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Original Research

How Soil-Applied Maltodextrin with Caraway (*Carum carvi* L.) Oil Affects Weed and Soil Microbiological Activity in Maize (*Zea mays* L.) Stands

Agnieszka Synowiec[1]*, Anna Lenart-Boroń[2], Jan Bocianowski [3], Andrzej Lepiarczyk[1], Danuta Kalemba[4]

[1]Department of Agroecology and Crop Production, University of Agriculture in Kraków, Poland
[2]Department of Microbiology, University of Agriculture in Kraków, Poland
[3]Department of Mathematical and Statistical Methods, Faculty of Agriculture and Bioengineering, Poznań University of Life Sciences, Poznań Poland
[4]Faculty of Biotechnology and Food Chemistry, Institute of General Food Chemistry, Łódź University of Technology, Łódź, Poland

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Abstract

The essential oil (EO) of caraway (*Carum carvi* L.) is a confirmed source of herbicidal compounds. Therefore, the object of this study was to determine the effect of soil-applied maltodextrin (MD) microcapsules containing approximately 12% caraway EO on weed infestation in maize (*Zea mays*) stands, the yield of maize and the number of colonies of soil microbes. A two-year field experiment was set up in a randomized block design on brown podzolic soil. It was shown that the MD-EO microcapsules affected the number of only some annual weeds, such as *G. quadriradiata*, *G. parviflora*, and *E. crus-galli*. The dry mass of monocotyledonous and dicotyledonous weeds was significantly reduced by all the MD treatments, on average by 45-65% and 35-65%, respectively, compared to the control. The number of maize plants and cobs per 1 m^2 was reduced by 17% and 21%, respectively, following the MD-EO treatments. The mass of the cobs was unaffected by the MD-EO treatments. The application of MD-EO caused a significant decrease in the number of soil mesophilic bacteria colonies but did not affect the amounts of phenolic compounds in the soil. In conclusion, the soil application of caraway EO within MD microcapsules has a potential as a natural weed control agent but should be studied further to optimize its dose and timing of application for weed control in maize stands.

Keywords: microcapsules, phenolic compounds, soil microbiota, yield

*e-mail: a.synowiec@urk.edu.pl

Introduction

The need for sustainable plant protection in agricultural and horticultural systems promotes the utilization of natural products, including essential oils (EOs), as natural pesticides. EOs can play a role as botanical pesticides or alleloherbicides used for pest control, and constitute an alternative to synthetic pesticides [1-3]. Furthermore, the biologically active components of EOs and other substances of plant origin might be valuable sources of new pesticidal substances in the future [4]. As several studies have shown, EOs inhibit weed seed germination [1, 3, 5] but also cause necrosis when applied to leaves [6]. Caraway EO is one of the candidates for the production of botanical herbicides. The fruits of caraway (*Carum carvi* L.) contain relatively high amounts of EO (3-5%), which is mainly composed of two phytotoxic monoterpene compounds, d-limonene and carvone [7, 8]. Caraway oil displays strong antigerminative effects against weed seeds germination in laboratory experiments [8, 9], and at the same time, this oil is one of the cheapest among the many EOs available on the market in Poland.

Unfortunately, most of the experiments that have tested the herbicidal potential of essential oils have been carried out in laboratory conditions and have very rarely been verified in field conditions. This is because the main restriction of applying EOs in the field is their volatility, which significantly reduces their span of biological activity. This problem may be overcome through the microencapsulation of essential oils, in which coating a micro-drop of essential oil is carried out with different solid compounds [10]. The purpose of microencapsulating an essential oil is to slow its release without losing its biological properties [11-13]. Microencapsulation is applied broadly for food preservation from food borne bacteria and fungi [10, 14]. There are also a few studies on the phytotoxic potential of microencapsulated essential oils. Scarfato et al. [15] tested the antigerminative activity of the vapors released from polyurea microcapsules containing EOs of sage (*Salvia officinalis* L.), lemon balm (*Melissa officinalis* L.), lavender (*Lavandula angustifolia* Mill.) or thyme (*Thymus vulgaris* L.) oils against the germination of *Raphanus sativus*, *Lepidium sativum*, and *Lactuca sativa*. Alipour et al. [16] found that encapsulated in starch and soil-applied rosemary EO caused a significant decrease in germination and morphophysiological features of *Amaranthus retroflexus* and *Raphanus sativus* under greenhouse conditions. Silicon dioxide microcapsules containing caraway oil were applied in field conditions by Synowiec et al. [17]. The authors found that the application of the microcapsules to the soil exerted an inhibitory effect on the number of dicotyledonous weeds, but also that there were significant injuries to the maize plants – especially during dry weather [17]. The above-mentioned experiments also point to the important role of carriers for the essential oils. One of the most common carriers used in food products is maltodextrin, which is usually combined with a small addition of arabic gum [18,19]. Maltodextrin is a polysaccharide with a high solubility in water, low viscosity and sugar content, and is colorless [20]. Synowiec et al. [21] analyzed, in a pot experiment, the phytotoxic activity of soil-applied maltodextrin microcapsules containing essential oils of peppermint (*Mentha x piperita* L.), caraway (*Carum carvi* L.) or calamus (*Acorus calamus* L.) on the initial growth of *Zea mays*, *Echinochloa crus-galli* and *Chenopodium album*. They found that the highest doses of microencapsulated EOs (200 kg ha^{-1}, containing approximately 24 kg of EO), especially caraway or peppermint oil, reduced the number and dry weight of weeds and did not impair the initial growth of maize [21].

