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Fate of Certain Pesticides in Presence of Biochar in Cultivated Soil

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Abstract Biochar from date palm by product was produced through slow pyrolysis at 450°C done in a batch retort. The identification of the chemical composition of biochar by using Fourier Transform Infrared spectroscopy, (FTIR) and elemental analysis show that there are function group found around the biochar structure. Its physical characterization by using X-Ray Diffraction (XRD). Surface area (BET analyse) found to be 96.4 m2/gm and its Particle Size Distribution (DLS) had mean size of 172.5 nm and 41.4 nm width. Biochar-amended soil were prepared by thoroughly mixing the soil with accurately weighed of biochar of percentages of 0, 10 and 25 % (w/w). Onion was plant in a sand soil. Two pesticides (Acetamprid and Oxamyl) were applied once after 60 days from planting . Residues were extracted inside onion blubs after 1, 3, 7 and 14 days from application. It is clearly proved that the half-life time of both pesticides are increased in the soil contain biochar. In addition to the leaching of pesticides from the agricultural soils to the run out water show statistically significant as the soil contain biochar had no pesticide residues after application.

Keywords: Biochar, Pesticide Residue, acetamprid, oxamyl

Introduction

Growers mainly relay on pesticides to suppress the population of pests that attack their crops Fathia *et al.* (1983) and El-adawy *et al.* (2001). Soil pollution with pesticides was occurred directly via soil treatment or indirectly as pesticides drift via aerial application. Farmers in Egypt prefer cultivating vegetable crops on their farms because the suitable environmental factor, beside the outcome is some what satisfactory for them. Also farms are planted many times through the year. Onion is one of these crops.

Onion is a preferable for both framers as cash crop and consumers as principle vegetable whether for cooking or as fresh product. Onion plants have rich sources of natural substance. The crop submitted to severe invasion by numerous pests stem and bulb nematode Ditylenchusdipsaci, root-knot nematode, Meloidogyne javanica and Meloidogyne chitwoodi, Rice root-knot

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nematode Meloidogyne graminicola, Onion thrips Thrips tabaci, Onion maggot Delia antique, Tobacco caterpillar Spodoptera exigua, Gram caterpillar Helicoverpa armigera, Bulb mites Rhizoglyphus-Tyrophagus and Leaf miner Liriomyza Mishra *et al.* (2012).Wide range of pesticides are marked to control these pests. Acetamiprid highly active neonicotinoid insecticides used to protect the various vegetable crops, by controlling numerous sucking and biting insects, including aphids, whiteflies, beetles and some lepidoptera species. Tomizawa and Casida (2003). Oxamyl is a high toxic carbamate pesticide used in controlling most nematode species and spider mites in addition to a large number of sucking and chewing insects such as aphids and thrips. It absorbed by the foliage and roots.

These pesticides lead to soil pollution and high concentration of chemical residues in onion after harvest. Soil contamination has become an increasing environmental problem. Pesticide residues are one group of contaminants in soils that have received great attention in the past decades. With heavy use of pesticides in modern agricultural production, pesticides have been frequently detected in vegetables and cause a great threat to the human health Sun *et al.* (2004).

The pesticides may be diffused to underground water. So, get rid of pesticides residues or at least minimize it is necessary.

Biochar is solid material composed of highly stable poly-aromatic bonds which generally resist weathering and decomposition by microbial communities. Biochar is capable of both absorption and adsorption. The surfaces of biochar, both internal and external, adsorb materials by electro-chemical bonds, working like an electric sponge.

Objectives of the present study is clarify the role of biochar in cultivated soil in presence pesticides.

Materials and methods

Preparation of biochar material

The biochar from date palm was obtained from Egyptian –Finnish project Eg. It was produced through slow pyrolysis at 450 ℃ done in a batch retort. The prepared biochar was ground into powder shape, then passed through a sieve. Mesh size of 0.841mm and 0.42 mm. The obtained material was stored in a dry place until use. The soil used in the experiment was collected from the farm of Agriculture Research Station in Ismailia soil layer (0-20 cm).It was sand texture grade (Sand 92%- Silt 5.8%-Clay 2.2%) and its organic matter was 0.13%. It was passed through a sieve.

