Abstract: Synthetic herbicides are commonly used in weed management, however, 70 years of use has led to weed resistance and environmental concerns. These problems have led scientists to consider alternative methods of weed management in order to reduce the inputs and impacts of synthetic herbicides. The aim of this experiment was to test the level of weed control using four weeding methods: glyphosate applied at an ultra-low volume, the organic herbicide nonanoic acid, flaming, and hot foam. The results showed that weed control was effective only when flaming and hot foam were applied (99% and 100% weed control, respectively). Nonanoic acid at a dose of 11 kg a.i. ha\(^{-1}\) diluted in 400 L of water did not control developed plants of *Cyperus esculentus* (L.), *Convolvulus arvensis* (L.) and *Poa annua* (L.). Glyphosate at a dose of 1080 g a.i. ha\(^{-1}\) (pure product) only controlled *P. annua* (L.), but had no effect on *C. esculentus* (L.) and *C. arvensis* (L.). After the aboveground tissues of weeds had died, regrowth began earlier after flaming compared to hot foam. There was no regrowth of *P. annua* (L.) only after using hot foam and glyphosate. Hot foam was generally better at damaging the meristems of the weeds. In one of the two experiment sites, significantly more time was needed after the hot foam to recover 10% and 50% of the ground compared to flaming. The time needed to recover 90% of the ground was on average 26–27 days for flaming and hot foam, which is the time that is assumed to be required before repeating the application. A total of 29 days after the treatments, weeds were smaller after flaming, glyphosate and hot foam compared to nonanoic acid and the control, where they had more time to grow.

Keywords: alternative methods; herbicide; organic; pelargonic acid; thermal tools; ultra-low volume
Alternative methods to the use of chemical-synthesis herbicides are alternative organic herbicides and thermal weed control [3,8,9]. Nonanoic acid (also called pelargonic acid) is a contact, non-selective, non-translocating, post-emergence non-synthetic herbicide [8,10]. It is a fatty acid, which kills plants by destroying the cell membranes, leading to rapid desiccation of plant tissues, and providing non-residual weed control [8,10]. Thermal weed control involves heat being transferred to plant material (leaves, stems, flowers, propagules, etc.) to destroy cell structures, and leads to the denaturation of proteins [9,11,12]. Flaming is the primary heat source for weed control in agriculture and on hard surfaces in urban areas [9,13–16]. Hot foam is an evolution of the hot water weed control method, modified by the addition of biodegradable foaming agents, and was first patented in 1995 [12,17]. Hot foam weed control is a non-toxic technique and is applicable to numerous weed species [3].

When a method of weed control is applied, in addition to its effectiveness, it is important to also evaluate the weed regrowth after the above-ground tissues of weeds have died [18]. In fact, most thermal methods affect the above-ground portion of the plants, however, some weeds (i.e., perennial weeds) may regrow from their below-ground components and thus a repeated application of the thermal control is required [3,19,20].

Hot foam has been used to control weeds in cotton fields [20], but the application of this high-energy demand weed control method (due to the high thermal capacity of water [21]) is more realistic in urban area contexts (e.g., on pavements) [22]. The growth of weeds on road pavements is different from that in a field, because the characteristics of the pavements affect the weed growth (i.e., fewer appear on frequently used roads with small joints than in infrequently used pavements with medium or wide joints) [22,23]. In Sweden hot foam was used to control weeds along railways [12]. Flaming can be used successfully for controlling weeds in both agricultural and urban area context [13,14,24,25].

The aim of this experiment was to test the weed control effect of different weeding methods: glyphosate applied at an ultra-low volume, organic herbicide nonanoic acid, flaming, and hot foam. Weed regrowth after the death of the vegetative weed tissues and weed dry biomass 29 days after treatment application were also evaluated.

2. Material and Methods

2.1. Experimental Set Up, Design and Treatment

A two-site experiment was conducted at the experimental farm of the University of Pisa (Pisa, Italy) (43°40′33.1″ N 10°18′41.2″ E). The study was replicated twice at each site. The two sites (sites I and II) differed in terms of weed population composition typology.

