Chapter-III

Evaluation of the Effect of Surfactants on the Adsorption and Movement of Carbaryl in soils of Divergent Texture
Chapter-III

EVALUATION OF THE EFFECT OF SURFACTANTS ON THE ADSORPTION AND MOVEMENT OF CARBARYL IN SOILS OF DIVERGENT TEXTURE

3.1. INTRODUCTION

Soil colloids are known to react with pesticides affecting their stability and biological activity in soils as well as environmental pollution. Environmental protection thus has become a major issue in agriculture during the last several years. The frequent detection of pesticides in soil, sediments and in surface and ground waters samples as pollutants has contributed to the need for experimental studies on pesticide adsorption and movement in soils so as to overcome the problems of environmental pollution. Carbaryl (1-naphthyl-N-methylcarbamate) is a systemic, non-ionic, contact insecticide which is widely used in Indian subcontinent against all major insects, pests of cereals, fruits, vegetables and other crops. Its structure can be represented as:

![Chemical structure of carbaryl](image)

Most of the data on carbaryl adsorption on soils and clays deals with aqueous solutions [1-3]. Recently, Singh et al. [4] have studied the effect of water miscible organic solvents (acetone and methanol) on the adsorption and movement of carbaryl in soils and verified the cosolvent theory. Much attention has now been paid to study the effect of surfactants on the adsorption of hydrophobic organic compounds (HOCs)/pesticides on soils. Several researchers [5-6] have shown that surfactant and pesticide compounds can interact under certain conditions and therefore influence the
pesticides adsorption and movement in soils. It has been investigated that the nonionic and anionic surfactants above their critical micelle concentrations (CMCs) strongly enhance the solubility of HOCs/pesticides by partitioning it into the hydrophobic cores of surfactant micelles and represents an important tool in chemical and biological remediation of contaminated soils and sediments [7-8]. Other type of surfactants such as cationic ones may be retained by soil colloids and may enhance a HOCs adsorption with a potential to increase the formation of bound residues [9]. Recently, Ahmad et al. [10] have reported the surfactant enhanced release of carbaryl from two long term contaminated soils. But information pertaining to the effect of surfactants on the adsorption and movement of carbaryl in soils is not available in the literature. Hence, in the present investigation, an attempt has been made to study the adsorption and movement of carbaryl in four Indian soils of divergent texture in the absence and presence of aqueous solutions of various surfactants of different charge properties viz. cationic (cetyl trimethyl ammonium bromide, CTAB), non-ionic (polyethylene glycol tert-octyl phenyl ether, TX-100) and anionic (sodium dodecyl sulphate, SDS) at different CMCs (0.1 \times \text{CMC}, 1 \times \text{CMC} and 10 \times \text{CMC}) with a view to understand how the adsorption and movement of carbaryl is altered by the presence of these surfactants when both coexist in soils as a result of human activity as well as possible development of the use of surfactants in solving pollution problems posed by carbaryl in soils.

3.2. EXPERIMENTAL

3.2.1. Chemicals and Reagents

Carbaryl Solution:

Carbaryl (technical grade; aqueous solubility 0.104 mg L$^{-1}$; log $K_{ow} = 2.35$) was obtained from M/S Bayer Crop Science India Ltd., Mumbai. A stock solution of carbaryl of 1000 μg mL$^{-1}$ concentration was prepared by dissolving requisite amount of carbaryl in 100 mL methanol (AR grade, E. Merck, India Ltd.). Methanol was the preferred solvent as it showed no effect on the surfactant solubilization; higher alcohols could not be utilized since they affect CMC values significantly [11].
0.03 % p-nitrobenzenediazonium tetrafluoroborate Solution:

0.03 g of p-nitrobenzenediazonium tetrafluoroborate (MERCK-Schuchardt, Germany) was dissolved in 100 mL methanol.

0.5 N Sodium hydroxide (NaOH) Solution:

20 g of NaOH (AR grade, CDH Chemicals Ltd. India) was dissolved in 1000 mL of distilled water.

Cetyl trimethyl ammonium bromide (CTAB) Solution:

0.68 g of CTAB (AR grade, CDH Chemicals Ltd. India) was dissolved in 100 mL distilled water. The selected properties of CTAB are given in Table 2.1 of Chapter-II.

Polyethylene glycol tert-octyl phenyl ether (TX-100) Solution:

0.30 g of TX-100 (AR grade, CDH Chemicals Ltd. India) was dissolved in 100 mL distilled water. The selected properties of TX-100 are given in Table 2.1 of Chapter-II.

Sodium dodecyl sulphate (SDS) Solution:

4.76 g of SDS (AR grade, CDH Chemicals Ltd. India) was dissolved in 100 mL distilled water. The selected properties of CTAB are given in Table 2.1 of Chapter-II.

3.2.2. Apparatus

The apparatus used in this study is same as given in section 2.2.2 of Chapter-II.

3.2.3. Collection of Soil Samples

The soils used in the present study were collected from the surface horizon (0–30 cm) of cultivated fields from the villages Alampur Rani (silt loam soil), Kalai (loam soil) and Bhoran (sandy loam soil) in the Aligarh district (U.P.), and Forest
Research Institute F.R.I. Farm (silt loam soil) in the Dehradoon district (Uttarakhand) in India. The soil samples were air-dried, ground to pass through a 2-mm sieve and stored in plastic bags at room temperature. The physico-chemical properties of the soils were determined by standard methods given in section 2.2.4. of Chapter-II and values obtained are listed in Table 2.2 of Chapter-II.

