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Comparative study of Removal of Malathion from Waste-Water by using Natural Adsorbent

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ABSTRACT

This work deals with the possibility of using adsorption method for the removal of Malathion. The effect of various operational parameters of the removal efficiency was investigated and optimized. The removal of Malathion using drumstick peel powder and bagasse fly ash was affected by the adsorbent dose, initial Malathion concentration, initial pH, contact time. The determination of removal Malathion from waste- water by using physicochemical characteristics like COD, BOD, TSS and Turbidity. The optimum contact needed to reach equilibrium was found to be 180 min. Maximum removal takes place at pH 12. The optimum adsorbent dose is 0.2 g/500ml of drumstick peel powder and 1g/500ml of bagasse fly ash. Optimum initial Malathion concentration 500ppm. The synthetic waste water by using drumstick peel powder & bagasse fly ash malathion removal with respect to COD, BOD, TSS , Turbidity. They were 69.56%, 88.57%, 96.66%, 96.15%and 65.94%, 85.71%, 95%, 95.93% respectively, under optimum condition. The adsorption equilibrium of the system at constant time was model by Langmuir and Freundlich isotherms. Freundlich model showed excellent correlation with experimental data. For kinetic study, the adsorption process fitted the pseudo-second order model. The thermodynamic study in temperature was constant. The Freundlich model in thermodynamic study in waste-water characterization of COD, BOD, TSS and Turbidity exothermic reaction of nature is same as DPP and BFA. A comparison of the adsorption capacity of drumstick peel powder and bagasse fly ash adsorbents shows that drumstick peel powder can be used for the removal of malathion from waste-water solutions.

Keywords: Adsorption, Malathion, Drumstick peel powder, Bagasse fly ash.

1. INTRODUCTION

Water pollution by organic and inorganic compounds is of great public concern. Adsorption technology is currently being used extensively for the removal of such pollutants from wastewaters. Among the various adsorbent systems available for the removal of pollutants from wastewater, activated carbon is being widely used in developed countries [1-3]. However, the high cost of activated carbon and difficulties associated with its regeneration [4], limits its use in developing countries. This has led to interest in the development of low cost alternatives to activated carbon. Thus a variety of materials, as adsorbents for the removal of different pollutants, have been tried by various workers [5,6]. All the materials studied have their own advantages and limitations and therefore, there is still a need for developing low cost adsorbents.

Malathion is very dangerous and harmful because of their toxic and carcinogenic nature. Poison information centre in National School of Occupational Health (NIOH), Ahmadabad, India, has reported that organophosphorous compounds are responsible for maximum number of poisoning (73%) among all agricultural pesticides. Patients of acute organophosphorus poisoning have been reported to suffer from problems like vomiting, nausea, miosis, excessive salivation, blurred vision, headache, giddiness, and disturbance in consciousness. In case of malathion, which is one of the widely used organophosphorus pesticide, almost all the effects observed are due to its active metabolite malaxon on the nervous system or is secondary to its primary action. Malathion is moderately toxic to birds and has a wide range of toxicity in fish and other aquatic invertebrates and is highly toxic to honeybees. The pesticides permissible concentrations limit in water for human consumption is 0.1 g/l. [7-12]

Malathion is the most commonly used pesticides in India and is very toxic. The pesticides contaminate water through agricultural, domestic and industrial activities and therefore their removal is important. This work aims to study the possibility of using drumstick peel powder (DPP) and bagasse fly ash (BFA) adsorbent for removal of malathion from synthetic waste water by adsorption method.

2. MATERIALS AND METHOD

2.1. Reagent and solution

All the reagents used in this study are of analytical grade. All glasswares used were of Borosil. Distilled water was used for making the synthetic samples. Technical grade malathion of 50 % (E.C.) pure. Acetone, Magnese Sulphate, Alkali iodide azide, Starch indicator, Sodium thiosulphate pentahydrate, $K_2Cr_2O_7$, Mercury Sulphate, Ferroin indicator, Ammonium iron Sulphate hexahydrate. Standard base of 0.1N NaOH and acid 0.1N H_2SO_4 solutions were used for pH adjustment.

