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Effect of Fatty Acid Methyl Esters on the Herbicidal Effect of Essential Oils on Corn and Weeds

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In this study, we tested whether the addition of fatty acid methyl esters (FAME) of edible oils would influence the herbicidal effect of the essential oils (EO) of fiber hemp and peppermint (*Mentha* × *piperita* L.) against common lambsquarters, barnyardgrass, and corn. The herbicidal properties of a 2.5% concentration of each EO in water mixtures with FAME were evaluated as sprays in a pot experiment. The oil-FAME mixtures showed phytotoxic effects against common lambsquarters and barnyardgrass expressed by a reduction in plant length and aboveground and root biomass, as measured three weeks after foliar spraying. Corn was the most tolerant species to the tested mixtures. Sunflower FAME alone was safe on corn but reduced the growth of weeds. Peppermint EO alone was the most phytotoxic on all tested species. In conclusion, the mixture of peppermint EO with oilseed rape FAME was the best treatment; however, improvement on *Ch. album* would be desirable for commercial-level control.

Nomenclature: Barnyardgrass, *Echinochloa crus-galli* (L.) Beauv. ECHCG; common lambsquarters, *Chenopodium album* L. CHEAL; common sunflower, *Helianthus annuus* L.; corn, *Zea mays* L.; hemp, *Cannabis sativa* L.; peppermint, *Mentha* × *piperita* L. Huds. var. *officinalis* Sole, f. *rubescens* Camus; rape, *Brassica napus* L.; soybean, *Glycine max* (L.) Merr.

Key word: Phytotoxicity.

En este estudio, evaluamos si la admisión de fatty acid methyl esters (FAME) de aceites comestibles influencia el efecto herbicida de los aceites esenciales (EO) del cáñamo y la menta contra *Chenopodium album*, *Echinochloa crus-galli*, y maíz. Las propiedades herbicidas de una concentración de 2.5% de cada EO en mezclas acuosas con FAME fueron evaluadas en un experimento con potes. Las mezclas de aceite-FAME mostraron efectos fitotóxicos contra *C. album* y *E. crus-galli* expresados como una reducción en el largo de la planta y la biomasa aérea y de raíz, medidas tres semanas después de la aspersión foliar. El maíz fue la especie más tolerante a las mezclas evaluadas. FAME de girasol solo fue seguro en el maíz, pero redujo el crecimiento de las malezas. EO de menta solo fue el más fitotóxico de todas las especies evaluadas. En conclusión, la mezcla de EO de menta con FAME de colza fue el mejor tratamiento. Sin embargo, una mejora en la actividad sobre *C. album* sería deseable para alcanzar un nivel de control comercial.

In light of recent literature, essential oils (EOs) can be used effectively to control weeds (Amri et al. 2013; De Almeida et al. 2010; Vasilakoglou et al. 2013), and these substances are a valuable alternate commercial opportunity for application in organic weed control in small areas, such as kitchen gardens or municipal greeneries, due to the relatively high costs of these products. There are a few other factors that limit a wider application of EOs in weed control. One of them is the chemical composition of the EOs, because it has been shown that the main components of EOs responsible for their phytotoxic effect, which inhibit the germination of weeds, are monoterpenes (Vokou et al. 2003). The EOs composed mainly of other chemical groups are characterized by a lower phytotoxicity

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(Rolli et al. 2014; Vasilakoglou et al. 2013). Regardless of the demonstrated ability of EOs to cause permanent damage to the aerial parts of weeds (Stokłosa et al. 2012), these substances are not easily used for foliar application due to their very poor water solubility (Baser and Buchbauer 2010) and their poor ability to create a uniform spray mixture (Synowiec and Drozdek, 2016). Furthermore, these compounds are highly volatile, so they are easier to study in well-controlled laboratory experiments carried out in closed dishes. There are significantly fewer experiments related to the phytotoxic effect of EOs after foliar applications in field conditions, such as that by Brainard et al. (2013). Therefore, to increase the effectiveness of a foliar application of EOs, it is necessary to find an adjuvant that improves their physicochemical properties. In the previously mentioned study by Brainard et al. (2013), the authors improved the efficacy of clove [*Syzygium aromaticum* (L.) Merr. & L.M. Perry] oil using an organic adjuvant, Humasol® (Agricar, Inc.), which is available on the US market.

