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Adsorptive Treatment of Dairy Waste Water Using Rubber Seed Shell Activated Carbon

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Abstract:
Background*: Dairy industries have grown in most of countries of the world due to the steady rise in demand of milk and milk products and India is one of the major milk producer in the world. The dairy industry generates strong wastewater characterized by high chemical oxygen demand (COD) and biochemical oxygen demand (BOD) and by high concentrations of nutrients, organic contents and pathogens along with considerable variation in pH (4.2– 9.4), relatively large load of suspended solids (0.4–2 g/l). It is estimated that about 2% of total milk processed is wasted into drains .The effluent waste water contain high COD concentration due to cleaning of floors and washing processin the day-to-day operation of the plant.Some of the process for the treatment of diary effluent includes osmosis treatment,ultrafiltration or electrochemical methodsandbiological treatment ,the limitation of these methods include high cost and fuel consumption . The present study is treatment of synthetic dairy waste water in terms of removal of chemical oxygen demand (COD) by means of adsorption using activated carbon made from rubber seed shell.*
Materials and Methods*: In the study rubber seed shell is used,The collected rubber seed shell is chemically activated as activated carbon.The batch study is conducted to study the optimum contact time and dosage.Adsorbent dosage (5g,10g,15g,20g) of activated rubber seed shell carbon was taken for analysis.the contact time between the adsorbent and synthetic dairy wastewater is taken as 15min,30min,45min,60miin.The percentage removal is obtained by determining the COD reduction.*
Results*: The effect of pH, contact time and adsorbent dosage in the removal of pollutants in dairy waste water was evaluated.The pH range was below 6 .The optimum dosage obtained was 10g/100ml and the optimum time was 15min.The adsorption efficiency obtained was above 95%.*
Key Word*: Adsorption ,Dairy waste water,rubber seed shell,chemical oxygen demand.*

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I. Introduction

Kerala is one of the leading rubber plantation state in India.It's account to 92 percent of country's total natural rubber production .Although the rubber kernel is sent to oil mills, there is still a huge amount of rubber seed shells available as agricultural waste which has become an environmental problem for rubber tree plantations . This environmental contamination problem destroys the rubber tree plantation. Therefore, rubber seed shells have chosen as a low-cost adsorbent for the treatment of dairy wastewater.Dairy industry is the main contributor to water pollution. The dairy industry wastewater are generated primarily from the cleaning and washing operations in the milk processing plants and is estimated to be 0.2-10 litres of effluent per litre of the milk processed milk with an average generation of about 2.5 litres of waste water per litre of the milk processed . The dairy industry involves processing raw milk into products such as consumer milk, butter, cheese, yogurt, condensed milk, dried milk (milk powder), and ice cream, using processes such as chilling, pasteurization, and homogenization. Due to high pollution load of dairy wastewater, the milk- processing industries discharging untreated/partially treated wastewater cause serious environmental problems . Nutrients present in dairy effluent such as nitrogen lead to eutrophication of receiving waters[1].

Dairy raw wastewater is characterized by high concentrations and fluctuations of organic matter and nutrient loads. The composition varies depending on the operations and products. The waste water of dairy contain large quantities of milk constituents such as casein, lactose, inorganic salt, besides detergents and sanitizers used for washing. With the rapid industrialization observed in the last century and the growing rate of milk production (around 2.8 % per annum), dairy processing is usually considered the largest industrial food wastewater source. Moreover, in around 50 % of the world's whey production, especially concerning acid whey, it is untreated prior to disposal. The effluents originating from various production technologies are not discharged simultaneously,thusforming a stream with wide qualitative and quantitative variations. Notwithstanding the differences in composition, attributable to the manufactured product and technological operations, dairy effluents are distinguished by their relatively increased temperature, high organic content and a wide pH range, which requires special purification in order to eliminate or reduce environmental damage. The volume of the wastewater produced depends largely on the quantity of milk processed and type of product manufactured. It appears white in colour with heavy black sludge and strong butyric acid odors due to the decomposition of casein. It is slightly alkaline in nature and becomes acidic quite rapidly, because of the fermentation of milk sugar to lactic acid. The COD of dairy wastewater is mainly due to milk, cream, or whey. Casein and whey are the main components of dairy wastewater which are relatively hydrophobic, making it poorly soluble in water, and have a negative charge in milk .The casein micelles exist in milk as insoluble particle[2].The above-mentioned phenomena make dairy industry, like many others, challenged with rising costs for wastewater treatment and disposal. Moreover industries have to meet the discharge standards mentioned by CPCB which becomes a great problem for the industrialists. Technologies such as coagulation/flocculation process and oxidation process have been developed over the years to remove organic matter (expressed as chemical oxygen demand, COD) from industrial wastewater. These methods are effective in fields of reduction and time but are expensive and require skilled personnel. They also become disadvantageous in terms of pH adjustment and generation of chemical sludge that must be treated before disposal. In addition to these various treatments which are already present in the dairy industry there are biological treatments including trickling filters and activated sludge process. Though they are effective for complete treatment of the wastewater but are non- economical, large power demand, more chemical consumption, and large area availability. Thus it is very much necessary for characterization of wastewater, treatability studies, and planning of proper units and processes for effluent treatment[2].

