

Studies on Preparation and Characterization of Solid Adsorbent for Removal of Pollutants from Spent Wash

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Abstract- This work focuses on the study on preparation and characterization of solid adsorbent. The present study investigates the methods of production of adsorbent (physical adsorption, chemical adsorption), characterization techniques (SEM, BET analysis), biological oxygen demand (BOD), chemical oxygen demand (COD), measurement of sulphur concentration, pH measurement. An experiment was carried out on a lab scale with sugar industry distillery spent wash with a variation of parameters like pH, time, temperature, mass. The results indicate that the prepared adsorbent from coconut shells gives better results for sulphur removal than other separation techniques.

Keywords- Distillery spent wash, coconut shell adsorbent.

I. INTRODUCTION

Distillery spent wash is a potential source of water pollution as it contains high content of organic & volatile matter. The characteristics of distillery spent wash depend on the quality of the feed stocks and the various aspects of the ethanol production process. The spent wash is generally dark brown liquid with an unbearable odor. It is acidic in nature with high biological oxygen demand (BOD), chemical oxygen demand (COD) and high concentration of mineral salts [1, 2]. Although it does not contain toxic substances, its discharge without proper treatment will impart color and lead to the depletion of dissolved oxygen of the receiving water stream [3].

In a developing country like India, distillery industries have become a major source of pollution, as 88% of its raw materials are converted into waste and discharged into the water bodies, causing water pollution[4]. A number of clean up technologies have been put into practice. It can be achieved by potential microbial (anaerobic and aerobic) as well as physicochemical processes such as coagulation, oxidation, adsorption, and membrane processes. In general most of these techniques are not suitable for treatment of spent wash due to their heterogeneous nature. Among these processes adsorption is a natural process. Activated carbons are carbonaceous materials that can be distinguished from elemental carbon by the oxidation of the carbon atoms found on the outer and inner surfaces [5]. These materials are characterized by their

extraordinary large specific surface areas, well-developed porosity and tunable surface-containing functional groups[6]. For these reasons, activated carbons are widely used as adsorbents for the removal of organic chemicals and metal ions of environmental or economic concern from air, gases, potable water and wastewater. [7]. Recently efforts have been made to use cheap and available agricultural wastes such as coconut shell, orange peel, rice husk, peanut husk and sawdust as adsorbents 3-5 to remove heavy metals from wastewater [8].

For this research activated carbon made from coconut shells is used as adsorbent. Adsorption capacity of adsorbent has been tested and compared for the removal of various compounds and heavy metals from distillery spent wash. Adsorption was brought to lab scale conditions and changes in pH, color, COD, BOD and sulphur concentrations of effluent using batch and continuous experimental methods. The prepared activated carbon is characterized by ash content, moisture content, surface area a (SEM, FTIR, BET analysis) and pore density. Treatment is done using this carbon and treated spent wash is analyzed by using pH meter, turbidity meter, and decolourization which gives values of pollutants quantitatively and qualitatively.

II. MATERIALS AND METHODS

Materials: Scanning electron microscope, fourier transformer, quantachrome analyzer, conical flasks, pH meter, incubator, burette, turbidity meter, glass bottles with stopper, muffle furnace, crucible

Chemicals: Sodium acetate, potassium permanganate, distilled water, glacial acetic acid, magnesium chloride, barium chloride, distillery spent wash from sugar industry from warananagar.

Experimental methods:

Production of activated carbon: The experimental work was divided into two major parts: (i) production of powder and granular activated carbon using coconut shell and characterization of manufactured activated carbon, and (ii)

treatment of brewery wastewater effluent using the produced activated carbon.

A. Characterization of manufactured activated carbon-

To measure the effectiveness of activated carbon produced based on its applications. The following methods are adopted such as percent ash, bulk density and moisture content. Surface area is characterized by FTIR, SEM, and BET analysis methods.

1. Bulk density: The bulk density of the prepared activated carbon is obtained by measuring the volume of distilled water displaced by a known mass of the experimentally produced activated carbon using measuring cylinder.

2. Ash content : Weighed oven dried samples are placed in a crucible and transferred into the muffle furnace at 900°C for about 3 hours, till no further weight loss is detectable. The crucible plus content is then weighed to obtain the weight of the ash. It is expressed as % of the oven dry weight.

