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EFFECT OF STORAGE IN DIFFERENT TEMPERATURE ON THE STABILITY OF CHEMICAL AND PHYSICAL PROPERTIES OF SOME BENZOYL UREA INSECTICIDE FORMULATION

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ABSTRACT

The present paper is an overview of the persistence of Chlorfluazuron (Atabron 5%), Lufenuron (Match 5%), Flufenoxuron (Cascade 10%) and Hexaflumuron (Consult 10%) under certain environmental factors, the present study was investigated the stability of active ingredients under accelerated storage through 54°C ± 2, 45°C ± 2, 40° ± 2, 35°C ± 2 , 30°C ± 2 for 14 days, 6 weeks, 8weeks, 12 week , 18 week respectively, also, including some consideration for developing studies physical properties for each unsorted and stored benzoyl urea formulation. Generally the chemical properties of the previous pesticides were estimated by HPLC. The results obtained could be summarized as follow: Data showed that the degradation of active ingredient not exceeds the international limit (acceptable limit) under pervious storage temperature. On the other hand, data showed that Lufenuron more persistence and Chlorfluazuron high degradable under temperature degrees (25°C, 35°C and 45°C). The different physical properties for unsorted and stored tested pesticides are in all allowed limits according to WHO and FAO.

INTRODUCTION

The term "urea pesticide" includes many compounds with important herbicide and insecticide activities. Chemically. Only the presence of a urea moiety is common to all them. Urea pesticides can be classified according to their chemical structures into substituted urea's, sulfonylurea (SUHS), benzoly urea's (BUIS). Most of the substituted urea's are phenyl urea's (PUHS) and the rest Contain heterocyclic groups. Worthing and walker (1983), Clark and Jwess(1990).

BUIS act as insect growth regulators, interfering with the chitin formation in the vital insect exo skeleton Tomlin (1994), Roberts and Huston(1999).

The physical and chemical properties of the urea pesticides determine the efficacy of the extraction, cleanup and detection processes, urea pesticides are colorless and odorless .Most Substituted urea herbicides are chlorinated ,and most BUIS contain fluorine atoms. Substituted urea herbicides have low molecular weights (usually less than 300), those of SUHS and BUIS are bigger, reaching 540 for BUI Chlorfluazuron. Substituted urea herbicides and BUIS are natural substances, while SUHS are acidic compounds. Urea pesticides manifest low acute toxicity for mammals and birds and high toxicity for the aquatic environment with prolonged use. Amfts. J.C.R. (2002), Nollet. B.L.(2004)

Concerning our study, this paper includes some development studies on the persistence of benzoyl urea e.g Cholerfluazuron, Lufenuron,

Flufenoxuron and Hexaflumuron in their formulation under some environmental components such as storage for different periods at 54°C ± 2, 45°C ± 2, 40° ± 2, 35°C ± 2 , 30°C ± 2 for 14 days, 6 weeks, 8weeks, 12 week , 18 week respectively., effect different temperature levels (25,35 and 45°C) then study the different physical and then study the stability and degradation of tested pesticides by estimate chemical properties (active ingredient value residues). Under supervision international limits according to WHO and FAO.

MATERIALS AND METHODS

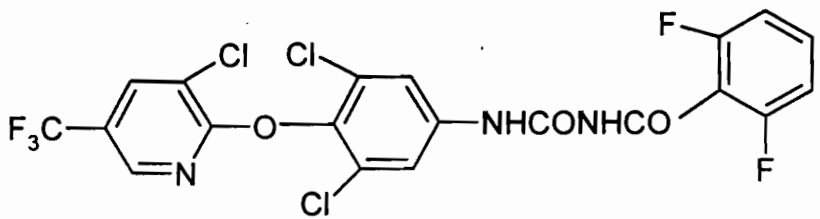
1. Pesticides test

A. Chlorfluazuron (Insecticide)

Common Name : Chlorfluazuron

IUPAC Name: 1-[3.5 dichloro-4- (3-chloro-5-trifluoromethy -2-pyridyloxy) phenyl] -3- (2.6- difluoro benzoyl) urea