At site I, the major weeds was Cyperus esculentus (L.), Convulvulus arvensis (L.) and Poa annua (L.), each accounting for 25% of the weed population. Other weeds randomly present in the field were Anagallis arvensis (L.), Avena fatua (L.), Cirsium arvense (L.), Conyza canadensis (L.), Eleusine indica (L.), Inula viscosa (L.), Lolium rigidum (Gaud.), Picris echioides (L.), Plantago major (L.), Silene vulgaris (Moench), Sonchus oleraceus (L.), Stellaria media (L.), Tordylium apulum (L.), Trifolium repens (L.), and Veronica persica (L.), with an overall total of 25% of the weed population. The majority of C. esculentus (L.) were at the 6-tiller visible growth stage, P. annua (L.) was at the inflorescence emergence (inflorescence fully emerged) growth stage, and C. arvensis (L.) was at the 8–9 true-leaf growth stage [26]. At site II, the major weed was C. esculentus (L.), which accounted for 90% of the weed population. Other weeds randomly present in the field were A. arvensis (L.), C. canadensis (L.), C. arvensis (L.), Erodium cicutarium (L.), Euphorbia prostrata (Aiton), P. echioides (L.), P. major (L.), S. oleraceus (L.), T. repens (L.), and V. persica (L.), with an overall total of 10% of the weed population. C. esculentus (L.) was at the 6-tiller visible growth stage [26].

Weed species and percentages of single species in the total weed population were identified based on visual estimates. The sites were uncultivated (i.e., meadows under orchards) and the weeds had been managed periodically with mowing before the experiments were carried out. The soil was loam in both sites.
Glyphosate (360.00 g L\(^{-1}\) of active ingredient) was applied pure (i.e., without diluting in water). The product used was GLIFENE HP (Diachem S.p.A., Caravaggio, Italy), which contained glyphosate as isopropylamine (IPA) salt and surfactants. The product was applied with an ultra-low volume sprayer (MANKAR-P 30–50 Flex, Mantis ULV\(^{\circledR}\), Geesthacht, Germany) (Figure 1b) at a dose of 3 L ha\(^{-1}\) (i.e., 1080 g a.i. ha\(^{-1}\)). The machine was equipped with a segment rotation atomizer, which produces small droplets with a uniform size of about 150 \(\mu\)m [27]. Flaming was applied manually with a prototype of a back-pack flaming machine developed at the University of Pisa [13] (Figure 1a). The dose was 150 kg ha\(^{-1}\) of liquefied petroleum gas (LPG) based on previous experiments where this dose was found to be effective in controlling developed weeds [14,15]. The burner was 0.3 m wide and operated at 6 cm above the ground. Hot foam was applied using a Foamstream\(^{\circledR}\) MW Series (Weedingtech Ltd., London, UK) [28]. The solution used (Foamstream V4) was a 100% blend of plant oils and sugar (e.g., alkyl polyglucoside surfactants) [29]. The emission class is equivalent to a Euro 5 [30]. The machine flow rate was 0.2 L s\(^{-1}\) (96% water and 4% Foamstream V4) and the dose applied was 8.33 kg m\(^{-2}\). The manufacturer advised that Foamstream V4 percentage in the total flow rate could be varied between 0.5% and 5% depending on the client’s application. This dose was based on a previous experiment where it provided the highest weed control effect and the slowest weed regrowth [18]. The hot foam distribution tool was 0.3 m wide and operated at 5 mm above the ground (Figure 1c). Pure nonanoic acid (Beloukha, Novamont, Novara, Italy) was applied using a sprayer (Acuspray, Techneat engineering ltd, Ely, Cambridgeshire, UK) (Figure 1d) at a dose of 16 L ha\(^{-1}\) (i.e., 11 kg a.i. ha\(^{-1}\)) diluted in 400 L of water.

