Adsorptive Removal of Sodium Lauryl Sulfate, Cetyl Trimethyl Ammonium Bromide & Triton X-100 using Non-Conventional Adsorbent through Chemical Kinetics & Thermodynamics: A Comparative Study

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Abstract : The object of present study is to examine the potential use of **c**arbonized **o**range **p**eel (COP) as an economical and non-conventional adsorbent for adsorptive removal of anionic, cationic and non-ionic surfactants from municipal effluent. Comparison has done among fresh COP, alkali and acid activated recycled COP. In addition, investigation of the chemical kinetics and thermodynamics has also been conducted. Pseudo second order rate equation is used to determine the equilibrium amount adsorbed and equilibrium concentration for different initial concentrations. Result shows that the chemical kinetics of adsorption of sodium lauryl sulfate (SLS), cetyltrimethyl ammonium bromide (CTAB) and Triton X-100 (TX-100) have the values of rate constant(K): 4.035 x 10^{-3}, 5.0 x 10^{-3} and 4.52 x 10^{-3} g.mg^{-1}min. respectively. The negative numerical values of Standard Gibb's Free Energy Change (ΔG^0) of SLS, CTAB and TX-100 are -2.969, -2.776 and -2.863 kJmol^{-1} respectively, which show that the adsorption of all the three surfactants on fresh and treated carbonized orange peel (COP) is found to be spontaneous, feasible and exothermic in nature.

***Index Terms*-- Non-conventional adsorbent, Adsorptive removal, Surfactants, Chemical kinetics, Thermodynamic, SLS, CTAB, TX-100, Standard Gibb's Free Energy Change.**

I. INTRODUCTION

Surfactants are the organic compounds that are best known for their wide use in many domestic and industrial applications. They are used as ingredients not only in detergents, soaps, cosmetics and personal care products, but also in paints, pesticides, textile, food, plastic, fibre, paper and leather industries and many more [1]. Surfactants are daily discharged to the environment from urban, rural and industrial activities. They are among widespread xenobiotics that may enter waste streams and the aquatic environment. Recent studies have demonstrated the persistence and accumulation of surfactants and their biodegradation products in the environment [2]. Surfactant adsorption is essential to be undertaken since effluent generated by surfactants as a result of their use shows an increase in proportion to the increase in the human's population. The pollution caused by surfactants in the environment such as foam emergence and their limited biodegradation by micro-organisms results in the disturbance of water eco-system [3]. Surfactants thus decrease water quality by lowering surface tension of water as well as decreasing biological oxygen demand (BOD) and by increasing chemical oxygen demand (COD). They are very harmful for aquatic flora and fauna and adversely affect all the living organisms through food chain ultimately. They affect aquatic flora by hindering their photosynthesis. Surfactants may cause chronic and acute effects on sensitive organisms. Moreover they affect the growth, motility and photosynthetic ability of algae [4]. Its long term exposure can cause cancer. In other words, surfactants are responsible for short term as well as long term effects on human beings, aquatic organisms, vegetation and environment. Therefore surfactant removal is necessary from aquatic environment for the elimination of surfactant contamination.

A variety of chemical, physical and biological techniques have been widely used to treat surfactant bearing wastewater, including adsorption [5], [6], [7], nano-filtration [8], [9], ion-exchange [8], biosorption, liquid-liquid extraction, advanced oxidation [10], [11], ozonation, coagulation/flocculation [12], ultraviolet photolysis [12], [11] etc. The advantages and disadvantages of every removal technique have been extensively reviewed. However, most of these methods have major drawbacks, such as- they require expensive and sophisticated equipments, pricey chemicals, high costs, trained technical staff and are more suitable for application in reference labs. Thus the removal of surfactants in an effective and economic way remains an important issue for researchers and environmentalists.

Overall adsorption has proven to be one of the most useful and effective among the control techniques. It is a separation technique in terms of initial cost, simplicity of design, ease of operation and execution, insensitive to toxic substances, easily adaptable, lower land area requirement, potential for significant removal of both organic and inorganic pollutants even at very low concentration, no sludge formation & adsorbent can be regenerated and reused again.

