Degradation of imidacloprid insecticide on three formulations types

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Abstract: Three different formulations of imidacloprid insecticide suspension concentrate (SC), flowable concentrate for seed treatment (FS) and oil dispersion (OD) were stored at 0 ^{O}C for 7 days and stored under accelerated storage temperature at 54 ^{O}C for 14 days to study the degradation of imidacloprid active ingredient which determined by HPLC-DAD. The physical properties such as pH values, suspensibility, wet sieve test, pourability, dispersion stability, spontaneity of dispersion and persistent foam test before and after storage were also studied according to FAO specification 2013. Results showed that the active ingredient for SC formulation was not effected by accelerated storage temperature but FS and OD formulations were effected and the degradation rate increase with increasing long time of storage. Physical properties of three imidacloprid formulations types passed successfully through all storage periods at 0 ^{O}C for 7 and at 54 ^{O}C for 14 days except suspensibility test.

Keywords: Storage, imidacloprid, insecticide, HPLC-DAD

1.Introduction:

Imidacloprid is a systemic insecticide which acts as an insect neurotoxin and belongs to a class of chemicals called the neonicotinoids which act on the central nervous system of insects, with much lower toxicity to mammals. The chemical works by interfering with the transmission of stimuli in the insect nervous system. Specifically, it causes a blockage of the nicotinergic neuronal pathway. By blocking nicotinic acetylcholine receptors, imidacloprid prevents acetylcholine from transmitting impulses between nerves, resulting in the insect's paralysis and eventual death. It is effective on contact and via stomach action. Because imidacloprid binds much more strongly to insect neuron receptors than to mammal neuron receptors, this insecticide is more toxic to insects than to mammals. Imidacloprid is currently the most widely used insecticide in the world. Although it is now off patent. It is sold under many names for many uses; it can be applied by soil injection, tree injection, application to the skin of the plant, broadcast foliar, ground application as a granular or liquid formulation, or as a pesticide-coated seed treatment. Imidacloprid is widely used for pest control in agriculture. Other uses include application to foundations to prevent termite damage, pest control for gardens and turf, treatment of domestic pets to control fleas, protection of trees from boring insects, and in preservative treatment of some types of lumber products. Recent research suggests that widespread agricultural use of imidacloprid and other pesticides may be contributing to honey bee colony collapse disorder, the decline of honey bee colonies in Europe and North America observed since 2006. As a result, several countries have restricted use of imidacloprid and other neonicotinoids. In January 2013, the European Food Safety Authority stated that neonicotinoids pose an unacceptably high risk to bees, and that the industry-sponsored science upon which regulatory agencies' claims of safety have relied, may be flawed, or even deceptive.

2. Material and methods:

2.1. Chemicals

All organic solvent were HPLC grade and supplied by Fisher Scientific Ltd. Deionized water was prepared by a Milli-Q water purification system. Anhydrous calcium chloride and magnesium chloride were of analytical grade and purchased from Merck Ltd. Buffer solution pH 4 and pH7 purchased from Panreac Applichem Germany. Imidacloprid standard purchased from Sigma Aldrich. Aquaprimo 35% SC from Coromandel International Limited (China), nuprid 60% FS from Nufarm Limited (Austria) and confidor 20% OD from Bayer CropScience (Germany).

2.2. Apparatus

Dionex UltiMate 3000 UHPLC systems GmbH, Germany with a quadrennial pump, an autosampler, column oven, and photodiode array detector was employed. The column C18 (15 cm length, 4.6 mm inner diameter, and 5.0 µm particles). Imidacloprid determined using mobile phase consisting of acetonitrile/water (80:20 v:v) at flow rate 1.0 mL/min with detector wavelength at 210 nm.

2.3. Determination of active ingredient percentage

Imidacloprid determined before and after storage according to method (582/SC/M2/2, CIPAC Handbook K, p.74, 2003), (582/FS/M/3, CIPAC Handbook K, p.75, 2003) and (582/OD/M/2, CIPAC Handbook L, p.89, 2006) for formulations SC, FS and OD, respectively. Total imi-

dacloprid content shall be declared (g/kg) and when determined the content obtained shall not differ from that declared by more than (±5 %, ±2.5% and ±6%) of the declared content for formulations SC, FS and OD, respectively.

