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Adsorption of metribuzin in tomato growing soils

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Abstract

A study was conducted to characterize the sorption behavior of metribuzin herbicide in 10 tomato growing soils. The adsorption isotherms were mainly parabolic in nature with ‘S’ shaped character in all ten soil samples and adsorption data fitted well with Freundlich equation. The Freundlich K_f values for the soils ranged from 0.062 (S-5) to 1.369 (S-6) and the n values varied between 0.82(S-7) to 1.12 (S-5). The values of the K_{foc} of the ten soils varied from 20.68 (S-7) to 197.10 (S-5). The soil water distribution quotient K_d values of the ten soils varied between 0.11 and 0.96. The highest K_d value was recorded in S-5 and lowest K_d value in S-4. The K_{doc} values for metribuzin in soils varied from 33.65 to 140.31. The lowest K_{doc} was noticed in S-6 and highest K_{doc} in S-5. The ‘K_f’ and K_{doc} values were positively and significantly correlated with organic carbon, clay content and clay + OC. There was a significant positive relationship between the metribuzin K_f and the OC content.

Keywords: metribuzin, freundlich constants, adsorption etc.

Introduction

Metribuzin belongs to chemical family of triazinones and it is applied pre-emergence or early post-emergence in tomato, carrot, potato and sugarcane crops. This herbicide is very effective against annual grasses and several broad-leaved weeds. Metribuzin is the highest consumed selective herbicide used in vegetable growing farmers of Telangana state. Fate of the herbicide in environment and plant system depends not only on herbicide properties *per se* but on soil properties, climatic conditions and crop characters also. Adsorption is important because it affects the availability of the herbicide to the plants and its mobility in the soil. Fate of the herbicide in the soil influence the bio-availability, movement of the herbicide and its environment contamination potential and the adsorption of herbicide largely depends on the soil properties (clay content, organic carbon content, CEC, pH etc) (Khoury *et al.*, 2003; Singh 2006) [13, 26]. Herbicide adsorption also influences the volatilization, physical distribution, breakdown and biological activity and even its susceptibility to microbial organisms. Barriuso *et al.* (1992) [3] studied the influence of the nature of soil constituents, the physico-chemical factors and the chemical structure of pesticides on the adsorption behavior of pesticides in soil.
Freundlich isotherms were used by Ouzna *et al.* (2013) [18] to study the adsorption of metribuzin and their results showed that as per the shape of the isotherms adsorption was not linear and decreases at higher concentrations. Rigi *et al.* (2015) [23] indicated that the adsorption isotherms of metribuzin were well described by the Freundlich equation. Adsorption of metribuzin by the soils was highly correlated with the organic matter content and EGME specific surface area measurements. (Peter and Weber, 1985) [20].
Several studies have shown the potential mobility of metribuzin in soil system (Peter and Weber, 1985; Sharom and Stephenson, 1976) [20, 25]. Prolonged persistence in environment and high water solubility (1,200 ppm at 20°C) of metribuzin necessitates a thorough understanding of adsorption in soil. Data on sorption behavior of metribuzin in tomoto growing soils of telangana is scanty. Hence an experiment was conducted to study the adsorption of metribuzin in tomato growing soils of Rangareddy district.

Material and methods

Collection of soil samples

Soil samples (10) were collected from different mandals of tomato growing soils of Ranga Reddy district in Telangana state with varying physico-chemical properties. Details of the sampling locations and their physico-chemical characteristics of the samples are presented in Table 1. At each selected location, composite soil samples were collected at different spots from a depth of 0-15 cm, quartered and about 1.0 kg of each soil sample was brought to the

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laboratory, air dried under shade and processed by passing through a 2 mm sieve. These 2 mm sieved soils were properly labeled and stored in cloth bags for further studies.

Physico-chemical properties of soils

The physico-chemical properties of soils like pH, EC, organic carbon, CEC and texture of soils were studied by using different standard procedures.

Spectral characteristics of Metribuzin

Analytical grade metribuzin (99% purity) was obtained from Dr. Ehrenstrofer GmbH, Germany and used for present study without any further purification. Metribuzin standard solution of 50 ppm was prepared by dissolving 50.5 mg of metribuzin in methanol and used as stock solution. Aliquots from this stock solution were diluted to prepare desired concentrations.

