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Sustainable Raecovery Of Sucrose And Glucose From Aqueous Solutions Using Green Carbon

M Mini Margaret[1*], G Rexin Thusnavis[1]

[1]Research Department of chemistry, Pioneer Kumaraswamy College, Nagercoil-629003,

Tamil Nadu, India.

Abstract

Replacement of fossil resources demands cost-effective and technically feasible solutions. Renewable carbon sources from lignocellulosic biomasses are among the effective options. Lignocellulosic wastes and agricultural waste materials contain multiple sugars. Large quantities of lignocellulosic wastes generated from agricultural residues, agro industrial practices are generally pollute the environment. This environmental waste can be minimized by converting them into valuable products. The aim of this study is more towards environmental friendly solutions by transforming the lignocellulosic waste into valuable material which improves and upgrades technology for waste management and minimization. Eco-friendly and economical adsorbents are desirable for removing pollutants from the environment. Production of natural adsorbents involves the use of waste from agricultural product which makes them cost effective and eco friendly. In the present study the glucose and sucrose were adsorbed on Prosopis Juliflora Green Carbon (PJGC) and desorbed in water and ethanol. This method can be suggested to fractionate the glucose and sucrose from lignocellulosic wastes. The adsorbent was charecterised by FT-IR and BET surface area studies. the maximum adsorption of glucose and sucrose on PJGC as analysed by adsoption studies in relation to contact time, pH, temperature, concentration and adsorbent dosage. The stability of glucose and sucrose on PJGC was determined by desorption studies using water and ethanol.

Key Words- lignocellulosic wastes, Adsorption , *Prosopis Juliflora*

I .Introduction

Lignocellulosic waste generation and their improper disposal has accelerated the problems associated with environmental pollution. Constructive ways to manage and mitigate the pollution associated with lignocellulosic waste has propelled the present work. Lignocellulosic biomass is a renewable and abundant natural resource that can be used to produce a wide range of bio based materials with a low carbon footprint compared to traditional fossil-based resources [1] Lignocellulosic wastes have been proposed as large

renewable resources for chemicals and sugars The low cost of readily available lignocellulosic biomass has caused increasing interest in the bioconversion of this feedstock into liquid fuels and chemical products [2-6]. Cellulose and hemicellulose are hydrolyzed into sugar monomers that can be converted liquid fuels [7–11] Ionic liquids (ILs) are useful for dissolving or solvating biomass and subsequent separation of lignin from hemicellulose and cellulose [12–14]. The high solubility of glucose in these aqueous ionic liquids makes a challenging separation problem [16–19] likely to encounter formidable problems in practice. After obtaining monomeric sugars from lignocellulosics through an appropriate pretreatment method and EH, the material is recovered and purified to extract the desired sugar(s)[20]. Sugar recovery methods may include solid/liquid systems, chromatographic and membrane separations, ion exchange or crystallization, among others. These strategies can be applied to separate the extracts from the non-treated biomass (physical separations); (2) recover the glucose and/or the xylose (the main monosaccharides resulting from the hydrolysis of the cellulosic and hemicellulosic fractions of the LB, respectively) [21] through purification approaches, aiming for their later use in the synthesis of a variety of products, including sugars, biofuels, and value-added chemicals.Research on biorefineries is active worldwide. The ultimate goal is to develop processes which could convert biomass efficiently into fuels, power, heat, and value-added products. One of the most studied concepts is the so-called sugar platform where biopolymers (cellulose and hemicellulose) are hydrolyzed into monomers (sugars). Fermentable sugars may then be converted biochemically into various products. Hydrolysis is carried out enzymatically with cellulases and hemicellulases or using an acid, most often dilute sulfuric acid. Lignocellulosic biomass contains various sugar monomers such as xylose, mannose, glucose, fructose, and galactose but also arabinose and rhamnose, which are released under hydrolysis [22] Adsorption equilibrium data have been published for glucose, fructose, sucrose, arabinose, xylose, and some oligosaccharides on strong acid cation (SAC) exchange resins in K+, Na+, Ca2+, or Fe2+ forms [23-26]. In this work, we consider adsorption of glucose and sucrose as a possible method for separation.

II. MATERIALS AND METHODS

The glucose (Loba chemie pvt. Ltd.) and sucrose (Loba chemie pvt. Ltd.) are used to study the adsorption process. Anthrone (Loba chemie pvt. Ltd.) and concentrated sulphuric acid (Merck, 98%) are used for the colour reaction of sugars. The Prosopis Juliflora barks were collected from local areas, for the preparation of green carbon which is used as an adsorbent for the adsorption process. Hydrochloric acid (Merck) and sodiumhydroxide (loba) were used to adjust the pH of the glucose and sucrose solution.

4.3 Glucose and Sucrose solution preparation

A stock solution of 1000mg/L is prepared by dissolving 1g of glucose and sucrose separately in distilled water in a 100ml standard measuring flask. The working solution of desired concentration is prepared by successive dilution of the stock solution. The concentration of glucoce and sucrose was analyzed by UV visible spectrometer (Perkin Elmer Lambda 25).

Green carbon preparation

The green carbon means that the carbon prepared from the cellulose based material by thermal method without using any chemicals. High temperature reactor used for the preparation of green carbon. *Prosopis Juliflora* barks were cut into chips and sun dried. The dried *P. Juliflora* chips were packed and supported either side by asbestos wool in a vertical type high temperature reactor. This reactor kept inside the tubular furnace. The furnace temperature is controlled by the digital temperature controller. The reactor temperature is increased up to 200°C and maintained the same temperature for 3 hours in the absences of air and reactor continuously evacuated during the carbonization reaction to remove volatile organics, hydrogen and moister. In further increase of temperature in the range of 250-350°C, the *P. Juliflora* chips becomes green carbon.

