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RESEARCH ARTICLE

EFFECTIVE REMOVAL OF HAZARDOUS CARBOXYLIC ACIDS BY NOVEL LOW COST ADSORBENTS

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ABSTRACT

Powdered (PTCRC) and Sulphonated Terminalia Chebula Retz carbon (STCRC) and Powdered (PMPC) and Sulphonated Mimosa pudica carbon (SMPC) adsorbents has been prepared and applied to the removal of environmental polluting carboxylic acids like formic acid, acetic acid and trichloroacetic acid. The adsorption characteristics have been investigated with the help of the various parameters like effect of initial concentration, contact time, dose of adsorbent and particle size. The adsorption capacity of adsorbents to adsorb Trichloroacetic acid is found to be higher than that of the other acids. Then, from the results of the present study, it is found that the SMPC and STCRC are a very good adsorbent than the PMPC and PTCRC for the removal of Formic acid, Acetic acid and Trichloroacetic acid from aqueous solutions at diluted condition. Hence this study will be useful in designing effluent treatment plant.

INTRODUCTION

Many procedures have been developed for the removal of hazardous formic acid, acetic acid and trichloroacetic acid from aqueous media such as reductive precipitation (Skogerboe *et al.*, 1985) and (Nakashima *et al.*, 1998), solvent extraction (Khalid *et al.*, 1996) and (Liu *et al.*, 2002), ion exchange (Hirata *et al.*, 1989) and (Caroli *et al.*, 1991), and adsorption. Interest in the adsorption of hazardous acids for recovery purpose has increased manifold in recent years. The adsorption process under certain conditions has a definite edge over other methods used for metal recovery, because of its simplicity, selectivity and efficiency (Qadeer *et al.*, 1994). Activated charcoal is a widely used material that is well known for its adsorption properties. Activated charcoal is non-hazardous, processed carbonaceous products having an intricate porous structure and a large surface area that is contained predominantly within the micropores. The adsorbent properties of the activated charcoal are essentially attributed to their large surface area, high degree of surface reactivity and favourable pore size distribution. The latter makes their internal surface accessible for adsorption and enhanced their adsorption rate without sacrificing their mechanical strength (Bansal *et al.*, 1988).

Due to these properties, activated charcoal has found many applications in industries for the separation and removal of hazardous acids from solutions. The present work describes our investigations on the adsorption of Formic acid, acetic acid and trichloroacetic acid on activated charcoal. No data are available on its adsorption on activated charcoal.

Experimental

MATERIALS

The following chemicals were employed for the present work:

- Trichloroacetic acid - Merck
- Acetic acid - Fisher
- Formic acid - Merck
- Mimosa pudica Sulphonated form (mesh size 105 microns)
- Mimosa pudica Powdered form (mesh size 105 microns)
- Terminalia Chebula Retz Sulphonated form (mesh size 105 microns)
- Terminalia Chebula Retz Powdered form (mesh size 105 microns)
- Sodium hydroxide - Merck

The adsorbents employed in the present work were Mimosa pudica and Terminalia Chebula Retz Sulphonated charcoal form and powdered form. Fresh sample of Mimosa pudica and

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Terminalia Chebula Retz was collected from Madurai and used as Powdered and Sulphonated form for all the experiments, after sieving for a constant particle size of 105 microns (Sharma *et al.*, 1976).

Solutions Prepared

Trichloroacetic acid

Approximately 0.1N solution of Trichloroacetic acid was prepared by diluting from 3ml of Trichloroacetic acid to 500ml SMF using double distilled water. The concentration of Trichloroacetic acid employed in the adsorption study was determined by acid-base titration using phenolphthalein as indicator.

Acetic acid

Exactly 3 ml of acetic acid was diluted with 500 ml double distilled water to get a solution of strength 0.1N.

Formic acid

Approximately, 0.1N solution of formic acid was prepared by diluting from 3ml of formic acid (LR) to 500ml SMF using double distilled water. The concentration of formic acid employed in each adsorption study was determined by acid-base titration using phenolphthalein as indicator.

Sodium Hydroxide

Stock solution of approximately, 0.1N sodium hydroxide was prepared by dissolving 2g of sodium hydroxide in 500ml of double distilled water. The strength of stock solution was determined by titrating against standard oxalic acid solution (0.05N) using phenolphthalein as indicator.

Activation of the Mimosa pudica and Terminalia Chebula Retz

The powdered Mimosa pudica and Terminalia Chebula Retz were carbonized and sulphonated by adding con.H₂SO₄ and heated to ~90°C in hot air-oven for 6 Hrs. It was then cooled, washed with distilled water for several times to remove the free acid and dried at 110°C for 12 Hrs. It was black in colour and labelled as Sulphonated Charcoal (SMPC & STCRC). It was sieved and used for a constant particle size of 105 microns.