Spectra of metribuzin solution (10 ug mL^{-1}) were scanned in UV range to determine the wave length of maximum absorbance on a UV-Vis spectrophotometer (UV 3000+ Model) using matched silica cuvettes. Metribuzin showed maximum absorbance in visible range at 455 nm wavelength. It was used as λ max for further studies and preparation of standard curve.

Adsorption

Adsorption studies were carried out in all the 10 soil samples collected from the farmer's fields. Five grams of soil was equilibrated with 20 mL of metribuzin solutions of various concentrations ranging from 0 to 50 µg $mL^{-1}$for 24 hours with intermittent shaking at constant temperature of 27^0C. $CaCl_2$ of 0.01M concentration was used to maintain the ionic strength. After 24 hours, the slurry was centrifuged at 4,000 rpm for 15 minutes. Identical soil blanks were maintained (without addition of herbicide). The absorbance for each treatment and corresponding blank values were measured at 455 nm. The difference between the treatment and blank was taken as actual equilibrium absorbance for which the concentration was calculated with reference to the calibration curve. Using the difference between initial and equilibrium concentration the amount of herbicide adsorbed per gram of soil was calculated.

Quantitative Treatment of Adsorption Data

The two commonly used mathematical models to explain isotherms are Freundlich and Langmuir isotherm equations.

Freundlich adsorption equation

The equation may be expressed as

$$x/m = K_f.\, C^{1/n} \qquad (1)$$

x/m = Amount adsorbed per unit mass of adsorbent
C = Equilibrium concentration
K_f = Freundlich constant related to the strength of binding
N = Constant which is less than 1

The constant K_f is related to strength of binding and depends on temperature.

K_f and n are determined from a logarithmic transformation of the equation, which is linear,

$$\text{Log } x/m = \text{Log } (K_f) + l/n.\ \log (C) \qquad (2)$$

The constant K_f and n were determined from the plot of log x/m vs. log C, the intercept and slope being log K and 1/n, respectively.

The empirical nature of Freundlich equation is shown by the fact that no limiting value of the surface concentration of the adsorbate is approached as the equilibrium solution concentrate of adsorbate is increased. This equation has no established theoretical basis and it works well for intermediate concentrations in most isotherm represents an initial slow increase in adsorption followed by a steep rising and ending with a slower increase and finally leveling off in adsorption.

To understand the role of organic carbon in adsorption of metribuzin in the soils K_{foc} for all the soil samples was computed using the following equation.

$$K_{foc} = \frac{K_f}{\text{\% Organic carbon}} \text{ X } 100 \qquad (3)$$

Hamaker and Thomson (1972) gave a relationship to describe the distribution of soil adsorbate between two phases, as simple partitioning between soil and water.

$$x/m = K_{d.}\ C \qquad (4)$$

Where,
x/m is the concentration of the adsorbate in µg g^{-1}
K_d is the soil – water distribution co-efficient.
C is Equilibrium Concentration µg mL^{-1}
The soil-water quotient was calculated as

$$Kd = \frac{\text{Amount adsorbed } (\mu g\ g^{-1})}{\text{Equilibrium Concentration } (\mu g\ mL^{-1})} \qquad (5)$$

The soil organic carbon- water quotient was calculated as

$$K_{doc} = \frac{K_d}{\text{\% Organic carbon}} \text{ X } 100 \qquad (6)$$

Results and discussion

Physico-chemical properties of the ten tomato growing soil samples collected from different locations in Ranga Reddy district are presented in the table 1.

The pH of soil samples varied from 6.48 to 8.98 *i.e.* slightly acidic to moderately alkaline in nature. Electrical conductivity varied from 0.10 to 0.34 dS m^{-1} indicating that all the soils were non-saline. Organic carbon content of most of the soils was low. The OC (%) content varied from 0.31 to 0.94 %. It was observed that, tomato is being grown in soils of wide ranging textures from sandy loams to clay. However, most of the soils were sandy clay loam in texture. Clay content in these soils varied from 15.8 % to 43.7%. Sand content was in the range of 26.1 % to 71.6%.

Wide variability of the soils in terms of the clay content, organic carbon content and pH (soil properties which show influence over sorption characteristics of herbicides) provided good opportunity for studying the sorption behavior of metribuzin, the most popular herbicide used in tomato crop in the district. These soil properties recorded in the present study are in good agreement with the range of properties reported in benchmark soils of Andhra Pradesh published by NBSS&LUP (Reddy *et al.,* 2005) [22].