Determination of Glucose and Sucrose concentration by Anthrone method

Carbohydrates are dehydrated by conc.H_2SO_4 to form furfural and its derivatives which condense with anthrone to form blue-green complex with an absorption maximum at 578nm. Concentration of glucose and sucrose were measured spectrophotometrically by Anthrone method briefly 2ml of chilled 75% H_2SO_4 and 4ml of chilled Anthrone solution were progressively added to 10ml of boiling tubes, and then add 1ml of Glucose and Sucrose solution to the tubes separately. Place the tubes in a boiling water bath for 15minutes , cool and measure the optical density at 578nm against a blank and converted to concentration from the calibration curve.

4.6 Adsorption experiment

About 0.5g of the adsorbent is taken and mixed with 50ml of glucose and sucrose solution in 100ml conical flask separately. The mixture was shaken in a mechanical shaker (KEMI) at rpm 150 to find out the equilibrium adsorption and at the end of the experiment the solution centrifuged off. Concentration of glucose and sucrose remaining in solution were measured spectrophotometrically by Anthrone method. The contact time was studied up to 90minutes to find out equilibrium adsorption. The pH effect of glucose and sucrose was studied in the range of 1-8. The adsorption process of glucose and sucrose on PJGC was studied in the concentration range of 10-50mg/L. The temperature effect of glucose and sucrose was studied in the range of 30-70°C. the adsorbent dosages were studied in the range of 0.5- 2.5g.

The percentage adsorption of Glucose and Sucrose at equilibrium and the amount of Glucose and Sucrose transferred on to the surface of the adsorbent, q_e (mg/g) was calculated using the following relationships.

The percentage of Glucose and sucrose adsorption is calculated from the following equation

$$(q_e) = 100(C_0\text{-}C_e)/C_0$$

Amount of adsorbed Glucose / Sucrose molecules per gram of solid is calculated from the equation

$$(q_e) = (C_0\text{-}C_e)V/W$$

Where,

C_0 → initial concentration of Glucose/Sucrose (mg/L)

C_e → equilibrium concentration of Glucose/Sucrose (mg/L)

V → volume of the solution (L)

W → mass of the adsorbent (g)

V → volume of the solution (L) , W → mass of the adsorbent (g)

III. RESULTS AND DISCUSSION

3.1 Characterization of Glucose and Sucrose solution

The maximum absorption of Glucose and Sucrose solution is determined spectrophotometricaly by using anthrone reagent.The Blue green complex formed is characterized by UV-Visible spectrometer.The maximum absorption of Glucose-anthrone complex is 578nm is shown in fig:1 and the maximum absorption of sucrose-anthrone complex is 580nm is shown in fig:2.

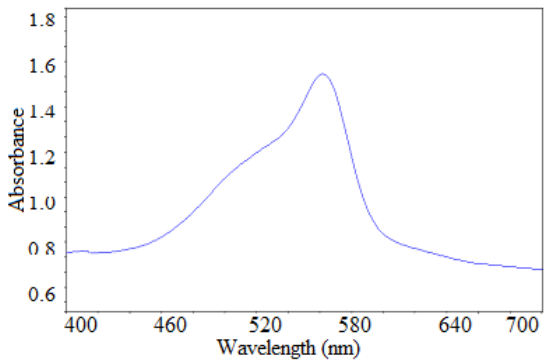


Fig: 1 UV spectrum of Glucose-anthrone complex

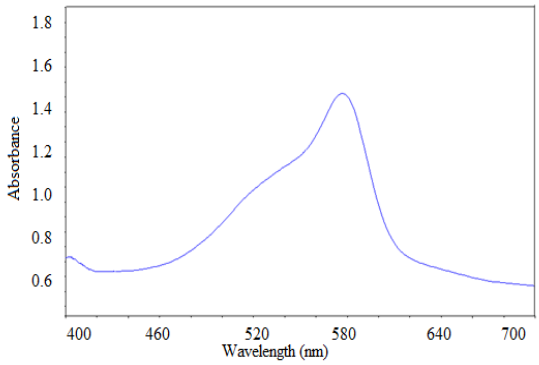


Fig: 2 UV of Sucrose anthrone complex

3.2 3.2 Characterization of the adsorbent

The adsorbent(Prosopis Juliflora Green Carbon) is characterized by FT-IR spectra and BET-surface area to find out the nature of the active site, structure, pore size and pore diameter.

3.2.1 FT-IR Spectrum of Green carbon

The FT-IR Spectrum of Green carbon is shown in fig. 3. The O-H stretching observed at 3430 cm^{-1} corresponds to the OH group present in the glucose unit of cellulose in Prosopis Juliflora chips. The peak at 1040 cm^{-1} corresponds to C-O-C stretching in cyclic form present in glucose unit. The peak at 1117 cm^{-1}corresponds to C-O-C stretching in between two glucose unit in the cellulose. The peak at 1014 and 1117 cm^{-1} are less intense in green carbon. This may be due to the cracking of glucose units in the cellulose polymers. Moreover, the peak at 1625 cm^{-1} in Green carbon is more intense, which may be due to the oxidation of alcoholic group to carbonyl group in the glucose unit of cellulose. Thus the characterization confirm the loss of OH group and have less number of ether linkage,which proved the formation of green carbon.