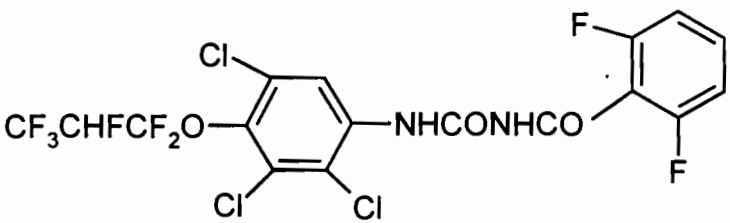
- Chemical Structure:



- Chemical Family: Benzoyl Urea.
- Trade Name : Atabron 5% EC

B. Lufenuron (Insecticide, Acricide)

- Common Name : Lufenuron
- IUPAC Name : (RS)-1 [2.5-dichloro-4- (1.1.2.3.3.3 hexafluoropropoxy) phenyl]-3- (2.6 difluoro benzoyl)
- Chemical Structure:



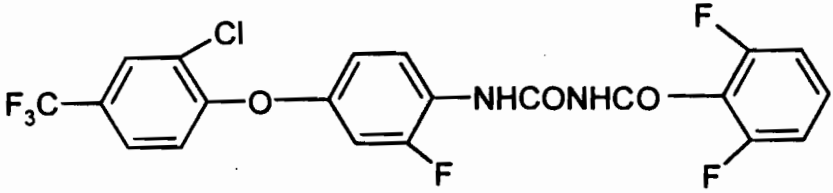
- Chemical Family: Benzoyl Urea
- Trade Name : Match 5% EC

C Flufenoxuron (Insecticide,Acaricide)

Common Name: Flufenoxuron

IUPAC Chemical Name : 1- [4-(2- chloro- α, α, α, - trifluoro p. tolyloxy). 2- fluoro phenyl] -3- (2.6- difluoro benzoyl urea)

- Chemical Structure:



Chemical Family Benzoyl Urea

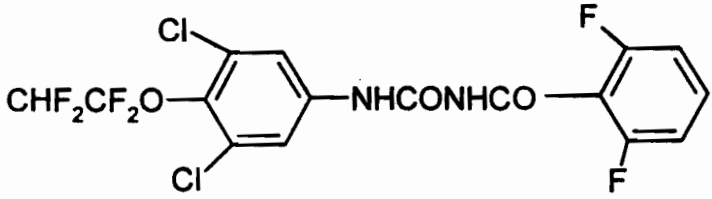
- Trade name : Cascade 10% EC

D. Hexaflumuron (Insecticide)

- Common Name: Hexaflumuron

IUPAC Name: 1-[3.5- dichloro-4- (1.1.2.2. tetrafluoro ethoxy) phenyl] -3- (2.6-difluoro benzoyl) urea

- Chemical Structure:



- Chemical Family: Benzoyl Urea
- Trade Name: Consult 10% EC
- EC: Emulsifiable Concentration.
- DC: Dispersible Concentration.

II. Effect Of Certain Environmental Condition On The Fate Of Chlorfluazuron, Lufenuron, Flufenoxuron and Hexaflumuron In Their Formulation

(1) Stability at O °C: The pervious benzoyl urea formulation. at O ± 2°C for 7 days. Mt. 39.2 : CIPAC F (1995), FAO(2007).

(2) Stability Of Storage At Elevated Temperatures:–

The pervious benzoyl urea formulation were stored in a dark glass package:-

In oven at 54°C ± 2, 45°C ± 2, 40° ± 2, 35°C ± 2 , 30°C ± 2 for 14 days, 6 weeks, 8weeks, 12 week , 18 week respectively. FAO / WHO Manual(2002), (2004) and (2006) , FAO(1988),(2007).

