Industrial Wastewater Sludge-Based Activated Carbon: Synthesis, Characterisation and Application in Wastewater Treatment

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***Abstract* - A novel activation process was adopted to produce highly porous activated carbon from activated sludge obtained from a common industrial wastewater treatment plant. The activated carbon was successfully used for COD removal from industrial wastewater. The physical properties of activated carbon produced by the activation of 1M Na_2CO_3 solution was investigated. COD removal from aqueous solution by adsorption onto the sludge-based activated carbon was studied under varying conditions of adsorption time and carbon dosage. The Freundlich equilibrium isotherm model fitted the adsorption data well with R^2 = 0.8593. Accordingly, it is concluded that the procedure of developing activated carbon used in this study could be effective and practical for utilizing in COD wastewater treatment.**

***Index Terms* - Activated Carbon, Industrial Wastewater Sludge, Pyrolysis.**

I. INTRODUCTION

The amount of activated sludge produced by treating wastewater has increased as the water treatment industry has developed rapidly [1]. Industrial wastewater sludge (IWWS) is carbonaceous in nature and rich in organic materials. Hence, it has the potential to be converted into activated carbon on pyrolysis under controlled conditions or with some chemical treatment. This conversion can offer the combined benefits of reducing the volume of sludge and producing a valuable adsorbent with lower cost than commercial activated carbon [2]. Conventionally, adsorbent materials from sludge were produced by chemical activation by H_2SO_4 impregnation followed by pyrolysis.

The parent sludges were initially oven dried at 105°C to constant mass and then subjected to chemical activation by impregnating with H_2SO_4. The ensuing activated sludges were then pyrolyzed under inert nitrogen, and subsequently washed with dilute HCl (10% by mass). These adsorbent particles were ground to desired particle sizes of greater porosity and higher surface area [3]. Evita 2013 et al [4] reported adsorption of 85.7% of potassium, 70% of chromium and 30% of arsenic from the leachate by the biochar synthesized from pyrolysis of sludge at 500 ºC and impregnation into K_2CO_3.

If sludge is impregnated in KOH then surface area is lower than that impregnated in K_2CO_3. The maximum surface area of 1352.86 m2/g was obtained at 800 ºC by K_2CO_3 and that of 618.54 m2/g was obtained at 800 ºC by KOH which is not acceptable for the adsorption operation in various chemical industries [5]. Commercial charcoal has iodine number in the range 600-1450 which indicates the capacity of adsorption [6]. When the ratio of the sludge to reagents (KOH, $ZnCl_2$) changes, the BET surface area of the sludge based activated carbon also changes. If the ratio is 1:3, the surface area is 1832 m2/g and at 1:2 ratio, the surface area is 495.75 m2/g at 750 ºC by KOH impregnation [7]. In the present study, 1M Na_2CO_3 solution was used for activation of the carbon.

II. EXPERIMENTAL

A. Production of Activated Carbon

The IWWS samples were used as a raw material for making activated carbon. The IWWS was taken from Enviro Technology Limited (ETL), a common effluent treatment plant in Ankleshwar Industrial Estate, Gujarat (India). Filtration was accustomed to dewater the untreated sludge, as was typical. It was then dried to a constant weight in a vacuum oven at 110°C up to 3 hrs, then compressed and sieved to a uniform size of less than 0.15 mm. The resulted granular particles were impregnated with a 1M Na_2CO_3 solution for 24 hr at room temperature. Sludge was ready to use after removing the liquid from it by heating it to 110ºC up to 4 hrs. The sludge was crushed once more and sieved (100 BSS). For the pyrolysis experiments, procedure was followed as described elsewhere [8] with nitrogen as a purge gas, 100 gm of IWWS is pyrolyzed. The heating temperature rose at a rate of 40 °C/min until it