**Figure 1.** Machines used in the experiments for weed control: (a) prototype back-pack flaming machine; (b) ultra low volume sprayer (MANKAR-P 30–50 Flex, Mantis ULV\(^{\circledR}\), Geesthacht, Germany) used for glyphosate application; (c) hot foam distribution tool (Weedingtech Ltd., London, UK); (d) sprayer (Acuspray, Techneat engineering ltd, Ely, Cambridgeshire, UK) used for nonanoic acid distribution.
Treatments were applied on 14 May 2019 (repetition I) and on 02 July 2019 (repetition II) in both sites. Cumulative rainfalls were 93, 4, 94 mm in May, June and July, respectively, and the average temperatures were 15, 23, 25 °C in May, June, and July, respectively.

The experimental design was a randomized block design with four blocks. The five treatments (control, flaming, glyphosate, hot foam, and nonanoic acid) were applied in each block for a total of 20 plots per site. Plots were 2 m long and 0.3 m wide. Plots were 0.3 m wide based on the width of the hot foam application tool and flaming burner. A space of 2.5 m between the plots has been left in order to avoid drift effect due to the use of the herbicides.

2.2. Data Collection

Measurements of ground covered by the total population of weeds were used to estimate weed control (i.e., from treatments application to death of weeds above-ground tissues) and weed regrowth (i.e., from death of weeds above-ground tissues to 27 days after the treatment application). These measurements were estimated from digital images using IMAGING Crop Response Analyser [31]. The digital images, one for each plot, were taken from an area of 0.075 m² (30 cm × 25 cm) at the center of each plot (with the same geographical coordinates). Photographs of the weed cover for evaluating the weed control were taken 1 day before, and 1 and 2 days after treatments. Weed cover photographs for the evaluation of the weed regrowth were taken 3, 7, 10, 17 and 27 days after treatments. The distance between the weeds and the camera was constant (i.e., 30 cm from the ground), and high contrast was prevented by using an umbrella. The brightness of the digital images was equalized before analysis. The digital image analysis was as described in Rasmussen et al. [32], which, summarizing, counted the percentage of green pixels on the whole pixels of the photograph. The green weed biomass was collected 29 days after treatment at the center of each plot (i.e., 0.075 m² area) by cutting the weeds at ground level. Cut plants were dried at 105 °C to a constant weight. The dry weight was then converted into g m⁻².

2.3. Statistical Analysis

Data normality was assessed using the Shapiro–Wilk test. Other tests consisted of the Student’s t-test to verify that the mean error was not significantly different from zero, the Breusch-Pagan test for homoscedasticity, and the Durbin–Watson test for autocorrelation.

The weed control in each site was modeled in a linear mixed model using the R software [33] extension package ‘lmerTest’ (tests in linear mixed effects models) [34]. A logit transformation of weed cover data was performed. The treatment, evaluation date and repetition of the experiment were fixed factors. Correlated random intercepts and slopes were fitted between blocks and fixed factors. An analysis of variance was performed for each model. The extension package ‘ggplot2’ (elegant graphics for data analysis) [35] was used to plot all the graphs.

The comparisons between pairs of estimated values were computed by estimating the 95% confidence interval of the difference between the values (Equation (1)):

$$CI \text{ (difference) } = (x_1 - x_2) \pm 1.96 \sqrt{(SE_{x_1})^2 + (SE_{x_2})^2},$$  

(1)
where \((x_1)\) is the mean of the first value, \((x_2)\) is the mean of the second value, \((\text{SE}x_1)\) is the standard error of \((x_1)\), and \((\text{SE}x_2)\) is the standard error of \((x_2)\). If the resulting 95% confidence interval (CI) of the difference between values did not cross the value 0, the null hypothesis that the compared values were not different was rejected.

3. Results

3.1. Weed Control

The \(p\)-values resulting from the analysis of variance are reported in Table 1. At site I, weed control was influenced by the treatment, evaluation date, and interaction between the two. At site II the interaction between the treatment, evaluation date, and repetition of the experiment were also significant. Tables 2 and 3 report the weed control least squares means and standard errors of weed cover percentage logit transformed one day before, and one and two days after treatments at sites I and II, respectively. The inverse transformed values and 95% confidence intervals are plotted in Figures 2 and 3, for site I and II, respectively.