3.2.4. Preparation of Standard Curve of Carbaryl

Standard curve (Figure 3.1) of carbaryl was prepared by using the method proposed by Stansbury and Miskus [12] in which varying volumes (0 to 5 mL) of 10 µg mL\(^{-1}\) carbaryl solution were pipetted into a number of 25 mL standard flasks. To these solutions 5 mL of 0.5 N NaOH and 5 mL of 0.03% methanolic solution of p-nitrobenzenediazonium tetrafluoroborate was added and the volume in each flask was made up to the mark with methanol and left for 30 min. The absorbance was recorded against reagent blank at 590 nm using T-70 UV/VIS Spectrophotometer.

3.2.5. Adsorption Studies

Batch equilibrium adsorption experiments of carbaryl on soils in the absence and presence of aqueous solutions of cationic, non-ionic and anionic surfactants at different CMCs (0.1 x CMC, 1 x CMC and 10 x CMC) were performed by taking eight concentrations of carbaryl (5, 10, 15, 20, 25, 30, 35, 40 µg mL\(^{-1}\)) in 100 mL glass stoppered conical flasks. The volume contained in each flask was made up to 20 mL by the addition of the requisite volume of methanol and surfactant solutions of different CMCs. To these solutions 1 g of each soil was added and the resulting suspensions were kept at 25±1°C for 24 hr in an incubator with intermittent shaking period of 3 hr. All the experiments were conducted in triplicate. Preliminary experiments revealed a contact time of 24 hr to be sufficient for equilibrium to be reached both in the absence or presence of various surfactants at the three CMC values employed. The suspensions were then centrifuged at 15000 rpm for 10 min using a Beckman model L3 – 50 Ultracentrifuge and the carbaryl in supernatant liquids estimated spectrophotometrically [12] using T-70 UV/VIS Spectrophotometer at 590 nm. This method has a detection limit of 0.1 µg mL\(^{-1}\). The instrument has a minimum detection limit of ±0.002 Abs. The amount of carbaryl
Figure: 3.1. Standard curve of carbaryl.
adsorbed was calculated as the difference between the initial and equilibrium concentrations in solution using the following expression:

\[ \frac{x}{m} = (C_0 - C_e) \frac{V}{W} \] (3.1)

where \( \frac{x}{m} \) is the surface concentration of carbaryl in the soil (\( \mu g \ text{ g}^{-1} \)), \( C_0 \) is the initial concentration of carbaryl in solution (\( \mu g \ text{ mL}^{-1} \)), \( C_e \) is the equilibrium concentration of carbaryl in solution (\( \mu g \ text{ mL}^{-1} \)), \( V \) is the volume of solution and \( W \) is the weight of the soil employed.

### 3.2.6. Soil Thin-Layer Chromatography (Soil TLC)

The movement of carbaryl in soils was studied using the soil TLC technique [13]. The detailed method for preparing the TLC plates is described in section 2.2.8 of Chapter-II. A 10\( \mu \)L volume of a carbaryl solution of 1000 \( \mu g \text{ mL}^{-1} \) concentration in methanol was applied as spot on the soil TLC plates with the help of a lambda pipette held 3 cm above the bottom of the plates. A 2 cm wide strip of paper towel moistened with eluents (water and surfactant solutions of different CMCs) was wrapped around the bottom of the plates to prevent disintegration of the soil layer when it came into contact with the eluents. The carbaryl spotted plates were eluted in distilled water and surfactant solutions of different critical micelle concentrations (0.1 \times \text{CMC}, 1 \times \text{CMC} and 10 \times \text{CMC}), positioning the plates at an angle of 45° in the glass tank. After the eluent had migrated to a distance of 10 cm from the base line, the plates were taken out and dried at room temperature. The movement of carbaryl was detected by spraying the developed plates with a 5% methanolic KOH solution followed by spraying with 0.1% methanolic solution of \( p \)-nitrobenzenediazonium tetrafluoroborate. The development of dark blue-black spots indicated the presence of carbaryl. The movement of carbaryl was expressed in terms of the \( R_f \) values [13] and the results obtained are summarized in Tables 3.2 – 3.4.

### 3.2.7. Statistical Analysis

Statistical analysis was performed using SPSS 11.0 for Windows to obtain correlation between adsorption constants (Freundlich constant and distribution coefficient) versus different physico-chemical properties of the soils and the values obtained are given in Tables 3.5 and 3.6.
3.3. RESULTS AND DISCUSSION

The adsorption isotherms of carbaryl on soils in the absence and presence of aqueous solutions of three surfactants at different CMCs are depicted in Figures 3.2 – 3.4. The corresponding adsorption isotherm data is given in Tables 3.1.1 – 3.1.40. All these isotherms indicate the amount of carbaryl adsorbed per unit mass of solid adsorbent (x/m, µg g⁻¹) versus the equilibrium concentration (Cₑ, µg mL⁻¹). From the isotherms it is clear that the adsorption follows the order as F.R.I. silt loam > Alampur silt loam > Kalai loam > Bhoran sandy loam soil in surfactant free as well as in aqueous surfactant solutions at all CMCs except at 1 x CMC and 10 x CMC level of aqueous non-ionic surfactant (TX-100) solutions. The higher adsorption on F.R.I. silt loam soil is may be due to higher amount of organic carbon, and clay content compared to Alampur silt loam, Kalai loam and Bhoran sandy loam soils (Tables 3.2 – 3.4). All the isotherms for both types of systems i.e. in the absence and presence of surfactants at varying CMCs are L-shaped [14] except at 10 x CMC of CTAB in F.R.I. silt loam, Alampur silt loam and Kalai loam soils, where the isotherms are H-shaped. The L-shaped isotherms indicate a high degree of affinity of the adsorbent for the adsorbate and suggest that as the active sites of the adsorbent became saturated, the adsorption of new molecules occurred with great difficulty. The H-shaped isotherms are the special case of L-shaped isotherms and indicate high affinity between the adsorbent and the adsorbate, such that adsorption is total and the final concentration of the solution is zero.