METHODS

The various experiments in this adsorption studies were carried out by employing the batch adsorption technique. In order to find out the optimum experimental conditions, adsorption studies were carried out by varying the following experimental conditions. Initial concentration of adsorbates viz., Formic acid, Acetic acid and Trichloroacetic acid at constant dose of powdered and Sulphonated form of Mimosa pudica & Terminalia Chebula Retz and contact time at 35 ± 1⁰ C. Contact time, at constant dose of powdered and sulphonated charcoals and initial concentration of adsorbates (acids) at 35 ± 1⁰ C. Dose of adsorbents (Powdered & Sulphonated) at constant optimum contact time and initial concentration of

adsorbates (acids) at 35 ± 1⁰ C. Particle size of the adsorbents (Powdered & Sulphonated) at constant dose, initial concentration and contact time at 35 ± 1⁰ C. The stock solution of the carboxylic acid (~ 0.1N) was suitably diluted to the required initial concentrations with double distilled water to a total volume of 50ml in 125ml leak-proof corning reagent bottles. The initial concentrations (C_i) of the acid were calculated from the concentration of stock solution or titrimetrically estimated. Required amount of adsorbents (Powdered & Sulphonated form) (5 – 20 gL⁻¹) was then exactly weighed and transferred into each one of these bottles. The bottles were then placed in a mechanical shaker and shaken vigorously for a required period of contact time (range 0-120 min.). After attaining equilibrium, the bottles were kept aside for 30 min. at room temperature (35 ± 1⁰C) in order to allow the adsorbents (Powdered & Sulphonated charcoal - mesh 105 microns) particles to settle down (Padma Vasudevan and Sharma, 1979). The solutions were then filtered through Whatmann No.1 filter paper. The first 10ml portion of the filtrate was rejected in each bottle in order to eliminate the effect of adsorption of adsorbates (acids) on filter paper. The filtration was then continued and the filtrate (acids) from each bottle was collected in a separate clean dry conical flask. Then, 10ml of filtrate (acid) from each bottle was pipetted out into a clean dry conical flask and one or two drops of phenolphthalein was added and titrated against standardized sodium hydroxide solution. The end point is the appearance of pale pink colour. The titrations were repeated for concordant titre values. The same procedure was followed for all the adsorption experiments under the specified experimental conditions. The titre values after attaining the equilibrium give the equilibrium concentration of adsorbate (acid) (C_e) remaining in solution after adsorption on the adsorbent at the room temperature (35±1°C) and other experimental conditions (Sharma and Bafna, 1953). In all the adsorption experiments, the percentage removal of adsorbate on the Powdered & Sulphonated charcoal has been calculated using the relationship.

$$\% \text{ Removal} = \left(\frac{C_i - C_e}{C_i} \right) \times 100$$

The amount adsorbed (q) per unit mass of the adsorbent have been calculated by using the following equation

$$\text{Amount adsorbed (q)} = \left(\frac{C_i - C_e}{m} \right) = \frac{x}{m}$$

Where, 'm' is the dose of adsorbent (in gL⁻¹) added in the adsorption experiment; C_i and C_e are the initial and equilibrium concentration of acids, in gqL⁻¹ and x is the amount of acid adsorbed by 'm' g of adsorbent.

Effect of Initial Concentration

In this adsorption experiments, dose of adsorbents (20gL⁻¹) was kept constant in all the bottles and different initial concentrations of Formic acid, Acetic acid and Trichloroacetic acid (0.1 – 0.01N) were taken. The bottles were then shaken for a period of 15 min. The equilibrium concentration (C_e, in g eq L⁻¹) of the acids had been estimated in each and every bottle and the readings were tabulated (Sharma and Bafna, 1953).

Table 1. Effect of Variation of Initial concentration of Formic acid on PMPC and PTCRC

S.No.	[HCOOH] (N)	% Removal acid by powdered form of MP $\left(\frac{C_i - C_e}{C_i}\right) \times 100$	% Removal acid by powdered form of TCR $\left(\frac{C_i - C_e}{C_i}\right) \times 100$
1	0.01	14.2	12.2
2	0.02	12.7	11.7
3	0.03	12.1	11.1
4	0.04	11.3	09.3
5	0.05	10.6	08.7
6	0.06	10.3	08.69
7	0.07	10.2	08.5
8	0.08	10.2	08.5

Dose of adsorbent = 20g L⁻¹; Contact Time = 5 min; Temperature = 35 ± 1°C.

Table 2. Effect of Variation of Initial concentration of Formic acid on SMPC and STCRC

S.No.	[HCOOH] (N)	% Removal acid by Sulphonated form of SMPC $\left(\frac{C_i - C_e}{C_i}\right) \times 100$	% Removal acid by Sulphonated form of STCRC $\left(\frac{C_i - C_e}{C_i}\right) \times 100$
1	0.01	52.6	40.2
2	0.02	44.9	36.7
3	0.03	41.7	31.1
4	0.04	37.7	29.3
5	0.05	36.2	28.7
6	0.06	34.1	26.6
7	0.07	32.6	26.5
8	0.08	27.9	25.9
9	0.09	24.3	24.9

Dose of adsorbent = 20g L⁻¹; Contact Time = 5 min; Temperature = 35 ± 1°C.

