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Adsorption and Mobility of Metribuzin Herbicide as Influenced by Certain Soil Properties

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TWO LABORATORY experiments were conducted to study the adsorption and mobility of the herbicide metribuzin, [4-amino-6-tert-butyl -3- methylthio -1,2,4- triazin -5 (4H) -one] herbicide as influenced by certain soil properties. Twenty seven artificial soils having different levels of clay, organic matter and calcium carbonate were used in order to study the effect of such parameters on the behaviour of herbicide metribuzin Eight different soils were selected for metribuzin adsorption with five concentrations , *i.e.*, 0, 20, 40, 60 and 100 µg/ml using Freundlich equation. Moreover, all prepared soils were used for studying the downward movement of metribuzin through soil columns technique.

The main findings show that the amount of metribuzin adsorbed in soils tended to increase, mainly with increasing the percentages of clay, followed by organic matter and calcium carbonate content. It was indicated that the adsorption of metribuzin was lower in sandy soil (x/m = 1.24-8.0 $\mu g.mg^{-1}$), while it was higher in clay rich soil (x/m = 2.2-12.2 $\mu g.mg^{-1}$).

On the other hand, amounts of metribuzin remained in soil column tended to increase in upper soil layers in the columns with increasing clay %, which estimated by 19. 26 and 35% of the amount of herbicide found in the soil column in case of 20 and 40% clay content as compared with zero level of clay, respectively. The mean R^2 values reveal that the contribution of clay on metribuzin movement through soil is about 44.6 %, while the organic matter and $CaCO_3$ contribution were only 1.2 and 0.01 %, respectively. Linear regression show that the amount of metribuzin moved to the lower section of soil was negatively correlated only with clay giving a contribution of 44.6% (r = - 0.6338).

The regression of the amounts of metribuzin recovered on the studied parameters, *i.e.*, clay, O.M. and $CaCO_3$ content using all studied soil samples resulted the following equation:

$$Y= a + b_1X_1 + b_2X_2 + b_3X_3$$

Where: Y = the amount recovered of metribuzin, µg, a (intercept) = 11.55, X 1, X2 and X3= clay %, O.M. % and $CaCO_3$ %, respectively.b1, b2 and b3 (regression coefficient)= 0.082**, -0.072 and 0.028 , respectively.

So, the results of adsorption and mobility of metribuzin are very useful to illustrate the differences in the activity and rates of metribuzin which are required to control target weeds from one soil to another depending on the differences in soil properties, especially clay content.

Keywords: Adsorption, Movement, Metribuzin, Clay, Organic matter, Calcium carbonate.

Adsorption appears to control herbicides movement, persistence, degradation, bioactivity and contamination of underground water supplies. Much of herbicides are adsorbed by soil surface and this can modify their activity. Some herbicides may be too tightly adsorbed to give proper weed control.

Metribuzin, [4-amino-6-tert-butyl-3-methylthio-1, 2, 4-triazin-5(4H)-one] is an effective herbicide against annual weeds in various fields and horticulture crops such as potatoes, tomatoes, wheat and sugarcane. Its weak soil sorption and high soil mobility make it an excellent example for studying herbicide movement within soil (Harper, 1988). Soil properties such as clay, organic matter, calcium carbonate and CEC play an important role in the adsorption and mobility of metribuzin. The effects of such soil parameters were subjected to study by many investigators. Daniel *et al.* (2002) indicated that only organic matter content was related positively to Kf coefficient. Meanwhile, Coultas & Harvey (1979) found that leaching of metribuzin was most highly correlated with soil organic matter, bulk density and field moisture capacity.

On the other hand, Sondhia & Yarduraju (2005); El Sawi (2000) and Khoury *et al.* (2003) found that metribuzin moved rapidly in sandy soil followed by clay loam and clay soils and vice versa with metribuzin adsorption. They found that metribuzin sorption was correlated with clay and organic matter content. In addition, Harper (1988) found that metribuzin was weakly adsorbed in different textured soils and sorbed significantly with relatively higher content of clay. Seeling (1994) mentioned that organic matter content of <2% in A horizon will have low potential to filter pesticide. He added that metribuzin had high leaching potential. Ladlie *et al.* (1976) reported that the high water solubility and weak sorption of metribuzin lead to high mobility. This added that soil properties can be used to predict the required rate of metribuzin.

