


# Influence of certain environmental conditions on degradation of several pendimethalin capsule suspensions now utilized in Egypt



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## ABSTRACT

Three commercial pendimethalin capsule suspension (CS) formulations were obtained from the Egyptian market, produced by three distinct companies to examine the impact of storage at room temperature and sunlight exposure for six months, as well as storage at 54 ± 2 °C for 70 days, on the stability of pendimethalin. Furthermore, GC-MS was employed to identify some breakdown products following exposure to sunlight. According to the obtained results, pendimethalin was stable after storage at room temperature and 54 ± 2 °C for 14 days, and the degradation rate was not affected even with increasing the storage period for all sources. Pendimethalin was less stable after being exposed to sunlight than storage at 54 ± 2 °C, and there was no difference in the degradation rate for all pendimethalin formulations. Pendimethalin photodecomposes by oxidative dealkylation and nitro reduction. Four degradation products were found using GC-MS analysis of samples exposed to the sunlight as follows: *N*-(1-ethylpropyl)-3-methyl-2,6-dinitroaniline, *N*-propyl-3,4-dimethyl-2,6-dinitroaniline, 4,5-dimethyl-3-nitro-*N*-(pentan-3-yl) benzene-1,2-diamine and 2,6-dinitro-3,4-dimethylaniline.

**Keywords:** Pendimethalin, Sunlight, Degradation, Degradation products, GC-MS.

## INTRODUCTION

Pendimethalin (*N*-(1-ethylpropyl)-2,6-dinitro-3,4-xylidine) is a dinitroaniline herbicide known for its significant activity against grasses and annual broadleaf weeds. It is commonly used to control weeds in various agricultural systems (soybeans, cotton, peanuts, potatoes, and maize), and many fruit trees (MacBean, 2011). Pendimethalin degradation exhibited an upward trend with increasing soil temperatures of 10, 20, 35, and 30 °C. The pace was equivalent to 75% and 100% field capacity, although it was comparatively slower at 50%. The impact of soil type was minimal under identical temperature and soil moisture conditions (Zimdahl *et al.*, 1984). Many studies have found that pendimethalin decomposes in several ways, including volatilization, photodecomposition, and biodegradation (WHO, 2003; Moza *et al.*, 1992, and Miller *et al.*, 1996). The presence of titanium dioxide significantly accelerates the degradation rate of pendimethalin through photolysis. A 500 ppm solution of pendimethalin exposed to UV light irradiation experienced approximately 50% decomposition within 7 hours when TiO<sub>2</sub> was present. In contrast, only 8% degradation was observed in its absence. This distinction demonstrates photolysis's effectiveness in facilitating the compound's decomposition (Pandit *et al.*, 1995). Pendimethalin's molecular structure confers its stability. It exhibits strong adsorption to soil while presenting challenges in desorption. The half-life of pendimethalin in soil is 90 days. (Mohan *et al.*, 2007).

Using Design Expert software, we conducted an ANOVA test. The results showed that four variables—irradiation period, pH, beginning herbicide concentrations, and photocatalyst dosage—had a significant impact on the degradation of pendimethalin. Among these variables, photocatalyst dosage emerged as the most influential factor in determining the removal efficiency of Pendimethalin. The optimal condition for accelerated decomposition of Pendimethalin is a pH of 10, which is highly alkaline (Ebrahimpour *et al.*, 2022). Pendimethalin photodegradation has been extensively studied in various conditions. In solution and on soil surfaces, pendimethalin undergoes rapid degradation through reductive cyclization, oxidative dealkylation, and nitro reduction (Dureja and Walia, 1989). The physical stability of high content pendimethalin capsule suspensions is influenced by various factors; emulsifier content, wall material composition, curing temperature, and suspending agents significantly affect microcapsule characteristics and stability. Better encapsulation efficiency, less water separation, and more stable storage can result from optimizing these parameters. The encapsulation process and the active ingredient's successful incorporation into the microcapsules can be characterized using techniques like infrared spectroscopy (Beixing *et al.*, 2013). The biodegradation of

pendimethalin resulted in the formation of two degradation products: 2-dimethyl-3,5-dinitro-4-N(buta-1,3-dien-2-yl)-dinitrobenzenamine-N-oxide and 1,2-dimethyl-3,5-dinitro-4-N(prop-1-en-2-yl)-dinitrobenzenamine-N-oxide (Han *et al.*, 2019). The photodegradation of pendimethalin was investigated across a range of solvents, excluding methanol and ethanol. As a result, two colored compounds were obtained from the irradiation n-hexane and aqueous solutions of pendimethalin. The substance has been designated as N-propyl-3,4-dimethyl-2,6-dinitroaniline. The latter, together with the unidentified metabolites, has been detected in soil samples after exposure to sunlight for 7, 15, and 30 days (Halder *et al.*, 1989).