2.2. Preparation of adsorbent

The Bagasse fly ash collected from Vasantdada Sahakari Sakhar Karkhana (Vithewadi, Nasik, Maharashtra, India) is used as a natural adsorbent. The drumsticks are collected from market. Remove peels kept in sunlight 2-3 days for drying. Dry peels crush in mixer grinder to create fine powder is used as a natural adsorbent. It is thoroughly washed with hot water (70°C), dried, and sieved by using standard sieves (IS 437 1979). The fractional sieve analysis of the particles of BFA and DPP were done and found as the average particle size of 425 μm and 150 μm (equivalent U.S. standard test sieves). Particle was used for the adsorption studies. The physico-chemical characteristics of DPP and BFA as shown in table 1 and 2 were determined in GEO-CHEM Laboratories Pvt.Ltd Mumbai.

3. Experiment Procedure

All the experiment is carried out in batch processes. A stock solution of Malathion is prepared by dissolving 5ml pesticides + 2 ml acetone add in 9993 ml of distilled water to create synthetic wastewater to get stock solution of 1000 ppm. A conventional jar test apparatus was used in the experiments to adsorbate sample of Malathion synthetic waste-water using natural adsorbent. It was carried out as a batch test, accommodating a series of six beakers together of 1 litre capacity with six spindle steel paddles. Before operating the jar test, the sample was mixed homogenously. This study consists of batch experiments involving rapid mixing. The apparatus consists of six beakers to be agitated simultaneously. 500 ppm of the synthetic waste-water samples was put in to each 6 one-litter beakers and placed under jar test apparatus. The required dose of drumstick peel powder 150 μm and bagasse fly ash 425 μm average size particle was added. The paddles were inserted in the jars,

Table - 1: Physicochemical Characteristics of BFA

Test	Method/Technique	Results
Particle size	IS 437 1979 sieves shaker	425μm
Proximate analysis: Moisture (%)	Ref.ASTM D-3173-11	0.29
Volatile matter (%)	Ref.ASTM D-3174-12	1.10
Ash (%)	Ref.ASTM D-3175-11	98.40
Fixed carbon (%)	Ref.ASTM D-3172-13	0.21
Bulk density (gm/cc)	REF.IS:33-2002	0.631
NS analysis:N (%)	Ref.ASTM D-4239-14	0.01
S (%)	Ref.ASTM D-5373-14	0.01
SiO_2 (%)	IS:1727-1967	75.95
MgO (%)	IS:1727-1967	3.37
CaO (%)	IS:1727-1967	3.24
Al_2O_3 (%)	IS:1727-1967	3.17
Fe_2O_3 (%)	IS:1727-1967	2.39
Loss on ignition (%)	IS:1727-1967	1.80

Table - 2: Physicochemical Characteristics of DPP		
Test	**Method/Technique**	**Results**
Partical size	IS 437 1979 sieves shaker	150 µm
Loss on ignition (%) @ $1000^{0}C$	REF.IS:1760(PART 1)1991	96.80
Fe_2O_3 (%)	ICP	0.084
Al_2O_3 (%)	ICP	0.27
CaO (%)	ICP	1.17
MgO (%)	ICP	0.42
SiO_2 (%)	REF.IS:1760(PART2) 1992	1
Proximate analysis: Bulk Density(gm/cc)	REF.IS:33-1992	0.22
Moisture(%) @$105^{0}C$	REF.IS:1350(PART 1)1984	11.92
Volatile Matter (%)	REF.IS:1350(PART 1)1984	71.18
Ash (%)	REF.IS:1350(PART 1)1984	1.71
Fixed carbon (%)	REF.IS:1350(PART 1)1984	15.19
NS analysis:N (%)	REF.IS:6092(PART 2)1985	1.46
S (%)	REF.IS:1350(PART 4)1984	0.005