With the exception of those of the H-shaped, all L-shaped isotherms could be described by the empirical Freundlich equation,

\[ \log x/m = \log K_F + 1/n \log C_e \]  

(3.2)

where \( K_F \) and \( 1/n \) are the constants associated with the affinity of the adsorbate for the adsorbent and the degree of curvature of the isotherms, respectively. The dimension of \( K_F \) is \( \mu g \ (1-n) \ mL^n \ g^{-1} \) whereas \( 1/n \) is dimensionless. In general the values of the coefficient of determination (\( r^2 \)) were greater than 0.95. The values of \( K_F \) and \( 1/n \) for carbaryl-soil combinations as estimated from the linear regression of the logarithmically transformed data are summarized in Tables 3.2 – 3.4. The
corresponding Freundlich adsorption isotherms are given in Figures 3.5 – 3.7. The values of $1/n < 1$ indicate a degree of non-linearity between solution equilibrium concentration and adsorption, and are in agreement with the L-shape of the isotherms. The lack of linearity may be attributed to specific interactions existing between compounds with polar groups and the organic matter or the mineral fraction of the soils [15-16].

As the values of $1/n$ depart from unity it was considered appropriate to use the distribution coefficient, $K_D$, as a measure of the soil adsorption capacity as it represents the adsorption under equilibrium conditions. The statistical average of the $K_D$ values for each soil in surfactant free and in surfactant-soil-water systems were evaluated from the relationship:

$$K_D = \frac{\sum x/m \cdot C_e}{\sum (C_e)^2}$$  \hspace{1cm} (3.3)

and the values obtained are summarized in Tables 3.2 – 3.4. The Freundlich constant and distribution coefficient in surfactant free systems are denoted as $K_F$ and $K_D$; whereas in surfactant-soil-water systems are denoted as $K_F^*$ and $K_D^*$ respectively.

The values of $K_F^*$ and $K_D^*$ in surfactant-soil-water systems shows that the adsorption of carbaryl was higher in presence of cationic surfactant followed by anionic and non-ionic surfactants (cationic > anionic > non-ionic) at all CMCs on all the four soils except at 10 x CMC level in Kalai loam and Bhoran sandy loam soils.

The frontal $R_f$ (FRf) values obtained from soil TLC studies are inversely proportional to the $K_F$, $K_D$ (surfactant free) and $K_F^*$, $K_D^*$ (surfactant-water systems) at all CMCs for all the soils studied (Tables 3.2 – 3.4) and confirmed the above adsorption pattern of carbaryl in different surfactants employed.

3.3.1. Effect of Cationic Surfactant (CTAB)

Figure 3.2 (a-d) and the adsorption isotherm data in Table 3.2 represents the carbaryl adsorption on four soils in the absence and presence of different CMCs (0.1 x CMC, 1 x CMC and 10 x CMC) of cationic surfactant CTAB.

The observed higher values of $K_F^*$ and $K_D^*$ for carbaryl at 0.1 x CMC compared to surfactant free system for all the four soils demonstrate that adsorption of
Fig. 3.2 (a) F.R.I. silt loam soil

Fig. 3.2 (b) Alampur silt loam soil

Figure: 3.2. Continued......

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**Fig. 3.2 (a) F.R.I. silt loam soil**

- Amount of carbaryl adsorbed (µg g⁻¹ soil)
- Ce
- Amount of carbaryl in equilibrium suspension (µg mL⁻¹)

- Without Surfactant
- 0.1 x CMC
- 1 x CMC
- 10 x CMC

**Fig. 3.2 (b) Alampur silt loam soil**

- Amount of carbaryl adsorbed (µg g⁻¹ soil)
- Ce
- Amount of carbaryl in equilibrium suspension (µg mL⁻¹)

- Without Surfactant
- 0.1 x CMC
- 1 x CMC
- 10 x CMC
Figure: 3.2 (a-d). Adsorption isotherms of carbaryl on four soils in different CMCs of cetyl trimethyl ammonium bromide (CTAB).
Figure: 3.3. Continued......
Figure: 3.3 (a-d). Adsorption isotherms of carbaryl on four soils in different CMCs of polyethylene glycol tert-octyl phenyl ether (TX-100).
Figure 3.4. Continued......
Figure: 3.4 (a-d). Adsorption isotherms of carbaryl on four soils in different CMCs of sodium dodecyl sulphate (SDS).
<table>
<thead>
<tr>
<th>Amount of carbaryl added (μg g⁻¹)</th>
<th>Amount of carbaryl in equilibrium suspension (μg/20 mL)</th>
<th>Cₓ/m (μg mL⁻¹)</th>
<th>(Cₓ)²</th>
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Table: 3.1.1. Adsorption of carbaryl on F.R.I. silt loam soil in aqueous (surfactant-free) systems.
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Table: 3.1.3. Adsorption of carbaryl on Kalai loam soil in aqueous (surfactant-free) systems.