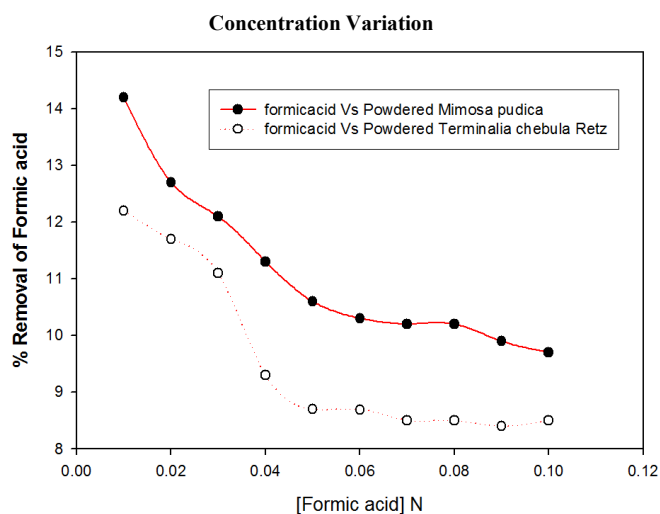


Fig. 1. Plot of % removal of formic acid Vs concentration of formic acid on PMPC and PTCRC at 298K

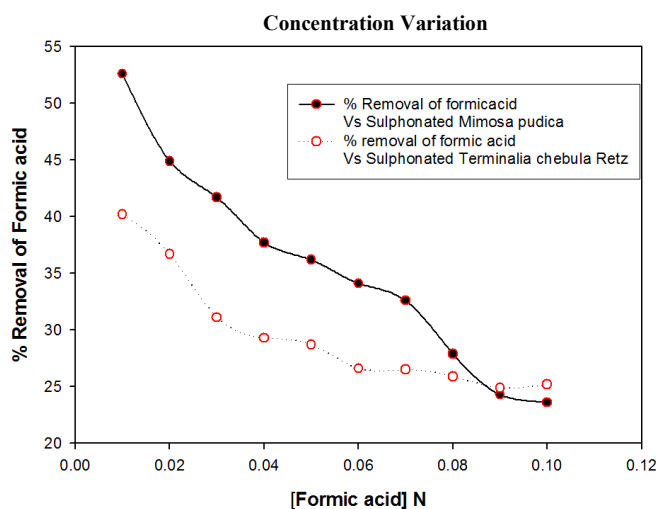


Fig. 2. Plot of % removal of formic acid Vs concentration of formic acid on SMPC and STCRC

Effect of contact time

In this adsorption experiments, an initial concentration of adsorbates viz., Formic acid, Acetic acid and Trichloroacetic acid (0.05N) and a constant dose of Sulphonated form charcoals (20g L⁻¹) were taken in all the bottles. All the bottles were placed in a mechanical shaker and while the shaking was started simultaneously, a stop watch was also started, to note the contact or shaking time. The bottles were then withdrawn at different time intervals i.e., 0, 5, 7, 10, 15, 20, 30, 45, 60 and 90 minutes and the other procedures were repeated to estimate the equilibrium concentration (C_e) of the acid. The results were noted and the percentage of removal was plotted against the contact time (Mohan Rao and Pillai, 1954).

Effect of dose of adsorbent: In this adsorption experiments, the initial concentration of adsorbates viz., Formic acid, Acetic acid and Trichloroacetic acid (0.05N) was kept constant with a varying amount of sulphonated charcoals (dose 5–30g L⁻¹) and constant contact time (15 min.). Other procedure followed was similar to the general procedure for adsorption studies. From the adsorption data, the optimum dose was obtained. The percentage of removal was plotted against the dose of adsorbent (Kathiresa Pandian and Krishnamoorthy, 1991).

Effect of particle size of adsorbent: In this adsorption experiments, an initial concentration of adsorbates viz., Formic acid, Acetic acid and Trichloroacetic acid (0.05N) was kept constant amount of sulphonated charcoal (dose 20g L⁻¹) and

constant contact time (15 min.) and with a varying of the particle size. Other procedure followed was similar to the general procedure for adsorption studies. From the adsorption data, the optimum particle size of adsorbent was obtained. The percentage of removal was plotted against the particle size of (Ramachandran and Krishnamoorthy, 1984).

RESULTS AND DISCUSSION

The present investigation deals with the study on the removal of carboxylic acids (Formic acid, Acetic acid and Trichloroacetic acid) by Powdered and Sulphonated Mimosa pudica carbon.

