



Organic waste removal from pharmaceutical and textile effluents using composite adsorbent

M. Khatun^{1,2}, G. Mohammad², Mir T. Rahman¹ and S. E. Kabir^{1*}

¹*Department of Chemistry, Jahangirnagar University, Savar, Dhaka 1342*

²*Gonoshasthaya Pharmaceuticals, Mirzanagar, Savar, Dhaka 1344*

Abstract

Pharmaceutical and textile industries widely use inorganic and organic components which undergo different chemical and biochemical reactions with the ground water systems. The removal of organic wastes from effluents was carried out using a composite adsorbent. Four different samples were collected from the Gonoshasthaya Antibiotic Limited, Gonoshasthaya Pharmaceuticals Limited, Pakiza Dyeing and Printing Industries Limited and Bangladesh Dyeing and Finishing Industries Limited. Synthetic effluents of four types were prepared maintaining a similar chemical average of the collected samples. All the samples contained a high concentration of TDS, TSS, TS, COD and BOD₅. The composite adsorbent performed excellently for all the samples. The concentrations of the components contained in the waste water were analyzed by UV-Visible spectrophotometer and High Performance Liquid Chromatography (HPLC). After the treatment, the decreasing trend of the absorbance and total peak area indicated that the significant amount of effluent materials were removed.

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Introduction

Pharmaceutical and textile industries in Bangladesh are widely distributed throughout the country and there is a consistent global increase in the use and production of potent pharmaceutical, distribution of yarn, cloth and clothing respectively (Aust *et al.*, 1990). These industries use both inorganic and organic compounds as raw materials, latter being either of synthetic or of vegetable and animal origin (Rao and Datta, 1979). The inorganic compounds are mainly metallic salts and various other basic and acidic compounds that undergo different chemical and biochemical reactions in the water systems (Safiullah *et al.*, 1989). The organic compounds can be of biodegradable and non-biodegradable in nature. The biodegradable organic component through its degradation creates several hazardous situations in the water system, such as depletion of dissolved oxygen (DO), production of odorous gases, change of pH etc. The non-biodegradable organic substances persist in the water

system for a long time and passes through the food chain (Khan, 2010). The pharmaceutical and textile industries have been playing a significant role in Bangladesh's economical growth for a long time. Currently, the textile industries in Bangladesh contributes 5% of the total national income (Mandal, 2013) and 46.98% gross outputs of top ten industrial sectors in Bangladesh (Sagris and Abbott, 2015). On the other hand, Bangladesh is the only least developed country in the world that meets up to 97% of its demand for pharmaceutical products through local companies (Mahmud, 2018). Among these industries, some do not discharge liquid waste at all, while some discharge concentrated liquid waste in very small scale. Most of other industries discharge highly alkaline and toxic liquid wastes. The discharged effluent contains high BOD, COD, dissolved and suspended matters, heavy metals, dye compounds (Sagris and Abbott, 2015; SWIG-Textile, 1997).

*Corresponding author e-mail: skabir_ju@yahoo.com

In order to prevent environmental pollution, there have been different methods to treat the waste water and reuse it. The ancient methods employed for the treatment of waste water includes physical, chemical and thermal methods. But these methods have disadvantages which includes huge labor requirement, high maintenance cost, low efficiency and large equipment. Adsorption technique has attracted a great amount of attention due to its cost effectiveness, easy operation, high efficiency, simplicity of the equipment, and easy regeneration of materials (Shan *et al.*, 2015). Activated charcoal is a form of carbon that is extremely porous and have a very large surface area available for adsorption and chemical reactions. Adsorption on activated carbon is selective, favoring non polar over polar substances (Ashraf *et al.*, 2019; Yagub *et al.*, 2014; Sun *et al.*, 2013). In this study, our objective was to modify the activated charcoal by manganese dioxide, ferric oxide, soil, sand and make a composite to improve the adsorption capacity and versatility.

