Persistence of some Systemic / Non-systemic Pesticides in Potato during Field Application and Food Processing

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ABSTRACT:

This research aimed to study the persistence of some systemic pesticides; dimethoate and methomyl, and non-systemic pesticides; diazinon and mancozeb in potato after field application and impact of different washing and soaking treatments and some thermal processes in removal of tested pesticides. Treated potato samples were collected at (3 hour), 1, 3, 5, 7, 14, and 21 days after pesticides treatment. Potato samples were soaked in tap water and different chemical solutions of acetic acid and NaCl, also potato slices were blanched, cooked by stewing and fried. Data indicated that the average recovery percent of dimethoate and diazinon determined by GC were 94.05 and 95.69. While, in the case of methomyl and mancozeb determined by HPLC were 85.86 and 96.01% in potato; respectively. Systemic pesticides; dimethoate and methomyl showed that high persistence rates in potato tubers after application which dissipated to (0.501 and 0.366 mg/kg) after 14 days of treatment compared with initial residues (3.682 and 3.190 mg/kg); respectively and there were still above the MRLs. Contrary, non-systemic pesticides; diazinon and mancozeb showed that high dissipation rates after application which decreased to (0.068 and 0.109 mg/kg) after 14 days of treatment compared with (4.211 and 3.820 mg/kg) at initial residues and there were below the MRLs. Also washing by soaking in 5% acetic acid solution more effective in pesticide residues removal which removed (51.75% - 61.93%) compared with other washing treatments. In addition, cooking and frying processes caused complete removing of tested systemic and non-systemic pesticide' residues from potato tubers.

Keywords: Pesticides; persistence; Residues analysis; Processing; Potato.

INTRODUCTION

Vegetables are important diet component in different societies, which provide necessary health components such as minerals, vitamin, fiber and phytochemicals. Therefore, vegetables are cultivated on a large scale (Ruzaidy and Azura, 2020). However, vegetables are major sources of pesticides hazards for humans, at a rate five times higher than other methods in ecological system such as such as water and air (Varela-Martinaze *et al.,* 2019 and Mahdavi *et al.,* 2022). Potato *(Solanum tuberosum L.)* is the most worldwide consumed vegetables due to its high nutritional value (Aloo *et al.,* 2020 and Guchi, 2020). During its growth period, the potato crop is exposed to many pests, which requires controlling them using various methods, including chemical pesticides (Yang *et al.,* 2020).

Pesticides are indispensable components of integrated pest management in some cases to control pest infestation that induced a significant loss of yield (Tiryaki and Temur, 2010). Pesticides classification depending on mechanism of action into two categories: a) Systemic pesticides i. e. (dimethoate, methomyl, carbofuran, carbendazim or penconazol) can penetrate into the treated plants tissues (roots, stem, leaves or fruits) through the leaf cuticle or vascular system and translocated throughout skin of plants, b) Non-systemic pesticides i. e. (diazinon, chlorpyrifos, mancozeb or malathion) occurred outside plants, no ability to penetrate into plant cuticle tissue (roots, stem, leaves or fruits) and cannot generally translocated (Tozowicka *et al.,* 2020; Heshmati *et al.,* 2020 and Polat, 2021). Therefore, these residues could easily be removal through soaking and washing process. On the other side, systemic pesticides can penetrate into the different plant tissues, thus it is highly difficult to remove systemic pesticides from different parts of the plants such as fruits, roots, tuber or leaves throughout washing or peeling treatments (Lozowicka *et al.,* 2016 and Acoğlu *et al.,* 2018). Therefore, it is a great interest in the removing or mitigation of pesticides residues in vegetables and decreasing human exposure to these contaminants (Gonzalez-Rodriguez *et al.,* 2011). Polat and Tiryaki, (2020) indicated that non-systemic insecticides (diazinon, malathion, chlorpyrifos) were more efficiently eliminated throughout washing processes than systemic insecticides (penconazole, methomyl, acetamiprid). In addition, Acoglu and Yolci, 2021) reported that pesticide residue reduction is thus largely designated by pesticide mode of action (systemic or non-systemic).