the apparatus was switched on and the whole procedures in the jar test were conducted of rapid mixing stirrer at a speed of 170 rpm for 180 minutes. Series of experiments are using operational parameter conducted to effect of adsorbent dose, initial Malathion concentration, effect of pH and contact time. After the agitation being stopped, the suspensions were allowed to settle. The samples are filtered to remove any fine particles and the concentration of Malathion in solution is then analysed by using HACH spectrophotometer (Total Suspended Solid:TSS), turbidity meter, (Chemical Oxygen Demand:COD) reflux condenser, (Biological Oxygen Demand:BOD) incubator.

4. RESULTS AND DISCUSSION

4.1. Effect of adsorbent dose

The effect of the adsorbent dose on removal of malathion is as shown in Figure 1 and figure 2. The adsorption of malathion increases in the drumstick peel powder range 0.2–1.2 g/500ml and bagasse fly ash range 0.5-5g/500ml then becomes constant indicating that DPP is 0.2g/500ml and BFA is 1 g/500ml of adsorbent is sufficient for the optimum removal of malathion. The synthetic waste water by using drumstick peel powder & bagasse fly ash filtrated sample of water to test on different water characteristics in laboratory was shown the result for malathion removal with respect to COD, BOD, TSS , Turbidity. They were 60.94%, 77.14%, 86.66%, 84% and 60.14%, 58.50%, 63.33%, 78.62% respectively.

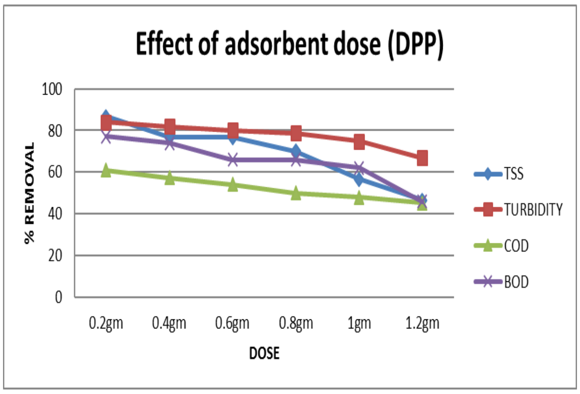

Figure - 1: Effect of adsorbent dose on the removal of malathion by using drumstick peel powder (t = 3 hr., malathion initial concentration =500 ppm at 298 K).

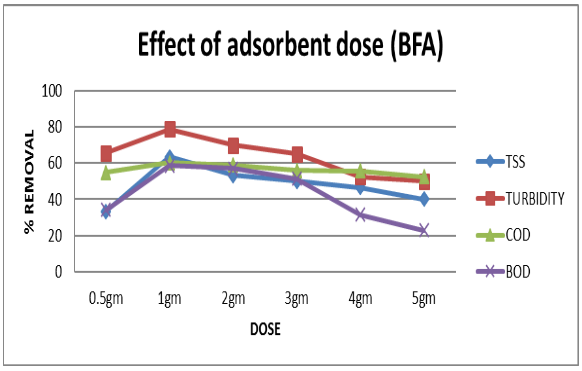

Figure - 2: Effect of adsorbent dose on the removal of malathion by using bagasse fly ash (t = 3 hr., malathion initial concentration =500 ppm at 298 K).

4.2. Effect of initial concentration

The effect of malathion initial concentration (500ppm -900 ppm) at fixed temperature 298 K on the equilibrium uptake of malathion by the DPP at m_{DPP} = 0.2g/500ml & BFA at m_{BFA}=1g/500ml and t =3hr was studied and a plot of the removal of malathion and the sorptive uptake of malathion by DPP and BFA versus malathion initial concentration with at fixed temperature. The synthetic waste water by using drumstick peel powder & bagasse fly ash filtrated sample of water to test on different water characteristics in laboratory was shown the result for malathion removal with respect to COD, BOD, TSS ,Turbidity. They were 62.98%, 79%, 86.66%, 86.25%and 62.86%, 60.38%, 63.33%,84.15% respectively. The effect of initial concentration on removal of malathion as shown in figure 3 and 4.

