

RESEARCH ARTICLE

## Performance Analysis of Dye Removal Efficiency on Sewage Sludge Activated Carbon

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### ABSTRACT

Removal of dyes from effluents is done by the physio-chemical method. It is very sumptuous, for this cause, there is a demand for inexpensive and effective alternative method. The main dictum of this paper is to draw up activated carbon from sewage sludge and utilize it as an adsorbent in water for the removal of dyes. In this research work activated carbon was synthesized from sewage sludge by various activation methods such as synergistic chemical and physical activation using zinc chloride, a sol-gel method of  $TiO_2$  and modified sodium method. In this strive, the effective size of the activated carbon be elected and the analysis was run out on the outcome of various parameters like adsorbent concentration, pH and temperature in activated carbon on dye removal. This experiment demonstrated that the activated carbon prepared by combining chemical and physical activation using chloride method is more effective in dye removal and optimum efficiency is attained at the lower range of pH and temperature. This endeavor is effective for the removal of dyes from wastewater and also it is a hopeful technology to resolve environmental problems.

**Keywords:** Dye removal, Activated carbon, Sludge, Zinc chloride, GAC activation.

### 1. INTRODUCTION

Dyes are organic compounds which provide brighter color than conventional pigments, widely used for imparting color to textiles and also in Pharmaceuticals, foods, plastics, ink, Photographic, Paper and Paint industries as colorants. After agriculture, the major polluter of clean water is a dye. Over 10,000 different dyes are used in industries throughout the globe. In the usage of dyes, textile industry ranks first and it is one of the most water, chemicals, and energy consuming industry.[1] It is the largest consumer of dyestuffs; more than  $10^7$  kg/year dye is consumed by the textile industry. The higher amount of water is required in the dyeing of textiles extensively for finishing and dyeing operations that are around 200 tons of water per ton of product, hence the large volume of wastewater is generated from those operations and therefore 1,000 tons / year or higher of the dyes are being discharged into the waste

streams. The effluent coming out from a dyeing industry contains various chemicals and coloring compounds. Such wastewaters contain color, high alkaline, high biochemical oxygen demand (BOD) as 100-4000 mg/L, high COD as 150-10,000 mg/L, suspended solids, solvents and detergents, dyes, dyestuff, toxic chemicals and other inorganic compounds like sodium hydroxide, sodium hypochlorite, sodium sulphide, hydrochloric acid and sodium chloride. Likewise, textile effluents got high temperature [2] and pH between 4 and 12. During dyeing, nearly 10-15 % of dyes are discharged into the environment. Referable to the discharge of dyes in water, undesired and toxic (carcinogenic) matters are obtained. When the concentration of dyes is too high in water bodies, then the capacity of oxygenation of the receiving water gets stopped, the biological activity of the aquatic life gets upset due to the less ingress of sunlight in to the water bodies, Subject to the exposure time and dye

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concentration, dyes may have acute and/or chronic effects on exposed organisms. On aquatic plants, the photosynthesis process gets affected and humans are prone to mutation, skin irritation, allergic dermatitis, cancer and damage in DNA are caused due to the decomposition of dyes into pollutants. Hence, before discharging the effluent into any water bodies, it requires proper treatment [3]. To remove dyes from wastewater a number of chemicals, physical and biological methods are employed, such as chemical photolysis, chemical precipitation, chemical oxidation and reduction, electrochemical precipitation and photolysis, however these technologies are usually not effective in color removal and lack practical utilization due to the high price. Therefore, an inexpensive, efficient and convenient method is required [4].

The treatment methods applied for the removal of dye from the wastewater such as Fenton process, photo/ferrioxalate system, electrochemical and photo-catalytic combined treatments, photo-catalytic degradation using UV/TiO<sub>2</sub>, sonochemical degradation, adsorption, electrochemical degradation, integrated chemical-biological degradation, photo-Fenton process, Fenton biological treatment, ion exchange, reverse osmosis, ultra-filtration, Nano-filtration and cloud point extraction. Among these, adsorption treatment technology is the one that rapidly gaining prominence as treating of dye wastewater due to its low cost of regeneration, sludge free operation, effective in removing color, availability of known process equipment, recovery of the sorbate and soluble organic pollutants include pesticides, cyanides, phenol, organic dye, toxic chemical. Activated carbon is used widely as an adsorbent for dye removal and other pollutants include heavy metal because use of its extended surface area, high adsorption capacity, micro-pore structures, and a high degree of surface reactivity. Therefore, commercially available activated carbon is much expensive with a high regeneration cost while being enervated. Based on the average particle size activated carbon is classified, as either powdered or granular activated carbon. GAC adsorbs relatively small amounts of soluble organic and inorganic compounds such as nitrogen, sulfides, and heavy metals, hence granular activated carbon (GAC) adsorption have been applied successfully for treating dye wastewater [5].

Commercial activated carbon is extremely expensive and it increases the cost of treatment processes, to overcome this economical drawback cheaper raw materials are employed to prepare activated carbon [6, 7]. Primarily, a broad diversity of agricultural by-products and wastes, hardwood and bituminous coal, coconut shell and wood, Olive stones, sugar cane bagasse, pecan shells, palm seed, apple pulp, rubber seeds and molasses, etc. Recently, activated sludge is obtained from waste water treatment activities and evolved as an interesting option for the creation of activated carbon. In the European Union, more than 10 million tons of sewage sludge were produced. The five Indian states viz. Maharashtra, Tamil Nadu, Uttar Pradesh, Delhi and Gujarat account for about 50% of the total sewage generated [8]. In that, Maharashtra state contribution is of 13% of the total sewage generation. Wastewater treatment plants generate sludge that creates problems of disposal, that accounts for half the total cost of wastewater treatment. Primarily, sludge is disposed of either by spreading on land or burial. Some cities solve their problems by incinerating after drying. The cities along the coastal region, convey the sludge in to the sea through pipes or barges, but this method is now being seriously questioned. Hence, we synthesis activated carbon from sewage sludge for the removal of dyes from wastewater [9-11]. The primary motto of this paper is to look on to the characteristics of the synthesized activated carbon from sewage sludge and obtain the percentage of dye removal on various parameters such as contact time, temperature, adsorbate concentration, adsorbent dosage and pH.