Identification the Properties of biochar

Chemical composition

A. Fourier Transform Infrared spectroscopy, (FTIR):

FT- IR spectroscopy of solid samples of biochar relied on a Bio-Rad FTIS - 40 model USA. Sample (100µg) was mixed with 100 µg of dried Potassium Bromide (KBr) and compressed to prepare a salt disc (10 mm diameter) for reading the spectrum further wave length between 450-4000 cm-1. Micro Analytical Center, Faculty of Science, Cairo University.

B. Elemental analysis of (C, H, N, S) done by using elemental analyzer vario el cube using thermal conductivity detector Micro Analytical Center, Faculty of Science, Cairo University. Biochar oxygen content were done after knowing the whole elements.

Physical characterization

A. X-Ray Diffraction (XRD) Instrument specification:-Model: X'Pert PRO PANalytical – Netherlands Measurements at XRD. X-Rays diffractometer through the range of 10-700 2θ Unit of Nanotechnology and Advanced Material Central Lab (NAMCL), Agriculture of Research Center (ARC). We use Bragg's law to determine the inter planar spacing d by using equation

$n\lambda = 2d \sin\theta$

where λ is the wave length of the X-Rays

d is the inter planar spacing

θ is the diffracted angle

B. The specific surface area (BET). It was measured by Brun-auer-Emmett-Teller method in which N2 adsorbtion was applied at 77K and Carlo Erba Sorptometer was used.

C. Particle Size Distribution (DLS).

Zeta sizer nano Company name: Malvern, UK. Model: Zeta sizer nano series (Nano ZS). Size range (nm):0.6:6000 nm, Zeta potential range (mV): (-200:200mV). Unit in Nanotechnology and Advanced Material Central Lab (NAMCL), Agriculture of Research Center (ARC).

Soil Processing and planting

Biochar-amended soil were prepared by thoroughly mixing the soil with accurately weighed of biochar. The percentages of the biochar by weight in the amended soil were 0, 10 and 25 % (w/w), of the total weight. S, 10%BC and 25%BC referred to soil free from biochar, soil contain 10% biochar and the soil contain 25% biochar respectively. Pots of each percentage were employed.

Onion (Allium cepa) was planted in the pots five plant in each pots at the 1st of in January 2015 and 2016. Irrigation scheduling has been done when it was necessity.

Pesticides: Two pesticides were used

1-Acetamiprid 20 % SP. Trade name Mosplane.(Neonicotinoid Insecticide). Systemic insecticide with contact and stomach action. It applies as soil application. Recommended dose is 25gm/100Lwater

Chemical Structure

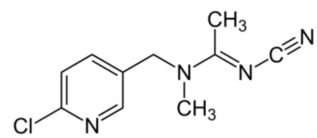

Figure 1. Acetamprid chemical structure

Name (IUPAC): E)-N1-[(6-chloro-3-pyridyl)methyl]-N2-cyano-N1-methyl acetamidine

2- Oxamyl 24 % SL. Trade name Vaydate (oxime carbamate pesticide). Soluble in water, cholinesterase inhibitor. It is contact and systemic pesticide .It applies as soil application. Recommended dose is 3L /100L water.

Chemical Structure

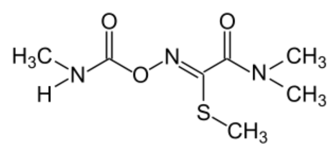

Figure 2. Oxamyl chemical structure

Name (IUPAC): Methyl 2-(dimethylamino)-N-[(methylcarbamoyl)oxy]-2-oxoethanimidothioate

The two pesticides were applied once after 60 days from planting.

