Phytochemical Analysis and Insecticidal Activities of Seed Extracts from *Oenanthe pimpinelloides* L. Treated Paper Samples *vs. Tribolium castaneum*

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The utilization of plant extraction products from *Oeneanthe pimpinelloides* (Apiaceae family) seeds were investigated in terms of their use as an insecticide control of packaging materials. The aim was to investigate their insecticidal effects against the flour beetle *Tribolium castaneum.* The *Oeneanthe pimpinelloides* seeds were extracted with methanol. By using the liquid-liquid extraction method, the hexane extract (II) was separated from the methanol extract (I) and hexane and methanol were evaporated. Then, the chemical composition of each sample was determined *via* gas chromatography–mass spectrometry. The methanol extract predominantly contained tetrahydrofuran, 1-methoxy-2-propanol, 1-methoxy, 2-butoxyethanol, 1-phenylethanone, cyclohexene carboxylate derivative, (3-phenyl-2-propynylidene) cyclopropane, diphenyldiazene, and dihydroxypropyl ester components, while the hexane fraction contained nonane, 1-octanol, decane, undecane, tridecane, alkyl benzene, benzene sulfonic acid, benzoxazine, and hexadecanoic acid components, as well as some derivatives of them. Each fraction was dissolved in DMSO for impregnation on filter paper. The insecticide effects of the paper samples were determined against *Tribolium castneum*. According to the results, the mortality started after 3 d for each fraction. After 4 d, the hexane fraction indicated total mortality in comparison with the methanol fraction, which showed partial mortality (3/5).

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INTRODUCTION

Pest infestation of stored products can contaminate the products and cause serious postharvest losses in agricultural products and packaged food products. *Tribolium castaneum*, which is one of the widespread pests of stored commodities, especially in the tropics and subtropics, causes considerable financial losses (Howe 1956; Sokolof 1972; Sokolof 1974; Sokolof 1977; Matthews 1993). *T. castaneum* adults have a flat curved sided body with red-brown color. The upper part including thorax are covered with punctures, elytra with ridged through their length, and wings supported by extracellular cuticle

(Pietyra and Wegierek 2017). *Tribolium castaneum* and *Tribolium confusum* are similar pests (Cebeci *et al*. 2018), from which the morphology, histology, and chemistry of the wings can be distinguished (Zohry and El-Sayed 2019) and morphological structure of T.C. determined by scanning electron microscopy (Zohry 2017). *Tribolium castaneum* is considered a strict secondary pest and causes millet damage throughout the Sahelian zone (Gahukar 1976; Roorda *et al.* 1982). In addition, grain residues can be used as an insulating material or furniture equipment for indoor and outdoor equipment (Mo *et al.* 2016).

For the control of these insects, synthetic pesticides and fumigants are predominantly used. However, the widespread application of synthetic insecticides has caused serious issues, *e.g.*, toxic residues in nutrition and toxicity to consumers. In addition, synthetic chemicals are restricted because of environmental pollution and the insect strains resistance (Zettler and Cuperus 1990; White 1995; Riebeiro *et al.* 2003; Marrone 2019). Therefore, there is an important demand for biodegradable, cost-effective, natural materials from plant resources that are suitable as alternative chemical insecticides (Ebadollahi 2013; Marrone 2019; Samada and Tambunan 2020).

Essential oils from various annual plants may have insects (Isman 2000; Regnault-Roger 1997) or mosquito control properties (Sukumar *et al.* 1991; Chantraine *et al.* 1998; Trabousli *et al*. 2002; Trabousli *et al*. 2005; Amer and Mehlhorn 2006). Essential oils from the Apiaceae family have pest control properties (Regnault-Roger and Hamraoui 1994; Tunc *et al*. 2000; Kim *et al.* 2003; Lee 2004; Miyazawa *et al*. 2004; Tsukamoto *et al.* 2005).

In addition to essential oils, various plant extracts also have a potential pesticide effect against *T. castaneum* (Jbilou *et al.* 2006). Compared with the plant extracts, the low yields of essential oils limit their utilization rate in large-scale applications.

The chemical composition of many species of the Apiaceae family, which is an inexpensive source of natural pest control agents, has been investigated in various studies (Pieroni *et al*. 2006; Baldini *et al*. 2009; Evergetis *et al.* 2009; Labed *et al.* 2011). *Oeneanthe pimpinelloides* (O.P.), a species from the Apiaceae family, is among the invasive species that are growing in its natural habitat in Turkey. The body morphology of *O. pimpinelloides* is circular, in which parenchymatic cortex cells are arranged in 2 to 4 series, sclerenchyma tissue cells in 10 to 12 incrassans, and 1 to 3 central bundles below peripheral bundles (Guner 2016).