## **2. MATERIALS**

The sewage sludge sample was collected from the sewage treatment plant to prepare activated carbon. The work on dye removal was carried out in various synthetic dyes like acid red, acid yellow, acid blue, acid black and acid orange.

## **3. PREPARATION OF GRANULAR ACTIVATED CARBON**

### **3.1. GAC1/S1: Combined chemical and physical activation using zinc chloride**

The wet sludge was kept in the oven overnight at 105°C for drying. 150 grams of dried sludge along with 5M of ZnCl<sub>2</sub> solution

was dissolved in 200ml of deionized water for Chemical activation. Physical Activation (Pyrolysis) was done at 500 °C in Muffle Furnace for 24 hrs. Then the sample was removed and soaked in 3M of Conc. HCL solution for an hour further it was cleaned with Deionised water. After cleaning, the sample is dried at 105 °C to remove the excess moisture content. Then the dried sample was soaked for 4 hours in 10 M of HNO<sub>3</sub> solution and redried at 105 °C.

**3.2. GAC 2 / S2: sol-gel method of tiO2 coating**

In this method, 100g of dried sludge was coated with 5ml of deionized water along with 28.7ml of Tio<sub>2</sub> prepared by dissolving titanium peroxide with 100 ml of isopropyl alcohol. Then Physical Activation (Pyrolysis)is done using Muffle Furnace at 500 °C. Further, the sample was rinsed with deionized water. After rinsing, the sample is dried for 24 hours in Oven at 100 °C.

**3.3. GAC 3/S3: modified sodium method**

The wet sludge was dried at 105 °C.10 M of sodium hydroxide solution is coated with 100g of dried sludge and kept in an oven at 180 °C for 24 hours. Then the dried sample is soaked with 3 M of Conc. HCL solution for an hour. Afterward, place the solution in oven at 180 °C for 8 hours and then rinse thoroughly with deionized water. After washing the sample dried for 24 hours in Oven at 100 °C.

**4. RESULTS AND DISCUSSION**

**4.1. Characteristics of sewage sludge**

The initial characteristics such as pH, Total Solids, TSS, TDS, BOD, TCOD, SCOD, Turbidity and moisture content of sewage sludge are examined and its depiction is presented in Table 1.

Table 1.Characteristics of sewage sludge

S.NO	Parameters	Unit	Value
1	pH	-	6.68
2	Total Solids	(mg/L)	23700
3	Total Suspended Solids	(mg/L)	20000
4	Total Dissolved Solids	(mg/L)	3700
5	Turbidity	nm	1.803
6	TCOD	(mg/L)	18000
7	SCOD	(mg/L)	1400

8	BOD	(mg/L)	50.1
9	Moisture content	%	7.25

**4.2. Characterisation of prepared activated carbon**

The characterization of the prepared activated carbon such as moisture, volatile, Ash and fixed carbon content is shown in table.

Table 2.Characterization of prepared activated carbon

S. No	Percentage	GAC 1	GAC 2	GAC 3
1	Moisture content	2.24	1.62	1.45
2	VM content	9.45	11.01	14.14
3	Ash Content	2.74	4.89	3.62
4	FC content	84.35	86.49	83.67

From the Table 2, it was observed that the amount of moisture content is high in GAC 1 as 2.24 %, which is 1.38 times greater than GAC 2 and 1.54 times greater than GAC 3. While in the case of volatile matter and ash content GAC 1 shows the least value as 9.45 % and 2.74 % respectively. The volatile matter and ash content present in GAC 1 are 1.17 and 1.5 times lesser than GAC 2, also 1.78 and 1.32times lesser than GAC 3. And the fixed carbon content maximum at GAC 2 as 86.49 %. From this, it was concluded that the carbon content is more in GAC 1 than GAC 2 and GAC 3 respectively.

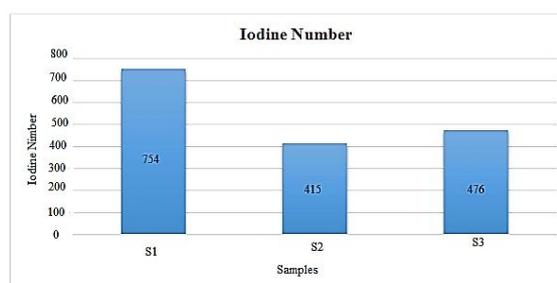


Figure 1.Iodine number of prepared activated carbon

From Figure 1 it was observed that, iodine number of activated carbon prepared from the combined method using zinc chloride reaches the maximum value of 754 which is 1.82 times greater than the sol-gel method of Tio<sub>2</sub> coating and 1.58 times greater than the modified sodium method, hence it concluded that Zinc chloride based Activated carbon has more micropores when compared to the activated carbon prepared from sol-gel method

of  $TiO_2$  and modified sodium method. Referable to the presence of high pores in zinc chloride based activated carbon, it prompts as a better adsorbent in the adsorption process.

### 5. EFFECTIVE SIZE OF GAC PREPARED

The activated carbon made from the blended physical and chemical activation method using zinc chloride, sol-gel method of  $TiO_2$  coating and modified sodium method is prepared in two different sizes such as 1-2 mm and 2-4 mm respectively. In Figure 2 and figure 3, the efficient size of the prepared activated carbon in removal efficiency is displayed.