(3) Stability Through Different Temperatures.

a) Aliquots of the tested pesticides representing I ml Acetone containing 500 µg a.i (Active ingredient)each were spread as uniform ally as possible on the surface of uncovered Petri dishes (5cm i,d). The acetone solvent was left to dry at room temperature treated Petri dishes were exposed to 25, 35 and 45°C for 1, 2, 4, 8, 12, 24, 48 and 192 hours inside a dark electric oven provided with temperature regulating system. Nasser N(2001)

(4) Physical properties were evaluated before and after different storage periods in this study:-

a) Density properties MT 3.2 CIPAC F(1995)
b) Emulsion and Dispersion stability MT 36 and MT180 CIPAC F(1995)
c) Free Acidity or Alkalinity MT 31 CIPACF(1995)

d) pH values MT 75 CIPAC F (1995)

(5) Chemical Analysis:-

- **Determination of active ingredient % in benzoyl urea pesticides as follow:-**
- **Stability at elevated temperatures**: determined test benzoyl urea formulation , before and after storage by HPLC system.
- **Stability through different temperatures** :residues of tested insecticides which were remained on exposed surface were quantitatively transferred to standard glass stopper test tubes with dichloromethane and the solvent was evaporated to dryness and the residues were determined by HPLC system.
- **High Pressure Liquid Chromatography (HPLC) Conditions:** the chromatographic system consisted of Jusco HPLC, diodearry detector Model 12015 and intelligent quaternary pump Model PU. 2089. UsingC_{18} stainless column (4.6 nm id 25cm) at 40°C. Chlorfluazuron, Lufenuron, Flufenoxuron and Hexaflummuron in their formulation were eluted isocratic ally Methanol –Acetonitril-Water (65:30:5), at the rate of 1 lm/min. under these conditions. The R_t were 3.10, 1.75, 2.9 and 2.7 min for Chlorfluazuron, Lufenuron, Flufenoxuron and Hexaflummuron in their formulation respectively. The result of tested insecticides were qunitively determined by comparison with standard solutions injected under the identical HPLC conditions according to the methods of Wendy and Denis (1990),Miliadis et al(1999), Shimazu, (2003) and FAO (2007)

(6) Kinetic Study: In order to study the rate of degradation of the tested active ingredients and half life periods RL_{50} for tested pesticides were calculated according to equation (Moye et al 1987).

$RL_{50} = \ln 2/k = 0.6932/k$ $k = 1/t_x . \ln a/b_x$

where k. rate of decomposition a: initial residue.

t_x: time reside b_x : residue at x time

RESULTS AND DISCUSSION

II. **Effect of Certain Environmental Condition Of The Fate Of Chlorfluazuron, Lufenuron, Flufenoxuron and Hexaflumuron In Their Formulation:**

1.Stability at 0 °C: After storage at 0 ± 2 °C for 7 days. The maximum amount of separated solid of benzoyl formulations are 0.3 ml

The applicability of this Results are established on the clause and limit are upon it MT 39.2, CIPAC F(1995)and FAO (2007)

2. Stability of storage at elevated temperatures:-

Data presented in tables (1-5) showed that degradation of the a i not exceeds 5% as quantified. Degradation products has acceptable levels of storage stability and meet the stated requirements. Avoidance of temperatures exceeding 50°C is likely to be necessary for the formulation during storage (b-e) (3) Stability Through Different Temperatures.The average active ingredient content should not decline to less than 95% of average content measured period to the test FAO/WHO Manuel(2002),(2004),(2006) ; FAO (1988), (2007).

Table 1: In oven at 54°C ± 2

Storage period days	Chlorfluazuron		Lufenuron		Flufenoxuron		Hexaflumuron	
	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%
0	5.00	0.00	5.00	0.00	10.00	00.0.	10.0	000
14	4.95	1.00	4.96	0.8	9.90	1.00	9.89	1.10

Zero = one hour before storage.

