Journal of Materials and Environmental Sciences
ISSN : 2028-2508
CODEN : JMESCN

J. Mater. Environ. Sci., 2018, Volume 9, Issue 9, Page 2660-2668

http://www.jmaterenvironsci.com

Screening of factors influencing the efficacy of *Pistacia lentiscus* (L.) essential oil from Tunisia for the control of *Tribolium castaneum*

Olfa Bachrouch[1]*, Abdelkarim Aydi[3,5], Iness Jabri Karoui[3], Soumaya Haouel Hamdi[4], Jazia Sriti[2], Nithal salem[2], Kamel msaada[2], Majdi Hamami[2], Emna Boushih[4], Manef Abderraba[3], Ferid Limam[2], Jouda Médiouni Ben Jemâa[4]

[1] *Laboratoire de Protection des Végétaux, Institut National de la Recherche Agronomique de Tunisie (INRAT), Rue Hedi Karray, 2049 Ariana, Tunis, Université de Carthage, Tunisia*
[2] *Laboratoire des Substances Bioactives, Centre de Biotechnologie à la Technopole de Borj Cedria, BP, 901, 2050 Hammam Lif, Tunisia.*
[3] *Laboratoire Matériaux- Molécules et applications, IPEST, route Sidi Bou Said, B.P:51, 2075. La Marsa. Tunisia.*
[4] *Laboratoire de Biotechnologie Appliquée à l'Agriculture, Institut National de la Recherche Agronomique de Tunisie (INRAT), Rue Hedi Karray, 2049 Ariana, Tunis, Université de Carthage, Tunisia.*
[5] *Chemical and Materials Engineering Department College of Engineering Northern Border University, Kingdom of Saudi Arabia, P.O. Box 1321 Arar*

Received 26 Dec 2017,
Revised 06 Apr 2018,
Accepted 01 May 2018

Keywords
- ✓ *Pistacia lentiscus,*
- ✓ *Tribolium castaneum*,
- ✓ occupation space
- ✓ fumigant toxicity.

bachrouch_olfa@yahoo.fr
Phone: +21653957049;
Fax: +216 71752897

Abstract
This study was aimed at assessing the chemical composition and fumigant toxicity of *Pistacia lentiscus* L. essential oil leaves against *Tribolium castaneum* adults in the floor mill conditions. GC/MS analysis showed that essential oil contains α-Pinene (18.48%), β-Myrcene (22.59%) and Sabinene (8.67%) as major compounds. Response surface methodology was used to optimize the fumigant toxicity of *Pistacia lentiscus* L. essential oil leaves from Tunisia against adults of *Tribolium castaneum* L. (Herbst, 1797) developed during wheat flour storage. The effects of two parameters namely storage period and wheat flour occupation space, on *T castaneum* mortality were studied. Different storage periods (15, 30 and 45 days) and occupation spaces (0, 50 and 100 %) were experimented. The fitted mathematical model allowed us to determine optimal conditions of *P. lentiscus* leaves essential oil fumigant toxicity. Results clearly indicated that the space occupation was the main factor influencing the mortality percentage of *T. castaneum* adults. The selected optimal conditions were obtained for an occupation space of 30% and a storage period situated between 15 and 45 days. In these optimal conditions, mortality percentage can reach 85%.

1. Introduction
The red flour beetle *Tribolium castaneum* (Herbst) is one of the most widespread destructive primary pests attacking grains in storage. In milling industry, it is difficult to manage because it has the ability to exploit the hidden refugia where food material accumulates [1]. *T. castaneum* infestations causes significant losses in both the quality and quantity of milled cereal products [2]. Moreover, *T. castaneum* consume storage material directly. So, this infestation led to the increase of the temperature and humidity of the storage environment which accelerates the growth of molds including toxigenic species [3]. Globally, 10 to 20% of all grain produced is lost due to stored product pests before it reaches the consumer [4].

Protecting crops against agricultural pests is based mainly on the use of chemical pesticides and synthetic fumigants. However, broad spectrum insecticides have been reported to cause damage on human health and environment. By the way, insects develop insecticidal resistance to fumigation with synthetic insecticide [5].

In this regards, natural pesticides are preferred because of their biodegradability. Essential oils of many medicinal and aromatic plants were screened for their insecticidal activities against stored product beetles and they have been proved in some case to be more efficient than traditionally used pesticides [6-7-8-9-10]. *Pistacia lentiscus* is an evergreen shrub belonging to the Anacardiaceae family, locally known under the Arabic name of "Dharw" it is also khown as ghathoum[11]. Several studies have reported the insecticidal activity or repellency of *P. lentiscus* essential oil [12-13-6].