In Egypt, metribuzin is registered for weed control for many crops. The efficiency of this herbicide varied greatly from one soil to another. For this reason the present work was designated to study the effect of certain soil properties namely clay, organic matter and calcium carbonate content on adsorption and mobility of the metribuzin herbicide.

Materials and Methods

Artificial soil preparation

Sand particles having 1 to 2mm diameter were first washed with 1:3 HCl: H_2O, tap water then distilled water. The washed sand received the following additions: Clay size fraction previously separated from an alluvial clayey soil, at the levels of 0, 20, and 40%; separation procedure is outlined by Shuman (1976) and El Sokkary (1980). (ii. Calcium carbonate at rates of 0, 3, and 18% and (iii. Organic matter (composted plant residues) with the rates of 0, 1, and 2 %.

Sand, clay, calcium carbonate and organic matter were weighed and mixed together according to the previously mentioned rates making 27 artificial soils.

Adsorption of metribuzin by soils

Eight different artificial soils were selected for characterizing adsorption of metribuzin using Freundlich equation according to Harper (1988), as follows: (sand), (sand + 2% O.M.), (sand + 18 % $CaCO_3$), (sand + 2% O.M. + 18% $CaCO_3$), (clay), (clay + 2% O.M.), (clay + 18% $CaCO_3$) and (clay + 2% O.M. + 18% $CaCO_3$). Soil samples were sieved (2-mm opening) and allowed to air dry.

Five gm soil and 25 ml of metribuzin in 0.01 M $CaCl_2$ were placed in a plactic centrifuge tube. The concentrations used were 0, 20, 40, 60, and 100 µg/ml dissolved in 0.01 M $CaCl_2$ solution. Calcium chloride solutions were used in adsorption measurements in order to minimize ionic strength changes. Each concentration was run in three replicates. The soil slurry was equilibrated for 24 hr on the shaker at room temperature (25 C°) to ensure that equilibrium had been reached. Preliminary adsorption time studies showed that most of the metribuzin reached equilibrium quickly, within one hr, without significant degradation over 48 hr. At the end of equilibration time the tubes were centrifuged at 1900 xg for 30 min, then 18 ml aliquots of the supernatant were taken into a 125 ml separatory funnel and partitioned with methylene chloride three times 50, 30 and 20 ml. The combined mehtylene chloride phases were dried by filtration through a pad of cotton and anhydrous sodium sulphate. Then, it was evaporated just to dryness using a rotary evaporator.

The residues of metribuzin were analyzed and determind using GLC and the adsorbed amount was calculated from the difference between concentration in solution before and after contact with the soil. Adsorption isotherms were prepared by plotting the logarithm of the adsorbed amounts of metribuzin versus the logarithm of the concentrations of solution after contact with the soil. Then, adsorption isotherms were determined by using the Freundlich adsorption isotherm equation:

$$x/m = KC^{1/n}$$

which could be turned to straight line equation as follows:

$$\log x/m = \log K + 1/n \log C,$$

Where: x/m is the µg of pesticide adsorbed per g of soil. C = equilibrium concentration of the adsorbate in solution, µg/ml. 1/n = the value of the adsorption isotherm, K and 1/n are constants and may be determined by the best line drawn through the plot of log x/m versus log C.

Mobility of metribuzin

Mobility of metribuzin was studied through soil columns, (30 cm height and 3.5 inner diameter) glass columns. The columns were supported by -9- cm Buchner funnels fitted with whatman No. 1 filter paper. The columns were packed with the twenty seven prepared artificial soils to depth of 20 cm and were divided into four layers by filter papers. Amount of metribuzin, 62.8 μg were added to each column equal to 210 g/fed and mixed thoroughly in the upper 1 cm layer of the soil in the column. Soils in the columns were watered to field capacity. After that they were carefully segmented, extracted and subjected for estimation of metribuzin in these segments. Each soil column was replicated three times.