The disposal of agro-waste materials is increasingly becoming a cause for concern because these wastes represent unused resources. A major part of these wastes is normally used as domestic fuel. However, for better utilization of these cheap and abundantly available agricultural waste products, it can be explored as a low-cost alternative adsorbent owing to relatively high fixed

carbon content and presence of porous structure. Thus interest has grown towards the use of orange peel, an agro-waste material, as non-conventional adsorbent for the adsorptive removal of surfactants due to its ease of availability, renewability and biodegradability, cost-effectiveness, non-toxicity. Orange peel as agricultural spent material is sustainable for the environment. The conversion of this agro-waste material into a useful commodity towards the removal of a potential contaminant seems to be an attractive way in economic as well as environmental point of view. The thermodynamics of the binding process is also investigated, which includes determining the thermodynamic equilibrium adsorption constant, its natural log and Standard Gibb's Free Energy Change. The kinetics of adsorption process is also studied.

II. MATERIALS AND METHOD

A. Adsorbate

Merck made (analytical reagent grade) SLS, CTAB and TX-100 are used as adsorbates.

B. Preparation of Adsorbent Bed

Orange peel used as the precursor is collected from local fruit market, Bhilai, CG, India. The procedure employed in producing carbonized peel is drawn below-

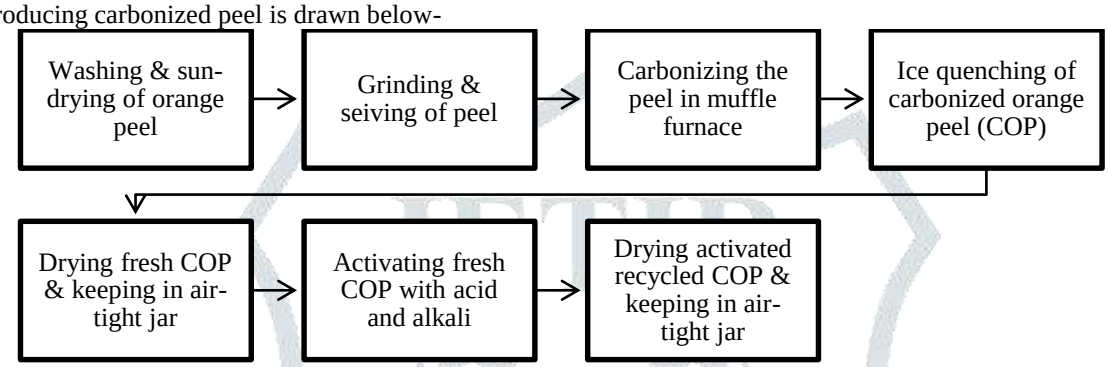

Fig.1: Flow diagram of preparation of adsorbent bed

Morphological image of COP is shown in figure 2, which shows that although the particles are of different sizes, but adsorb surfactant solutions efficiently.

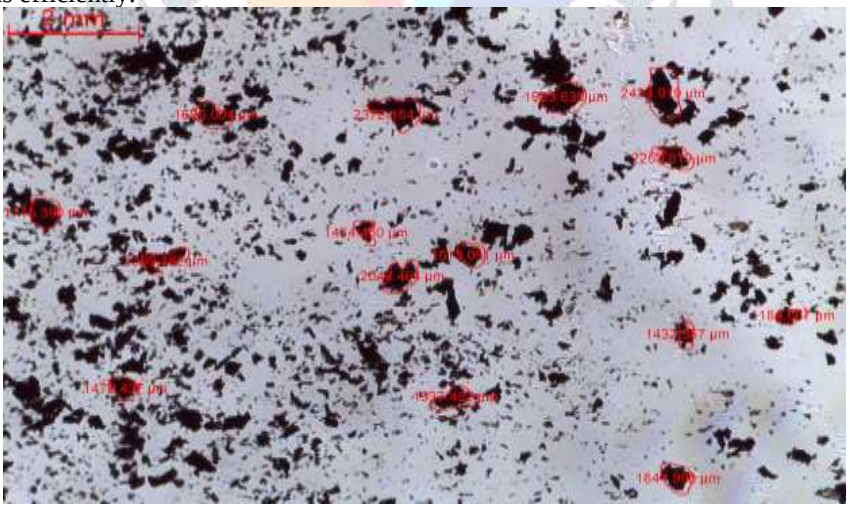

Fig. 2: Morphological image of carbonized orange peel (COP) with particle size

C. Chemicals Used

Orange-G, sodium chloride, chloroform, crystal violet, ortho-phosphoric acid, benzene, eosin-Y, borate buffer etc. chemicals are received from Merck (AR grade) and used without purification.