2.4. Stability at 54 ^{Ó}C

Storage at 54 ± 2 ^{Ó}C for 14 days according to **FAO** specification by the method (MT 46.3, CIPAC Handbook J, p.128, 2000) after storage at 54 ^{Ó}C the samples was determined and the average of active ingredient percentage must not be lower than 97%, 95% and 95% for imidacloprid formulations (SC, FS and OD, respectively) relative to the determined average content found before storage, the product shall continue to comply with physical properties of pesticides.

2.5. Stability at 0^{Ó}C

Storage at 0 ^{Ó}C for 7 days according to FAO specification by the method (MT 39.3, CIPAC Handbook J, p.126, 2000), the formulation shall continue to comply with the clauses for, suspensibility, Dispersion stability and wet sieve test.

2.6. Physical properties

Physical properties determined one day before storage and after different storage periods as the following Pourability (MT 148.1, CIPAC Handbook F, p.348, 1995), Wet sieve test (MT 185, CIPAC Handbook K, p.148, 2003), Persistent foam (MT 47.2, CIPAC Handbook F, p.152, 1995), Suspensibility (MT 184, CIPAC Handbook K, p.142, 2003), Spontaneity of dispersion (MT 160, CIPAC Handbook F, p.391, 1995), pH range (MT 75.3, CIPAC Handbook J, p.131, 2000) and Dispersion stability (MT 180, CIPAC Handbook H, p.310, 1998).

2.7. Standard solution preparation and standard curve plotting.

Stock standard solution of imidacloprid 100 ppm was prepared in acetonitrile. Different concentrations was then prepared from stock solution by dilution using mobile phase as diluents. The following diluted concentrations were prepared 0.5, 1.0, 5.0, 10, 25 and 50 ppm. Each of these solutions was injected into UHPLC and peak areas were recorded and plotted versus the concentration of the pesticide.

3. Results and discussion:

3.1. Stability at elevated temperature 54 ^{Ó}C for 14 days according to FAO specification (2013).

3.1.1. Degradation of imidacloprid formulations under accelerated storage temperature at 54 ^{Ó}C.

Table (1) showed the degradation of three formulations types SC, FS and OD of imidacloprid when stored for 14 days at 54 ^{Ó}C according to FAO specification (2013). The initial deposits one day before storage of imidacloprid percentage for formulations SC, FS and OD were 34.93% recording loss 0.20%, 59.86% recording loss 0.23% and 19.75% recording loss 1.25%, respectively. Degradation of imidacloprid increased with increasing long time of storage to reach 33.28% recording loss 4.91% and 58.45 recording loss 2.58% for SC, FS formulations, respectively after 14 days of storage and it was 18.78% recording loss 6.10% for OD formulation after 12 days of storage. According to tolerances of imidacloprid formulations SC, FS and OD (±5%, ±2.5% and ±6%, respectively) in **FAO specifications (2013),** the used pesticide SC formulation become conformity with this specification when stored for 14 days at 54^{Ó}C but FS and OD formulations become non conformity when stored for 14 and 12 days at 54 ^{Ó}C, respectively. Results are in line with Marei et al (1979), El-Sayed et al (1980), Hegazy et al (1982), Singh et al (1999), Shereen (2008), El-badry and kamal El-din. (2007), Kamal El-Din and Ola (2007), Ismail (2010), Kamal El-Din and Ramadan (2011), Mohamed (2013) and Mohamed et al (2016).

Table (1) degradation of imidacloprid formulations SC, FS and OD at 54 ^{Ó}C for 14 days of storage.

Storage period (days)	Imidacloprid Active ingredient 35% SC		Imidacloprid Active ingredient 60% FS		Imidacloprid Active ingredient 20% OD	
	%	Loss %	%	Loss %	%	Loss %
Initial	34.93	0.20	59.86	0.23	19.75	1.25
2	34.61	1.11	59.81	0.32	19.56	2.20
4	34.24	2.17	59.59	0.68	19.30	3.50
6	34.01	2.86	59.31	1.15	19.03	4.85
8	33.86	3.26	58.98	1.70	18.92	5.40
10	33.59	4.03	58.79	2.02	18.89	5.55
12	33.40	4.57	58.64	2.27	18.78	6.10
14	33.28	4.91	58.45	2.58	18.71	6.45

3.1.2. Effect of accelerated storage temperature on Limits of wet sieve test of imidacloprid formulations SC, FS and OD at 54 ^{Ó}C.