Materials and methods

Preparation of composite adsorbent

A 100 g composite material was prepared by mixing of 10% activated charcoal (Source – BDH Chemicals Ltd. England, granular size 10-18 mesh), 10% manganese dioxide (Source – BDH Chemicals Ltd. England), 20% ferric oxide (Source – Qualikems Fine Chemicals Pvt. Ltd. India), 40 % soil (Source – Jahangirnagar University, Bangladesh, granular size 10-15 mesh) and 20% sand (Source – Padma River, Pakshi, Kushtia, Bangladesh, granular size 250-355 μm). Following the uniform mixing, it was dried at 105 °C for two hours and kept in a desiccator to avoid the gaining of moisture. Then, the content of composite materials was taken in a column made up of glass tube having an internal diameter 22 mm and height of content 270 mm.

Sample collection

Four different types of samples were collected from the discharge points of Gonoshasthaya Antibiotic Limited, Mirzanagar, Dhaka (GAL); Gonoshasthaya Pharmaceuticals Limited, Mirzanagar, Dhaka (GPL); Pakiza Dyeing and Printing Industries Ltd., Choto Bali Mehor, Savar, Dhaka (PDPI) and Bangladesh Dyeing and Finishing Ind. Ltd., DOEL group, Karanpara, Savar, Dhaka (BDFI). 2.0 l of the effluent liquid samples were collected from each sampling location approximately 500 cm depth from the surface of the water to avoid the contamination from any floating materials. The standard water sampler (Hydro Bios, Germany) was used with four pre-cleaned 500 ml plastic containers to collect the water from each sampling site. Then waters from four

containers (4 x 500 ml = 2 l) were mixed together to obtain a mixture. The effluent mixture was filtered through 0.45 μm membrane filters.

Preparation of synthetic effluents

Four synthetic effluents were prepared which represent an average character of the samples collected from GAL, GPL, PDPI and BDFI sampling locations. The synthetic effluents were marked as GAL-SYN, GPL-SYN, PDPI-SYN and BDFI-SYN respectively.

Preparation of GAL-SYN

2.0 g each of the materials such as methylene chloride, isopropyl alcohol, 2,6-lutidine, 2-ethylhexanoic acid, hydrochloric acid, N, N-dimethylacetamide, triethylamine, acetone, ethyl acetate, ethanol, methyl isobutyl ketone, n-butanol and methanol were diluted to 200 ml with distilled water. This solution was termed as ‘Solution – A’. ‘Solution – B’ was prepared by dissolving 6-aminopenicillanic acid (0.2052 g), ethylene diamine tetraacetic acid (0.2026 g), charcoal (0.2010 g), sodium chloride (0.2006 g), sodium sulfate (0.2002 g), amoxicillin trihydrate (0.2049 g), flucloxacillin sodium (0.2037 g), cefradine (0.2040 g), cloxacillin sodium (0.2094 g), ciprofloxacin hydrochloride (0.2020 g), 3-(2-chloro-6-fluorophenyl)-5-methyl isoxazole-4-carbonyl chloride (0.2025 g), D(-) alpha para hydroxyl phenyl glycine methyl potassium (0.2052 g) in 2000 ml distilled water. Then, 10 ml of *Solution – A* and 90 ml of *Solution – B* were mixed and diluted to 200 ml by distilled water. Again, 40 ml of this solution was diluted to 1000 ml using distilled water.

Preparation of GPL-SYN

GPL-SYN solution was prepared by dissolving amoxicillin sodium (0.210 g), chlorphenamine maleate (0.202 g), atropine sulfate (0.206 g), amlodipine besilate (0.242 g), atorvastatin as calcium (0.205 g), bupivacaine hydrochloride (0.211 g), chloramphenicol (0.203 g), trimethoprim (0.201 g), calcium gluconate (0.207 g), ciprofloxacin lactate (0.205 g), cefixime (0.202 g), diazepam (0.204 g), diclofenac sodium (0.202 g), dexamethasone sodium phosphate (0.205 g), furosemide (0.207 g), flucloxacillin sodium (0.207 g), griseofulvin (0.2022 g), haloperidol (0.208 g), methylparaben (0.204 g), providone (0.2075 g), magnesium stearate (0.204 g), sodium saccharin (0.109 g), folic acid (0.025 g), ketamine hydrochloride (0.203 g), lidocaine hydrochloride (0.208 g), metronidazole (0.209 g), misoprostol 1% (0.206 g), metformin hydrochloride (0.204 g), neostigmine methyl sulfate (0.205 g), paracetamol (0.200 g), phenobarbital

sodium (0.201 g), prednisolone (0.205 g), salbutamol sulfate (0.2073 g), hydrochlorothiazide (0.203 g), thiamine hydrochloride (0.207 g), riboflavin (0.010 g), maize starch (0.210 g) and lactose (0.192 g) in 2000 ml distilled water. Then 28 ml of this solution was diluted to 1000 ml using distilled water.

