EFFICIENCY OF ACETOCHLOR AGAINST ANNUAL WEEDS IN MAIZE(*Zea mays* L.) AND IT'S PERSISTENCE IN THE SOIL.

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Abstract: A field experiment was carried out in the experimental farm of Sids Agricultural Research Station, Beni-Suef Governorate, Egypt. to study the efficacy of pre- emergence application of acetochlor (Harness 84% EC) applied to the soil surface at 0.5L./fed + one hoeing and 1 L./fed. for controlling of seven annual weeds *Portulaca oleracea* L., *Corchorus olitorius* L., *Amaranthus hybridus* L., *Euphorbia helioscopia* L., *Xanthium pungens* wallr., *Brachiaria eruciformis* L., and *Echinochloa colonum* L.in maize during two seasons (2013 and 2014). A considerable reduction was observed in fresh weight of *Corchorus olitorius* L., *Amaranthus hybridus* L., *Xanthium pungens* wallr. and *Echinochloa colonum* L. with no fresh weight (0.0 g/m^2) after acetochlor application of 0.5 L./fed. + one hand hoeing. The second order of fresh weight of other weeds treated with the same treatment was *Portulaca oleraceea* L., *Brachiaria eruciformis* L. and *Euphorbia helioscopia* L. with values 23.3, 46.7 and 81.3 g/m^2 in season 2013 and 53.3, 77.3 and 98.0 g/m^2 in season 2014. Acetochlor at 0.5 L./fed. plus one hoeing significantly increased plant height, 100 grain weight and grain yield of maize in the two seasons. The highest chlorophyll a, b and carotenoids of maize was obtained from acetochlor at 1.0L./fed., as compared with the other treatments.

The highest total NPK of straw was obtained at hand weeding treatment with values 2.33 and 2.18 in the two successive seasons. Acetochlor alone gave less effect on N of grains than acetochlor plus hoeing. There was no difference between acetochlor applied alone or when combined with hand hoeing on P and K concentration of grains. The recommended dose of acetochlor 1.0L./fed was more effective on protein and oil content in maize grains than half dose + hoeing. It gave 9.25 and 9.0% of protein, 3.48 and 3.94% of oil in both seasons, respectively.

Determination the persistence of acetochlor under real field condition on maize crop was conducted using HPLC analysis. Since the herbicide was applied to the soil surface, it dissipation will vary depending on the concentration, soil type, pH, organic matter and environmental conditions. Extraction of field soil samples taken from different depths (15 and 30 cm) at different times after herbicide application showed that all applied doses moved deeper.
The statistically half-life times (RL_{50}) for acetochlor were 10.11 and 12.4 days at half and recommended dose, respectively.

Key words: Weed control, Acetochlor, Residues, Maize, persistence

1.Introduction

Weeds compete with crop plants for nutrients, light, space, moisture and many other growth factors through competition and allelopathy, resulting in direct loss to quantity and quality of the production **(Gupta, 2004).** A part from increasing the production cost, they also intensify the disease and insect pest problem by serving as alternative hosts **(Marwat *et al.*, 2008).** Weeds competition with maize could be either of broadleaf or grasses.

Portulaca pleracea L. and *Xanthium pungens* wallr are annual summer weeds, which grow in maize fields. Maize is one of the most important cereal crops in Egypt, whereas it is a multipurpose crop e.g. used as human food, animals and poultry feed, also it produces row materials for starch industry and also used in the preparation of other products **(Shaban *et al.*, 2015).**

Currently, chemical weed control has emerged as an effective tool for weed management it is approachable, less time consuming as well as economical **(Duke and Lydon, 1987; Jarwar *et al.*, 1999 and Baghestani *et al.*, 2007).** A large number of herbicides such as acetochlor are applied directly to the soil **(Huertas-Perez *et al.*, 2006)**. Acetochlor is used as pre-emergence or pre-planting to control annual grasses and certain annual broad leaved weeds. It is absorbed by shoots (less so by the roots) of germination plants and inhibits protein synthesis in susceptible plants **(Anonymous, 2004).**

Accordingly, the aim of the present study was to investigate: a- efficacy of acetochlor against seven annual weeds namely: *Portulaca oleracea* L., *Corchorus olitorius* L., *Amaranthus hybridus* L., *Euphorbia helioscopia* L., *Xanthium pungens* wallr, *Brachiaria eruciformis* L., and *Echinochloa colonum* L. in maize during two seasons

(2013 and 2014). b- The integration between acetochlor and hand hoeing on weed control and maize components. c- Persistence of acetochlor in soil and maize using HPLC analysis.