Based on the literature survey, it is seen that the essential oils have been isolated from the freshly collected plant materials, including *Oenanthe pimpinelloides* from stems, leaves, and flowers in different habitats throughout Greece, and their larvicidal activity against the West Nile virus vector *Culex pipiens* have been investigated by Evergetis *et al.* (2009).

O. pimpinelloides essential oils are known to have insecticidal activities against various common insects, *e.g.*, *Culex pipiens* L., and constitute a potential class of natural compounds (Evergetis *et al.* 2009; Labed *et al.* 2011). However, the literature survey indicates that there are limited studies about the seed parts of *Oenanthe* plants. Depending on this literature survey related with *Oenanthe* plants extracts, it can be concluded that the investigations mainly have dealt with the ethanoic and aqueous extracts of the aerial parts of the plant as: The clean *Oenanthe* species was cut into small pieces and then dried. The dried samples were crushed and were subsequently extracted with distilled water or alcohol (He *et al.* 2020). In addition, research has been done on the extract's antioxidant (Bae *et al.* 2022; Sulaiman *et al*. 2022), antimicrobial (Abdusalam *et al.* 2022), anti-inflammatory (Lee *et al.* 2006; Park *et al.* 2019) *etc.* properties. Additionally literature work shows that there are investigations related with the determination of the nutrient content of *Oenanthe* *pimpinelloides* (Demir *et al.* 2020), their traditional use in the human nutrition (Kayabasi *et al.* 2018), and the essential oil composition of *O. pimpinelloides* retrieved from Greek herbal biodiversity (Evergetis and Haroutounian 2014). As there have been limited studies about the larvicidal activity of *Oenanthe pimpinelloides* L., the present manuscript focuses on the seed parts, the larvicidal activity of which has not been investigated yet. Therefore, extracts from *O*. *pimpinelloides* seed parts were selected for the current study.

Oeneanthe pimpinelloides seed extracts were impregnated in filter paper, and the toxic effects of these papers are investigated in the current study. Filter paper was used, since papers are formed from a suitable material for carrying the active ingredients without losing the sample properties. Also, it is appropriate to absorb the extract in the filter paper material. In addition, thanks to some advantages of filter paper, *e.g.*, being inexpensive, deployable, and absorbent, it became an increasingly important parallel with the development of technologic devices (Shen *et al.* 2000; Guan *et al*. 2014; Ozer *et al.* 2020; Zhang *et al.* 2020). In this respect, the immersing method is one of the useful methods for imparting the desired properties to the paper surface, *e.g.*, water-resistant features (Shen *et al*. 2000; Li *et al*. 2008; Seo *et al.* 2008; Li *et al*. 2009). Thus, paper materials can be developed for paper packaging for storing grains. In addition, the can serve as an alternative to protecting the product from stored-product insects that develop in wrapping paper in a viable, cost effective way that does not harm the environment.

EXPERIMENTAL

Plant Collection

The *O*. *pimpinelloides* samples were collected from the Istanbul University-Cerrahpasa, Faculty of Forestry campus garden in their natural habitat. The samples were diagnosed by Prof. Dr. Unal Akkemik (Istanbul University-Cerrahpasa, Faculty of Forestry, Department of Forest Botany). The seeds were separated from the body of the plant. The seed was separated from its umbrella part and washed, dried, and ground with a grinder.

Extractions of 20 g dried seed were carried out by reflux with methanol for 4 h (VELP Scientifica, SER 148 model). Extracts were prepared with 150 mL of solvent. Then, the hexane part was separated *via* liquid-liquid extraction with n-hexane. The solvents were eliminated using a rotary evaporator.

Chemical Composition Identification

Gas-phase chromatography was used to show the relative composition and concentration of various compounds that were present in the extracts of the seeds. The photochemical analysis of the seed extracts was analyzed by the TUBITAK-Marmara Research Center (Gebze, Istanbul) using gas chromatography–mass spectrometry (GC-MS) (Xcalibur, Thermo Scientific, Waltham, MA), equipped with head space (the processing method was 59H4407/Aroma). The relative amounts of the substances were calculated based on the peak-area ratios, and the peak areas of the substances were calculated with the provided Xcalibur software. The ethanolic and hexane extract-treated paper samples were analyzed *via* Fourier-transform infrared spectroscopy (FT-IR) with an ATR attachment (Perkin Elmer Spectrum 100, Istanbul University-Cerrahpasa, Forest Product Chemistry and Technology Lab.) and compared to base paper samples. The resolution value was 16 cm^{-1} and for each sample, 32 scans per spectrum were recorded.