The side effect of soil-applied essential oils could be an increase in the content of phenolic compounds in the soil, which could be an allelopathic response of the soil microbes to the treatments. According to Inderjit [22] and Li et al. [23], phenolic compounds are the main indicators of the response of plants to allelopathic stress.

Therefore, the objectives of this study were to evaluate the influence of soil-applied maltodextrin microcapsules containing the essential oil of caraway on i) the weeds in the maize stand, ii) the maize yield and iii) the microbiological activity and phenolic content in the soil. The research hypothesis was that soil-applied essential oil of caraway encapsulated in a maltodextrin solid carrier inhibits the growth of weeds but does not affect the growth of maize or the soil microbiological composition.

Material and Methods

Chemical and Morphometric Analyses of the Microcapsules

The EO of caraway fruits was purchased from the Avicenna Oil company (Wrocław, Poland), and maltodextrin (MD, 85.5 % with 4.5 % of arabic gum E 414) was used as the carrier. The MD microcapsules containing caraway EO were prepared on an industrial scale by the Hoffmann Aroma company (Zamysłowo, Poland) via the process of spray drying. The final content of caraway EO in the microcapsules was measured by the hydrodistillation method (20 g of microcapsules and 60 mL of water) for 3 h using a Clevenger-type apparatus in the Institute of General Food Chemistry, Lodz University of Technology. The volume of the separated EO was multiplied by the specific density of the microcapsules, which was determined by the pycnometer method.

The chemical composition of the EO in the MD microcapsules was analyzed by gas chromatography-mass spectrometry (GC-MS) using a Trace GC Ultra apparatus (Thermo Electron Corporation, Milan, Italy) equipped with a flame ionization detector

(FID) and MS DSQ II detector. A simultaneous GC-MS/FID analysis was performed using an MS-FID splitter (SGE, Analytical Science). The operating conditions were as follows: apolar capillary column Rtx-1ms (Restek), 60 m × 0.25 mm i.d., film thickness 0.25 µm; temperature program, 50-310ºC at 4ºC min^{-1}; SSL injector temperature 280ºC; FID detector temperature 300ºC; split ratio 1:20; helium carrier gas at 300 kPa pressure. Mass spectra were acquired over the mass range of 30-400 Da with an ionization voltage of 70 eV and ion source temperature of 200ºC. The identification of the components was based on a comparison of their MS data and retention indices (RI) with data stored in the computer libraries, NIST 98.1, Wiley 275.1, and Mass Finder 4.1. The RI were determined according to homologous series of alkanes (C8-C26) analyzed under the same conditions with linear interpolation. The percentages were obtained from the FID response without using correction factors [24].

The microcapsule morphology was studied with scanning electron microscopy (SEM) (JSM-5800 LV, JEOL, Tokyo, Japan) at a 15 kV acceleration voltage.

Field Experiment

In 2014 and 2015, a one-factorial field experiment was carried out at the Experimental Units of the University of Agriculture in Mydlniki-Krakow (50°4'49''N and 19°50'43''E). This experiment was established on brown podzolic soil [25] (pH 7.5, organic C content 5.6%, P_2O_5 11.2 mg 100 g^{-1} soil and K_2O 14.0 mg 100 g^{-1} soil). In November 2013, the soil was fertilized with manure (30 t ha^{-1}) and mineral fertilizers (47.9 kg P ha^{-1} and 74.7 kg K ha^{-1}), and the mineral fertilizer was applied again in November 2014. The experimental design was a randomized block design with four replications on 9 m^2 plots. Maize cv. Wilga (FAO 180) was sown on May 5, 2014, and on May 8, 2015, with 40 cm row spacing; there were 7 rows of maize per plot. Nitrogen (urea) fertilizer was applied in two doses: before sowing at a dose of 100 kg N ha^{-1} and as a dressing (urea form) in BBCH 15-17 [26] at a dose of 40 kg ha^{-1}.