There are a limited number of food preparations or processing treatments involved into pesticide residues elimination in vegetables. Effective removing methods include washing, soaking, ozone treatment, thermal processes and ultrasonic cleaning (Lozowicka *et al.,* 2016). Also, there are various chemical agents such as acetic acid, citric acid, sodium carbonate and sodium hydroxide could be used in washing and soaking treatments. The efficacy of method used in pesticides residues removal dependent on major factors such as chemical and physical properties of the pesticide, pesticide solubility in water, type of processing, temperature, and agricultural commodity produced (Polat and Tiryaki, 2020 and Zhao *et al.,* 2020).

Several studies demonstrates that washing treatments by soaking in different chemical solutions such as citric acid, acetic acid, sodium chloride and ozonated water more than effectiveness in pesticide residues removal compared to washing by tap water (Kentish and Feng 2014; Lozowicha *et al.,* 2016; Anita *et al.,* 2018; Polta, 2021 and Tiryaki and Polat 2023).

Therefore, this research was carried out to investigate the persistence/degradation behavior of some systemic pesticides; dimethoate and methomyl and non-systemic pesticides; diazinon and mancozeb in potato tubers at different intervals post field treatment and study efficiency of several washing treatments and some thermal processes; boiling, cooking and frying in elimination of tested pesticides residues.

MATERIALS AND METHODS

Materials:

Potato tubers *(Solanum tuberosum)* variety (Spunta) was obtained from the experimental field of Central Agricultural Pesticides Laboratory, Giza, Egypt.

Systemic pesticides: (dimethoate and methomyl) and non-systemic pesticides: (diazinon and mancozeb), were purchased from Elhelb Pesticides and Chemicals Company, Damietta, Egypt.

All chemicals, solvents and reagents (with analytical grade) were obtained from Misr Chemical Industries Company, Cairo, Egypt.

Methods:

Field Sampling:

Potatoes were treated with tested pesticides; dimethoate, methomyl, diazinon and mancozeb individually according to the recommended rats of Pesticides Manual (2012). Treated potato samples were separately collected at initial (zero time); at 3 hour after tested pesticides application. Subsequent samples collected at intervals of 1, 3, 5, 7, 14, and 21 days after tested pesticides treatment for investigated the persistence/dissipation behavior of tested systemic and non-systemic pesticides in potato tubers. Also, treated samples at zero time (3 hours) after pesticides treatment were collected for determination the impact of some house-hold treatments and cooking methods on the removal of tested pesticides residues in tested potato samples. Then, the harvested potato samples were kept in polyethylene bags and transferred in the ice boxes to the laboratory for analysis (Romeh *et al.,* 2009).

Methods for pesticides residues removal from tested vegetable:-

Tested potato samples were divided into two parts; one part was analyzed as (contaminated raw sample) without any processing treatments and the anther part was subjected to different washing and some thermal processing treatments to removal the pesticides under investigation as follows:

Washing and soaking treatments:

A peeled potato slice was soaked in tap water, aqueous solutions of acetic acid (2.5 and 5%) and sodium chloride (5 and 10%) at ambient temperature for 5 minutes. Then, soaked samples were rinsed well with tap water until removal acid or salt traces. After that, they drained on a clean paper for 10 minutes at ambient temperature and kept in polyethylene bags and stored under frozen storage condition (at -18±2 ^{o} C) until time of analysis for tested pesticides residues (Sattar *et al.,* 2013).

Thermal processing:

Blanching: Potato were washed in tap water and blanched in hot water at 90±5 ^{o} C for 10 min according to (Kumar *et al.,* 2021).

Cooking process: potato samples were cooked by stewing method at 98 ^{o} C for 30 minutes in open kettle under the atmospheric conditions. After that, the cooked samples were mixed well and let to cool into the ambient temperature and then they kept in polyethylene bags and stored under frozen storage condition (at -18±2 ^{o}C) according to the method of (Thanki *et al.,* 2012).

Deep fat frying process: in this procedure a batch of 100g of potato slices was deep-fried independently in 1L of sunflower oil at temperature of 175±5 ° C. Then potato chips left until reach room temperature. Then samples were kept in polyethylene bags and stored under frozen storage condition (at -18±2° C) (Kaundal *et al.*, 2022).