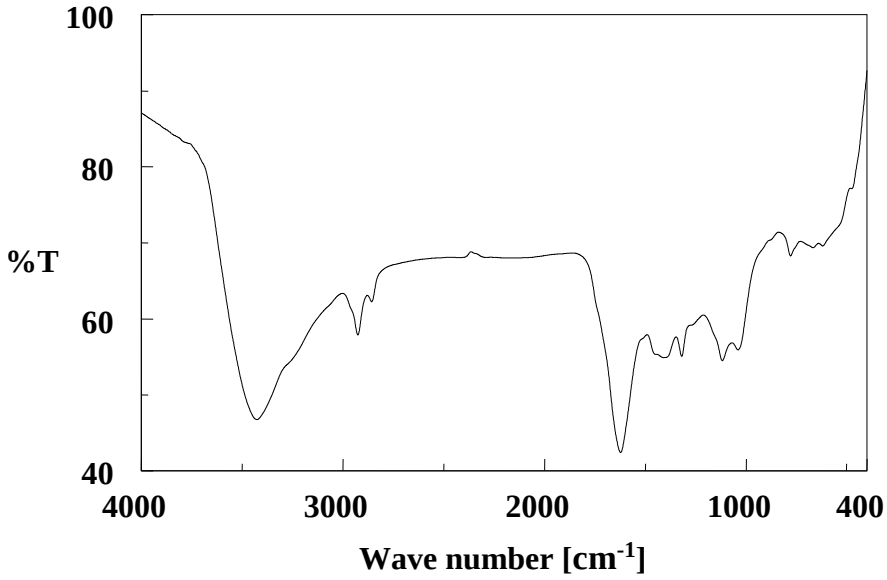


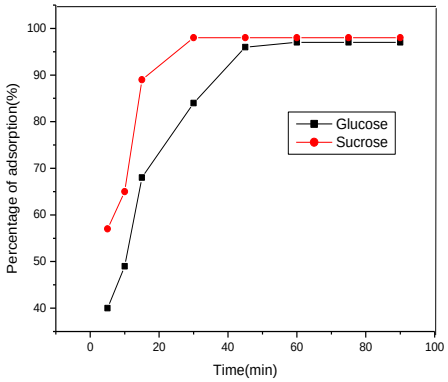
Figure 3: FT-IR Spectrum of Green carbon

3.2.2 BET-Surface area

The BET- Surface area of PJGC was measured by nitrogen adsorption isotherm method. The BET-Surface area was found to be 2.7219m^2/g. The pore size of green carbon is in the range of 12–15Å.Density of green carbon is 0.4857 and the particle size of green carbon is 0.667mm.

3.3 Adsorption studies

To investigate the value added sugar separation, the adsorption of both glucose and sucrose have been studied on *Prosopis juliflora* Green Carbon (PJGC). The experimental conditions like contact time, concentration, pH, adsorbent dosage, temperature are optimized for maximum adsorption. These experimental data are used to investigate the adsorption mechanism and sugar separation.



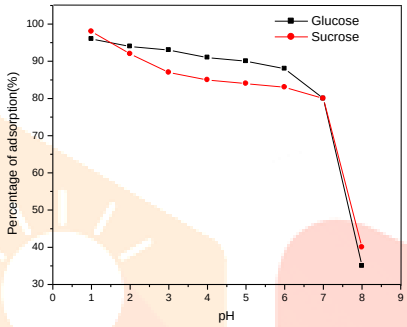
Glucose: Contact time- 60 Min, Temperature- 30°C, Concentration-10 ppm,
Adsorbent Dosage- 0.5 g, adsorption of glucose by PJGC is 96%.

Sucrose: Contact time- 30 Min, Temperature- 30°C, Concentration-10 ppm,
Adsorbent Dosage- 0.5 g, adsorption of sucrose by PJGC is 98%.

Figure 4: Effect of contact time on the adsorption of glucose and sucrose on PJGC

3.3.1 Effect of pH

The pH effects on the adsorption of glucose and sucrose on PJGC shown in figure: 5. The pH of the glucose and sucrose solution was varied from 1-8 by using 0.1N HCl and 0.1NaOH. The results revealed that the amount of adsorbed glucose and sucrose increase with decreasing the pH of the suspension and attain a maximum adsorption at pH 1, below the pH 7 the green carbon surface acquires a net positive charge. The partial negative charge oxygen of the -OH groups in Glucose and Sucrose interact with the positive charge surface of PJGC. However, there is a decrease in adsorbed amount beyond the pH 7, for a further increase in the pH value the surface acquires a negative charge, an electrostatic repulsion begins to operate between the green carbon surfaces and -OH groups of the Glucose and Sucrose. Thus with further increase in the pH the amount of adsorption decreases.