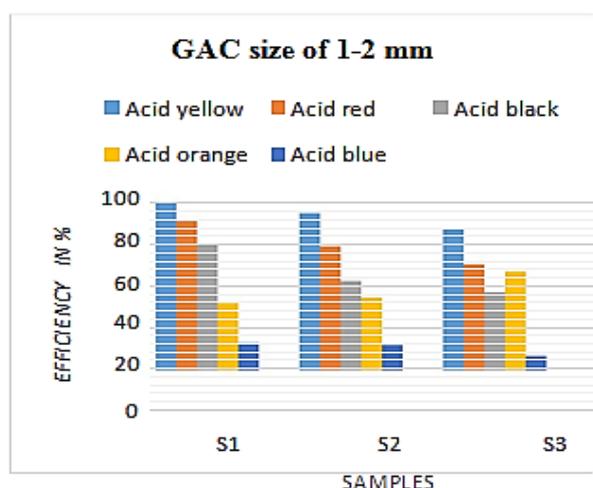


Figure 2. Optimum efficiency for GAC size 1-2 mm

From Figure 2 the prepared granular activated carbon (GAC) at size 1-2 mm was achieved the greater efficiency at GAC 1 in acid yellow dye as 92 %, which is 1.1 times greater than GAC 2 and 1.18 times greater than GAC 3 and the less efficiency was observed in acid blue dye as 8 % at GAC 3. The GAC 1 shows a more sizable performance in the removal of acid yellow, acid red, acid black and acid blue dyes and for acid orange dye GAC 3 given high removal efficiency, which is 1.35 times greater than GAC 2 and 1.46 times greater than GAC 1.

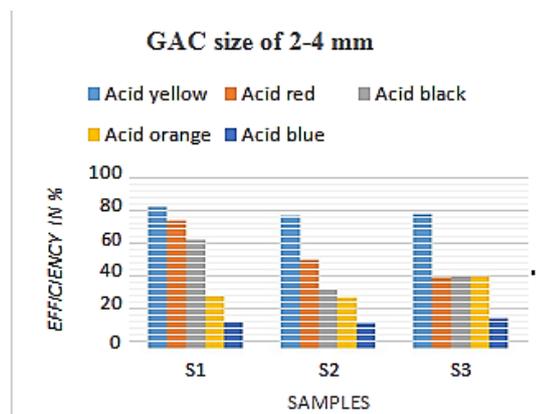


Figure 3. Optimum efficiency for GAC size 2-4 mm

From Figure 3 at GAC of size 2-4 mm, the greater efficiency was achieved by GAC 1 in acid yellow dye as 86 % and least efficiency of 15 % was achieved by GAC 2 in an acid blue dye. GAC 1 shows good performance in acid yellow, acid red, and acid black dyes. For acid orange and acid blue dye, the removal efficiency is greater in GAC 3. From Figures 2 and 3 it was concluded that the GAC of size 1-2 mm shows higher efficiency in dye removal than GAC of size 2-4 mm. In the GAC of size 1-2 mm, the acid yellow, acid red and acid blue dye shows greater removal efficiency in GAC 1. In the case of acid orange dye GAC 3 shows better results. Hence it is clear that to remove acid yellow, acid red and acid blue dyes GAC 1 is preferable, and for acid orange dye GAC 3 is desirable.

#### 5.1. Effect of various parameters on the prepared activated carbon

The various parameters including concentration, temperature, pH is taken into consideration in order to determine the various removal efficiency of dyes.

#### 5.2. Effect of adsorbate concentration

The adsorption study was conducted for the concentration of 100 mg/l in Acid red, Acid black, Acid blue, Acid yellow and Acid orange dyes by keeping other parameters are set as constant (pH=7, room temperature). The removal efficiency of various dyes using GAC with respect to time is testified in the following Figures.

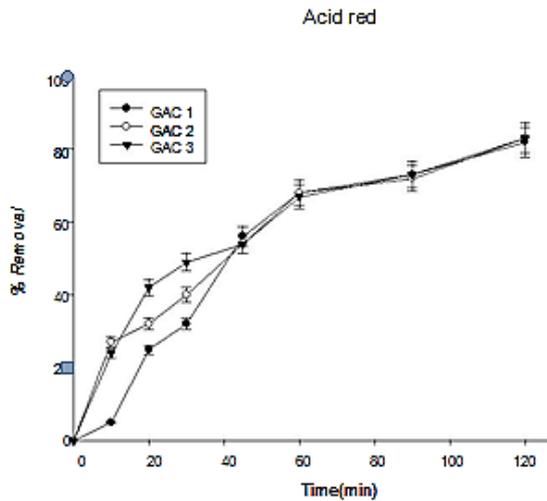


Figure 4. Effect of acid red concentrations in removal efficiency

From Figure 4, GAC 1, GAC 2, GAC 3 linear increase in removal efficiency with regard to increasing in time. The GAC 1, GAC 2, and GAC 3 reach a maximum removal efficiency of 82 %. From this, it is stated that the activated carbon made from the three methods is effective in acid red removal.