2. Material and Methods

2.1. Field evaluation experiment:

Field experiment was conducted at the Experimental Station, Sids Agricultural Research, Beni-Suef governorate during 2013 and 2014 seasons. Maize of triple way cross 314 (TWC 314) was sowing on July 9 and July 11 in 2013 and 2014 seasons and harvest at 120 days after sowing. The experimental was laid out in randomized complete block design (RCBD) having four replications with a net plot size was 19.6 m^2 and consisted of 7 ridges each 4 m long and 0.7 m width. The following treatment of herbicide as pre-emergence was applied: acetochlor (Harness 84% EC) 2-chloro-N-(ethoxymethyl)-N-(2-ethyl-6-methylphenyl) acetamide was applied at the two rates of 1.0 and 0.5 L. /fed. Herbicide was applied on soil surface directly after sowing and before irrigation using knapsack sprayer with 200L water. /fed. Ther were four treatments: 1- acetochlor 0.5L/fed with one hand hoeing at 30 days after sowing. 2- acetochlor 1.0L/fed. 3- Hand hoeing twice at 18 and 30 days after sowing. 4- Untreated control (Weedy check) to evaluate the effects of these treatments on seven studied weed species namely: *Portulaca oleracea* L.(Purslane), *Corchorus olitorius* L.(Wild jute), *Amaranthus hybridu*s L.(Pig weed), *Euphorbia helioscopia* L.(Spurge), *Xanthium pungens* wallr. (Cocklebur), *Brachiaria eruciformis* L.(Broadleaf), and *Echinochloa colonaum* L.(Grass jungle). The experimental soil was clay in texture with pH 7.90- 8.0, organic matter 1.68 - 1.71% and available nitrogen 33.10 – 33.0 ppm in the two seasons.

2.2.Data recorded:

All weed species in the different treatments were identified at each evaluation time. Weeds were collected after 45 days from one square meter in each plot. Weed population was measured separately for each weed species by each plot. Fresh weights of weeds were weighed and average weight was calculated.

2.3. Maize yield and its components:

Ten guarded plants were taken randomly from the two central rows of each plot to determine the following characters:

Plant height (cm.), Ear length (cm.), Ear diameter (cm.), Ear weight (gm.), 100 grain weight (gm.) and Grain yield (ardab/fed.) at 120 days after sowing.

Parameter was individually subjected to the ANOVA technique by using computer software. Means were separated by using LSD test at 5% level **(Snedecor and Cochran, 1980).**

2.4.Pigment content determination in maize leaves:

To study the effect of acetochlor on maize pigments, after 15 days of herbicide application the leaf top of plants were taken to determine chlorophyll a, chlorophyll b and carotenoids. Five plants were randomly collected from each treatment. Chlorophyll a, chlorophyll b and carotenoids were determined according to **(Robbelen, 1957)** with little modification by **(Ritchie, 2008),** where 0.2 gm. of fresh leaves was mixed with 10 ml. acetone 85% and ground in mortar in presence of pure sand and calcium carbonate till exhausting green color by washing several times and repeating the extraction if required. The total extraction was made up to 100ml.

The pigments concentration was calculated as mg/ L. by the following formula:

Chlorophyll (a) = 10.3(O.D.) 663- 0.918(O.D.) 644.
Chlorophyll (b) = 19.7(O.D.) 644- 3.87(O.D.) 663.
Carotenoids = 4.75(O.D.) 452- Total chlorophyll x 0.226.

The optical density (O.D.) was determination at 663, 644 and 452 nm of Chlorophyll a, b and carotenoids, respectively by Shimadza Spectrophotometer UV 120-02. The calculated concentration as mg. /L. were converted to mg. /gm. Fresh weight leaves according to **(Wettstein, 1957):**

mg. /gm. =C. V / W. 1000

Where:
C = Concentration of any pigment content as mg. /L.
V = the volume of extraction.
W = the fresh weight of used leaf sample.

2.5.Determination of N, P and K in ear leaves:

At 65 days post planting, samples of ten ear leaves were randomly collected from each plot, taken to the laboratory and oven dried at 50^{o}C until the weight become constant. After complete dryness, samples were grounded in stainless steel mill. The ground samples digested using sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2) as described by **Page *et al.* (1982)**. Total nitrogen was determined using the method of Kjeldahl as modified by **Hillebrand *et al.* (1953)** as follows:

$$\% N = (V – B) X N X V X 14 X 100 / Wt X V_1$$

Where:
N = Normality of HCl solution. (V-B) = Blank
V = Volume of sample 14 = Atomic weight of N.
Wt = weight of plant (g). V_1 = Volume of plant digest used.

Phosphorus was determined using spectrophotometer at 660nm as described by **Dickman and Bray (1940) as follows:**

$$\% P = P_{ppm} (V1 / Wt x 25/ V_2 x 1/ 10000)$$

Where:
V_1 = Volume of sample. Wt = weight of plant (g).
V_2=Volume of plant digest used. P_{ppm} =from calibration curve.

