

## Wastewater Treatment of Textile Industry via Adsorption and Electrochemical Regeneration

S. N. Hussain<sup>1+</sup>, A. Ahmad<sup>1</sup>, A. Ali<sup>1</sup>, H. Sattar<sup>1</sup> and H. M. A. Asghar<sup>1</sup>

<sup>1</sup>Institute of Chemical Engg.& Tech., University of the Punjab, Lahore

**Abstract.** In Pakistan, industries are producing wastewater that carries pernicious pollutants and this concern has been of yearning interest to the researcher's today. In this milieu, the wastewater beget from textile industry comprise large amount of dyes and chemicals containing trace metals such as Cr, Ar, Cu, Zn. The present study deals with the treatment of textile industry effluent by adsorption using graphite adsorbents with electrochemical regeneration. The results of this work have revealed that graphite based adsorbent is fully capable of removing chemical oxygen demand (COD) from the effluent of wastewater of textile industry. The adsorbent showed regeneration efficiencies greater than 95% at 1.5 A current for 20 min.

**Keywords:** Electrochemical regeneration, textile wastewater, COD

### 1. Introduction

Water contamination has been a major problem not only in a developing country like Pakistan but also in many other developed countries around the globe. Industrial and Agricultural waste water are allowed to drain in the nearby ponds lakes or water channels that not only poisons that particular water, killing the marine life but has other alarming affects as well. With active NGO's and different other awareness campaigns the people are very well familiar with the hazards different forms of pollution can cause so the industries today are focusing too on the pollution menace their waste is causing and are ready to invest in effluent free waste projects.

Wastewater of textile includes a wide range of dyes and chemical additives that make this industry as a pollution source. Wastewater, in the textile industry is generated from the processes of dyeing and finishing. Wide range of dyestuff and chemicals like organic compounds having complex structures are required as an input in these processes. Excess water is used for the application of different chemicals and dyes for the finishing process. Among the major contaminants in textile wastewaters are COD, colour, acidity, high suspended solids and other soluble substances. The major contaminants that need to be eliminated from the textile wastewater are nitrogen, BOD, COD, dyestuffs and heavy metals. The textile waste characteristics varies and it is dependent on the type of textile produced and different chemicals used. The textile wastewater can cause serious damage to the environment and human health because it contains huge amounts of agents including suspended and dissolved solids, BOD, COD, chemicals, odour and color. The majority of the ratios of BOD/COD are found about 1:4 and it represents non-biodegradable substances existence [1], [2].

Significant amount of organic substances are present in textile dyestuff which cause hindrance in degradation process and are difficult to discard aerobically. Under anaerobic situation organic substances are observed to reduce into carcinogenic agents [3]. Interaction to textile dyes has causes problems to humans including congenital malformations, skin irritations, lungs problem, nausea and headaches [4], [5]. Studies regarding the viability of treating textile wastewater are significant [1]. Many techniques like biochemical,

<sup>+</sup> Corresponding author. Tel.: +924299232048; fax: +924299231159.  
E-mail address: hussain\_nadir@hotmail.com.

physiochemical and other combined treatment techniques have been developed to find an economic and efficient way to treat the textile wastewater. Adsorption is mainly used method in physicochemical wastewater treatment, which can merge the wastewater and the porous material powder or granules, such as activated carbon and clay, or allow the wastewater through its filter bed composed of granular materials. Through this process, effluents in the wastewater are adsorbed and removed on the surface of the porous material or filter.

High efficiency of activated carbon makes adsorption a suitable technique for the removal of pollutants from wastewater. However, the economics of the process utilizing adsorption rely on the regeneration and the re-use of the depleted adsorbent. Considering the economics of the wastewater treatment technologies, adsorption with electrochemical regeneration of the graphite adsorbents has reaped significant importance. Electrochemical regeneration refers to the adsorbent regeneration within an electrolytic cell where the adsorptive capacity of the adsorbent is restored, so that it can be used for multiple adsorption cycles. This method has been used for the treatment of waste waters contaminated with dissolved organics [6]-[9]. The present appraisal deals with the adsorption of dissolved organics present in the wastewater of textile industries using graphite based adsorbent called Nyex®1000. The selected adsorbent is cheap, gives quick adsorption equilibrium times and has better adsorption properties compared to other available adsorbents [6].