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<th>Amount of carbaryl in equilibrium suspension (µg mL⁻¹) (Cₑ)</th>
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Table: 3.1.4. Adsorption of carbaryl on Bhoran sandy loam soil in aqueous (surfactant-free) systems.

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Table: 3.1.8. Adsorption of carbaryl on Alampur silt loam soil at 0.1 x CMC of cationic surfactant ‘CTAB’.

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Table: 3.1.11. Adsorption of carbaryl on Kalai loam soil at 0.1 x CMC of cationic surfactant ‘CTAB’.

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Table: 3.1.12. Adsorption of carbaryl on Kalai loam soil at 1 x CMC of cationic surfactant ‘CTAB’.

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Table: 3.1.13. Adsorption of carbaryl on Kalai loam soil at 10 x CMC of cationic surfactant ‘CTAB’.

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109
Table: 3.1.14. Adsorption of carbaryl on Bhoran sandy loam soil at 0.1 x CMC of cationic surfactant ‘CTAB’.

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Table: 3.1.16. Adsorption of carbaryl on Bhara sand loam soil at 10 x CMC of cationic surfactant - "CTAB".
Table: 3.1.17. Adsorption of carbaryl on F.R.I. silt loam soil at 0.1 x CMC of non-ionic surfactant ‘TX-100’.

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Table: 3.1.18. Adsorption of carbaryl on F.R.I. silt loam soil at 1 x CMC of non-ionic surfactant 'TX-100'
Table: 3.1.19. Adsorption of carbaryl on F.R.I. silt loam soil at 10 x CMC of non-ionic surfactant ‘TX-100’.

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<th>Amount of carbaryl in equilibrium suspension per g soil (µg g⁻¹)</th>
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Note: The table data is presented in a standard format for readability.
Table: 3.1.21. Adsorption of carbaryl on Alampur silt loam soil at 1 x CMC of non-ionic surfactant 'TX-100'.

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<th>Amount of carbaryl in equilibrium suspension (μg mL⁻¹) (Cₑ)</th>
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Table: 3.1.22. Adsorption of carbaryl on Alampur silt loam soil at 10 x CMC of non-ionic surfactant ‘TX-100’.

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Table 3.1.23. Adsorption of carbaryl on Kalai loam soil at 0.1 x CMC of non-ionic surfactant 'TX-100'.

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Table: 3.1.24. Adsorption of carbaryl on Kalai loam soil at 1 x CMC of non-ionic surfactant ‘TX-100’.

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<th>Amount of carbaryl adsorbed per g soil (µg g⁻¹) (x/m)</th>
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Table: 3.1.25. Adsorption of carbaryl on Kalai loam soil at 10 x CMC of non-ionic surfactant ‘TX-100’.

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Table: 3.1.26. Adsorption of carbaryl on Bhoran sandy loam soil at 0.1 x CMC of non-ionic surfactant 'TX-100'.

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Table: 3.1.27. Adsorption of carbaryl on Bhoran sandy loam soil at 1 x CMC of non-ionic surfactant 'TX-100'.

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Table: 3.1.28. Adsorption of carbaryl on Bhoran sandy loam soil at 10 x CMC of non-ionic surfactant 'TX-100'.

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Table: 3.1.29. Adsorption of carbaryl on F.R.I. silt loam soil at 0.1 x CMC of anionic surfactant ‘SDS’.

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Table 3.1: Adsorption of carbaryl on F.R.I. silt loam soil at 1 x CMC of anionic surfactant 'SDS'.
Table: 3.1.31. Adsorption of carbaryl on F.R.I. silt loam soil at 10 x CMC of anionic surfactant ‘SDS’.

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Table: 3.1.32. Adsorption of carbaryl on Alampur silt loam soil at 0.1 x CMC of anionic surfactant ‘SDS’.

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<th>Amount of carbaryl adsorbed per g soil (µg g⁻¹) (x/m)</th>
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Table 3.1.33. Adsorption of carbaryl on Alampur silt loam soil at 1 x CMC of anionic surfactant 'SDS'.

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<th>Amount of carbaryl in equilibrium suspension (µg mL⁻¹) (Ce)</th>
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Table: 3.1.34. Adsorption of carbaryl on Alampur silt loam soil at 10 x CMC of anionic surfactant 'SDS'.

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Table: 3.1.35. Adsorption of carbaryl on Kailai loam soil at 0.1 x CMC of anionic surfactant ‘SDS’.

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Table: 3.1.36. Adsorption of carbaryl on Kalai loam soil at 1 x CMC of anionic surfactant ‘SDS’.

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<th>Amount of carbaryl in equilibrium suspension (µg mL⁻¹) (Cₑ)</th>
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Table: 3.1.37. Adsorption of carbaryl on Kalai loam soil at 10 x CMC of anionic surfactant ‘SDS’.

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<th>Amount of carbaryl adsorbed per g soil (µg g⁻¹) (x/m)</th>
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Table: 3.1.38. Adsorption of carbaryl on Bhoran sandy loam soil at 0.1 x CMC of Anionic surfactant ‘SDS’.