Adsorption technique emerges as promising technique in the removal efficiency. The use of adsorbent made from rice husk[2],Baggasse fly ash[5],coconut shell activated carbon [7],groundnut shell[29],orange peel[30],orange and banana peel[8] and marl and stone cutting solid waste [9]are explored for the removal of organic pollutant from dairy waste water. The use of RSS activated carbon activated chemically is used as the adsorbent in our study .The project aim is to determine the efficiency of removal of pollutants from dairy wastewater using low cost effective adsorbent made from rubber seed shell.

II. Material And Methods

Preparation of Adsorbent

The rubber seed shell collected is washed with water repeatedly to remove the earthy materials and dried in oven at 80 degree Celsius overnight .it is then crushed in to pieces approximately 1.18 to 0.6 mm in size, corresponding to standard sieves 4-16. Once this step was completed to activate the pieces it is soaked in a 5% solution of sulphuric acid for 24 hours. They were then rinsed thoroughly and either allowed to dry in the sun. After drying 100ml of freshly prepared 30% phosphoric acid is added to 50g of sample and kept in furnace for 1hr at 500degree Celsius . Acid content is removed by washing with distilled water[3].

Test of Activated Carbon

Activated carbon was tested by means of a qualitative, visual colorimetric method using a common dye indicator (methyl orange) as a proxy chemical for pesticides and herbicide.Methyl orange, a highly soluble organic dye, was used to preliminarily assess whether the chemical activation had been successful. A dilute methyl orange solution was made to test once it had been chemically activated as mentioned above. Approximately one gram of activated carbon was placed into the test solution and 10 minutes were allowed for adsorption to take place before the treated solution was passed through a common coffee filter. The observed decrease in the colour intensity of the methyl orange solution following this final filtration step was an indication that the carbon has been successfully activated[4].

Batch Study

Synthetic dairy waste water was generated in the laboratory by dissolving 4 g of milk powder (diary sure, Allepy) per litre of distilled water in order to generate a constant wastewater composition throughout the experiments[5] .The characteristics of SDW is given in Table No 1.For each experiment, a known amount of the adsorbent was introduced into 250 ml Erlenmeyer flasks in which 100 ml of the dairy wastewater of COD,BOD and pH determined. This mixture was kept in a stirrer at a constant speed of 150 rpm . The adsorbent was filtered from the treated wastewater sample and was analysed for COD. COD test will be conducted to study the percentage removal of COD from diary waste water using rubber seed shell activated carbon. The batch adsorption experiments are conducted to study optimum time and dosage. The different doses (5 g, 10 g, 15 g, 20g) of adsorbents taken for analysis. The contact time between the adsorbent and the effluent solution is taken as 15min, 30min, 45min, 60 min.

Cod Test Procedure

Wash digestion vessels and sealer with 20% sulphuric acid before first use to prevent contamination. Take 10ml of sample or an aliquot to 10ml with distilled water and add 6ml digestion solution.. Carefully run 14ml sulphuric acid reagent down inside of vessel, so an acid layer is formed under the sample digestion solution layer. Place the sealer and mix the contents thoroughly. Place the vessel with the content in the oven, which is already preheated to 150^0C and reflux for 2 hours. Cool to room temperature and remove the sealer.Put the Teflon covered magnetic stirring bar. Add 1 to 2 drops of ferroin indicator and stir rapidly on magnetic stirrer while titrating with 0.1M FAS.The end point is a sharp colour change from blue green to reddish brown, although the blue green may reappear within minutes. In the same manner reflux and titrate a blank (distilled water) containing the reagents (a volume of distilled water equal to that of sample).Titrate a blank (distilled water) containing the reagents (a volume of distilled water equal to that of sample) for the determination of molarity of the titrant (cold blank)[38].