Two types of microcapsules were used in the field experiment: with or without caraway (MD-EO or MD), and each was applied at two doses: 0.5 t ha^{-1} (containing 60 kg ha^{-1} of EO; dose 1) or 1.0 t ha^{-1} (containing 120 kg EO ha^{-1}; dose 2), based on the previous pot experiment [21]. The microcapsules were first manually spread on the soil surface and then, on the same day, mixed with the soil using a cultivator at a depth of approximately 10 cm. The MD was applied the same day as the maize was sown; MD-EO, the day before. In addition, there were two control treatments, C, untreated control; and herbicide control (H), foramsulfuron and iodosulfuron-methyl-sodium (Maister 310 WG, Bayer Sp. z o.o., PL, of 22.5 and 0.75 g a.i. ha^{-1}, respectively), which were applied according to recommendations by the producer for timing and dose.

Weed Analysis

The weed infestation of the maize plots was evaluated twice during the growing season using the standard methods of primary and secondary weed infestation assessment [27]: 1) at an early growth stage of the weeds and maize (June 1, 2014 and June 5, 2015) by counting the number of weed seedling in a 0.25 m^2 area (frame 250 am long and 10 cm wide); the results were calculated per 1 m^2 and 2) a week before maize harvesting, when all weeds were cut from 1 m^2, separated into monocotyledonous and dicotyledonous species, air dried and weighed.

Analysis of the Physiological State of the Maize

On July 1, 2014, and July 16, 2015, during the developmental stage of the maize stem (BBCH 35) [26], the physiological state of the maize was analyzed by measuring i) the leaf greenness index (SPAD), which indicates the nitrogen content in the leaves [28] and ii) the parameters of chlorophyll *a* fluorescence (FL), which indicate the early stress response in the plants [29]. The measured FL parameters included F_v/F_m (maximum yield of photosystem II) and PI (performance index). F_v/F_m shows the proportion of light used in the photochemical processes that is absorbed by chlorophyll in photosystem II, whereas PI is a complex parameter of the overall photosynthetic performance of plants under different types of stresses [29]. These measurements were carried out on the 6th leaf of six plants from the middle rows; SPAD was evaluated using a SPAD 502DL meter (Konika-Minolta Europe), and FL was determined with a Handy PEA fluorometer (Hansatech Instruments, UK) 20 min after covering the fragment of leaf using special clips. The duration of light was 1 s; gain, 0.7; and actinic light, 3000 µmol $m^{-2}s^{-1}$.

Yield of Maize Cobs and Plant Biomass

Maize was harvested when the cobs were fully developed (BBCH 89) [26] on 18 September 2014 and 1 September 2015. During the harvest, the four middle rows of maize were cut, and all plants and cobs were counted and weighed; the results were expressed as g m^{-2}.

Analysis of Soil Microorganisms, Soil pH and Total Phenolic Content of the Soil

The number of colonies of select soil microorganisms was analyzed on July 9, 2014 and 2015, which was 65 and 62 days after applying the microcapsules to the soil, respectively. A soil sample (0-30 cm) was taken from each of the four replications (plots) using a soil probe, and the samples were thoroughly mixed in a bucket. Then, soil samples that were approximately 0.5 kg were packed

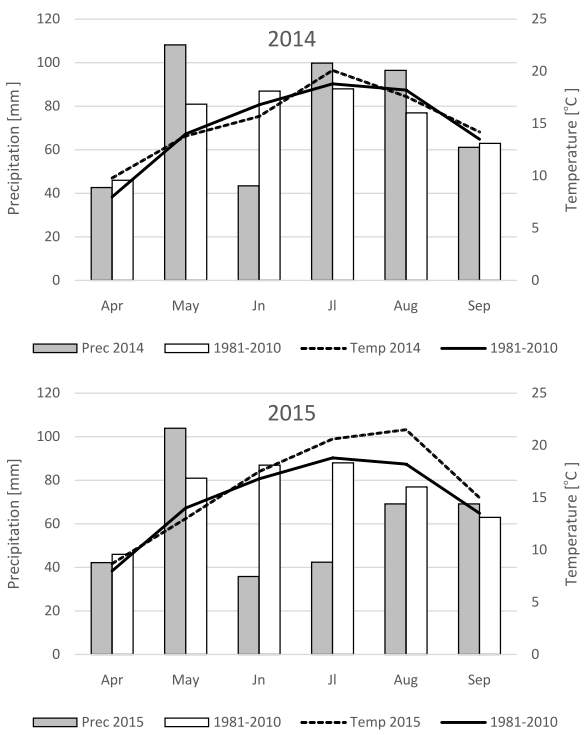

Fig. 1. Weather course in Mydlniki Experimental Station during vegetation time of maize in the seasons 2014-2015, in comparison to a multiyear period 1961-1990.
Apr – April; Jn – June; Jl – July; Aug – August; Sep – September; Prec – precipitation; Temp – temperature.

in zip-lock plastic bags and transferred to the laboratory for analyses on the same day. Each bulk sample of soil was divided into three 10 g sub samples, and before the analysis, the soil samples were sieved through a 2 mm sieve. The number of colonies of microorganisms was determined by plating with the soil dilution method. For the sterile saline solutions, serial dilutions from 10^{-1} to 10^{-6} were prepared, 1 mL of each was poured into Petri dishes with microbiological media suitable for culturing the microorganisms, and the total number of mesophilic bacteria was determined on nutrient agar following a 48 h incubation at 37ºC. Counts of fungal colonies were evaluated after a 3-day incubation at 28ºC on malt agar and those of actinomycetes following a 7-day incubation at 28ºC on Pochon agar. The results (means of three replicates) are shown as CFU (colonies forming units) of microorganisms per 1 g of soil. The pH of the soil was determined in distilled water in a 1:2.5 solution using an electronic pH meter with a glass electrode (model CP-205, Elmetron, Poland).