3. Moisture content: About one gram of the powdered air dried activated carbon powder was taken in a previously weighed crucible. The crucible was placed in an electric hot air oven maintained at about 110 °C. After one hour the crucible is taken out, cooled in desiccators and weighed again. The loss in weight of the powder reported on percentage basis gives moisture content in the sample.

4. Scanning Electron Microscopy: The surface morphology of the activated carbon samples are studied by scanning electron microscopy.

5. Surface area and porosity: The BET surface area of activated carbon powder is determined by nitrogen adsorption desorption method. The instrument used is quantachrome. Nitrogen gas was allowed to adsorb on the powder at 77 K. The nitrogen adsorption and desorption isotherms are used to determine parameters like surface area, pore volume and pore size.

III.EXPERIMENTAL WORK

Experiment consists of adsorption carried out with different adsorbent dosage and contact time with the three different dilutions of sample as shown in Table 1. Adsorbent dosage of 1g, 2.5g, and 5g with the corresponding contact time of 6hr, 12hr and 24hr for each sample was used with each dilution. Each diluted sample of 600ml quantity is divided into three parts of 200ml quantity each. There are total nine samples three from each diluted sample of 100ml quantity each. Each concentration each sample is taken in a volumetric flask of 250ml and added with a desired adsorbent dose. Each flask was shaken for one hour and the samples was filtered and collected for analysis after desired contact time. Whole adsorption test was performed at room temperature.

IV.RESULTS AND DISCUSSION

Characterization results:

Bulk density is an important parameter of powdered solids. The American Water Work Association has set a lower limit on bulk density at 0.25 gm/ml for activated carbon to be of practical use. The bulk density values of the prepared are given in table 1 of prepared samples satisfies this condition.

The moisture content values of the prepared activated carbon samples are reported in table 1. The commercially available samples contain about 5-8% moisture.

From table 1, it can be seen that the ash content in activated carbon samples is low. The low ash content makes activated carbon an attractive candidate for adsorption studies. The ash content of commercial samples is around 2% . If ash content is high it will interfere with pore structure development and hence adsorption will be less.

The above results show that surface area of activated carbons is in agreement with other studies. Average pore volume of 0.4 cm³ /g suggests good pore structure development. The BET surface area increases with increase in temperature of activation, soaking time and porosity as well. Increase in surface area results in an increase in chromium adsorption. This can be explained as due to the increases in the number of adsorption sites.

Figures 1 and 2 shows that SEM micrographs of the sample before and after. The porosity is less in case of figure 2 compared to that in case of figure 1.

Table no. 1 Showing various characteristics parameters results-

| Sr. No | Sample no. | Bulk Density gm/cc | % Moisture content | % Ash content | BET surface area m ² /g | Total pore volume Cc/g | Average size A° |
|--------|------------|--------------------|--------------------|---------------|------------------------------------|------------------------|-----------------|
| 1 | 1 | 0.2630 | 5.10 | 1.52 | 590 | 0.36631 | 15.52 |
| 2 | 2 | 0.2589 | 5.28 | 1.42 | 650 | 0.39652 | 16.22 |
| 3 | 3 | 0.2625 | 5.15 | 1.34 | 690 | 0.40854 | 18.82 |

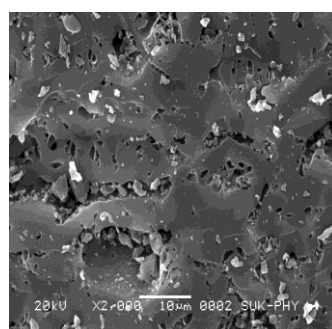


Fig. 1 SEM micrograph before adsorption

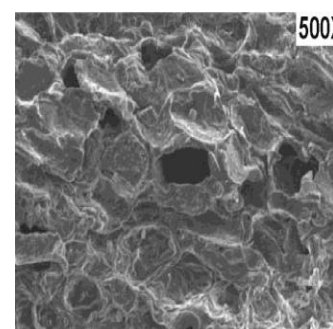


Fig.2. SEM micrograph after adsorption

Analysis of Spent Wash before Adsorption

Following are the parameters in a tabular form (Table 2) which were analyzed initially i.e. before adsorption for the three dilutions of 25%, 50% and 100% pure sample and with different adsorbent dose and contact time.