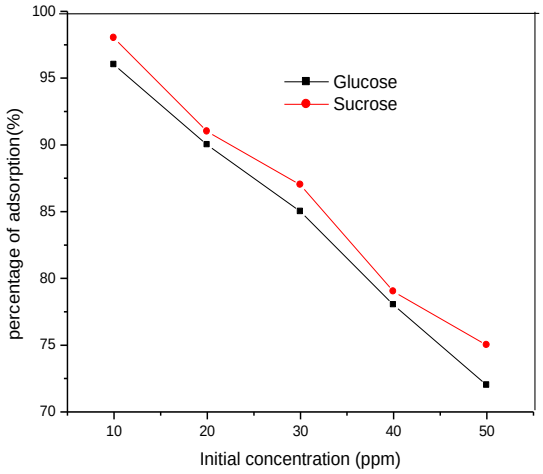
Glucose: Contact time- 60 Min, Temperature- 30˚C, Concentration-10 ppm, Adsorbent Dosage- 0.5 g,

Sucrose: Contact time- 30 Min, Temperature- 30˚C, Concentration-10 ppm, Adsorbent Dosage- 0.5 g,

Figure 5: Effect of pH on the adsorption of Glucose and sucrose on PJGC

3.3.2 Effect of concentration

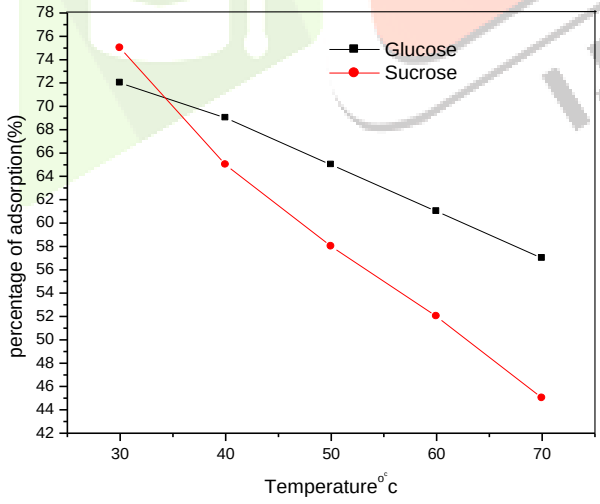
The effect of sugar concentration in the range of 10-50mg/L of glucose and sucrose adsorption on PJGC is shown in the figure 6. Percentage adsorption of glucose decreased with increase of glucose concentration from 96-72%. Percentage adsorption of sucrose decreased with increase of initial sucrose concentration from 98-75%. The adsorption decreased with increase of sugar concentration due to the saturation of surface area and active sites on green carbon. This means that the adsorption is highly dependent on initial concentration of glucose and sucrose. At lower concentration, the ratio of the initial number of glucose and sucrose molecules to the available surface area is low. Subsequently, the fractional adsorption becomes independent of initial concentration. However, at high concentration the available sites of adsorption become fewer and hence the percentage adsorption of glucose and sucrose is dependent upon initial concentration [188,189].



Glucose: Contact time- 60 Min, Temperature- 30°C, Adsorbent Dosage- 0.5 g, pH 1
Sucrose: Contact time- 30 Min, Temperature- 30°C, Adsorbent Dosage- 0.5 g, pH 1
Figure 6: Effect of concentration on the adsorption of glucose and sucrose on PJGC

3.3.4Effect of temperature

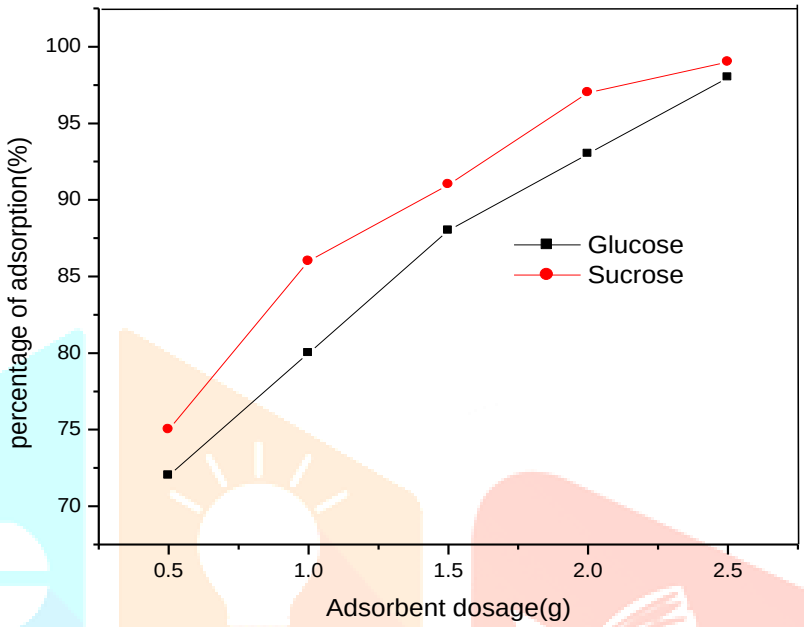
The effect of temperature on the uptake of Glucose and sucrose on green carbon at 30-70°C is shown in figure 7. The decrease of adsorption with rise of temperature may be due to the enhanced escaping tendency of Glucose and sucrose molecules from the surface of the adsorbent. This decrease of adsorption may be due to the collapse of hydrogen bond or physisorption of glucose and sucrose on green carbon.



Glucose: Contact time- 60 Min, Concentration-50 ppm, Adsorbent Dosage- 0.5 g, pH 1
Sucrose: Contact time- 30 Min, Concentration-50 ppm, Adsorbent Dosage- 0.5 g, pH
Figure 7: Effect of Temperature on the adsorption of glucose and sucrose on PJGC

3.3.5 Effect of adsorbent dosage

The effect of adsorbent dosage for the sugar adsorption is shown in the figure 8. The percentage adsorption increases with increase of adsorbent dosage. It is apparent that by increasing the adsorbent dosage increases the number of valuable adsorption sites as well as the surface area. Therefore adsorption increases with increase of dosage.