Table 1. Weed control type III analysis of variance with Satterthwaite’s method resulting from the linear mixed model where the treatments (control, flaming, glyphosate, hot foam, and nonanoic acid), evaluation date (one day before, and one and two days after the treatments) and repetition of the experiment (I and II) were used as fixed factors at sites I and II, respectively. Significant \(p\)-values are shown in italics.

|                     | Site I       | Site II      |
|---------------------|--------------|--------------|
|                     | \(p\)-Value  | \(p\)-Value  |
| Treatment           | <0.001       | <0.001       |
| Date                | <0.001       | <0.001       |
| Repetition          | 0.161        | 0.134        |
| Treatment: Date     | <0.001       | <0.001       |
| Treatment: repetition| 0.588        | 0.112        |
| Date: repetition    | 0.896        | 0.160        |
| Treatment: date: repetition | 0.865 | 0.037 |

Table 2. Weed control least squares means and standard errors (SE) of weed cover percentage logit transformed as affected by the different treatments, repetition of the experiment, and evaluation date (one day before, and one and two days after treatments) at site I.

| Treatment           | Logit [Weed Cover (%)] (±SE) |
|---------------------|------------------------------|
|                     | 1 DBT | 1 DAT | 2 DAT |
| Control             | 1.55 (0.365) | 1.72 (0.399) | 1.07 (0.365) |
| Flaming             | 1.18 (0.400) | −4.53 (0.381) | −5.16 (0.399) |
| Glyphosate          | 1.55 (0.415) | 0.85 (0.375) | 1.11 (0.397) |
| Hot foam            | 1.97 (0.476) | −6.98 (0.507) | −7.07 (0.466) |
| Nonanoic acid       | 1.84 (0.430) | 1.31 (0.451) | 1.18 (0.417) |

|                     | 1 DBT | 1 DAT | 2 DAT |
|---------------------|-------|-------|-------|
| Control             | 1.11 (0.409) | 1.40 (0.483) | 0.97 (0.426) |
| Flaming             | 0.89 (0.468) | −5.04 (0.494) | −5.15 (0.482) |
| Glyphosate          | 1.29 (0.367) | 0.82 (0.378) | 0.52 (0.366) |
| Hot foam            | 1.50 (0.437) | −8.34 (0.511) | −7.73 (0.442) |
| Nonanoic acid       | 1.50 (0.406) | 1.00 (0.473) | 0.26 (0.410) |

1 DBT, 1 DAT, and 2 DAT: one day before, and one and two days after the treatments, respectively.
Table 3. Weed control least squares means and SE of weed cover percentage logit transformed as affected by the different treatments, repetition of the experiment, and evaluation date (one day before, and one and two days after treatments) at site II.

| Treatment          | Repetition I   |          |          |
|--------------------|----------------|----------|----------|
|                    | logit [Weed Cover (%)] (±SE) | 1 DBT | 1 DAT | 2 DAT |
| Control            | 0.50 (0.175)   | 0.53 (0.234) | 0.64 (0.197) |
| Flaming            | 1.19 (0.240)   | −5.70 (0.218) | −4.59 (0.262) |
| Glyphosate         | 0.90 (0.298)   | 1.36 (0.207) | 1.36 (0.316) |
| Hot foam           | 1.07 (0.264)   | −5.48 (0.351) | −5.37 (0.270) |
| Nonanoic acid      | 0.89 (0.226)   | 0.18 (0.172) | 0.28 (0.229) |

|                  | Repetition II  |          |          |
|                  |                | 1 DBT | 1 DAT | 2 DAT |
| Control          | 0.54 (0.183)   | 0.64 (0.252) | 0.75 (0.177) |
| Flaming          | 0.49 (0.208)   | −5.82 (0.198) | −4.17 (0.209) |
| Glyphosate       | 0.59 (0.280)   | 0.98 (0.194) | 0.99 (0.280) |
| Hot foam         | 1.25 (0.271)   | −6.30 (0.364) | −5.41 (0.257) |
| Nonanoic acid    | 0.38 (0.240)   | −0.18 (0.204) | −0.04 (0.220) |