A recovery test was carried out for certain studied soils according to the method of Polese *et al.*, (2002) and resulted 90 to 96 % metribuzin recovery . According to Jarezyk (1983) the residue (R), expressed in mg/kg, is calculated by applying the following equation:

$$R = \frac{F_A \, . \, V_{End} \, . W_{St} \, . \, F}{F_{St} \, . \, V_i \, . \, G}$$

Where:

F_A= peak area of sample, mm^2, V_{End} = terminal volume of ethyl acetate solution, ml.
W_{St}= amount of parent compound injected with standard solution, mg.
F= recovery factor determined by analyst, F_{St}= peak area of standard, mm^2, V_i= portion of terminal volume injected into gas chromatograph, μl and G= sample weight.

The herbicide residues were dissolved into 2 ml ethyl acetate and analyzed using gas liquid chromatography (GLC). HP HEWLETT PACKARD 5890 SERIES II equipped with computer.

Results and Discussion

Adsorption of metribuzin as affected by soil properties

Effects of soil clay, organic matter and calcium carbonate contents on adsorption of metribuzin are shown in Table 1.

Sorption of herbicide was compared by means of the Freundlich equation since herbicide sorption isotherms are typically non-linear. Data show that the amounts of sorbed metribuzin markedly increased with increasing clay content in soils by about 91.6%, while organic matter and calcium carbonate contents show relatively slight increase of 14.9 and 3.6%, respectively. Presence of 18% calcium carbonate seems to be inactive for metribuzin adsorption. Similar findings were also found by Sheta (1982) for sorption of the herbicide paraquat by soils containing different amounts of $CaCO_3$. However, previous workers have found a positive relationship between soil organic matter and metribuzin adsorption in soil (Bouchard *et al.*, 1982 and Savage, 1976).

TABLE 1. Values of equilibrium metribuzin concentration, C($\mu g.ml^{-1}$) and metribuzin adsorbed by soil, x/m ($\mu g.mg^{-1}$) as affected by clay, O.M. and $CaCO_3$content in the studied artificial soils .

Clay,	O.M,	$CaCO_3$,	C, $\mu g.ml^{-1}$				x/m, $\mu g.mg^{-1}$			
	%		1	2	3	4	1	2	3	4
0	0	0	0.55	1.30	1.90	3.20	1.24	1.40	2.44	4.00
	2	0	0.50	1.30	1.70	2.80	1.60	1.28	3.38	6.00
	0	18	0.40	1.30	1.60	2.70	1.80	1.12	4.00	6.40
	2	18	0.50	1.16	1.80	2.40	1.64	2.20	3.20	8.00
40	0	0	0.40	1.00	1.10	1.70	2.20	2.96	6.80	11.6
	2	0	0.30	0.80	0.90	1.60	2.60	3.80	7.60	12.2
	0	18	0.30	0.50	1.40	2.40	2.60	5.54	5.20	8.00
	2	18	0.50	0.70	0.80	2.30	2.60	4.48	8.00	9.00

On the other hand, amounts of sorbed metribuzin obviously increased with increasing the amount of the herbicide equilibrated with the tested soils regardless of their content from clay, O.M. or $CaCO_3$. However, the highest adsorption rate was observed for the rich clay soil (Fig. 1).