The percentage removal of COD was calculated using the following relationship:

Percent COD removal= $(C_i-C_t/C_i)*100$……………………………………Eq(1)

where, C_i is the equilibrium COD concentration (mg/l).

The amount of adsorbate adsorbed, qe (mg/g), at any time t was calculated as:

$q_e = (C_o-C_t/w)v$ …………………………………………………………Eq(2)

here, Ct is the COD concentration (mg/l) at time t, V is the volume of the solution (l) and w is the mass of the adsorbent(g)[5]

Parameter	Range
Ph	5.94
Turbidity	1970NTU
BOD	6548mg/l
COD	9600mg/l

Table No 1: parameter of synthetic dairy waste waster

III. Result

Effect Of pH

In biosorption studies pH of the solution is the most important factor influencing the process It influences not only the surface charge of the biosorbent but also the degree of ionization of the organic substances present in the solution and the dissociation of functional groups on the active sites of the sorbent. This shows that organic removal is favoured at lower pH. Due to dissociation of functional groups at higher pH the adsorbent surface carries a net negative charge while at lower pH it carries a net positive charge ,Thus in our study, the pH was kept acidic range keeping other parameters like adsorbent dosage at 10 g/100ml and rotational speed of 150- rpm. The removal was favoured at a lower and there is a sharp decrease in the percentage removal with increase of pH . At low pH values, the RSS activated carbon surface would be protonated and became positive, and the surface will be surrounded by the hydrogen ions, which enhances the interactions between the organic substances and binding sites through attractive forces[2].

Effect On Adsorbent Dosage

The optimum adsorbent dosage was studied at optimum pH and optimum time of 15min, different dosages of 5, 10, 15 and 20g respectively. Figure 1 shows that the COD decreased as the mass of adsorbent dosage was increased. This result indicates that more surface area was made due to increased mass of adsorbent. According to the test result , a significant decrease in value of COD was observed effectively in adsorbent dosage of 10g which satisfy the limit of discharge standard of treated wastewater in to stream and use of the amount of adsorbent can be reduced while meeting the limit , therefore by considering the factor, we can say the optimum dosage as 10g.