This study examines and analyses the stability and degradation rate of three commercially available pendimethalin capsule suspension (CS) formulations obtained from the Egyptian market, each manufactured by a different company. It will calculate the shelf lives for the formulations under investigation. The formulations will be stored at varying temperatures ( $54 \pm 2$  °C and room temperature) and exposed to sunlight at various intervals. Specific breakdowns by-products based on sunlight exposure will also be identified using GC-MS.

## MATERIAL AND METHODS

### Chemicals:

The pendimethalin analytical standard, which has a CAS RN of [40487–42–1] and a purity of 98%, was acquired from LGC Standards (Dr. Ehrenstorfer). The pendimethalin commercial formulations with concentrations of 45%, 45.5%, and 45.6% (w/v) CS were acquired from three distinct companies in Egypt, identified as sources I, II, and III. The HPLC-grade methanol from Merck was utilized. **Fig. 1** illustrates the chemical structure of pendimethalin.

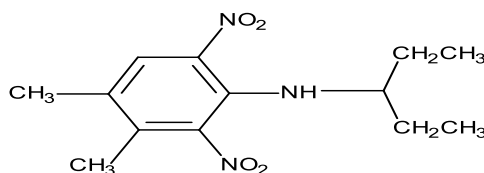


Fig. 1. Chemical structure of pendimethalin

### Storage methods:

Pendimethalin capsule suspension formulations were placed in approximately 50 ml bottles. The bottles were categorized into three groups and exposed to different procedures as outlined below: The initial group was subjected to storage at  $54 \pm 2$  °C for 70 days, while the second and third groups were subjected to storage under ambient temperature and sunlight for six months. Samples from the first experimental group were collected at intervals of 0, 14, 28, 42, 56, and 70 days, whereas samples from the second and third experimental groups were collected at intervals of 0, 1, 2, 3, 4, 5, and 6 months (CIPAC MT 46, 1995).

### Preparation of standard:

Weigh 10 mg of pendimethalin analytical standard into a 25 ml volumetric flask. Using methanol, dissolve the substance and fill the flask to its final volume.

### Preparation of samples:

Weigh an appropriate amount of pendimethalin formulations at concentrations of 45%, 45.5%, and 45.6% (w/v) to match 10 mg of the pendimethalin analytical standard. Transfer the weighed samples into a 25 ml volumetric flask and mix them slowly with methanol. Finally, add enough methanol to get the desired volume.

### Measurements:

#### 1. GC Determination:

In accordance with (Engebretson *et al.*, 2001), the procedure made use of an Agilent 7890B gas chromatograph coupled with an autosampler 7693, which was fitted with a Flame Ionization Detector (FID) functioning at 250 °C, and a capillary column HP-50+ (30 m x 0.53 mm I.D., 1 µm film thickness). The carrier gas was nitrogen, which was continuously pumped at a rate of 8 ml/min. After holding at 200 °C for 1 minute, the oven temperature was increased to 260 °C with a 20 °C increment per minute ramp. We used splitless mode and set the injector temperature to 250 °C. A volume of one microliter was injected. Under these circumstances, pendimethalin had a typical retention time of 4.073 minutes, and the standard for pendimethalin is depicted in **Fig. 2**. Excellent linearity was achieved within the range of 10-400 ng µl<sup>-1</sup> for the active ingredient, exhibiting a correlation coefficient of 0.99990, as illustrated in **Fig. 3**.