Potassium content was determined using flame photometric procedure as described by **Chapman and Pratt (1961)** as follows:

% K = K_{ppm} (V/Wt x 1 / 10000)
Where:
V = Volume of sample Wt = weight of plant (g).
K_{ppm} = from calibration curve

2.6. Determination of protein and oil content:

Protein was determined as total nitrogen was determined by micro kjeldahl/method and crude protein was obtained by multiplying nitrogen content by 6.25 according to **(A.O.A.C, 2000).** Two gm dry maize grains were extracted with 10 ml petroleum ether at 60^{o}C for 10 hours using a Soxhlet apparatus the extraction was evaporated under reduced pressure to remove solvent, and then total lipids content was calculated according to **(A.O.A.C., 2000).**

2.7.Determination of acetochlor residues in soil using HPLC analysis:

Soil samples were randomly collected from each treatment at 0, 5, 10, 15, 30, 45 and 60 days after spraying. Fifteen grams soil sample was extracted with 10 ml acetonitrile and 2 ml of acetic acid 1% for 1 min, using a vortex mixer at high speed. After that, 1 g sodium chloride and 4 g magnesium sulfate anhydrous were added. The extract was vortexes for 0.5 min, and centrifuged for 5 min at 3800 rpm and 40^{o}C. A 4 ml. aliquot of the upper layer was taken to clean up by dissolved solid phase and extracted with 100 mg. Primary Secondary Amine (PSA), 20 mg Graphitized Carbon Black (GCB) and 600 mg $MgSO_4$. The extract was vortexes again for 1 min. 1 ml. of the upper layer was taken mixed with 2 ml. toluene, and then evaporated to dryness at 40^{o}C using rotary evaporators. The residues were redissolved in 1 ml. toluene for HPLC determination **(QUECHERS methodology Anastassiades *et al*., 2003)**. Quantitative analysis of acetochlor was performed by Hewlett Packard (HP series 1100), quaternary pump, U V – PDA (Photo Diodarray) detector with rheodyne injection system and computer (model acer), U V detector wave length monitored at 220 nm. An ODS Hypersil Agilent Zorbax eclipse plus C18 (3.5µm (3.6 x 150 mm) was used and the column temperature was 40^{o}C. Acetochlor was eluted isocratically with two solvent systems: acetonitrile – methanol 40:60. Flow rate was used at 1.5 ml. /min. A 20µl injector was used to choose the most suitable conditions for acetochlor separation and determination **(Lehotay, 2007).**

2.8.Method validation:

The validation of the proposed analytical method (HPLC - PDA) was carried out according to the **SANCO document 10684/2009.** Linearity was evaluated by constructing matrix matched calibration curves in the range of 0.1–100 µg / L for HPLC- PDA. Method sensitivity and recovery were determined by using samples spiked with the acetochlor at two different levels. Fortified samples were extracted as described earlier and the average recovery percentages for fortified samples were determined. Limits of detection (LOD) and quantification (LOQ) were evaluated as the acetochlor concentration that produces a peak signal-to-noise ratio of 3/1 and 10/1, respectively. The rate of degradation (K) and half-life (RL_{50}) period in soil were calculated according to the equation of **(Moye *et al*, 1987).**

$RL_{50} = Ln_2/K = 0.6932/K$
$K = (1 /tx) \times Ln (a/bx)$
Where:
K = rate of decomposition tx = time in days
a = initial residue bx = residue at x time

3.Results and Discussion

3.1.Field evaluation experiment:

Field experiments were conducted to determine the efficacy of acetochlor applied alone and in combination with one hoeing on seven annual weed species, namely : *Portulaca oleracea* L., *Corchorus olitorius* L., *Amaranthus hybridu*s L., *Euphorbia helioscopia* L., *Xanthium pungens* wallr., *Brachiaria eruciformis* L., and *Echinochloa colonum* L .

Data in Table (1) show the efficiency of acetochlor spraying at different rates of application against seven annual weeds in maize field. Results indicated that there were differences between the untreated treatments and herbicide treatment and also differences occurred between the different treatment during two growing season 2013 and 2014. Generally, the fresh weight (0.0g/m^2) of *Corchorus olitorius* L., *Amaranthus hybridu*s L., *Xanthium pungens* wallr. and *Echinochloa colonum* L. in acetochlor treatment (0.5 L./fed.) with hoeing were less than in acetochlor treatment alone at 1 L./fed. during the two seasons 2013 and 2014. The second order of fresh weight of other weeds treated with 0.5L./fed. + hoeing of acetochlor was *Portulaca oleracea* L., followed by *Brachiaria eruciformis* L. and *Euphorbia helioscopia* L. with values 23.3, 46.7 and 81.3 g/m^2 in season 2013, 53.3 , 77.3 and 98.0 g/m^2 in season 2014.