Several other commercial products, also available on the US market and based on the herbicidal activity of EOs, contain edible oils or their fatty acid methyl esters (FAMEs), as adjuvants (e.g., Matran® EC, EcoSmart Technologies, Inc.). In our own glasshouse experiment, we obtained a satisfactory herbicidal effect of caraway (*Carum carvi* L.) EO mixed with a Polish commercial adjuvant, Atpolan® Bio (Agromix ZPH, Niepołomice, Poland), which contains 80% of the methyl esters of rapeseed oil (Synowiec and Drozdek 2016).

In various industries, FAMEs are considered to be good adjuvants (Salimon et al. 2012; Wang et al. 2015), as well as effective solvents for EOs (Abo El-Seoud et al. 2005). FAMEs are also important components of modern biodiesels (Giakoumis 2013). Most of these substances are currently increasingly popular because they are environmentally friendly and are therefore classified as "all-green adjuvants". Due to their construction, FAMEs have a high affinity for fatty compounds (Wang et al. 2015), and therefore, they easily penetrate the plant cuticle and also improve the adhesion of the spray mixture to the leaf surface. The herbicidal effect of commercial herbicides can be significantly improved by the addition of edible oils or FAMEs to a tank mixture (Anyszka and Dobrzański, 2002; Lim et al. 2012; Manthey et al. 1992), or by the addition of a commercial adjuvant containing FAME as a component (Menendez et al. 2011).

The aim of this study was to evaluate the herbicidal effectiveness of the EOs of fiber hemp or peppermint with FAMEs of edible oils, using foliar application in a mixture with water, against selected weeds and corn in a pot experiment. The EOs were selected based on their contrasting chemical compositions: monoterpenes in the EO of peppermint (Mahdavikia and Saharkhiz 2015), and hydrocarbons in the EO of fiber hemp (Synowiec et al. 2016). The weeds tested in this experiment represent the most troublesome mono- and dicotyledonous weeds in corn (Gołębiowska et al. 2015). Corn was selected for this study because of the growing popularity of its cultivation in Central Europe (Elsgaard et al. 2012).

Material and Methods

Preparation of the Substrates. The EOs of two species, grown in a temperate climate of Poland and collected from arable fields in the growing season of 2013, were used: fiber hemp ('Białobrzeskie') and peppermint. The EO of fiber hemp inflorescences was steam-distilled on an industrial scale at the Institute of Natural Fibres and Medicinal Plants in Poznan, Poland. The EO of peppermint herb in full vegetative growth was steam-distilled using a Clevenger-type apparatus in the Department of Chemistry at the University of Agriculture in Krakow, Poland. After distillation, the EOs were stored in vials of dark glass in cool conditions until the start of the experiment.

FAMEs were obtained from the edible oils commercially available in Europe, oilseed rape, soybean, and sunflower (all from ZT Kruszwica SA, Poland), in the laboratory of the Chair of Food Plant Chemistry and Processing, Faculty of Food Sciences, University of Warmia and Mazury in Olsztyn, Poland, by the methylation of vegetable oil with methyl alcohol in the presence of a potassium hydroxide catalyst. A 500-mL round-bottom flask was filled with 400 mL of vegetable oil and heated to 60 C using a heating mantle. The flask content was stirred with a magnetic stirrer. Next, 90 mL of a 1 N KOH solution was added to the heated oil and stirred for 30 min. The reaction mixture was then transferred to a separatory funnel, and the top fraction containing FAMEs was separated off.

The resulting product was filtered through activated bleaching earth (Tonsil® Supreme 112FF, TS, Clariant, Munich, Germany) in order to clean it (Richardson 1978).

Chemical Analysis of EOs and Methyl Esters. The chemical composition of the EOs was analyzed using gas chromatography–mass spectrometry (GC-MS). After dilution in diethyl ether (10 µL in 1 mL), the EOs were analyzed using a Trace® GC Ultra apparatus (Thermo Electron Corporation, Milan, Italy) with a flame ionization detector (FID) and mass spectrometry DSQ II detector. A simultaneous GC-FID and MS analysis was performed using an MS-FID splitter (SGE, Analytical Science, Victoria, Australia). The operating conditions were as follows: apolar capillary column Rtx-1ms (Restek), 60 m length, 0.25 mm internal diameter, 0.25 µm film thickness; temperature program, 50 to 310 C at 4 C min^{-1}; split/splitless injector temperature, 280 C; FID detector temperature, 300 C; split ratio, 1:20; and helium carrier gas at regular pressure, 300 kPa. Mass spectra were acquired over the mass range of 30 to 400 Da, ionization voltage of 70 eV, and an ion source temperature of 200 C. Identification of the components was based on a comparison of their MS data with computer libraries NIST 98.1, Wiley 275.1, and MassFinder 4.1, along with the relative retention indices (apolar column) associated with a series of alkanes (C_8 to C_{26}), with linear interpolation. Percentages were obtained from the FID response without the use of correction factors.