Table 3. Effect of Variation of Initial concentration of Acetic acid on PMPC and PTCRC

S.No.	[CH ₃ COOH] (N)	% Removal acid by powdered form of MP $\left(\frac{C_i - C_e}{C_i}\right) \times 100$	% Removal acid by powdered form of TCR $\left(\frac{C_i - C_e}{C_i}\right) \times 100$
1	0.01	51.2	45.45
2	0.02	42.7	28.57
3	0.03	36.1	26.26
4	0.04	28.3	19.16
5	0.05	22.6	18.03
6	0.06	22.3	18.03
7	0.07	22.2	12.73
8	0.08	18.2	12.38
9	0.09	18.9	12.07

Dose of adsorbent = 20g L⁻¹; Contact Time = 5 min; Temperature = 35 ± 1°C.

Table 4. Effect of Variation of Initial concentration of Acetic acid on SMPC and STCRC

S.No.	[CH ₃ COOH] (N)	% Removal acid by Sulphonated form of MP $\left(\frac{C_i - C_e}{C_i}\right) \times 100$	% Removal acid by Sulphonated form of TCR $\left(\frac{C_i - C_e}{C_i}\right) \times 100$
1	0.01	53.2	48.45
2	0.02	45.7	38.57
3	0.03	39.1	24.26
4	0.04	38.9	20.16
5	0.05	38.6	27.03
6	0.06	32.3	27.03
7	0.07	32.2	26.73
8	0.08	28.2	26.38
9	0.09	28.9	26.07

Dose of adsorbent = 20g L⁻¹; Contact Time = 5 min; Temperature = 35 ± 1°C.

Table 5. Effect of Variation of Initial concentration of Trichloroacetic acid on PMPC and PTCRC

S.No.	[Cl ₃ CCOOH] (N)	% Removal acid by powdered form of MP $\left(\frac{C_i - C_e}{C_i}\right) \times 100$	% Removal acid by powdered form of TCR $\left(\frac{C_i - C_e}{C_i}\right) \times 100$
1	0.01	42.31	36.2
2	0.02	36.5	25.46
3	0.03	32.8	19.65
4	0.04	28.2	14.13
5	0.05	26.21	22.31
6	0.06	26.01	11.16
7	0.07	25.3	10.68
8	0.08	25.25	10.80
9	0.09	22.9	10.17

Dose of adsorbent = 20g L⁻¹; Contact Time = 5 min; Temperature = 35 ± 1°C.

Table 6. Effect of Variation of Initial concentration of Trichloroacetic acid on SMPC and STCRC

S.No.	[Cl ₃ CCOOH] (N)	% Removal acid by Sulphonated form of MP $\left(\frac{C_i - C_e}{C_i}\right) \times 100$	% Removal acid by Sulphonated form of TCR $\left(\frac{C_i - C_e}{C_i}\right) \times 100$
1	0.01	86.1	85.0
2	0.02	76.9	71.61
3	0.03	73.9	71.42
4	0.04	71.8	67.50
5	0.05	70.2	65.52
6	0.06	70.25	62.81
7	0.07	70.2	61.57
8	0.08	69.5	63.58
9	0.09	69.6	63.56

Dose of adsorbent = 20g L⁻¹; Contact Time = 5 min; Temperature = 35 ± 1°C.

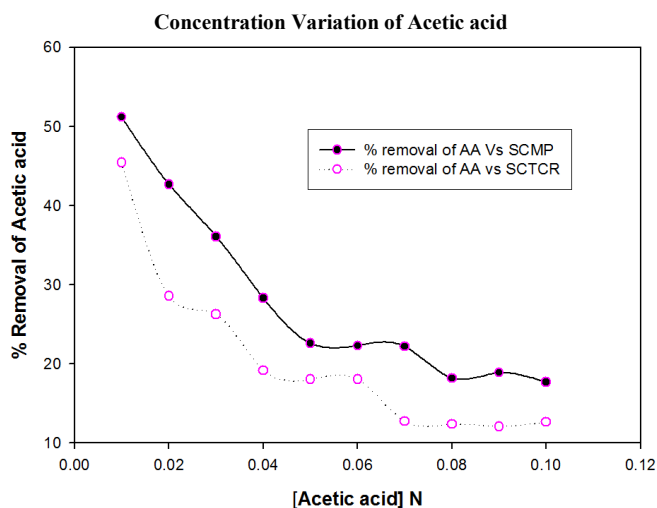


Fig. 3. Plot of % removal of acetic acid Vs concentration of acetic acid on PMPC and PTCRC at 298K