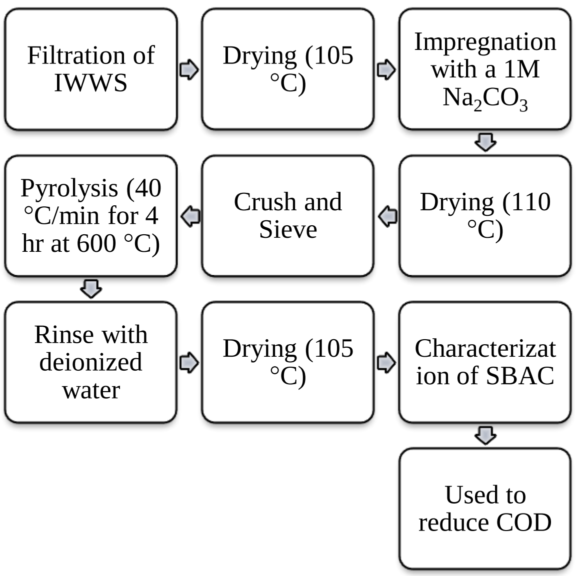

Fig.1: Sequence of operations for making Sludge Based Activated

reached 600 °C. The nitrogen flow was held at 5 LPH during the four- hour dwell period at the final temperature. After pyrolysis, the product was rinsed 10 times with 50 mL deionized water to remove any residual sodium carbonate or inorganic matter, and then dried to a constant weight for use. The sequence of operations to making sludge based activated carbon is shown in Figure No. 1.

B. Characterization of Activated Carbon

To determine the adsorption ability of the developed sludge based activated carbon (SBAC), the iodine number was estimated using the technique developed by the American Society for Testing Materials [6]. Solutions used in the Iodine no. test were KIO_3, HCl, Iodine Solution, Sodium Thiosulphate and Starch Solution.

C. Adsorption Study

COD of wastewater from the ETL plant was used as the adsorbate in this study to determine the applicability of the developed activated carbon as an adsorbent. A typical experiment used the initial adsorbate concentration of 2682 mg/L. In a 200 mL sealed flask, different quantities of SBAC were added, along with 100 mL of COD wastewater. The SBAC was isolated from the solution by filtration after shaking the flasks in an orbital shaker (REMI RS-24 BL) at a rolling speed of 180 rpm at a steady temperature of 38 ± 1 °C for 4 hrs. It was earlier ascertained that equilibrium is attained after 3 hours of shaking. The concentration of COD remaining in solution was determined using the open reflux method. Each adsorption experiment with a different dose of SBAC was carried out three times.

III. RESULTS AND DISCUSSION

A. Characteristics of the Sludge-Based Activated Carbon

The activated carbon was produced by pyrolysis of activated sludge and activating it with a 1M Na_2CO_3 solution. The iodine number of the sludge-based activated carbon was 941.7145 mg/g, which is falls within the range of 500-1200 mg/g [9]. It is equivalent to surface area of carbon between 900 and 1100 m^2/g. Iodine number is an alternative indication of the porosity of activated carbon. The adsorption of aqueous I_2 is considered a simple and quick test for getting an idea of the surface area of activated carbon associated with pores with d > 1 nm [10].

B. Effect of carbon dosage on COD removal efficiency

SBAC dose was varied to adsorb COD from the effluent in adsorption experiments. For every experiment the adsorption time is 4 hr. 2682 mg/L initial COD concentration in solution, the effect of carbon dosage on adsorption efficiency was investigated with SBAC doses ranging from 1 to 5 gm in 0.1 L which is shown in the Table No. I. and Figure No. 2 gives the graphical description.

Table I. Results of experiments for removal of COD by SBAC

Sample	SBAC Weight (gm)	COD (mg/L)		
		Initial	Final	% Removal

S1	1	2682	2203	17.86%
S2	2		2142	20.13%
S3	3		2040	23.94%
S4	4		1960	26.92%
S5	5		1880	29.90%

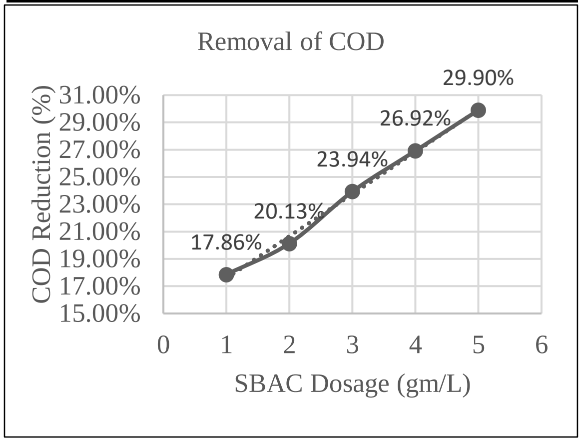

Fig.2 Removal of COD by SBAC.