The experiment was finished in September 2015, and then the amount of total phenolic compounds in the soil was measured. From each plot, three samples of soil, taken with a soil probe at a depth of 0-30 cm, were collected and mixed. The soil was then air dried and sieved using a 2 mm sieve. Next, 5.0 g of the soil sample was extracted with 10 mL of 80% ethanol and shaken for 1 h, and then 2 mL of the soil extract was centrifuged for 15 min at 4,000 *g* at 3ºC. The extract was condensed to 1 mL in a CentriVap concentrator (LABCONCO, Kansas City, Missouri, USA), half of which, together with 0.5 mL of water, 0.5 mL of carbonate (0.25 H %) and 0.125 mL of Folin-Ciocalteu [30], was used to determine the phenolic concentration (µg g^{-1} dry weight, DW) with a spectrophotometer (Perkin Elmer, Norwalk, CT, USA). The absorbance was measured at 290 nm, and gallic acid was used as a standard.

Weather

The temperature and precipitation during the 2014 and 2015 growing seasons were compared to those in the 1981-2010 period (Fig. 1) using weather station data from Mydlniki [31]. The weather conditions for maize growth were more favorable in 2014 than in 2015. In April 2014 and 2015, the weather conditions were comparable with those of 1981-2010, whereas May was more humid. In 2015, June and August were hot and dry with higher mean temperatures and lower precipitation (approximately 2ºC and 8-50 mm, respectively) compared to those of 1981-2010.

Statistical Analysis

Two-way ANOVA was performed using GenStat v. 18 (VSN International Ltd, UK). The data were tested for both normality of distribution and homogeneity of variance, according to Shapiro-Wilk and Levene tests, respectively. The nonhomogenic data were log-transformed and analyzed. The means were compared with Fisher's LSD test ($p \leq 0.05$).

Results

Physical and Chemical Characteristics of the Microcapsules

The microcapsules contained 7.5% caraway EO composed of d-limonene (40 %) and carvone (60%) (Fig. 2). Scanning electron microscopy showed that

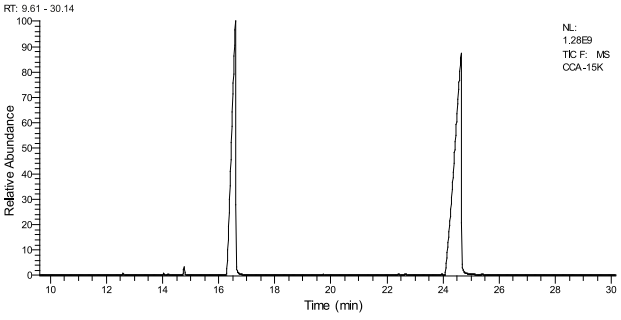

Fig. 2. Chromatogram of the chemical composition of caraway (*Carum carvi* L.) essential oil isolated from the maltodextrin microcapsules

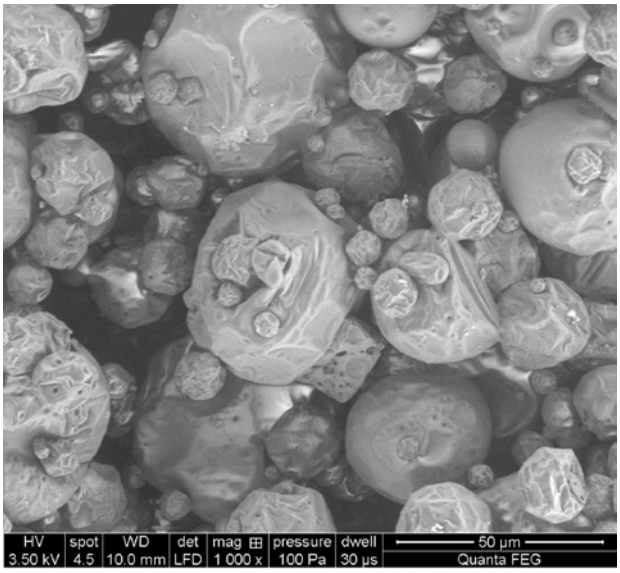

Fig. 3. SEM photos of the microcapsules with maltodextrin as the carrier.

morphologically, the microcapsules were a mixture of differently sized particles (Fig. 3).