The ethanol and hexane extracts were diluted with DMSO at a 1 to 5 ratio (w/w) and mixed in an ultrasound bath for 30 min. It was transferred into 1 mL portions in 5 separate Petri dishes. The extracted samples were transferred to a glass petri dish and filter paper was dipped into the extracts diluted with DMSO. The base paper samples were directly impregnated with DMSO. Whatman Filter Paper No. 2 was selected because of its pure structure. The paper samples were kept under the hood until completely dry.

Insecticidal Experiment

The breeding of the *Tribolium castaneum* was maintained in the natural laboratory conditions of the University of Ghardaia-Algeria. Insect-fed semolina samples were taken from a warehouse belonging to the Ghardaia storage cooperatives during a period of three months and were brought back in jars for propagation until new adults of the next generation were used for the experiments. The *T. castaneum* individuals were maintained under laboratory conditions. It should be noted that breeding of these insects was carried out in an oven set at a temperature of 30 °C / 35 °C and a relative humidity ranging from 65% to 70%, in glass jars containing semolina.

Toxicity Bioassay

The treatment for the *T. castaneum* adults was applied with a filter paper that was emerged in the hexane and methanol extracts in Petri dishes. The latter consists of 10 g of date flour and 15 *T. castaneum* adults. A control group was treated with DMSO. Three repetitions were applied for each treatment trial. After spraying, all the boxes were closed with the help of film, to prevent the individuals from escaping.

Toxicological Parameters: Mortality Rate

Mortality is the first criterion for judging the effectiveness of a chemical or biological treatment. The percentage of mortality observed in the control adults treated with the plant extract was estimated by applying the following formula, as shown in Eq. 1,

$$\text{Observed mortality} = \frac{\text{Number of deaths}}{\text{Total number of individuals}} \times 100 \qquad (1)$$

(Hadj *et al.* 2006).

The concentration of Efficacy CE50 CE refers to the “concentration of efficacy”, the EC50 is the quantity of a single-dose substance that causes mortality in 50% (half) of a treated group. The EC50 is a way of measuring the short-term toxic potential (acute toxicity) of a material (Kemassi 2014).

Statistical Analysis

Probability analysis of the corrected mortality percentage based on the doses of extracts applied to *Tribolium castaneum* were conducted to determine the values of the lethal dose 50 and 90 (LD 50 and LD 90) of the tested extracts. The mortality rate was subjected to the analysis of the variance (ANOVA). When the results were significant at a p-value equal to 0.5, Tukey (HSD) and Kruskal-Wallis tests were used. The XLSTAT software (version 2012, Microsoft, Redmond, WA) was used to interpret the experimental results.

RESULTS AND DISCUSSION

Phytochemical Analysis of the Extract Oil

The GS-MS chromatograms of the *O. pimpinelloides* seed extracts were examined, and it was found that O. *pimpinelloides* seeds methanol extract (I) consisted of tetrahydrofuran, 1-methoxy-2-propanol, 2-butoxyethanol, 1-phenylethanone, cyclohexene carboxylate derivative, (3-phenyl-2-propynylidene) cyclopropane, diphenyldiazene, and dihydroxypropyl ester components (as shown in Table 1). The methanolic extract was 23% yield.

Table 1. Gas Chromatography–mass Spectrometry (GC-MS) Chromatogram Results of the MeOH Extraction of the *O. pimpinelloides* Sample