Methyl esters were dissolved in hexane and analyzed by gas chromatography with a GC-MS QP2010 PLUS system (Shimadzu, Japan). Separation was performed on a BPX 70 capillary column (25 m by 0.22 mm by 0.25 µm; SGE Analytical Science, Victoria, Australia) with helium as the carrier gas, at a flow rate of 0.9 mL min^{-1}. The column temperature was programmed as follows: an increase from 150 C to 180 C at the rate of 10 C min^{-1}, to 185 C at the rate of 1.5 C min^{-1}, and to 250 C at the rate of 30 C min^{-1}, and then held constant for 10 min. The interface temperature of the GC-MS was set at 240 C. The temperature of the ion source was 240 C, and the electron energy was 70 eV. The total ion current mode was used in 50 to 500 m/z range. The fatty acids were identified based on the retention times of fatty acid standards purchased from Sigma-Aldrich (Poznan, Poland).

Preparation of Spray Mixture. Mixtures containing suspensions of each of the EOs and FAMEs were prepared using tap water. The given amounts relate to the doses per hectare. EO at 2.5% (w/w) was mixed with methyl esters of a selected fatty acid in the amount equal to 1.5 or 2.0 L ha^{-1} (w/w) and with tap water (200 L ha^{-1}); the resulting mixtures were visually transparent. The mixture was prepared just prior to spraying; it was intensively shaken by hand a few times and applied manually on the leaves of corn and weeds using a TeeJet® flat nozzle placed at the tip of a syringe.

Pot Experiment. A pot experiment with three replications was established in the series starting on April 7, 2014 and April 1, 2015. Pots with an upper diameter of 17 cm and a capacity of 1.5 L were filled with a sieved top layer of arable sandy soil (pH_{KCl}, 5.25; P_2O_5, medium; K_2O, low; Mg, very low).

In each pot, several seeds of each species were sown: corn ('Wilga') and two common species of weeds of corn, common lambsquarters and barnyard grass. After emergence, the number of seedlings in each pot was reduced to five. When the plants reached the three- to five-leaf stage, they were sprayed with an appropriate spray mixture. In total, there were twelve different treatment combinations: water (control), water plus oilseed rape FAME, water plus soybean FAME, water plus sunflower FAME, water plus fiber hemp EO, water plus fiber hemp EO plus oilseed rape FAME, water plus fiber hemp EO plus soybean FAME, water plus fiber hemp EO plus sunflower FAME, water plus peppermint EO, water plus peppermint EO plus oilseed rape FAME, water plus peppermint EO plus soybean FAME, and water plus peppermint EO plus sunflower FAME.

Plant Measurements. Symptoms of leaf damage (injured tissues, necroses on the leaves) were observed at two time points: 3 h and 24 h after spraying. Three weeks after spraying, the plants were cut at the ground level, and their length was measured (in the case of corn and barnyardgrass, from the base to the end of the straightened leaf, and in the case of common lambsquarters, to the apex). Roots were shaken to remove the soil and washed thoroughly. The aboveground parts and roots of plants in each pot were packed separately in paper bags and dried at 50 C in an oven for 72 h. The dried parts of the plants were weighed on an electronic scale with an accuracy of 0.01 g. Then, the average length of plants, the average weight of the aboveground parts per plant, and the average weight of roots per plant were calculated.

Statistical Analysis. In a first step, the significant difference between the experiment series was tested, and because it turned out to be statistically insignificant, both series were pooled. Similarly, because no statistically significant differences between the doses of FAMEs (1.5 or 2.0 L ha^{-1}) were found, the results were pooled. The effects of the treatment combinations on the dry weight of plants were analyzed using a one-way ANOVA for the main effects. The significant differences between the mean values were tested by Tukey's test at $P \leq 0.05$.