Table 2: In oven at 45°C

Storage period weeks	Chlorfluazuron		Lufenuron		Flufenoxuron		Hexaflumuron	
	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%
0	5.00	0.00	5.00	0.00	10.00	00.0.	10.0	000
6	4.96	0.8	4.97	0.6	9.96	0.4	9.96	0.4

Zero = one hour before storage

Table 3: In oven at 40 °C

Storage period weeks	Chlorfluazuron		Lufenuron		Flufenoxuron		Hexaflumuron	
	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%
0	5.00	0.00	5.00	0.00	10.00	00.0.	10.0	000
8	4.98	0.4	4.98	0.4	9.98	0.2	9.98	0.2

Zero = one hour before storage

Table 4: In oven at 35°C

Storage period weeks	Chlefluozuron		Lufenuron		Flufenoxuron		Hexaflumuron	
	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%
0	5.00	0.00	5.00	0.00	10.00	00.0.	10.0	000
12	4.99	0.2	4.99	0.2	9.99	0.1	9.99	0.1

Zero = one hour before storage

Table 5: In oven at 30°C

Storage period weeks	Chlefluozuron		Lufenuron		Flufenoxuron		Hexaflumuron	
	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%	a.i%	Loss%
0	5.00	0.00	5 00	0.00	10.00	00.0.	10.0	000
18	4.99	0.2	4.99	0.2	9.99	0.1	9.99	0.1

Zero = one hour before storage

Generally, data in tables(6-9), illustrate that the benzoyl formulation high stable at 25°C and this stability decrease with increasing temperature conditions, So, Lufenuron most stable than the other three bezoyl urea and the Chlorfluazuron more degradable than other benzoyl urea formulations. It is clearly evident that there is affirmative relationship between temperature and rate of degradation. The previously mentioned results clearly showed that the rate of persistence of tested benzoyl urea were influenced by many factors such as: chemical structures, vapor pressure, concentration of pesticides applied and period of exposure. In general increasing the period of exposure increased the rate of residues degradations. The statically half-lives for tested pesticides support that results. The results obtained agreed with findings of *Hong and pehkonen (1998), Barakat et al., (1999); (2001) and Nasser et al. (2003)*

Table 6: Effect of different temperature degrease on the persistence of Chlorfluazuron residues:-

Time periods(hrs)	25%		35°C		45°C	
	µg	Loss%	µg	Loss%	µg	Loss%
Initial	500	-	500	-	500	-
1	499.50	0.1	492.28	1.54	445.40	10.92
2	499.00	0.2	486.88	2.62	437.37	12.53
4	488.92	2.22	453.22	9.36	428.22	14.36
8	475.63	4.87	398.45	20.31	411.77	17.65
12	448.36	10.33	388.64	22.27	407.36	18.53
24	345.88	30.52	387.93	22.41	397.26	20.55
48	338.98	32.20	318.86	36.23	356.47	28.71
96	332.77	33.45	280.87	43.83	297.79	40.44
192	312.95	37.41	280.88	43.94	286.69	54.66
$t_{0.5}$ (hours)	100.90		38.3		18.24	

Initial = 1 hour before storage

Table 7:Effect of different temperature degrees on the persistence of Lufenuron residues:-

Time periods(hrs)	25°C		35 °C		45°C	
	µg	Loss%	µg	Loss%	µg	Loss%
Initial	500	-	500	-	500	
1	49450	1.10	493.51	1.30	481.16	3.77
2	477.56	4.49	473.63	5.27	475.26	4.95
4	467.00	6.60	446.46	10.71	433.52	13.30
8	436.01	12.80	408.42	18.32	415.41	16.92
12	427.53	15.49	392.86	21.43	393.52	21.30
24	389.38	22.17	387.96	22.41	366.74	26.65
48	388.66	22.27	383.85	23.34	354.31	29.14
96	382.99	23.40	382.20	23.56	345.38	30.92
192	354.09	29.18	307.27	38.55	330.11	33.98
$t_{0.5}$ (hours)	60.44		53.86		40.82	