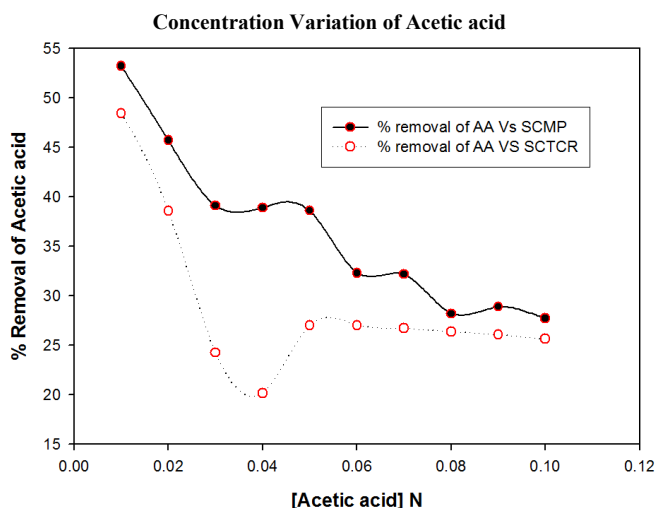


Fig. 4. Plot of % removal of acetic acid Vs concentration of acetic acid on SMPC and STCRC at 298K

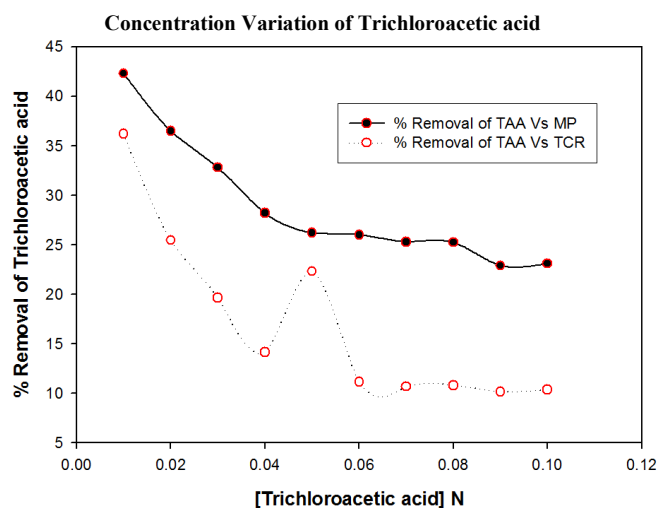


Fig.5. Plot of % removal of Trichloroacetic acid Vs concentration of Trichloroacetic acid on PMPC and PTCRC at 298K

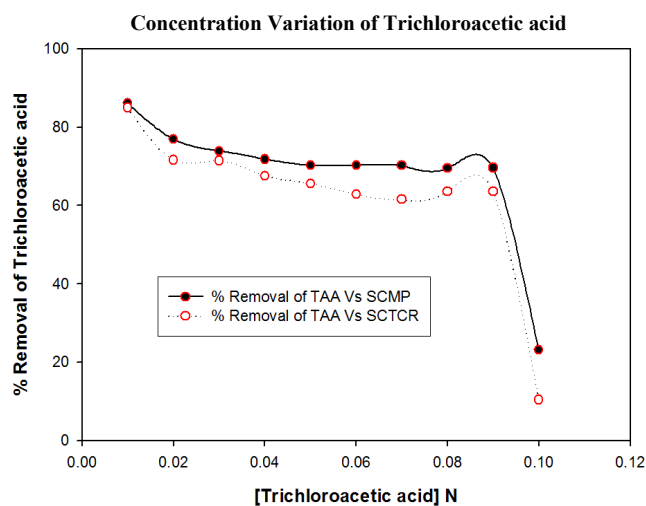


Fig. 6. Plot of % removal of Trichloroacetic acid Vs concentration of Trichloroacetic acid at 298K

This is an endeavour to present data for the design of an economical and flexible wastewater treatment unit for the removal of carboxylic acid from the effluents discharged from the industries (Duraisamy and Krishnamoorthy, 1981). The parameters which warrant the evaluation for investigating the mechanism of adsorption and deciding the efficiency of removal of carboxylic acids are,

- The initial concentration of the solution.
- The time of contact between adsorbate and adsorbent
- The particle size of adsorbent
- The dose of adsorbent used.

Procedures for the Evaluation of the Parameters are given below:

- Effect of initial concentration of carboxylic acids viz., formic acid, acetic acid and trichloroacetic acid with fixed dose of powdered and sulphonated form of Mimosa pudica (MP) and Terminalia Chebula Retz (TCR) and fixed contact time at $35 \pm 1^{\circ} \text{C}$.

- Effect of contact time at which constant initial concentration of carboxylic acids with fixed doses of sulphonated charcoals at $35 \pm 1^{\circ} \text{C}$.
- Effect of particle size of adsorbents at which the fixed initial concentration, contact time and dose at $35 \pm 1^{\circ} \text{C}$.
- Effect of dose of adsorbents at which the fixed initial concentration and fixed contact time at $35 \pm 1^{\circ} \text{C}$.
- Initial Concentration of Carboxylic acids = range 0.01 – 0.1N
- Dose of Adsorbents = range 5 – 20 gL^{-1}
- Particle size = range 90–220 mesh.
- Contact Time = range 0 – 120 min.