Possible Compounds	Molecular Formula	RT[a]	Area(%)	SI/RSI[b]	Peak Area
Furan, tetrahydro-(CAS)	$(CH_2)_4O$	1.65	36.57	921/932	7.054.708
2-Propanol, 1-methoxy-	$C_4H_{10}O_2$	3.77	2.80	632/811	540.117
Ethanol, 2-butoxy-(CAS)	$C_6H_{14}O_2$	11.38	30.22	898/931	5.831.041
Ethanone, 1-phenyl-(CAS)	C_8H_8O	20.29	2.78	644/885	537.239
Methyl 2-(3 ',3 '-dimethyl-1 '-butyn-1-yl)-1–cyclohexenecarboxylate	$C_{14}H_{20}O_2$	29.6	11.08	927/937	2.137.422
(3-Phenyl-2-propynylidene)cyclopropane	$C_{12}H_{10}$	6.39	31.73	722/929	1.232.104
Diazene, diphenyl- (CAS)	$C_{12}H_{10}N_2$	43.07	2.81	545/813	541.458
Octadecanoic acid, 2,3-dihydroxypropyl ester (CAS)	$C_{21}H_{42}O_4$	55.71	7.35	639/670	1.418.899
Note: [a]RT Retention time (min); and [b]SI/RSI Search index/Reverse search index					

The O. *pimpinelloides* seeds hexane extract (II) consisted of nonane, 1-octanol, decane, undecane, tridecane, alkyl benzene, benzene sulfonic acid, benzoxazine, and hexadecanoic acid components, as well as some derivatives of them (as shown in Table 2).

Table 2. Gas Chromatography–mass Spectrometry (GC-MS) Results of the Hexane Extraction of the *O. pimpinelloides* Sample

Possible Compounds	Molecular Formula	RT[a]	Area (%)	SI/RSI[b]	Peak Area
Nonane	C_9H_{20}	1.52	0.86	844/903	1.050.584
Nonane, 4-methyl-	$C_{10}H_{22}$	1.82	4.31	875/898	5.259.325
Decane	$CH_3(CH_2)_8CH_3$	2.14	8.73	904/927	10.658.044
Decane	$CH_3(CH_2)_8CH_3$	2.33	8.64	922/936	10.553.363
1-Octanol, 2-butyl-	$C_{12}H_{26}O$	1.82	4.31	784/815	5.259.325
Undecane	$CH_3(CH_2)_9CH_3$	3.46	13.42	901/918	16.382.794
2,3-Dimethyldecane	$C_{12}H_{26}$	4.38	2.65	794/899	3.229.899
Decane, 1,1'-oxybis-	$C_{20}H_{42}O$	4.58	0.38	793/809	458.061

Tridecane	$CH_3(CH_2)_{11}CH_3$	5.27	3.98	824/893	4.862.687
Benzene, 1,3,5-trimethyl-	C_9H_{12}	6	2.12	721/782	2.585.592
Benzene, 1-ethyl-3-methyl-	C_9H_{12}	6.95	1.42	824/885	1.729.204
Benzene, 1,3,5-trimethyl-	C_9H_{12}	7.49	3.35	910/921	4.085.482
Benzene, 1-methyl-3-propyl-	$C_{10}H_{14}$	8.08	2.06	821/883	2.519.932
Benzene, 1,2,3,4-tetramethyl-	$C_{10}H_{14}$	8.74	0.80	846/903	981.642
Benzene, 1,2,4-trimethyl-	C_9H_{12}	9.07	3.00	830/915	3.663.473
Benzene, 2-ethyl-1,4-dimethyl-	$C_{10}H_{14}$	9.61	1.19	885/897	1.455.444
Benzene, 1-ethyl-2,4-dimethyl-	$C_{10}H_{14}$	9.8	1.47	891/905	1.789.811
Benzene, 1-ethyl-3,5-dimethyl-	$C_{10}H_{14}$	10.07	0.90	862/879	1.101.009
Ethanol, 2-butoxy-	$C_{20}H_{14}O_2$	11.33	6.87	908/916	8.392.201
Benzene, 1,3-diethyl-5-methyl-	$C_{11}H_{16}$	11.92	0.71	770/835	870.045
Benzene, 1,2,3,4-tetramethyl-	$C_{10}H_{14}$	12.32	0.93	856/918	1.140.165
Benzene, 1,2,3,4-tetramethyl-	$C_{10}H_{14}$	14.14	0.61	850/904	743.614
Acetophenone	C_8H_8O	20.21	7.12	940/947	8.691.342
Quinoline, 2-methyl-	$C_{10}H_9N$	30.89	2.63	910/944	3.213.290
1,1-Biphenyl	$C_{12}H_{10}$	31.65	1.37	922/945	1.669.196
Benzenesulfonic acid, 4-hydroxy-	$C_6H_6O_4S$	32.85	3.30	897/899	40.332.003
2-N-cyanoimino-1 H,4H-3,1 –benzoxazine	----	41.68	7.72	924/975	9.424.803
Hexadecanoic acid	$CH_3(CH_2)_{14}COOH$	58.47	3.23	770/832	3.938.261

Note: [a]RT Retention time (min); and [b]SI/RSI Search index/Reverse search index

According to the GC-MS results, the hexane extract was rich in aromatic and aliphatic hydrocarbons and the methanolic extract was rich in cyclic and acyclic ethers and aliphatic alcohols.

