, 200, 250, and 300 mg/L mixed with the 50 mL dye solution at 100 rpm (rapid shake) for 1 minute (slow shake) and 30 rpm for 30 minutes at room temperature (25 ± 2°C). Controls without adsorbent were prepared simultaneously to understand that results were obtained only from the adsorption process but not due to the walls of the centrifuge bottles. The percentage uptake of combined dye and coffee wastewater is:

\[
\text{Percentage Uptake (\%)} = \frac{C_o - C_t}{C_o} \times 100
\]

where \(C_o\) is the initial concentration, and \(C_t\) is the concentration at a time \(t\).

The dye concentrations were analyzed using the Carolina UV-VIS spectrophotometer. All the measurements were taken at the wavelength corresponding to the maximum absorption of acid black 48, i.e., \(\lambda_{\text{max}} = 663\ \text{nm}\).
Results and Discussion

The main criteria describing the performance of adsorption and microfiltration are adsorbent particle size, dosage, and non-purgeable organic carbon (NPOC) based on absorbance and transmittance. During batch experiments, peanut hull particle size was defined using sieve analysis with size ranging from 3.327 to 0.425 mm and dosage ranging from 0.2 to 2 g. Adsorbent dosage ranging from 0.4 to 2 g was used for onion peel without sieving. Apart from adsorbent size and dosage, NPOC was used to measure the performance of organic carbon removal in adsorption (using low-cost adsorbents) followed by microfiltration. The efficiency of binary mixture treatment was observed based on transmittance. For treatment performance comparison, transmittance was categorized as before treatment (BT) and after treatment (AT) with fixed dye concentration and varying coffee concentration ranging from 0, 100, 150, 200, 250, and 300 mg/L respectively for a batch as shown in Figs. 2 to 4. Three batches were tested by categorizing dye concentration at low (50 mg/L), medium (100 mg/L), and high (200 mg/L). After comparing transmittance, isotherms were used to select and optimize the use of adsorbents in the treatment process. It was noteworthy to mention that the current treatment approach can be upscaled to the actual industrial conditions at the tertiary treatment stage.

Adsorbent particle size and dosage on adsorption

Transmittance is a parameter used to quantify the treatment of wastewater in terms of color removal. Transmittance before treatment (BT) and after treatment (AT) of a combined binary mixture of dye and coffee wastewater at varying concentrations were compared. This comparison gives a relationship between adsorbent dosage and transmittance. As shown in Figs. 2, 3, and 4, the following bar graphs with different adsorbents, including PAC, peanut hull, and onion peel show that an increase in adsorbent size and dosage improve the transmittance.

As seen in Fig. 2 binary mixture of acid black 48 dye and coffee wastewater were removed with an increase in adsorbent dosage. Being an excellent adsorbent, PAC has the highest transmittance, touching 80 to 100% with dosage levels from 0.4 to 1 g.

Fig. 2. Comparison of transmittance with binary wastewater at 200 mg/L using PAC

![Comparison of transmittance with binary wastewater at 200 mg/L using PAC](image-url)
Fig. 3. Comparison of transmittance with the binary mixture at 200 mg/L using peanut hull

Fig. 4. Comparison of transmittance with the binary mixture at 200 mg/L using onion peel
Although low-cost adsorbents do not have the potential to remove color like PAC, peanut hull at different adsorbent sizes could improve transmittance from 30 to 70% of a binary mixture of dye and coffee wastewater as illustrated in Fig. 3. Therefore, using low-cost adsorbents seems to be a good pre-treatment option, which can reduce the use of PAC in the treatment process. Since different peanut hull sizes do not seem to affect the adsorption of combined dye and coffee waste from aqueous solution, onion peel at different concentrations were used. Based on the results, as shown in Fig. 4, 1.2 g per 50 mL seems to be an optimum dosage for the binary mixture to remove color. Onion peel at optimum dosage of 1.2 g seems to transmit 2.42%, and 3.84% more light than peanut hull at low and medium concentration of acid black 48, indicating better color removal in terms of transmittance. At high concentrations of acid black 48 in binary wastewater solution, peanut hull seems to adsorb better. At high concentrations, 11.21% more light was transmitted by wastewater while using peanut hull as an adsorbent, as shown in Fig. 5. Onion peel seems to be a good low-cost adsorbent when compared with peanut hull.

**Fig. 5. Comparison of transmittance of acid black 48 and coffee wastewater at optimum adsorbent size and dosage**

| Adsorbent                  | Low concentration (50 mg/L) | Medium concentration (100 mg/L) | High concentration (200 mg/L) |
|----------------------------|-----------------------------|---------------------------------|-------------------------------|
| Powdered activated carbon  | 96                          | 97.3                            | 97.2                          |
| Peanut hull                | 78.2                        | 72.8                            | 69.4                          |
| Onion peel                | 80.1                        | 75.6                            | 62.4                          |

NPOC
Non-purgeable organic carbon (NPOC) is the organic carbon left and purges out the purgeable carbon using HCl acid. To better understand the quality of treatment using low-cost adsorbents in terms of organic carbon, the NPOC parameter was used. The current study shows the amount of coffee that was removed from the binary mix of acid black 48 and coffee wastewater using low-cost adsorbents. NPOC for a high concentration of acid black 48 at 200 mg/L with increasing coffee concentration from 100 to 300 mg/L was shown in the comparative plot of Fig. 6. From Fig. 6, it can be observed that activated carbon best removes the organic carbon better than the low-cost adsorbents. While comparing low-cost adsorbents, onion peel was better than peanut hull in removing organic carbon. Therefore, onion peel can remove organic carbon from binary wastewater.
Adsorption isotherm

The Beer-Lambert law was used to find the final concentration of wastewater after treatment. The amount of dye adsorbed at the equilibrium can be calculated as follows:

\[ q_e = \frac{(C_0 - C_e)V}{W} \]  

(2)

where \( C_0 \) is initial concentration (mg/L), \( C_e \) is equilibrium concentration (mg/L), and \( W \) is weight of the adsorbent used (g).