In order to study their effects of process parameters on the adsorption of carboxylic acids on powdered and sulphonated charcoals, the adsorption experiments were carried out at room temperature ($35 \pm 1^{\circ} \text{C}$). The results are analyzed and discussed in this chapter.

Effect of initial concentration:

The adsorption study of carboxylic acids (Formic acid, Acetic acid and Trichloroacetic acid) on powdered and Sulphonated form of MP and TCR at fixed amount of adsorbent i.e., 10g/L-1 which means that 1g per 100ml of volume at different initial concentration (0.01 - 0.1 N) of carboxylic acids were carried out. The data obtained in these experiments are given in Tables 1 - 6. The variation of percentage of removal of acid with respect to the initial concentration is graphically represented in Figures 1-6. The adsorption study of carboxylic acids (Formic acid, Acetic acid and Trichloroacetic acid) on Powdered and Sulphonated form of Mimosa pudica (MP) & Terminalia Chebula Retz (TCR) at fixed amount of adsorbent i.e., 20g/L⁻¹ which means that 1g per 50ml of volume at different initial concentration (0.01 - 0.1N) of carboxylic acids were carried out.

Table 7. Effect of Variation of contact time for Formic acid on SMPC and STCRC

S.No.	[HCOOH] (N)	% Removal of Acid by SMPC	% Removal of Acid by STCRC
1	0	09.5	08.04
2	5	13.8	17.6
3	7	25.5	14.5
4	10	33.5	27.8
5	15	36.2	28.7
6	20	36.3	28.2
7	30	35.2	28.5
8	45	35.5	27.9
9	60	35.5	26.9
10	90	34.9	26.8

Initial concentration = 0.05 N ; Sulphonated form of Mimosa Pudica and Terminalia Chebula Retz (gm) = 20g L⁻¹; Temperature = 35 ± 1°C

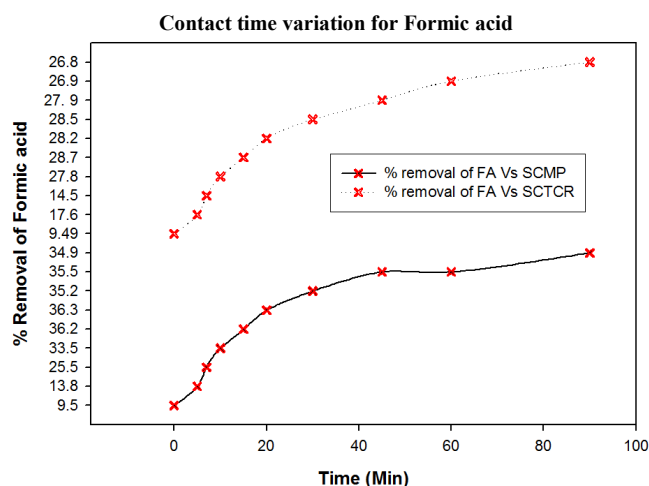


Fig. 7. Plot of % removal of Formic acid Vs contact time at 298K

The data obtained in these experiments are given in Tables (1 - 6). The variation of percentage of removal of acid with respect to the initial concentration is graphically represented in Fig.1-6. The range of percentage of removal of Formic acid by powdered form of adsorbent in the range of 10.2 - 14.2 Acetic acid in the range of 24.3 - 52.6 and Trichloroacetic acid in the range 12.7 - 51.2. The range of percentage of removal of Formic acid by sulphonated form of adsorbent in the range of 26.7- 53.2, Acetic acid in the range of 10.17- 42.31 and Trichloroacetic acid in the range 63.56- 86.1.

An increase in the initial concentration of carboxylic acid results in the reduction in the percentage of adsorption. This indicates the reduction is immediate solute adsorption due to the lack of available active site on the adsorbent surface compared (Chandrasekaran and Krishnamoorthy, 1987).

Table 8. Effect of Variation of contact time for Acetic acid on SMPC and STCRC

S.No.	[CH ₃ COOH] (N)/Min	% Removal of Acid by SMPC	% Removal of Acid by STCRC
1	0	13.2	12.45
2	5	25.7	18.57
3	7	29.1	24.26
4	10	36.9	26.16
5	15	38.6	27.03
6	20	32.3	27.03
7	30	32.2	26.13
8	45	32.2	26.18
9	60	31.9	26.07
10	90	31.7	25.63

Initial concentration = 0.05 N ; Sulphonated form of Mimosa Pudica and Terminalia Chebula Retz (gm) = 20g L⁻¹; Temperature = 35 ± 1°C