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<th>Amount of carbaryl in equilibrium suspension (µg mL⁻¹) (Cₑ)</th>
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Table: 3.1.39. Adsorption of carbaryl on Bhoran sandy loam soil at 1 x CMC of anionic surfactant ‘SDS’.

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<th>Amount of carbaryl adsorbed per g soil (µg g⁻¹) (x/m)</th>
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Table: 3.1.40. Adsorption of carbaryl on Bhoran sandy loam soil at 10 x CMC of anionic surfactant ‘SDS’.

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<th>Amount of carbaryl adsorbed per g soil (µg g⁻¹) (x/m)</th>
<th>Amount of carbaryl in equilibrium suspension (µg mL⁻¹) (Cₑ)</th>
<th>Cₑx/m</th>
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Figure 3.5. Continued....
Figure: 3.5 (a-d). Freundlich adsorption isotherms of carbaryl on four soils in different CMCs of cetyl trimethyl ammonium bromide (CTAB).
Fig. 3.6 (b) Alampur silt loam soil

Fig. 3.6 (g) F.R.I. silt loam soil

Figure: 3.6. Continued......
Figure 3.6 (a-d). Freundlich adsorption isotherms of carbaryl on four soils in different CMCs of polyethylene glycol tert-octyl phenyl ether (TX-100).
Figure 3.7. Continued...
Figure: 3.7 (a-d). Freundlich adsorption isotherms of carbaryl on four soils in different CMCs of sodium dodecyl sulphate (SDS).
carbaryl increase at this CMC value because CTAB is cationic in nature and adsorbed (cation exchanged) by soils in the form of hemimicelle/admicelle [17]. This soil bound surfactant increases the hydrophobicity of the soil surfaces and increase the adsorption of carbaryl. However, at 1 x CMC and 10 x CMC the observed Kp* and Kp* values increased markedly compared to the surfactant free system (Table 3.2). This marked increase in adsorption may be due to the fact that at 1 x CMC and 10 x CMC the amount of CTAB adsorbed by the soils must be greater than 0.1 x CMC together with the fact that the adsorption capacity of CTAB in micellar form is greater than monomer form. Apart from the great adsorption capacity of the cationic surfactants it has also been reported by several researchers [18-19] that the application of surfactants results in drastic increase in soil dispersion, thus increasing the soil surface area and the amount of available sites for adsorption. The mechanical disintegration of particles in batch experiments due to sample shaking may also increase available sites for adsorption resulting in an increase in the adsorption capacity of the soil. The results obtained are in accordance with the work of Iglesias-Jimenez et al. [6] and Singh and Rajkumar [20] who studied the effect of surfactants on adsorption of pesticides in soil-water systems.

3.3.2. Effect of Non-Ionic Surfactant (TX-100)

The adsorption isotherm data in Table 3.3 and Figure 3.3 (a-d) represents the carbaryl adsorption on four soils in the absence and presence of different CMCs (0.1 x CMC, 1 x CMC and 10 x CMC) of non-ionic surfactant TX-100.

In F.R.I. silt loam and Alampur silt loam soils the Kp* and Kp* values of carbaryl decrease as the concentration of TX-100 increases from 0.1 x CMC to 10 x CMC. Whereas in Kalai loam and Bhoran sandy loam soils the Kp* and Kp* values of carbaryl increase as the concentration increases from 0.1 x CMC to 10 x CMC of TX-100 with respect to its adsorption in surfactant free systems (Table 3.3).

In F.R.I. silt loam and Alampur silt loam soils decrease in adsorption with increase in CMC may be attributed to the inefficiency of TX-100 to adsorb on high organic matter soils [21-22]. The similar results were reported by Ahmad et al. [10]. They reported an increase in desorption of carbaryl with increase in TX-100 concentration.
In Kalai loam and Bhoran sandy loam soils the $K_F^*$ and $K_D^*$ values of carbaryl increase with increase in CMC showing an increase in the adsorption of carbaryl with respect to its adsorption in surfactant free system. The increase in adsorption in Kalai loam soil may be attributed to the largest amount of clay content in this soil and TX-100 is known to adsorb readily on the soil mineral surfaces [22]. In Bhoran sandy loam soil increase in adsorption may be attributed to the lesser amount of organic matter associated with this soil. The results obtained are in accordance with the work of Rodriguez-Cruz et al. [23]. They reported an increase in desorption of pesticides (atrazine and diuron) with increase in TX-100 concentration in soils with highest organic matter content (as is the case with F.R.I. and Alampur silt loam soils). Whereas in soils with lowest organic matter content, desorption was not favourable (as in Kalai and Bhoran sandy loam soils). Generally, the non-ionic surfactant adsorption on soils is correlated mainly with the composition of the mineral phase on which the adsorption/desorption of the contaminants is similarly dependent [24-25]. Non-ionic surfactants adsorbed to low organic matter soils (such as Bhoran sandy loam soil) are mainly correlated with the mineral properties of the soils [22]. The Kalai loam soil with a relatively higher clay mineral fraction and Bhoran sandy loam soil with least organic matter content adsorbs a greater amount of surfactant, to which carbaryl can partition on the soil surface leading to an obvious increase in $K_F^*$ and $K_D^*$ values. Variations in surfactant effect as a function of soil organic matter content have also been observed by Werkheiser and Anderson [26] for the adsorption of herbicide primisulfuron in the presence of Triton X-77. Other authors have also reported variations in the effect of non-ionic surfactants as being due to soil clay mineralogy [27].