Data in table (2) illustrated the effect of accelerated storage temperature at 54 ^{Ó}C for 14 days on stability of wet sieve test of imidacloprid insecticide on different formulations types. The residue percentage of imidacloprid formulations SC, FS and OD before one day of storage were none for three types of formulations and at the end of experiment became 0.05%, 0.1% and 0.15% for SC, FS and OD formulations, respectively. According to FAO specification (2013) which reported that a maximum of 0.1%, 0.1% and 0.2% retained on a 75 µm test sieve for SC, FS and OD formulations of imidacloprid, respectively. The three formulations become conformity with

this specification. The results are in line with Smith (1976), Morpeth (1995), Mohamed (2009), Mohamed (2013) and Mohamed et al (2016).

Table (2) Thermal stability of imidacloprid formulations SC, FS and OD during storage at 54 ^{O}C according to FAO specifications (2013).

Storage period (days)	Pourability Residue %			Wet sieve test %			Persistent foam ml			pH	Suspensi-bility %	Spontaneity of dispersion %	Dispersion stability ml
	SC	FS	OD	SC	FS	OD	SC	FS	OD	FS	SC	SC	OD
Initial	1.0	0.5	2.0	None	None	None	3.5	6.0	11.0	6.79	100.0	100.0	None
2	1.0	0.5	2.0	None	None	None	3.5	6.0	11.0	6.73	100.0	100.0	None
4	1.0	1.0	2.0	None	None	0.05	3.5	6.0	10.5	6.68	99.00	100.0	None
6	1.3	1.0	2.5	None	0.03	0.05	3.0	5.5	10.3	6.61	98.00	99.00	None
8	1.3	1.0	2.5	0.03	0.05	0.10	3.0	5.5	10.0	5.93	97.50	99.00	None
10	1.5	1.5	3.0	0.05	0.05	0.10	2.0	5.0	9.5	5.88	96.00	98.00	0.1
12	1.7	1.5	3.0	0.05	0.10	0.15	2.0	4.0	8.0	5.83	95.00	97.00	0.1
14	1.7	1.5	3.5	0.05	0.10	0.15	1.5	4.0	7.5	5.76	94.50	97.00	0.2

3.1.3. Effect of accelerated storage temperature on persistent foam limits of imidacloprid formulations SC, FS and OD at 54 ^{O}C.

Data in table (2) showed limits of stability of persistent foam of imidacloprid formulations SC, FS and OD during storage for 14 days at 54 ^{O}C. Foam volume after 1 min of the test one day before storage were 3.5ml, 6ml and 11ml for imidacloprid formulations SC, FS and OD, respectively. By increasing time of storage the foam amount slightly decreased to the end of experiment to reach 1.5ml, 4ml and 7.5ml after 14 days storage for imidacloprid formulations SC, FS and OD, respectively. Results showed that three types of imidacloprid formulations SC, FS and OD becomes conformity with FAO specification (2013) which reported that maximum 40ml, 50ml and 50ml of foam after 1 min for imidacloprid formulations SC, FS and OD, respectively. The obtained results are agree with El-Attal (1979), Ismail (2010), Kamal El-Din and Ramadan (2011) and Mohamed et al (2016).

3.1.4. Effect of accelerated storage temperature on pourability limits of imidacloprid formulations SC, FS and OD at 54 ^{O}C.

Data in table (2) showed residue percent of pourability test of imidacloprid SC, FS and OD during storage for 14 days at 54 ^{O}C. The residue percentage of imidacloprid formulations SC, FS and OD before one day of storage were 1%, 0.5% and 2%, respectively, and it increasing by long time of storage to reach 1.7%, 1.5% and 3.5% after 14 days of storage at 54 ^{O}C for imidacloprid formulations SC, FS and OD, respectively. This test pass successfully according to FAO specification (2013) which reported that maximum residue are 4%, 4% and 5% for imidacloprid formulations SC, FS and OD, respectively.