D. Instruments Used

The PC based Double Beam UV-Visible Spectrophotometer (Systronics, Model-2202), Digital Water and Soil Analysis Kit (Model no. 161) and Traube's Stalagmometer are used for measuring OD, pH, conductivity and surface tension respectively. Carbonized peel is prepared in muffle furnace (Tempstar ISO 9001:2000 Company). Research microscope (Leica DM750) is used to study the morphology of COP.

E. Experimental Protocol

The batch adsorption experiments are conducted in a set of 250 ml of shaking bottle containing adsorbent bed and 100-100 ml of SLS, CTAB and TX-100 solutions with different initial concentrations. Comparative study of adsorption of all the three surfactant solutions on COP has done for checking the capacity of fresh COP, acid and alkali activated recycled COP.

F. Optimization of Experimental Conditions

Optimization is done to check the efficacy of adsorbent bed, by changing three experimental conditions: (1) initial concentration of surfactant solutions; (2) doses of adsorbent bed; (3) shaking time. Adsorption of SLS, CTAB and TX-100 is studied by UV-Visible spectrophotometric, pH, electrical conductivity and stalagmometric methods. Experiments are independently performed and repeated again and again to get the accurate values.

G. Kinetic Study:

The study of kinetics of adsorption process is done by determining the average adsorption rate (Ms^{-1}) for each initial concentration of surfactant solutions [13]. It is necessary to know the rate of adsorption as adsorption is a time dependent procedure [14]. Adsorption rate can be calculated by using equation (1)-

$$\text{Rate of adsorption} = K\ x\ C_i \quad (1)$$

Where, K = Rate constant

C_i = Initial concentration

The values of K for SLS, CTAB and TX-100 are 4.035 x 10^{-3}, 5.0 x 10^{-3} and 4.52 x 10^{-3} g.mg $^{-1}$min. respectively.

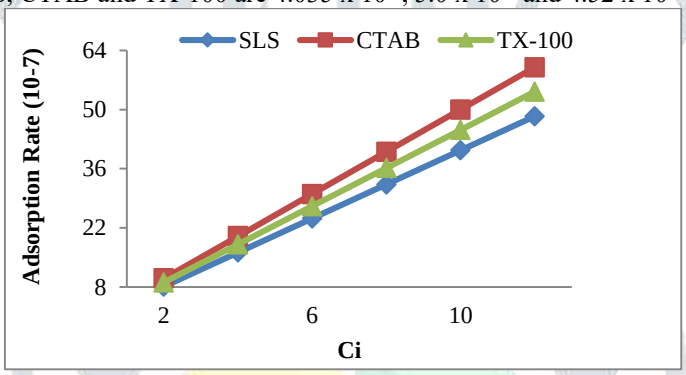

Fig.3: A plot of Cᵢ Vs Adsorption Rate

H. Thermodynamic Study:

The adsorption characteristics of surfactants can be expressed in terms of thermodynamic parameters such as thermodynamic equilibrium adsorption constant (K_c), its natural log (lnK_c) and Standard Gibb's Free Energy Change (ΔG^0) [15], [16], [17], [18]. The value of thermodynamic equilibrium constant (K_c) at 303 K temperature is calculated in accordance with the following equation (2)-

$$K_c = \frac{Cad}{Ce} \quad (2)$$

where, K_c be the equilibrium adsorption constant, Cad be the equilibrium concentration of surfactant on adsorbent bed surface and Ce be the equilibrium concentration of surfactant in solution. Calculation of Standard Gibb's Free Energy Change (ΔG^0) is done by using equation (3)-

$$\Delta G^0 = -RTlnK_c \quad (3)$$

where, ΔG^0 be the Standard Gibb's Free Energy Change, R be the Universal gas constant (8.314 joule/⁰/mole), T be the absolute temperature and lnK_c be the natural log of equilibrium adsorption constant.