On the other hand, many researchers demonstrated the significant effect of different occupation space of the stored product on the effectiveness of the essential oil against stored grain pests [14-15-16-17].

Essential oils have drawn the greatest attention for fumigant activity against stored product insects [18]. However, [19]; [20] reported that only few researchers have been adopted essential oil as a grain protectant because there are many barriers under biological, technical, legal, and commercial categories.

So, the focus of this work was to optimize the experimental conditions of the essential oil application . In this context, an efficient way of optimization might be to systematically create prototypes around the key ingredient levels of the product via some type of response surface experimental design [21]. Response Surface Methodology (RSM) is a collection of mathematical and statistical techniques that makes a full description of independent variables effect in the vicinity of the optimum conditions [22-23-24]. Several classes of treatment structures can be used as RSM experiments [25]. The most widely used class is very similar to a factorial experiment to investigate linear effects of variables.

The aim of this study was to look for the experimental conditions leading to the maximum mortality of *T. castaneum* adults developed during wheat flour storage. As many factors can influence the mortality yield, screening design was applied to determine and exploit a mathematical model representing the relationship between the response (*T. castaneum* mortality) and variables (occupation space and storage period). Moreover, the chemical composition of *P. lentiscus* essential oil was investigated.

2. Material and Methods

2.1. Plant material

Pistacia lentiscus fresh leaves were collected from Nefza, Beja, North Tunisia (36°59'N; 9°4'E) in February 2014 and were identified by a taxonomic specialist. Pant material were air-dried at ambient temperature (20-25 °C) during one week and then conserved in cloth bags until the time of oil extraction.

2.2. Culture of insects

Tribolium castaneum was reared on an artificial diet based on wheat floor. Insect rearing was maintained in plastic boxes (15 x 22 x 10 cm) in an automatically regulated rearing room at temperature (28 ±1°C), relative humidity (65±5%) and darkness.

2.3. Extraction and analysis of the essential oil

The air-dried leaves of *P. lentiscus* were subjected to hydrodistillation for 3 hours using a Clevenger type apparatus. The obtained essential oil was stored in glass vials in darkness at 4°C until used. Essential oil analysis by GC–MS was performed on an Agilent 7890A GC system, coupled to an Agilent 5972C mass spectroscopy detector with electron impact ionization (70 eV). A HP-5 MS capillary column (30 m x 0.25 mm, coated with 5% phenyl methyl silicone, 95% dimethylpolysiloxane, 0.25 mm film thickness; Hewlett-Packard, CA, USA) was used. The column temperature was programmed to rise from 40 to 240 °C with a 5 °C/min rate, the carrier gas was Helium N60 with a 0.9 mL/min flow rate; split ratio was 100:1. Scan time and mass range were 1s and 50-550 m/z, respectively.

The compound identification was based on mass spectra (compared with Wiley Registry 9th Edition/NIST 2011 edition mass spectral library) and by comparison of their kovats retention indices (Ri) with either those in the literature (26) or with those of authentic compounds available in our laboratories. Kovats retention indices were determined in relation to a homologous series of n-alkanes (C_8–C_{40}) under the same conditions according with the definition of Van den Dool and Kratz [27].

2.4. Fumigant toxicity bioassays

2.4.1. Fumigant bioassays in spaces differently occupied with wheat floor

These trials were conduced to evaluate the effectiveness of *P. lentiscus* essential oil in spaces differently occupied by wheat flour: empty space, 10, 50 and 100 %. The concentration used in this trial correspond to the lethal concentration CL_{50} = 54.5 µL/L air screened in our previous work [6]. Bioassays were conducted in mini silos in plastic with 20 L of volume. Each trial was replicated three times and infested with tribolium adults as an artificial infestation. We put 160, 160, 800 and 1600 insects respectively in empty space, 10%, 50% and 100% occupied with wheat flour. Then, mortality was noted after 15, 30 and 45 days after the treatment. The mortality was calculated using the Abbott correction formula [28].

2.4.2. Experimental design

When many factors affect a desired response, it can be an exhausting task to optimize a process. Therefore, response surface methodology (RSM) can be an effective tool for optimizing the response [29-30-31-24]. Response surface methodology is defined as statistical method that uses quantitative data from appropriate experimental design to determine optimal conditions [32].

RSM was selected for the present study to optimize the fumigant toxicity of *P. lentiscus* leaves essential oil against adults of *T. castaneum* developed during wheat flour storage. The individual and interactive effects of storage periods (15, 30 and 45 days) and occupation spaces (0, 50 and 100 %) on *T. castaneum* mortality (Y) as response variable, was studied. In order to determine the significant experimental variables and develop a response surface for the optimization of *T. castaneum* mortality, the major factors mentioned above were further investigated by screening design.