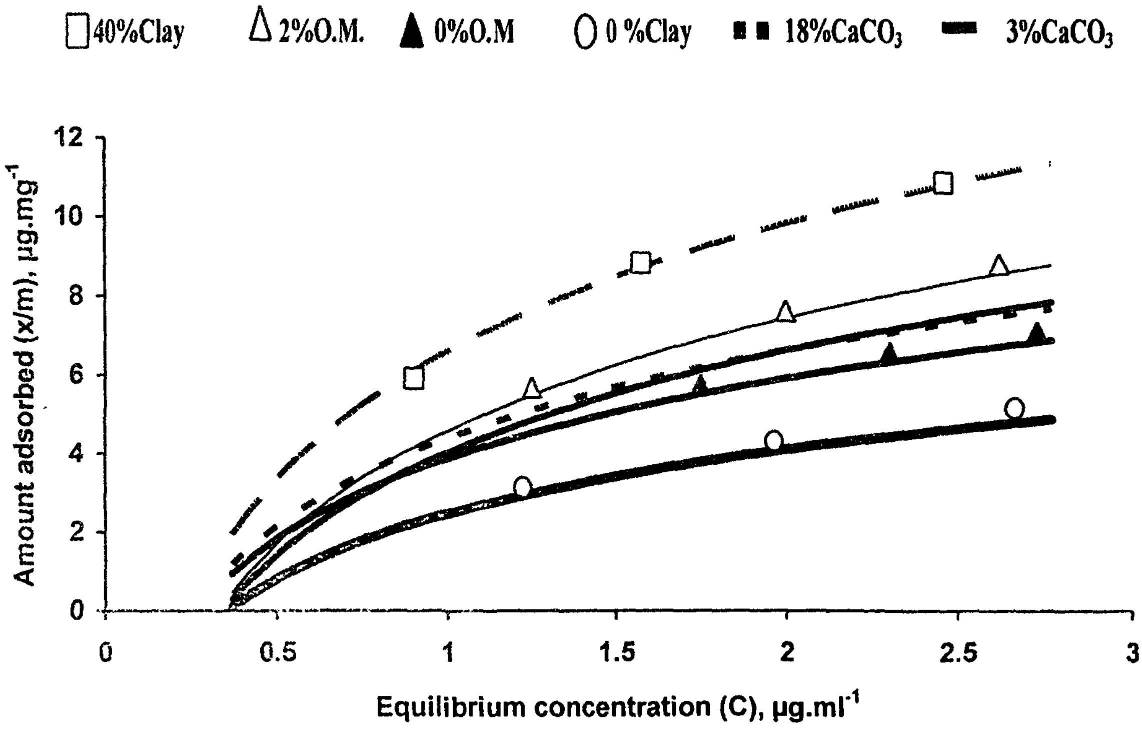

Fig.1. Adsorption rate for metribuzin in different soils.

Freundlich coefficients of metribuzin sorption shown in Table 2, *i.e.*, K and 1/n constants belonging to the tested soils were calculated from logarithmic form of the adsorption equation. It is clear that marked differences were found between the clay rich soil and the clay free soil for both intercept and slope of adsorption line. While those soils containing different levels of both O.M. and $CaCO_3$ were relatively lower. Freundlich coefficient (K) ranged from 0.183 to 0.587 and decreased with increasing clay, O.M. or $CaCO_3$ content in the tested soils. Slope values (1/n constants) that were determined from linear form of Freundlich equation ranged from 0.776 to 0.990. Slope values of the clay rich soil were lower than that of the clay free soil. Similarly, slope values of the soils rich in O.M. or $CaCO_3$ were lower than that of the free O.M. and calcium carbonate contents. A similar trend was also found by Osgerby (1970) who stated that low 1/n constant was correlated with high organic matter content.

TABLE 2. Mean values of metribuzin adsorbed by soil, μg/mg soil and Freundlich coefficient as affected by clay, organic matter and $CaCO_3$ content.

	Clay,%		Organic matter,%		$CaCO_3$,%	
Parameter level	0	40	0	2	0	18
Metribuzin adsorbed	3.10	5.94	4.21	4.84	4.45	4.61
Intercept (K)	0.58	0.18	0.40	0.20	0.43	0.34
Slope (1/n)	0.95	0.77	0.92	0.81	0.99	0.85

The results of the present study show that clay content is strongly involved in adsorption of metribuzin followed by organic matter. The calcium carbonate content showed the lowest effect in herbicide adsorption by the tested soils.

Therefore, clay content should be considered when metribuzin rate is applied in farming practice. On the other hand, adsorption of metribuzin by soil clay or even with organic matter can prevent or minimize its downward movement through soil profile and accumulating in ground water.