Table 1: Thermodynamic Parameters of Adsorption of SLS, CTAB and TX-100 on COP

S.#	Type of Surfactant	T (Kelvin)	K_c	lnK_c	ΔG^0 (kJmol-1)
1.	SLS	303	3.25	1.179	-2.969
2.	CTAB	303	3.01	1.102	-2.776
3.	TX-100	303	3.12	1.138	-2.863

Table 1 represents the calculated values of ΔG^0 of SLS, CTAB and TX-100. The negative values of ΔG^0 indicate that the system is spontaneous and exothermic. The shaking process and normal condition of temperature are enough to supply the required energy for the adsorption process.

III. Results and Discussion

The graphs of pH, electrical conductivity, optical density (OD) and surface tension are plotted against different initial concentrations. Figures are shown below. Fig. 4.1, 4.2 and 4.3 show the comparison among pH values of SLS, CTAB and TX-100 at different initial concentrations using fresh COP, alkali and acid activated recycled COP respectively. Results reveal that at 8x10^{-4}

molar concentration highest adsorption take place. Alkali activated recycled COP is found to be a better adsorbent bed for SLS because the sulphate ions (acid radicals) get adsorb at alkali surface, whereas acid activated recycled COP is better for CTAB adsorption because the ammonium ions (basic radicals) get adsorb at acid surface easily. Adsorption of TX-100 takes place due to weak van der waals force of attraction. No chemical or ionic bond is formed between adsorbate and adsorbent bed. Similar results are found for electrical conductivity, which are shown in fig 5.1, 5.2 and 5.3.

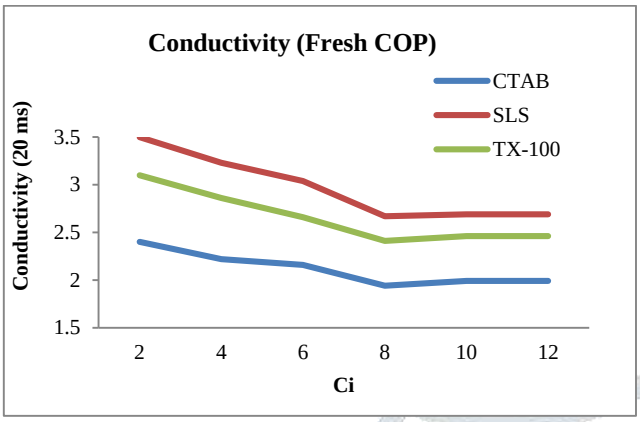

Fig.5.1: A plot of Initial Concentration Vs Conductivity using Fresh COP

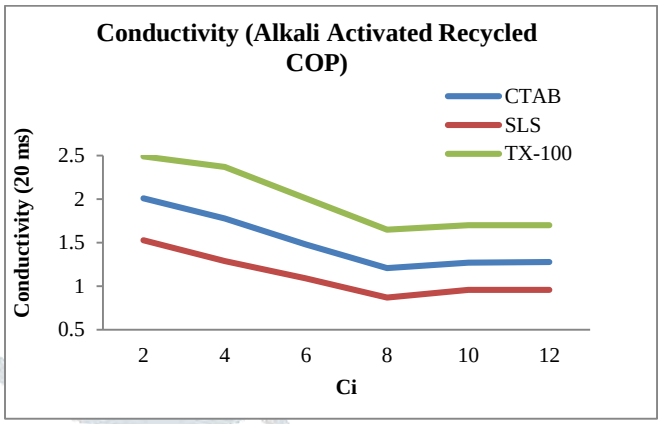

Fig.5.2: A plot of Initial Concentration Vs Conductivity using Alkali Activated Recycled COP