Influence of MD Microcapsules on Weed Infestation in Maize Stands

We observed that some weed species were more sensitive to MD-EO microcapsules than others. Specifically, in spring 2014, *Galinsoga quadriradiata*, *Cirsium arvense* and *Myosotis arvensis* were most sensitive to the higher dose of microcapsules, whereas two main monocotyledonous species – *Elymus repens* and *Echinochloa crus-galli* – were sensitive to both doses of microcapsules with EO. In spring 2015, *Galinsoga parviflora* was the most sensitive species to the higher dose of microcapsules with EO (Table 1).

The overall number of monocotyledonous weeds following the application of herbicides and microcapsules was similar for both studied years ($p = 0.9$) (Fig. 4a). In both years, compared to the untreated control (C), the application of the herbicide was the most effective weed control method, reducing the number of monocotyledonous weeds by 80%. The application of the microcapsules with the caraway EO reduced, on average, the number of monocotyledonous weeds by approximately 20-40% compared to the control (Fig. 4a). The number of dicotyledonous weeds was different for both years, with treatments being significantly more effective, by about 30%, in the cooler 2014 season (Fig. 4b). Similar to the monocotyledonous weeds, the herbicide treatment was most effective for the control of dicotyledonous weeds; the application of herbicide reduced weeds by about 60% compared to the control. Interestingly, the application of 2 MD-EO had a similar effect on weeds as the control (C) and application of maltodextrin alone (Fig. 4b).

By the end of the maize vegetative period, the weed biomass was affected by the application of the microcapsules compared to the C (Fig. 5a-b). At that stage, the long-term effect of the herbicide on the weeds was much more profound compared to that of both MD-EO treatments. The dry mass of monocotyledonous and dicotyledonous weeds was significantly reduced by all the MD treatments, on average by 45-65% and 35-65% compared to C, respectively (Fig. 5a-b). Nevertheless, the most effective treatment against weeds was the application of the herbicide (Fig. 5a-b).

Table 1. List of the dominating weed species per 1m^2 in spring 2014 and 2015 as their percentage amount in relation to the control

Year	2014						2015					
Treatment	C	H	1MD	2MD	1MD-EO	2MD-EO	C	H	1MD	2MD	1MD-EO	2MD-EO
	Monocotyledonous weeds percentage											
Echinochloa crus-galli L. (Beauv.)	100	70	175	71	67	31	100	46	70	124	134	95
Elymus repens (L.) Gould.	100	68	88	72	41	41	100	93	130	100	60	80
	Dicotyledonous weeds percentage											
Amaranthus retroflexus L.	100	80	140	70	93	80	100	38	138	58	63	88
Chenopodium album L.	100	100	275	333	150	250	100	40	125	140	75	80
Cirsium arvense (L.) Scop.	100	56	50	0	67	0	100	107	0	0	40	80
Galinsoga parviflora Cav.	100	181	124	83	60	95	100	37	122	94	89	56
Galinsoga quadriradiata Ruiz & Pav.	100	87	290	228	174	65	100	0	0	0	0	0
Myosotis arvensis (L.) Hill	100	0	150	50	50	0	0	0	0	0	0	0

Abbreviations: C – control; H – herbicide control, MD –maltodextrin carrier; MD-EO – maltodextrin with caraway essential oil; 1 and 2 – a lower and a higher dose of powder.