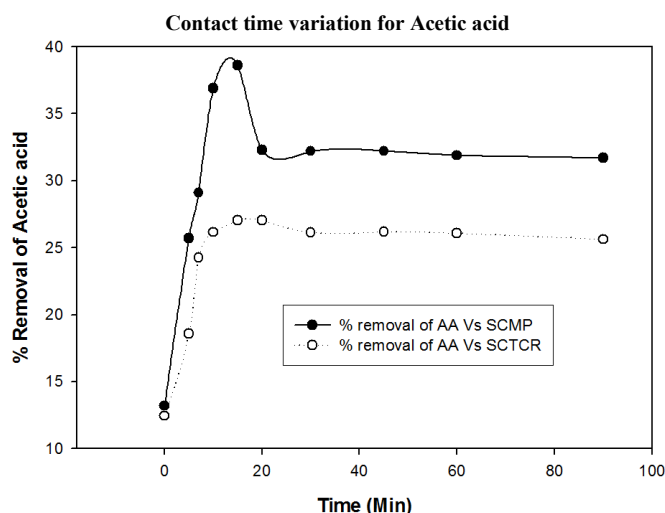


Fig. 8. Plot of % removal of acetic acid Vs contact time at 298K

Effect of contact time: The uptake of carboxylic acid by SMPC and STCRC is found to be rapid at the initial phase of adsorption, but it is becoming more or less constant or attains the equilibrium state, as it is expected in such cases of both acids. The percentage of removal of carboxylic acids by adsorption on Saraca asoca (Rexb) de wilde (SSAD) slightly increases with increase in contact time and reaches a limiting value (Zagorodni *et al.*, 2002).

Table 9. Effect of Variation of contact time for Trichloroacetic acid on SMPC and STCRC

S.No.	[Cl ₃ CCOOH] (N)	% Removal of Acid by SMPC	% Removal of Acid by STCRC
1	0	16.1	11.3
2	5	36.9	23.89
3	7	43.9	38.44
4	10	61.8	49.09
5	15	70.2	66.23
6	20	70.25	65.28
7	30	70.2	66.23
8	45	69.59	66.18
9	60	69.65	67.53
10	90	68.98	66.18

Initial concentration = 0.05 N ; Sulphonated form of Mimosa Pudica and Terminalia Chebula Retz (gm) = 20g L⁻¹; Temperature = 35 ± 1°C

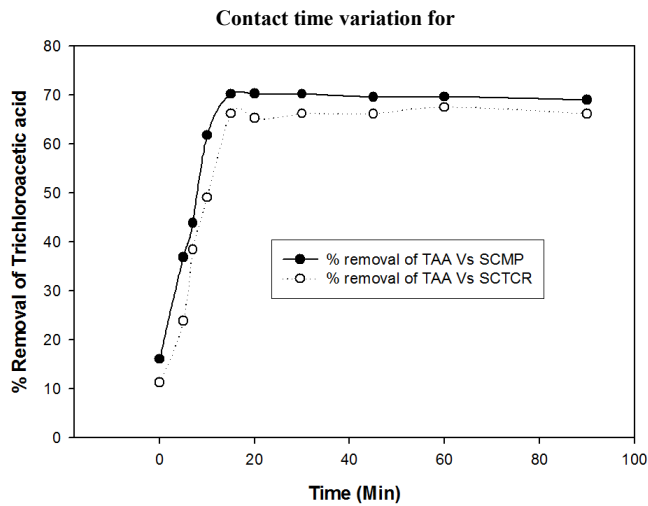


Fig. 9. Plot of % removal of trichloroacetic acid Vs contact time at 298K

Table 10. Effect of Variation of Dose for Formic acid on SMPC and STCRC

S.No.	Amount of SC (g/L)	% Removal of Acid by SMPC	% Removal of Acid by STCRC
1	0.25	10.55	11.5
2	0.50	13.30	18.5
3	0.75	16.51	21.0
4	1.00	26.21	22.31
5	1.50	36.85	32.0
6	2.00	45.32	41.5

Initial concentration = 0.05 N; Temperature = 35 ± 1°C; Contact Time = 15 Min.

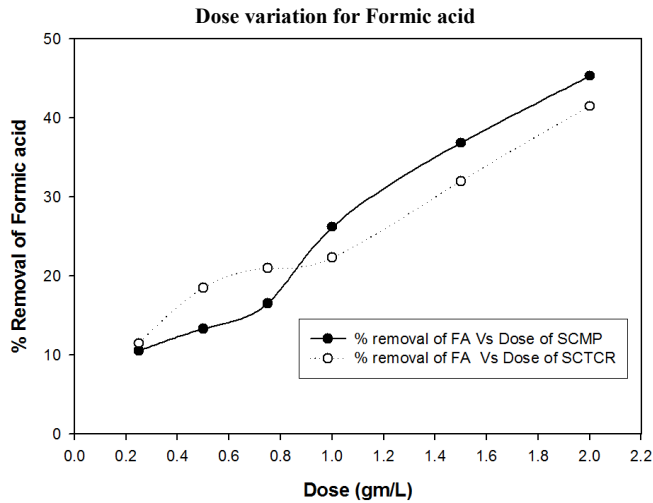


Fig. 10. Plot of % removal of Formic acid vs dose at 298K

Table 11. Effect of Variation of Dose for Acetic acid on SMPC and STCRC

S.No.	Amount of SC (g/L)	% Removal of Acid by SMPC	% Removal of Acid by STCRC
1	0.25	10.55	11.5
2	0.50	15.30	14.5
3	0.75	26.51	21.0
4	1.00	38.6	27.03
5	1.50	36.85	38.0
6	2.00	66.32	52.5