Sampling

Five ripe onion blubs from each pot were collected in plastic bags at 1, 3, 7 and 14 days after application with the tested pesticides. Samples of the run out water from irrigation (50ml) were taken in a glass bottle at 1, 3, 7 and 14 days .

Samples transferred to the laboratory of plant protection division in Agriculture Research Station at Ismailia Government. The bags were kept in the refrigerator - 4◦C until analysis.

Residues analysis

Acetamiprid

Residues were extracted according to the method of Masanori and Gomyo (1994). Onion samples of 50 g were homogenized with 200ml methanol, then filtered. The filtrate was shaken with 10ml of sodium chloride solution then we add 100 ml hexane, the hexane layer was discarded. The aqueous methanol was extracted with 100 ml dichloromethane. The anhydrous sodium sulfate was used to dry dichloromethane layer. The extract was concentrated to near dryness under reduced pressure. The extract was cleaned up by column chromatography using florisil activated 60-100 mesh (10g). The column was first eluted with 150 ml of mixed solvent of acetone and hexane (20:80) and it was discarded. st Then it was eluted with 150ml of mixture of acetone and hexane (50:50). The eluted was collected and concentrated to dryness by rotary evaporator at 40 °C.

Oxamyl

Residues were extracted according to the Holt and Pease, (1976). Onion samples of 50 g were homogenized with 200ml ethyl acetate for 5 min. Transfere the homogenized mixture into centrifuge tube and centrifuge at 1500 r.p.m for 10- 15 min. Suction - filter.the liquid through a fast slow – rate filter paper covered with 5 gram filter aid contained in a Buchner porcelain funnel, and collect in round bottomed flask.Extract the residue in centrifuge tube two more time in the same way using ethyl acetate. Add 50 ml water to the combined filtrates and rotary evaporator at 40 °C to an aqueous residue.

Each sample transferred quantitatively to small glass vials which has a glass stopper to analysis. The residues of acetamiprid were estimated by high performance liquid chromatograph (HPLC) and the residues of Oxamyl were estimated by Gas liquid chromatograph. (GlC)Samples of each of blubs and irrigation water were transferred to Central Agriculture Pesticides Laboratory, Doke, Giza.

Degradation of the two pesticide acetamipride and oxamyl were calculated according to the method described by Ahmed (1989). The Half-life time, Mean time and Decay constant was calculated from the equation.

$t\ ½ = \frac{\ln ½}{\ln Ct - \ln C0}\ X\ t$ $\quad t_{1/2} = \ln(2)T = \ln(2)/\lambda$

t ½ =ln½/(lnCt-lnC0) X t $\quad$ t1/2 = ln(2)T = ln(2)/λ

Where:

C_0 is the initial concentration

C_t is the concentration that still remains after a time t
$t_{1/2}$ is the half-life T is the mean lifetime λ is the decay constant

Statistical Analysis

Data obtained in the experiment were subjected to computerized statistical analysis. Duncan's multiple range tests was used to determine the significant differences between the mean values of the pesticides residues.

Results

Identification the Properties of biochar

Chemical composition

A. Fourier Transform Infrared spectroscopy, (FTIR):

It was necessary to investigate the Infrared spectroscopy (IR) figures (3) to show the main function group found around the biochar structure. It has 6 main bands. Sample showed a strong broad band 2 at ~ 3400 cm-1 which indicate the presence of free hydroxyl (O-H) ,the two band 5 and 6 at ~ 1600 and 1440 cm -1 indicated the stretching of the aromatic ring .Also, bands 7 at ~ 1110 cm-1 indicate the stretching of (C-N) . The two bands 11 and 12 < 660 cm-1 indicated the presence (C-X).