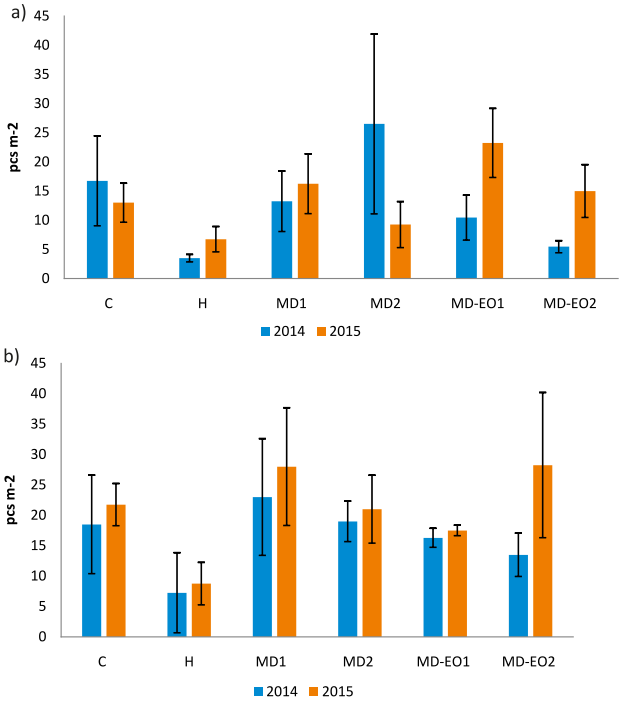

Fig. 4. Number of a) monocotyledonous and b) dicotyledonous weeds per 1 m^2 following the soil application of MD microcapsules with or without the addition of caraway EO; whiskers represent ± standard error (n = 4)
Pand LSD values for two-way ANOVA performed for monocotyledonous weeds: p_{year} = 0.87; $p_{treatment}$<0.001***, LSD = 6.2; $p_{interaction}$ = 0.08.
P and LSD values for two-way ANOVA performed for dicotyledonous weeds: p_{year} = 0.02*, LSD = 3.5; $p_{treatment}$=0.005***, LSD = 6.0; $p_{interaction}$ = 0.08.
Abbreviations: C – control; H – herbicide control, MD –maltodextrin carrier; MD-EO – maltodextrin with caraway essential oil; 1 and 2 – a lower and a higher dose of powder; p = significance level; LSD – least significant difference.

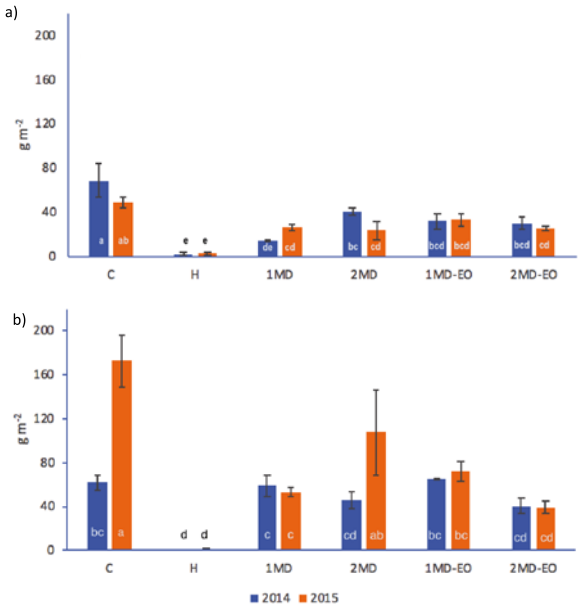

Fig. 5. Dry mass of a) monocotyledonous and b) dicotyledonous weeds (gm^{-2}) as measured prior to the harvest of maize cv. Wilga and following the spring soil-application of maltodextrin microcapsules with or without the addition of caraway essential oil; whiskers represent ± standard error n = 4
P and LSD values for two-way ANOVA performed for monocotyledonous weeds: p_{year} = 0.3; $p_{treatment}$<0.001***, LSD = 14.8; $p_{interaction}$ = 0.05*, LSD = 20.9.
P and LSD values for two-way ANOVA performed for dicotyledonous weeds: p_{year} = 0.004***, LSD = 19.3; $p_{treatment}$<0.001, LSD = 33.4; $p_{interaction}$ = 0.005, LSD = 47.3.
Abbreviations: C – control; H – herbicide control, MD – maltodextrin powder; MD-EO – maltodextrin powder with caraway essential oil; 1 and 2 – a lower and a higher dose of powders; p = significance level; LSD – least significant difference.

Effect of the Microcapsules on Maize Physiological State

Significantly higher values of physiological parameters (Fv/Fm, PI and SPAD) were noted in 2014 for the maize treated with the herbicide (H) compared to those for the C, MD and MD-EO treatments (Table 2). However, the soil application of the MD and MD-EO microcapsules did not negatively affect the physiological parameters of maize during both growing seasons, and all the tested parameters were in the optimal range (Table 2).

Effect of the Microcapsules on Maize Yield

The number of maize plants per 1 m^2 was significantly higher in the more weather-favorable season of 2014 and ranged between 7.8 and 10.6 plants per 1 m^2; the range was 5.5-8.5 plants per 1 m^2 during the less weather-favorable 2015 season (Table 3). The 2MD-EO microcapsule treatment significantly decreased the number of maize plants and cobs per 1 m^2 by 17% and 21%, respectively, compared to C. However, the mass of the cobs per 1 m^2 was similar and not significantly different among the MD-EO treatments and C and H controls (Table 3).

The Number of Microbiological Colonies and Concentrations of Phenolic Compounds in the Soil

The soil pH in the second year of the application of MD and MD-EO microcapsules was in the pH range 6.6-6.7 and was not significantly different from that of the control plots (C and H).

The soil-applied MD and MD-EO microcapsules significantly affected the number of colonies of soil microbes during both years (Table 4). On average, a significantly higher number of bacterial and actinomycete colonies was observed in September 2015 than in 2014. In September 2014 and 2015, the soil content of mesophilic bacteria colonies in the plots treated with MD-EO microcapsules was significantly lower than that of the C plots, by 80% and 70%,

Table 2. Selected physiological parameters of maize following the soil application of maltodextrin microcapsules with or without the addition of caraway essential oil.