Acetochlor at recommended dose (1.0 L./fed.) gave the lowest fresh weight (0.0 g/m^2) of *Portulaca oleracea* L., *Amaranthus hybridu*s L. and *Xanthium pungens* wallr. followed by *Echinochloa colonum* L., *Corchorus olitorius* L., *Euphorbia helioscopia* L. and *Brachiaria eruciformis* L. during the two seasons.

Furthermore, it was observed that acetochlor at half recommended dose plus one hoeing gave maximum efficiency than the recommended dose on the tested weeds after 45 days of application. These results are in analogy with the results of **Hassan (2012)** who indicated that acetochlor resulted in fresh weight highest effect as for the full and half dose on *Portulaca oleracea* L**.** and *Xanthium pungens* wallr**.** Also these results are in harmony with those obtained by **Dalley *et al.,* (2006)** and **Abouziena *et al.,* (2008)** showed that the pre-emergence acetochlor was more efficient in eliminating maize weeds. Acetochlor plus one hand hoeing was effective in controlling *Corchorus olitorius* L., *Xanthium pungens* wallr., *Amaranthus hybridu* L., *Portulaca oleracea* L. and *Echinochola colonum* L. The reduction in weeds was ranged from 91.3-88.5% at 60-80 days after sowing **(Soliman and Hamz, 2014).** Finally, the broad leaved weeds were more sensitive than the narrow leaved weeds to the herbicides (**Abouziena *et al*., 2013).**

Table (1). Effect of acetochlor on fresh weight of tested weeds after 45 days from sowing

Treatments	Rate (L. /Fed.)	Weeds fresh weight (g/m^2)						
		2013						
		***Portulacao leracea* L.**	***Corchorus Olitorius* L.**	***Amaranthushybridu* L.**	***Euphorbia helioscopia* L.**	***Xanthium pungens* Wallr**	***Brachiaria eruciformis* L.**	***Echinochloa colonum* L.**
Acetochlor	**0.5 +one hoeing**	23.3 b	0 c	0 b	81.3 c	0 b	46.7 c	0 c
	1.0	0 c	12.0 c	0 b	156.0 b	0 b	186.7 b	10.3 b
Hand weeding	**Two times**	21.3 b	24.7 b	0 b	28.0 d	0 b	34.0 cd	8.0 c
Weedy check	**Unweeded**	2808 a	1198.7 a	390.7 a	572.0 a	348.0 a	1448.0 a	650.7 a
L.S.D $_{0.05}$		18.3	16.5	9.1	36.3	17.4	15.6	8.9
		2014						
Acetochlor	**0.5+onehoeing**	53.3 b	0 c	0 b	98.0 c	0 b	77.3 c	0 c
	1.0	0 d	24.0 b	0 b	182.0 b	0 b	319.3 b	14.0 b
Hand weeding	**Two times**	24.7 c	25.3 b	0 b	32.7 d	0 b	29.3 d	0 c
Weedy check	**Unweeded**	2880.7 a	1292.0 a	454.7 a	758.7 a	544.7 a	1485.3 a	870.0 a
L.S.D $_{0.05}$		9.7	6.3	6.8	5.8	5.6	9.3	12.2

3.2.Maize yield and its components:

According to the results in Table (2) yield and yield components of maize plants were significantly affected by all treatments compared with weedy check. Acetochlor at 0.5L./fed.+ one hoeing and hand weeding treatments significantly increased the plant height, ear length, ear diameter, 100 grain weight and grain yield. The lowest plant height, 100 grain weight and grain yield resulted from maize at 1.0L./fed. of acetochlor. Acetochlor at 0.5L./fed.+one hoeing gave (271.67, 283.33 cm) of plant height, (37.01, 34.97 gm) of 100 grain weight and (26.28, 29.85 ardab) of grain yield in the two seasons, respectively. Insignificant differences were recorded in ear diameter between acetochlor at 0.5L./ fed.plus one hoeing and 1L./fed.alone. The ranged of ear diameter was from 4.20- 4.40cm in season 2013 and 2014. Hand weeding (two times) surpassed the acetochlor treatments for increasing plant height, ear length, ear diameter and grain yield in both seasons. On the other hand, the lowest grain yield was recorded from weedy check treatment 12.87, 13.81 ardab/fed., respectively in two successive seasons. Similar results were obtained with **Shaban *et al.*, (2015)** who reported that the acetochlor at different rates 840, 1280 and 1680 g a.i/fed. increased grain weight per ear as compared with the control and the maximum weight of 100 grains was obtained by the application of acetochlor at 1680 g a.i/fed. and hand weeding twice. These results agree with those of **Khan and Haq (2004)** who found that the increase in maize grain yield was directly correlated with increase in yield components and decrease in density of weeds. While, uncontrolling weeds caused a significant reduction in grain yield compared to hand weeding (two times). **Dalley *et al.*,(2004)** and **Abouziena *et al*., (2007)** found that 66-90% reduction in maize grain yield was due to weed infestation. Reduced grain yield due to weeds may be attributed to several factors, e.g., competition between maize and weeds for water, nutrients and allelopathic effects of weeds **(EL-Metwally *et al.,* 2012).**