Pesticide residue removal (%) = Initial residue – retained residue / Initial residue x 100

Analytical procedure for determination of tested pesticides residues:

Extraction:

Extraction of organophosphorus pesticides (diazinon and dimethoate) residues from tested potato samples was performed according to the procedure of (Bowman, 1980). Extraction of carbamate pesticides (methomyl and mancozeb) residues from potato samples was determined according to the method of (Ahmed and Ismail 1995).

Clean up procedure of pesticide residues extract

The cleanup process of tested pesticides' residues extract is performed to remove any interfering substances co-extracted with pesticide residues. For this purpose, a florisil column chromatographic technique was used according to the procedure of (Krynitsky *et al.*, 1988).

Quantitative determination:

Gas chromatography a Philips PU GC Model, 4500, equipped with flame photometric detector operated in the phosphorus mode (526 nm filter) was used for determination of dimethoate and diazinon residues.

Whereas, methomyl and mancozeb residues were determined using HPLC in isocratic system using a Shimadzu Chromatograph including LC-10AS pumps, 20-µl Reodyne injector, SPD-10A UV detector operating at 190-370 nm and a Supelco c18 analytical column (25 cm x 4.6 mm (i. d)).

Recovery assays of tested pesticides:

Control samples of potato tubers were spiked with a known amount (1.0 and 2.0 mg/kg) of dimethoate, diazinon, methomyl and mancozeb before the extraction and clean-up for recovery assay of tested pesticides. The recovery percentages of tested pesticides were calculated by the following equation:

% Recovery = ((µg) present / (µg) added) x 100.

Calculation of the residues:

The residues were calculated using the equation of (Möllhoff, 1975).

Statistical Analysis:

The recorded data were expressed as mean values of three replicates and standard error. The statistical comparison between treatments was performed using a one-way analysis of variance and test significant differences tests (ANOVA) according to the method described by McClave and Benson (1991). Duncan's multiple range tests was also used to test the significant differences between the mean values by using SPSS (version 20.0 software Inc. Chicago, USA).

RESULTS

Validation of the methods used for analysis of tested pesticide residues:

The performance of analytical method used for determination of dimethoate, methomyl, diazinon and mancozeb residues in potato tubers was evaluated and the data were recorded in table (2). Data indicated that the average recovery percentage of organophosphorus pesticides dimethoate and diazinon determined by GLC were 94.05 and 95.69 for tested vegetables. On the other side, in the case of carbamate pesticides methomyl and mancozeb determined by HPLC, these values were 85.86 and 96.01% for potato samples; respectively. Our results are consistent with those of Mohamed (2015) observed that the average recovery percent ranged between 88.34 – 82.31%, 90.22 – 82.55%, 91.38–81.10% and 93.53- 83.35% for methomyl, chlorpyrifos, diazinon and profenofos, respectively in green bean and squash. Also, Aung *et al.* (2016) indicated that a good mean recovery percentage between 87.38 and 88.32% for dimethoate and 85.78 and 92.01% for diazinon in spinach. Saraji *et al.* (2021) reported that the recovery of diazinon and chlorpyrifos were 95.76–99.87% and 90.85– 99.07% in potatoes, respectively.

Residual degradation behavior of tested pesticides in/on potatoes tubers at different intervals post treatment.

The residual levels of the tested pesticides in potatoes tubers at different intervals post field treatment are presented in Table (3). The initial residues of dimethoate, methomyl, diazinon and mancozeb were 3.682, 3.190, 4.211, and 3.820 mg/kg which decreased till reached 3.110, 3.144, 2.236 and 3.216 mg/kg; respectively after one day from pesticides treatment. The residues of tested systemic pesticides; dimethoate and methomyl declined slowly to 1.102 and 0.736 mg/kg at dissipation rates of 70.07 and 76.92 of the initial residue after 7 days from treatment; respectively. Subsequent samples of dimethoate and methomyl residues were (0.501 and 0.366 mg/kg) and (0.92 and 0.189 mg/kg) with loss percentage (86.39 and 88.52%) and (94.78 and 94.07) after 14 and 21 days of field applications; respectively. On the other side, diazinon and mancozeb which are non-systemic pesticides showed that low persistence (high degradable) and dissipated to (0.321 and 0.301 mg/kg) and (0.068 and 0.109 mg/kg) with loss rate (92.37 and 92.12%) and (98.38 and 97.14%) after 7 and 14 days of pesticides treatment; respectively. Diazinon completely dissipated (100%) after 21 days from the field application. Comparing the dissipation rates of the tested pesticides to reach the maximum residue limits (MRLs) it was found to be that dimethoate and methomyl reached below the MRLs (0.5 and 0.2 mg/kg) after 14 and 16.4 days from application, whereas diazinon and mancozeb residues reached below MRLs (0.2 mg/kg) after 8.6 and 8.8 days from application; respectively. Accordingly, potatoes treated with systemic pesticides such as dimethoate and methomyl need a long period (14 to 16 day) to reach the safe limit and therefore they must be not harvested for human consumption until reach the permissible residual limit.

