

Preparation of Activated Carbon From Date Seeds And Evaluation of It Application A Review Paper

K. V. Priya^{*}, C. Anitha¹, V. Sakthipriya², S. Saranya³, S. J. Shebatini Seljin⁴

**Assistant Professor, Department Of Civil Engineering, Panimalar Engineering College, Chennai.*

^{1, 2, 3, 4} Student, Department Of Civil Engineering, Panimalar Engineering College, Chennai.

Corresponding Author: K. V. Priya

Abstract: Activated carbon (AC) is frequently used for purification due to its ability to remove pollutants either from air or water. Its porous structure allowed it to capture the pollutants and is widely recommended for numerous applications in water and wastewater treatment. However, AC application fields are restricted due to high cost. In this project, AC prepared from locally available date seeds is used for the removal of Methylene blue (MB) from Textile Water, in order to find alternative to commercial activated carbon (CAC). Physical properties of date seeds activated carbon (DSAC) and CAC were compared and batch adsorption processes were obtained to find the best condition for using DSAC in the removal of MBA. The experimental outcomes found that DSAC got high percentage removal and it has a good potential for economic removal of MB.

Keywords: Activated Carbon, Adsorption Isotherm, Date Seeds, Textile Wastewater, Phosphoric Acid

Date of Submission: 16-02-2018

Date of acceptance: 03-03-2018

I. Introduction

Date seeds are easily available in India especially in Kachch region of Gujarat, which is economically viable material for the production of activated carbon (AC) from date seeds (DS). The date seeds activated carbon (DSAC) is also used as a filtering medium for automobiles exhaust gases and as an adsorbent of toxic organic and inorganic compounds.

Colored wastewater generated from textile dyeing industries is a significant cause of concern in developing countries owing to its potential to escalate environmental pollution. Many complicated processes in textile manufacturing such as washing, dyeing, printing, finishing etc. use an assortment of dyes, which are responsible for producing colored wastewater. Color or dyes have recalcitrant and non-biodegradable characteristics and generally cannot be removed efficiently through conventional treatment processes. If released in the environment, they tend to persist over long distances in flowing water, can affect sunlight penetration through the water column, can retard photosynthesis and reduce the availability of dissolved oxygen in water. Composite textile effluent is characterized by significantly high Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD) and Total Dissolved Solids (TDS) content along with a large number of non-biodegradable organic dyes and other chemicals used in manufacturing. Inefficiencies in dyeing can also result in large amounts of dyestuff being directly lost to the wastewater during textile processing, which ultimately finds its way into the environment.

Different methods of color removal from industrial effluents have been proposed which include aerobic and anaerobic microbial degradation, coagulation, flotation, membrane filtration and chemical oxidation but none of these methods have been found to be effective enough for the industrialists to adopt for the treatment of colored effluents. For example, biological treatment processes remove BOD, COD and suspended solids to some extent, but they have been found to be largely ineffective in removing color from wastewater. Also, coagulation process effectively decolorizes insoluble dyes but fails to work well with soluble ones. Adsorption, on the other hand, has been found to be very efficient in treating wastewater containing chemically stable pollutants is a sludge free process and can completely remove even very minute amount of dyes from waste water.

There could be other regulatory issues deterring efficient removal of color from industrial wastewater in developing countries. For example, in Bangladesh the industrialists are reluctant to invest in additional treatment processes to remove color after applying conventional treatment to reduce BOD, COD, TSS and TDS particularly when the discharge standard for textile effluent does not specify color of effluent as a parameter to be regulated (ECR, 1997). An affordable solution to color removal can encourage these industrialists to adopt such methods and reduce the environmental burden of their effluent wastewater.



Fig.1

II. Experimental

2.1materials And Methods

The DS was collected from locally available dates in India. The MB concentration used was 0.995 g/ml. It has a molecular weight of 319.86 g/mol, density of 0.995 g/cm³, molecular formula is C₁₆H₁₈CIN₃S. The MB and all other chemicals, such as NaOH and HCl, were laboratory grade. The instruments used for DSAC preparation were: muffle furnace (Nabertherm more than heat 30-3000 °C), conductivity meter (Elico, CM183EC-TDS Analyser) and digital magnetic stirrer (WiseStir MS-D-Digital magnetic stirrer). A spectrophotometer (Spectronic BioMate 5 UV-Visible Spectrophotometers) used to find λ_{max} for MB and for absorbance determination. Filter papers were used to remove the AC from the solution before determining the absorbance from the spectrophotometer. The standard test method ASTM-D3838 -80 was used [4] to determine the pH of AC by using pH meter (Eutech instruments, CyberScan pH 300). The samples were tested in duplicates. Determination of moisture content was according to ASTM -D1762 [5]. In determining the ash content, 0.1 g of the AC heated at 500 °C for 4 hours, cooled in a desiccator and weigh.