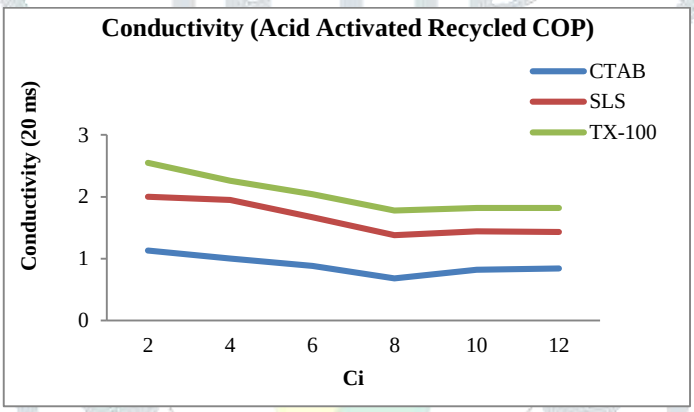

Fig.5.3: A plot of Initial Concentration Vs Conductivity using Acid Activated Recycled COP

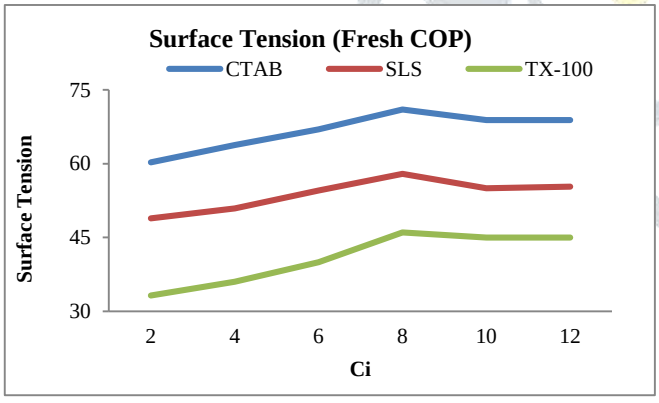

Fig.6.1: A plot of Initial Concentration Vs Surface Tension using Fresh COP

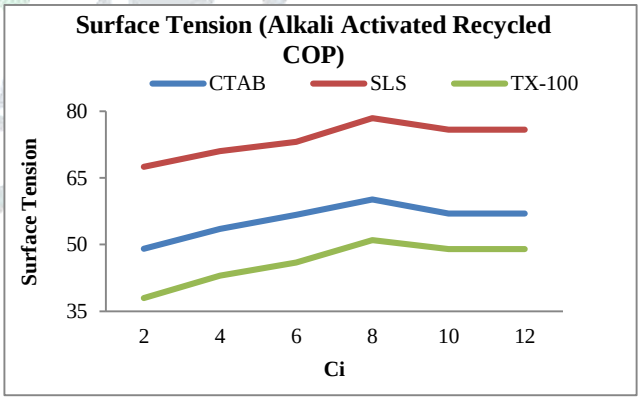

Fig.6.2: A plot of Initial Concentration Vs Surface Tension using Alkali Activated Recycled COP

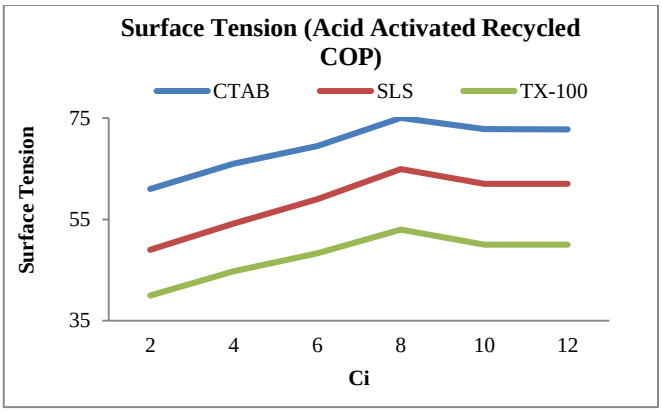

Fig.6.3: A plot of Initial Concentration Vs Surface Tension using Acid Activated Recycled COP

The surface tension values of all the three surfactants for comparative adsorption are shown in fig 6.1, 6.2 and 6.3. Results reveal that SLS is highly adsorbed by alkali activated recycled COP, since alkali can chemically react with sulphate ions (acid radicals) very easily. Greater the value of surface tension, smaller is the concentration of surfactant in the shook solution. Adsorption of CTAB takes place by using acid activated recycled COP, since acid can chemically react with ammonium ions (basic radicals) very easily. TX-100 gets adsorbed through van der waals force of attraction

Fig 7.1, 7.2 and 7.3 are the plots of initial concentration versus OD values of SLS, CTAB and TX-100 solutions, which show the comparative adsorption. Alkali activated recycled COP is found to be a better adsorbent bed for SLS and acid activated recycled COP is found to be a better adsorbent bed for CTAB adsorption.