Table 1 Physico –chemical properties of soil samples (S-1 to S-10)**:**

Sample No& Village Name		pH	EC (dS m^{-1})	CEC c. mol (p+) kg^{-1}	OC (Mg/m3)	Particle size distribution (%)			Texture
						Clay	Silt	Sand	
S-1	Malkapur	8.98	0.33	26.61	0.45	34.8	25.4	39.8	Clay loam
S-2	Nagariguda	8.84	0.27	31.3	0.94	43.7	30.2	26.1	Clay
S-3	Chellapur	8.46	0.23	32.1	0.72	42.3	25.6	32.1	Clay
S-4	Rangapur	6.52	0.13	13.91	0.32	15.8	13.5	70.7	Sandy loam
S-5	Pargi	8.14	0.18	24.62	0.69	35.7	29.5	34.8	Clay loam
S-6	Near Pargi	6.48	0.12	16.8	0.47	22.5	17.8	59.7	Sandy clay loam
S-7	Lingaipally	7.06	0.10	14.1	0.31	16.9	11.5	71.6	Sandy loam
S-8	Kotturu	8.48	0.26	21.4	0.49	27.4	17.8	54.8	Sandy clay loam
S-9	Taramathipet	8.61	0.34	16.74	0.36	31.3	24.2	44.5	Clay loam
S-10	Ravalkollu	8.27	0.26	15.28	0.42	20.1	15.4	64.5	Sandy clay loam

Adsorption of metribuzin on 2.0 mm sieved soils

Adsorption of metribuzin was carried out in all the ten soil samples with initial concentration ranging from 0 to 50 µg mL^{-1}. As explained in the materials and methods, slurry method of equilibrium concentration was adopted and metribuzin was measured spectrophotometrically, after preparing appropriate dilutions. The amount adsorbed was calculated as the difference between the initial concentration and equilibrium concentration after correcting for blanks.

The extent of adsorption on the soil samples at initial concentration of 5.0 µg mL^{-1} varied from 0.41 µg g^{-1} to 5.01 µg g^{-1}. While at initial concentration, of the adsorbed metribuzin was 5.01 µg g^{-1} to 35.29 µg g^{-1}. In general, the metribuzin adsorption was higher in soils with higher clay content and higher organic matter content. The adsorption was slower at the lower initial concentrations (<15 µg ml^{-1}) and increased rapidly at the intermediate concentrations and tended to slow down at higher concentrations of >40 µg mL^{-1}. These results are in good agreement with the findings of Khoury *et al.* (2003) [13] who found that a clay soil retains more metribuzin than a sandy loam soil. Results of Delle Site (2001) [5] and Singh (2006) [26] also indicated that organic carbon and clay content of the soil positively influence the most metribuzin adsorption.

Adsorption isotherms were plotted by taking adsorbed herbicide on soil on Y-axis against the equilibrium concentration on X- axis. The adsorption isotherms were mainly parabolic in nature with 'S' shaped character in all soil samples. In The adsorption data fitted well with Freundlich equation. The Freundlich parameter K_f, obtained as the intercept and 1/n the slope of the isotherm were calculated by plotting log values of the equilibrium concentration and amount of metribuzin adsorbed on the soils. Isotherms for all the soils samples are presented in the fig.1.

Adsorption was not linear (1/n <1) and the shape of the isotherms showed that adsorption decreased at higher concentrations which could be explained by a decrease in affinity of adsorption sites or competition with water molecules for the same adsorption sites (Ouzna *et al.* (2013) [18]. Adsorption isotherms of soils low in organic carbon content (in general less than 6 %) showed a tendency for 'S' shaped character, indicating a stronger initial competition of water molecules to the adsorbent as compared to the herbicide, thereby indicating the initial resistance to the adsorption of herbicides to be overcome later by the co-operative effect of the adsorbed molecule. These appear when the solid molecule is mono-functional, has moderate inter-molecular attraction and needs a strong competition for adsorbent site from the molecules of solvent or other adsorbed species. The relationship between the nature of adsorption isotherm and the organic carbon content of soils indicate that the soils low in organic carbon have a tendency to give 'S' shaped isotherms on account of their hydrophilic nature as compared to the soils high in organic carbon content which tend to be hydrophobic. Similar 'S' shaped isotherms for soils low in organic carbon content have been obtained for various herbicides by several workers. (Cox *et al,* 2000; Morrica *et al*, 2000; Nagamadhuri, 2003 ; Sireesha, 2008) [4, 16, 27].