Preparation of PDPI-SYN

1.0 g each of the materials sodium bicarbonate, urea, soda ash, sodium chlorate, resis salt, alginate gum and 10.0 g each of the silicon softener (liquid), detergent (liquid), acetic acid (10%), sodium hydroxide (10%) were dissolved in 100 ml distilled water. 50 ml of this solution was mixed with 50 ml red BP (reactive), 0.1 % and yellow – H4G (reactive), 0.1 %. Then, the solution was diluted to 1000 ml using distilled water.

Preparation of BDFI-SYN

10.0 g each of the materials detergent (liquid), sequestering agent KEB/SED (liquid), anti creasing agent Ciba – C (liquid), peroxide killer (liquid) and 10 ml of each of hydrogen peroxide (10% solution), sodium hydroxide (10% solution), hydrosol (10% solution), acetic acid (10% solution), glauber's salt (10% solution), sodium bicarbonate (10% solution) were dissolved to 100 ml distilled water. Then, 50 ml of this solution was mixed with 50 ml reacto bond red 3BX, Kiri, India (0.1 % solution) and 50 ml selazol blue BRF, setaschemiya, China (0.1% solution). The solution was then diluted to 1000 ml using distilled water.

Treatment of practical and synthetic effluent by composite adsorbent (column method)

The effluent treatment ability of the composite adsorbent was examined through column method. In this method, a fixed amount of composite material (100 g) was taken in a column made up of glass tube having an internal diameter 22 mm and height of content 270 mm. The lower end of the column is bent in the form of a burette having a stop cock for controlling out flow. Glass fiber filter is placed at the lower end of the column, first to support the adsorbent and second to allow only liquid to pass through it. A 200 ml effluent from different samples was passed through the column containing the adsorbent. The flow rate was maintained 2.0 ml/minute. The treated water was collected in a plastic container for further analysis.

Analytical method

The principal equipments used in this study were High Performance Liquid Chromatography (HPLC) (Shimadzu, Japan, model – CTO – 10 AS), LC – 20AT (solvent delivery module), SPD – 20A (uv visible detector); UV-Visible spectrophotometer (Shimadzu, Japan, model – 1601PC). TDS, DO, BOD and COD were analyzed using APHA method (APHA *et al.*, 2017).

Results and discussion

Physical appearance and pH

The physical appearance of the samples collected from the GAL and GPL were found yellowish brown with objectionable odor. But a deep reddish brown and deep violet colors were found in the samples collected from PDPI and BDFI respectively. In the textile-dyeing industries, a mixture