1 DBT, 1 DAT, and 2 DAT: one day before, and one and two days after the treatments, respectively.

Figure 2. Weed control bar graph of back-transformed means (Table 2) and the 95% confidence interval as affected by the treatments (control, flaming, glyphosate, hot foam and nonanoic acid), repetition (I and II) and evaluation date at site I; 1 DBT, 1 DAT and 2 DAT: one day before, and one and two days after the treatment, respectively.
In both sites and repetitions of the experiment, only the flaming and hot foam treatments were able to control weeds and showed a significant reduction in weed cover one and two days after treatments compared with one day before their application. On the other hand, after the nonanoic acid and glyphosate applications, there was no weed cover reduction (Figures 2 and 3, Tables 2 and 3).

In the two repetitions at site I, one and two days after the application of flaming, weed cover was statistically similar and was on average 0.7%. Also after hot foam application, there were no statistical differences the weed cover in the two repetitions, both one and two days after the treatments, which on average was 0% (Figure 2, Table 2). At site II, weed cover one day after flaming was similar in the two experiment replications (on average 0.3%), and significantly lower compared with two days after its application in both replications, which was on average 1.3%. This thus suggests that there was an early start of the regrowth already two days after the treatment application. Weed cover after hot foam was similar between one and two days after the treatment application in both replications, which was on average 0.4% (Figure 3, Table 3).

Weed cover estimated one and two days after the application of treatments was statistically lower in plots where hot foam was applied compared with the flamed plots in both repetitions of site I. At site II, in both repetitions, weed cover one day after the treatments was similar between flaming and hot foam, whereas two days after treatments, weed cover after hot foam was significantly lower than after flaming (Figures 2 and 3, Tables 2 and 3).

3.2. Weed Regrowth

At site I, the weed composition observed 27 days after the application of the treatments showed a shift in the plots (both repetitions) where glyphosate and hot foam were applied compared to that observed before the start of the experiment. In these plots *P. annua* (L.) was no longer present, whereas it was still observed in the plots where the other treatments had been applied. At site II, an increase in *C. arvensis* (L.) was observed, which represented 30% of the final weed population (27 days after treatments) in all the treated plots and the control. In both sites and repetitions, regrowth was observed starting from the meristems, and the weed coverage was not due to a new weed infestation from seeds.
The p-values from the analysis of variance are reported in Table 4, and the coefficients of the regressions in Table 5. The regression lines with all the points and 95% confidence interval bands of percentage weed cover as affected by the treatments (control, flaming, glyphosate, hot foam and nonanoic acid), the repetitions (I and II) and the evaluation dates (3, 7, 10, 13, 17 and 27 days after the treatments) for both sites are shown in Figure 5. At site I, weed cover regrowth was affected by the treatments, evaluation date, and their interaction, whereas at site II, the interaction between treatments, evaluation date and the repetition was also significant (Table 4).

Table 4. Weed regrowth analysis of variance resulting from the linear model, where weed coverage was affected by the treatments (control, flaming, glyphosate, hot foam, and nonanoic acid), the evaluation date (3, 7, 10, 13, 17 and 27 days after the treatments) and the repetition of the experiment (I and II) were used as factors. Significant p-values are shown in italics.

| p-Values          | Site I | Site II |
|-------------------|--------|---------|
| Treatment         | <0.001 | <0.001  |
| Date              | <0.001 | <0.001  |
| Repetition        | 0.447  | 0.492   |
| Treatment: Date   | <0.001 | <0.001  |
| Treatment: repetition | 0.834  | 0.476   |
| Date: repetition  | 0.494  | 0.056   |
| Treatment: date: repetition | 0.914  | 0.019   |

Figure 4. Weed dry biomass bar graph and 95% confidence interval as affected by the treatments (control, flaming, glyphosate, hot foam and nonanoic acid), repetition and evaluation date at sites I and II, respectively.
The time estimated after hot foam treatments, which reached 10% weed cover about three days later (Table 6, Figure 4).