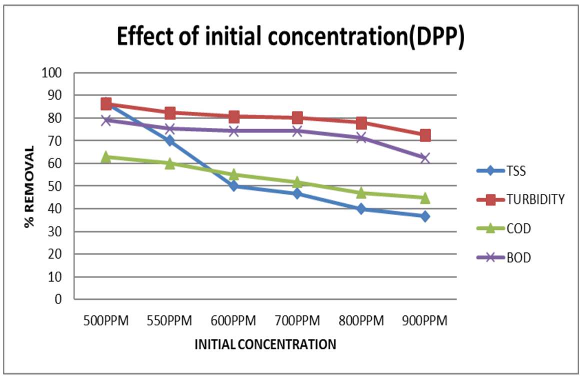

Figure - 3: Effect of initial concentration on the removal of malathion by using drum stick peel powder (t = 3 hr., DPP Dose = 0.2 gm at 298 K).

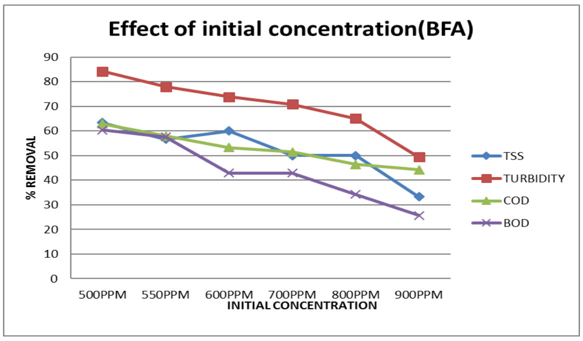

Figure - 4: Effect of initial concentration on the removal of malathion by using bagasse fly ash (t = 3 hr., BFA Dose = 1 gm at 298 K).

4.3 Effect of initial pH

The pH value can affect the structural stability of malathion and its concentrations. Effects of initial pH (pH_0), range 2-12, on the removal of malathion as shown in figure 5 and 6. At fixed 298 K for malathion initial concentration 500ppm, m_{DPP} = 0.2 g/500ml & m_{BFA} =1g/500ml. Equilibrium was attained in t = 3 h. The maximum affinity to malathion was found at pH_o~12. The synthetic waste water by using drumstick peel powder & bagasse fly ash filtrated sample of water to test on different water characteristics in laboratory was shown the result for malathion removal with respect to COD, BOD, TSS , Turbidity. They were 65.04%, 86%, 93.33%, 92.96% and 63.69%, 83.33%, 93.33%, 86.66% respectively.

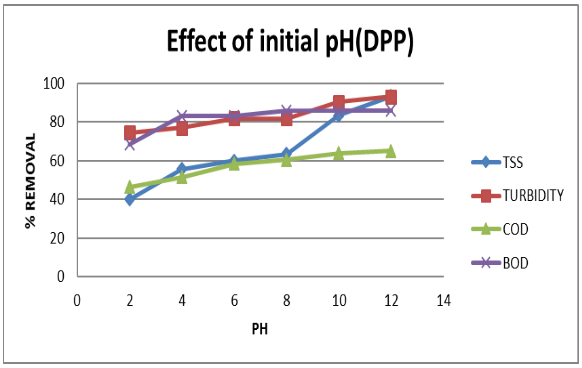

Figure - 5: Effect of initial pH on the removal of malathion by using drum stick peel powder (t = 3 hr., DPP Dose = 0.2 gm, malathion initial concentration =500ppm at 298 K).