Glucose: Contact time- 60 Min, Concentration-50 ppm, pH 1
Sucrose: Contact time- 30 Min, Concentration-50 ppm, pH 1

Figure: 8 Effect of Adsorbent dosages on the adsorption of glucose and sucrose on PJGC

3.4 Adsorption isotherms

The adsorption isotherm of glucose and sucrose on PJGC at different concentrations were studied and it was observed that both are well matched with the Langmuir and Freundlich adsorption isotherms (fig: 9, 10, 11, and 12).

The Langmuir equation is represented as

$$\frac{C_e}{Q_e} = \frac{1}{Q_{max} K_L} + \frac{C_e}{Q_{max}}$$

Where,

Qe → equilibrium saccharide concentration on the adsorbent (mg/g)

Ce → equilibrium saccharide concentration in solution (mg/L)

Q_{max} → monolayer capacity of the adsorbent (mg/g)

K_L → Langmuir constant (L/g)

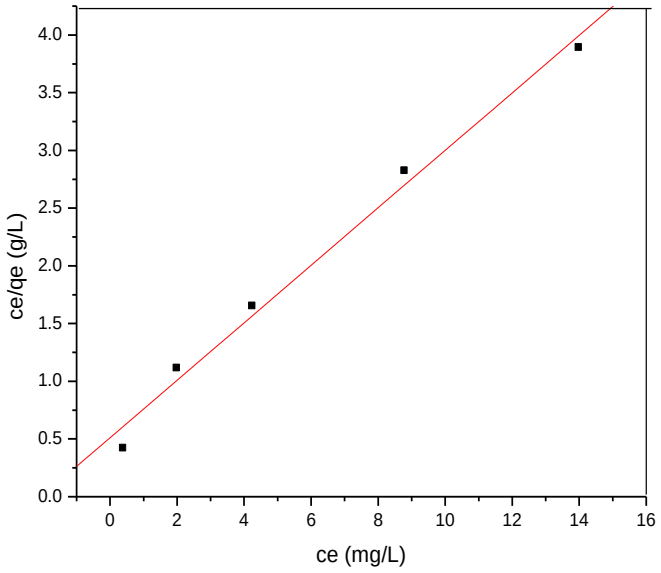


Figure 9: Langmuir Isotherm for adsorption of Glucose on PJGC

Figure 10: Langmuir Isotherm for adsorption of Sucrose on PJGC

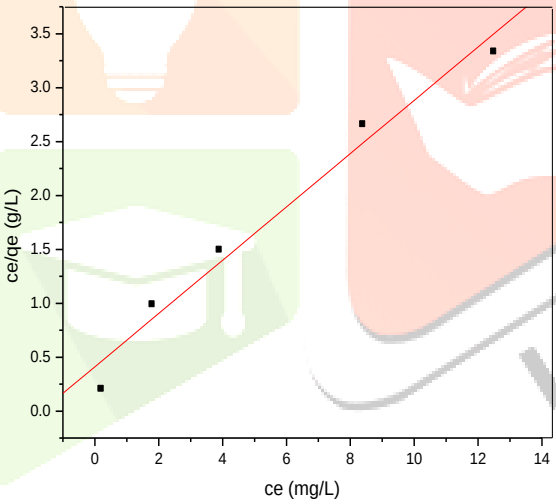


Figure 10: Langmuir Isotherm for adsorption of Sucrose on PJGC

Table 1: Langmuir adsorption isotherm plot values for the adsorption of Glucose and Sucrose

Saccharides	**Q_{max}**	**K_L**	**R^2**
Glucose	4.0159	0.4874	0.9947
Sucrose	4.0457	0.6015	0.9877

The Freundlich equation is represented as

$$\ln Q_e = \ln K_F + \frac{1}{n} \ln C_e$$

Where,

Qe → equilibrium saccharide concentration on the adsorbent (mg/g)

Ce → equilibrium saccharide concentration in solution (mg/L)

K_f → Freundlich constant (L/g)