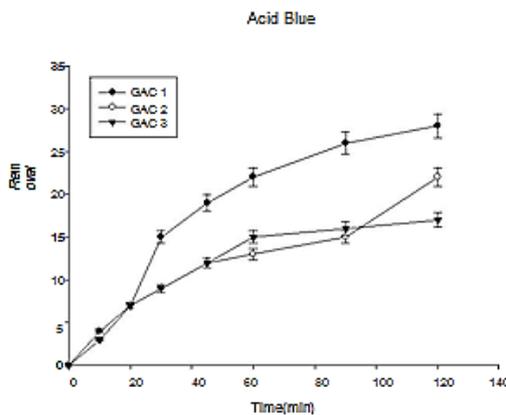


Figure 5. Effect of acid blue concentrations in removal efficiency

From Figure 5, it is clear that for removal of acid blue dye GAC 1 is fitted, it shows a linear increase in removal efficiency than GAC 2 and GAC 3, GAC 1 attained maximum removal efficiency. It is 1.27 times greater than GAC 2 and 1.75 times greater than GAC 3. Hence, for removal of acid blue dye GAC 1 is suited.

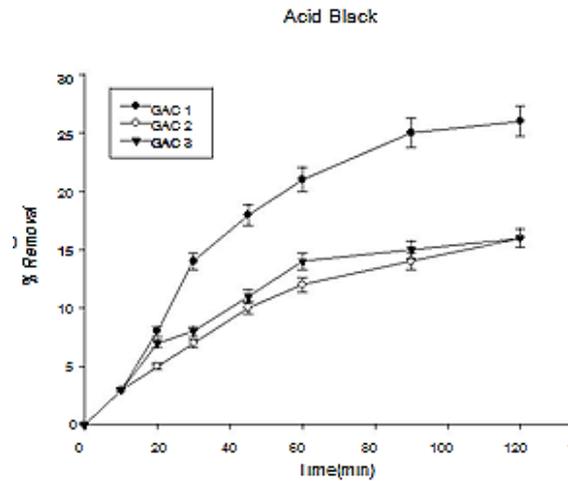


Figure 6. Effect of acid black concentrations in removal efficiency

From Figure 6, it shows that the removal efficiency of acid black dye is more in GAC 1 which is 10 % greater than GAC 2 and GAC 3 respectively. Hence GAC 1 is suited to remove acid black dyes.

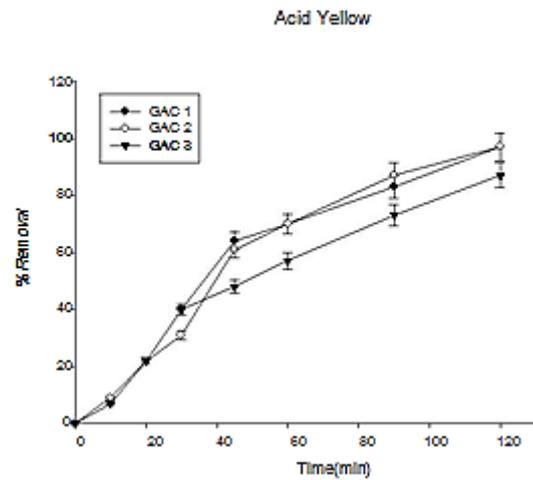


Figure 7. Effect of acid yellow concentrations in removal efficiency

Figure 7 shows that the removal efficiency of acid yellow dye is high in GAC 1 as 97 %, which is 1.1 times greater than GAC 2 and GAC 3. Hence, for removal of acid yellow dye, GAC 1 is suitable than GAC 2 and GAC 3.

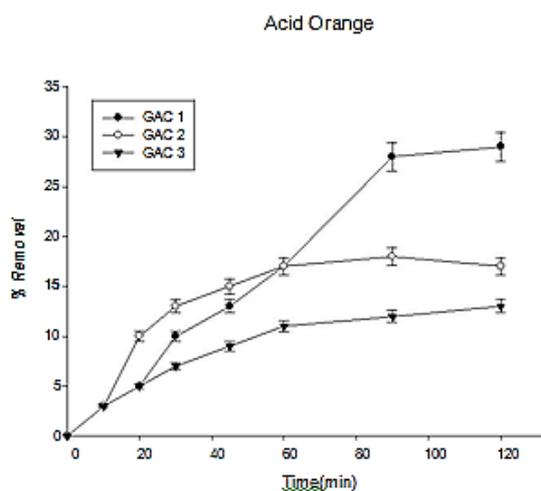


Figure 8. Effect of acid orange concentrations in removal efficiency

Figure 8, shows that the removal efficiency of acid orange dye is greater in GAC 1, which is 1.71 times greater than GAC 2 and 2.42 times higher than GAC 3. As a result, GAC 1 is highly preferable to remove acid orange dyes. It is evident from the Figures 4,5,6,7 and 8 that the absorbance is superior in the Acid Red and Acid yellow dyes compared to that of the acid black, acid blue and acid orange. GAC 1 shows greater removal efficiency for all the dyes. GAC 1 shows better performance in removal efficiency as 96 % and 82 % in acid yellow and acid red dyes respectively at the time interval of 120 min.

### 5.3. Effect of temperature

The important factor takes place in adsorption process is temperature. The research is done to find the influence of temperature in prepared activated carbon and also in adsorption of dyes from water. The experimental work is extended out under various temperatures from 25 °C to 40 °C in the interval of 5 °C and the percentage of dye removal is analyzed for acid red, acid black, acid blue, acid yellow and acid orange dyes.

The Figure A1, states that the granular activated carbon prepared by the combined method using zinc chloride is more effective and the amount of removal efficiency as 99 %, which reach maximum at 120 minutes at room temperature for acid red dye.

The Figure A2, shows that the granular activated carbon prepared by the combined method using zinc chloride is more effective and

the amount of removal efficiency is high at 120 minutes at room temperature for acid blue dye. Next, to that of the combined method using zinc chloride, sodium method shows the better result at 35 °C at 90 minutes.

From the Figure A3, it is stated that the amount of removal efficiency is high when the granular activated carbon prepared by the combined method using zinc chloride is used. The efficiency reached the optimum condition in acid black dye when it is in the room temperature at 120 minutes time interval.