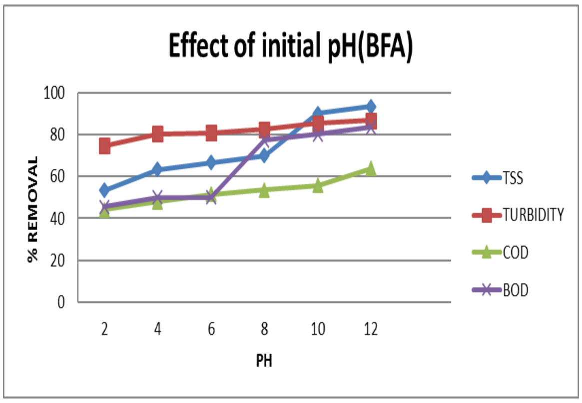

Figure - 6: Effect of initial pH on the removal of malathion by using bagasse fly ash (t = 3 hr., BFA Dose = 1 gm, malathion initial concentration =500ppm at 298 K).

4.4 Effect of contact time

The effect of contact time on the removal of malathion by DPP for m_{DPP} =0.2g/500ml and BFA for m_{BFA} = 1 g/500ml and malathion initial concentration 500ppm as shown in figure 7 and 8. Initially the adsorption rate of malathion onto DPP & BFA is found to be instantaneous because of the availability of more adsorption sites. The steady state adsorption is assumed after 30 min.,60min.,90min.,120min.,150min.& 180 min. and further experiments were carried out at t=3 h(180 min.). The synthetic waste water by using drumstick peel powder & bagasse fly ash filtrated sample of water to test on different water characteristics in laboratory was shown the result for malathion removal with respect to COD, BOD, TSS , Turbidity. They were 69.56%, 88.57%, 96.66%, 96.15%and 65.94%, 85.71%, 95%, 95.93% respectively.

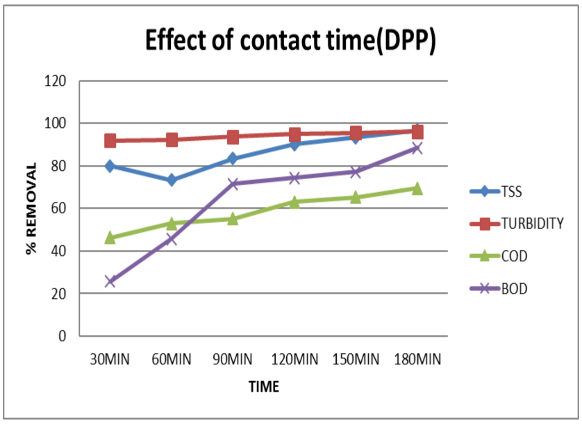

Figure - 7: Effect of contact time on the removal of malathion by using drumstick peel powder (t = 3 hr., DPP Dose = 0.2 gm, pH=12 ,malathion initial concentration =500ppm at 298 K).

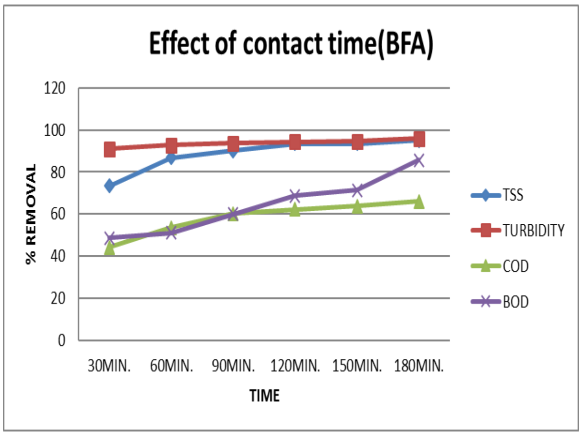

Figure - 8: Effect of contact time on the removal of malathion by using bagasse fly ash (t = 3 hr., BFA Dose = 1 gm,pH=12 ,malathion initial concentration =500ppm at 298 K).

Comparative studies of adsorbent

Figure 9 shows drumstick peel powder adsorbent more efficient than bagasse fly ash under optimum condition to removal of malathion.