Table no. 2: Showing parameter analyzed before adsorption.

| Sr no. | Parameters | For 25% sample dilution | For 50% sample dilution | For 100% sample dilution |
|--------|------------|-------------------------|-------------------------|--------------------------|
| 1. | pH | 5.3 | 5.74 | 4.3 |
| 2. | Sulphate | 165.4 | 210.11 | 264.8 |

Graphs after Adsorption

The following graphs will give better illustration of the results obtained following the analysis of spent wash. Here the results obtained are graphically represented for various wastewater quality parameters.

For 25% dilution-

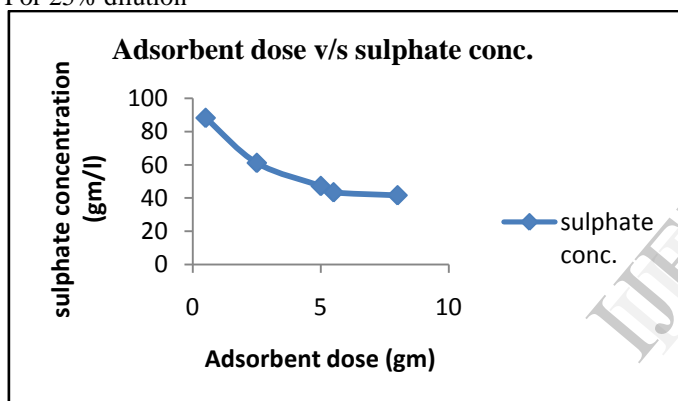


Fig.3 Variation of sulphate conc. with adsorbent dose

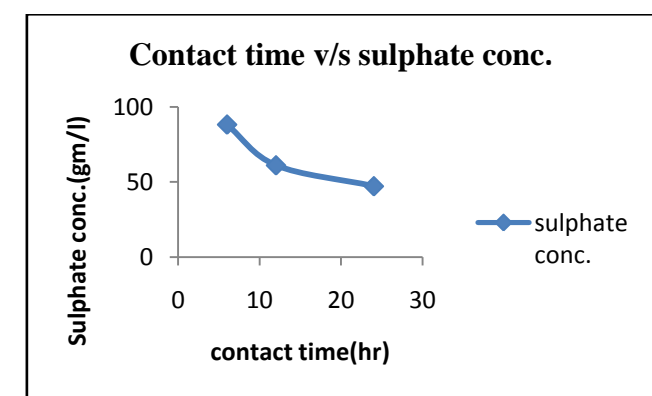


Fig. 4 Variation of sulphate conc. With contact time

In Figure 3 and 4 sulphate concentration decreases with increase in adsorbent dose up to 5gm and contact time of 24hr with increase in adsorbent dose and contact time. The maximum percent removal 76.23% has been observed at an adsorbent dose of 5gm/100ml, contact time of 24hr with 25%

diluted sample. The result can also be summarized in. table no.3

pH graphs-

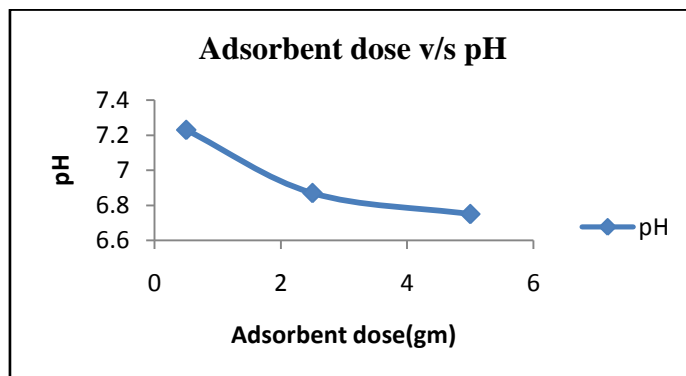


Fig. 5 Variation of pH with adsorbent dose

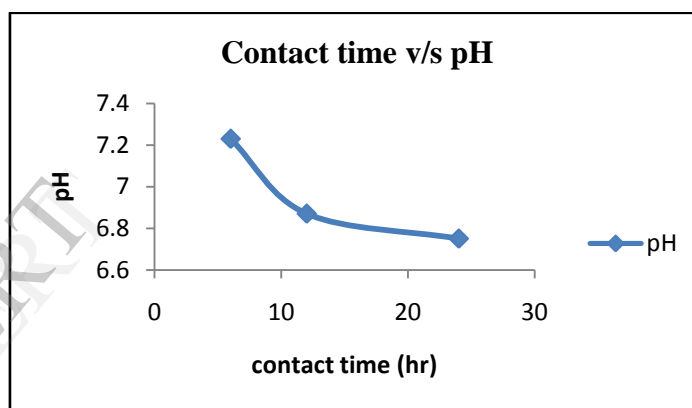


Fig. 6 Variation of pH with contact time

Fig. 5 and 6 shows variation of pH with adsorbent dose and contact time. Graphs shows that pH increases with increase in adsorbent dose up to 0.5 gm and contact time of 6hr and then decreases with increase in adsorbent dose and contact time. The maximum percent removal 25.31% has been observed at an adsorbent dose of 0.5gm/100ml and contact time of 6hr with 25% diluted sample.