Table (2). Effect of acetochlor on maize yield and its component

Treatments	Rate (L. /Fed.)	plant height (cm.)	Ear length (cm.)	Ear diameter (cm.)	Ears weight (gm.)	100 grain weight (gm.)	Grain yield (ardab/fed)
			2013				
Acetochlor	**0.5+one hoeing**	271.67 b	23.27 b	4.40 b	368.33 b	37.01 ab	26.28 b
	1.0	256.7 c	22.07 c	4.40 b	376.79 a	36.63 b	23.70 c
Hand weeding	**Two times**	286.67 a	24.53 a	4.93 a	340.46 c	37.37 a	26.49 a
Weedy check	**Unweeded**	243.33 d	18.00 d	4.53 b	279.26 d	23.61 c	12.87 d
L.S.D $_{0.05}$		4.86	0.57	0.24	5.02	0.55	0.060
			2014				
Acetochlor	**0.5+one hoeing**	283.33 a	21.00 b	4.40 ab	388.15 a	34.97 a	29.85 b
	1.0	268.33 b	21.20 b	4.20 b	382.68 b	34.02 b	25.17 c
Hand weeding	**Two times**	288.33 a	22.27 a	4.53 a	348.37 c	32.71 c	29.98 a
Weedy check	**Unweeded**	236.67 c	17.87 c	3.73 c	290.78 d	25.59 d	13.81 d
L.S.D $_{0.05}$		5.95	0.29	0.19	5.32	0.26	0.084

3.3. Pigment content determination in maize leaves:

Regarding, the effect of treatments on chlorophyll content and carotenoide of maize during both seasons were tabulated in Table (3). Some of the treatments increased, significantly the chlorophyll a, b and carotenoides of the maize, where the highest chlorophyll content assured through acetochlor at 1.0 L./fed. as compared with the other treatments. Its values ranged from 1.039-1.105mg./gm. of chlorophyll a, 0.312-0.365 mg./gm. of chlorophyll b and 0.319-0.273 mg./gm. of carotenoides in both seasons. While, the lowest content of chlorophyll a, b and carotenoides occurred by weedy check treatment during both seasons of the study.

In general chlorophyll pigments were not affected by any weed control treatments indicating the safety of acetochlor on photosynthetic apparatus. Similar results were obtained by **Hassanien(1996)** and **Mekky *et al.,* (2002).** Also, **Safawo *et al.,* (2010)** found that carotenoides are derived from the isoprenoid biosynthetic pathway and are precursors of the plant hormone abscisic acid and of other opocarotenoids. Weed interference for the entire growing season significantly decreased the carotenoides content by 42.9% relative to hoeing treatment.

Table (3). Effect of Acetochlor treatments on maize chlorophyll a, b and carotenoids

Treatments	Rate (L./Fed.)	Chlorophyll a	Chlorophyll b	Carotenoids
		2013		
Acetochlor	**0.5+one hoeing**	0.760 b	0.197 b	0.270 b
	1.0	1.039 a	0.312 a	0.319 a
Hand weeding	**Two times**	0.674 c	0.179 c	0.230 c
Weedy check	**Unweeded**	0.459 d	0.114 d	0.179 d
L.S.D $_{0.05}$		0.19	0.10	0.044
		2014		
Acetochlor	**0.5+one hoeing**	0.782 b	0.268 b	0.249 b
	1.0	1.105 a	0,365 a	0.273 a
Hand weeding	**Two times**	0.634 c	0,192 c	0.249 b
Weedy check	**Unweeded**	0.468 d	0.079 d	0.179 c
L.S.D $_{0.05}$		0.59	0.044	0.014

3.4. Determination of N, P and K concentrations in ear leaves:

The N concentration in straw increased significantly in all the treatments compared with weedy check. The highest value was obtained at hand weeding treatment followed by others (Table 4) in season 2013 and 2014. The P concentration of straw showed significantly in all the treatments from control. The highest value was 0.32 at acetochlor (0.5L./fed. + one hoeing) and 0.31 at acetochlor 1L./fed. in the first season. In the second season acetochlor treatments gave 0.29 of P concentration. Acetochlor at the recommended rate decreased the K concentration in straw were 0.31 and 0.34, respectively in both seasons. Concerning N: P: K of straw, N, P and K concentration was found to be maximum at hand weeding in comparison with the weedy check of the two seasons.