	Fv/Fm						SPAD		
	2014	2015	Mean	2014	2015	Mean	2014	2015	Mean
C	0.768ab±0.004	0.719bc±0.004	0.744ab	2.33bc±0.15	2.77bc±0.14	2.55b	36.7b ±1.0	40.8ab±1.4	38.8ab
H	0.767ab±0.009	0.759ab±0.012	0.763a	2.18c±0.17	4.50a±0.29	3.34a	38.3ab±0.5	44.0a±1.9	41.1a
1MD	0.779a±0.003	0.678d±0.039	0.729b	2.39bc±0.14	2.88b±0.37	2.64b	38.8ab±3.4	38.1ab±1.9	38.5ab
2MD	0.784a±0.002	0.713bc±0.024	0.748ab	2.42bc±0.12	2.45bc±0.39	2.44b	36.8b±1.7	35.9b±1.5	36.4b
1MD-EO	0.774ab±0.004	0.716bc±0.005	0.745ab	2.21bc±0.27	2.52bc±0.31	2.36b	37.1b±1.9	40.0ab±3.1	38.5ab
2MD-EO	0.771ab±0.004	0.736bc±0.009	0.753a	2.16c±0.07	2.73bc±0.23	2.44b	38.5ab±3.3	37.6b±1.2	38.1ab
$LSD_{0.05}$	0.042	0.029	0.69	0.28	6.02	4.26			
Mean	0.774a	0.772a		2.28b	2.97a		37.69a	39.41a	

Table contains mean values ± standard error (n = 6).
C – control; H – herbicide control, MD – maltodextrin carrier; MD-EO – maltodextrin with caraway essential oil; 1 and 2 – a lower and a higher dose of powder. Fv/Fm - the maximum yield of photosystem II; PI – Performance Index, SPAD – relative chlorophyll content in leaves.

respectively. However, changes in the number of colonies of soil fungi and actinomycetes treated with MD-EO microcapsules in relation to the C soil were not clear and depended on the season (Table 4).

In September 2015, after 2 years of applying MD and MD-EO microcapsules, the lowest content of phenolic compounds was noted in the soil treated with the high concentration of the carrier (2MD, Table 4).

Discussion

The process of coating the caraway EO with maltodextrin did not affect the chemical composition of the EO in the resulting microcapsules. The prevailing compounds in the tested caraway EO were carvone and limonene, and their concentrations in oil were similar to those found by other authors [32,33].

The effect of microencapsulated caraway EO against the weeds in this field experiment was much less profound than the effect of microencapsulated caraway oil in the pot experiment as observed by Synowiec et al. [21]. The number of weeds in the spring, following the application of microcapsules, was different in each of the studied years. However, we observed that some weed species, i.e., *Galinsoga* sp., were more sensitive than others to the soil application of MD-EO microcapsules. It is difficult to determine the reason

Table 3. Number of plants and yield of cobs of maize (per 1 m^2) following the soil application of maltodextrin microcapsules with or without the addition of caraway essential oil.

	Number of plants (pcs m^{-2})			Number of cobs (pcs m^{-2})			Mass of cobs [kg]		
	2014	2015	Mean	2014	2015	Mean	2014	2015	Mean
C	10.6a±0.6	6.9def±0.5	8.8a	8.1ab±0.2	5.9cde±0.4	7.0ab	0.8bcd±0.03	0.5def±0.04	0.63abc
H	10.0ab±0.4	6.9def±0.8	8.5ab	8.7a±0.7	6.0cde±0.7	7.3a	1.0ab±0.1	0.7bcde±0.08	0.83ab
1MD	7.8cde±0.8	6.4ef±0.1	7.1c	6.4bcde±0.9	4.9e±0.5	5.7c	0.6cdef±0.1	0.4ef±0.03	0.46c
2MD	9.3abc±0.8	8.5bcd±0.1	8.9a	8.0ab±0.2	5.5de±0.8	6.7a	0.8bcd±0.1	0.4ef±0.06	0.63abc
1MD-EO	9.4abc±0.4	6.2ef±0.8	7.8abc	7.0abcd±0.3	4.5e±0.7	5.8bc	1.3a±0.3	0.4ef±0.08	0.86a
2MD-EO	9.2abc±0.6	5.5f±1.3	7.3bc	7.5abc±0.7	3.4f±0.9	5.5c	0.9bc±0.1	0.3f±0.09	0.60bc
$LSD_{0.05}$	1.95	1.38	1.81	1.28	0.33	0.24			
Mean	9.4a	6.7b		7.6a	5.0b		0.9a	0.5b	

Table contains mean values ± standard error (n = 4).
C – control; H – herbicide control, MD –maltodextrin carrier; MD-EO – maltodextrin with caraway essential oil; 1 and 2 – a lower and a higher dose of powder.

Table 4. Number of selected groups of microorganisms [*cfu*×10^5/g of soil] and total phenolics [µg/g dm of soil] in soil after maize harvest (September) following the soil application of maltodextrin microcapsules with or without the addition of caraway essential oil.