The levels of the independent variables storage periods (X_1), occupation spaces (X_2), were coded respectively as: -1, 0 and +1 (Table1) represents the minimum, center and maximum for each parameter in the Central Composite Designs CCD. Dependent variable evaluated was *T. castaneum* mortality (Y).

A second order polynomial model was fitted for tribolium mortality (Y), giving an equation of the following form:

$$\mathbf{Y} = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{12} X_1 X_2 + e$$

Where (Y) is the calculated response function, X_1 and X_2 are the levels of the independent variables, β_0 is the intercept term, β_1 and β_2 are the linear coefficients, β_{11} and β_{22} are the quadratic coefficient, β_{12} is the interaction coefficient and e is the global error . Nemrod-w software package was used for the regression analysis of the experimental data obtained [33]. Fit quality of the polynomial model equation was expressed by the determination coefficient R^2, and its statistical significance was checked by an F-test. The significance of the regression coefficient was tested by a t-test. Significance level was given as *** $P< 0.001$, **$P< 0.01$, *$P< 0.05$. Differences with p-value superior to 0.05 were not considered significant. For CCD validation, optimum conditions were fixed on the basis of the data obtained from experimental design.

Table 1 presents experimental codes, ranges and levels of the independent variables levels of the screening design.

Table 1 Experimental codes, ranges and levels of the independent variables of the screening design

	Interval		
Parameter	**-1**	**0**	**1**
Storage period	15	30	45
Occupation space	0	50	100

Table 2 presents independent variables levels in coded and encoded form according to the experimental design. A total of 27 experiments with different combinations were carried out according to this table. Then, each experience was repeated three times.

3. Results and discussion

3.1. Composition of the essential oil

GC/MS analysis of *P. lentiscus* essential oil leaves collected from Nefza (Beja, Tunisia) site revealed that thirty nine compounds were identified. Results showed that Nefza chemotype presented an essential oil rich in beta-myrcene (22.59 %), α-pinene (18.48 %) and 2-β-pinene (13.5%) (Table 3). Studies concerning the variability of *P. lentiscus* essential oil composition from different regions have been reviewed. Our results are different from those obtained by [34] showed that Tunisian *P. lentiscus* EO from Zaghouan region was rich in α-pinene (17%), δ-terpinene (9%) and terpinene-4-ol (12%). Furthermore, [35] revealed the presence of limonene in the first time in Tunisian *P. lentiscus* essential oil only from Jebel Mansour (North of Tunisia) and Siliana (North Ouest of Tunisia).

sites. Previous studies reported that *P. lentiscus* essential oil from Morocco was characterized by the predominance of terpinene-4-ol (14.5 – 19.3%), caryophyllene oxide (6.5–10.3%) and limonene (6.7–10.3%) [36]. In this context, [37] reported that Greek oil contains 57 constituents where α-pinene (9.4–24.9%) and limonene (9.01–17.8%) were the major compounds. Moreover, sesquiterpene hydrocarbon fraction characterizes the spanich *P. lentiscus* essential oil [38]. Many factors such as geographic areas, individual chemotypes, harvest time and plant part distilled [39] could explain variability in chemical composition of the oil.

Table 2 Coded and experimental matrix for the central composite design

Essay	X_1	X_2
1	15 (-1)	0 (-1)
2	15 (-1)	0 (-1)
3	15 (-1)	0 (-1)
4	45 (1)	0 (-1)
5	45 (1)	0 (-1)
6	45 (1)	0 (-1)
7	15 (-1)	100 (1)
8	15 (-1)	100 (1)
9	15 (-1)	100 (1)
10	45 (1)	100 (1)
11	45 (1)	100 (1)
12	45 (1)	100 (1)
13	15 (-1)	50 (0)
14	15 (-1)	50 (0)
15	15 (-1)	50 (0)
16	45 (1)	50 (0)
17	45 (1)	50 (0)
18	45 (1)	50 (0)
19	30 (0)	0 (-1)
20	30 (0)	0 (-1)
21	30 (0)	0 (-1)
22	30 (0)	100 (1)
23	30 (0)	100 (1)
24	30 (0)	100 (1)
25	30 (0)	50 (0)
26	30 (0)	50 (0)
27	30 (0)	50 (0)

3.2. Fumigant toxicity

Adult mortality varied with storage duration and percentage of space occupation with wheat flour. Figure 1 revealed that high mortalities were obtained with spaces less occupied with wheat flour (100%) mortality with empty space) whatever storage duration (15, 30 and 45 days). Moreover, results showed that there is a significant difference between mortalities obtained for 0%, 10%, 50% and 100% occupation space percentage. Fumigation with *P. lentiscus* essential oil in an empty space induced nearly 100% mortality. However, fumigation in space filled with 10% and 50% of wheat flour led to 86.66% and 84.5% mortality respectively. Finally, the lowest mortality was recorded for 100% occupation space rate corresponding to 31%. For each storage period, statistical analyses revealed that the presence of three significantly different groups: 0%, 10 %-50% and 100% space occupation.