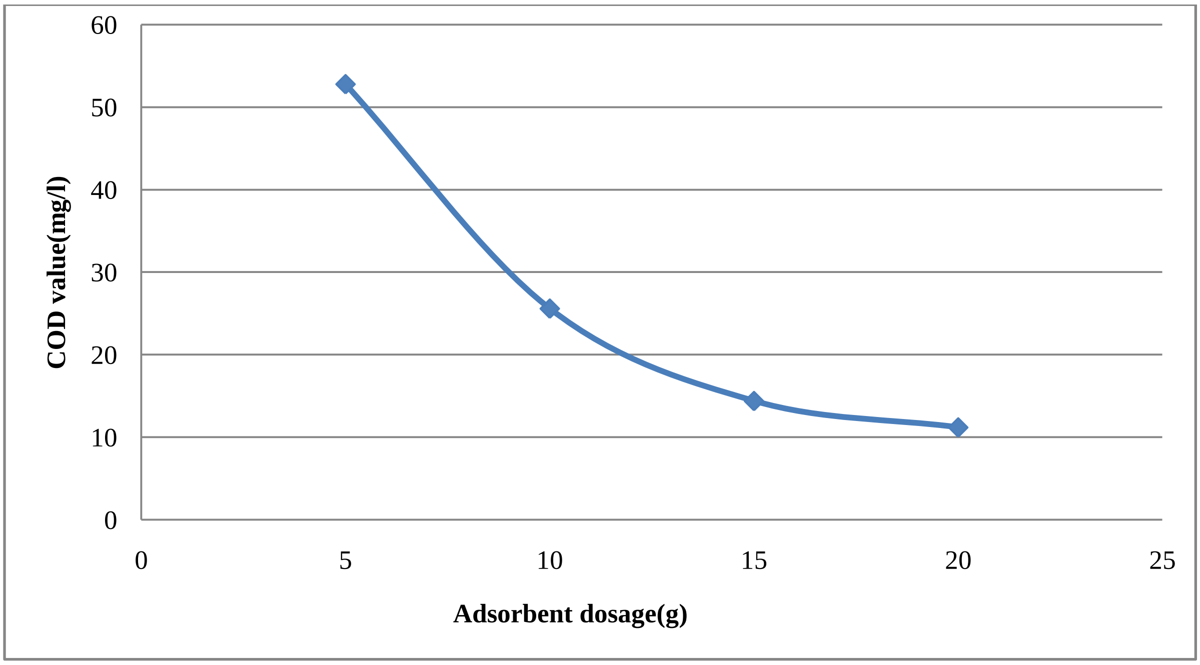

Fig No. 1: graph showing the COD value Vs adsorbent dosage.

Effect of Contact Time

Adsorption Test was conducted by varying contact time for 15min,30min,45min and 60min respectively for a constant adsorbent dosage of 10g. In a 250ml of Erlenmeyer flask 10g of rubber shell activated carbon was added to 100ml sample of synthetic diary wastewater . The suspension is stirred using a magnetic stirrer at a rotation speed of 150rpm .After the time period,the sample is filtered and tested for COD.

According to the standard limit of COD for effluent discharge is 100mg/l for discharging into streams and rivers ,the result indicate that within 15min the COD decreased to 25.6mg/l. Thus we can take the optimum time as 15min.