Results tabulated in Table (4) show that the N concentration of grains decreased significantly in weedy check compared with other treatments. Acetochlor alone resulted less effect (1.44 and 1.48) on N than acetochlor + hoeing (1.51 and 1.61) in two seasons, respectively. At the same time, there was no difference between acetochlor alone and with hand hoeing on P and K concentration of grains. Generally, N and P concentrations of straw less than N and P of grains. On the other hand K concentration of straw was more than K of grains in the two seasons. These results agree with **Hossain and Rahman (2013)** who reported that the individual increase of N, P and K was found in all the treatments from the unweeded control but no definite trend of increase was observed.

3.5. Determination of protein and oil content:

Data presented in (Table 5) showed that controlling maize weeds significantly increased the concentration of protein and oil percentage in maize grain in comparison to unweeded control. The lowest values of protein and oil percentage in maize grains were recorded in weedy check. On applying the recommend dose (1L. / fed.), the results differed than those of half the dose + hoeing except for control. Acetochlor applied alone at the recommended dose had highest effect on protein (9.25 and 9.0%) in two seasons, respectively.

Table (4). Effect of acetochlor on N, P and K concentrations on straw and grains of maize.

Treatments	Rate (L. /Fed.)	Straw					Grain				
		2013									
		N	P	K	Total NPK	N:P:K	N	P	K	Total NPK	N:P:K
Acetochlor	0.5+one hoeing	1.37 b	0.32 a	0.35 ab	2.04 b	67.16:15.69:17.16	1.51 ab	0.39 a	0.16 b	1.99 c	72.36:19.60:8.04
	1.0	1.19 c	0.31a	0.31 b	1.81 c	65.75:17.13:17.13	1.44 b	0.38 ab	0.15 b	2.04 b	74.02:18.63:7.35
Hand weeding	Two times	1.63 a	0.32a	0.38 a	2.33 a	69.96:13.73:16.31	1.58 ab	0.40 a	0.18 a	2.16 a	73.15:18.52:8.33
Weedy check	Unweeded	0.93 d	0.29 b	0.26 c	1.48 d	62.84:19.59:17.57	1.12 c	034 b	0.09 c	1.55 d	72.26:21.94:5.81
L.S.D $_{0.05}$		0.12	0.015	0.049	0.18		0.097	0.049	0.015	0.099	
		2014									
Acetochlor	0.5+one hoeing	1.31 b	0.29 ab	0.35 ab	1.95 b	67.18:14.87:17.95	1.61 b	0.39 a	0.15 ab	2.15 b	74.88:18.14:6.98
	1.0	1.22 c	0.29 ab	0.34 b	1.85 c	65.95:15.68:18.38	1.48 d	0.38 a	0.14 b	2.00 c	74.00:19.00:7.00
Hand weeding	Two times	1.51 a	0.31 a	0.36 a	2.18 a	69.27:14.22:16.51	1.67 a	0.39 a	0.16 a	2.22 a	75.23:17.57:7.21
Weedy check	Unweeded	0.81 d	0.28 c	0.31 c	1.40 d	57.86:20.00:22.14	1.21 d	0.33 b	0.08 c	1.62 d	74.88:20.37:4.94
L.S.D $_{0.05}$		0.084	0.015	0.015	0.12		0.095	0.015	0.013	0.10	

In the case of applying the recommended dose, the values of oil content were lower in general than the treatment with half recommend dose + hoeing of acetochlor. These results are in analogy with the results of Soliman and Hamz (2014) who indicated that hand hoeing twice recorded the highest increase in grain protein and oil content, followed by acetochlor + hoeing. While, El-Metwaly, (2002) stated that protein and oil content in maize grains were decreased by 8.0 and 9.2% due to the weed interference and controlling weeds mechanically by hoeing or chemically using acetochlor at the recommended rate produce the greatest grain yield.

3.6.Method validation:

The calibration curve of acetochlor showed strong correlation between concentrations and area in the studied range (0–100 ng/ ml; $r^2 > 0.990$). The LODs and LOQs were sufficiently low; 0.05 µg / kg and 0.1 µg/ kg, respectively. These limits are, in all cases, below the maximum residue limits (MRLs) established by [EU] at 0.01 mg/ kg for fruits. The method had a good repeatability expressed by the relative standard deviation (RSDs) < 12 % and The limits of detection and quantification were found to be 0.2 ng /g and 0.67 ng/ g of dry soil, respectively. The average recoveries ranged from 88.3%–89.4% in all cases, with RSD lower than 8.5 %.