The kinetics involved in the adsorption process of binary wastewater was investigated using two adsorption isotherms, mainly Langmuir and Freundlich isotherms. The equations involved in the isotherms are as follows:

\[ \frac{1}{q_e} = \frac{1}{K_L Q_m} + \frac{1}{C_e Q_m} \]  

(Langmuir isotherm equation)

\[ \log q_e = \log K_f + \frac{1}{n} \log C_e \]  

(Freundlich isotherm equation)

where \( q_e \) is the amount of dye adsorbed at time \( t \) (mg/g), \( K_L \) is the Langmuir adsorption coefficient, \( Q_m \) is the maximum adsorption capacity (mg/g), \( K_f \) is the Freundlich constant for adsorption capacity, and \( n \) is the Freundlich constant for intensity. The coefficients of the linearized form of both Langmuir and Freundlich isotherms were mentioned in Table 2 and Table 3. Only Langmuir isotherm seems to have a reasonable fit to the observed data using low-cost adsorbents, as shown in Table 2.

As we can see that the Freundlich constant was less than 1, which indicated the intercept was negative, which was highly unusual for a linear fit model. So, the Freundlich isotherm cannot fit the observed concentration uptake data of acid black 48 wastewater, combined with coffee wastewater, using low-cost adsorbents. Also, the \( R^2 \) values were not close to 1 compared with Langmuir isotherm linear fit, as shown in Table 2.
Table 2. Adsorption isotherms coefficients and capacity using low-cost adsorbents

| Adsorbent  | Langmuir | Freundlich |
|------------|----------|------------|
|            | $Q_m$ (mg/g) | $K_L$ (L/mg) | $R^2$ | $K_f$ | $n$ | $R^2$ |
| Peanut hull | 46.3     | 0.15       | 0.83 | 0.08 | 0.90 | 0.73 |
| Onion peel | 58.1     | 0.17       | 0.82 | 1.04 | 0.09 | 0.73 |

The maximum uptake for onion peel was 58.1 (mg/g), compared with the peanut hull being 46.3 (mg/g). This shows that onion peel has a better adsorption capacity than peanut hull. Based on plotting and comparing $R^2$ values, the best linear fit of equilibrium data with both Langmuir and Freundlich isotherms, using peanut hull and onion peel, were observed in the linearized plots from Fig. 7 to Fig. 10.

Absorbance and transmittance parameters used in the current study were obtained under controlled conditions at 25°C temperature in the laboratory. Also, wastewater samples were not exposed to atmospheric oxygen because of the possibility of getting oxidized. Further investigation based on sensitivity analysis with variable temperatures may improve the treatment efficiency. The results indicated that Langmuir isotherm was the best fit for peanut hull and onion peel in the adsorption process. Adsorbent uptake of 58.1 mg/g and 46.3 mg/g using onion peel and peanut hull, respectively, can be used to upscale the current treatment approach for full-scale application.

Fig. 7. Langmuir isotherm for acid black 48 adsorbed on peanut hull adsorbent
Fig. 8. **Langmuir isotherm for acid black 48 adsorbed on onion peel adsorbent**

\[ y = 10.268x + 0.0172 \]
\[ R^2 = 0.8239 \]

Fig. 9. **Freundlich isotherm for acid black 48 adsorbed on peanut hull adsorbent**

\[ y = 1.1086x - 1.0656 \]
\[ R^2 = 0.7372 \]
**Conclusions**

Transmittance is an important parameter that can estimate color removal from combined dye and coffee wastewater. Maximum transmittance in the treated sample indicates the highest treatment efficiency in removing color from the combined wastewater sample. Based on this study, it is found that both the adsorbents, including onion peel and peanut hull, have the potential to remove color in combined acid black 48 and coffee wastewater from aqueous solution. Onion peel can treat combined wastewater with a maximum transmittance of 80.1% and 75.6% at low and medium concentrations of combined wastewater. In comparison, peanut could achieve transmittance of 69.4% at high concentrations of combined acid black 48 and coffee wastewater. The optimum pH in the adsorption process for combined acid black 48 wastewater and coffee wastewater solution was found to be around 5.7. The isotherm study indicated that equilibrium sorption data is the best fit for Langmuir isotherm but not for Freundlich isotherm with an $R^2$ value of 0.83 and 0.82 for peanut hull and onion peel, respectively. Modeled equations for the color removal of combined wastewater using peanut hull was $y = 7.306X + 0.0216$ and using onion peel was $y = 10.268X + 0.0172$. The higher slope with onion peel indicated better color removal, using onion peel as a low-cost adsorbent when compared with peanut hull. The maximum adsorption capacities estimated from both peanut hull and onion peel were 46.3 mg/g and 58.1 mg/g, respectively.

**Acknowledgment**

The authors are grateful to the research facilities provided by Cleveland State University, Ohio, USA.
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