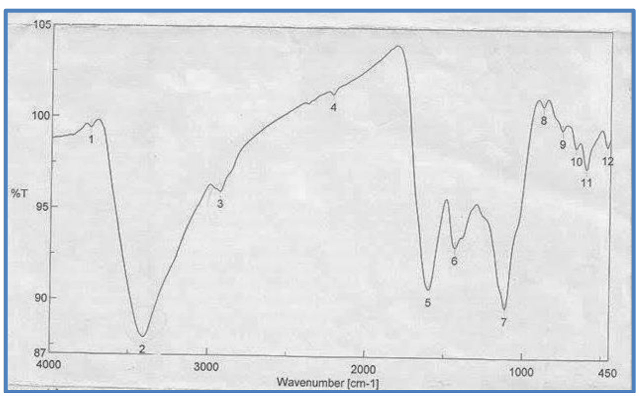

Figure 3. FTIR spectroscopy biochar

B. Elemental analysis of biochar obtained from palm tree

Table 1. Elemental analysis of biochar obtained from palm tree.

C%	**80.1%**
H %	6.2%
N %	2.5%
O %	5.5 %

H/C (aromaticiy)	0.21
O + N /C (polarity)	0.099

Physical characterization

A. X-Ray Diffraction (XRD). We used Bragg's law to determine the inter planar spacing d .This is experimental d value depend on 2θ measured experimentally. These pattern identified sharp peaks at 2θ of about at 20.10, 26.610, 31.6710, 39.40, 50.10 and 59.89 which had accounts number of 268.84, 1669.66, 313.07, 305.05, 277.13 and 129.11 respectively. The calculated d where ~ 4.2671, 3.3349, 2.8251, 2.2853, 1.8208 and 1.5442.

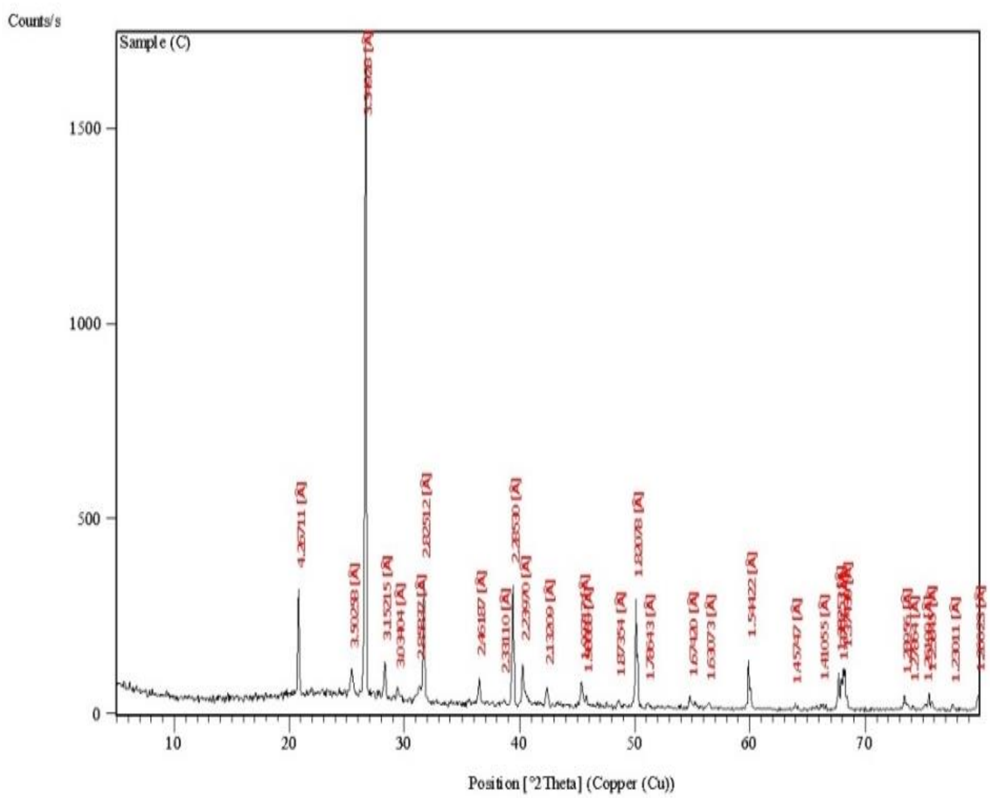

Figure 4. X-Ray Diffraction(XRD) of solid biochar

B. The specific surface area (BET)) found to be 96.4 m2/gm

C. Particle Size Distribution (DLS). It was noticed from figure 5 that the ~22 % of biochar sample had mean size of 172.5 nm and 41.4 nm width.