n → (dimensionless) is the heterogeneity factor

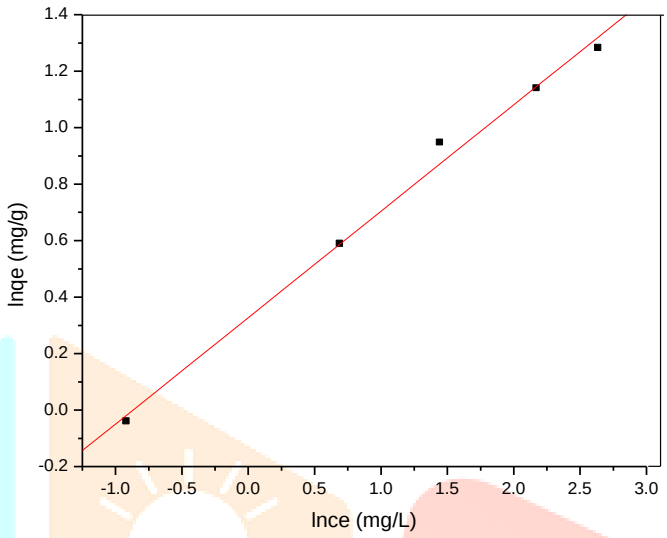


Figure 11: Freundlich isotherm for adsorption of Glucose on PJGC

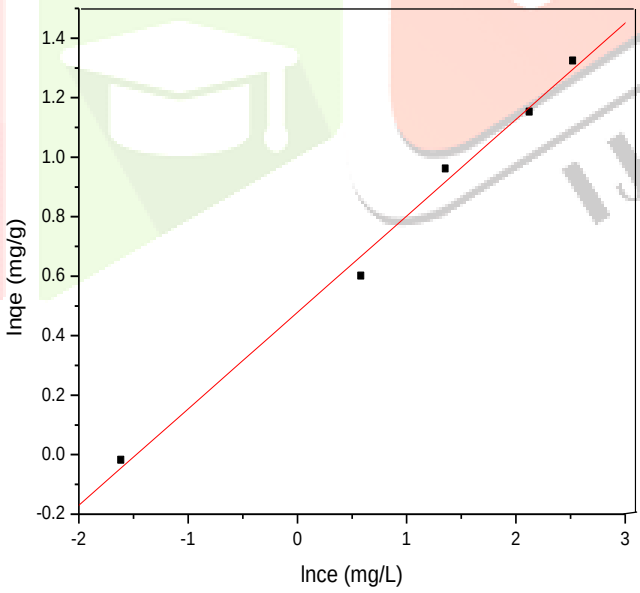


Figure 12: Freundlich isotherm for adsorption of Sucrose on PJGC

Table 2: Freundlich adsorption isotherm plot values for the adsorption of Glucose and Sucrose

Saccharides	n	K_F	R^2
Glucose	2.6543	1.3873	0.99657
Sucrose	3.0845	1.6132	0.9964

The various parameters obtained from Langmuir and Freundlich adsorption isotherm are given in Table 1&2. The R^2 values for both the adsorption models of glucose and sucrose on PJGC are close to 1 indicated that the adsorption of glucose and sucrose followed the Langmuir and Freundlich adsorption isotherms. The value of n more than one corresponds to multilayer adsorption of glucose and sucrose.

3.5 Adsorption kinetics:

The adsorption kinetics glucose and sucrose on PJGC has been studied in regular time interval at room temperature to determine the kinetics of the adsorption.

The pseudo first order equation is [190]

$$\log(q_e - q) = \log q_e - \frac{K_1 t}{2.303}$$

Where,

q_e & q → amount of saccharides adsorbed at equilibrium and at any time t, respectively (mg/g)

k_1 → the rate constant of adsorption (min^{-1})

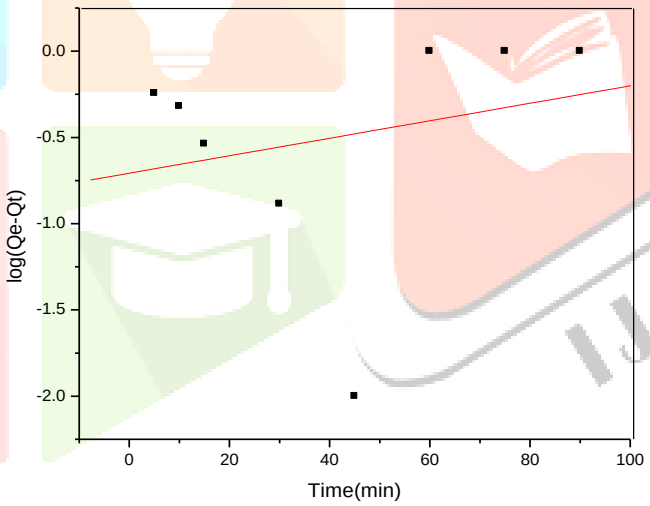


Figure 13: Pseudo-first-order kinetics of Glucose on PJGC

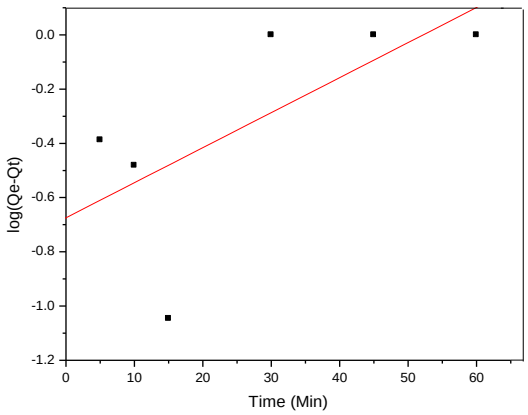


Figure 14: Pseudo-first-ordr kinetics of Sucrose on PJGC

Table 3: Pseudo-first-order kinetic values for the adsorption of Glucose and Sucrose on PJGC

Saccharides	Q_e	K	R^2
Glucose	0.1962	0.01165	0.2348
Sucrose	0.21164	0.0297	0.6720

The pseudo second order equation is [191]

$$t / Q_t = 1/ k Q^2_e + 1 / Q_e t$$

where,

k is the rate constant,

Q_e is the amount of dye adsorbed per unit mass of the adsorbent at equilibrium

Q_t is the amount of dye adsorbed per unit mass of the adsorbent at time t

T is time in minutes.