The Freundlich constants K_f and 'n' for the tomato growing soils are presented in the table 2. The K_f values for the soils ranged from 0.062 (S-5) to 1.369 (S-6) and the n values varied between 0.82(S-7) to 1.12 (S-5). The K_f values reported in the present study are closely correlated to the K_f value proposed by the Footprint PPDB (2001) [6] for metribuzin (0.959), also referring to the range of values of K_f mentioned in the EU certification records that range from 0.018 to 1.9. Further, the K_f values reported by Johnson and Pepperman (1995) [10] and Lagath *et al*. (2011) are in the range of K_f coefficients for metribuzin obtained in the present study.

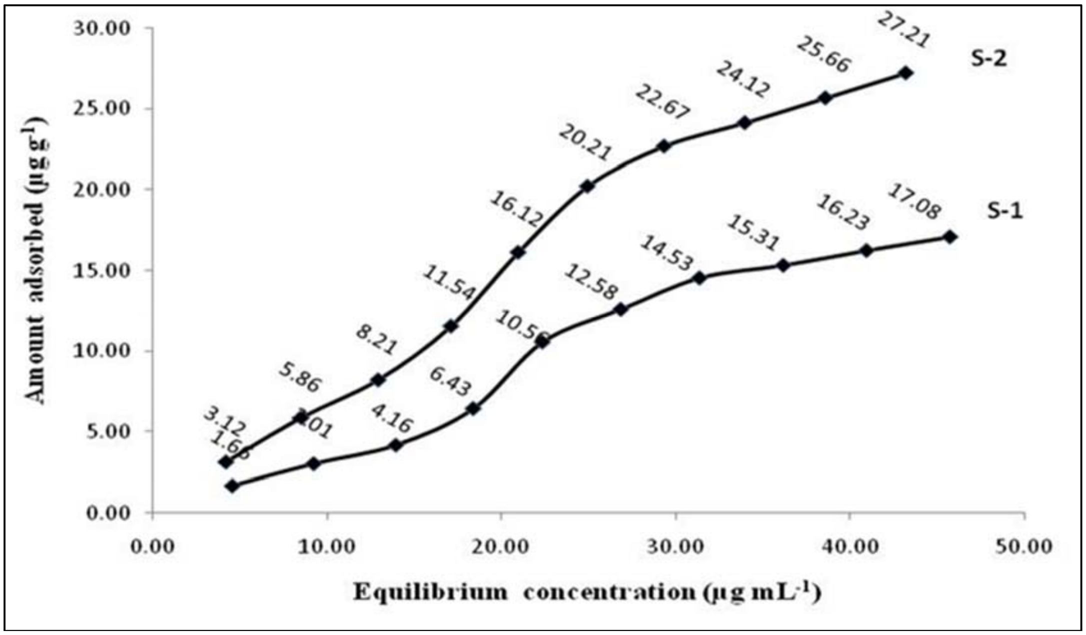
Freundlich equation has been used effectively to determine the adsorption of herbicides on soil, where the fraction of adsorbed herbicide is low (Pandey and Agnihotri, 2000) [19]. The co-efficient of determination (R^2 values) for the log x/m and log Ce plots were greater than 0.97, indicating the excellent fit of the adsorption data by Freundlich equation. The values of 1/n suggest the existence of non – linear adsorption. There could be a decrease in available sites as the adsorption increases. This is particularly true in soils with low organic matter and clay content. Another factor that affects the fraction of herbicide adsorbed is the type of adsorption sites. Organic molecules tend to adsorb to high energy sites first followed by the progressively weaker sites (Sonon and Schwab, 1995) [28].