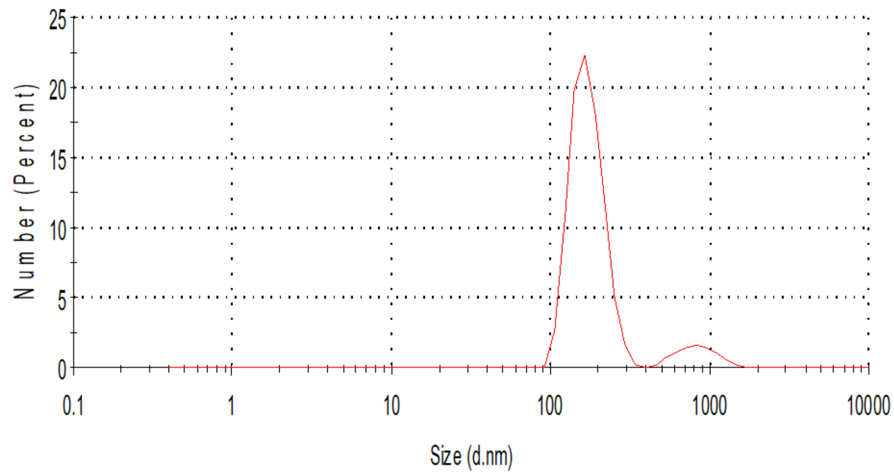

Figure 5. Particle Size Distribution of solid biochar

Residue values of the two studied pesticides Oxamyl and Acetamiprid

Onion plants were sprayed once with Acetamiprid: and Oxamyl with the recommended doses 25gm/100L for the acetamiprid and 3L/100L for the Oxamyl to clarify the results. Samples of onion were collected at random from treated and untreated pots after 1, 3, 7 and 14 days of application to determine the residues. The purpose of pesticide residues monitoring to show the fate of pesticides in onion that could be unexpected residues in the presence of biochar.

The result in table 2 showed that the persistence of acetamiprid inside onion bulb after three day of application was 6.46, 53.78 and 64.79 % for Soil, 10% BC and 25% BC respectively. After seven days of application there isn't persist of acetamipride for soil free from biochar but founded to be 15.15 and 26.18% for 10% BC and 25% BC respectively. The results also clarified that after 14 days there were a persist of acetamiprid inside onion bulb by 3.788 and 5.1 % for 10%BC and 25% B C, respectively.

The half-life times for acetamipride inside the onion were 12.14 , 53.64 and 76.66 hours for S, 10% BC and 25% BC respectively. The result also showed that the mean life time which were 17.52, 77.38 and 110.6 hours for S ,10% BC and 25% BC respectively. The decay constant (λ) found to be 1.37, 0.312 and 0. 216 days for S, 10 % BC and 25% BC respectively.

Table 2. Residues of acetamiprid in onion bulb (corrected according to the recoveries percent).

Type soil	Time (day)	Mean Residue(PPm)	Loss %	Persistence %	T½ (hrs)	T (hrs)	λ (day)
S	1	10.6	-	100	12.14	17.52	1.37
	3	1.03	93.54	6.46			
	7	ND	100	-			
	14	ND	100	-			
S + 10% BC	1	10.32	-	100	53.64	77.38	0.312
	3	7.1	46.212	53.78			
	7	2	84.84	15.15			
	14	0.5	96.212	3.788			
S + 25% BC	1	14.4	-	100	76.66	110.06	0.216
	3	9.33	35.208	64.79			
	7	3.77	73.819	26.18			
	14	0.74	94.86	5.1			

% Loss $=\frac{residue\ at\ first\ day - residue\ found\ at\ different\ time}{residue\ at\ first\ day}\ X100$ S=Soil

BC= Biochar
Persistence = 100 - % Loss $T_{1/2}$ = Half life time T=Mean life time λ = Decay constant

There was high significant (f=53.54 , df =2, P 0.001).