## 2. Materials and Methods

### 2.1. Materials

In this study, a graphite intercalation compound (GIC) adsorbent with flake morphology was used. This material under the trade name of Nyex®1000 was supplied by Arvia Technology Ltd. As the material is non-porous in character, therefore, possesses a low surface area of around  $1.0 \text{ m}^2/\text{g}$ . The mean particle diameter of the particles is in the range of 450-500 micrometer. The sample of the water to be treated was received from a textile industry located in Lahore, Pakistan. Analytical grades of all the chemicals are used in this study.

### 2.2. Equipment

A sequential batch reactor (SBR) as shown in Fig. 1 was used to carry out the adsorption and electrochemical regeneration experiments in the laboratory. It had an anode current feeder (a graphite plate of 5 mm thick) and a stainless steel cathode with 3mm diameter perforations. Both of these electrodes are being separated by a microporous polyethylene membrane. Area of each electrode was  $50 \text{ cm}^2$ . The details of this equipment has been described elsewhere (9). For the adsorbent and water to mix, air was supplied through an array of orifices at the base of the cell during adsorption. The air flow was maintained at such a level that the splashing of water from the top of the reactor could not take place.

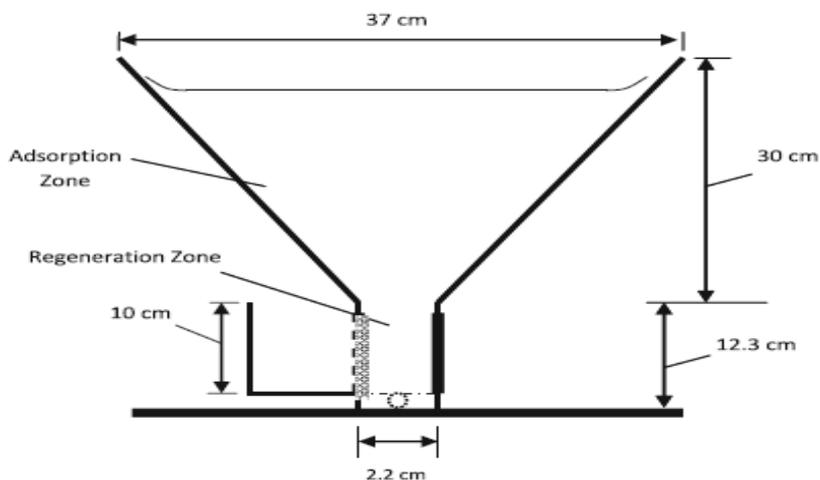


Fig. 1: Diagram showing Sequential batch reactor (SBR) used for adsorption and electrochemical regeneration

## 2.3. Methods

Initially, the water sample to be treated was analyzed for COD (see analysis). 1L of effluent water was poured in the SBR and a measured quantity of adsorbent was added to it. This was followed by the mixing of adsorbent and water via air sparging for 30min. Once the adsorption time was completed, the air supply was switched off and the adsorbent particles were allowed to settle down for 5min in the anode compartment of the SBR as shown in Fig. 1. Once the adsorbent has settled, sample of the adsorbed water is collected, filtered and analyzed for the COD content that remains after adsorption. Approximately 350mL of electrolyte i.e.0.3% NaCl + 0.125MHCl solution was added to the cathode compartment of the SBR in such a way that the level of the electrolyte and the settled bed of the adsorbent are the same. Then a suitable amount of current for a certain time period was applied across the electrodes for regeneration of the adsorbent. The current was kept constant while the changes in the voltage were monitored.

Once the current has been supplied to the electrodes for the desired time, the power supply was switched off and sample of water was taken to analyze its COD after regeneration. The water present above the adsorbent bed was then siphoned off along with the electrolyte in the cathode compartment. Fresh sample was then added to the SBR and the process of adsorption and regeneration discussed above was repeated for a number of times.

## 2.4. COD Analysis [10]

A known volume of the water sample (20 mL) to be analysed was poured in a rounded flask connected with a reflux condenser. A measured volume of potassium dichromate solution (0.1N, 30 mL) was added to the flask. Afterwards, concentrated sulphuric acid (30 mL) was gradually added to the contents of the flask from the open end of the condenser. Make sure that the acid is added gradually to avoid the escape of volatile matter to the surrounding. Afterwards, the contents of the flask were digested at 150 ° C for 2h. On the completion of digestion, the contents of the flask were allowed to cool down to room temperature. Afterwards, flask material was transferred to conical flask followed by the addition of 2-3 drops of ferroin indicator and titrate the the flask contents against standard solution of Mohr salt till the orange colour turns to red. In this way, the amount of of  $K_2Cr_2O_7$  consumed and the oxidizable matter was calculated in terms of oxygen equivalent.