From the obtained data it could be observed that, tested systemic pesticides were more persistence after application than non-systemic. Heshmati *et al.* (2020) reported that degradation rate of non-systemic pesticides like diazinon was greater than that of systemic ones such as dimethoate. Our results are harmony to those reported in previous studies; in tomatoes (Chen *et al.,* 2022); in potatoes (Saraji *et al.,* 2021); in eggplant (Rowayshed *et al.,* 2013) and in grape (Morsy *et al.*, 2022).

Efficiency of different washing treatments and thermal processes on the elimination of tested pesticides residues from potato tubers.

The results for efficiency of various washing treatments and some thermal processes in pesticide residues removal from potato tubers are presented in Table (4). Data indicated that contaminated unprocessed samples contained dimethoate, methomyl, diazinon and mancozeb residues of 3.682, 3.190, 4.211 and 3.820 mg/kg; respectively which are above recommended MRLs. In addition, the effect of washing treatments with tap water and soaking in different chemical solution such as acetic acid and NaCl solutions at different concentration on the tested pesticides in potato tubers as given in Table (4), it could be observed that soaking in acetic acid solutions at concentration of either 2.5 or 5% caused highest removal ranged between (51.75% - 61.93%) for 5% acetic acid and (43.19% - 53.19%) for 2.5% acetic acid from the initial residue in the contaminated fresh samples. With regard, washing by soaking in sodium chloride solutions come in the second order for pesticide residues removal after soaking in acetic acid solutions which removed between (42.35% - 52.29% loss) for 10% NaCl and (34.95% - 44.31% loss) for 5%NaCl in all tested samples. Contrarily, tap water washing recorded a lowest removal of tested pesticides in potato samples between (21.97% - 39.84% loss) of those present in the contaminated fresh samples.

On the other side, thermal processes including blanching, cooking by stewing and deep-fat frying caused complete removing of tested systemic and non-systemic pesticide' residues from potato tubers, with the exception of methomyl residues which elimination by 96.11 in tubers throughout blanching process.

Removal rates of pesticides residues during washing treatments depending on some factors; residues location (outer or inner), mode of action (systemic or non-systemic) and water solubility of pesticide (Acoglue *et al.,* 2018; Polat, 2021 and Tiryaki and Polat, 2023). Also, the elimination effect of the thermal cooking process may be attributed mainly to the oxidative degradation, thermal distruction, hydrolysis and volatilization of pesticides (Kaushik *et al.,* 2009).

Similar results were observed in other studies Liang *et al.* (2012) found that washing by chemical solutions for 20 minutes removed 31.10% to 89.80% of organophosphorus pesticides. Sheikh *et al.* (2015) reported that imidaclopride was eliminated during tap water washing and chemically washing by 42.68% and 45.73%; respectively. Anita *et al.* (2018) observed that cooking and frying processes reduced the pesticide residues by 99.20%. Polat and Tiryaki (2020) indicated that non-systemic pesticides were more efficiently reduced through different washing treatments. While, ultrasonic cleaning process was more effective in elimination of systemic pesticides.

Tiryaki and Polat (2023) found that boiling process more effective in pesticide residues removal compared with washing treatments.

CONCLUSION

Application of systemic pesticides with potatoes needs a long period (14 to 16 day) to reach the safe limit. While, non-systemic pesticides fast dissipated from potato during a short period (8 days), as well as washing by soaking in different chemical solutions play a great role in the elimination of pesticide residues from potato tubers. Soaking in acetic acid solutions was found to be the most effective method in pesticide residues removal compared with other soaking and washing treatments. Also, thermal processes such as boiling, cooking and frying caused a complete removing of systemic and non-systemic pesticides from potato tubers.