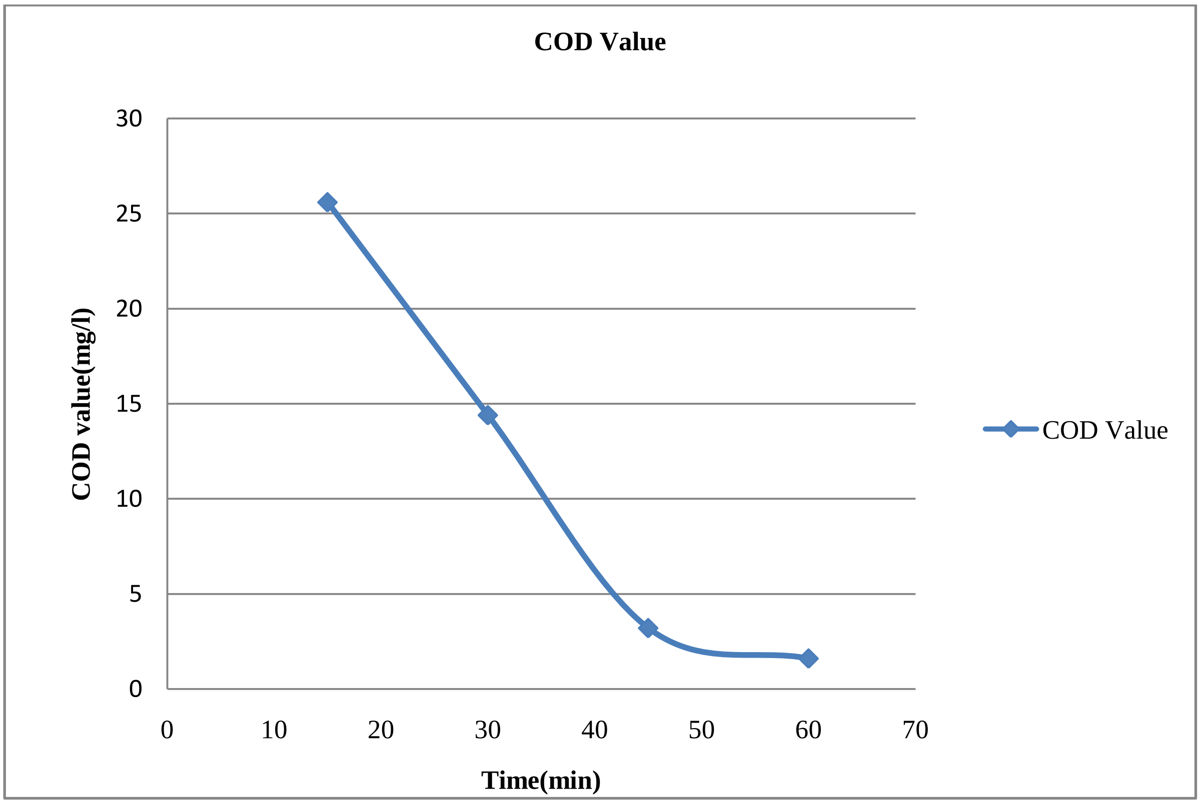

Fig No. 2: Graph showing the COD value Vs time

Adsorption Efficiency

The COD of dairy wastewater samples treated with RSS activated carbon is calculated .It indicates a continuous removal of organic pollutants by adsorption on the surface of rubber seed shell activated carbon. Fig No.2 showed the COD concentration decreased with time .The percentage removal of COD was calculated using Eq.(2), and removal verses time is shown in Fig No.3. Fig No. 3 shows that, the percent removal increases gradually as the time is increased. It can be attributed to the fact that more time becomes available for the organic substances to stick with the adsorbent surface, as well as surface adsorption increases with time. At the chosen conditions, the maximum percentage reduction in COD using RSS activated carbon was found to be above 90%. RSS was found to be highly efficient for removing Pollutant from wastewater. A percentage removal of above 90% within 15min using a dose of 10g/100 ml was obtained (at pH below 6.0).