Multivariate statistical analysis was performed, which included all species and all treatment combinations based on canonical variate analysis (CVA). In order to verify the differences between groups, multivariate analysis of variation (MANOVA) was applied because the groups of variables were independent. Verification of the assumptions was performed using Wilk's λ test. Additionally, a Hotelling test was used to compare the pairs of variables, with a Bonferroni correction. To determine the treatment combinations that showed a reduction in the length and weight of plants, ternary graphs were plotted. The analyses were performed using the statistical software STATISTICA version 12.0 (StatSoft, Inc., Tulsa, OK) and PAST (Hamer 2001).

Results and Discussion

Chemical Compositions of EOs and Fatty Acids. Chemically, the tested EOs contrasted with each other. The main fraction of fiber hemp EO comprised monoterpene hydrocarbons (73.3%) and sesquiterpene hydrocarbons (21.8%). In the peppermint EO, oxygenated monoterpenes constituted 91.2% of the total oil. The composition of EO is similar to previous reports (Costa et al. 2013; Ross and ElSohly 1996).

Among the fatty acid components of edible oils, the oleic and linoleic fatty acids dominated, in ratios of 1:2, 1:2, and 3:1 for soybean, sunflower, and rapeseed, respectively. The other fatty acids accounted for 12.8% to 24.8% of the total oils (Table 1).

Effect of Treatment on the Injuries of Plants. Fifteen minutes following the treatment, clusters of larger liquid droplets of spray formed on leaves of the plants. Thus, no treatment combination evenly coated the leaf surface (Figure 1), which suggests that the mixtures had poor wetting abilities. The first symptoms of injury resulting from treatment—dying leaf tissues—appeared 3 h following spraying, and symptoms were of comparable intensity at 24 h after spraying (Figure 2). As proven by Tworkoski (2002), EOs show toxic effects similar to those of the contact herbicides, which result in a dying of tissue in the

Table 1. Composition (%), formula, and molecular weight of fatty acid methyl esters of three edible oils.

Fatty acid/ Methyl ester	Formula	Molecular weight	Oilseed rape avg	Oilseed rape SD	Soybean avg	Soybean SD	Sunflower avg	Sunflower SD
Palmitic/ methyl palmitate	$C_{16}H_{32}O_2$/ $C_{17}H_{34}O_2$	256.432/ 270.459	5.24	0.38	10.36	0.85	7.77	0.49
Palmitooleinic/ methyl palmitooleate	$C_{16}H_{30}O_2$/ $C_{17}H_{32}O_2$	254.432/ 268.459	0.26	0.01	0.09	0.01	0.15	0.09
Stearic/ methyl stearate	$C_{18}H_{36}O_2$/ $C_{19}H_{38}O_2$	284.486/ 298.513	1.99	0.12	5.91	0.41	4.64	0.12
Oleinic/ methyl oleate	$C_{18}H_{34}O_2$/ $C_{19}H_{36}O_2$	296.513/ 338.594	61.87	4.96	25.39	1.24	28.79	1.25
Linoleic/ methyl linoleate	$C_{18}H_{32}O_2$/ $C_{19}H_{34}O_2$	280.486/ 294.513	20.47	1.00	49.82	2.95	58.38	3.98
Gamma-linolenic/ methyl linolenate	$C_{18}H_{30}O_2$/ $C_{19}H_{32}O_2$	278.486/ 292.513	-	-	0.44	0.02		-
Alpha-linolenic/ methyl linolenate	$C_{18}H_{30}O_2$/ $C_{19}H_{32}O_2$	278.486/ 292.513	8.57	0.59	7.80	0.53	0.08	0.01
Eicozenic/ methyl eicozenate	$C_{20}H_{38}O_2$/ $C_{21}H_{40}O_2$	310.540/ 324.567	1.60	0.07	0.19	0.04	0.19	0.01

Abbreviations: avg, average; SD, standard deviation.