C. Freundlich Adsorption Isotherm

The amount of adsorbate that can be absorbed by the adsorbent depends on the concentration and temperature of the adsorbate in the adsorption isotherm. Solubility, molecular structure, molecular weight, polarity, and hydrocarbon saturation are some of the characteristics of the adsorbate [11]. The Freundlich isotherm is described as follows:

$$\frac{x}{m} = K_f C_e^{\frac{1}{n}} \qquad \text{……………………………………..(4.1)}$$

Where x/m is the adsorbate mass adsorbed per unit mass of adsorbent, (mg adsorbate/gram activated carbon). K_f is the Freundlich capacity/constant factor, C_e is the equilibrium concentration of the adsorbate in solution after adsorption, (mg/L) and 1/n is Freundlich's intensity parameter. The constant in the Freundlich isotherm can be determined by plotting log (x/m) versus log Ce according to Eq. 4.2.

$$\log\frac{x}{m} = \log K_f + \frac{1}{n}\log C_e \qquad \text{……………………..(4.2)}$$

The equation for determining x/m is shown in Eq. 4.3.

$$\frac{x}{m} = \frac{(Ci - Ce) \times V}{1000 \times m} \qquad \text{………..……………………..(4.3)}$$

The graphical representation of the Freundlich adsorption isotherm is shown in Figure No.3. Freundlich's equation is obtained log (x/m) = 6.2311, log Ce = -19.244 with R^2 = 0.8593 from Table No. II. The constant values of the Freundlich equation are K_f = 5.7016 × 10-20 and n = 0.1608.

Table II. The value of x/m, log (x/m) and log Ce

Weight of SBAC (m) (gm)	Initial COD (C_i) (mg/L)	Final COD (C_e) (mg/L)	x/m (mg/g)	Log(C_e)	Log(x/m)
1	2682	2203	47.9	3.3430	1.6803
2		2142	27	3.3308	1.4313
3		2040	21.4	3.3096	1.3304
4		1960	18.05	3.2922	1.2564
5		1880	16.04	3.2741	1.2052

IV. CONCLUSIONS

Converting IWWS to activated carbon not only eliminates the need for additional sludge treatment, lowers the expense of hauling, landfilling, and transporting sludge, but also produces a valuable adsorbent at a lower cost than industrial activated carbons. A new approach is developed by impregnating sodium carbonate in IWWS and pyrolyzing it in an inert atmosphere. The findings revealed that the activated carbon made from IWWS under the preparation conditions of 600 °C pyrolysis temperature and 4 h heating time was mainly microporous and mesoporous in nature, with an iodine amount of 941.7145 mg/g.

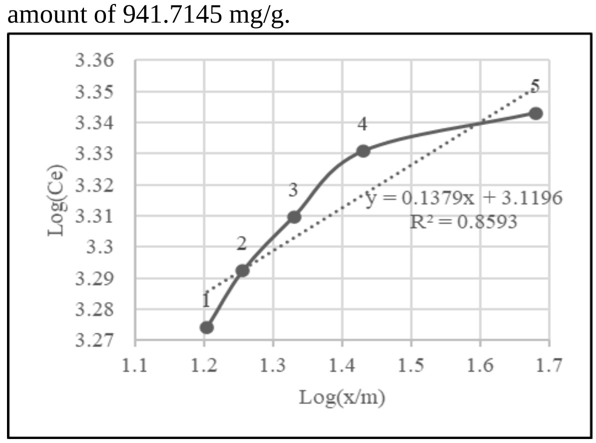

Fig.3 Determination of Parameters of Freundlich Adsorption Isotherm.

Adsorption time and carbon dosage were the most important factors in the removal of COD by adsorption on SBAC. The removal of maximum COD in the effluent with an initial concentration of 2682 mg/L was at 5 g per 100 mL of carbon dosage. The COD removal efficiency remained above 29.90% over the range of initial concentrations examined, suggesting that the emitted carbon had moderate adsorption potential. The data on COD adsorption on the activated carbon created matched the Freundlich equilibrium isotherm model [11].

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