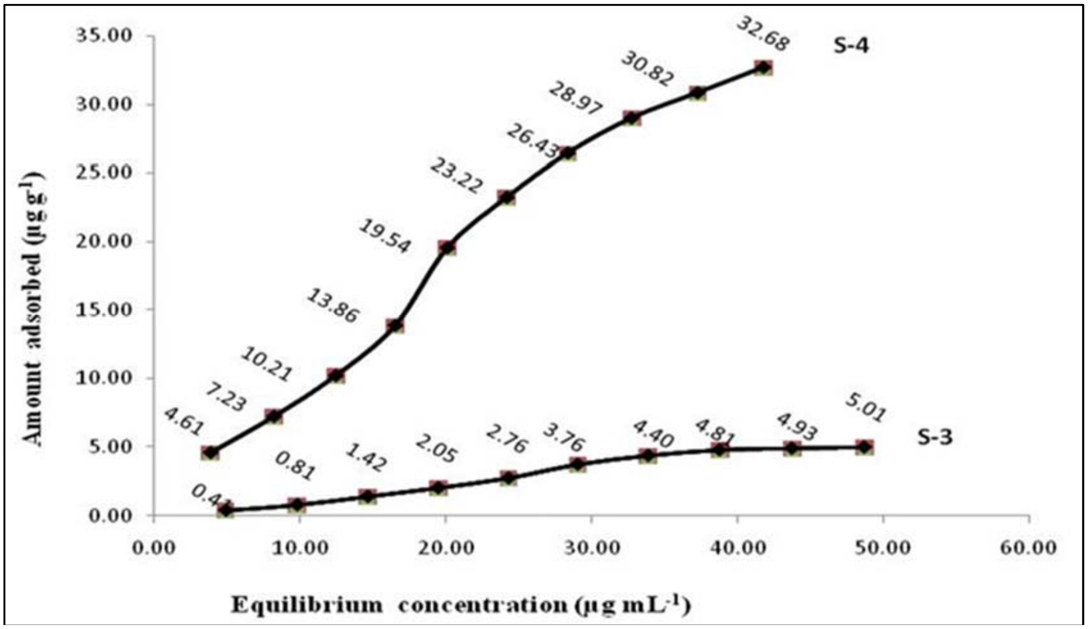
The values of the K_{foc} calculated from the K_f and organic carbon content are presented in the table 2. The values of the K_{foc} of the ten soils varied from 20.68 (S-7) to 197.10 (S-5). These K_{oc} values are closely related to values reported by Rigi *et al.* (2015) [23] where in Koc values for the metribuzin in eight soils varied from 33.68 to 291.93.

The soil water distribution quotient K_d values of the ten soils varied between 0.11 and 0.96. The values of the K_{doc} calculated from the K_d and organic carbon content are presented in the table 2. The highest K_d value was recorded in S-5 and lowest K_d value in S-4. The soil organic carbon-water distribution coefficient was found to be helpful in estimation of the leaching of metribuzin in soil and adsorption properties of the herbicide in soil. The K_{doc} values for metribuzin in soils varied from 33.65 to 140.31. The lowest K_{doc} was noticed in S-6 and highest K_{doc} in S-5. the highest and the lowest K_{doc} values were in close correspondence to the organic carbon content of the soils. These values are in the range of K_{oc} coefficients for metribuzin described by Johnson (2001) [11]. The K_{oc} values of metribuzin computed by Bakhsh *et al*. (2004) [2] and Kah and Brown (2007) [12] were 109 and 66 for different soils. These values were considered to be low, indicating a high risk of mobility of metribuzin. The K_{oc} values are in agreement with the Koc values between 9 and 120 reported in the literature cited above.

Table 2: Freundlich constants K_{f}, n, K_{foc}, K_d and K_{doc} values for adsorption on soil samples (S-1 to S-10)

Sample. No.	ƟC (Mg/m3)	K_f	n	K_{foc}	K_d	K_{doc}
S-1	0.45	0.279	0.90	62.0	0.40	90.40
S-2	0.94	0.729	1.01	77.34	0.70	74.64
S-3	0.72	1.230	1.11	170.83	0.87	121.62
S-4	0.32	0.062	0.85	19.37	0.11	35.48
S-5	0.69	1.369	1.12	197.10	0.96	140.31
S-6	0.47	0.099	0.87	21.06	0.15	33.65
S-7	0.31	0.061	0.82	20.68	0.13	43.12
S-8	0.49	0.149	0.90	30.49	0.22	44.25
S-9	0.36	0.218	0.86	60.53	0.38	105.36
S-10	0.42	0.0936	0.87	22.22	0.15	35.86