Downward movement of metribuzin herbicide as affected by certain soil properties

The main advantages to use artificial soils is to study the effect of clay, organic matter and calcium carbonate on herbicide metribuzin movement without various complexes and side interactions that may interfere with the studied parameters as under natural soil conditions. Average values of metribuzin content in the different layers of the tested soils under the interaction effects of clay, organic matter and calcium carbonate contents are shown in Table 3.

TABLE 3. Average values of the effect of clay, organic matter and calcium carbonate content on the downward movement of metribuzin herbicide in soil columns.

Column depth, cm	Clay, %			Organic matter, %			Calcium carbonate, %		
	0	20	40	0	1	2	0	3	18
	Metribuzin remained, µg								
0-5	8.10	15.8	20.1	14.9	13.7	15.4	15.4	17.5	16.4
5-10	7.20	26.5	14.7	15.2	18.2	16.7	11.2	16.4	16.0
10-15	8.50	8.60	15.1	13.2	9.70	9.20	11.4	9.90	8.20
15-20	19.1	9.10	6.42	9.12	13.1	12.4	15.6	8.30	10.8
Average	10.7	15.0	14.1	13.1	13.7	13.4	13.4	13.0	12.8

It is clear that the prepared soil without clay addition (sand) contained the lowest amounts from the remained herbicide, with the highest amounts of metribuzin were then, leached out from soil column. However, metribuzin detected in the upper layer (0-5 cm) represented about 19, 26 and 35 % of the total amount of the herbicide found in the soil column in case of 0, 20 and 40% clay content, respectively. This indicates the faster movement of metribuzin in the sandy soil compared to the other two soils. In the contrarily, the amounts of metribuzin reached the lower layer (15-20 cm) represented 45, 15 and 11% of the total amount of the detected herbicide. Fleming *et al* (1992) found that only 7% of the applied metribuzin remained in the surface and 44% leached below 5 cm and in leachate. On an average, the total amounts of metribuzin found in the soils of 0, 20 and 40 % represented 68, 96 and 90 % of the added dose (62.8 µg), respectively.

Thus, it could be concluded that the high existence of metribuzin in the (0-15cm) layers of heavy or medium textured soils increases herbicide activity for weed control as compared with sandy soil which losses considerable amounts of metribuzin. The obtained results are in a good agreement with that of El-Sawi (2000) who found that in sandy soil metribuzin moved rapidly through the column, meanwhile in clay loam soil it persist in the top 10 cm after 7 days. Also, Khoury *et al.* (2003) and Sondhia & Yaduraju (2005) reported the high solubility and mobility of metribuzin, particularly in the coarse textured soils.

Data in the same table show that the relative increase in the upper 10 cm layer recorded about 6% due to the presence of organic matter comparing with soil without organic matter. So, the levels of organic matter occurred in such soils

(1 and 2%) did not show strong effect on metribuzin movement or leach abilities. Similarly, Thomson (1982) and Seeling (1994) mentioned that the levels of organic matter in soil below 2% can not prevent metribuzin to be filtrate in the upper layer of soils.

On the other hand , 3 and 18 % calcium carbonate can partially hinder metribuzin movement in various layers of soils. The detected amounts of metribuzin were 26.6, 33.9 and 32.4 µg in 0-10 cm layers of soils having 0, 3 and 18% calcium carbonate, respectively. The relative increase in herbicide, in the upper 10 cm, due to presence of 3 and 18% calcium carbonate were 27.4 and 21.8% compared to soil without calcium carbonate, respectively. The highest amount of metribuzin was detected in the lower layer (15-20 cm) of the soil without calcium carbonate indicating the higher downward movement of herbicide in such soil.

The present findings indicate that there are marked differences in the herbicide remaining amounts in the upper layers (0-10 cm) due to differences in $CaCO_3$ content, with an opposite trend in the lower layers (10-20 cm). However, the general averages showed slight differences.