Initial concentration = 0.05 N; Temperature = 35 ± 1°C; Contact Time = 15 Min.

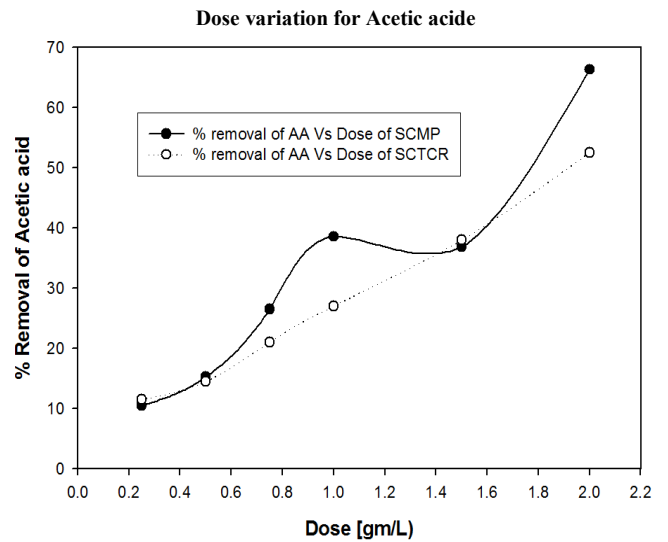


Table 12. Effect of Variation of Dose for Trichloroacetic acid on SMPC and STCRC

S.No.	Amount of SC (g/L)	% Removal of Acid by SMPC	% Removal of Acid by STCRC
1	0.25	15.55	14.5
2	0.50	35.30	32.5
3	0.75	46.51	41.0
4	1.00	70.2	65.52
5	1.50	96.85	89.0
6	2.00	136.32	115.5

Initial concentration = 0.05 N; Temperature = 35 ± 1°C; Contact Time = 15 Min.

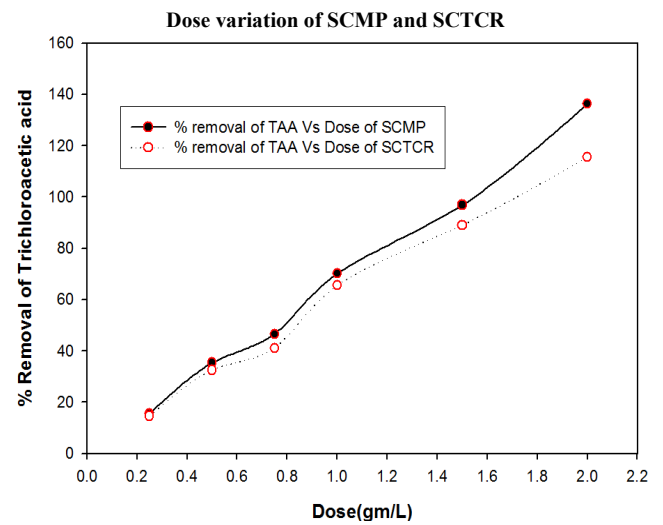


Fig. 12 Plot of % removal of trichloro acetic acid vs dose at 298K

Effect of dosage

The variation in the percentage of removal with the dose of adsorbent are raphically represented. The percentage of removal of Formic acid, Acetic acid and Trichloroacetic acid is found to increase with increase in the dose of the adsorbent (TCR). This is due to the increased availability of adsorption sites (Anderson, 1962). The increase in the effective surface area resulting from the conglomeration of the adsorbent especially at higher adsorbent concentration is also responsible for this observation (Tables 9-12 and Figures 9-12)

Conclusion

The results of the present investigation concluded that the SMPC is the superior adsorbent while compared to the STCRC for the removal of Formic acid, Acetic acid and Trichloroacetic acid from the industrial wastewater. Then, the adsorption capacity of SMPC to adsorb Trichloroacetic acid is found to be higher than that of the Formic acid and Acetic acid. Moreover, from the results of the present study, it is found that the Sulphonated form of SMPC is a very good adsorbent than the Powdered form of SMPC for the removal of Formic acid, Acetic acid and Trichloroacetic acid from aqueous solutions at diluted condition. This result of this study will be useful in designing the effluent treatment plant.

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