The Figure A4, states that when we use the combined method using zinc chloride in acid yellow dye, then the percentage of removal efficiency reaches to the maximum in 120 minutes time interval at 30 °C.

The Figure A5 explains that in the acid orange dye the greater removal efficiency was achieved at 40°C at the time interval of 120 minutes for the combined method using zinc chloride. The experimental study on temperature reveals that the increase in temperature results in decreasing of removal efficiency and moreover for many dyes combined method using zinc chloride gives optimum efficiency

## 6. EFFECT OF pH

Adsorption process is highly influenced by the pH medium. The removal efficiency for various dyes was carried out using granular activated carbon at a pH range of 3, 7, and 9. The results were plotted in graphs and shown in the following Figures 9-13.

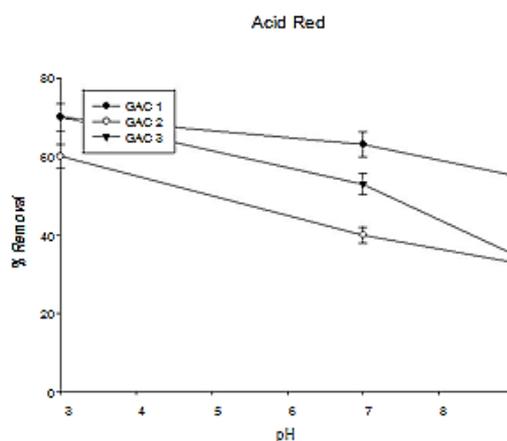


Figure 9. Effect of pH in acid red dye

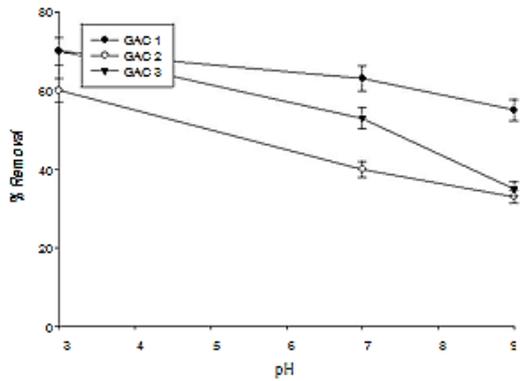


Figure 10.Effect of pH in acid blue dye

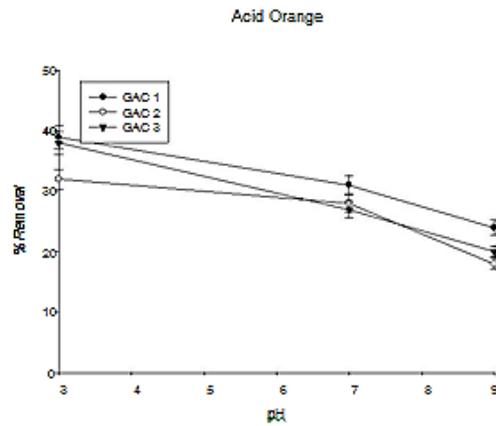


Figure 13.Effect of pH in acid orange dye

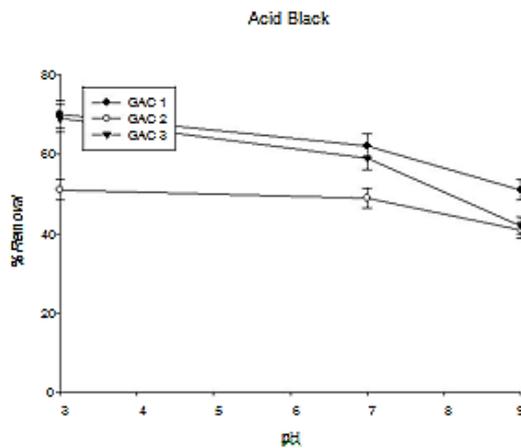


Figure 11.Effect of pH in Acid black Dye

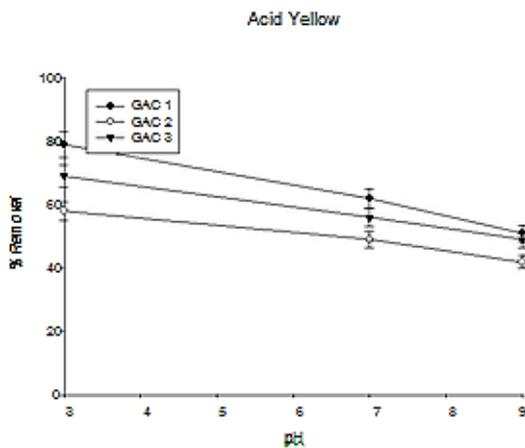


Figure 12.Effect of pH in acid yellow dye

From the Figures, it is observed that the GAC 1 has more removal efficiency than GAC 2 and GAC 3 respectively at pH 3. In GAC 1, acid yellow dye removes in greater efficiency as 80 %. Next to acid yellow, acid red and acid black show better efficiency as 70 %. While, acid blue and acid orange show least efficiency as 40 %. From this experimental study, it is clear that for acid red, acid blue, acid black, acid yellow and acid orange dyes the greater removal efficiency was found at low pH. When the pH increases the removal efficiency started to thin out, the same tendency is noted in all the dyes. And the optimum efficiency was attained when the combined method using zinc chloride was applied.

### 6.1. Optimum efficiency of GAC prepared

The prepared granular activated carbon is said to be effective and the optimum efficiency is attained in the acid yellow dye for the different parameters like pH, temperature and concentration, and also synergistic chemical and physical activation using zinc chloride gives a better result compared to that of the sol-gel method of tio<sub>2</sub> coating and modified sodium method. The Figure 14 shows the optimum efficiency for the acid yellow dye.