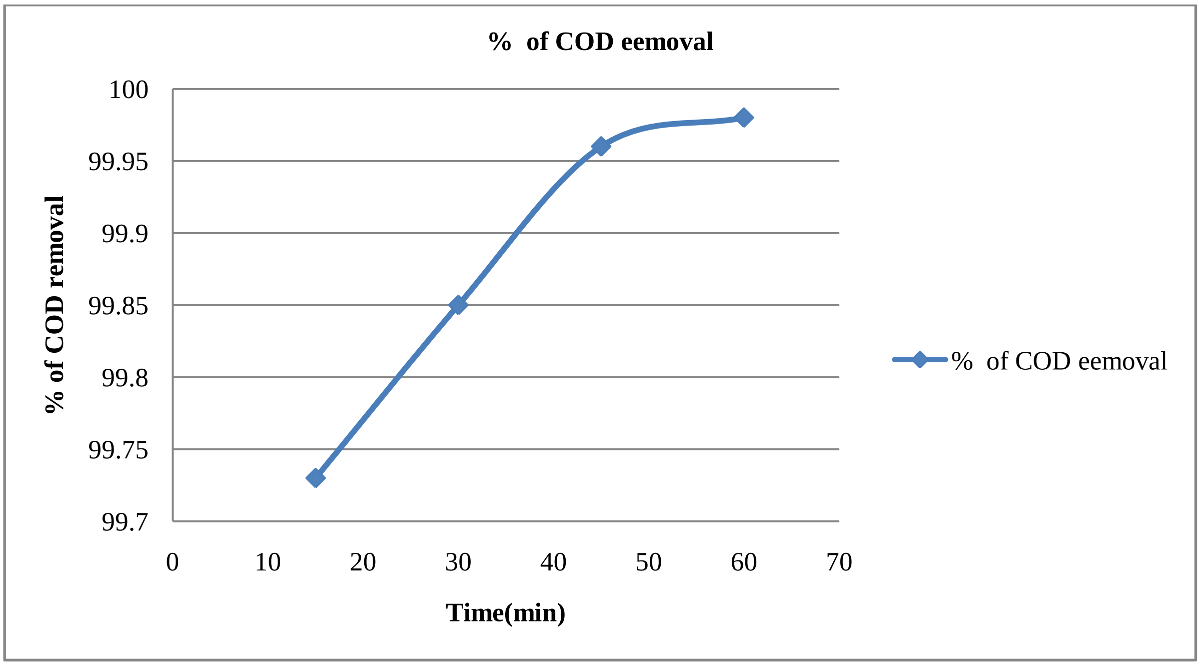

Fig No. 3: Graph showing the %COD removal Vs time

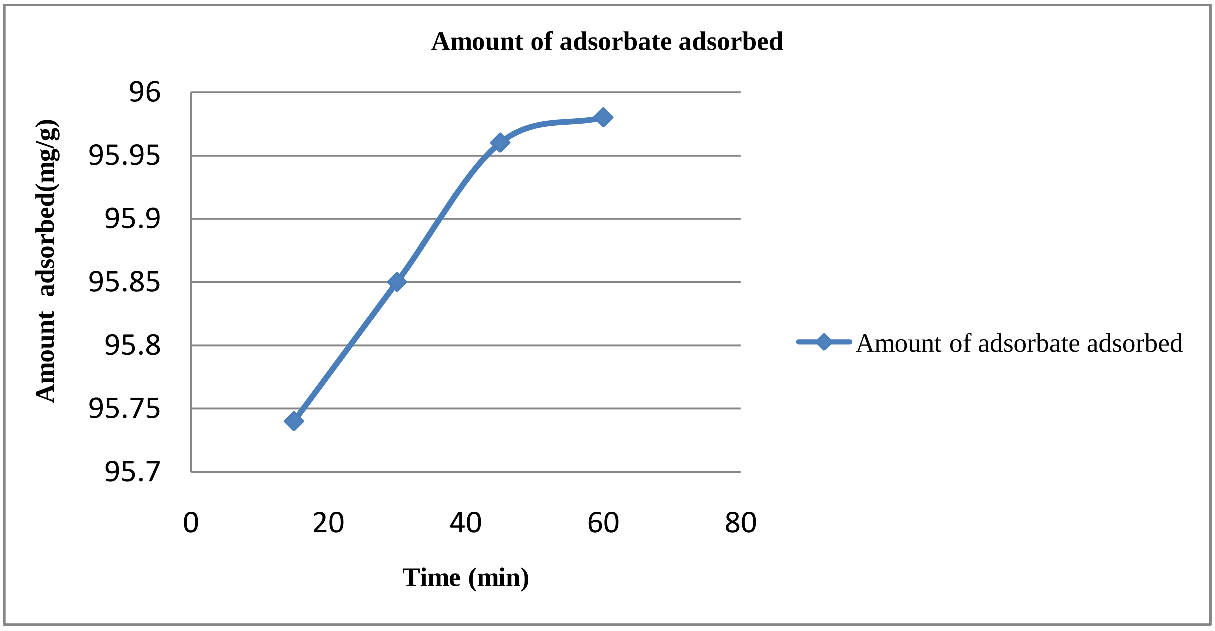

Fig No. 4: Graph showing the amount of adsorbate adsorbed Vs time

Interpretation of Data

Regresssion analysis was done using Microsoft excel version 2010,the correlation obtained was -0.9623,if r value obtained as negative, we can say their exist a relationship between COD and time. As time increases, the COD value decreases . In statistical hypothesis, we assume that the null hypothesis is correct until we have evidence to suggest. After performing the test, there are two possible outcome.

When p value is less than or equal to significance level, reject the null hypothesis. The data favors the alternative hypothesis.

When p value is greater than significance level, fail to reject the null hypothesis. Result is not significant.

The anova test result shows that the p value is 0.080443, which is less than the significance value F of value 0.260147,thus the result is statically significant.

ANOVA

	df	SS	MS	F	ignificance F
Regression	1	378.9474	378.9474	5.333333	0.260147
Residual	1	71.05263	71.05263		
Total	2	450			

	Coefficients	andard Err	t Stat	P-value	Lower 95%	Upper 95%	ower 95.0%	Jpper 95.0%
Intercept	57.63158	7.321278	7.871792	0.080443	-35.3941	150.6572	-35.3941	150.6572
25.6	-1.97368	0.85463	-2.3094	0.260147	-12.8328	8.885424	-12.8328	8.885424

Fig No.5: ANOVA test result.

Adsorption Isotherm

Experimental adsorption data were subjected to Langmuir and the Freundlich adsorption isotherms to evaluate adsorption.Freundlich equilibrium constants were determined from the plot of Inq_e versus InC_e from Fig No.7, on the basis of the linear form of Freundlich equation. 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; if n>1, then adsorption is a physical process[6]. The n value in Freundlich equation was found to be less than 1, showing it as chemical process.The linear plot of specific adsorption (Ce/qe) against the equilibrium concentration (Ce)(Fig No.6) shows that the adsorption obeys the Langmuir model. The constants b and q_{max} relate to the energy of adsorption and maximum adsorption capacity, and their values are obtained from the slope and interception of the plot. Where C_e(mg/l) is the COD concentration in the solution at equilibrium, q_e (mg/g) is the amount adsorbed per unit mass of adsorbent at equilibrium time, q_0(mg/g) and b(l/mg) are the Langmuir constants related to maximum monolayer adsorption capacity and energy of adsorption that is affinity of binding sites respectively.Further, separation factor which is defined below was also applied to get insights of the isotherm[6].