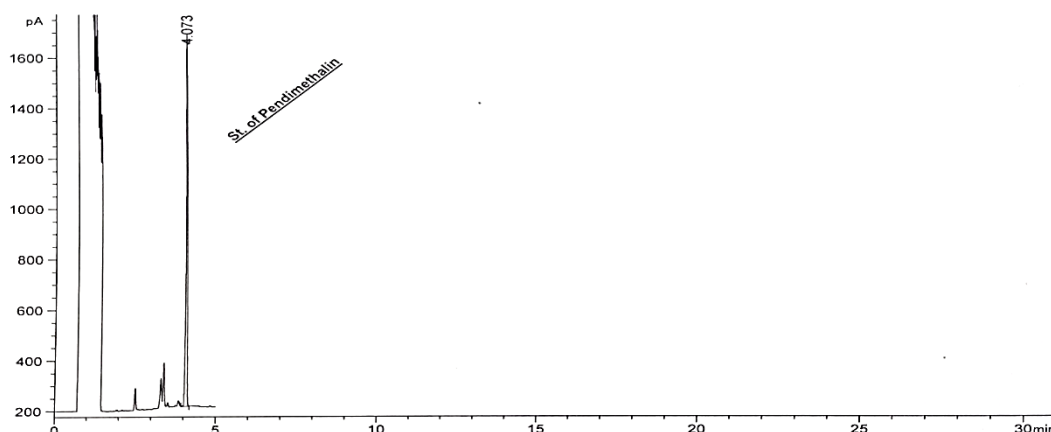


Fig. 2. Chromatogram of pendimethalin Standard

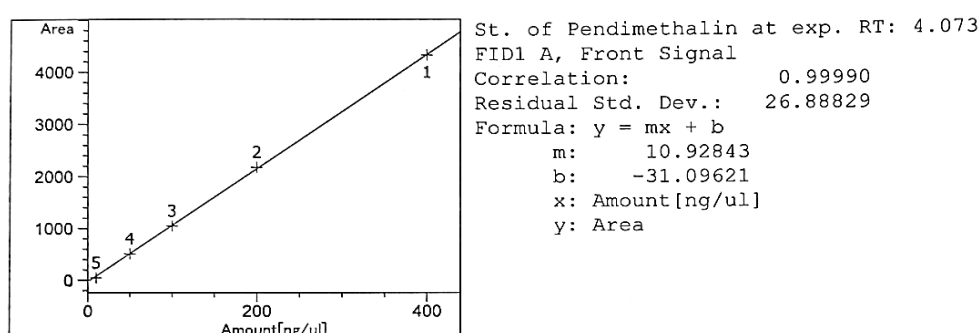


Fig. 3. Standard calibration curve of pendimethalin using GC

## 2. GC-MS Conditions:

An Agilent 7890B gas chromatograph equipped with a 5977 A MSD Agilent mass spectrometric detector was utilized for the analysis of samples after sunlight exposure. The HP-5MS fused silica capillary column (30 m x 0.25 mm x 0.25  $\mu$ m film thickness) and direct capillary interface were installed in the chromatograph. The procedures described in (Wang and Wu, 2020) were adhered to in the analysis. Helium was the carrier gas at a roughly 1.0 ml/min flow rate in pulsed split mode. The volume of the injection was 1  $\mu$ l. The temperature program for the GC consisted of a holding period at 50°C for 0.5 minutes, increased at a rate of 10°C per minute until reaching 190°C, maintained for 1 minute. A subsequent ramping rate of 10°C per minute was applied until reaching 300°C, which was held for 2 minutes. The total run time for this program was 29.5 minutes. The temperature of the injector was adjusted to 280 °C. The mass spectra were discovered utilizing the Wiley mass spectral database Library.

## 3. Kinetic study:

As per the FAO tolerances, pendimethalin should be within  $\pm 5\%$  of the declared amount. Therefore, pendimethalin should not be less than 95% of the original amount after storage. (El-Dars *et al.*, 2023 and Mansour *et al.*, 2024) determined the degradation rate and shelf life following first-order kinetics. We used the first-order rate equation to find the degradation rate constants and shelf lives:

$$C_t = C_0 e^{-kt}$$

$C_t$  is the pesticide concentration at time  $t$ ,  $C_0$  is the concentration at the start, and  $k$  is the degradation rate constant in (days<sup>-1</sup>). We calculated the  $k$  value by plotting the logarithm of concentration against time and using the  $k$  value to calculate the shelf life ( $t_{95}$ ) according to the following equation:

$$t_{95} = \ln(0.95)/k$$

## RESULTS

### 1. Effect of storage on the stability of pendimethalin CS content:

Table (1) shows the impact of accelerated storage at  $54 \pm 2^\circ\text{C}$  for 70 days on the stability of three commercial pendimethalin CS formulations. Stored at  $54 \pm 2^\circ\text{C}$  for 14 days, pendimethalin remained unchanged for all sources under investigation. However, as the storage period increased, there was a slight rise in the degradation rate for all sources, resulting in loss percentages of 4.2, 2.85, and 4.22%. The shelf life for sources I, II, and III were 85.5, 102.6, and 85.5 days, respectively. Pendimethalin exhibited lower stability when exposed

to sunlight compared to storage at  $54 \pm 2$  °C for 70 days. The data presented in **Table (2)** illustrates the impact of six months of sunlight exposure on the stability of pendimethalin CS. The results revealed that the percentages of loss after six months were 8.58, 8.03, and 7.72%. The shelf life for sources I, II, and III were 109.93, 87.44, and 112.34 days, respectively. Pendimethalin exhibited exceptional stability and remained unaffected by storage at ambient temperature. **Table (3)** examined the impact of a 6-month storage period on the stability of pendimethalin CS at ambient temperature. The results indicated that the percentages of loss after six months were 1.74, 1.78, and 1.8%. In addition, sources I, II, and III had estimated shelf lives of 496.45, 513, and 530.69 days, respectively.

**Table 1.** Thermal stability of pendimethalin CS at  $54 \pm 2$  °C.

Storage Periods (Days)	I		II		III	
	Pendimethalin concentration (wt/vol.) 45.5%	Loss %	Pendimethalin concentration (wt/vol.) 45%	Loss %	Pendimethalin concentration (wt/vol.) 45.6%	Loss %
0	45.46*	0	44.96	0	45.47	0
14	45.23	0.51	44.85	0.24	44.91	1.23
28	44.54	2.02	44.79	0.38	44.75	1.58
42	44.25	2.66	44.15	1.8	44.51	2.11
56	43.87	3.5	43.78	2.62	43.84	3.58
70	43.55	4.2	43.68	2.85	43.55	4.22
t <sub>95</sub> (days)	85.5**		102.6**		85.5**	

\* All analyses are conducted simultaneously to prevent analytical errors.

\* Each value represents an average of three replicates: FAO tolerance  $\pm 5\%$  of the declared content of Pendimethalin.

\*\* The duration needed to get 95% of the initial Pendimethalin content.

**Table 2.** Sunlight exposure of pendimethalin CS.

Storage Periods (months)	I		II		III	
	Pendimethalin concentration (wt/vol.) 45.5%	Loss %	Pendimethalin concentration (wt/vol.) 45%	Loss %	Pendimethalin concentration (wt/vol.) 45.6%	Loss %
0	45.46*	0	44.96	0	45.47	0
1	44.68	1.72	44.48	1.07	44.93	1.19
2	44.24	2.68	43.85	2.47	44.38	2.4
3	44.12	2.95	42.95	4.47	43.89	3.47
4	43.05	5.3	42.11	6.34	43.13	5.15
5	42.62	6.25	41.75	7.14	42.43	6.69
6	41.56	8.58	41.35	8.03	41.96	7.72
t <sub>95</sub> (days)	109.93**		87.44*		112.34*	

\* Each value represents an average of three replicates: FAO tolerance  $\pm 5\%$  of the declared content of Pendimethalin.

\*\* The duration needed to get 95% of the initial Pendimethalin content.