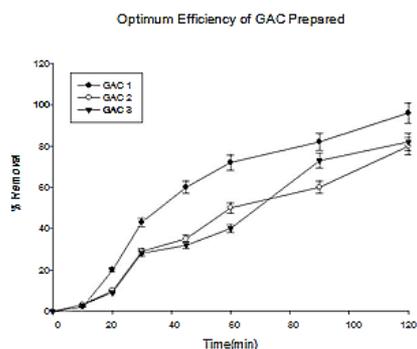


Figure 14. Optimum efficiency for GAC

## 7. CONCLUSION

In this research work, the effect of prepared granular activated carbon is investigated. As a result, the synergistic physical and chemical activation using zinc chloride gives better result on dye removal when compared to other methods. In this research work, the GAC of size 1-2 mm shows better performance than GAC of size 2-4 mm, from this it is clear that smaller particles of activated carbon involve greater adsorption. In the GAC of size 1-2 mm, the acid yellow, acid red and acid blue dye shows greater removal efficiency in GAC 1. In the case of acid orange dye GAC 3 shows better results. Various parameters including concentration, temperature, and pH are analyzed. The analysis reveals that the removal efficiency of acid yellow dye is greater than other dyes. It shows removal efficiency as 97 % on the effect of concentration and 80 % efficiency at pH 3. But in effect on temperature, acid red dye shows the efficiency of 98 %, which is greater than acid yellow dye. From this experimental study, it is stated that the activated carbon prepared from sewage sludge is a promising adsorbent for the removal of dyes from wastewater.

## REFERENCES

- [1] I.Abe, T.Fukuhara, S.Iwasaki, K.Yasuda, Y.Iwata, H.Kominami and Y.Kera, Development of a High-Density Carbonaceous Adsorbent from Compressed Wood Carbon, Vol. 39, No. 10, 2001, pp. 1485-1490, [https://doi.org/10.1016/S0008-6223\(00\)00273-6](https://doi.org/10.1016/S0008-6223(00)00273-6).
- [2] M.P.Islam and T.Morimoto, Thermodynamic Performances of a Solar Driven Adsorption System. Solar Energy, Vol. 139, 2016, pp. 266–277, <https://doi.org/10.1016/j.solener.2016.09.003>.
- [3] P.M.Diaz, A Study on Anaerobic Co-digestion of Sewage Sludge for Bio-gas Production, DJ International Journal of Advances in Microbiology and Microbiological Research, Vol. 2, No. 1, 2017, pp. 1-12, <https://dx.doi.org/10.18831/djmicro.org/2017011001>.
- [4] D.Angin, Utilisation of Activated Carbon Produced from Fruit Juice Industry Solid Waste for the Adsorption of Yellow 18 from Aqueous Solutions Bioresource Technology, Vol. 168, 2014, pp. 259-266, <https://doi.org/10.1016/j.biortech.2014.02.100>.
- [5] D.Mohan, V.K.Gupta, S.K.Srivastava and S.Chander, Kinetics of Mercury Adsorption from Wastewater using Activated Carbon Derived from Fertilizer Waste, Physicochemical and Engineering Aspects, Vol. 177, No. 2-3, 2001, pp. 169-181, [https://doi.org/10.1016/S0927-7757\(00\)00669-5](https://doi.org/10.1016/S0927-7757(00)00669-5).
- [6] A.M.Ahmed, Yassin A Aggor, M.A.Darweesh and Mahmoud I Noureldeen, Removal of Ferrous Ions from City Water by Activated Carbon prepared from Palm fronds, DJ Journal of Engineering Chemistry and Fuel, Vol. 3, No. 1, 2018, pp. 46-63, <https://dx.doi.org/10.18831/djchem.org/2018011005>.
- [7] J.Torres-Perez, C.Gerente and Y.Andres, Sustainable Activated Carbons from Agricultural Residues Dedicated to Antibiotic Removal by Adsorption, Vol. 20, No. 3, 2012, pp. 524–529, [https://doi.org/10.1016/S1004-9541\(11\)60214-0](https://doi.org/10.1016/S1004-9541(11)60214-0).
- [8] N.Pourreza, M.R.Fathi and A.Hatami, Indirect Cloud Point Extraction and Spectrophotometric Determination of

- Nitrite in Water and Meat Products, Microchemical Journal, Vol. 104, 2012, pp. 22–25, <https://doi.org/10.1016/j.microc.2012.03.026>.
- [9] J.H.Ko, J.Wang and Q.Xu, Characterization of Particulate Matter Formed During Sewage Sludge Pyrolysis. Fuel, Vol. 224, 2018, pp. 210–218, <https://doi.org/10.1016/j.fuel.2018.02.189>.
- [10] G.Kalaivani and L.Lakshmi, Kinetic and Isotherm Studies of Dyes Removal from Aqueous Solution by Adsorption on Low Cost Arachis Hypogea (Groundnut Shell), DJ Journal of Engineering Chemistry and Fuel, Vol. 2, No. 2, 2017, pp. 36-47, <https://dx.doi.org/10.18831/djchem.org/2017021004>.
- [11] A.Maleki, M.Mohammad, Z.Emdadi N.Asim, M.Azizi and J.Safaei, Adsorbent Materials Based on a Geopolymer Paste for Dye Removal from Aqueous Solutions. Arabian Journal of Chemistry, 2018, <https://doi.org/10.1016/j.arabjc.2018.08.011>.