Initial = 1 hours before storage

Table 8: Effect of different temperature degrees on the persistence of Hexaflumuron

Time periods(hrs)	25°C		35°C		45°C	
	µg	Loss%	µg	Loss%	µg	Loss%
Initial	500	-	500	-	500	-
1	492.65	1.47	490.77	1.85	477.36	4.53
2	489.37	2.13	489.25	2.15	465.79	6.84
4	483.72	3.26	479.66	4.07	451.55	9.61
8	477.36	4.53	458.34	8.33	437.79	12.44
12	474.83	5.03	442.90	11.42	431.22	13.76
24	471.38	5.73	440.046	11.91	423.20	15.36
48	418.45	16.31	380.53	23.89	360.92	27.82
96	398.01	20.40	376.63	24.67	344.54	31.09
192	347.95	30.41	344.00	31.20	173.55	62.29
$t_{0.5}$ (hours)	78.77		78.51		33.04	

Initial = 1 hours before storage

Table 9: Effect of different temperature degrees on the persistence of Flufenoxuron

Time periods(hrs)	25°C		35°C		45°C	
	µg	Loss%	µg	Loss%	µg	Loss%
Initial	500	-	500	-	500	-
1	499.60	0.08	481.00	3.8	473.78	5.24
2	499.00	0.2	476.31	4.74	461.73	7.65
4	484.00	3.2	462.37	7.53	442.00	11.60
8	422.57	15.49	418.08	16.39	401.01	19.80
12	417.79	16.44	396.79	20.64	388.42	22.32
24	387.11	22.58	379.97	24.01	374.87	25.03
48	383.10	23.38	338.09	32.38	333.71	33.36
96	328.03	34.39	279.97	44.01	151.37	69.73
192	192.11	61.58	187.50	62.50	141.53	71.61
$t_{0.5}$ (hours)	42.42		40.82		34.63	

Initial = 1 hours before storage.

(4) Effect of storage in different temperatures on physical properties.

The data presented in tables (10-13) under different storage at 54°C ±2, 45°C ± 2, 40°C±2, 35°C ± 2 and 30°C ± 2 indicated that tested benzoyl urea pesticides passed successfully through (Density, Free Acidity, pH and(Emulsion and Dispersible Stability)

Comment:- Acidity and pH: These results are in agreed with finding Claylon (1943), EL-Attal and EL-Halofawy (1974) and EL-Attal and EL-sisi (1979), , Abd EL-All *et al.* (1993a, b).FAO/WHO Manual (2002); (2006)and FAO (2007). and the active ingredient and formulations are stable over acceptable range of pH values.

Density properties : the result had justified.

Emulsion and Dispersible Stability: the data presented in table 13Indicated that all benzoyl urea pesticides passed successfully through this test indifferent type of storage comply with specification WHO (1979), FAO/WHO (2002), (2006)and FAO (2007).

Table 10: Effect of storage on Free Acidity for used Formulated benzoyl urea at different periods.

Type of storage	Period	Atabron g /kg	Match g/ kg	Consult g /kg	Cascade g /kg
Initial	Initial	Neutral	Neutral	Neutral	Neutral
54°C	14 days	0.028	0.264	0.028	0.221
45 °C	6 week	0.40	2.400	0.06	0.45
40 °C	8 week	0.36	2.470	0.06	0.325
35 °C	12 week	0.21	2.479	0.29	0.323
30 °C	18 week	0.20	2.478	0.29	0.322

Initial = one hour before storage

Table 11: Effect of storage on Density for used Formulated benzoyl urea to different periods

Type of storage	Period	Atabron g /cm^3	Match g /cm^3	Consult g /cm^3	Cascade g /cm^3
Initial	Initial	1.03	0.9244	1.00	1.04
54°C	14 days	1.02	0.929	0.99	1.05
45 °C	6 week	1.00	0.924	0.98	1.03
40 °C	8 week	1.01	0.925	0.99	1.04
35 °C	12 week	1.01	0.924	1.00	1.05
30 °C	18 week	1.00	0.924	1.00	1.05