2.1.1 Preparation Of Ac From Ds

DS are used for the preparation of AC. Many researchers used different methodologies to prepare DSAC. According to [7] research, the following procedure is considered.

2.1.1.1. *BIOMASS* The date palm seeds were washed with deionized water to remove foreign materials and dried in an oven at 105 °C for 24 h. The dried seeds were crushed and sieved using a standard sieve to collect the precursor with the size lower than 2 mm and were stored in a desiccator for further use [7].



Fig.2

2.1.1.2. Carbonization And Activation

Carbonization and activation of sieved DS will be carried out simultaneously. Activation will take place by the activated agent H₃PO₄. 30 g of crushed DS was mixed with H₃PO₄ (60%) at an impregnation ratio (IR) (grams of 100% H₃PO₄/gram of dried precursor) of 3.1[7].



Fig.3

2.2. Calibration Curve

A 1000 mg/L stock solution was prepared by dissolving 1 ml of 0.995 g/ml MB dye concentration in 1 L distilled water. The different concentrations of MB for the entire study were prepared from that stock solution. The calibration curve was prepared between absorbance against the known concentrations of MB in the aqueous solutions, using a spectrophotometer at an optimum wavelength of 665 nm.

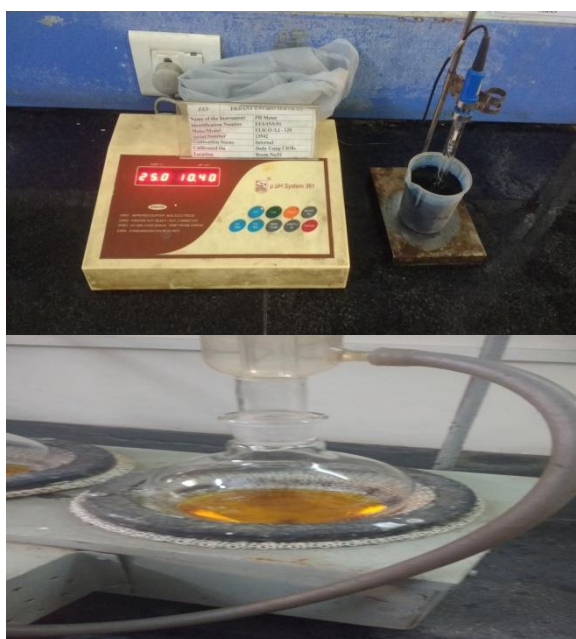


Fig.4

2.3. Batch Adsorption Process

The adsorption experiments were conducted to find the optimum conditions of DSAC to be used. This study shows investigation of three parameters: adsorbent dose, pH and contact time. There were fixed conditions in each experiment, the solution pH was 7 except in effect of pH experiment, the shaking time and speed was 6 hours and 120 RPM, respectively except in contact time influence the contact time was varied. Moreover, all experiments were conducted at room temperature between 22 °C to 26 °C. After shaking, the samples were filtered using filter paper. The final concentration of MB was determined by using the calibration curve. The amount of adsorbed MB onto AC, q_e (mg/g), was found by Equation (1). Comparison between DSAC and CAC were investigated under the same experimental conditions. All the experiments were conducted in duplicate.

$$q_e = ((C_0 - C_e)V)/W \quad (1)$$

Where C_0 and C_e are the initial and final equilibrium liquid-phase concentrations of MB, respectively (mg/L), V the volume of the solution (L), and W is the weight of the AC used (g).

The percentage removal of MB from water (R%) were determined using Equation (2).

$$R\% = ((C_0 - C_e)/C_0) \times 100 \quad (2)$$

2.3.1. Effect Of Adsorbent Concentration

The effect of adsorbent concentration on the adsorption capacity was studied by mixing various amounts of AC i.e. 0.1, 0.2 and 0.3 g of CAC and DSAC separately to the fixed MB concentration of 300 mg/L, pH of 7, shaking time of 6 hours with a rotating speed of 120 RPM held in room temperature.

2.3.2. Effect Of Ph

The effect of pH on the MB adsorption capacity was carried at different MB concentrations of 100, 300 and 500 mg/L, 6 hours equilibrium time, 120 RPM, held in room temperature and the adsorbent dose was 0.2 g of either DSAC or CAC. The initial pH values were 2 and 10 pH adjusted by adding a few drops of 0.1 M of HCl or 0.1 M of NaOH, respectively.