**Table 3.** The impact of storage surroundings on the stability of pendimethalin CS at ambient temperature.

Storage Periods (months)	I		II		III	
	Pendimethalin concentration (wt/vol.) 45.5%	Loss %	Pendimethalin concentration (wt/vol.) 45%	Loss %	Pendimethalin concentration (wt/vol.) 45.6%	Loss %
0	45.46*	0	44.96	0	45.47	0
1	45.35	0.24	44.9	0.13	45.25	0.48
2	45.25	0.46	44.85	0.24	45.01	1.01
3	45.01	0.99	44.65	0.69	44.9	1.25
4	44.91	1.21	44.55	0.91	44.82	1.43
5	44.75	1.56	44.35	1.35	44.75	1.58
6	44.67	1.74	44.16	1.78	44.65	1.8
t <sub>95</sub> (days)	496.45*		513*		530.69*	

\* Each value represents an average of three replicates: FAO tolerance  $\pm 5\%$  of the declared content of Pendimethalin

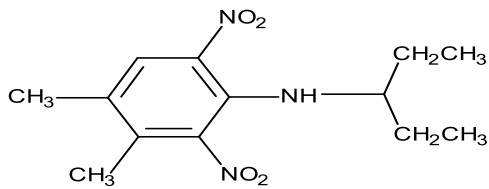
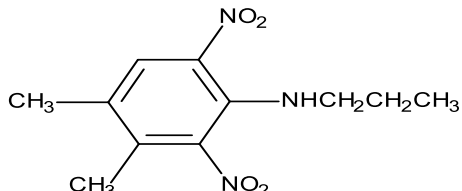
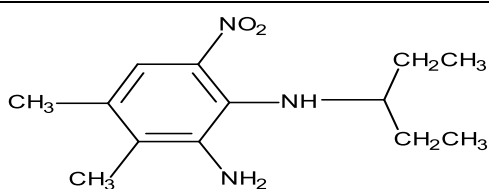
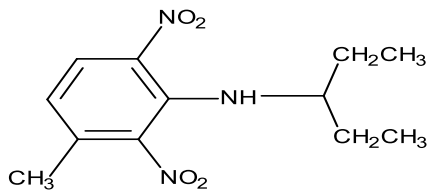
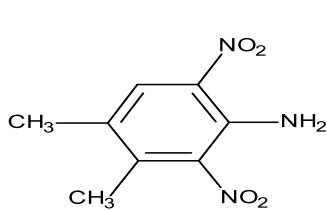
\*\* The duration needed to get 95% of the initial Pendimethalin content.

## 2. Identification of the photodegradation products of pendimethalin by GC-MS:

The degradation products of pendimethalin CS formulations were identified by GC-MS analysis of samples taken before and after exposure to sunlight. The molecular ions of pendimethalin, N-(1-ethylpropyl)-3-methyl-2,6-dinitroaniline, N-propyl-3,4-dimethyl-2,6-dinitroaniline, 4,5-dimethyl-3-nitro-N2-(pentan-3-yl)benzene-1,2-

diamine, and 2,6-dinitro-3,4-dimethylaniline were identified at  $m/z$  281, 266, 253, 251, and 211, respectively, however, **Table (4)** indicated that, identification of the degradation products of pendimethalin by GC-MS.

**Table 4.** Identification of the photodegradation products of pendimethalin by GC-MS

Structure	RT (min)	Characteristic ions ( $m/z$ )
	19.64	$[M]^+ = 281, 252, 236, 208, 194, 162, 146, 119$ pendimethalin
	21.85	$[M]^+ = 253, 238, 207, 191, 133, 117$ N-propyl-3,4-dimethyl-2,6-dinitroaniline
	19.15	$[M]^+ = 251, 221, 207, 191, 162, 117$ 4,5-dimethyl-3-nitro-N <sup>2</sup> -(pentan-3-yl)benzene-1,2-diamine
	21.53	$[M]^+ = 266, 252, 237, 207, 191, 162, 146, 117$ N-(1-ethylpropyl)-3-methyl-2,6-dinitroaniline
	20.74	$[M]^+ = 211, 105, 91, 77$ 2,6-dinitro-3,4-dimethylaniline