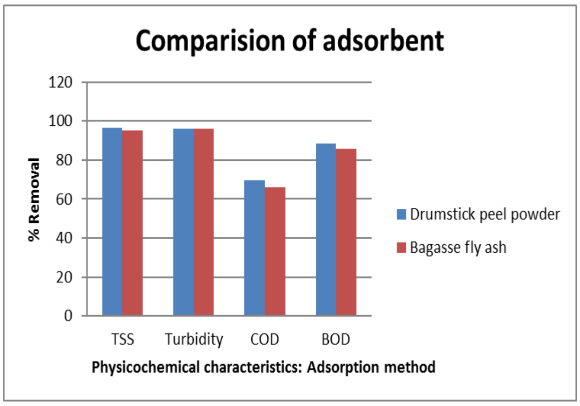

Figure – 9: Comparative studies: Malathion removal of adsorption method.

4.6. Adsorption isotherm

Adsorption isotherm gives an idea about the feasibility of an adsorbate–adsorbent system. In this work Langmuir and Freundlich were tested for describing the experimental results. The Langmuir model can be expressed as [13]:

(Non-linear form)

$$q_e = [q_m K_L C_e / (1 + K_L C_e)] \quad (1)$$

or

$$1/q_e = (1/ K_L q_m C_e) + (1/q_m) \quad (2)$$

or

$$C_e/q_e = (1/K_L q_m) + (C_e/q_m) \quad (3)$$

Where, q_m = monolayer sorption capacity (mg /L) and is a constant,

K_L = constant related to the free energy of sorption.

From the linear plot of an experimental sorption data C_e / q_e Vs C_e at 298 K. The isotherm parameters K_L and q_m are determined at fixed 298K. All these values are shown in Table 3. The vales of K_L increases and q_m decreases with increasing the temperature. K_L indicates the affinity of the adsorbents to the adsorbate. High K_L indicates a higher affinity. The maximum adsorption capacity of the adsorption (q_m) is the monolayer saturation at equilibrium.

In the Freundlich adsorption isotherm, an extent of adsorption is directly proportional to pressure at low pressure. And it is independent at high pressure. Therefore at intermediate value of pressure, adsorption is a directly proportional to the pressure raised to power *1/n* where, *n* is a variable whose value is greater than one. Using constant of proportionality *k* also known as adsorption constant.

The Freundlich equation as:

$$q_e = k_f c_e^{(1/n)} \quad (4)$$

or

$$\ln q_e = \ln k_f + (1/n) \ln C_e \quad (5)$$

Where, k_f is the sorption capacity of the adsorbent (mg/g), *1/n* is a constant which gives the intensity of the adsorption.

Hence, the linear representation of the Freundlich model is plot of qe Vs C_e at 298 K. The isotherm parameters such as k_f and *1/n* are shown in Table 3. Higher the value of 1/n, highest is the affinity between the adsorbate and adsorbent, and the heterogeneity of the adsorbent sites. The *1/n* values indicate the relative distribution of energy sites and depend on the nature and strength of the adsorption process. The Freundlich isotherm does not predict the saturation of the adsorbent surface

Table - 3: Adsorption isotherm parameters for removal of malathion at C_0 = 500 mg/L								
Isotherm equation	**Langmuir isotherm model $qe = q_m K_L C_e /(1+K_L C_e)$**							
Adsorbent	DPP				BFA			
Physico- Chemical characteristics	TSS	Turbidity	COD	BOD	TSS	Turbidity	COD	BOD
Constants K_L (L/ mg)	-0.5393	0.6787	-3.9623	1.40256 x10^{-3}	-0.513347	0.5974	1.6504	-0.07072
q_m (mg /g)	-104166.67	100000	-952.380	20000000	-2000000	23255.82	454.54	-16666.67
R^2	0.994	0.9999	0.9873	0.9122	0.9947	0.9997	0.9647	0.5223
Isotherm equation	**Freundlich isotherm model $q_e = K_F C^{1/n}$**							
Adsorbent	**DPP**				**BFA**			
Physico- Chemical characteristics	TSS	Turbidity	COD	BOD	TSS	Turbidity	COD	BOD
Constants K_F (L/ mg)	75000	78000	24500	8750	15000	15500	4900	1750
n	-0.4	-4 x10^{-4}	-4 x 10^{-4}	-4 x10^{-5}	-2 x10^{-3}	-0.0347	-2 x10^{-3}	-2 x10^{-3}
1/n	-2.5	-2500	-2500	-25000	-500	-29.34	-500	-500
R^2	1	1	1	1	1	1	1	1