Fig.5

2.3.3. Effect Of Contact Time

The concentration of MB solutions used were 100, 300 and 500 mg/L at pH of 7. 0.2 g of either CAC or DSAC were added. The samples was shacked for 0, 1 and 3 hours for each MB concentrations at 120 RPM and held in room temperature 22 °C to 26 °C.

III. Results And Discussions

3.1. Physical Properties Of Dsac Compared With Cac

The physical properties are listed in Table 1. The DSAC showing pH 6.7 but CAC is more than pH 7. The CAC showing lesser moisture content than DSAC. On the other hand, ash content of both CAC and DSAC are 5% and 6%, respectively which shows that DSAC has slightly more ash content. Likewise, both activated carbons have almost same bulk density of 0.3 g/cm³.

3.2. Batch Adsorption Processes

3.2.1. Effect Of Adsorbent Concentration

In order to study the effect of adsorbent dose on MB removal, various amounts of CAC and DSAC were contacted with a fixed initial MB concentration of 300 mg/l, fixed RPM 120 and contact time 6 hours at room temperature. The amount adsorbed at an equilibrium and the percentage removal versus the adsorbent concentration by using CAC and DSAC. It is readily understood that the increase in percentage removal of MB resulted by the increase of CAC dose. Therefore, increasing CAC dose from 0.1 to 0.3 g leads to an increase of the adsorption sites and resulting a high percentage removal up to 90%.

The DSAC dose varied from 0.1g to 0.3 g and it is evident from Fig.2 that the maximum percentage removal occurred when the concentration of DSAC was 0.3 g. In comparison, DSAC showing 100% removal which is higher percentage removal of MB than CAC at 0.3 g dose. The optimum DSAC dosage was at 0.3 g. It is because of the increasing the available overall surface area of the DSAC.

The change of adsorption capacity rate might be due to fact that at first all the adsorbent sites are available and MB concentration is very high in the solution. Later, adsorption rate was lowered which indicates the possible monolayer formation of MB on the AC surface. That might indorse to the lack of available active sites required for further uptake after reaching the equilibrium. In the effect of AC dose, the DSAC is fitted with Langmuir isotherm showing regression value 0.9049.

3.2.2. Effect Of Ph

Effect of pH on removal of color using date seeds PAC was investigated over a range of pH from 5 to 11. Batch adsorption experiment was carried out with the sample of initial color concentration 1200 Pt-Co (contact time: 30 min, dosage: 7 g/l, temperature: 30°C, particle size: 150-300 mm, agitation speed: 200 rpm). The removal percentage of color increased with increase of pH from 5 to 11. Maximum color removal (92%) was observed at pH 11 and the minimum removal (55%) was at pH 5. The increasing trend of removal of color

with increasing pH is dependent on the nature of the adsorbent. Date seeds PA was activated using $ZnCl_2$. pH of date seeds PAC paste was measured as 4.47 which denotes that the activated carbon is acidic in nature and will have a tendency to react better with aqueous solution of basic nature. This result indicates that removal of contaminants using activated carbon having different surface characteristics (acidic or basic) will have an influence of pH.

3.2.3.Effect Of Contact Time And Adsorbent Dosage

Effect of contact time with initial color concentration ranging from 800 to 1200 Pt-Co on removal of color of textile wastewater using date seeds PAC is presented in Fig. 1(a). The adsorption of color was rapid in first 5 minutes for all three initial concentrations (800, 1000 and 1200 Pt-Co) and after 30 minutes, the incremental amount of color adsorbed was insignificant with the increase of contact time. Therefore, for further experiments 30 minutes was considered as the equilibrium contact time with these wastewater samples. At equilibrium contact time, a 75% color removal was obtained for both the samples having initial color concentration of 800 and 1000 Pt-Co and 70% color removal was achieved for the wastewater having 1200 Pt-Co initial concentration. At the start of experiment, the available sites present in adsorbent are more, so the adsorption rate is high. As the time progresses the availability of active sites inside or outside of adsorbent gradually decreases, so the color removal gradually decreases and after the equilibrium time, the adsorption rate was not significant. Adsorption rate typically increases with the increase in initial color concentration as increased color concentration increases the mass transfer driving force and hence the rate at which color induced molecules pass from the bulk solution to the particle surface. For 800 Pt-Co initial color concentrations, color adsorption rate was 120 Pt-Co per gram of adsorbent per litre; whereas adsorption rate was 150 Pt-Co per gram of adsorbent per litre for 1000 Pt-Co initial concentration and 168 Pt-Co per gram of adsorbent per litre for 1200 Pt-Co color concentration.