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Table 1: The Identification Knowledge of tested pesticides:-

Pesticide	Chemical group	Rate of application*	Mode of action
Dimethoate	Organophosphorus	250 m/100 L/fed	Systemic insecticide and acaricide
Methomyl	Carbamates	300 g/ 100 L/ fed	Systemic insecticide and acaricide
Diazinon	Organophosphorus	150 m /100L /fed	Non-systemic insecticide
Mancozeb	Carbamates	200 g/ 100L /fed	Non-systemic fungicide

* The recommended rates of application according to the Pesticides Manual (2012)

Table 2: Recovery percent of tested pesticides in spiked potato samples.

Pesticides	Spiked level (mg/kg)	Recovery *(%)	Average(%)
Dimethoate	1.0	92.38	94.05
	2.0	95.72	
Methomyl	1.0	82.25	85.86
	2.0	89.48	
Diazinon	1.0	94.15	95.69
	2.0	97.23	
Mancozeb	1.0	94.12	96.01
	2.0	97.90	

* Means with three replicates.

Table 3: Residual dissipation rate of tested pesticides in potatoes tubers at various intervals post treatment:-

Period after treatment (Days)	Systemic pesticides				Non-systemic pesticides			
	Dimethoate		Methomyl		Diazinon		Mancozeb	
	Residue* (mg/kg)	Dissipation (%)	Residue* (mg/kg)	Dissipation (%)	Residue* (mg/kg)	Dissipation (%)	Residue* (mg/kg)	Dissipation (%)
Initial▲	3.682 ±0.057^{a}	0	3.190 ±0.014^{a}	0	4.211 ±0.079^{a}	0	3.820 ±0.290^{a}	0
1	3.110 ±0.105^{b}	15.53	2.236 ±0.025^{b}	29.90	3.144 ±0.024^{b}	25.33	2.955 ±0.130^{b}	22.64
3	2.279 ±0.025^{c}	38.10	1.930 ±0.017^{c}	39.49	2.202 ±0.009^{c}	47.70	1.659 ± 0.088^{c}	56.57
5	1.720 ±0.116^{d}	53.28	1.244 ±0.010^{d}	61.00	1.770 ±0.045^{d}	57.96	0.705 ± 0.046^{d}	81.54
7	1.102 ±0.051^{e}	70.07	0.736 ±0.029^{e}	76.92	0.321 ±0.013^{e}	92.37	0.301 ± 0.015^{e}	92.12
14	0.501 ±0.107^{f}	86.39	0.366 ±0.012^{f}	88.52	0.068 ±0.016^{f}	98.38	0.109± 0.004^{f}	97.14
21	0.192 ±0.006^{g}	94.78	0.189 ±0.005^{g}	94.07	ND	100	0.072± 0.002^{g}	98.11
MRLs (mg/kg)	0.5		0.2		0.2		0.2	
PHI ▲▲ (Days)	14		16.4		8.6		8.8	

*Mean of three replicates ± Standard error; ▲ Three hour after pesticides application; (MRLs): Maximum residue limit according to FAO/WHO Codex Alimentarius Commission (2010); ▲▲ PHI: Pre-Harvest Intervals.

Table 4: Effect of washing treatments and some thermal processes on pesticide residues removal from potatoes tubers:-