Table (5). Effect of acetochlor treatments on protein and oil percentage of grain maize

Treatments	Rate (L. /Fed.)	2013		2014	
		Protein %	Oil %	Protein %	Oil %
Acetochlor	0.5+one hoeing	10.00 a	3.85 b	9.44 ab	4.17 b
	1.0	9.25 b	3.48 c	9.00 b	3.94 c
Hand weeding	Two times	10.44 a	4.13 a	9.88 a	4.34 a
Weedy check	Unweeded	7.56 c	3.35 d	7.00 c	3.42 d
L.S.D $_{0.05}$		0.51	0.049	0.58	0.069

3.7.Persistence of acetochlor residues in soil:

The level residue of the tested herbicide was dependent on the time after application and depth of soil. The remaining amounts of acetochlor after different days of application to soil were tabulated in Table (6). The initial deposit of acetochlor extracted from soil depth 0.5L. /fed.) decreased further with time to 2.99µg/gm at 45 days after application representing a loss of 63.40%.

At 30 cm depth the % migration of acetochlor at 0.5L./fed. was ranged from 43.45 and 35.13% after 60 days of application. The percentage amount loss from acetochlor (1L./fed.) at 15 cm depth were from 35.05 to 83.74% from 10 to 30 days post application. The rapid degradation continued for acetochlor until the 15 days from application reaching 73.97%, and then degradation became slower and gradual.

Data in Table (6) indicated that the amount loss from acetochlor (1L. /fed.) at 30 cm depth. It increased sharply from zero to 5 days after spraying, whereas the % migration 25.29% and then gradual increased to 39.12, 34.66, 30.69 and 16.91% after 10, 15, 30 and 60 days, respectively.

The appearance of the herbicide in the 5-10 cm layer could not be explained on the basis of the classic- convection- dispersion equation using the measured rainfall. However, temperature had a significant influence on degradation of acetochlor, biodegradation was an important dissipation pathway for acetochlor, but biodegradation alone could not adequately describe dissipation of the acetochlor in the field, soil and moisture had little effect on biodegradation of herbicide **(Qing *et al*., 2000).** The statistical half-life times (RL_{50}) of acetochlor was 10.11 and 12.4 days at 0.5 and 1L. /fed, respectively.

These results agree with those of **Dictor *et al.* (2008)** who found that the half-lives (DT_{50}) of acetochlor varied from 1.4 to 14.9 days depending on the soil temperature and applied concentration. While **Zhen and Deng (2011)** reported that half-life times (t ½) for acetochlor in soil was 6.074 days. **Ma *et al*, 2004** found that the time for 50% (DT_{50}) of initial acetochlor loss was approximately 9 and 56 days, 18 and 63 days at low and high application rates, respectively. They also stated that acetochlor loss in the Horotiu soil possibly resulted from the higher soil organic carbon content that retained more acetochlor near the soil surface where higher temperature and photolysis accelerated the loss.

Residue analysis of acetochlor (0.5 and 1.0 L./fed) at harvest of corn grain showed that no detectable amounts of acetochlor residues, so corn grains could be safely marketed for human consumption after treatment with acetochlor under the normal field conditions. The dissipation of the herbicide residues in/on crops depends on environmental condition, type of application, plant species, dosage, and interval between application, the relation between the treated surface and its weight and living state of the plant surface, in addition to harvest time **(Abdel-Rahman, and Abdell Seid, 2014).**

Table (6). Persistance of acetochlor applied in soil at two different depths.

Time (days)	Application rate (L. /fed.)							
	0.5				1.0			
	15cm	% Migration	30cm	% Migration	15cm	% Migration	30cm	%Migration
0	8.17	0.0	8.17	0.0	20.17	0.0	20.17	0.0
5	6.40	21.66	3.55	43.45	18.26	9.47	5.10	25.29
10	4.13	49.45	3.61	44.19	13.10	35.05	7.89	39.12
15	3.37	58.75	3.45	42.23	5.25	73.97	6.99	34.66
30	3.13	61.69	3.38	41.37	3.28	83.74	6.19	30.69
45	2.99	63.40	3.29	40.27	2.08	89.68	5.10	25.29
60	0.35	95.72	2.87	35.13	0.98	95.14	3.41	16.91
RL_{50}(days)	10.11				12.40			