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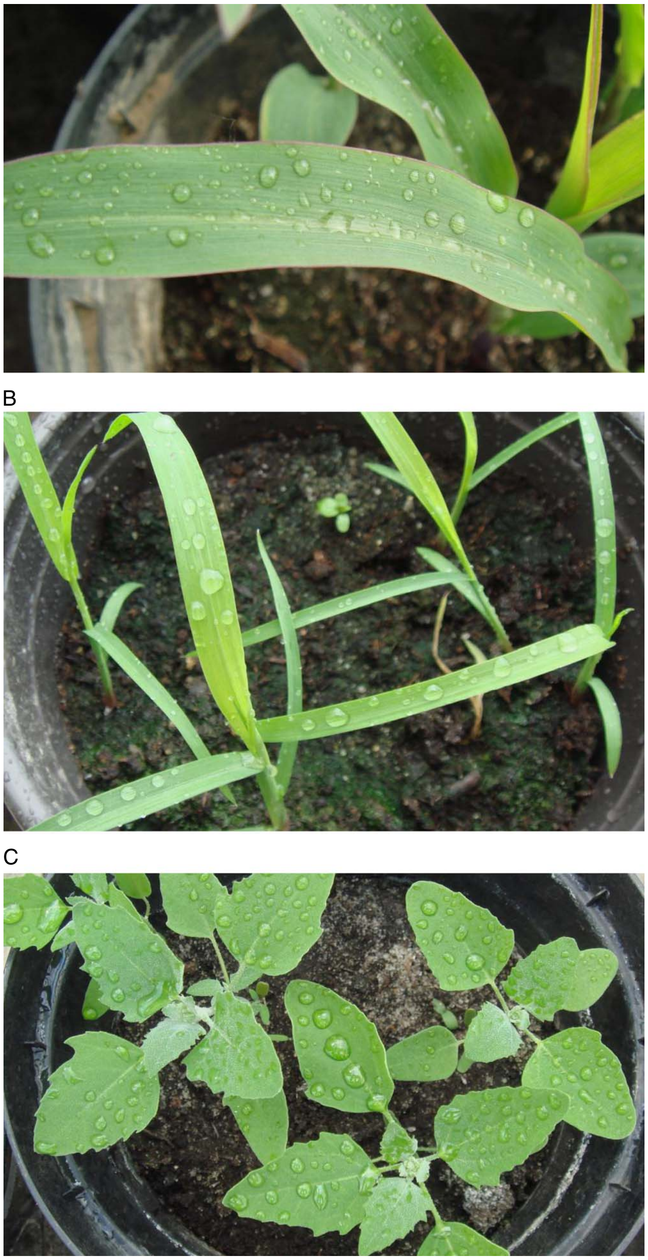
A
B
C


Figure 1. Leaves sprayed with mixtures of water plus essential oils (EOs) and fatty acid methyl esters (FAMEs), 15 minutes after treatment. A) Corn treated with peppermint EO plus oilseed rape FAME, B) barnyardgrass treated with water plus fiber hemp EO; C) common lambsquarters treated with fiber hemp EO plus soybean FAME.

spot of a direct contact with the leaf. According to Synowiec et al. (2015), in the case of highly phytotoxic clove EO, which is a component of a few natural herbicides, the first sign of injury on broccoli leaves appears as early as 20 min following its application. Generally, the degree of leaf injury was higher in all plants treated with peppermint EO, regardless of the combination. In this case, visible necroses were observed on leaves where the drops of the mixture contacted the leaf. More injuries were observed on the leaves of common lambsquarters, which were positioned horizontally, compared to injuries on the leaves of grasses, which were positioned at an acute angle relative to the shoot (Figure 2). Synowiec and Drozdek (2016) also observed that peppermint EO, alone or in mixtures with commercial adjuvants, causes stronger injuries to leaf tissues of common lambsquarters, and causes much weaker injuries to the monocotyledonous weed wild oat (*Avena fatua* L.) In corn, the drops of spray mixtures were observed on leaves for a longer time only on the flat, wide leaves; in the case of narrow leaves of barnyardgrass, the drops of a treatment mixture either dripped from them or flowed along the leaf-blade to accumulate at the base. Three weeks after spraying, no clear injuries were noted on plants anymore, and new, undamaged leaves grew.

Effect of Spraying on Length and Dry Mass of Plants. Spraying the plants with the treatment combinations caused short-term growth inhibition and a reduction in biomass accumulation, which ultimately affected the length and dry weight of plants, as measured 3 wk after spraying. Canonical variate analysis enabled an isolation of groups of species differing in relation to the action of the treatment combinations (MANOVA, $F = 79.58$, Wilk's test, $\lambda = 0.22$). Additionally, a Hotelling test with Bonferroni correction was used to compare the pairs of species. The most varied results were obtained for corn ($P < 0.01$), while the least varied were obtained for common lambsquarters, indicating that common lambsquarters was the most sensitive to the applied spray combinations. Barnyardgrass showed an intermediate susceptibility to the spray combinations (Figure 3).