The time (days) estimated to reach 10%, 50% and 90% weed cover regrowth, is reported in Table 6. Regression lines with all the points and 95% confidence interval bands of percentage weed cover as affected by the treatments (control, flaming, glyphosate, hot foam and nonanoic acid), the repetition (I,II) and the evaluation date (time) in the two sites. (Site I) residual standard error = 0.123; multiple R-squared = 0.859; adjusted R-squared = 0.846. (Site II) residual standard error = 0.089; multiple R-squared = 0.916; adjusted R-squared = 0.909.

In both sites and repetitions, except for glyphosate at Site I, the weeds grew again. Glyphosate at site I followed a different trend, showing a slight weed cover decrease of 15% (±7%) 27 days after the application of the treatment in repetition I, whereas in repetition II, the weed cover did not increase and remained statistically similar for 27 days (Figure 5).

At site I, the application of treatments showed three response trends. Glyphosate followed the trend described above. Hot foam and flaming started with a weed cover of 0% and 1%, respectively,
and reached a similar average of 90–94% (±5%) after 27 days (i.e., regrowth). Nonanoic acid and the control started with an average weed cover of 73–81% (±4%) and reached 98–100% (±5%) after 27 days. Flaming after 27 days was also similar to the control and nonanoic acid, whereas the hot foam was significantly lower (Figure 5).

At site II, after hot foam and flaming, weed cover regrew from 0% (hot foam) and 7–11% ± 3% (flaming). After the other two treatments, the weeds did not die and continued to grow, starting with a weed cover percentage of 61–79% ± 3%. The hot foam in repetition I showed the lowest significant weed coverage after 27 days (71% ± 4%). In repetition I, weed cover after flaming treatment reached 88% (±4%) after 27 days. This was similar to flaming in repetition II (95% ± 4%), hot foam in repetition II (90% ± 4%), glyphosate (89% and 90% ± 4%, respectively for Replication I and II) and the control in repetition II (95% ± 4%). However, it was different from nonanoic acid (98% and 99% ± 4% for repetitions I and II) and the control in repetition I (99% ± 4%), respectively. The other treatments showed similar results to each other (Figure 5).

The time (days) estimated to reach 10%, 50% and 90% weed cover regrowth, is reported in Table 6. The time to reach 10% and 50% weed cover regrowth was only biologically significant for the flaming and hot foam because, in the control, glyphosate and nonanoic acid plot weeds did not die and weed cover was already above 10% and 50%, respectively (Figure 5).

Table 6. Estimated time (days) to reach 10%, 50% and 90% weed cover regrowth (ET10, ET50, and ET90, respectively) as affected by the different treatments (control, flaming, glyphosate, hot foam, and nonanoic acid), the repetition (I and II) and the evaluation date in the two sites. The linear regression lines are plotted in Figure 5.

| Treatment      | ET10 (±SE) | ET50 (±SE) | ET90 (±SE) | ET10 (±SE) | ET50 (±SE) | ET90 (±SE) |
|----------------|------------|------------|------------|------------|------------|------------|
| Site I         |            |            |            |            |            |            |
| Control        | NA         | NA         | 17.6 (±3.69) | NA         | NA         | 15.8 (±4.00) |
| Flaming        | 4.7 (±0.97) | 15.3 (±0.70) | 26.0 (±1.32) | 4.8 (±0.96) | 15.3 (±0.70) | 25.9 (±1.30) |
| Glyphosate     | NA         | NA         | NA         | NA         | NA         | NA         |
| Hot foam       | 5.7 (±0.89) | 16.0 (±0.70) | 26.4 (±1.31) | 6.6 (±0.83) | 16.8 (0.73) | 27.1 (±1.35) |
| Nonanoic acid  | NA         | NA         | 17.3 (±3.55) | NA         | NA         | 16.7 (±2.28) |
| Site II        |            |            |            |            |            |            |
| Control        | NA         | NA         | 19.2 (±2.12) | NA         | NA         | 19.8 (±3.79) |
| Flaming        | 2.8 (±0.93) | 15.3 (±0.60) | 27.8 (±1.24) | 3.9 (±0.75) | 14.7 (±0.51) | 25.6 (±0.95) |
| Glyphosate     | NA         | NA         | 28.0 (±7.48) | NA         | NA         | 26.8 (±6.52) |
| Hot foam       | 5.4 (±0.89) | 19.3 (±1.85) | 33.7 (±1.85) | 6.7 (±0.59) | 16.8 (±0.52) | 27.0 (±0.97) |
| Nonanoic acid  | NA         | NA         | 20.7 (±2.01) | NA         | NA         | 21.2 (±1.71) |