A previous study by Evergetis *et al.* (2009) indicated that the essential oil of *Oenanthe pimpinelloides* L. fresh aerial parts contained nonoxygenated monoterpenes (y-terpinene (43.2%), o-cymene (14.4%), p-sesquiphellandrene (8.2%), and p-pinene (6.7%)) that had larvicidal activity against *Culex pipiens* larvae. However, the cited study did not contain any results regarding the chemical composition of the seed parts of *Oenanthe pimpinelloides*.

Fourier-Transform Infrared Spectroscopy (FT-IR) Results of the O. *pimpinelloides* Seed Extract Treated Paper

After treatment, the amount of extraction products absorbed by the paper samples was 5.31%. A comparison of the FTIR results of the extract impregnated paper and base paper samples are shown in Fig. 1.

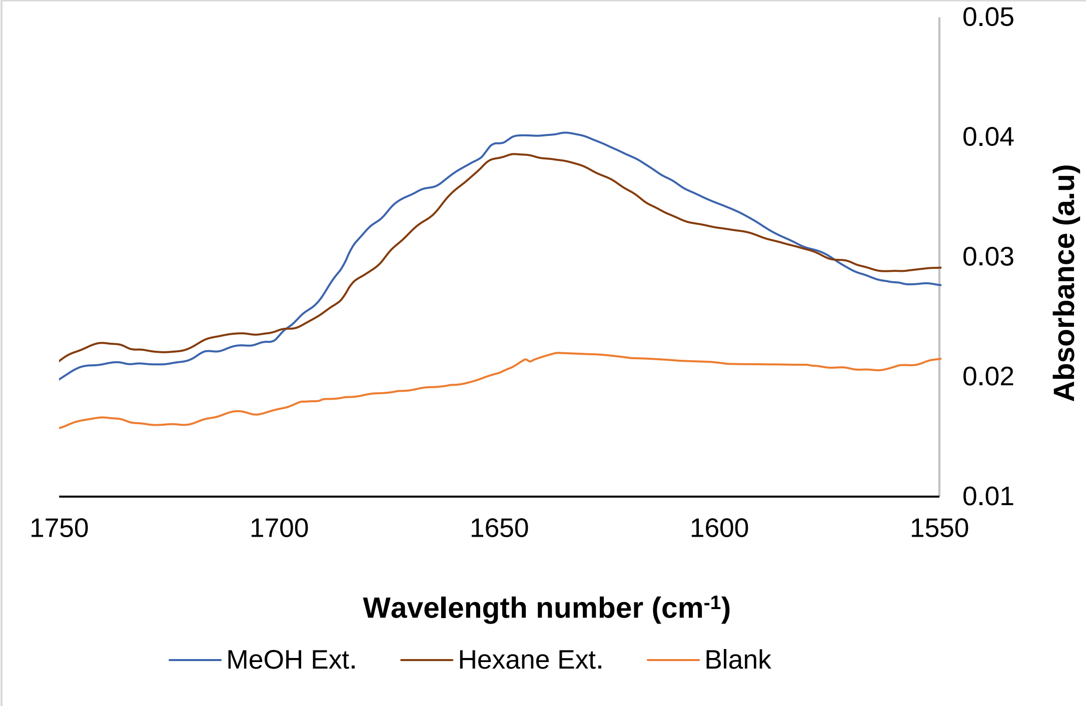

Fig. 1. FTIR results of the MeOH and Hexane extract-treated paper and base paper samples

A 1650 cm^{-1} band is associated with a strong stretching C=O bond. The FTIR results indicated that the bonds seen in the ethers and alcohols in the carbonyl compounds were retained by the paper samples. The base paper had a chemical bonding with both methanol and hexane extract with reactive C=O and COOH groups which is highly visible, whereas other chemical bonding between the base papers and extracts was not sufficiently visible.