3.1.5. Effect of accelerated storage temperature on suspensibility percentage of imidacloprid SC formulations at 54 ^{O}C.

Data in table (2) showed effect of accelerated storage temperature at 54 ^{O}C for 14 days on suspensibility of imidacloprid SC formulation during storage. Suspensibility percentage of imidacloprid one day before storage was 100% and it started decease by increasing long time of storage to reach 94.5% after 14 days of storage at 54 ^{O}C. According to FAO (2013) which reported that A minimum of 95% of the imidacloprid shall be in suspension after 30 min. The imidacloprid SC formulation become non conformity with this specification when stored after 12 days at 54^{O}C. Results are agree with Duraipandian and Regupathy (1989) and Morpth (1995), Kamal El-Din (2007), El-badry and Mohsin (2007) and Kamal El-Din and Ramadan (2009), Mohamed (2009) and mohamed (2013).

3.1.6. Effect of accelerated storage temperature on spontaneity of dispersion percentage of imidacloprid SC formulations at 54 ^{O}C.

Data in table (2) showed the effect of accelerated storage temperature at 54 ^{O}C for 14 days on spontaneity of dispersion percentage of imidacloprid SC formulation during storage. Spontaneity of dispersion percentage of imidacloprid one day before storage after 5 min was 100%

and it started decease by increasing long time of storage to reach 97% at end of the experiment after 14 days of storage at 54 ^{O}C. According to FAO (2013) which reported that a minimum of 90% of imidacloprid shall be in suspension after 5 min. The imidacloprid SC formulation become conformity with this specification when stored for 14 days at 54 ^{O}C.

3.1.7. Effect of accelerated storage temperature on pH value of imidacloprid FS formulation at 54 ^{O}C.

Data in table (2) indicated the effect of storage temperature at 54 ^{O}C for 14 days on pH value for imidacloprid insecticide FS formulation. The pH value was 6.79 one day before storage and reached to 5.76 after 14 days from storage. According to FAO specifications (2013) which reported that pH for imidacloprid FS formulation ranged from 5 to 9. The FS formulation of imidacloprid become conformity with this specification when stored for 14 days at 54 ^{O}C. Results agree with Hirahara et al (1997), Abou–Donia et al (1985), Barakat et al (1999), El-badry and Mohsin (2007), Kamal El-Din and Ola (2007), Ola and Sheren (2007), Mohamed (2009), Mohamed (2013) and Mohamed et al (2016).

3.1.8. Effect of accelerated storage temperature on limits of dispersion stability of imidacloprid OD formulations at 54 ^{O}C for 14 days.

Data presented in the Table (2) indicate that imidacloprid OD formulation passed successfully through dispersion stability test during 14 days of storage at 54 ^{O}C while, the cream volume or free oil after half hour one day before storage was none and it increased to 0.2 mL at the end of experiment after 14 days of storage. From these results imidacloprid OD formulation become conformity with FAO specifications (2013) which reported that time after dilution were zero and 0.5 hr, limits of stability must be dispersion complete and maximum of cream volume 0.5ml , respectively. These results are in line with El-badry and Kamal El-Din (2007), El-badry and Mohsin (2007), Ola and Sheren (2007), Shereen (2008), Mohamed (2009), Mohamed (2013) and Mohamed et al (2016).

3.2. Stability at zero ^{O}C for 7 days according to FAO specification (2013).

3.2.1. Effect of storage on Limits of wet sieve test of imidacloprid formulations SC, FS and OD at ^{O}C.

Data in table (3) showed that limits of wet sieve test of imidacloprid pass successfully in this test. Where, the retained percentage of imidacloprid formulations SC, FS and OD one day before storage until the end of experiment after 7 days of storage at 0^{O}C were none and results pass successfully according to FAO specification (2013) which reported that a maximum of 0.1% , 0.1% and 0.2% retained on a 75 µm test sieve for imidacloprid SC, FS and OD formulations, respectively. These results are in line with Smith (1976), Morpeth (1995), Mohamed (2009) and Mohamed (2013).