Table no. 3 Results analyzed from graphs for 25% sample dilution after adsorption

| Parameter s | For 25% conc. dilution | | | | | | |
|---------------|------------------------|-------------------|-------|-------|-------------------|-------|-------|
| | Initial conc. | Final conc. After | | | Removal (%) after | | |
| | | 6 hr | 12 hr | 24 hr | 6 hr | 12 hr | 24 hr |
| Sulfate conc. | 198.6 | 88.2 | 61.1 | 47.2 | 55.58 | 69.23 | 76.23 |
| pH | 5.4 | 7.23 | 6.87 | 6.75 | 25.31 | 21.39 | 0.20 |

For 50% dilution-

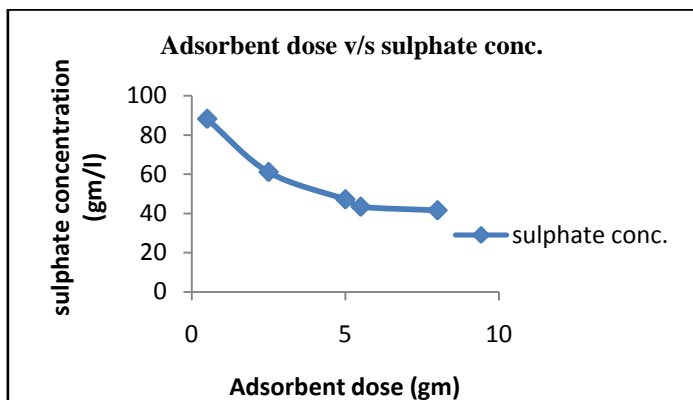


Fig. 7 Variation of sulphate conc. With adsorbent dose

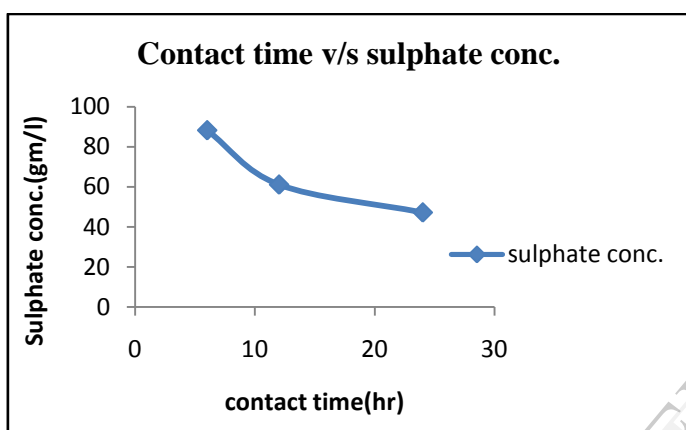


Fig. 8 Variation of sulphate conc. With contact time

In fig. 7 and 8 sulfate concentration decreases up to 39.2(mg/l) with increase in adsorbent dose up to 5gm and contact time of 24hr. The maximum percent removal 70.54% has been observed at an adsorbent dose of 5gm/100ml, contact time of 24 hr with 50% diluted sample.