The insecticidal constituents of many plant extracts and essential oils exhibited fumigant activity against several target stored pest insects [40-41-9]. Moreover, many researchers have reviewed the importance of the amount of space occupancy on essential oil effectiveness [15-16]. [14] showed that concentration of cineole of 50g m (-3) in empty space induced nearly 100% mortality of *T. castaneum*. However, fumigation in space 50% and 95% occupied with wheat was effective only with 11% and 4.5% mortality respectively. In this context, investigation on fumigant toxicity of two *Eucalyptus* species on stored dates revealed that for both oils, high mortalities were obtained with spaces less occupied with dates (100% mortality with empty space) [17]. Thus, grain absorption of the vapor of essential oil compounds and the weakness of its penetration into seed interspaces could explain the lower efficiency of essential oil in filled spaces [42]. A multiple regression analysis between *T. castaneum* mortality as the dependent variable and the occupation space and storage period as independent variables indicated a highly positive significant ($P \leq 1$) relationship ($R^2 = 0.827$) between mortality and occupation space parameters (Table 4). About 82% of the variation was accounted for the regression.

Table 3 Chemical composition of *Pistacia lentiscus* essential oil leaves collected from Nefza region

Volatil compounds	RI[a]	RI[b]	Identification	%
Tricyclene	924	1014	GC–MS, Co-GC	0.19
α-thujene	928	1035	GC–MS, Co-GC	0.31
α-Pinene	939	1032	GC–MS, Co-GC	**18.48**
Camphene	954	1076	GC–MS, Co-GC	0.94
Sabinene	975	1132	GC–MS, Co-GC	2.66
2-β-pinene	980	1118	GC–MS, Co-GC	**13.50**
β-Myrcene	991	1174	GC–MS, Co-GC	**22.59**
l-phellandrene	1006	1176	GC–MS, Co-GC	0.93
α-Terpinene	1018	1188	GC–MS, Co-GC	3.69
p-cymene	1026	1280	GC–MS, Co-GC	0.58
Sabinene	975	1132	GC–MS, Co-GC	8.67
Cis-ocimene	1050	1245	GC–MS	0.20
β-ocimene	995	1240	GC–MS	0.66
Isoamylbutyrate	1045	1259	GC–MS	0.28
γ-terpinene	1053	1243	GC–MS, Co-GC	5.23
α-terpinolene	1088	1282	GC–MS, Co-GC	2.21
Butanoic acid, 3-methyl-, 3 methyl butyl ester	1048	1256	GC–MS	0.37
Terpineol-4	1172	1601	GC–MS, Co-GC	4.99
α-terpineol	1189	1709	GC–MS, Co-GC	0.86
Bornyl acetate	1295	1597	GC–MS, Co-GC	0.30
2-undecanone	1293	1598	GC–MS	0.55
α -copaene	1372	1490	GC–MS	0.27
β-Elemene	1390	1590	GC–MS	0.25
Caryophyllene	1434	1594	GC–MS, Co-GC	1.24
Aromadenrene	1474	1661	GC–MS	0.16
Cis-muurola 3,5 diene	1438	1606	GC–MS	0.15
α-humulene	1454	1687	GC–MS, Co-GC	0.56
Aromadendrene	1474	1661	GC–MS	0.23
Germacrene-D	1480	1726	GC–MS, Co-GC	0.26
α-amorphene	1485	1679192	GC–MS	0.54
β-cubebene	1386	1541	GC–MS	3.25
(+) epi-bicyclosesquiphellandrene	1482	1720	GC–MS	0.22
α-muurolene	1523	1714	GC–MS	0.64
γ-cadinene	1512	1763	GC–MS	0.14
Delta-cadinene	1527	1755	GC–MS	1.80
Zingiberene	1495	1720	GC–MS	0.18
Tau-muurolol	1608	2145	GC–MS	0.42
α-cadinol	1651	2227	GC–MS	0.25
Heptacosane	2700	2700	GC–MS	1.23

RI[a], RI[b]: Retention indices calculated using respectively an apolar column (HP-5) and polar column (HP-Innowax)

3.3. Validation of the model

The analysis of variance for the fitted model showed that the regression sum of squares was statistically significant at the level 99.9%. The regression coefficients and the analysis of the variance (ANOVA) indicate the high significance of the model (Table 5). The high R^2 value 0.993 showed the good agreement between the experimental results and the theoretical values predicted by the model (Pred R^2 = 0.989) [43]. The R^2 value is always between 0 and 1. The closer the R^2 is to 1.0, the stronger the model and the better it predicts the response [44]. The value of the adjusted determination coefficient (Adj R^2 = 0.989) was also very high to advocate for a high significance of the model [44].