Calculation of COD:

$$\text{COD as mg } \frac{O_2}{L} = \frac{(A - B) \times M \times 8000}{\text{mL sample}}$$

where:

A = volume of FAS used for blank,

B = volume of FAS used for sample,

M = molarity of FAS,

8000 = milli equivalent weight of oxygen  $\times$  1000 mL/L.

## 3. Results and Discussions

### 3.1. Kinetic Studies

The kinetic studies were carried out in which the effluvia water was treated with the GIC adsorbent at room temperature for ascertaining the inceptive COD of the effluvia water and the adsorbent effect on the effluvia water. At room temperature the effluvia water was subjected to a 2h batch adsorption process in which 1litre of filtered water (from which fibers were removed) was spout in a large beaker in which a known measured quantity of the Nyex<sup>®</sup>1000 was added. Than the beaker was placed on a magnetic stirrer and was stirred at the covet r.p.m at which the mixing of adsorbent and the effluvia water was ample. The sample was stirred for 2h during which adsorbed water samples were withdrawn at 5, 10, 20, 30, 60, 120 and 180 min time interludes and were later anatomized for the COD probe The acquired results of COD and percentage adsorption from the above kinetic experiment is shown in the Fig. 2-3 below:

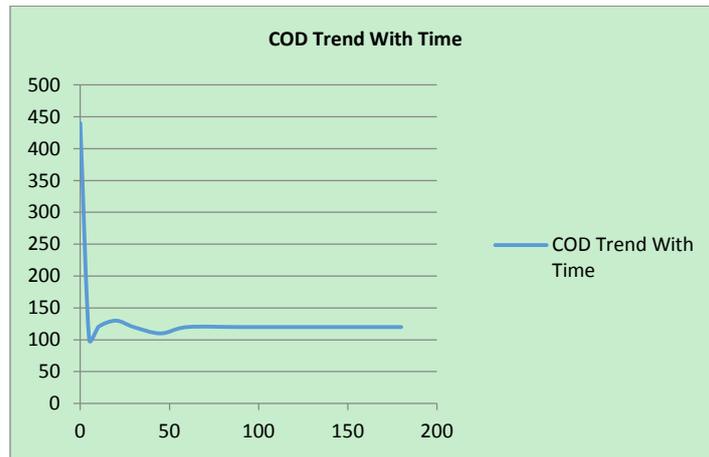


Fig. 2: COD variation with time at Room Temperature

From Fig. 2, it can be clearly seen that the adsorbent has been effective with the effluvia water of the textile industry as the inceptive COD of 440mg/L was reduced to 100mg/L in very short span. This substantial curtailment in COD was due to the high driving force for the transfer of the COD from the solution to the surface of Nyex®1000 at the start of adsorption followed by the expeditious mass transfer to the surface of the adsorbent. Adsorption can be considered to be expeditious when compared to porous activated carbon adsorbents, which take various days to reach equilibrium. The adsorption equilibrium was achieved within the first 30 minutes with no change in COD curtailments up to the 2h adsorption process carried out at the inceptive stage of study. The slight jump in the COD values at t=10 and t=20 might have been due to an experimental error that might have been incurred during the course of experimentation as the system again reaches equilibrium at t=30 onwards.

Percentage Adsorption was calculated by the following formula:

$$\% \text{ age Adsorption} = \frac{C_o - C_t}{C_o} \times 100$$

where:

$C_o$  = inceptive COD.

$C_t$  = COD at any time.

It was then observed from Fig. 3 that the adsorption efficiency is about 73% and this efficiency is achieved within no time indicating that the adsorbents affinity with the covet contaminant of the effluvia water.