APPENDIX

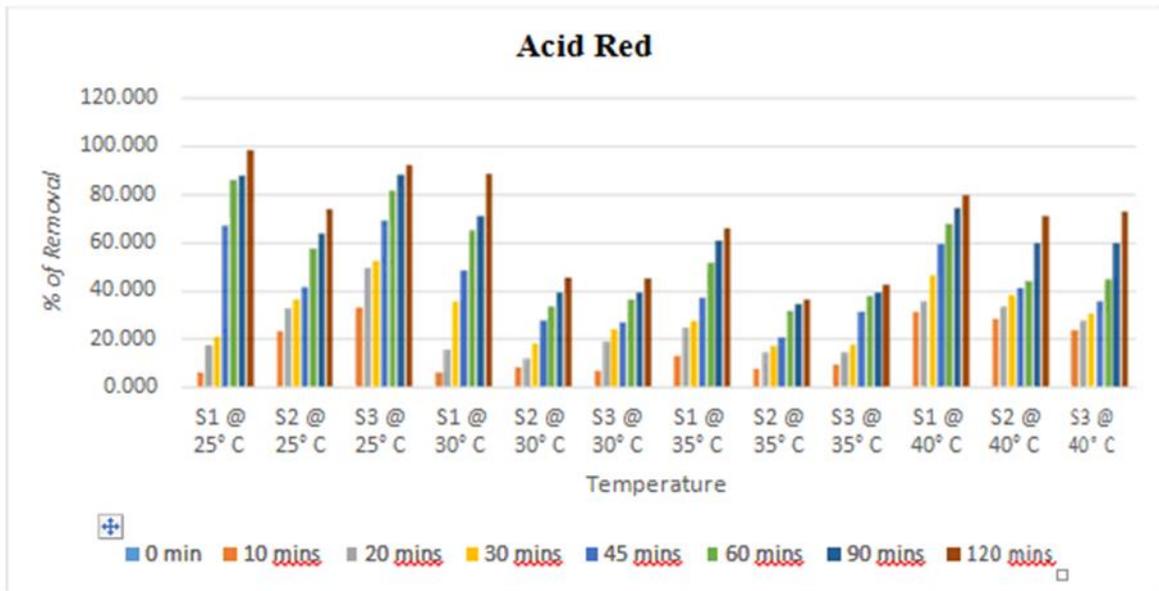


Figure A1. Effect of temperature in acid red dye

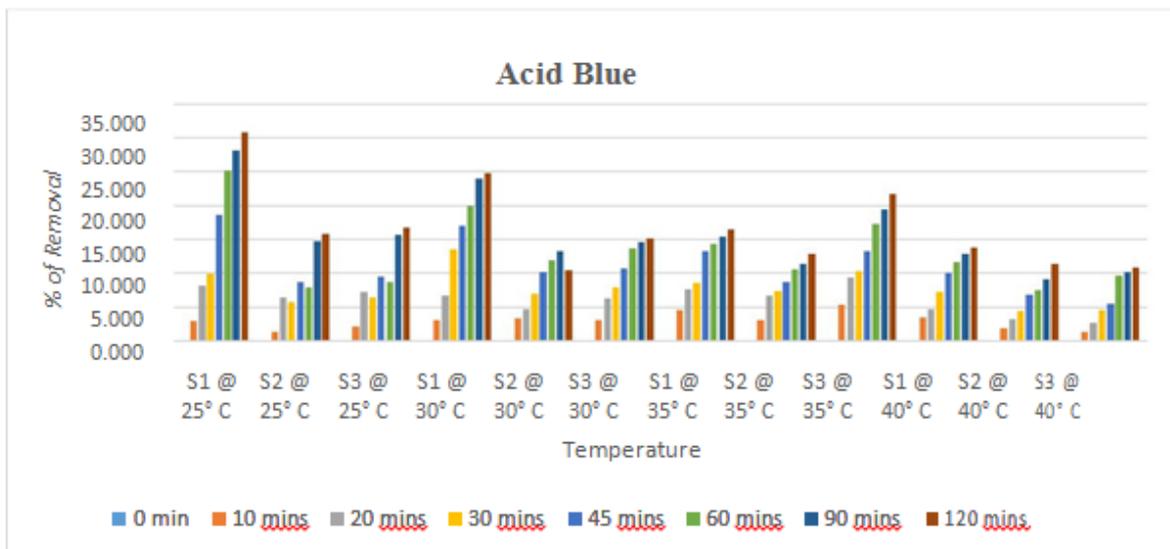


Figure A2. Effect of temperature in acid blue dye

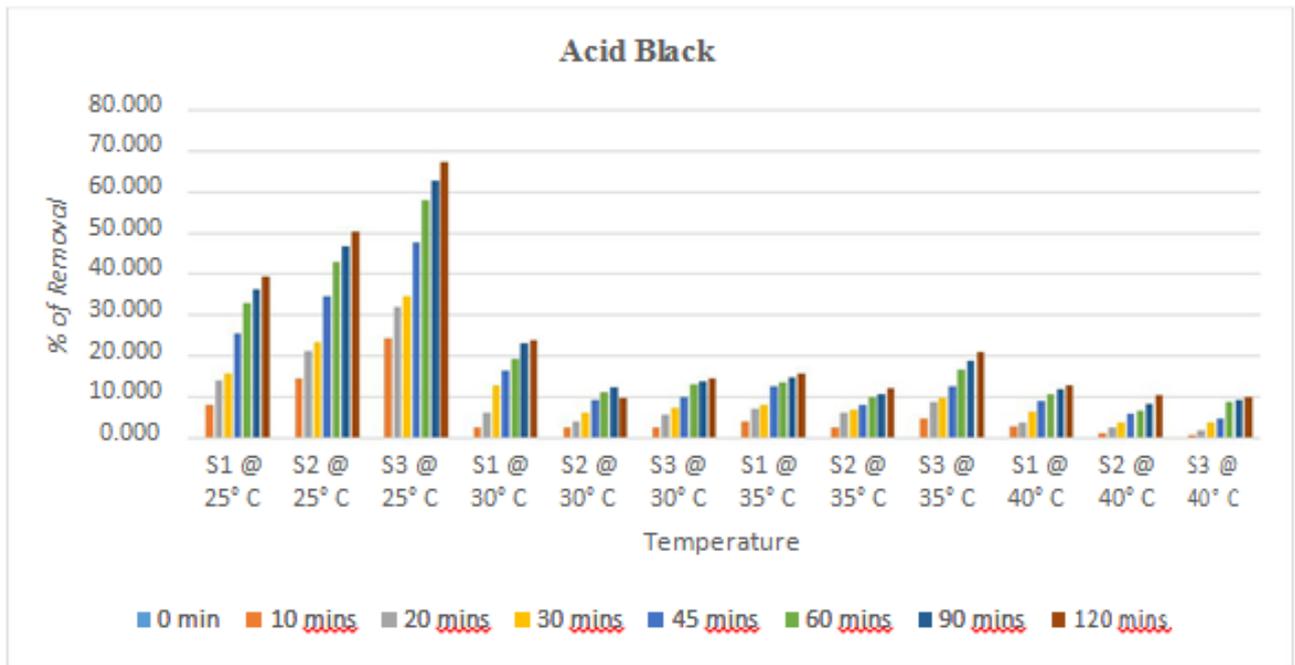