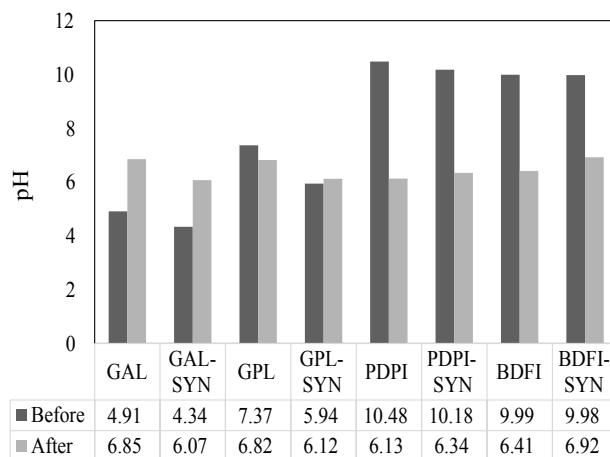


Fig. 1. Change of pH in different samples before and after treatment

of azoic (for example, fast violet B base, fast violet K base, fast blue RR base) and vat (for example, algol blue 3R, indenthren yellow 5FK, indigo) dyes were used in the different steps of the production (Khan, 2010). Therefore, a lot of dye containing effluent are released from these industries. In case of synthetic effluent, it was observed that all the samples were clear but different in colors. A colorless, light yellowish, deep red and deep violet blue colors were observed for GAL-SYN, GPL-SYN, PDPI-SYN and BDFI-SYN samples respectively. All the samples (both practical and synthetic) were found clear, colorless and odorless after the treatment with composite adsorbent. A wide range of pH variation was observed in Fig. 1 for

different samples. The effluents collected from GAL and GPL, both practical and synthetic were found acidic in nature. These effluents contain a wide variety of chemicals such as acids, drugs, salts etc. pH values generally depend on the nature of chemicals used in synthesis and other compounds and their reaction mechanism (Satyanarayana *et al.*, 2015). Satyanarayana *et al.* (2015) observed that the effluents collected from different pharmaceutical industries were commonly acidic in nature. On the other hand, effluents from PDPI and BDFI, both practical and synthetic were alkaline in nature (Fig. 1). The pH of textile effluent is generally high due to use of many alkaline substances in textile processing

(Islam and Guha, 2013). After the treatment, all samples were well within in the range of the standards established by different organizations (Table I).

Total dissolved solids (TDS)

Fig. 2 shows that the total dissolved solids in GAL, GPL, PDPI and BDFI were 685, 259, 1890 and 4760 mg/l respectively and in GAL-SYN, GPL-SYN, PDPI-SYN and BDFI-SYN were 19.9, 76.4, 5712 and 4891 mg/l respectively. The concentrations were significantly high in textile and dyeing related samples, for example, PDPI, BDFI, PDPI-SYN and

Table I. Water quality standard of physico-chemical parameters from different organizations

Parameter	Unit	DoE, BD		MoE, JP	EPA
		Inland surface water	Irrigatedl and	National effluent standard	Surface water
pH		6 - 9	6 - 9	5.8 - 8.6	5.5 - 9.0
TDS	mg/l	2100	2100		
TSS	mg/l	150	200	200	50
BOD ₅ , 20 ^o C	mg/l	50	100	160	5
COD	mg/l	200	400	160	40
DO	mg/l	4.5 - 8	4.5 - 8		

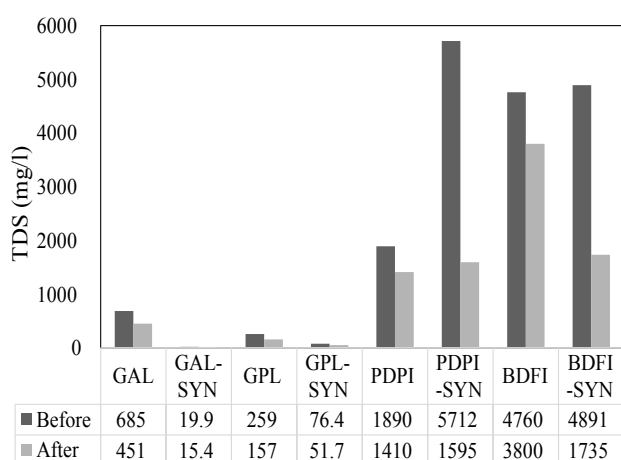


Fig. 2. Concentration of TDS in different samples before and after treatment

BDFI-SYN. The composite adsorbent performed in removing the dissolved solids 20-40% for the samples. But, the samples PDPI-SYN and BDFI-SYN showed a higher removal of 65-2%. The total dissolved solids comprise inorganic salts and some small amount of organic matters which are dissolved in water. In case of inorganic salt, the principal components present as cations are: calcium, magnesium, sodium and potassium, while for anions are: carbonate, bicarbonate, chloride, sulfate and nitrate (DNHW 1993). Beside these, a wide variety of organic and inorganic components are present in the practical waste water. Some of these are complex in nature, especially in the different types of bond formation. This might be the reason for lower removal in the practical effluent than the synthetic. According to the guideline of DoE, BD (Table I), the permissible limit of TDS is 2100 mg/l for both inland