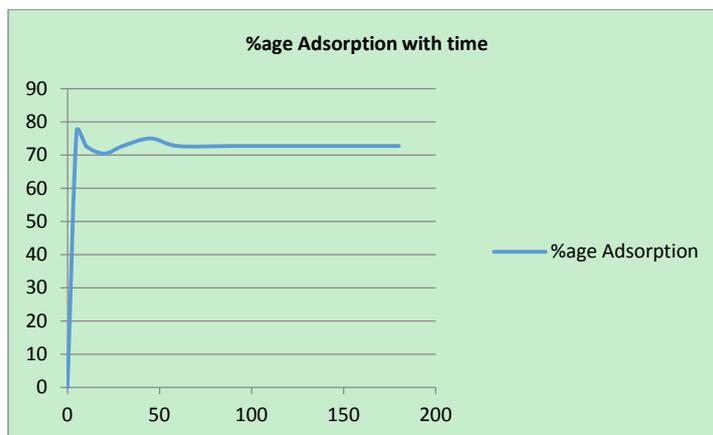


Fig. 3: Percentage Adsorption with respect to time

### 3.2. Adsorption and Electrochemical Regeneration Using SBR

The effluvia was treated for the adsorption effect in two different ways. Initially, the first cycle was run for six adsorptions and five Regenerations in which each time fresh effluvia water was poured in the SBR and was adsorbed via air sparging for 30min. The effluvia had to first undergo adsorption where the

effluvium and adsorbent were mixed for 30min and then the adsorbent had to undergo a 20min regeneration process. Afterwards, the treated water was siphoned off and new water was added in the SBR and the cycle continued for another cycle of adsorption and regeneration. Each time after the adsorption and after regeneration, samples of water were withdrawn for COD probe. The adsorbent is regenerated at 1.0 Amp current. The results of these experiments are shown in Fig. 4.

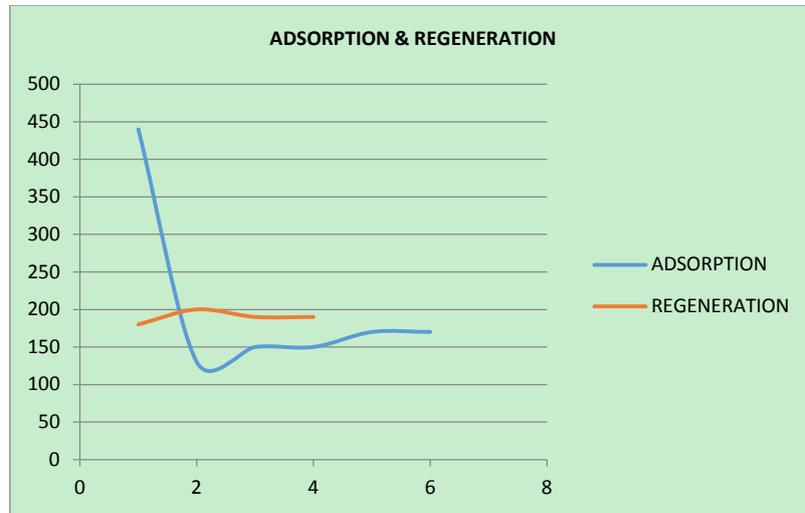


Fig 4: Adsorption and Regeneration using SBR

It can be observed from Fig. 4 that for every 30 min adsorption, the attainable COD of the effluvium water was as low as 130 mg L<sup>-1</sup>. Then to further investigate the effect of adsorbent and its regeneration capacity a run was carried out in which 1L sample was poured in the SBR with a known quantity of the adsorbent and adsorption followed by regeneration experiment was carried out. This time it was the same 1L sample from which different samples were withdrawn for COD probe after every 30min of adsorption and 20min of regeneration. Results of this experiment are shown in Fig. 5.

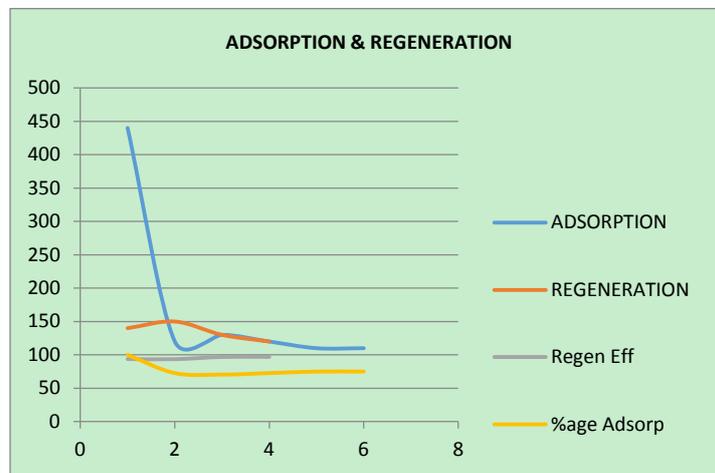


Fig. 5: Adsorption and Regeneration Statistics using different effluent Sample at 1 Amp

The results from this experiment are almost the same except one addition that the curtailment in COD is much greater as compared to all the experiments performed as the COD achieved is as low as 110mg/L and high adsorption efficiencies approaching to 75% as shown in Fig. 5.