Table - 4: Pseudo second order kinetic parameters at malathion initial concentration = 500ppm							
Pseudo second order kinetic parameters							
Adsorbent		**DPP**			**BFA**		
Physico-chemical characteristics	Malathion Initial concentration	h (mg/l min.)	K_S (g/l min.)	q_e (mg/l)	h (mg/l min.)	K_S (g/l min.)	q_e (mg/l)
COD	500 mg/l(ppm)	—	—	13586.01	500	939548950.7	1370.80
BOD		333.33	3.7 x 10^{10}	10536.39	116.28	185531147.3	1263.16
TSS		2222.22	4.37 x 10^{12}	44355.143	1219.52	2.97 x10^{11}	15615.38
Turbidity		—	—	70003.5	—	—	7609.34

Table - 5: Thermodynamics parameters for removal of malathion by using physicochemical characteristics

Isotherm	**Langmuir isotherm model(298 K)**							
Adsorbent	**DPP**				**BFA**			
Physico-Chemical characteristics	TSS	Turbidity	COD	BOD	TSS	Turbidity	COD	BOD
$\Delta G°$ (KJ/mol)	29.95502	-30.5247	34.8959	-15.2085	29.8327	-30.2085	-32.72608	24.9214
ΔH° (KJ/mol)	31.48	-31.4837	38.3069	-32.5526	31.4847	-31.4848001	-31.4847	31.4845
ΔS° (KJ/mol K)	0.105637	-0.10565	0.1285466	-0.109236912	0.105653355	-0.105653691	-0.105653355	0.105652684
Reaction of Nature	En[a].	Ex[b].	En[a].	Ex[b].	En[a].	Ex[b].	Ex[b].	En[a].
Isotherm	**Freundlich isotherm model(298K)**							
Adsorbent	**DPP**				**BFA**			
Physico-Chemical characteristics	TSS	Turbidity	COD	BOD	TSS	Turbidity	COD	BOD
$\Delta G°$ (KJ/mol)	-59.29610	-59.3933	-56.5242	-53.9732	-55.3086	-55.3898	-52.5366	-49.9857
ΔH° (KJ/mol)	-31.47	-31.4845	-31.4846	-31.4847	-31.4847	-31.4847	-31.4845	-31.4847
ΔS° (KJ/mol K)	-0.105604	-0.1056526	-0.10565302	-0.105653355	-0.105653355	-0.105653355	-0.105652684	-0.105653355
Reaction of Nature	Ex[b].	Ex[b].	Ex[b].	Ex[b].	Ex[b].	Ex[b].	Ex[b].	Ex[b].

Note: Endothermic: a;Exothermic:b

by the adsorbate. The k_f value can be taken as a relative indicator of the adsorption capacity. The magnitude of k_f showed the higher uptake at higher temperature indicating endothermic in nature of the adsorption process.

It can be observed from the Table 3 that the Freundlich adsorption isotherm could be used to represent the equilibrium adsorption at contact time.

Where, h is the initial sorption rate (mg/g min) at $t \rightarrow 0$. The value of h and q_e can be determined experimentally from the slope and intercept of the plot of t/q_t versus t. k_s value can then be estimated from the value of h.