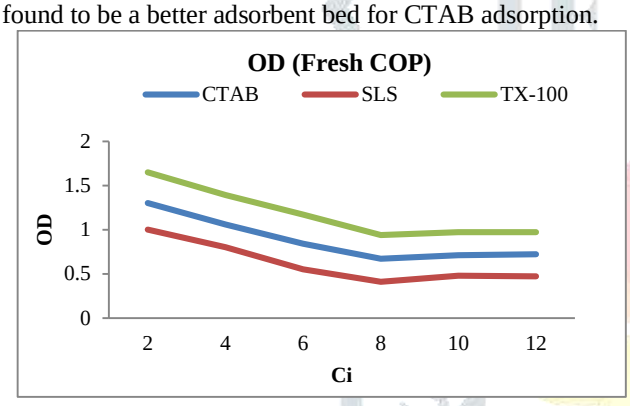

Fig.7.1: A plot of Initial Concentration Vs OD using Fresh COP

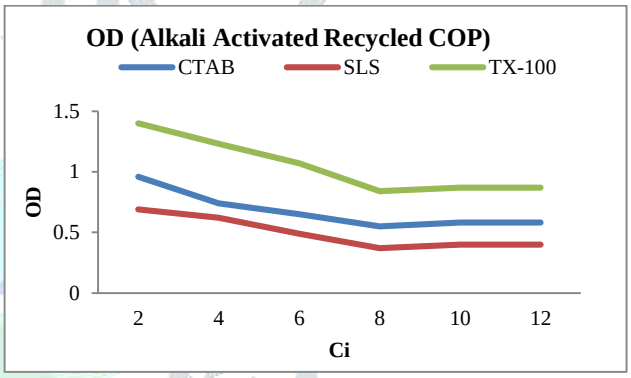

Fig.7.2: A plot of Initial Concentration Vs OD using Alkali Activated Recycled COP

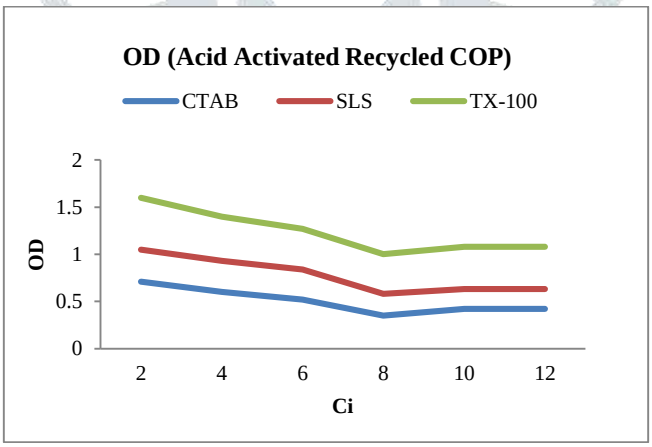

Fig.7.3: A plot of Initial Concentration Vs OD using Acid Activated Recycled COP

The mechanism of comparative adsorption of SLS, CTAB and TX-100 on COP are considered by measuring change in pH, conductivity, surface tension and OD values for different adsorbent bed doses and different shaking time. Related tables are shown below.

Highest adsorption takes place by using 2.0 gm adsorbent bed dose. This is due to the increase in surface area with increase in mass of adsorbent, which results in greater availability of the exchangeable sites or active sorption sites. 45 minute time is found sufficient shaking time to adsorb anionic, cationic and non-ionic surfactants and reaches saturation in about 60 minutes. As the number of adsorption sites was more in the beginning, more and more surfactant molecules get adsorbed, but with the passage of time, the adsorption sites get exhausted and thus lead to slower adsorption and after sometime, equilibrium is attained.