Apparently, significant positive correlations were found for clay content in 0-5, 5-10 and 10-15 cm of the total soil column sections (Table 4). Results show positive correlation between clay or $CaCO_3$ contents in upper layers 0-5 and 5-10 cm of soil columns versus the amount of recovered metribuzin. The positive significant correlations for clay versus the amount of recovered metribuzin may be explained by the high amount of metribuzin adsorbed on clay surfaces and this was in agreement with the findings of Witt & Thomas (1985); Peter & Weber (1985) and Harper (1988). Contrarily the negative correlation of clay content and the amount of recovered metribuzin is attributed to the low amount adsorbed in this layer (15-20 cm).

TABLE 4.Values of simple correlation coefficients of metribuzin amounts recovered in different soil column depth, cm as affected by some soil properties.

Soil properties	Soil column depth, cm				All soil depths
	0 – 5	5 -10	10- 15	15 – 20	
Clay	0.568**	0.270*	0.463**	-0.633**	0.149**
O.M.	0.022	- 0.010	-0.285**	0.165**	-0.007
$CaCO_3$	- 0.191	0.313**	-0.005	-0.118	0.024

* and **: Significant at 5% and 1% level, respectively.

On the average, the contribution of clay on metribuzin movement through soil is about 44.6%, while the organic matter and $CaCO_3$ contributions are only 1.2% and 0.01%, respectively (Table 5).

Stepwise multiple linear regression had been calculated to represent the effect of each factor in the presence of the other factors and the order of magnitude effect of the studied soil properties. Values of the regression coefficients, intercepts and the multiple coefficient of determination (R^2) for the different depths of soil column are given in Table 5.

TABLE 5. Contribution of studied factors on metribuzin movement in different sections of soil columns.

Soil Properties	Soil column depth, cm								All soil depths	
	0 – 5		5 -10		10- 15		15 – 20			
	R	R^2	R	R^2	R	R^2	R	R^2	R	R^2
Clay	0.57**	32.2%	0.27*	7.3%	0.46**	21.4%	0.63**	40%	0.67**	44.6%
O.M.	0.02	0.05%	-0.01	0.01%	-0.29**	8.1%	0.17	2.7%	-0.11	1.2%
$CaCO_3$	-0.19	3.6%	0.31**	9.8%	-0.01	0.01%	-0.12	1.4%	0.01	0.01%

* and **: Significant at 5% and 1% level, respectively.

The regression coefficient (b) indicated the extent and direction of the influence of soil parameters on the amount of metribuzin recovered in various column sections of the studied soils. The effect of clay contribute positive values in 0-5, 5-10, 10-15 cm which were 0.295, 0.185 and 0.166 and negative with 15-20cm. Values of the (R^2) indicated that the contribution of the three studied soil properties, clay, organic matter and $CaCO_3$ content in metribuzin movement in the upper layer (0-5 cm) were estimated by 36%, 17.1% in 5-10 cm, 29.6% in 10-15 cm and 44.2 % in 15-20 cm layer (Table 6) .

TABLE 6. Multiple linear models for the mobility of metribuzin as affected by some soil properties.

Independent Variables	Soil column depth, cm				All soil depths
	0 – 5	5 -10	10- 15	15 – 20	
Regression coefficients:					
Clay	0.295**	0.185**	0.166**	-0.319**	0.082**
O.M.	0.229	-0.136	-2.041**	1.659*	-0.072
$CaCO_3$	-0.205*	0.443**	-3.924	-0.123	0.028
Intercept:	10.062**	9.487**	9.513**	17.124**	11.55**
Multiple coefficient of determination (R^2) :	0.360	0.171	0.296	0.442	0.023

* and **: Significant at 5% and 1% level, respectively.

Accordingly, we can use the equation derived from the regression of the amount of metribuzin recovered on clay, organic matter and calcium carbonate using all studied soil samples in the order of magnitude effect as follows:

$$Y = a + b_1X_1 + b_2X_2 + b_3X_3$$

Where, Y = the amount recovered of metribuzin (µg/g), a (intercept) = 11.55, X_1, X_2 and X_3 = clay %, organic matter % and $CaCO_3$ % respectively. b_1, b_2 and b_3 (regression coefficients) = 0.0827, [illegible] and [illegible] respectively. Clay, organic matter and $CaCO_3$ resulted (R^2) values of 44 [illegible], [illegible] and 6.01% respectively

The results of adsorption and mobility of metribuzin are very useful to illustrate the differences in the activity and rates of metribuzin which required to control target weeds from one soil to another depending on the differences in soil properties, especially clay content.