Current is among the parameters that can affect the regeneration process so to check the current affect, 1L sample was treated but this time the adsorbent was regenerated at 1.5Amp to investigate the current effect on the process whose results are shown in Fig. 6 which is a graph showing the adsorption and regeneration COD variation and Fig. 7 which is a graph of adsorption and regeneration efficiency. The results as shown by the mentioned graphs show that the process as far as efficiency is concerned has been effective as the COD has dropped down to 90mg/L with an adsorption efficiency approaching to 80% and regeneration

efficiencies going high up to 97%. This indicates that the oxidation of organic contamination of the effluent of textile waste has been better at high current.

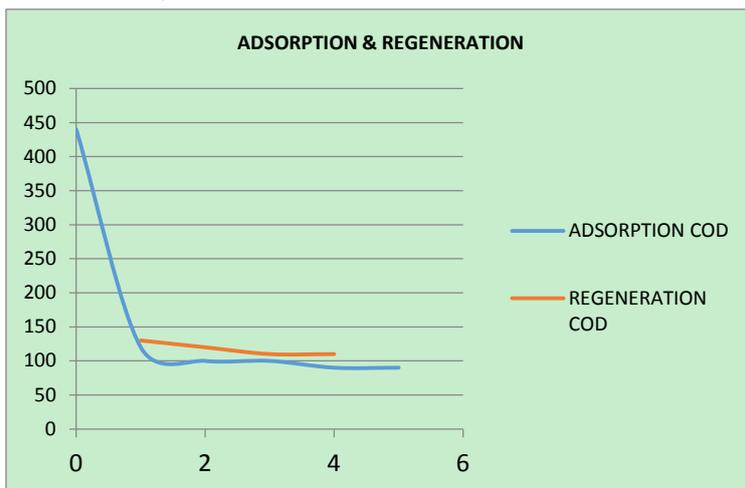


Fig. 6: Adsorption and Regeneration at 1.5 Amps

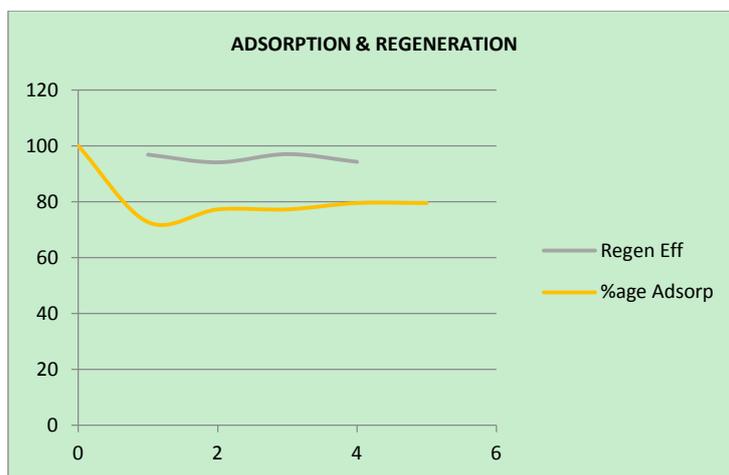


Fig. 7: Adsorption and Regeneration Efficiency at 1.5 Amps

#### 4. Conclusion

The present study shows that Nyex<sup>®</sup>1000, a bisulphate graphite intercalation compound has the capability of removing the COD from the effluent water of the textile industry with a percentage adsorption of almost 80% with 30 minutes of adsorption. The adsorbent regeneration in an electrochemical cell achieved regeneration efficiencies of greater than 95%. There is no loss of the adsorbent capacity on regeneration as multiple adsorption and regeneration cycles are carried out with the Nyex<sup>®</sup>1000.

#### 5. References

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