Increasing the dosage of date seeds PAC increases the availability of surface active sites for adsorption and results in higher color removal amount of color removal increased from 77 to 96 percent with increase in dosage of adsorbent from 4 to 14 g/L for 1200 Pt-Co initial color concentration. These results are quite comparable to that using commercially available PAC as it was previously found that a COD removal of 88 to 98 percent could be obtained for low and medium strength textile wastewater with a PAC dose of 15 g/L (Yeh *et al.*, 2002). The adsorbent dosage corresponding to 87% color removal (i.e. 7 g/L) has been used as a reference dose in subsequent experiments.

3.2.4.Effect Of Temperature

Thermodynamic parameters such as heat of adsorption and energy of activation play an important role in modulating adsorption behavior and both the parameters are strongly dependent on temperature. Adsorption of color at three different temperatures (30°C, 35°C and 45°C) onto date seeds PAC was studied for 800, 1000 and 1200 Pt-Co initial color concentrations (Fig. 1(c)). It is observed that with the increase in temperature color removal percentage increased. Color removal increased from 67.5 to 75%, 69 to 76% and 72 to 78.33% with increase in temperature from 30°C to 45°C for samples having initial color concentration of 1200, 1000 and 800 Pt-Co respectively. The increase in color removal percentage with the increasing experimental temperature may be attributed to the fact that as the temperature increases, rate of diffusion of adsorbate molecules across the external boundary layer and the internal pores of date seeds PAC also increases.

3.2.5.Effect Of Agitation Speed

The adsorption of color by date seeds PAC for different agitation speeds of shaker ranging from 150 to 250 rpm with varying initial concentration from 800 to 1200 Pt-Co were studied. The amount adsorbed was found to increase from 120 to 130 Pt-Co per gram of adsorbent per litre with increase in agitation speed of shaker from 150 to 250 rpm for a sample of 1200 Pt-Co initial color concentration. The color removal percentage increased with the increase of agitation speed of the shaker as shown in Fig. 1(d). This may be due to the fact that with low agitation speed of shaker, greater contact time is required to attain the equilibrium. With the increase of initial color concentration, the % color removal was generally found to decrease (Fig. 1(d)). Color removal increased from 65 to 71.25%, 68 to 74% and 70 to 76% with increase in agitation speed of shaker from 150 rpm to 250 rpm for samples having initial color concentration of 1200, 1000 and 800 Pt-Co respectively.

3.3adsorption Isotherm

Batch adsorption studies with different initial concentrations of colored sample were performed in order to determine adsorption isotherms for color removal using date seeds PAC. Isotherm is an empirical relation which is the presentation of the amount of solute (colored particle) adsorbed per unit weight of

adsorbent (date seeds PAC). The adsorbent phase concentration after equilibrium is computed using the Eq.(1) as:

$$q_e = (C_o - C_e)V/m \dots\dots\dots (1)$$

Where, q_e is the amount of solute adsorbed per unit weight of adsorbent (mg/g), C_o is the initial concentration of adsorbate (mg/L), C_e is the final equilibrium concentration of solute (mg/L), m is the mass of adsorbent (g) and V is the volume of wastewater (Litre).

In this current study, the Freundlich and Langmuir isotherm models were examined. The Freundlich adsorption model illustrated in Eq. (2) is the most widely used mathematical model in aqueous systems. The equation is written as:

$$q_e = K_f C_e^{1/n} \dots\dots\dots (2)$$

Where, q_e is the amount adsorbed at equilibrium (mg/g), C_e is the equilibrium concentration of adsorbate (mg/L), K_f is the adsorption capacity constant (L/g), n is the intensity constant. The Freundlich equation is modified in logarithmic form as:

$$\log(q_e) = \log(K_f) + 1/n \log(C_e) \dots\dots\dots (3)$$

The logarithmic form of Freundlich isotherms for color adsorption using date seeds PAC for different temperatures are represented in Fig. 2(a). The isotherm values fitted well with R^2 value of 0.9967, 0.9979 and 0.9991 for color adsorption under temperatures 30°C, 35°C and 45°C respectively. The adsorption capacity constant K_f and intensity constant (n) at 30°C, 35°C and 45°C temperature are presented in Table I. Using Freundlich model, the isotherm Eqs. (4), (5) and (6) were obtained for color adsorption at 30°C, 35°C and 45°C temperature respectively

$$q_e = 2.80x C_e^{0.6216} \dots\dots\dots (4)$$

$$q_e = 1.57x C_e^{0.7346} \dots\dots\dots (5)$$

$$q_e = 3.85x C_e^{0.6098} \dots\dots\dots (6)$$

The n value indicates the degree of nonlinearity between solution concentration and adsorption as follows: if $n = 1$, then adsorption is linear; if $n < 1$, then adsorption is a chemical process (chemisorption); if $n > 1$, then adsorption is a physical process (physisorption) (Mohan and Karthikeyan, 1997). Here, the values of n obtained from the slopes of the plots were from 1.36 to 1.64 indicating physisorption has taken place. Specifically, the linear least-squares method and the linearly transformed equations have been widely applied to correlate sorption data, where $1/n$ is a heterogeneity parameter. The smaller the $1/n$, the greater the expected heterogeneity is and values of n between 1 to 10 represents favorable adsorption.