Table 2: Measurement of pH, conductivity, OD and surface tension between adsorption of SLS, CTAB & TX-100 and various adsorbent bed doses

S. #	Surfactant	Charcoal Type	Adsorbent Bed Doses (gm) for pH Measurement						Adsorbent Bed Doses (gm) for Conductivity Measurement						Adsorbent Bed Doses (gm) for OD Measurement						Adsorbent Bed Doses (gm) for Surface Tension Measurement					
			0.4	0.8	1.2	1.6	2.0	2.4	0.4	0.8	1.2	1.6	2.0	2.4	0.4	0.8	1.2	1.6	2.0	2.4	0.4	0.8	1.2	1.6	2.0	2.4
1	SLS CTAB TX-100	Fresh COP	4.32	4.76	4.98	5.11	5.33	5.29	3.2	2.9	2.73	2.46	2.03	2.12	1.5	1.2	0.83	0.71	0.48	0.62	48.12	52.45	56.34	61.24	66.0	63.54
			9.02	8.91	8.68	8.44	8.13	8.16	2.4	2.22	2.16	1.98	1.53	1.62	1.7	1.52	1.36	1.01	1.25		60.04	64.21	68.57	72.1	74.78	72.91
			7.21	7.06	6.72	6.43	6.02	6.07	3.1	2.86	2.66	2.41	2.02	2.17	1.65	1.46	1.22	1.05	0.78	1.01	33.31	37.13	41.0	46.32	50.01	47.35
2	SLS CTAB TX-100	Alkali Activated Recycled COP	6.3	6.72	7.0	7.59	8.14	8.09	2.31	2.02	1.78	1.59	1.22	1.48	0.99	0.83	0.61	0.46	0.22	0.39	50.06	54.7	60.07	65.5	70.85	67.03
			8.07	7.65	7.32	6.89	6.44	6.47	1.98	1.58	1.35	1.21	1.01	1.18	1.37	1.05	0.93	0.77	0.55	0.62	63.12	67.0	69.53	72.67	75.0	74.83
			7.1	6.78	6.55	6.21	6.0	6.11	2.49	2.27	2.03	1.89	1.55	1.64	1.68	1.51	1.35	1.21	1.01	1.16	35.71	39.31	43.0	46.98	51.01	48.53
3	SLS CTAB TX-100	Acid Activated Recycled COP	5.05	5.77	6.25	6.78	7.0	6.97	2.02	1.95	1.67	1.48	1.16	1.27	1.3	1.19	1.07	0.92	0.63	0.76	49.34	52.46	57.21	59.89	63.0	61.25
			7.0	6.65	6.26	6.0	5.76	5.88	1.86	1.54	1.27	1.07	0.89	1.04	1.01	0.87	0.68	0.47	0.23	0.36	60.0	64.67	67.89	71.03	76.24	74.32
			7.17	7.01	6.76	6.47	6.33	6.41	2.55	2.36	2.17	2.02	1.77	1.91	1.5	1.36	1.19	1.03	0.82	0.95	36.0	39.57	43.0	45.23	52.02	49.0

The general pattern of adsorption is shown in figure 8. Figure 8 represents the common trend of typical adsorption of SLS, CTAB and TX-100 on COP. It is clear from the graph that initially all the three types of surfactants get adsorb rapidly at the solid-liquid interface. Maximum adsorption is taking place at C_e 100 $mg.g^{-1}$, after that equilibrium is attained. The reason is, in the beginning number of adsorption sites was greater, thus the extent of adsorption was greater, but with the passage of time, the adsorption sites get exhausted and thus lead to slower adsorption and after sometime, saturation reached.