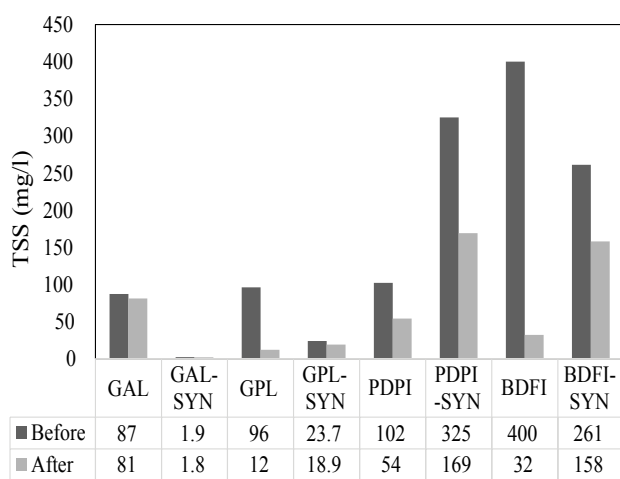


Fig. 3. Concentration of TSS in different samples before and after treatment

surface water and irrigated land. After the treatment, it is observed that the concentration of TDS in all the samples are within the limit, except BDFI sample.

Total suspended solids (TSS)

Fig. 3 shows that the total suspended solids in GAL, GPL, PDPI and BDFI were 87, 96, 102 and 400 mg/l respectively and in GAL-SYN, GPL-SYN, PDPI-SYN and BDFI-SYN were 1.9, 23.7, 325 and 261 mg/l respectively. According to the guideline of DoE, BD and MoE, JP (Table I), the permissible limit of TSS is 200 mg/l for both irrigated land and national effluents. After the treatment with composite adsorbent, it is observed that the concentrations of TSS in all the samples are within the limit. TSS are solids in water that can be trapped by filtration. High concentrations of suspended solids can cause many problems for stream and health of aquatic life. It causes to, reduce the photosynthesis and thus the water plants are not capable to produce the oxygen, therefore, decaying occurs. High TSS can also cause an increase in surface water temperature, because the suspended particles absorb heat from the sunlight (Mohammad, 2003).

Chemical oxygen demand (COD)

The chemical oxygen demand of the samples collected from GAL, GPL, PDPI and BDFI was 1590, 38.4, 585.6 and 192 mg/l respectively, whereas, the synthetic waste water contained the COD of 134.4, 590.4, 2428 and 3043 mg/l

for the samples GAL-SYN, GPL-SYN, PDPI-SYN and BDFI-SYN respectively (Fig. 4). From Fig. 4, it is clearly observed that the removal of COD was more than 85% for the synthetic waste water, but different removals were found for the practical waste water. On the other hand, a high removal was observed from the high concentration of COD containing waste water. Chemical oxygen demand (COD) is the total amount of oxidizable organics (biodegradable and nonbiodegradable and both dissolved and particulate), measured by the amount of oxygen in the

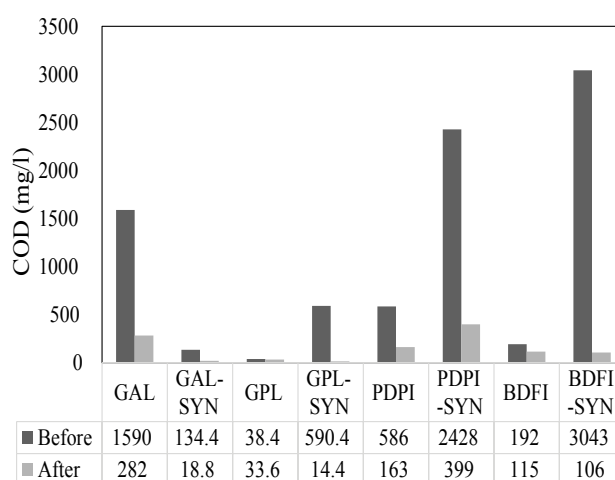


Fig. 4. Concentration of COD in different samples before and after treatment

form of oxidizing agent required for the oxidation of organic matters (Bergem *et al.*, 2018). Table I explains that the permissible limit for the COD in the inland surface water and irrigated land were set 200 and 400 mg/l according to the DoE, BD. Our treatment experiment shows that most of the samples followed inland surface water standard, whereas, GAL and PDPI-SYN followed irrigated land standard.