	Mesophilic bacteria			Fungi			Actinomycetes			Total phenolics
	2014	2015	Mean	2014	2015	Mean	2014	2015	Mean	2015
C	12.7±0.21 bc	54.3±1.27 a	33.5 a	4.34±0.37 a	1.93±0.23 bc	3.14 a	2.31±0.10 g	140±8.43 b	71.2 b	1.42±0.06 a
H	5.77±0.49 d	6.50±0.70 cd	6.14 d	1.12±0.10 c	2.55±0.47 ab	1.84 b	3.02±0.31 g	209±5.00 a	106 a	1.13±0.03 b
1MD	9.05±0.59 c	23.2±1.59 b	16.1 b	1.26±0.58 c	0.50±0.10 d	0.88 c	2.50±0.54 g	68.9±1.85 d	35.7 d	1.21±0.03 b
2MD	12.2±0.99 bc	8.43±1.44 c	10.3 c	2.61±0.61 ab	0.93±0.07 c	1.77 b	2.25±0.55 g	87.4±4.30 c	44.8 c	0.90±0.03 c
1MD-EO	2.15±0.55 e	6.20±0.58 cd	4.18 d	0.59±0.06 d	2.95±0.41 ab	1.77 b	12.7±0.21 f	32.5±2.41 e	22.6 e	1.21±0.04 b
2MD-EO	1.28±0.60 e	19.6±0.56 b	10.4 c	0.80±0.06 cd	0.83±0.07 cd	0.82 c	2.14±0.55 g	97.5±3.67 c	49.8 c	1.51±0.05 a
Mean	7.19b	19.7a		1.79a	1.62b		4.15b	105a		

Statistical analysis (2-way ANOVA) was performed on log-transformed data. Table contains rough data, mean values ± standard error (n = 3). Numbers followed by different letters differ significantly
C – control; H – herbicide control, MD –maltodextrin carrier; MD-EO – maltodextrin with caraway essential oil; 1 and 2 – a lower and a higher dose of powder; *cfu* – colonies forming units.

of different weed species sensitivity to the soil-applied microcapsules with caraway oil. This phenomenon could be partially explained by the higher susceptibility of small-sized seeds of weeds to the essential oils. As observed by Synowiec et al. [8] in laboratory conditions, weeds with smaller seeds are more prone to be inhibited by essential oils, which would be the case for both *Galinsoga* species.

Similar to weed numbers, the soil-applied microcapsules caused a reduction in weed biomass, and the monocotyledonous weeds were more sensitive than the dicotyledonous weeds. The inhibitory effect of the MD microcapsules on weed biomass was also observed in the pot experiment carried out by Synowiec et al. [21], but in the controlled conditions of the pot experiment, where the inhibitory potential of the microencapsulated essential oils was more visible than that in the field experiments.

Soil application of the MD and MD-EO microcapsules did not negatively affect the physiological parameters of chlorophyll content and photosynthesis in maize leaves in both growing seasons, and all the tested parameters (namely, Fv/Fm, PI and SPAD) were within the range of the optimal values [29]. In contrast, an inhibitory effect of the application of the essential oils was observed on the number of maize plants and cobs in 2015. This effect could partially be a result of the unfavorable weather conditions, especially drought, during the 2015 season. Maize is drought-sensitive, and a decrease in maize yield of approximately 34 % was observed in the Czech Republic during the dry season in 2015 compared to that in 2014 [34].

The soil-applied microcapsules containing caraway essential oil also caused a shift in the number of microbiological colonies in the soil. The soil content of mesophilic bacteria colonies in the plots treated with MD-EO microcapsules was significantly lower than that of the C plots. However, the number of colonies of fungi and actinomycetes in the soil treated with MD-EO microcapsules depended more on the seasonal effects. Vokou et al. [35] showed that soil microorganisms might use EOs as a source of carbon and energy. According to their research, the application of EOs to the soil may shift the balance of microorganisms in the soil, thereby increasing the number of bacteria and simultaneously decreasing the number of soil fungi [35]. However, we did not observe any influence of the application of microcapsules on the content of phenolic compounds in the soil. An increased content of phenolic compounds in the soil is indicative of the allelopathic activity of the soil [36]; therefore, our results show that the application of microcapsules did not affect the soil biological balance.

Conclusions

The research hypothesis stated in this work can be partially accepted, as soil-applied MD-EO microcapsules act selectively against weed species. On the other hand, the microcapsules with caraway oil impair the yield of maize cobs. In the temperate climate of southern Poland, if the level of precipitation during the vegetative season (April-September) is typical for the region, the soil application of microcapsules containing

caraway essential oil inhibits the number of colonies of soil mesophilic bacteria and fungi. Regarding both statements above, the research hypothesis assuming lack of effect of MD-EO microcapsules against maize growth and microorganism numbers has to be rejected. Nevertheless, the use of microcapsules with caraway oil and maltodextrin as the carrier should be studied further in order to optimize the dose and timing of application for weed control in maize.

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Conflict of Interest

The authors declare no conflict of interest.

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