Table 3: Measurement of pH, conductivity, OD and surface tension between adsorption of SLS, CTAB & TX-100 and various shaking time

S. #	Surfactant	Charcoal Type	Shaking Time (min.) for pH Measurement				Shaking Time (min.) for Conductivity Measurement				Shaking Time (min.) for OD Measurement				Shaking Time (min.) for Surface Tension Measurement			
			15	30	45	60	15	30	45	60	15	30	45	60	15	30	45	60
1	SLS CTAB TX-100	Fresh COP	4.5	4.84	5.33	5.12	2.8	2.6	2.15	2.38	1.4	1.28	0.97	1.16	55.27	63.24	69.25	65.54
			8.91	8.68	8.13	8.36	2.4	2.27	1.85	2.16	1.76	1.53	1.17	1.44	66.57	71.41	78.78	73.61
			7.06	6.72	6.02	6.41	2.78	2.56	2.39	2.44	1.57	1.31	1.15	1.25	42.50	46.32	53.61	50.15
2	SLS CTAB TX-100	Alkali Activated Recycled COP	6.77	7.02	8.19	7.46	1.94	1.67	1.02	1.38	1.01	0.83	0.42	0.66	61.17	67.33	78.75	63.23
			7.59	7.05	6.24	6.67	2.3	2.12	1.76	2.0	1.37	1.19	1.0	1.09	59.53	62.67	67.0	64.83
			6.46	6.28	6.0	6.11	2.59	2.37	2.13	2.24	1.48	1.34	1.11	1.25	45.00	48.88	57.01	53.00
3	SLS CTAB TX-100	Acid Activated Recycled COP	6.37	6.88	7.43	6.97	2.02	1.8	1.27	1.59	1.21	1.06	0.73	0.96	57.21	59.89	63.0	61.25
			6.75	6.26	5.76	5.92	2.14	2.01	1.59	1.91	1.12	1.03	0.78	0.96	69.89	72.34	79.24	74.11
			6.43	6.33	6.06	6.27	2.48	2.27	2.06	2.19	1.39	1.20	1.02	1.13	44.1	49.33	55.01	50.00

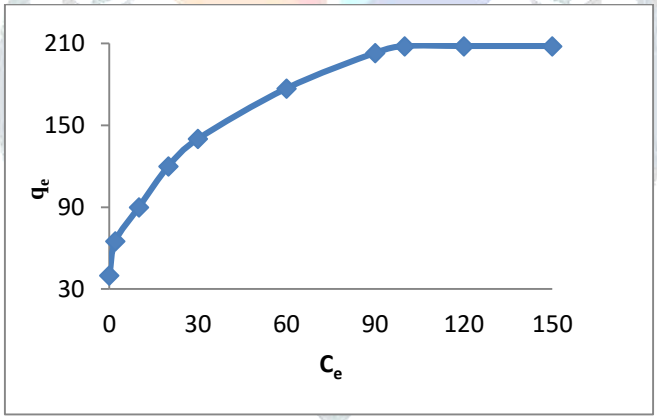

Fig.8: Common Trend of Adsorption of Surfactants (SLS, CTAB, TX-100)

IV. CONCLUSIONS

Results reveal that maximum adsorption takes place by using 2.0 gm adsorbent bed dose, 45 minute shaking time at 8 x 10^{-4} M initial concentration and after that equilibrium is attained. Alkali activated recycled COP is found to be a better adsorbent bed for the adsorption of SLS because the sulphate ions (acid radicals) of SLS, get adsorb at alkali surface readily, whereas acid activated recycled COP is better enough for adsorbing CTAB because the ammonium ions (basic radicals) of CTAB, get adsorb at acid surface easily. TX-100 is adsorbed through bed by physical adsorption. Weak van der waals force of attraction is responsible for the adsorption of TX-100. Spontaneous, feasible and exothermic adsorption is shown by thermodynamic study. The adsorption kinetics shows pseudo second order reaction. Adsorption process represents an effective method for all the three kinds of surfactants. It's a faster, convenient and less energy-consuming procedure for the effective exclusion of surfactants present in effluent intended for reuse.

Apart from utilizing cheap and abundant agricultural waste product, it is promising indeed to convert orange peel into activated charcoal. This conversion will address problems associated with unwanted agricultural wastes been converted into useful, value-added alternative adsorbent. After use, the adsorbent bed can be reused as domestic fuel, without any harm. This can also be used as additives with conventional bed with no side effects. It may be a better replacement of synthetic adsorbent beds.

V. ACKNOWLEDGMENT

We gratefully appreciate the support of management of Rungta College of Engineering & Technology, Bhilai and Chhattisgarh Council of Science & Technology, Raipur for providing lab facilities and essential prop up.

VI. REFERENCES

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