The result in table 3 showed that the persistence of oxamyl inside onion bulb after three day of application was 30.78, 70.88 and 77.16 % for soil free from biochar, soil contain 10% biochar and the soil contain 25% biochar respectively. After seven day of application the persist of oxamyl was 7.12, 45.88 and 56.09 % for soil free from biochar, soil contain 10% biochar and the soil contain 25% biochar respectively.

After 14 days of application there was not persisted of oxamyl inside onion bulb planted in soil free from biochar but founded to be 17.48 and 17.766% for soil contained 10% biochar and the soil contained 25% biochar respectively.

The half-life times for oxamyl inside the onion were 1.18 , 5.33 and 7.19 days for S, 10% BC and 25% BC respectively. The result also showed that the mean life time were 1.7., 7.67 and 10.38 days for S, 10% BC and 25% BC respectively. The decay constant (λ) found to be 0.6, 0.12 and 0.096 days for S, 10% BC and 25% BC respectively.

Table 3. Residues of oxamyl in onion bulb (corrected according to the recoveries percent).

Type soil [a]	Time (day) [b]	Residue Mean(PPm)	Loss %	Persistence %	T½ (day)	T (day)	λ (day)
S	1	131	-	100	1.18	1.7	0.6
	3	40.1	69.211	30.78			
	7	9.33	92.878	7.12			
	14	ND	100	0			
S + 10% BC	1	137.3	-	100	5.33	7.67	0.12
	3	97.33	29.11	70.88			
	7	63	54.11	45.88			
	14	24	82.52	17.48			
S + 25% BC	1	131.33	-	100	7.19	10.38	0.096
	3	101.33	22.84	77.156			
	7	73.66	43.90	56.09			
	14	23.33	82.23	17.766			

% Loss $= \frac{residue\ at\ first\ day - residue\ found\ at\ different\ time}{residue\ at\ first\ day} X100$ S=Soil BC= Biochar

Persistence = 100 - % Loss $T_{1/2}$ = Half life time T=Mean life time λ = Decay constant

[a] There was high significant difference (f = 196.99 df= 2 p = 0.0001)

[b] There was high significant difference (f = 1369.58 df = 3 p = 0.0001)

The result in table 4 showed that residues of acetamiprid and oxamyl of the run out water after application were 16.17 and 189 ppm for soil free from biochar but the soil contain 10% biochar and the soil contain 25% biochar was not detectable in water for acetamipride and found to be 29 and 4.4 ppm for oxamyl. The residues of oxamyl after one day of application 5.3 and 54.33 ppm for soil free from biochar biochar but the soil contain 10% biochar and the soil contain 25% biochar was not detectable in water.

Table 4. Concentration (ppm) values of acetamiprid and Oxamyl of the run out water.

Type	Time	Pesticide concentration (PPm)	
		Acetamipride Mean	Oxamyl Mean
S	0*	16.17	189
	1	5.3	54.33
	3	ND	4.6
	7	ND	ND
S + 10% BC	0*	ND	29
	1	ND	
	3	ND	ND
	7	ND	ND
S + 25% BC	0*	ND	4.4
	1	ND	ND
	3	ND	ND
	7	ND	ND

ND: Not detectable

Discussion

The Properties of biochar

The IR chart showed the complete combustion of waste to form aromatic fused carbon ring with some free active group. It showed clearly the presence of amine group in the structure of biochar and free hydroxyl groups. This showed the high adsorbtion ability of biochar. Biochar obtained at low pyrolysis temperatures are characterized by a lower surface area and aromaticity but higher polarity and the amount of oxygen-containing functional groups on its surfaces, and may thus be more suitable in removing inorganic/polar organic contaminants Ahmad *et al.* (2014).