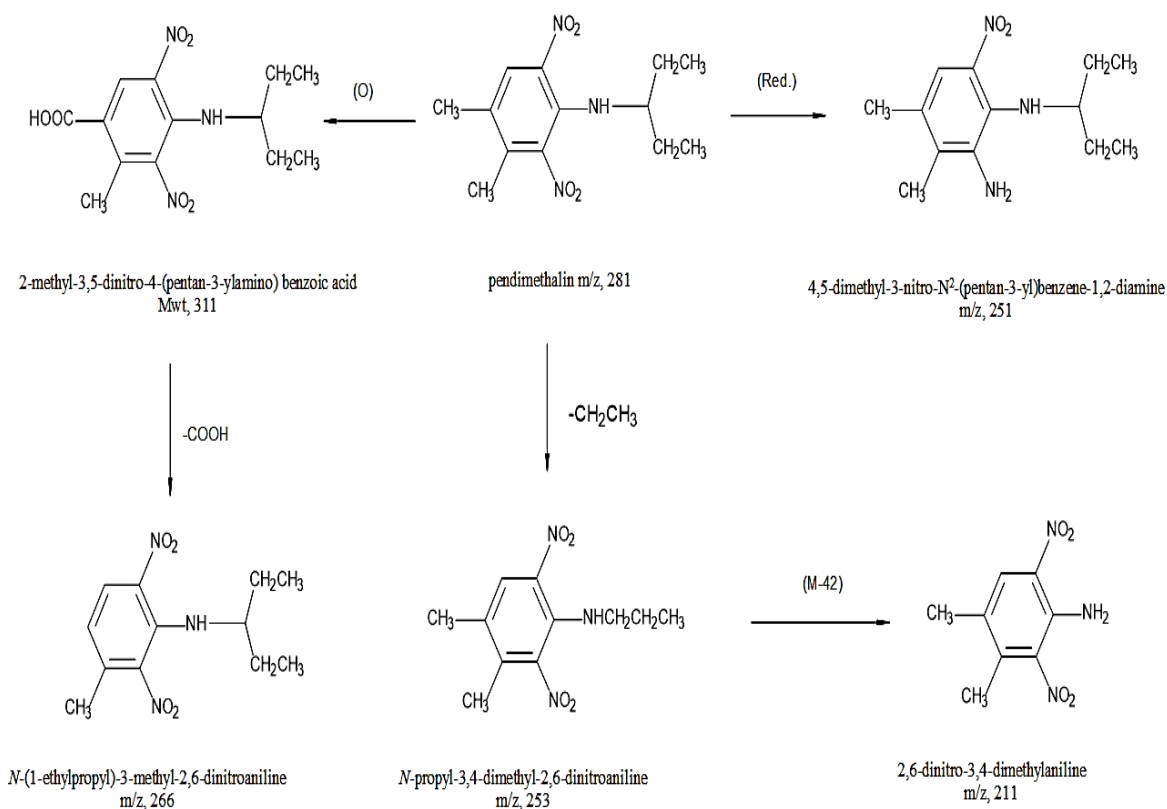
## DISCUSSION

The findings mentioned above from storage at  $54 \pm 2$  °C and sunlight exposure also at room temperature unambiguously show no notable disparity in the degradation rate of the three pendimethalin CS formulations. The results indicated that pendimethalin remained thermally stable even after being stored for an extended period at  $54 \pm 2$  °C. Also, the results unequivocally demonstrated that the degradation rate of the three pendimethalin CS formulations under investigation was significantly and noticeably increased when exposed to sunlight, compared to accelerated storage at  $54 \pm 2$  °C and storage at room temperature. Several aspects, including the manufacturing method, components, adjuvants, chemical composition, environmental factors, and light and air exposure, influence the quality of pendimethalin CS formulations. Our findings are in harmony with (Hennecke *et al.*, 2020) who reported that under certain experimental conditions, pendimethalin exhibited a notable degradation rate when exposed to simulated sunlight, surpassing the degradation rate observed in the dark studies. Also, this study demonstrated that pendimethalin is sensitive to photolysis, and when exposed to simulated sunlight, its degradation half-life in the surface water is much shorter than that of the dark control. During a 15-day photolysis experiment, pendimethalin was hydrolytically stable but vulnerable to aqueous photolysis with half-lives ranging from 1.5 to 5 days when subjected to continuous exposure to light (EFSA 2016). Pendimethalin is characterized by high stability during storage and gradual degradation under light exposure;  $DT_{50}$  in water within 21 days (MacBean, 2011). Pendimethalin is stable due

to its molecular structure and properties. The presence of two nitro groups (-NO<sub>2</sub>) and a substituted aniline ring in its molecular structure sometimes gives it stability. Pendimethalin's high melting point and low vapor pressure contribute to its thermal stability, making it less susceptible to degradation at elevated temperatures.