Multiple regression analysis revealed a significant, positive correlation between the length of the plants and the aboveground dry weight for each species: corn, $r = 0.74$, $P = 0.001$; barnyardgrass, $r = 0.81$, $P = 0.001$; and common lambsquarters, $r = 0.66$, $P = 0.001$. Thus, the aboveground dry weight parameter is shown in the bar graphs because it best represents the phytotoxic effect of treatment combinations against the tested species (Figure 4). Spraying

Figure 2. Leaf injury of selected species 12 hours after treatment with selected essential oils.

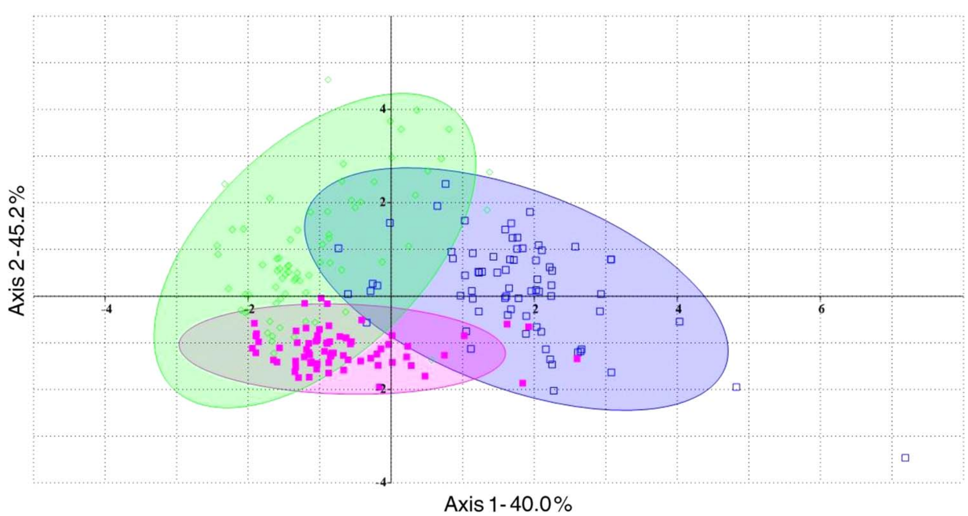


Figure 3. Canonical variate analysis showing a correlation between length and dry matter of corn □, common lambsquarters ◇, and barnyardgrass ■ following treatment with water mixtures containing the essential oils of peppermint or fiber hemp with the addition of methyl esters of fatty acids as adjuvants.