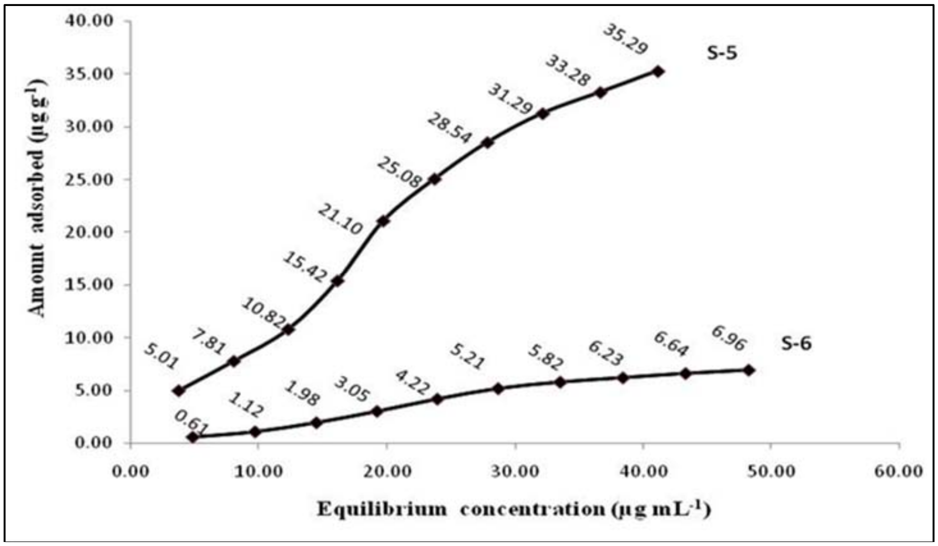


Fig 1 (a): Adsorption isotherms of metribuzin on 2 mm sieved soil samples (S-1 to S-10)