$$\mathbf{R_L} = \frac{\mathbf{1}}{\mathbf{1+bC_0}} \quad \text{...Eq(3)}$$

Where C_0 (mg/l) is the initial concentration and *b* (l/mg) is the Langmuir constant. Four $R_L$scenarios are possible for any adsorption: R_L>1 (unfavourable), R_L= 1(linear), R_L = 0 (irreversible) and 0 < R_L < 1 (favourable). Fig No.7 represent plot of Ce/$q_e$versus $C_e$for the adsorption[6]. R_L = 0.129 indicates that the adsorption is favourable[6]. The experimental data showed a good fit to Langmuir model with correlation coefficient R^2 = 0.997,indicating that the model was appropriate to describe the adsorption process using RSS activated carbon.

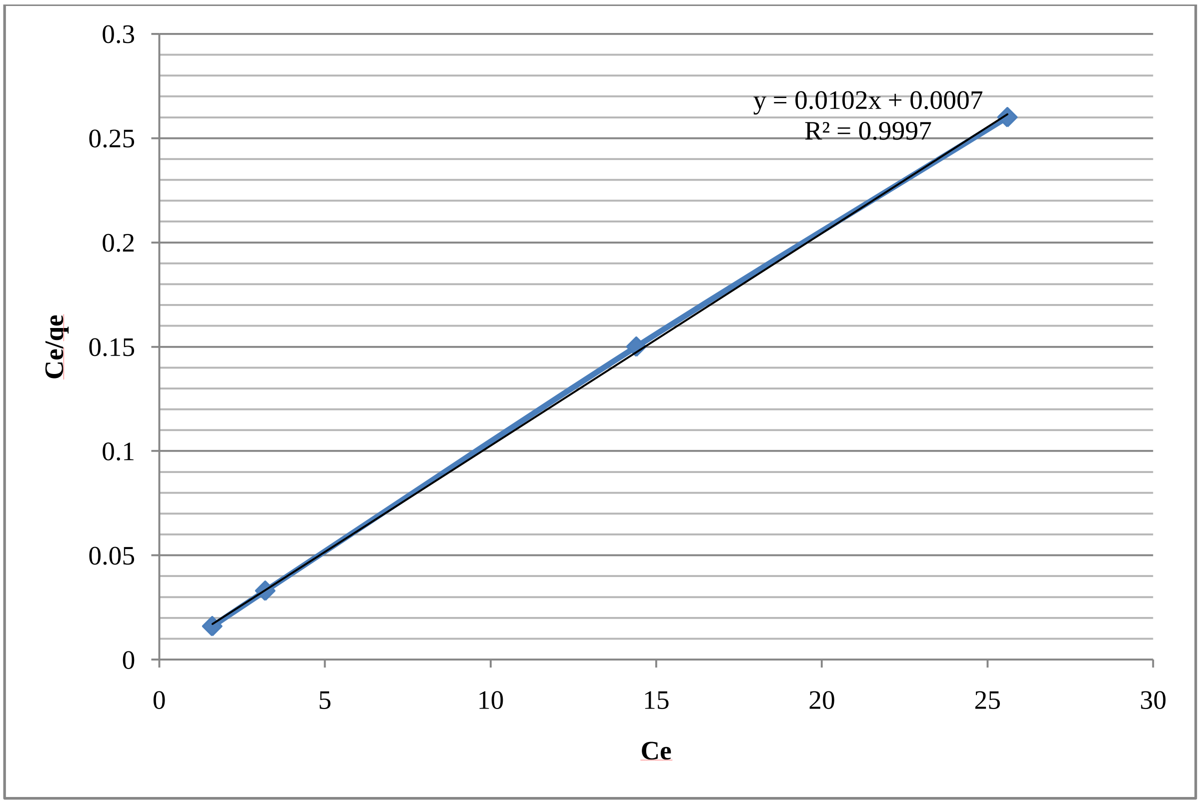

Fig 4.6: Langmuir isotherm showing the variation of adsorption (Ce/qe) against the equilibrium concentration(Ce) for adsorption