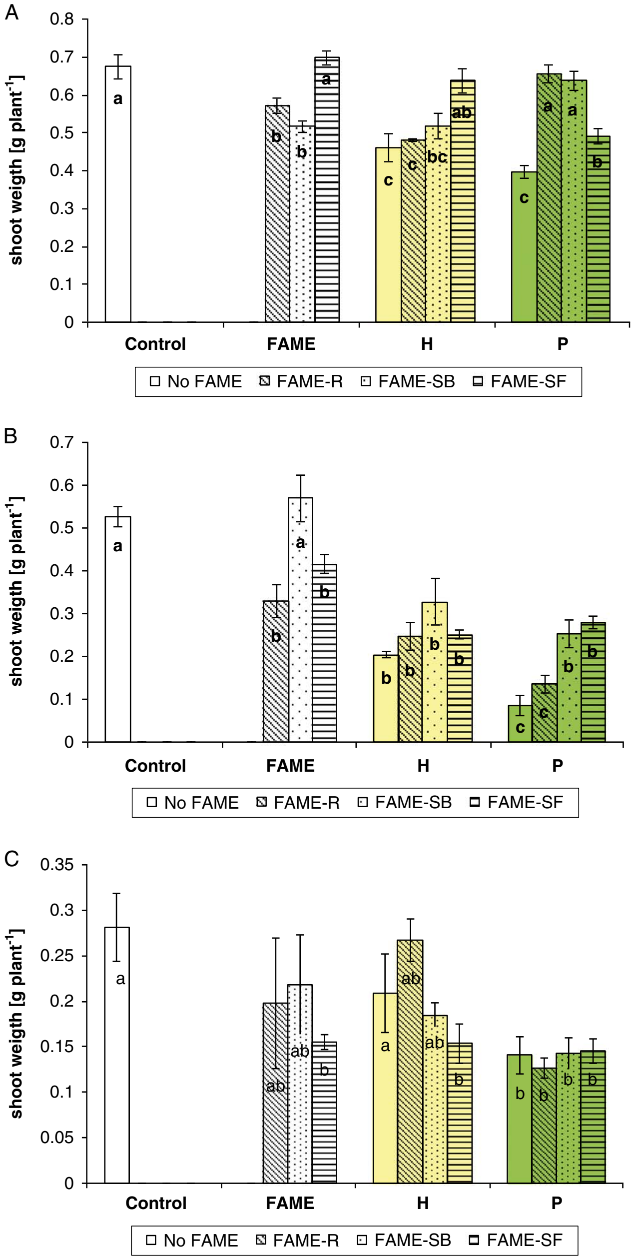


Figure 4. Effect of spray mixtures on the dry mass of a single plant of A) corn, B) barnyardgrass, and C) common lambsquarters, 3 weeks after spraying. The bars represent the mean value for two series of experiments ± SE (n = 6). Different letters indicate significant differences in relation to the control sprayed with water, according to Tukey's test at $P < 0.05$. Abbreviations: FAME, fatty acid methyl esters; H, fiber hemp essential oil; P, peppermint essential oil; R, oilseed rape; SB, soybean; SF, sunflower.

plants with a mixture of water and FAME caused a significant reduction in the plants' dry weight compared to that of plants receiving the control treatment. Only sunflower FAME was safe on corn (Figure 4A). Both rapeseed and soybean FAMEs caused a 30% reduction of corn dry weight. Additionally, soybean FAME was safe on barnyardgrass (Figure 4B). Common lambsquarters was the most susceptible to the sunflower FAME treatment (Figure 4C). Peppermint EO alone was the most active treatment, with the most phytotoxicity on each of the three species, and was more active than any mixture (Figure 4A–C). The average weight reduction following the peppermint EO treatment was 12%, 58%, and 51%, for corn, barnyardgrass, and common lambsquarters, respectively, whereas in the case of the fiber hemp EO, there were reductions of 20%, 48%, and 28% for corn, barnyardgrass, and common lambsquarters, respectively. The other authors demonstrated a strong herbicidal potential of peppermint EO against weed germination (Cavalieri and Caporali 2010; Mahdavikia and Saharkhiz 2015), which is due to its chemical composition—mainly oxygenated monoterpenes (Rolli et al. 2014). Fiber hemp EO, containing mainly mono- and sesquiterpene hydrocarbons, displays a weaker phytotoxic effect against germinating seeds (Synowiec et al. 2016).

The addition of EOs to methyl esters caused a further reduction in the dry weight of these species, compared to the control treatment and FAMEs alone. Diversity in the sensitivity of species to combinations of EOs and FAMEs was noted. In general, the reduction in dry weight of corn was lower than that of the weeds. Moreover, both oilseed rape as well as soybean FAME were safe with peppermint EO on corn and barnyardgrass. Among the weeds, a greater reduction in aboveground weight was observed for barnyardgrass, especially when the peppermint EO was used with oilseed rape FAME (Figure 4B). Interestingly, the sunflower FAME sprayed on common lambsquarters had a comparable effect on the reduction of the dry weight of this species as did the sunflower FAME with fiber hemp EO or peppermint EO (Figure 4C).

Ternary graphs (Figure 5) display all three measured characteristics of the tested species—plant length and weight of shoots and roots, in percentage values. Based on the presented data, it is clear that in the case of corn and barnyardgrass, peppermint EO alone was the most phytotoxic treatment, especially in relation to the root weight (corn) and shoot weight (barnyardgrass). For barnyardgrass, the most effective mixture was peppermint EO combined with oilseed