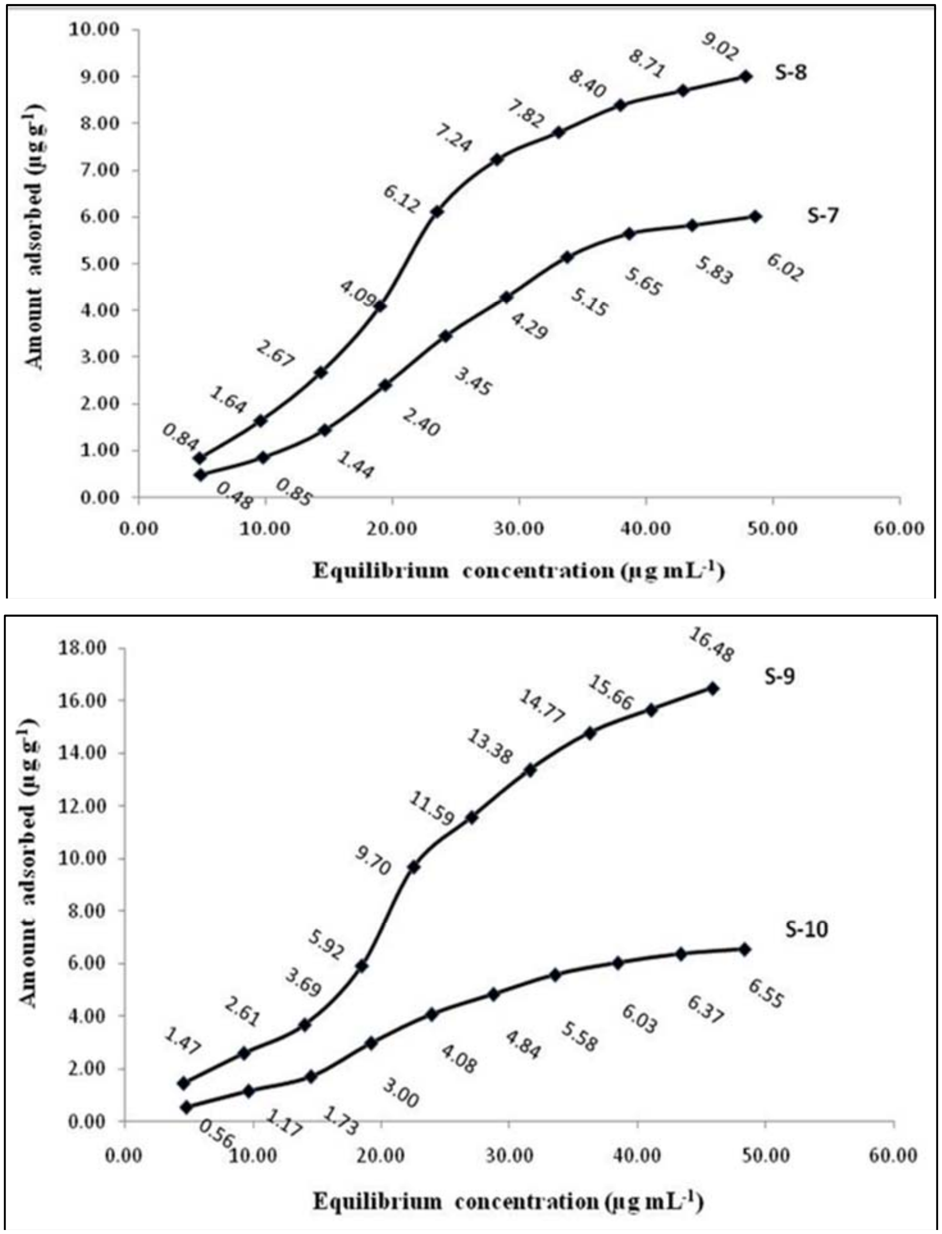


Fig 1 (b): Adsorption isotherms of metribuzin on 2 mm sieved soil samples (S-1 to S-10)

Correlation of adsorption constants with soil properties

Simple correlations were worked out between the extent of adsorption represented by Freundlich constant K_f with soil properties. The Freundlich 'K_f' values which indicate the extent of binding of herbicide to the soil constituents were positively and significantly correlated with organic carbon (r = 0.802** P<0.01), clay content (r = 0.737** P<0.01) and clay + OC (r = 0.741** P<0.01). There was a significant positive relationship between the metribuzin K_f and the OC content (r= 0.809, P <0.01) (Rigi *et al*, 2015) [23]. Soil organic carbon has a main role in adsorption of pesticides (Arias-Estévez *et al*., 2008) [1]. Similar results were reported by Graham and Conn (1992) [8], Ma *et al.* (1993) [15]. Organic carbon content has been shown to be the first critical parameter positively and significantly correlated with adsorption of metribuzin in soils (Sharom and Stephenson, 1976) [25].

Clay content of soil is said to be another critical parameter in adsorption of metribuzin in soils often masked by that of organic matter and this can become a significant factor when organic carbon content decreases (Savage, 1977; Kah and Brown, 2007) [12, 24]. Khoury *et al.* (2003) [13] who found that a clay soil retained more metribuzin than a sandy loam soil.

Clay minerals have high reactive surfaces mainly due to the negative sites present on them. Binding of organic compounds to these sites takes place through metallic cations, which directly react with these organic compounds and bridged to them through water molecules. The existence of such mechanism has been demonstrated by many workers in a large number of simple and complex organic compounds. (Raman and Rao, 1984; Gowri Priya *et al.*, 2006) [21, 7]. Sireesha (2008) [27] brought out the importance of clay-organo complexes in the tropical and sub-tropical soils, where organic matter content is often less than 2 per cent, clays have been found to be the predominant factor in the adsorption of soil applied herbicides. Hamaker and Goring (1976) [9] reported that the soil can be considered to be a unique complex adsorbing surface incorporating new features like clay organic interactions. Raman and Rao (1984) [21] demonstrated that the adsorption of herbicides on model clay–organo complexes was much higher than the corresponding homoionic clays suggesting that the naturally occurring clay organo complexes in soils play a very significant role in the adsorption of soil applied herbicides.

Conclusion

Metribuzin adsorption was lesser at the lower initial concentrations (<15 µg ml^{-1}) and increased rapidly at the intermediate concentrations and tended to slow-down at higher concentrations (>40 µg mL^{-1}). The K_f values for the soils varied from 0.062 (S-5) to 1.369 (S-6) and the n values varied between 0.82(S-7) to 1.12 (S-5). The values of the K_{foc} of the ten soils were in the range of 20.68 (S-7) to 197.10 (S-5). Higher metribuzin adsorption and larger Freundlich K values was observed in soils with higher clay content and organic carbon content. Freundlich K values were significantly correlated with organic carbon per cent Clay percent, clay + organic carbon percent.

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