3.3.3. Effect of Anionic Surfactant (SDS)

Figure 3.4 (a-d) and the adsorption isotherm data in Table 3.4 represents the carbaryl adsorption on four soils in the absence and presence of different CMCs (0.1 x CMC, 1 x CMC and 10 x CMC) of anionic surfactant SDS.

In all the four soils the $K_F^*$ and $K_D^*$ values of carbaryl increase at 0.1 x CMC and 1 x CMC of SDS with respect to its adsorption in surfactant free systems (Table 3.4). But at 10 x CMC the $K_F^*$ and $K_D^*$ values for carbaryl decrease in all the four
soils. The increase in carbaryl adsorption at 0.1 x CMC and 1 x CMC is might be due to the adsorption of SDS by soils. Notwithstanding its anionic character, it has been reported that SDS is adsorbed by soils and clays [28]. At higher SDS concentration i.e. 10 x CMC, \( K_F^* \) and \( K_D^* \) values decreases considerably, indicating a decrease in carbaryl adsorption with respect to its adsorption in surfactant free system in all the four soils. This is due to the formation of aqueous surfactant micelles which effectively compete with the solid phase as a sorptive medium for hydrophobic compounds like carbaryl. The similar results were reported by Iglesias-Jimenez et al. [6] for the adsorption of pesticide ethofumesate in presence of different concentration of SDS; and Lee et al. [22] who studied the effect of surfactants on the distribution of organic compounds in the soil solid/water system.

Wang and Keller [29] also reported similar adsorption pattern for the two pesticides, atrazine and diuron in presence of anionic surfactant LAS. In this study, with an increase in LAS concentration upto the CMC the adsorption of these pesticides increase due to soil adsorption and co-precipitation of LAS with the divalent cations, followed by partitioning of pesticide into the adsorbed and precipitated LAS. However after the CMC the decrease in the pesticide adsorption was due to the increasing micelle concentration and the release of pesticides from the precipitated LAS.

### 3.3.4. Remediation Efficiency

The \( K_D^*/K_D \) ratios were used to define the remediation efficiency of various surfactants in soils studied. If the \( K_D^*/K_D \) ratio exceeds one, remediation of a pesticide contaminated soil is considered to be inefficient for the system involved. Conversely, a ratio of less than one means that the soil remediation is favourable [22]. The observed \( K_D^*/K_D \) values are shown in Tables 3.2 – 3.4.

The ratios of \( K_D^*/K_D \) for CTAB-soil-water systems are greater than one, and increases with increase in CMC for all the four soils (Table 3.2). This shows the poor remediation efficiency of this surfactant as CTAB is cationic in nature and aggregate readily on the negatively charged soil surfaces.
Table 3.2: Carbaryl adsorption data on four soils in different CMCs of cetyl trimethyl ammonium bromide (CTAB).

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<tbody>
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<td>K(_D^*)</td>
<td>(r^2)</td>
<td>K(_F)</td>
<td>K(_F^*)</td>
<td>1/n</td>
<td>K(_OC)</td>
<td>K(_C)</td>
<td>R(_f)</td>
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<td>95.50</td>
<td>-</td>
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<td>-</td>
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<td>CMC (g L⁻¹)</td>
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<td>K₁ Sistema</td>
<td>Kₒ</td>
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</table>

Table: 3.3. Carbaryl adsorption data on four soils in different CMCs of polyethylene glycol tert-octyl phenyl ether (T-OX-100).
Table: 3.4. Carbaryl adsorption data on four soils in different CMCs of sodium dodecyl sulphate (SDS).

<table>
<thead>
<tr>
<th>Soils</th>
<th>CMC (g L⁻¹)</th>
<th>Parameters</th>
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<th></th>
<th></th>
<th></th>
<th></th>
<th>Rₚ</th>
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<tbody>
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<td>K_D</td>
<td>K_D*</td>
<td>r²</td>
<td>K_F</td>
<td>K_F*</td>
<td>1/n</td>
<td>K_OC</td>
<td>K_C</td>
<td>Rₚ</td>
<td>K_D*/K_D</td>
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<td>F.R.I. silt loam</td>
<td>0.00</td>
<td>46.59</td>
<td>-</td>
<td>0.99</td>
<td>95.50</td>
<td>-</td>
<td>0.69</td>
<td>1694</td>
<td>222</td>
<td>0.37</td>
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<td>-</td>
<td>0.99</td>
<td>28.84</td>
<td>-</td>
<td>0.75</td>
<td>2233</td>
<td>88</td>
<td>0.59</td>
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<tr>
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<td>1229</td>
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<td>Kalai loam</td>
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<td>-</td>
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<td>22.39</td>
<td>-</td>
<td>0.81</td>
<td>1788</td>
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<td>3.96</td>
<td>-</td>
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<td>1082</td>
<td>19</td>
<td>0.86</td>
<td>0.53</td>
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</table>
In presence of non-ionic surfactant TX-100, $K_{D^*}/K_D$ ratios are less than one in F.R.I. silt loam and Alampur silt loam soils and greater than one in Kalai loam and Bhoran sandy loam soils at all CMCs studied (Table 3.3). This shows that the remediation efficiency of TX-100 is higher for the soils with high organic matter content and low clay content (F.R.I. and Alampur silt loam soils, respectively) and lower for soils with high clay and low organic matter content (as Kalai loam and Bhoran sandy loam soils respectively). The Kalai loam soil with a relatively higher clay mineral fraction and Bhoran sandy loam soil with least organic matter content adsorbs a greater amount of surfactant, to which carbaryl can partition on the soil surface leading to an obvious increase in $K_{D^*}/K_D$ ratios in these two soils.