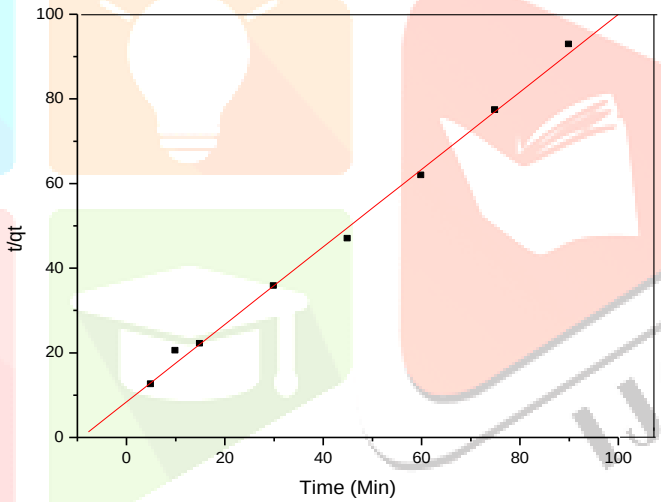


Figure 15: Pseudo-second-order kinetics of Glucose on PJGC

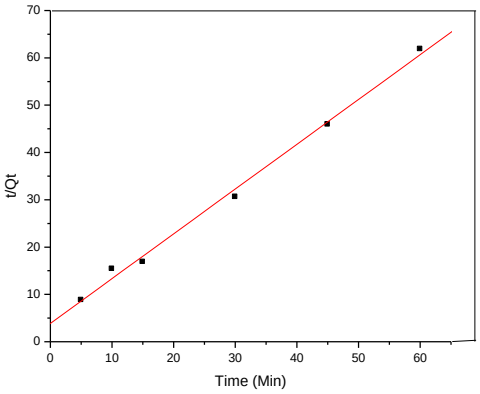


Figure 16: Pseudo-second-order kinetics of Sucrose on PJGC

Table 4: Pseudo-second-order kinetic values for the adsorption of Glucose and Sucrose on PJGC

Saccharides	Q_e	K	R^2
Glucose	1.0923	0.099	0.99813
Sucrose	1.0562	0.2320	0.99759

The plot of t / Q_t Vs t gives straight lines for Glucose and Sucrose adsorption on PJGC. The linear regression coefficient (R^2) near to 1 (Table 4) indicated that the adsorption of glucose and sucrose on PJGC follows a pseudo-second order kinetics (Fig.15 and 16). The results also applied with a pseudo-first order kinetics (Fig.13 and 14) model, but the R^2 value is 0.2348 for Glucose and 0.6720 for Sucrose indicating that the adsorption of Glucose and sucrose on PJGC was not perfectly follow a pseudo- first order kinetics.(Table 3)

3.6 Adsorption Thermodynamics:

The thermodynamics parameters like free energy (ΔG^o), enthalpy (ΔH^o) and Entropy (ΔS^o) of Glucose and sucrose adsorption on PJGC can be calculated by using Van't Hoff relationship.

$$\ln K_d = \frac{-\Delta H}{RT} + \frac{n\Delta S}{R}$$

The equilibrium constant (K_d) calculated from the following equation

$$K_D = \frac{q_e}{c_e}$$

$$\Delta G = - RT\ln K_D$$

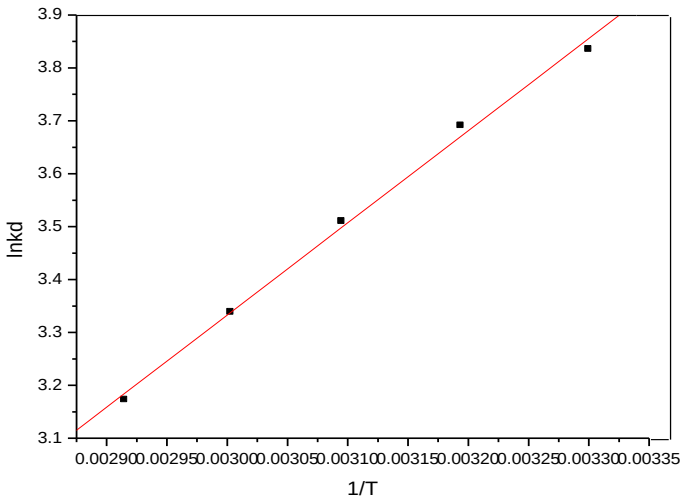


Figure17: Van't Hoff plot for the adsorption of Glucose on PJGC

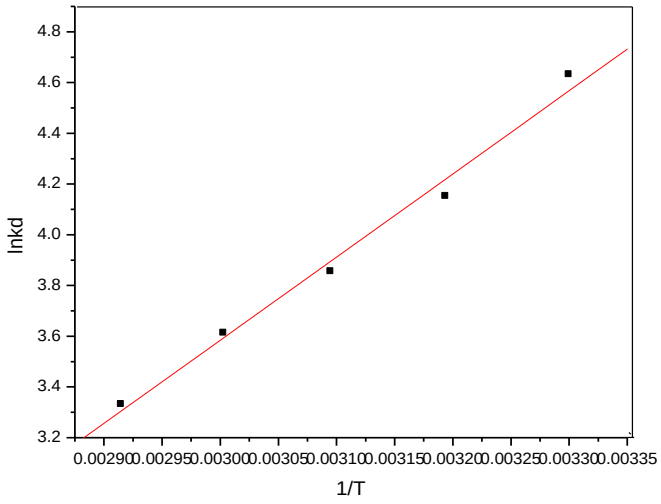


Figure 18: Van't Hoff plot for the adsorption of Sucrose on PJGC

Table 5: Thermodynamics parameters for the adsorption of Glucose on PJGC

Temperature(K)	ΔG(KJmol^{-1})	ΔH(KJmol^{-1})	ΔS(KJmol^{-1})
303	-9.7139	-14.488	-15.756
313	-9.5563		
323	-9.3988		
333	-9.2412		
343	-9.0836		