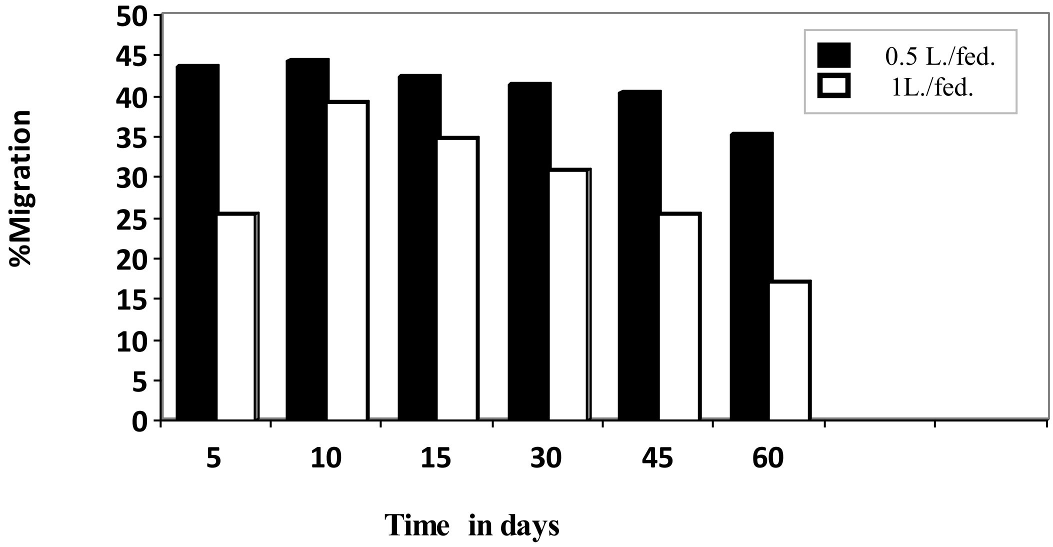

Figure (1). Percentage migration of acetochlor into depth 30 cm.

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كفاءة الاسيتوكلور ضد الحشائش الحوليه في الذرة الشاميه وبقائه في التربه

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الملخص العربى

اجريت التجربه لدراسه كفاءة تطبيق مبيدات ماقبل الانبات (الاسيتوكلور) بمعدل ٠,٥ لتر/فدان + عزقه واحدة و ١ لتر / فدان لمكافحه سبعه حشائش حوليه : الرجله، الملوخيه، عرف الديك، أم اللبن، الشبيط، حشيشه الارانب وأبو ركبه في الذرة الشاميه موسمي ٢٠١٣ و ٢٠١٤. لوحظ وجود إنخفاض كبير في الوزن الطازج لحشيشه الملوخيه، عرف الديك، الشبيط و ابوركبه (٠,٠ جم/م٢) بعد تطبيق الاسيتوكلور بمعدل ٠,٥ لتر/فدان+عزقه واحدة.

كان الترتيب التصاعدي من الوزن الطازج للحشائش الاخري المعامله بنفس المبيد هو الرجله، حشيشه الارانب وأم اللبن (٢٣,٣، ٤٦,٧٢ و ٨١,٣ جم/م٢) في موسم ٢٠١٣. و (٥٣,٣، ٧٧,٣ و ٩٨ جم/م٢) في موسم ٢٠١٤.

الاسيتوكلور بمعدل ٠,٥لتر/فدان متبوعا بعزقه واحدة أدي إلي زيادة معنويه كبيرة في طول النبات، وزن١٠٠ حبه ومحصول الحبوب للذرة الشاميه في الموسمين.

تاكد أعلي تركيز للكلورفيل أ، ب والكاروتينات للذرة من خلال الاسيتوكلور بمعدل ١اتر/فدان وذلك بالمقارنه بالمعاملات الأخري.

كان اعلي مجموع للنيتروجين، الفسفور والبوتاسيوم في القش تم الحصول عليه في معامله العزيق (٢,٣٣ و ٢,١٨) في الموسمين علي التوالي. الاسيتوكلور منفردا أعطي تأثيرا أقل علي النيتروجين في الحبوب من الاسيتوكلور متبوعا بعزقه واحدة. لم يكن هناك أي فروق بين الاسيتوكلور منفردا أو متبوعا بعزقه واحدة علي تركيز الفسفور والبوتاسيوم في الحبوب.

بينما الجرعه الموصي بها من الاسيتوكلور أكثر فاعليه علي البروتين والزيت في حبوب الذرة الشاميه من نصف الجرعه متبوعه بعزقه واحدة حيث أعطت (٩,٢٥ و ٩ %) بروتين ، (٣,٤٨، ٣,٩٤%) زيت في كلا الموسمين علي التوالي.

تقدير ثبات الاسيتوكلور تحت ظروف الحقل في محصول الذرة باستخدام كروماتوجرافي السائل عالي الضغط. منذ تطبيق علي سطح التربه تكون نسبه الفقد علي حسب التركيز، نوع التربه، درجه الحموضه، المواد العضويه والظروف البيئيه.

تم استخراج عينات التربه من أعماق مختلفه(١٥ و ٣٠ سم) في أوقات مختلفه بعد تطبيق المبيد. أظهرت أن جميع الجرعات المطبقه تنتقل إلي العمق. وكانت فترة نصف العمر للاسيتوكلور (١٠,١١ و ١٢,٤) يوم عند نصف الجرعه والجرعه الوصي بها علي التوالي.