NA: not available (i.e., the estimation had no biological meaning).

In both sites and repetitions, weeds re-covered 10% of the ground in a similar average time of four days after flaming, and in a similar average time of six days after hot foam. At site I, there were no statistical differences between flaming and hot foam in re-covering 10% of the ground after treatment. The times estimated after flaming at site II were, instead, statistically lower than that estimated after hot foam treatments, which reached 10% weed cover about three days later (Table 6, Figure 5).

A total of 50% weed cover regrowth was reached after flaming in a similar average time of 16 days in the two repetitions and in both sites. Also after the hot foam, there were no statistical differences between repetitions and sites, and 50% weed cover was reached after an average time of 17 days. At site I, again, there were no statistical differences between flaming and hot foam in re-covering 50% of the ground after treatment, whereas at site II, 50% weed cover after hot foam was reached three days later than the time needed after flaming (Table 6, Figure 5).
A total of 90% weed cover regrowth (or natural growth where weeds were not dead) was reached after all the treatments, except for glyphosate at site I. After an average of 18 days from the start of the experiment, the control reached 90% of the ground covered by weeds in the two repetitions and in both sites. The nonanoic acid plots reached it after an average time (average between repetitions and sites which were similar) of 19 days. The control and nonanoic acid times were statistically similar. A total amount of 90% weed cover was reached in an average time (average between similar values of replicates and sites) of 26 days after flaming. After the hot foam, repetition I of site II showed a significantly higher time (average of 34 days) to reach 90% weed cover regrowth compared to repetition II of site II and repetitions I and II of site I, which showed a similar average time of 27 days. At site II, the resulting high standard errors due to the high variability in the plots after the glyphosate application averaged 27 days, which was similar to the times estimated for all the other treatments to reach 90% weed cover. At site I, the time to reach 90% weed cover after flaming was significantly higher compared with the control (+8 days) and nonanoic acid (+9 days), and similar to that of hot foam. At site II, the time after flaming was similar to that of the control and hot foam in repetition II, higher than nonanoic acid (+4 days) and lower (~8 days) than hot foam in repetition I. At site I, after the hot foam, the significant time delay to reach 90% weed cover compared with the control and nonanoic acid was 9 days and 10–11 days, in repetition I and II, respectively. At site II, the significant delay after hot foam in repetition I compared to the control and nonanoic acid was 14 days and 13 days, respectively. On the other hand, after the hot foam in repetition II, the time was similar to the control and higher (+6 days) than the nonanoic acid (Table 6, Figure 5).

3.3. Weed Dry Biomass

Weed dry biomass collected 29 days after treatment application was influenced by the treatment and the interaction between treatment and site (p-values < 0.001, respectively). The other factors and interactions were not significant. Least squares means and 95% confidence intervals for each treatment, repetition and site are plotted in Figure 4.

At site I, the weed dry biomass in both repetitions of the control and nonanoic acid plots were similar and significantly higher compared with both the repetitions of glyphosate, flaming and hot foam, whereas the dry biomass was similar (Figure 4).

At site II, the weed dry biomass in both repetitions of the control was similar to that estimated in both repetitions of nonanoic acid and higher compared with both repetitions of flaming, glyphosate and hot foam. Both repetitions of hot foam were significantly lower than both repetitions of nonanoic acid, whereas both repetitions of flaming and glyphosate were significantly lower only than repetition I of nonanoic acid. Both repetitions of glyphosate and repetition II of flaming were similar, whereas repetition I of flaming was lower than repetition II of nonanoic acid. Weed dry biomass in both repetitions of glyphosate was similar to those of flaming and hot foam in repetition I, whereas glyphosate in repetition I was significantly higher than hot foam in repetition II. Weed dry biomass in both repetitions of flaming was similar to those of hot foam.