Biochar from palm tree physical characterization clarify that it had a great surface area and 22% had a nano size in addition to the distance between molecule in the crystal lattice was 0.33 nm this clarify the high ability of biochar to make absorption.

Residues in onion

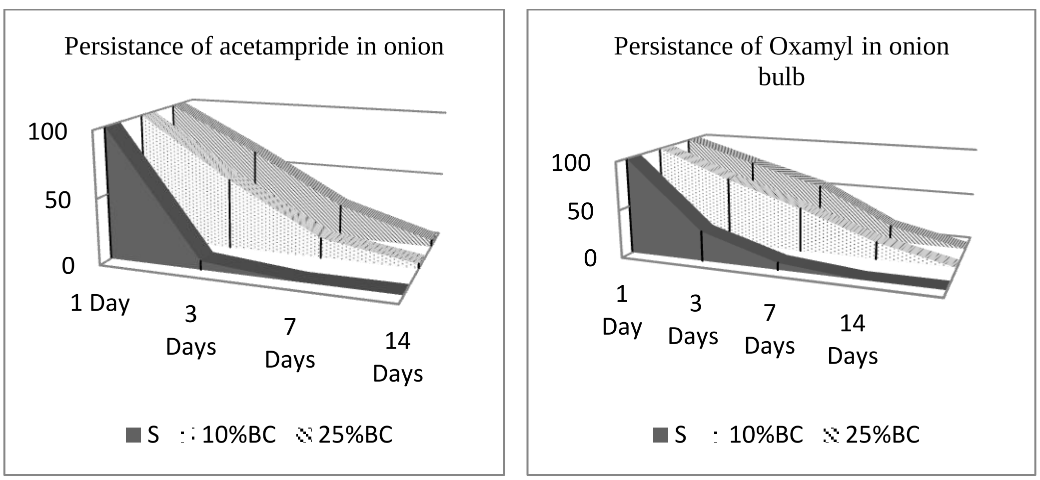

Figure 6. Persistance of acetampride and Oxamyl

Biochar obtained from wastes of Palm trees appeared to have an effect in the persistence of both acetampride and oxamyl in onion bulb. The results showed that there is a statistically significant between the soil free from biochar and the soil contain 10 or 25 % biochar. It is clearly proved that the half-life time of both pesticides are increased in the soil contain biochar. These results agreed with Yang *et al.* (2010) which showed that the loss of chlorpyrifos and fipronil pesticides in soils decreased significantly with increasing amounts of the biochars in the soil. Also biochar reduced the leaching of glyphosate (herbicides) from the soil by 24–27% Hagner (2013). Ogbonnaya and Semple (2013) said that the presence of biochar in the soil increased the half-life of the pesticides. The results obtained may due to the both the absorption capacity of biochar which results from the high surface area and its pores structure ,also the adsorption due to electro chemical bonding which results of the free active group on the surface of biochar.

The leaching of pesticides from the agricultural soils to the run out water show statistically significant as the soil contain biochar had no pesticide residues after application.

These results clarified the rule of biochar which has high absorption property by having a great surface area and have huge amount of pores, and also the role of various functional groups. So, its structural and electrochemical properties were affected biochar sorption capacity (Hagner *et al.*, 2015). In sandy soil the degradation of pesticides decreased as biochar quantity increased (Diez *et al.*, 2013). Also, in the study of Jones *et al.* (2011) stated that biochar also reduced the downward movement of pesticides in response to artificial rainwater, thus potentially reducing the risk of groundwater contamination.

Our results indicated that applications rates of pesticides should be revised for use in farming systems with biochar. Lower doses may give good control because of the longer duration of the pesticide in root systems. This would enable lower costs for farmers and lower environmental risks because of lowered use of pesticides in long run. However, if the use rates are not lowered pesticides residues in products such as onion may be unacceptable and cause severe health problems as well as the products will not be marketable.

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