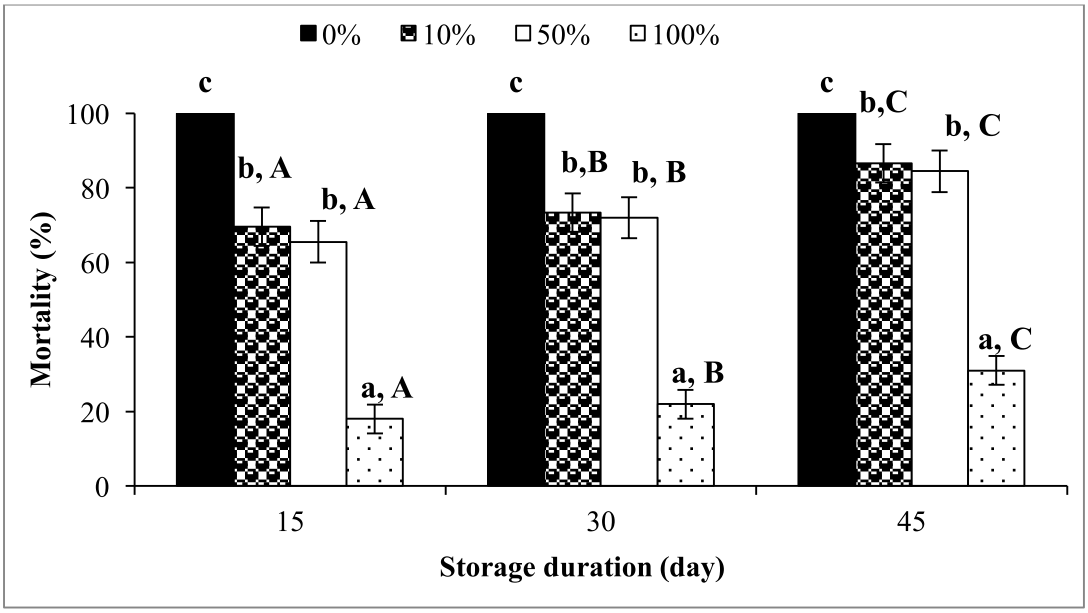

Figure 1 Percentage mortality of *T. castaneum* adults exposed to *P. lentiscus* essential oil after various periods of storage and under spaces differently occupied with wheat flour (For each storage period, comparisons were made between percentage mortality of the different occupation spaces (lower case). For each occupation space, comparisons were made between percentage mortality at different storage periods (upper case).Values followed by the same equivalent letter are not significantly different according to Duncan's multiple range test P < 0.05.

Table 4 Linear regressions of *Tribolium castaneum* adult mortality (%) after application of *Pistacia lentiscus* essential oil under different occupation space levels and storage periods

Parameters	N	R^2	R^2 ajusted	F	P	Equation
Occupation space	36	0.827	0.822	162.202	0.000	$Y=10.583+ 23.150X_1$
Storage period	36	0.031	0.003	1.106	0.114	$Y=56.083+ 6.188 X_2$

Table 5 Regression analysis

Standard deviation of the response	0.8
R^2	0.993
R^2A	0.991
R^2 pred	0.989
PRESS	314.933
Degree of freedom number	18

3.4. Statistical analysis of coefficients

The significance of each coefficient was determined by p-values which were listed in Table 6. The ANOVA analysis of the optimization study indicated that all the coefficients of the model were significant (p < 0.001%). Moreover, occupation space had a more important effect than the others variables on *T. castaneum* mortality (Y).

Table 6 Statistical analysis of coefficients

term	Y	
	Coefficients	p-value
β 0	72.7	*** (< 0.001%)
β 1	5.4	***
β 2	-38.2	***
β 11	1.8	***
β 22	-12.1	***
β 12	3.3	***

The final equations for optimization of *P. lentiscus* essential oil toxicity against *T. castaneum* derived from the application of the method (after eliminating non-significant terms) are given below:

$$Y= 72.7+ 5.4\ X_1 - 38.2X_2 + 1.8\ X_1^2 -12.1\ X_2^2+ 3.3\ X_1X_2$$

According to the model, a simulation was undertaken and results are presented in Table 7 in column 3. Results revealed that the difference between the expected response and the calculated one where week ranging between 0.7 and 5.1 which minimize the error. Then, the model seems to be efficient to express what happened in reality and could be considered reliable.