pH graphs -

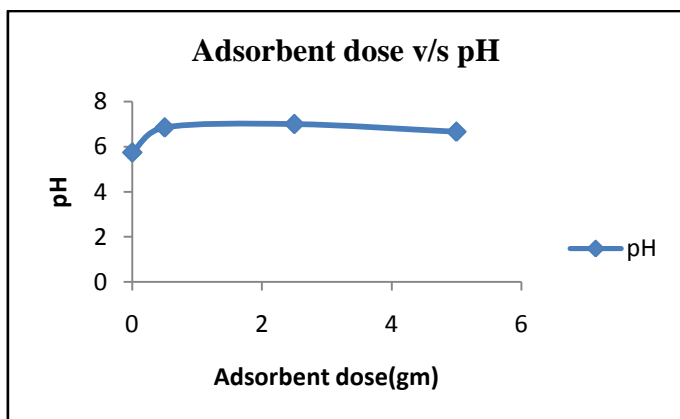


Fig. 9 Variation of pH with adsorbent dose

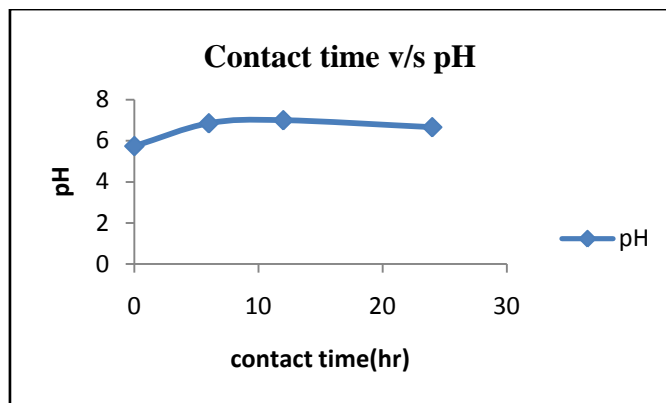


Fig. 10 Variation of pH with contact time

In fig. 9 and 10 pH increases with increase in adsorbent dose and contact time of 12hr and then decreases with increase in adsorbent dose and contact time. The maximum percent removal 18% has been observed at an adsorbent dose of 2.5gm/100ml, contact time of 12hr with 50% diluted sample

Table no. 4 Results analyzed from graphs for 50% sample dilution after adsorption

| Parameters | For 50% solution(mg/l) | | | | | | |
|----------------------------|------------------------|-------------------|-------|-------|------------------|-------|-------|
| | Initial conc | Final conc. after | | | Removal(%) after | | |
| | | 6 hr | 12 hr | 24 hr | 6 hr | 12 hr | 24 hr |
| Sulfate conc.(mg/l) | 132.4 | 99.2 | 60.32 | 39.2 | 25.07 | 54.59 | 70.54 |
| pH | 5.74 | 6.85 | 7.0 | 6.66 | 16.20 | 18 | 13.81 |

For 100% dilution

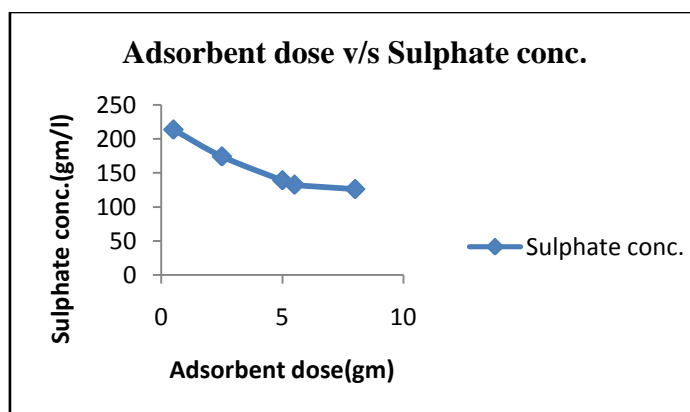


Fig. 11 Variation of sulphate conc. With adsorbent dose

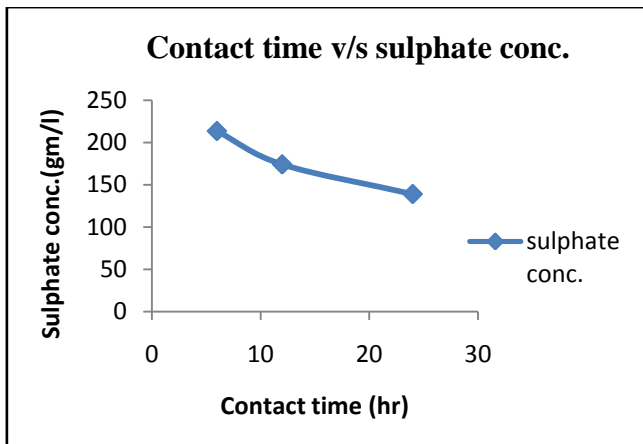


Fig. 12 Variation of sulphate conc. With contact time

In fig. 11 and 12 Sulfate concentration decreases with increase in adsorbent dose up to 5gm and contact time of 24hr. The maximum percent removal 47.46% has been observed at an adsorbent dose of 5gm/100ml, contact time of 24hr with 100% sample.