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دراسة ادمصاص وحركة مبيد الميتروبزين ومدي تأثرها ببعض خواص التربة

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قسم الأراضى كلية الزراعة – جامعة عين شمس و [**]المعمل المركزى لبحوث الحشائش – مركز البحوث الزراعية – القاهرة – مصر .

أجريت تجربتان بالمعمل بهدف دراسة ادمصاص وحركة مبيد الميتروبزين ٤-أمينو-٦ ترت- بيوتايل-٣-ميثيل- ثيوميثيل- ترايازين-٥-(٤ أتش)- ١ ومدي تأثرها ببعض خواص التربة . تم تكوين ٢٧ تربة مخلقة صناعيا استخدمت فيها مستويات مختلفة من الطين والمادة العضوية وكربونات الكالسيوم بغرض دراسة تأثيرها على سلوك مبيد الميتروبزين وتم اختيار ثماني أراض مختلفة لدراسة إدمصاص الميتروبزيـــن مع خمس تركيزات من المبيـــد وهـــي صفر، ٢٠، ٤٠، ٦٠، ١٠٠ (µg/ml) باستخدام معادلة فريندليش. وفى التجربة الثانية استخدمت جميع الأراضى المخلقة صناعيا من أجل دراسة حركة مبيد الميتروبزين باستخدام طريقة أعمدة التربة.

أوضحت النتائج أن كميات الميتروبزين المدمصة على أسطح التربة اتجهت إلى الارتفاع بزيادة محتوي الطين يليه المادة العضويه ثم كربونات الكالسيوم. وقد وجـد أن ادمصاص الميتروبزين كان منخفضا فـى التربة الرملية (١,٢ - ٨ µg/mg) بينما كان أكثر إرتفاعا في التربة ذات المحتوي العالي من الطين (٢,٢-٢,٢-١٢,٢ µg/mg). ومن جهة أخري أوضحت النتائج أن الكمية الموجودة من المبيد في الجزء العلوي من أعمدة التربة اتجهت إلي الزيادة بزيادة محتوى الطين والتي قدرت بـ ١٩ و ٢٦ و ٣٥٪ من كمية المبيد الموجودة في أعمدة التربة في حالة التركيزات ٢٠و ٤٠٪ مقارنة بالتربة الخالية تماما من الطين علي التوالي. وقد أوضحت قيم معامل الأرتباط (R^2) مقدار مساهمة الطين في حركة الميتروبزين بالتربة والتى تقدر بحوالي ٤٤,٦٪ ، في حين كانت مساهمة المادة العضوية وكربونات الكالسيوم تقدر فقط بحوالي ١,٢ و ٠,٠١٪ علي التوالي.

كما أشارت دراسات الارتباط أن كمية الميتروبزين في الطبقات السفلى من الأعمدة كانت مرتبطة عكسيا مع كمية الطين حيث وصل مقدار تأثير الطين الى ٤٤,٦٪ (معامل الارتباط = - ٠,٦٣٣٨). وتم حساب معامل الأنحدارلكمية مبيد الميتروبزين المستخلصة من الطين والمادة العضوية وكربونات الكالسيوم باستخدام المعادلة التالية:

$$Y= a + b_1X_1 + b_2X_2 + b_3X_3$$

حيث Y الكمية المستخلصة من الميتروبزين µg ، a = 11.55 و X_1 , X_2 , X_3 = النسبة المئوية للطين والمادة العضوية وكربونات الكالسيوم على التوالي

b_1 ، b_2 ، b_3 = معامل الانحدار.

هذا وتعتبر نتائج الإدمصاص وحركة مبيد الميتروبزين مفيدة جدا في توضيح مدى نشاط المبيد ومعدلات استخدامه المطلوبة لمكافحة الحشائش المستهدفة من تربة إلي آخرى تبعا لاختلاف خواصها وبخاصة محتواها من الطين .