Table 7 Residue table

N° Exp	Yexp.	Ycalc.	Difference	Norms	dU	Student-R	R-Student	D-Cook
1	100.0	98.4	1.6	2.081	0.269	2.4	0.6	0.4
2	100.0	98.4	1.6	2.081	0.269	2.4	0.6	0.4
3	100.0	98.4	1.6	2.081	0.269	2.4	0.6	0.4
4	100.0	102.8	-2.8	-3.620	0.269	-4.2	-1.1	1.1
5	100.0	102.8	-2.8	-3.620	0.269	-4.2	-1.1	1.1
6	100.0	102.8	-2.8	-3.620	0.269	-4.2	-1.1	1.1
7	17.0	15.6	1.4	1.864	0.269	2.2	0.5	0.3
8	18.0	15.6	2.4	3.163	0.269	3.7	0.9	0.8
9	19.0	15.6	3.4	4.462	0.269	5.2	1.3	1.7
10	31.0	33.0	-2.0	-2.538	0.269	-3.0	-0.7	0.5
11	32.0	33.0	-1.0	-1.239	0.269	-1.4	-0.4	0.1
12	30.0	33.0	-3.0	-3.837	0.269	-4.5	-1.1	1.2
13	65.0	69.0	-4.0	-5.244	0.185	-5.8	-1.5	1.3
14	64.0	69.0	-5.0	-6.543	0.185	-7.2	-2.0	2.0
15	66.0	69.0	-3.0	-3.945	0.185	-4.4	-1.1	0.7
16	84.0	79.9	4.1	5.292	0.185	5.9	1.5	1.3
17	85.0	79.9	5.1	6.591	0.185	7.3	2.0	2.0
18	85.0	79.9	5.1	6.591	0.185	7.3	2.0	2.0
19	100.0	98.8	1.2	1.540	0.185	1.7	0.4	0.1
20	100.0	98.8	1.2	1.540	0.185	1.7	0.4	0.1
21	100.0	98.8	1.2	1.540	0.185	1.7	0.4	0.1
22	22.0	22.5	-0.5	-0.625	0.185	-0.7	-0.2	0.0
23	21.0	22.5	-1.5	-1.925	0.185	-2.1	-0.5	0.2
24	23.0	22.5	0.5	0.674	0.185	0.7	0.2	0.0
25	73.0	72.7	0.3	0.385	0.185	0.4	0.1	0.0
26	71.0	72.7	-1.7	-2.213	0.185	-2.5	-0.6	0.2
27	72.0	72.7	-0.7	-0.914	0.185	-1.0	-0.3	0.0

3.5. Interpretation of the response surface model

The relationship between the responses and the experimental variable can be showed graphically by three dimensional response surface plots (Figure.2). The vertical axes showed occupation space X_2 and the horizontal axe corresponds to the independent variable storage period X_1. The topography of these response surfaces are also illustrated by isoresponse contours representing lines of constant response in a two variable planes. Such plots are helpful in studying the effects of the variation of the factors in the domain studied and consequently, in determining the optimal experimental conditions [45]. In Figure 2, the examination of the isoresponse contours and three-dimensional plots showed that tribolium mortality percentage increasing when occupation space decreased independent of the storage period. Results revealed that the higher mortality 100% was recorded in empty spaces 0% whatever storage period. So, this result seems to be interesting but it cannot be applicable in storage environment. However, results showed that when occupation space was fixed in 30% and storage period in 40 days, the tribolium mortality percentage recorded were 85%. Thus, X_2 seems to be not influent.

In conclusion, the originality of this study consist on assessing for the first time on insecticidial activity of essential oil from *Pistacia lentiscus* (L.) against *Tribolium castaneum* (Herbst) adults in flour mill conditions. So, our results revealed that occupation space parameter could significantly affect Tribolium mortality whatever storage period. Accordingly, mortality optimal response was reached for 30% occupation space. This study demonstrated that the optimizing of the occupation space and the storage period parameters could contribute to the success of *T. castaneum* management under mill environment.Thus, the use of *P. lentiscus* essential oil under industrial scale could be successful.