Table 6: Thermodynamics parameters for the adsorption of Sucrose on PJGC

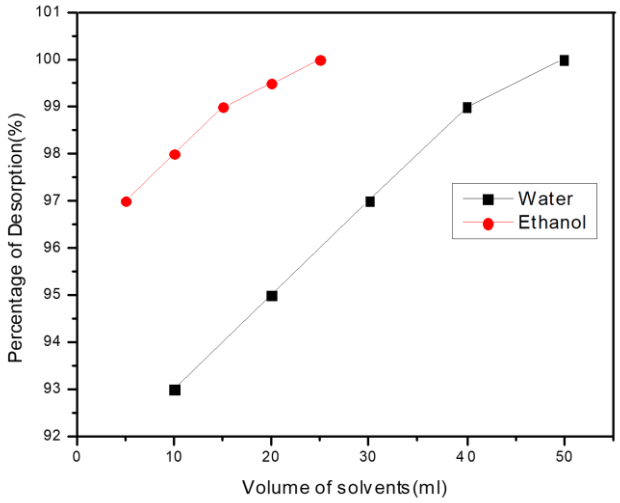
Temperature(K)	ΔG(KJmol$^{-1)}$	ΔH(KJmol^{-1})	ΔS(KJmol^{-1})
303	-11.5089	-27.2415	-51.9225
313	-10.9897		
323	-10.4705		
333	-9.95130		
343	-9.43208		

The enthalpy and entropy can be calculated from Van't Hoff Plot as shown in the figure 17&18. The ΔG^o, ΔH^o and ΔS^o values calculated for the adsorption of glucose and sucrose are given in Table 5 &6. The negative value of ΔG^o is an indication of spontaneous of the process. The negative value of ΔH^o confirms the exothermic nature of the adsorption process and the values of ΔH^o lower than 40KJ/mole indicated that the adsorption was physisorption for Glucose and Sucrose.

3.7 Desorption studies

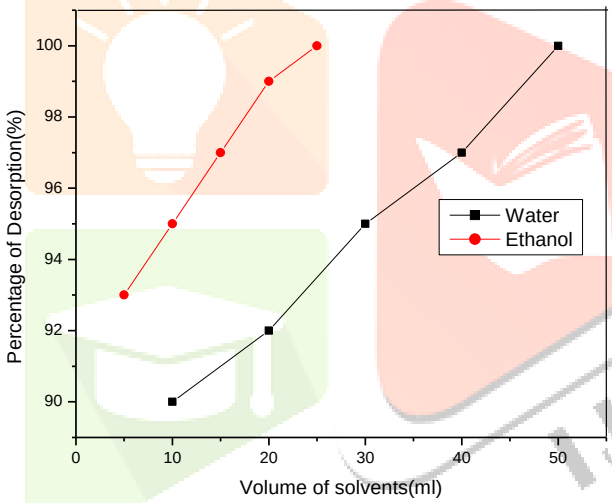
Desorption studies of glucose and sucrose are shown in the figure 19& 20. Desorption studies of glucose and sucrose have been carried out in the presence of water and ethanol at room temperature. Mild conditions are satisfactory for reaching complete glucose recovery after adsorption [192]. The complete sucrose desorption was reached in 20 minutes, while only 10 minutes were sufficient for desorption of glucose, when compared with water the amount of ethanol required to desorb both glucose and sucrose was decreased. The high desorption of sugars from PJGC indicated that the sugars were weakly adsorbed

(physisorption) on PJGC. The high desorption are promising for separation of sugars by adsorption. The PJGC is the best adsorbent for the recovery of sugars from the aqueous solutions.



Time: 10minutes, Temperature: 30°C

Figure 19: Desorption of Glucose



Time: 20minutes, Temperature: 30°C

Figure 20: Desorption of Sucrose

V. CONCLUSION

Prosopis Juliflora Green Carbon is used as an adsorbent for the adsorption of glucose and sucrose. It is characterized by FT-IR spectra and BET surface area. The adsorption study is carried out under the optimization conditions like contact time, pH, concentration, temperature and adsorbent dosage for finding the maximum adsorption. The contact time study proved that the adsorption equilibrium attained at 30min for the adsorption of sucrose and 60min for the adsorption of glucose on PJGC. The percentage of adsorption decreased with an increase in the concentration of glucose and sucrose. This is attributed to the saturation of active sites and surface area of the adsorbent. The temperature study explained that the percentage of adsorption on the adsorbent decreases as the temperature increases. The adsorbent dosage results showed a significant increase in the adsorption amount of glucose and sucrose. From the adsorption studies, adsorption isotherms, adsorption kinetics and adsorption thermodynamics were calculated.

The adsorption isotherm data are well matched with Langmuir and Freundlich adsorption isotherm model. Adsorption isotherm studies used to find out the maximum adsorption, the number of maximum layers and the adsorption capacity of each layer. Adsorption kinetic studies were studied to find out the rate of the process. The adsorption results were well matched with pseudo second order kinetics. From the adsorption thermodynamics, the free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) can be calculated.

The negative values of ΔG for a very low energy barrier present in the adsorption interface and suggested a rapid adsorption of glucose and sucrose at room temperature. The ΔH values for the adsorption of glucose and sucrose on adsorbent is lower than 40kJ mol^{-1} indicated that the adsorption is physisorption.

Desorption study is carried out by using the solvents like water and ethanol to find out the binding stability of glucose and sucrose on adsorbent. Glucose and sucrose were completely desorbed in both water and ethanol. This high desorption of sugars from PJGC indicated that the sugars were physisorbed on PJGC.

The experimental methods carried out in the present study can be suggested to fractionate the glucose and sucrose from the lignocellulosic wastes in food industries.

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