Initial = one hour before storage

Table 12: Effect of storage on pH for used formulated benzoyl urea at different periods

Type of storage	Period	Atabron	Match	Consult	Cascade
Initial	Initial	4.8	6.2	6.2	6.1
54°C	14 days	4.75	6.6	6.19	6.09
45 °C	6 week	4.79	6.58	6.19	6.09
40 °C	8 week	4.78	6.59	6.18	6.08
35 °C	12 week	4.77	6.57	6.17	6.07
30°C	18week	4.76	6.56	6.16	6.06

Initial = one hour before storage

Table 13: Effect of storage on Emulsion and Dispersible stability for used Formulated benzoyl urea at different periods

Type of storage	Period	Atabron	Match	Consult	Cascade
Initial	Initial	Acceptable Limit	Acceptable Limit	Acceptable Limit	Acceptable Limit
54°C	14 days	Acceptable Limit	Acceptable Limit	Acceptable Limit	Acceptable Limit
45 °C	6 week	Acceptable Limit	Acceptable Limit	Acceptable Limit	Acceptable Limit
40 °C	8week	Acceptable Limit	Acceptable Limit	Acceptable Limit	Acceptable Limit
35 °C	12 week	Acceptable Limit	Acceptable Limit	Acceptable Limit	Acceptable Limit
30 °C	18 week	Acceptable Limit	Acceptable Limit	Acceptable Limit	Acceptable Limit

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تأثير ظروف التخزين الحرارية على ثبات الخواص الكيميائية والطبيعية لمستحضرات مبيدات البنزويل يوريا

باسم السيد السيدمحمد البدرى و إبراهيم الدسوقى محمد محمد عطا الله

مركز البحوث الزراعية – المعمل المركزي للمبيدات – قسم بحوث تحليل المبيدات – الدقي – الجيزة

هذا البحث يلقى الضوء على دراسة ثبات الكلورفلوازيرون (أتابرون ٥%) ليفنيورن (ماتش ٥%) فليوفينوكسيورون (كاسكيد ١٠%) وهكسافليوميرون (كونصلت ١٠%) تحت تأثير بعض الظروف البيئية المناسبة المختلفة كالتخزين مثلا على درجات حرارة ٥٤ درجة مئوية، ٤٥ درجة مئوية، ٤٠ درجة مئوية، ٣٥ درجة مئوية، ٣٠ درجة مئوية لمدة ١٤ يوم، ٦ أسابيع، ٨ أسبوع ، ١٢ أسبوع، ١٨ أسبوع على التوالى.

أيضا هذه الدراسة تضع فى اعتبارنا مقدار التغير فى الخواص الطبيعية المناسبة فى الدراسة على المبيدات المختبرة قبل وبعد التخزين.

وقد أثبت التحليل الكيميائى بواسطة جهاز HPLC أن تحطم المواد الفعالة للمبيدات المختبرة تحت درجات التخزين المختلفة لا تتعدى الخطوط الارشاديه التى أقرت بهامنظمه الاغذيه والزراعهFAO، منظمة الصحة العالميةWHO .

أظهرت النتائج أيضا أن مبيد الكلورفلوازيرون (أتابرون) كان أكثر تأثر عند التعرض لدرجات حرارة مختلفة وزيادة مدة التعريض يليه مبيد الهكسافليوميرون (كونصلت) وكان مبيد ليفنيورن (ماتش) أكثر ثباتا عن المبيدات الاخرى تحت الدراسة عند درجات الحرارة المختلفة.

وفى النهاية كانت نتائج اختبارات الخواص الطبيعية للمبيدات المختبرة قبل وبعد التخزين فى الحدود الدوليه المسموح بها طبقا للخطوط الارشاديه لل FAO وWHO.