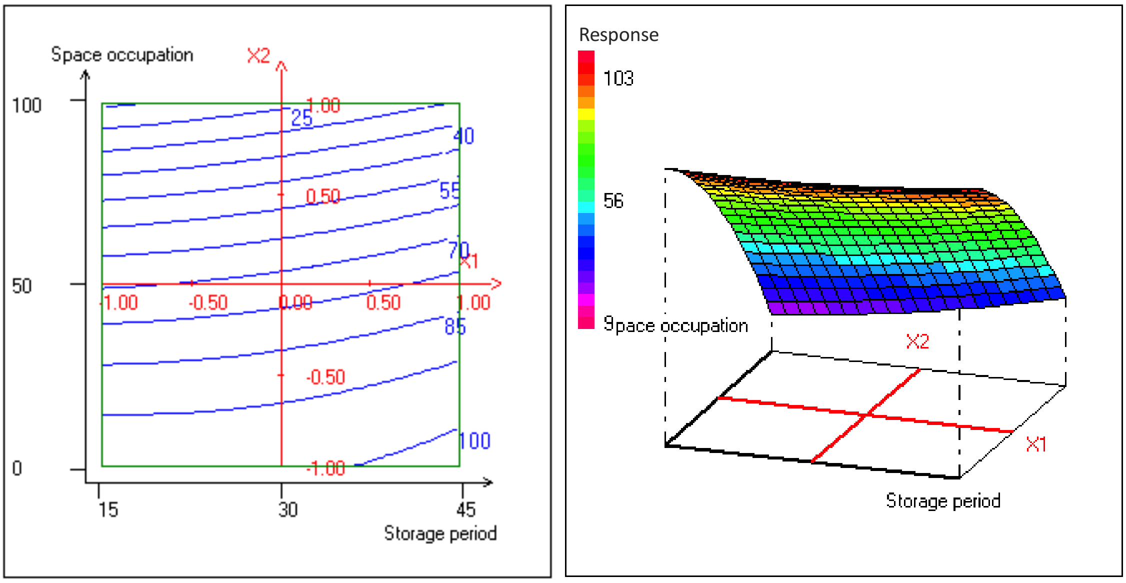

Figure 2: Three-dimensional response surface and contour plots for the effect of storage period and occupation space on Tribolium mortality

References

1. J. F. Campbell, M. D. Toews, F. H. Arthur, R.T. Arbogast, *J. Econ. Entomol.* 103 (2010) 991-1001.
2. D. Rees, *Insects of Stored Products*, 9780643101128 (2004) 192.
3. N. Magan, R. Hope, V. Cairns, D. Aldred, *Eur. J. Plant. Pathol.* 109 (2003) 723–730.
4. T.W. Phillips, J.E. Throne, *Ann. Rev. Entomol.* 55 (2010) 375-397.
5. G. D. Rossi, C. D. Santos, M. G. Cardoso, A. D. Corrêa, C. M. P. Abreu, L. V. Paiva, *Ciênc. Agrotec.* 34 (2010) 361–366.
6. O. Bachrouch, J. Mediouni-Ben Jemâa, T. Talou, B. Marzouk, M. Abderraba, *Bull. Insectology* 63 (2010) 129-135.
7. M. Ahmadi, A. M. M. Abd-Alla, S. Moharramipour, *Appl. Radiat. Isot.* 78 (2013) 16-20.
8. S. Kim, D. Lee, *J. Asia Pac. Entomol.* 17 (2014) 13–17.
9. O. Bachrouch, N. Ferjani, S. Haouel, J. Mediouni Ben Jemâa, *J. Stored. Prod. Res.* 65 (2015) 127–133.
10. D. Yu, J. Wang, X. Shao, F. Xu, H. Wang, *J. Appl. Microbiol.* 119 (2015) 1253–1262.
11. M, Hmamouchi, *Les plantes médicinales et aromatiques marocaines: utilisations traditionnelles, marchés, biologies, écologies, chimie, pharmacologie, toxicologie, lexiques*, 9954800700 (1999) 387.
12. M. S. Pascual-villalobos, A. Robledo, *Ind. Crops. Prod.* 8 (1998) 115–120.
13. A. Lamiri, S. Lhaloui, B. Benjilali, M. Berrada, *Field. Crops. Res.* 71 (2001) 9-15.
14. V. Rozman, Z. Korunic, I. Kalinovic, Effect of different quantities of wheat on the effectiveness of the essential oil cineole against stored grain insect pests. In: Proceedings of the 8th International Conference on Controlled Atmosphere and Fumigation in Stored Products, Chengdu, China, September 21-26, pp. 503-506 (2008).
15. E. Shaaya, M. Kostjukovski, J. Eilberg, C. Sukprakarn, *J. Stored. Prod. Res.* 33 (1997) 7–15.
16. B. H. Lee, P. C. Annis, F. Tumaalii, S. E. Lee, *Phytoparasitica.* 32 (2004) 498–506.
17. J. Mediouni Ben Jemâa, S. Haouel, M.L. Khouja, *J. Stored. Prod. Res.* 53 (2013) 67-71
18. S. Rajendran, V. Sriranjini, *J. Stored. Prod. Res.* 44 (2008) 126–35.
19. Z. Korunić, V. Rozman, I. Kalinović, The potential use of natural essential oils in the fumigation of stored agricultural products. In: Proceedings of the 8th International Conference on Controlled Atmosphere and Fumigation in Stored Product- CAF2008. 22-26 September, Chengdu, China (eds. Daolin,G.,Navarro, S.,) (2008).
20. G. J. Daglish, Opportunities and barriers to the adoption of new grain protectants and fumigants, Proceeding 9th International Working conference on Stored Product Protection. Sao Paolo Brazil, pp. 209-216 (2006).