Upon analysis of the chemical composition of pendimethalin, it is evident that it possesses two nitro groups, two methyl groups, and two ethyl groups. These groups are highly susceptible to breakdown through various mechanisms. The oxidation of one of the methyl groups in pendimethalin can readily result in the formation of 2-methyl-3,5-dinitro-4-(pentan-3-ylamino) benzoic acid m.wt 311. The acid product can then lose the carboxylic group to create *N*-(1-ethylpropyl)-3-methyl-2,6-dinitroaniline m/z 266. Second, the 4,5-dimethyl-3-nitro-*N*<sup>2</sup>-(pentan-3-yl) benzene-1,2-diamine m/z 251 is obtained by reducing one of the nitro groups to an amine group (EFSA 2016 and Vighi *et al.*, 2016). Furthermore, pendimethalin can readily undergo photolysis and lose its alkyl chain (-CH<sub>2</sub>CH<sub>3</sub>) to produce *N*-propyl-3,4-dimethyl-2,6-dinitroaniline m/z 253. This is followed by losing the alkyl chain (M<sup>+</sup>-CH-CH<sub>2</sub>-CH<sub>3</sub>) to generate 2,6-dinitro-3,4-dimethylaniline m/z 211 (Halder *et al.*, 1989).

Based on the provided information, the various reactions observed can be elucidated by referring to **Fig. 4**. This figure illustrates possible degradation pathways of pendimethalin.



**Fig. 4.** The possible degradation pathways of pendimethalin after exposure to sunlight

## CONCLUSION

Pendimethalin exhibits considerable thermal stability and maintains excellent stability throughout storage at various temperatures. Exposure to sunlight causes a progressive increase in its degradation rate, leading to several degradation products. Several variables influence the quality of pendimethalin, including the production method, origins of technical components, composition of adjuvants, and extended storage periods in sun-exposed open areas. To minimize degradation, the formulation should be stored in opaque containers or protected from direct sunlight. Using herbicides past their expiration date may result in reduced germination rates compared to non-expired ones. The rapid degradation of pendimethalin can lead to the expiration of the active ingredient. Furthermore, expired herbicides can pollute soil and water, harm treated crops through phytotoxicity, or leave harmful residues in food or the environment.

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**Conflict of Interest:** The authors declare no conflict of interest

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## تأثير بعض العوامل البيئية علي تحليل العديد من معلقات كبسولات البنديميثالين المستخدمة حاليا في مصر

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تم جمع ثلاثة مستحضرات تجارية (CS) من مبيد البنديميثالين من السوق المصرية ( مصنعة بواسطة ثلاث شركات مختلفة) لفحص تأثير التخزين في درجة حرارة الغرفة والتعرض لأشعة الشمس لمدة ستة أشهر، وكذلك تخزينها عند  $2 \pm 54$  م لمدة 70 يومًا، على ثبات البنديميثالين، بالإضافة الى استخدام جهاز GC-MS لتحديد بعض نواتج التحطم بعد التعرض لأشعة الشمس. وفقًا للنتائج، كان البنديميثالين مستقرًا بعد التخزين في درجة حرارة الغرفة و  $2 \pm 54$  درجة مئوية لمدة 14 يومًا، ولم يتأثر معدل الانهيار حتى مع زيادة فترة التخزين لجميع المصادر الثلاثة. كان البنديميثالين أقل استقرارًا بعد التعرض لأشعة الشمس من التخزين عند  $2 \pm 54$  م ولم يكن هناك فرق في معدل الانهيار لجميع مستحضرات البنديميثالين. ينهار البنديميثالين ضوئيًا عن طريق نزع الألكيل المؤكسد واختزال النيترو. تم التعرف على أربعة نواتج تكسير باستخدام جهاز GC-MS للعينات المعرضة لأشعة الشمس على النحو التالي:

أ. *N*-(1-ethylpropyl)-3-methyl-2,6-dinitroaniline

ب. *N*-propyl-3,4-dimethyl-2,6-dinitroaniline

ج. 4,5-dimethyl-3-nitro-*N*<sup>2</sup>-(pentan-3-yl)benzene-1,2-diamine

د. 2,6-dinitro-3,4-dimethylalaniline

الكلمات المفتاحية: بنديميثالين ، أشعة الشمس، الانهيار ، نواتج التحطم، GC-MS