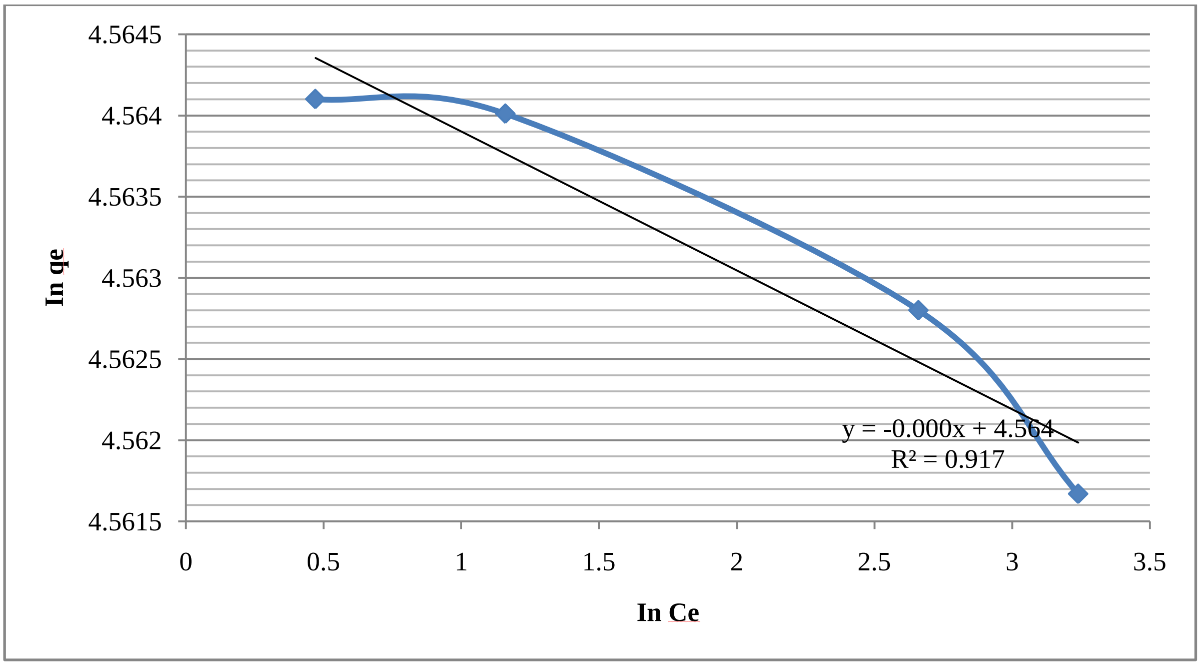

Fig No.7:Freundlich isotherm representing variation of In(qe)with respect to In(Ce) for adsorption

IV. Discussion

This paper demonstrates that activated rubber seed shell can be used as adsorbents for reducing COD in wastewater resulting from dairy industry. The removal of organic substance from the dairy wastewater was studied by investigating the effect of time and adsorbent dosage. The maximum percentage reduction in COD using Rubber seed shell activated carbon found to be above 95%. Rubber seed shell activated carbon was found to be highly efficient for removing pollutant from diary wastewater. A percentage removal of 99% within 1hours using a dose of 10g/100 ml was obtained (at pH below 6.0).The permissible limit of COD is less than

100mg/l .According to IS:2490,Part-I-1981, the value obtained after the test meets the maximum tolerance limits for industrial effluents discharged in to onland surface water and marine/coastal area . The optimum time duration required for removal was 15 minutes.. The correlation coefficient for Freundlich isotherm model was obtained as R^2 = 0.9176. In the case of Langmuir isotherm separation factor R_L obtained is less than 1, which indicates that the adsorption process is chemical process. Langmuir isotherm plot is fitted with correlation coefficient R^2 = 0.9997. These findings demonstrated that Rubber seed shell activated carbon is suitable and efficient adsorbent for the adsorptive treatment of dairy wastewater. Thus, the present study concludes that these adsorbents could be employed as eco-friendly adsorbents

V. Conclusion

The present study concludes that activated carbon made from the rubber seed shell can be used as a low cost adsorbent material in adsorption process for the removal of organic pollutants in dairy waste water .

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