Biochemical oxygen demand (BOD)

The biochemical oxygen demand (5-days, 20 °C incubation) of the samples collected from GAL, GPL, PDPI and BDFI was 1280, 2.3, 110 and 24 mg/l respectively, whereas, the synthetic waste water contained the BOD (5-days, 20°C) of 120, 330, 1467 and 400 mg/l for the samples GAL-SYN, GPL-SYN, PDPI-SYN and BDFI-SYN respectively (Fig. 5). After treatment with composite adsorbent, it was found that the removal was

more than 80 % in most of the samples. The maximum permissible limit for BOD (5-days, 20 °C) is 100 mg/l for the irrigated land by DoE, BD (Table I). After treatment, the samples of GAL, PDPI-SYN and BDFI-SYN contained BOD₅ higher than the permissible limit due to high initial concentration. Nevertheless, the removal was observed more than 80% for these samples. BOD₅ indicates the amount of putrescible organic matter present in water. Therefore, a high BOD₅ indicates polluted water. Dissolved oxygen (DO) is consumed by bacteria when large amounts of organic matter from sewage or other discharges is present in the water.

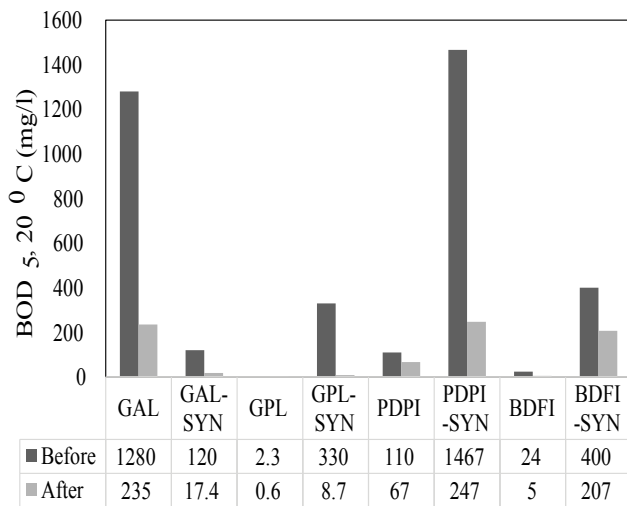


Fig. 5. Concentration of BOD₅ in different samples before and after treatment

Dissolved oxygen (DO)

Fig. 6 shows that the DO values were very low initially. But after the treatment, the DO value increased and contained within the range set by the DoE, BD (Table I). The values in PDPI-SYN and BDFI-SYN still low. Actually, the DO in water is very related with BOD. In these samples, the BOD values were very high, therefore, the demand for oxygen by the bacteria was high and they were taking that oxygen from the oxygen dissolved in the water. When the DO drops below a certain level, the life forms in that water are unable to continue at a normal rate. The decrease in the oxygen supply in the water has a negative effect on the fish and other aquatic life.