21. M. Maldao-Martins, S. Beirao-da-Costa, C. Neves, C. Cavaleiro, L. Salgueiro, M.L. Beirao-da-Costa, *Food. Qual Prefer*. 15 (2004) 447-452.
22. E. T. S. Chow, L. S. Wei, R. E. DeVor, M. P. Steinberg. *J. Food. Sci.* 53 (1988) 1761–1765.
23. A. A. Guillou, J. D. Floros, *J Food Sci*. 58 (1993) 1381–1389.
24. D. C. Montgomery, *Design and analysis of experiments*, 978-1118146927 (1991) 680
25. M. Meilgaard, G. Civille, B.T. Carr, "*Sensory Evaluation Techniques*", 1439832277 (1991) 416.
26. R.P. Adams, *Identification of Essential Oil Components by Gas Chromatography/Quadrupole Mass Spectroscopy*, 1932633219 (2001) 804.
27. H. Van Den Dool and P. D. Kratz, J. Chromatogr. 11 (1963), 463
28. W.S. Abbott, J. Econ. Entomol. 18 (1925) 265-267.
29. E. P. Box, W. G. Hunter, J. S. Hunter, *Statistics of experimenters*, 978-0-471-71813-0, (1978) 664.
30. R. Carlson, *Design and optimization in organic synthesis*, 0-444-89201-X (1992).
31. J. Goupy, *Plans d'Expériences pour Surfaces de Réponse*, 2100039938 (1999) 409.
32. M. Giovanni, *Food Technol* 37 (1983) 41–45.
33. D. Mathieu, J. Nony, R. Phan-Tan-Luu, New efficient methodology for research using new Optimal Design (Nemrodw) Software-L.P.R.A.I. (2000).
34. F. Ben Douissa, A-M. Mariotte, N. Hayder, L. Chekir-Ghedira, M-G. Dijoux-Franca, M. Hammami, K. Ghedira, *Flavour. Frag. J*. 20 (2005) 410-414.
35. O. Bachrouch, T. Talou, W. Aidi Wannes, R. Ksouri, N. Salem, M. Abderraba, B. Marzouk, *Plant. Biosyst.* (2013).
36. S. Zrira, A. Elamran, B. Benjilali, *Flavour. Frag. J.* 18 (2003) 475–480.
37. G. Chryssavgi, P. Vassiliki, M. Athanasios, T. Kibouris, K. Michael, *Food. Chem.* 107 (2008) 1120– 1130
38. A. Fernandez, A. Camacho, C. Fernandez, J. Altarejos, *J. Essent. Oil. Res*. 12 (2000) 19– 23.
39. N. B. Perry, R. A. Anderson, N. J. Brennan, M. H. Douglas, A. J. Heaney, J. A. Mcgimpsey, B. M. Smallfield, *J. Agric. Food. Chem.* 47 (1999) 2048 – 2054.
40. J. Wang, F. Zhu, X. M. Zhou, C. Y. Niu, C. L. Lei, *J. Stored. Prod. Res.* 42 (2006) 339-347.
41. A. Ayvaz, O. Sagdic, S. Karaborklu, I. Ozturk, *J. Insect. Sci.* 10 (2010) 1-13.
42. A. Liska, V. Rozman, I. Kalinovic, A. Eced, S. Slavica Mustac, B. Perhoc, *Poljoprivreda*. 1 (2011) 58-63.
43. S. Weisberg, *Applied linear regression*, 0-471-66379-4 (1985) 305.
44. A. I. Khuri, J. A. Cornell, *Response Surfaces: Design and Analysis*, 0-82477653-4 (1987) 536.
45. A. Kamoun, B. Samet, J. Bouaziz, M. Chaabouni, *Analusis*. 27 (1999) 91–96.

(2018) ; http://www.jmaterenvironsci.com