Figure A3.Effect of temperature in acid black dye

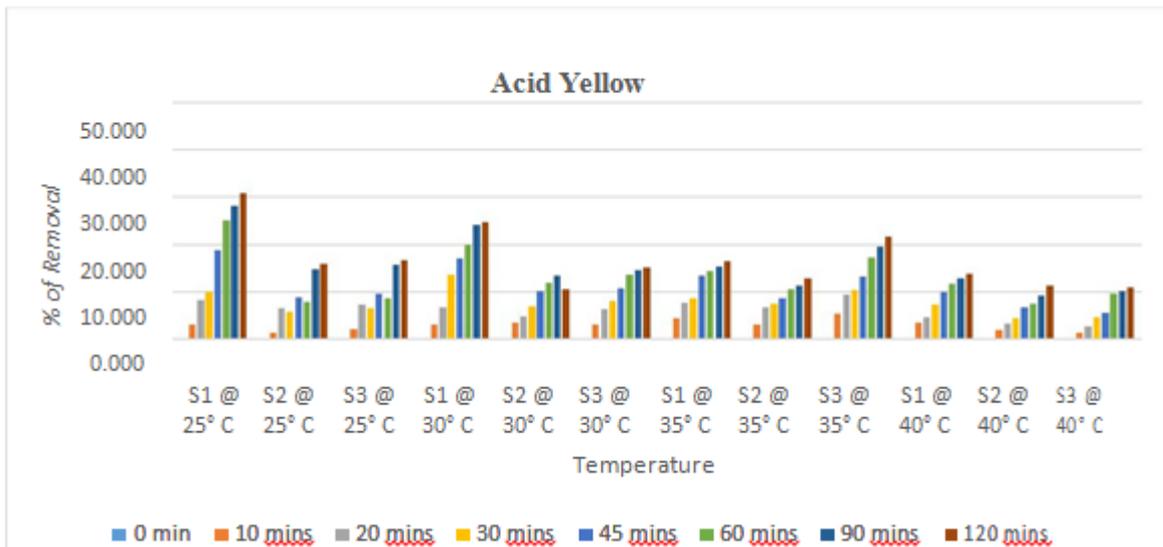


Figure A4.Effect of temperature in acid yellow dye

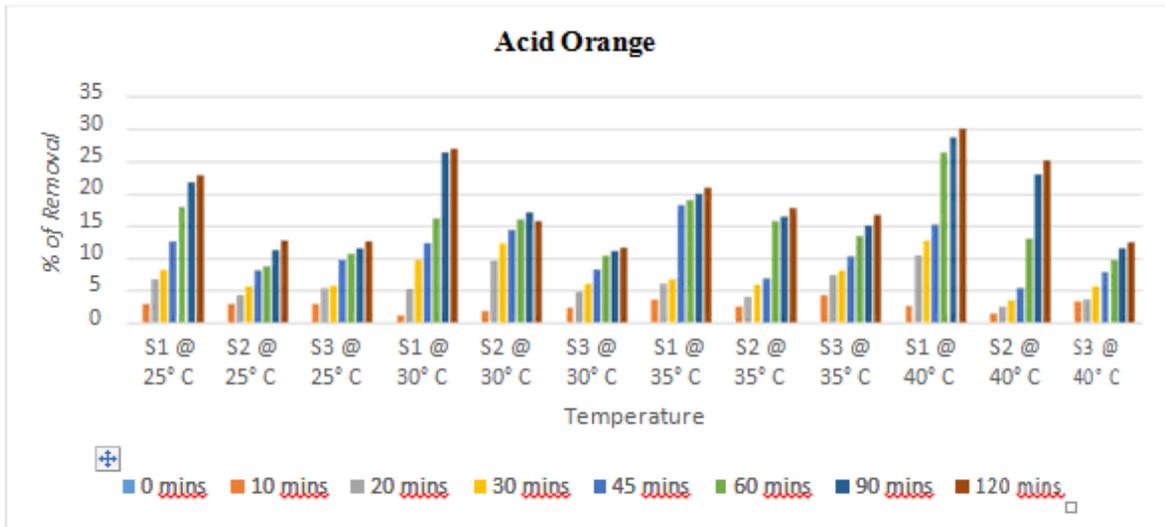


Figure A5.Effect of temperature in acid orange dye