Insecticidal Activity of Plant Extracts

The insecticidal activity of the seed extracts of *O. pimpinelloides* against the *T. castaneum* adults was studied at the Pedagogical Laboratory/ University of Ghardaïa (Algeria). The work was conducted under controlled conditions of temperature at 32 ± 2 °C, and humidity at 60 ± 4%. The filter paper that had been treated with seed extracts of *O. pimpinelloides* hexan and ethanolic extracts was placed petri dish and directly contacted with 15 adults of *T. castaneum*. For each sample, three repetitions were performed. The synthetic Pyritrinoides group (Decis®EC50) was used as a positive control under the same conditions. Besides, the insects of the control groups were examined with *T. castaneum.* Kinetic monitoring of the mortality was observed. The estimation of the mortality rates and the lethal doses were 50 and 90.

The mortality rate of the control batches was compared to the samples. The strong insecticidal effect of the *O. pimpinelloides* seed extracts was confirmed with respects to *T. castaneum*. The rate of mortality observed in the *T. castaneum* treated with hexane and ethanolic extracts from O. *pimpinelloides* was 100%. A similar study by Zardi-Bergaoui *et al.* (2008) indicated that ethyl acetate extract of crude *Anacyclus* and its eight fractions had A3, A4, P8, P10, F2, F3, and F7.

At the insecticidal level, positive results for the contact activity of the tested extracts were obtained against the red flour beetle adults. It was very clear that the mortality percentage was high for both pure extracts after 5 days. However, cryptocleidids showed a considerable inhibitory effect on T. *confusum* growth. The adult mortality reached 12 days after treatment. The corrected mortality properties as a function of the logarithms of the applied concentrations allowed for easy determination of the efficacy concentrations, *i.e.*, the Lethal Dose, LD_{50} and LD_{90}. LD_{50} represened acute toxicity and potential of short term toxicity of the sample and mortality rate was as a function of the logarithms of the application concentrations. Multiple comparison analysis of variance involved comparing of the average of the hexane and methanolic extracts of O. *pimpinelloides* group with the average of another.

Table 3. Regression Equation, Regression Coefficient, and LD_{50} Values for the Methanolic and Hexane Extract from *Oeneanthe pimpinelloides* on *Tribolium castaneum*

Extraction	Regression Equation	Lethal Dose LD_{50} (µg)
Hexane extract	y = 8.8889x - 10.164	50.81
Methanolic extract	y = 8.8905x - 10.167	48.81

Table 3 shows the resulting values of the lethal doses of methanolic and hexane extract of *O*. *pimpinelloides* against the individuals of *T. castaneum*. The methanolic extract from *O. pimpinelloides* was more toxic to *T. castaneum* than the reported 50 lethal doses of 48.8 (µg) for individuals exposed to contact with the methanolic extract from *O. pimpinelloides* after 5 days. This dose was in the order of 50.8 (µg) for the individuals exposed *via* contact with the hexane extract of *O. pimpinelloides.* Statistical analysis were analyzed with "XLSTAT software. The variance analysis examined with ANOVA that compare the means of the results after the normality test and multiple comparison analysis of variance.

Soufi (2016) reports lethal doses in the order of 30.4 µL/cm² for *Citrullus colocynthis* seed oils against white scale of date palm, while it is 0.0643 mg/cm² for the aqueous extract of the pulp of this same plant after a 24 h exposure period.

CONCLUSIONS

1. In the current study, the phytotoxic effect of the seed extracts of *Oeneanthe pimpinelloides*, a member of the broad Apiaceae family, was determined and its utilization as a crop protectant for pest management in paper packaging applications was investigated.
2. The methanolic and hexane phase-separated extracts were collected from *O. pimpinelloides* seeds. Gas chromatography–mass spectrometry analysis of the extracts was performed, and chemical substances contained in the hexane extract were rich in aromatic and aliphatic hydrocarbons and carbonyl compounds, while the methanol extract was rich in cyclic and acyclic ethers and aliphatic alcohols. To benefit from its pure cellulosic structure and absorbent properties, the extracts were applied to pure filter paper *via* the immersion method. The FTIR of the paper impregnated extracts and base paper samples were compared, and the 1650 cm^{-1} band was characterized as a C=O

stretching vibration of the bond. The FTIR results indicated that the bonds seen in the ethers and alcohols in the carbonyl compounds were also present in the treated paper samples.

3. In particular, the paper samples gained a lethal effect against *Tribolium Castaneum* Herbst. Favorable paper materials provide effective protection for seed paper packaging and grain waste insulation material.

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