IV. Conclusion

In the present study, optimization of MB removal from aqueous solution using DSAC prepared by chemical activation using phosphoric acid (H_3PO_2) as activated agent was investigated. The DSAC was compared with CAC. The physical properties and influence of adsorbent dose, pH and contact time of both ACs were determined. In conclusions, the characteristics of the prepared DSAC were pH 6.7, moisture content 7.99%, ash content 6% and bulk density 0.36 g/cm^3 can be replaced with CAC. The percentage removal of MB by DSAC was higher than CAC, 100% and 90% respectively i.e. with optimum dosage 0.3 g for 300 mg/L MB concentration. The optimum pH and contact time of DSAC was found to be pH 10 and 3 hours respectively. Moreover, the pH 10 showing optimum percentage removal 100, 100 and 96% for 100, 300 and 500 mg/L respectively. The optimum contact time was 3 h showing percentage removal 90, 86 and 80% for 100, 300 and 500 mg/L MB concentrations respectively. All adsorption data from CAC are best fits with Freundlich isotherm. However, DSAC adsorption data did not fit with one isotherm only but it fits with both Langmuir and Freundlich isotherms in different batch experiments. The adsorbent dose and 10 pH data fits with Langmuir isotherm and didn't fit with Freundlich isotherm and vice versa with pH 2. Moreover, the DSAC best fits with Freundlich isotherm in contact time adsorption data. Finally, from these results it is understood that DSAC can be replaced with CAC using the optimum conditions for best results.

References

- [1]. Chafia Bouchelta et al. (2008). Preparation and characterization of activated carbon from date stones. Science direct.
- [2]. M.K. Sharma, R.C. Sobti. (2000). Rec effect of certain textile dyes in *Bacillus subtilis*. 27–38. [https://doi.org/10.1016/s1383-5718\(99\)00201-6](https://doi.org/10.1016/s1383-5718(99)00201-6)
- [3]. Kayode Adesina Adegoke, Olugbenga Solomon. (2015). Dye sequestration using agricultural wastes as adsorbents. Water Resources and Industry. <https://doi.org/10.1016/j.wri.2015.09.002>
- [4]. ASTM. (1996). *American Society for Testing and Materials Annual Book of ASTM Standard*. Philadelphia PA.
- [6]. Test, A. S. (1991). Methods for moisture in activated carbon. Philadelphia: PA: ASTM Committee on Standards.
- [7]. Abdul Halim Abdullah et al. (2000). Preparation and Characterization of Activated Carbon from Gelam Wood Bark (Melaleuca cajuputi). Malaysian Journal of Analytical Sciences, 68.
- [8]. K. Suresh Kumar Reddy et al. (2012). A comparison of microstructure and adsorption characteristics of activated carbons by CO₂ and H₃PO₄ activation from date palm pits. NEW CARBON MATERIALS, 344–351. [https://doi.org/10.1016/S1872-5805\(12\)60020-1](https://doi.org/10.1016/S1872-5805(12)60020-1)
- [9]. Abde et al. (2009). Adsorption, kinetic and equilibrium studies on removal of basic dye from aqueous solutions using hydrolyzed oak sawdust. Desaline. Water Treat, 252–268.
- [10]. Ahmed El Nemr et al. (2009). Removal of direct blue-86 from aqueous solution by new activated carbon developed from orange peel. Journal of Hazardous Materials, 102–110. <https://doi.org/10.1016/j.jhazmat.2008.03.060>
- [11]. Sha Liang et al. (2010). Isotherms, kinetics and thermodynamic studies of adsorption of Cu²⁺ from aqueous solutions by Mg²⁺/K⁺ type orange peel adsorbents. Journal of Hazardous Materials, 756–762. <https://doi.org/10.1016/j.jhazmat.2009.09.116>

K. V. Priya " Preparation of Activated Carbon From Date Seeds And Evaluation of It Application A Review Paper." IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE), vol. 15, no. 1, 2018, pp. 81-87