Hence, the linear nature of the graph for pseudo-second order kinetic model at malathion initial concentration 500 ppm and 298 K and hence the data are fitted with this model. The kinetic parameters, k_s and q_e are determined for 500 ppm and shown in Table 4.

It can be observed from the Table 4 that for kinetic study, the adsorption process fitted the pseudo-second order model.

The Thermodynamic parameters such as Gibbs free energy change of the adsorption process is related to the adsorption equilibrium constant by the classical Van't Hoff equation [16]:

$$\Delta G°_{ads} = -RT \ln K_{ads} \quad (10)$$

The Gibbs free energy change is also related to the change in the entropy and the heat of adsorption at a constant temperature as given by the equation:

$\Delta G°ads = \Delta H° - T\Delta S°$ (11)

The above two equations give

$\ln K_{ads} = -(\Delta G°_{ads}/ RT) = (\Delta S°/R) - (\Delta H°/R)(1/T)$ (12)

$\ln k = \ln k' - \Delta H^o/RT$ (13)

$\Delta S° = \Delta H^o/T$ 14)

Where,

$\Delta G°_{ads}$ = Free energy change (kJ/ mol),

$\Delta H°$ = Change in enthalpy (kJ/ mol),

$\Delta S°$ = Entropy change (kJ/ mol K),

K_{ads} = Equilibrium constant of interaction between the adsorbate and the AGCA surface,

T = Absolute temperature (K) and

R = Universal gas constant (8.314 J/ mol K).

Table 5 shows Van't Hoff's equation for the Langmuir and Freundlich adsorption isotherm, if positive $\Delta H°$ and $\Delta S°$ value indicates that the overall-sorption process is endothermic and if it is negative the process is exothermic in nature. Negative values of $\Delta S°$ show a decreased randomness or increased order at the solid/solution interface, with some structural changes in the adsorbate, adsorbent and malathion affinity to the DPP and BFA surface. But in this case temperature is constant at 298 K.

$\Delta G°_{ads}$ values at fixed 298 K were determined and also shown in Table 5. For significant adsorption to occur, the free energy changes of adsorption, $\Delta G°$, must be negative. So, from Table 5, $\Delta G°$ values were negative indicating that the sorption process lead to decrease in Gibb's free energy. It also indicates the feasibility and spontaneity of the adsorption process. But the situation becomes less favorable as $\Delta G°$ increases in the positive direction. It should be borne in mind that many reactions with positive values of $\Delta G°$ are certainly feasible from standpoint of industrial operation.

It can be observed from the Table 5 that the Freundlich adsorption isotherm model in exothermic reaction of nature is same as DPP and BFA at constant temperature.

5. CONCLUSION

Adsorption process was tested for the removal of malathion from the synthetic waste water. The effect of various operational parameters on the malathion removal efficiency was investigated and optimized. The synthetic waste water by using drumstick peel powder & bagasse fly ash filtrated sample of water to test on different water characteristics in laboratory was shown the result for malathion removal with respect to COD, BOD, TSS , Turbidity. They were 69.56%, 88.57%, 96.66%, 96.15% and 65.94%, 85.71%, 95%, 95.93% respectively. It is observed that drumstick peel powder more efficient than bagasse fly ash to removal of Malathion.

The obtained results showed that pseudo-second-order equation model was found to be in a good agreement with the experimental results. Adsorption isotherm study showed that the Freundlich model fitted the experimental data well as compared to other models. Thermodynamic study in observed Langmuir model in waste-water characterization COD, TSS is endothermic reaction of nature and exothermic reaction of nature is Turbidity, BOD in DPP. The BFA adsorbent using of Langmuir model in COD, Turbidity is exothermic reaction of nature and BOD, TSS is endothermic reaction of nature. The Freundlich model in thermodynamic study in waste-water characterization of COD, BOD, TSS and Turbidity exothermic reaction of nature is same as DPP and BFA. It also indicates the feasibility and spontaneity of the adsorption process.

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