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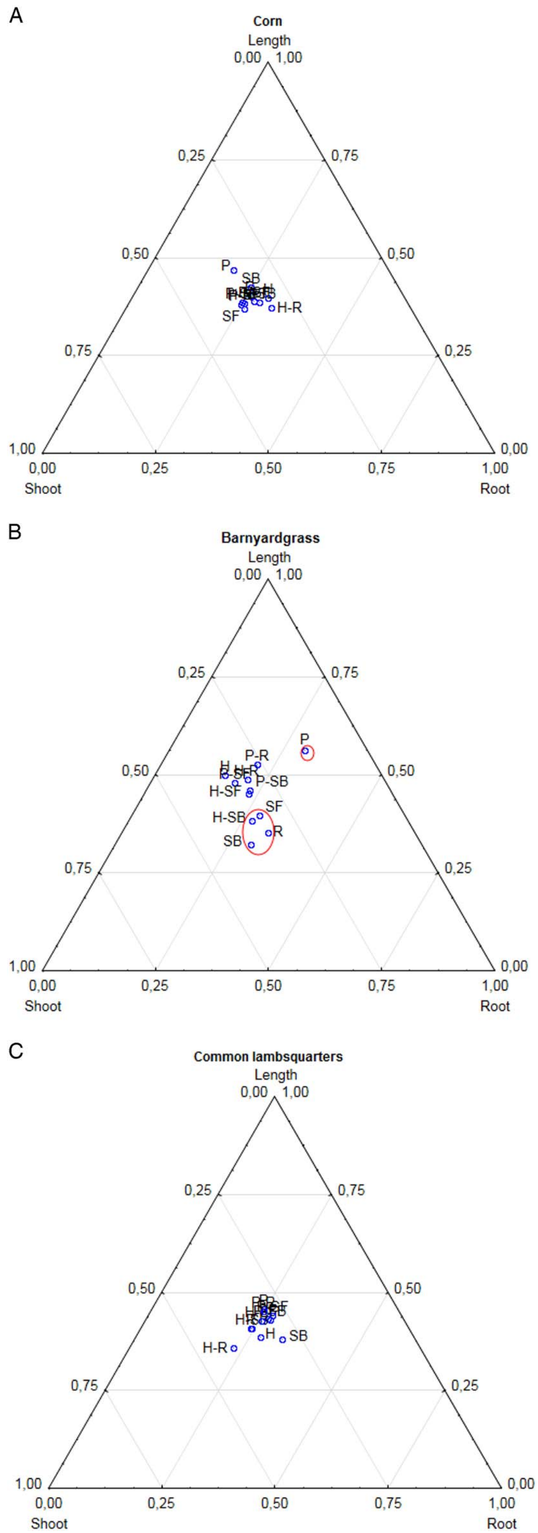
rape FAME, whereas soybean FAME was the least phytotoxic (Figure 5B). In case of common lambsquarters, the least phytotoxic was a mixture of fiber hemp EO and oilseed rape FAME (Figure 5C).



Manthey et al. (1992) found that a donor species of FAME may affect both the herbicide efficiency, when applied as an herbicide adjuvant, and the plant susceptibility. In the cited study, the addition of sunflower FAME to quizalofop herbicide improved herbicide performance against oats; on the other hand, the addition of linseed (*Linum usitatissimum* L.) FAME to the same herbicide decreased its performance against oats (Manthey et al. 1992). In our experiment, it was shown that the addition of FAMEs to peppermint EO reduces it phytotoxicity. However, the mixture of peppermint EO and oilseed rape FAME was the most effective against weeds and safe on corn. Among the treatments with fiber hemp EO, the most effective treatment against weeds that was also safe on corn was the mixture with sunflower FAME. This finding could indicate that the phytotoxic properties of a particular EO can be improved by mixing it with an appropriate adjuvant. Similar conclusions were reached by Synowiec and Drozdek (2016), who applied foliar sprays containing EOs of peppermint or caraway with commercial adjuvants as alleloherbicides against weeds. These authors found that the herbicidal performance of caraway oil can be improved by an appropriate adjuvant, in this case Atpolan® Bio (Agromix). At the same time, these studies showed that peppermint EO showed a strong phytotoxicity against weeds, regardless of the adjuvant used (Synowiec and Drozdek 2016).

In summary, the use of FAMEs of edible oils alone, particularly soybean FAME, showed a phytotoxic effect in foliar applications against weeds. Sunflower FAME was safe on corn. Peppermint EO alone was the most active treatment against corn and weeds, and was more active than any mixture. Oilseed rape FAME provided the best combination overall, with less than 10% injury to corn and more than 84% injury to barnyardgrass and 50% injury to common lambsquarters. These results show that it is possible to improve the phytotoxicity of an EO by mixing it with a proper FAME.

Figure 5. Ternary graphs presenting the measured characteristics of A) corn, B) barnyardgrass, and C) common lambsquarters after treatment with combinations of the essential oils of hemp or peppermint with fatty acid methyl esters, or with treatments containing fatty acid methyl esters only. Abbreviations: H, fiber hemp essential oil; P, peppermint essential oil; R, oilseed rape fatty acid methyl ester; SB, soybean fatty acid methyl ester; SF, sunflower fatty acid methyl ester.

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