pH graph-

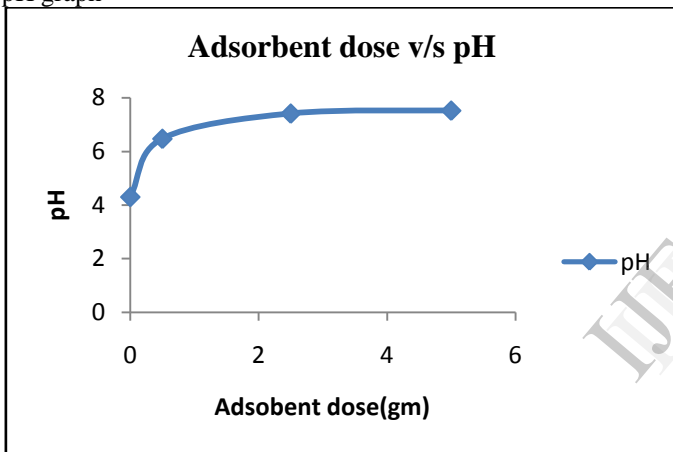


Fig. 13 variation of pH With adsorbent dose

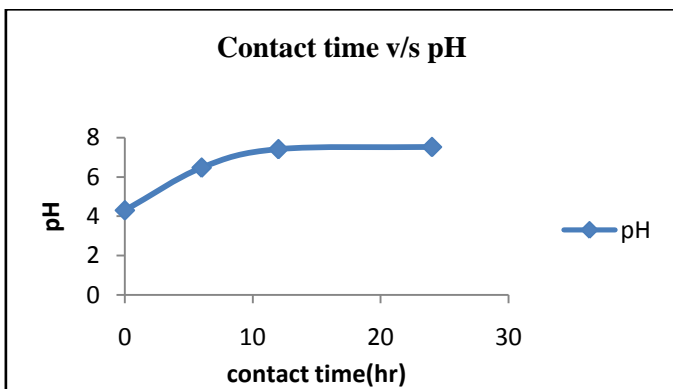


Fig.14 variation of pH With contact time

In fig. 13 and 14 pH increases with increase in adsorbent dose and contact time. The maximum percent removal 42.9% has been observed at an adsorbent dose of 5gm/100ml and contact time of 24 hr with 100% sample.

Table no. 5 Results analyzed from graphs for 100% sample dilution after adsorption

| Parameter | For 100% dilution(mg/l) | | | | | | |
|---------------------|-------------------------|-------------------|-------|-------|------------------|-------|-------|
| | Initial conc. | Final conc. after | | | Removal(%) after | | |
| | | 6 hr | 12 hr | 24 hr | 6 hr | 12 hr | 24 hr |
| Sulfate conc.(mg/l) | 264.67 | 213.67 | 174.2 | 139.1 | 19.30 | 34.21 | 47.46 |
| pH | 4.3 | 6.47 | 7.41 | 7.52 | 33.69 | 42.10 | 42.9 |

Table no. 6 Final results for Maximum removal for particular parameters

| Pollutants | Sample dilution | Adsorbent dose | Contact time | % removal |
|--------------|-----------------|----------------|--------------|-----------|
| Sulfate conc | 25% | 5mg/l | 24 hr | 76.23% |
| pH | 100% | 5mg/l | 24 hr | 42.9% |

V. CONCLUSIONS

1. The activated carbon is found to be effective adsorbent.
2. It has been observed that adsorbent dose of 5 gram and contact time of 24hr for 100ml of sample is found to be most effective for different dilutions for removal of most pollutants.
3. On increasing the adsorbent dose and contact time after this limit there is not much effect on the removal of pollutants.
4. This is because of the adsorptive capacity of the adsorbent is reached to an end.
5. The maximum removal (42.9%) of pH has been observed at 5gm/100ml of adsorbent dose and a contact time of 24 hr for 100% pure sample.
6. The maximum removal (76.23%) of sulfate has been observed at 5gm/100ml of adsorbent dose and a contact time of 24 hr for 25% diluted sample.
7. It is also found that there is a certain limit for increasing the adsorbent doses for given amount of effluent. This has to be carefully controlled. An excess use can leads to uneconomical process. The initial concentration plays an important role throughout the adsorption.
8. Regeneration and disposal depends on the amount and the availability of the adsorbent.

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