Table (3): Cold stability of imidacloprid formulations SC, FS and OD during storage at 0 ^{O}C according to FAO specifications (2013).

Storage period (days)	Wet sieve test %			Suspensibility %	Dispersion stability ml
	SC	FS	OD	SC	OD
Initial	None	None	None	100.0	None
1	None	None	None	100.0	None
2	None	None	None	100.0	None
3	None	None	None	100.0	None
4	None	None	None	99.00	None
5	None	None	None	99.00	None
6	None	None	None	99.00	None
7	None	None	None	99.00	None

3.2.2. Effect of storage on suspensibility percentage of imidacloprid SC formulations at 0^{O}C.

Data in table (3) showed that imidacloprid SC formulation pass successfully through suspensibility test. Suspensibility percentage of imidacloprid one day before storage was 100% and it still stable to reach 99% at end of the experiment after 7 days of storage at 0^{O}C. According to FAO (2013) which reported that A minimum of 95% of the imidacloprid shall be in suspension after 30 min. The imidacloprid SC formulation become conformity with this specification when stored for 7 days at 0^{O}C. Results are agree with Duraipandian and Regupathy (1989) and Morpth (1995), Kamal El-Din (2007), El-badry and Mohsin (2007) and Mohamed et al (2016).

3.2.3. Effect of storage on limits of dispersion stability of imidacloprid OD formulations at ^{O}C.

Data presented in the Table (3) indicate that imidacloprid OD formulation passed successfully through dispersion stability test during 7 days of storage at 0^{O}C. The volume of cream or free oil after half hour one day before storage to the end of experiment was none. From the results imidacloprid OD formulation become conformity with FAO specifications (2013) which reported that time after dilution were zero and 0.5 hr, limits of stability must be dispersion complete and maximum of cream volume 0.5ml , respectively. These results are in line with El-badry and Kamal El-Din (2007), El-badry and Mohsin (2007) , Kamal El-Din and Ola (2007), and Ola and Shereen (2007).

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الملخص العربي

تحطم مبيد الايميداكلوبراید فی ثلاثة مستحضرات

محمد فتحى عبدالرحمن رمضان

مركز البحوث الزراعية – المعمل المركزى للمبيدات – قسم بحوث تحليل المبيدات – الدقى – الجيزة

تهدف هذة الدراسة الى القاء الضوء عن تأثير حرارة التخزين المختلفة طبقا لمواصفات FAO لسنة (٢٠١٣) على ثبات الخواص الكيميائية والطبيعية لمركب الايميداكلوبراید في ثلاثة مستحضرات وهى مركز معلق لمبيد أكوابريمو ٣٥% و مركز انسيابي لمعاملة البذور لمبيد نوبراید ٦٠% و منتشر زيتي لمبيد كونفيدور ٢٠% على درجة حرارة ٥٤ م° لمدة ١٤ يوم درجة الصفر المئوي لمدة ٧ أيام لدراسة ثبات نسبة المادة الفعالة من الايميداكلوبراید التي تم تقديرها بواسطة جهاز التحليل الكروماتوجرافى السائل HPLC ودراسة الخواص الطبيعية مثل اختبار التعلق ومدى pH واختبار النعومة المبتلة واختبار ثبات الانتشار واختبار ثبات الرغاوى.

تبين من النتائج الأتي :

- لم تتأثر نسبة المادة الفعالة لمبيد الايميداكلوبراید بحرارة التخزين لمستحضر مركز معلق ولكنها تأثرت لمستحضر مركز انسيابي لمعاملة البذور بعد ١٢ يوم و مستحضر منتشر زيتي بعد ١٠ يوم من التخزين على درجة حرارة ٥٤ م° .
- الخواص الطبيعية للثلاثة مستحضرات بعد التخزين لمدة ١٤ يوم على درجة ٥٤ م° جميعها لم تتأثر بحرارة التخزين ماعدا اختبار التعلق لمستحضر مركز معلق الذي تأثر بحرارة التخزين بعد ١٢ يوم.
- جميع اختبارات الخواص الطبيعية مرت بنجاح عند تخزينها على درجة الصفر المئوي لمدة ٧ أيام.