The $K_{D^*}/K_D$ ratios in SDS-soil-water systems are greater than one at 0.1 x CMC and 1 x CMC level in all the four soils and indicates lower efficiency of SDS to remediate the soils at these concentrations (Table 3.4). However, the $K_{D^*}/K_D$ ratios drop significantly at 10 x CMC of SDS. This shows that remediation of soils is favourable at this concentration of SDS.

3.3.5. Importance of Soil Organic Carbon and Clay Content on Carbaryl Adsorption

The adsorption coefficient normalized to organic carbon, $K_{OC}$ and clay content, $K_C$ were evaluated from the equations proposed by Gerstl [30]. The values thus obtained are listed in Tables 3.2 – 3.4. They are the important parameters which play a significant role in the environmental fate assessment of organic chemicals. They provide an indication of the extent to which chemical partitioning occurs between the solid and solution phases in the soil and suggest whether the chemical is likely to leach through the soil or be rendered immobile. The affinity of carbaryl towards organic carbon and clay content of the soils may be compared through the use of the $K_{OC}$ and $K_C$ values (Tables 3.2 – 3.4). In the present study the $K_{OC}$ values are higher than the $K_C$ values in both surfactant free and surfactant-soil-water systems and this may lead to the conclusion that the organic matter is the main parameter for carbaryl adsorption. Use of these parameters without realizing their limitations may result in incorrect decisions regarding the pollution hazard of these dangerous
Table: 3.5. Correlation coefficients between Freundlich coefficient, $K_F$ (surfactant free) and $K_F^*$ (surfactant-soil-water systems) versus soil characteristics.

<table>
<thead>
<tr>
<th>CMC g L$^{-1}$</th>
<th>OC</th>
<th>Clay</th>
<th>CEC</th>
<th>S+C</th>
<th>pH</th>
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<tr>
<td>Cetyl trimethyl ammonium bromide (CTAB)</td>
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<td></td>
<td></td>
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</tr>
<tr>
<td>0.00</td>
<td>0.999**</td>
<td>0.348</td>
<td>0.972*</td>
<td>0.700</td>
<td>-0.968*</td>
</tr>
<tr>
<td>0.10 x CMC</td>
<td>1.000**</td>
<td>0.371</td>
<td>0.982*</td>
<td>0.683</td>
<td>-0.954*</td>
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<tr>
<td>1.00 x CMC</td>
<td>0.999**</td>
<td>0.320</td>
<td>0.972*</td>
<td>0.650</td>
<td>-0.963*</td>
</tr>
<tr>
<td>10.00 x CMC</td>
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<tr>
<td>Polyethylene glycol tert-octyl phenyl ether (TX-100)</td>
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<td>0.00</td>
<td>0.999**</td>
<td>0.348</td>
<td>0.972*</td>
<td>0.702</td>
<td>-0.968*</td>
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<tr>
<td>0.10 x CMC</td>
<td>0.978*</td>
<td>0.509</td>
<td>0.999**</td>
<td>0.678</td>
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<td>1.00 x CMC</td>
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<td>10.00 x CMC</td>
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<td>Sodium dodecyl sulphate (SDS)</td>
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</tr>
<tr>
<td>0.00</td>
<td>0.999**</td>
<td>0.348</td>
<td>0.972*</td>
<td>0.702</td>
<td>-0.968*</td>
</tr>
<tr>
<td>0.10 x CMC</td>
<td>1.000**</td>
<td>0.366</td>
<td>0.979*</td>
<td>0.694</td>
<td>-0.959*</td>
</tr>
<tr>
<td>1.00 x CMC</td>
<td>0.999**</td>
<td>0.334</td>
<td>0.972*</td>
<td>0.676</td>
<td>0.966*</td>
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<tr>
<td>10.00 x CMC</td>
<td>0.989*</td>
<td>0.462</td>
<td>0.983*</td>
<td>0.779</td>
<td>-0.937</td>
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*Correlation is significant at 0.05 level. ** Correlation is significant at 0.01 level.
Table: 3.6. Correlation coefficients between distribution coefficient, $K_D$ (surfactant free) and $K_D^*$ (surfactant-soil-water systems) versus soil characteristics.