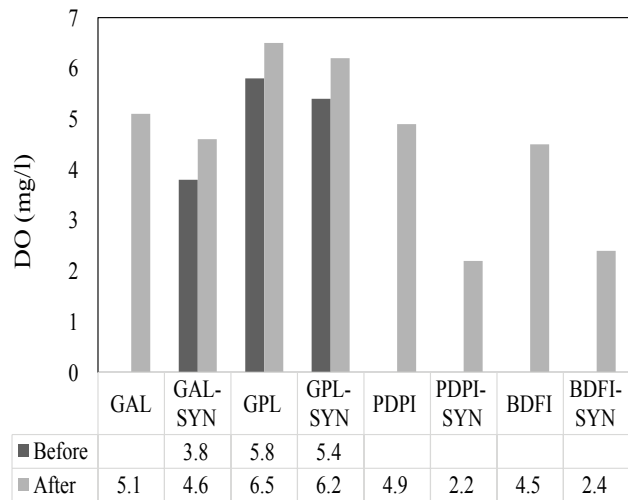


Fig. 6. Concentration of DO in different samples before and after treatment

Total concentration of the substances in the waste water

The concentration of different components contained in the waste water was analyzed by UV-Visible spectrophotometer and High Performance Liquid Chromatography (HPLC). The samples employed in this study contained various types of pharmaceuticals and textile related components. All or most of the components response to spectrophotometric absorbance and HPLC chromatogram. Table II shows that all the samples exhibits absorption peak at different wavelengths. For example, the sample GAL exhibits absorbance 1.2831 and 1.5939 at the wavelengths of 302.5 and 271nm respectively. But after the treatment with composite adsorbent, it is clearly observed that the absorbance peaks were reduced or disappeared for all the samples. In case of HPLC analysis, the samples displayed different peak area for different components present in the samples. Table III summarizes the total peak areas for each of the samples. The total peak areas in GAL, GAL-SYN, GPL, GPL-SYN, PDPI, PDPI-SYN, BDFI and BDFI-SYN were observed 1210278, 650574, 249470, 599737, 4305432, 339371, 743910 and 11633041 mV-min respectively, whereas after the treatment the values of the total peak areas were reduced to 52510, 230243, 46354, 58924, 79725, 130915, 225909 and 222161 mV-min respectively. The UV-Vis absorbance and HPLC total peak areas represent the total

Table II. Spectrophotometric data of different industrial effluents before and after treatment

Sample	Type of treatment	Wavelength (nm)	Absorbance (%)
GAL	Before	302.5	1.2831
		271.0	1.5939
	After		No peak
GAL -SYN	Before	270.5	1.9615
		315.0	0.0499
	After	270.5	0.1313
		315.0	0.0126
GPL	Before	278.0	0.123
	After		No peak
GPL -SYN	Before	267.5	1.1106
	After	267.5	0.0149
PDPI	Before	669.0	0.3365
		512.0	1.3784
	After		No peak
PDPI -SYN	Before	534.0	1.3926
		515.0	1.3939
		425.5	1.0098
	After		No peak
BDFI	Before	542.5	1.350
	After	532.0	0.0176
		288.5	0.0234
BDFI -SYN	Before	330.0	1.4478
		527.0	0.3843
	After		No peak

Table III. The total peak area (HPLC analysis) of different industrial effluents before and after treatment

Sample	Type of treatment	Total peak area (mV -min)
GAL	Before	1210278
	After	52510
GAL -SYN	Before	650574
	After	230243
GPL	Before	249470
	After	46354
GPL -SYN	Before	599737
	After	58924
PDPI	Before	4305432
	After	79725
PDPI -SYN	Before	339371
	After	130915
BDFI	Before	743910
	After	225909
BDFI -SYN	Before	11633041
	After	222161

components present in the samples. Therefore, the decreasing trend of the absorbance and total peak area indicated that the significant amount of effluent materials were removed.

Conclusion

The removal of organic wastes from effluent of pharmaceuticals and textile industries was carried out using a composite adsorbent. Four different types of samples were collected from the discharge points of Gonoshasthaya Antibiotic Limited, Gonoshasthaya Pharmaceuticals Limited, Pakiza Dyeing and Printing Industries Limited and Bangladesh Dyeing and Finishing Industries Limited. Four synthetic effluents were prepared which represent the average character of the collected samples. All the samples contained a high concentration of TDS, TSS, TS, COD and BOD₅ with a low value of DO. The composite adsorbent showed excellent treatment capacity for all the samples. The results of UV-Visible and HPLC analysis showed high absorbance and an increase in total peak areas respectively representing considerable amount of components present in the untreated samples. After the treatment with composite adsorbent, it is clearly observed that the absorbance peaks were reduced or disappeared for all the samples. The decreasing trend of the absorbance and total peak area indicated that the significant amount of effluents materials were removed. The findings of this study are important for assessing water purification technologies. Moreover, this work addresses our further understanding of the mechanisms behind the removal processes.

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