Pesticide / Treatment			Systemic pesticides				Non-systemic pesticides			
			Dimethoate		Methomyl		Diazinon		Mancozeb	
			Residue (mg/kg)	Removal (%)	Residue (mg/kg)	Removal (%)	Residue (mg/kg)	Removal (%)	Residue (mg/kg)	Removal (%)
Contaminated fresh samples *			3.682 ^{a} ± 0.057	0	3.190 ^{a} ± 0.014	0	4.211^{a} ± 0.079	0	3.820^{a} ± 0.290	0
MRLs (mg/kg) **			0.5		0.2		0.2		0.2	
Tap water washing			2.714 ^{b} ± 0.320	26.29	2.489 ^{b} ± 0.216	21.97	2.533 ^{b} ± 0.240	39.84	2.587 ^{b} ±0.113	32.27
Chemically washing (Soaking for 5 min. in)	Acetic acid solution	2.5%	1.994 ^{d} ± 0.118	45.84	1.812 ^{d} ± 0.052	43.19	1.971 ^{c} ± 0.214	53.19	1.998 ^{c} ± 0.102	47.69
		5%	1.592 ^{e} ± 0.215	56.76	1.539 ^{e} ± 0.056	51.75	1.603 ^{d} ± 0.110	61.93	1.596 ^{d} ± 0.045	58.21
	NaCl solution	5%	2.311^{c} ± 0.118	37.23	2.075 ^{c} ± 0.023	34.95	2.345 ^{b} ± 0.102	44.31	2.309 ^{b} ± 0.080	39.55
		10%	2.014 ^{d} ± 0.025	45.30	1.839 ^{d} ± 0.074	42.35	2.009 ^{c} ± 0.024	52.29	1.981^{c} ± 0.010	48.14
Blanching (at 90±5 ° C for 10 min)			ND	100	0.124 ^{f} ± 0.003	96.11	ND	100	ND	100
Cooking (open kettle) (at 98 ° C for 30 minutes)			ND	100	ND	100	ND	100	ND	100
Deep fat frying (at175±5 ° C for 5 min)			ND	100	ND	100	ND	100	ND	100

* Mean of three replicates ± Standard error for pesticide residues; the means within the same column having different superscripts are significant varied. ** MRLs: Maximum Residue limit for pesticides according to Codex Alimentarius Commission FAO/WHO, (2010).

ثباتية بعض المبيدات الجهازية وغير الجهازية فى البطاطس خلال التطبيق الحقلى والتصنيع الغذائى

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الملخص العربي:

يهدف هذا البحث إلى دراسة ثباتية بعض المبيدات الجهازية (ديمثويت و ميثوميل) وغير الجهازية (ديازينون و مانكوزيب) فى البطاطس بعد المعاملة فى الحقل ومدى تأثير بعض معاملات الغسيل والنقع المختلفة وكذلك بعض المعاملات الحرارية فى إزالة بقايا المبيدات المختبرة. تم جمع عينات البطاطس على فترات مختلفة (3 ساعات) ، 1 ، 3 ، 5 ، 7 ، 14 ، 21 يوم بعد المعاملة بالمبيدات. تم إجراء نقع للبطاطس فى ماء الصنبور ومحاليل كيميائية مختلفة من حمض الخليك وكلوريد الصوديوم ، أيضاً تم اجراء عمليات السلق والطهى والقلى لشرائح البطاطس المختبرة. أوضحت النتائج أن متوسط نسبة الإسترجاع لكل من الديمثويت والديازينون كانت 94,05% و 95,69% بينما كانت تلك النسبة 85,86% و96,01% فى الميثوميل والمانكوزيب فى عينات البطاطس على التوالى. أظهرت المبيدات الجهازية (ديمثويت ، ميثوميل) معدلات ثبات عالية فى درنات البطاطس بعد المعاملة حيث تلاشت تلك المبيدات الى 0,501مجم/كجم ، 0,366 مجم/كجم بعد 14 يوم من المعاملة مقارنة بالتركيز المبدئى 3,682 مجم/كجم ، 3,190 مجم/كجم على التوالى وكانت لا تزال فوق الحدود القصوى المسموح بها. على العكس من ذلك أظهرت المبيدات غير الجهازية (ديازينون ، مانكوزيب) معدلات تحطم او تلاشى عالية حيث تناقصت بمعدلات اسرع الى 0,068مجم/كجم ، 0,109مجم/كجم بعد 14 يوم من المعاملة مقارنة بمحتواها 4,211 مجم/كجم ، 3,820 مجم/كجم فى العينة الاولية على التوالى وكانت اقل من الحدود القصوى المسموح بها. أيضاً لوحظ أن الغسيل بالنقع فى محلول 5% حمض خليك كانت أكثر فاعلية فى ازالة بقايا المبيدات المختبرة حيث ازالت حوالى (51,75% - 61,93 %) مقارنة بمعاملات الغسيل الأخرى. فى هذا السياق لوحظ أن عمليات الطهى والقلى أحدثت إزالة كاملة لبقايا المبيدات الجهازية وغير الجهازية المختبرة من درنات البطاطس.

الكلمات الاسترشادية: المبيدات الجهازية, تصنيع البطاطس.