<table>
<thead>
<tr>
<th>CMC g L$^{-1}$</th>
<th>OC</th>
<th>Clay</th>
<th>CEC</th>
<th>S+C</th>
<th>pH</th>
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<tr>
<td>Cetyl trimethyl ammonium bromide (CTAB)</td>
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</tr>
<tr>
<td>0.00</td>
<td>0.997**</td>
<td>0.344</td>
<td>0.968*</td>
<td>0.710</td>
<td>-0.971*</td>
</tr>
<tr>
<td>0.10 x CMC</td>
<td>0.993**</td>
<td>0.311</td>
<td>0.955**</td>
<td>0.710</td>
<td>-0.981*</td>
</tr>
<tr>
<td>1.00 x CMC</td>
<td>0.995**</td>
<td>0.262</td>
<td>0.953*</td>
<td>0.635</td>
<td>-0.979*</td>
</tr>
<tr>
<td>10.00 x CMC</td>
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<td>0.707</td>
<td>0.687</td>
<td>0.999**</td>
<td>-0.601</td>
</tr>
<tr>
<td>Polyethylene glycol tert-octyl phenyl ether (TX-100)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.00</td>
<td>0.997**</td>
<td>0.344</td>
<td>0.968*</td>
<td>0.710</td>
<td>-0.971*</td>
</tr>
<tr>
<td>0.10 x CMC</td>
<td>0.991**</td>
<td>0.450</td>
<td>0.983*</td>
<td>0.769</td>
<td>-0.941</td>
</tr>
<tr>
<td>1.00 x CMC</td>
<td>0.955*</td>
<td>0.611</td>
<td>0.995**</td>
<td>0.748</td>
<td>-0.836</td>
</tr>
<tr>
<td>10.00 x CMC</td>
<td>0.173</td>
<td>0.844</td>
<td>0.367</td>
<td>0.240</td>
<td>0.117</td>
</tr>
<tr>
<td>Sodium dodecyl sulphate (SDS)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.00</td>
<td>0.997**</td>
<td>0.344</td>
<td>0.968*</td>
<td>0.710</td>
<td>-0.971*</td>
</tr>
<tr>
<td>0.10 x CMC</td>
<td>0.999**</td>
<td>0.325</td>
<td>0.974*</td>
<td>0.640</td>
<td>-0.958*</td>
</tr>
<tr>
<td>1.00 x CMC</td>
<td>0.998**</td>
<td>0.332</td>
<td>0.976*</td>
<td>0.632</td>
<td>-0.952*</td>
</tr>
<tr>
<td>10.00 x CMC</td>
<td>0.987*</td>
<td>0.262</td>
<td>0.939*</td>
<td>0.690</td>
<td>-0.990*</td>
</tr>
</tbody>
</table>

*Correlation is significant at 0.05 level. ** Correlation is significant at 0.01 level.
chemicals [30]. Therefore, by means of a simple statistical approach the data obtained were subjected to simple correlation analysis between $K_F$, $K_D$ (surfactant free) and $K_F^*$, $K_D^*$ (surfactant-soil-water systems) versus organic carbon, clay and other soil parameters with a view to knowing the relative importance of these soil parameters on adsorption of carbaryl on soils. The values obtained are listed in Tables 3.5 - 3.6. On examination of these tables it can be noted that the correlation of adsorption coefficients with the soil clay content for carbaryl was rather poor while the correlation with the soil organic carbon content was highly significant ($r^2 < 1.00; \ p < 0.01$). This confirms the high affinity of carbaryl towards organic carbon than the clay content of the soils. The highly significant correlation with organic carbon content also indicates that adsorption of carbaryl occurs primarily through hydrophobic processes and partitioning to soil organic carbon. The highly significant correlations (Tables 3.5 – 3.6) were also obtained with cation exchange capacity, CEC ($r^2 < 0.98; \ p < 0.05$) of the soils. This shows that however the organic carbon content is of major importance in the adsorption of many organic chemicals in soils, other soil components may also contribute significantly to adsorption in certain cases [31]. Higher correlation with the CEC also suggests that the role of clay content is also important especially for the soils (Kalai loam and Bhoran sandy loam) where the organic matter content is low [4]. The influence of both soil fractions is due to the structure of the carbaryl molecule which contains both a hydrophobic part (1-naphthyl-N-methyl) and a polar part (-COO). The colloidal fractions, organic carbon and clay frequently influence the adsorption of pesticides with hydrophobic and polar groups in their molecular structure [32, 33]. Sheng et al. [2] also reported that due to its two-ring n-electron system carbaryl may participate in the formation of an electron donor-acceptor complex with the siloxane surfaces depending on the inductive and resonance properties of the N-methylcarbamate (−OCONHCH₃) moiety. The results are in accordance with the work of Walker and Crawford [34] and Stevenson [35] who reported that, up to an organic matter content of ca. 6%, both organic and mineral surfaces are involved in adsorption. The variability of $K_{OC}$ values among four soils in surfactant free as well as in surfactant-soil-water systems at different CMCs ($K_{OC}$ ranging from 595-180148) also showed that adsorption was not predictable exclusively from the organic matter content of the soils [36].
The results obtained indicate that the behaviour of carbaryl in soil-water systems in presence of cationic, non-ionic and anionic surfactants depends on the charge properties of the surfactants, the degree of hydrophobicity or polarity of the pesticide (carbaryl) and the soil properties. Higher adsorption of carbaryl was obtained in presence of cationic surfactants followed by anionic and non-ionic surfactants. The FRf values obtained from soil TLC studies were inversely proportional to $K_F$, $K_F^*$ and $K_D$, $K_D^*$ values and confirmed the adsorption pattern of carbaryl in the absence and presence of surfactants. The values of $K_F^*/K_D$ ratios were used to evaluate the remediation efficiency of soils and it was found that non-ionic surfactant, TX-100 was preferable for remediation of high organic matter and low clay containing soils (F.R.I. silt loam and Alampur silt loam) at all CMC values, while anionic surfactant, SDS is preferable for remediation of all the four soils only at above CMC level (10 x CMC). From an environmental viewpoint, the findings from this study with the applied surfactant levels should facilitate evaluation of the potential impact of these and similar surfactants on the pesticide distribution behaviour in natural water and/or at waste-disposal sites.
REFERENCES