4. Discussion

Weed control was effective only when flaming and hot foam were applied. Hot foam was the most effective method, leading to 100% weed control one and two days after the treatment in both sites and replications. At site I, flaming was statistically a little less effective than hot foam, but in any case, provided 99% of weed control. At site II, also flaming led to 100% weed control, but this effect lasted only one day (Figures 2 and 3).

Although the effectiveness of a herbicide should increase if the droplet size is reduced (i.e., an increase in droplet number obtained with ultra-low volume applications increases the likelihood of impacting the weed leaf surface) [4], a dose of 1080 g a.i. glyphosate per ha was probably not high enough to control *C. esculentus* (L.) and *C. arvensis* (L.), whereas this dose was effective against *P. annua* (L.). This effect of glyphosate on *P. annua* (L.) was visible in the photographs taken 13 days
after the treatments, which showed the delayed death of this weed species. In Figure 4, the decrease in the total weed cover in repetition I of glyphosate was due to the death of *P. annua* (L.). This decrease was not significant in repetition II (where the weed cover was similar to that three days after treatment). This was probably because the simultaneous growth of *C. esculentus* (L.) and *C. arvensis* (L.) minimized the reduction in the total weed population coverage due to the death of *P. annua* (L.). Because only *P. annua* (L.) died, and the total weed population coverage was never lower that an average of 50%, the weed control due to the use of glyphosate cannot be considered effective in this experiment.

Nonanoic acid was not effective in controlling weeds probably because the species in these experiments were too developed for the herbicide to have an effect. Previous research reported that nonanoic acid needs to be applied to very young or small plants for acceptable weed control [36], and repeated applications are suggested [8]. Rowley et al. [37] obtained a moderate reduction in weed coverage, density, and dry biomass compared to the untreated control, but the dose of nonanoic acid used (39 L a.i. ha$^{-1}$) was above that indicated on the product label. Other authors [38] found a reduction in *Microstegium vimineum* (Trin.) coverage compared to the untreated control when pelargonic acid was applied at 11.8 kg a.i. ha$^{-1}$, 5% volume.

The regrowth of weeds after the death of the aboveground vegetative tissues is an important indicator to validate the effectiveness of a weed control technique. In fact, it determines how many times a weeding method needs to be applied during a weed management program. Given that a technique should kill the weeds after being applied, the time weeds take to regrow and cover the ground again is an indicator of how many times the technique needs to be repeated in the annual management of weeds. This management depends on whether the weeds grow in urban areas or agricultural fields, with crops that may vary in sensitivity to competition from weeds.

Weed regrowth started earlier after flaming than after hot foam, in fact, at site II, just two days after the flaming application, the weed cover was higher than one day after. Three days after flaming, the weed cover estimated at site II was already 7–11% (±3%), whereas in the hot foam plots, the weed cover was still 0%. At site I, 27 days after treatments, the weed cover after hot foam was still significantly lower than the control and nonanoic acid. However, in the flaming plots, the weed cover was similar to hot foam, but also to the control and nonanoic acid, thus suggesting greater damage of the hot foam to the weeds’ meristems. This was more evident in the repetition I at site II, where the weed cover after 27 days from the hot foam application was still significantly lower than the other treatments (Figure 5).

At site II, the delay of time needed after hot foam to recover 10% and 50% of the ground compared to flaming was also significant, and this delay was still significant in repetition I to re-cover 90% (Table 6). *P. annua* (L.) did not regrow after hot foam, suggesting that the meristems of this species were severely damaged, which flaming did not achieve.

The time needed to recover 90% of the ground was on average 26–27 days for flaming and hot foam. The time of 34 days was estimated only for hot foam in repetition I of site II (Table 6). A time of 26–27 days was estimated to be the time after which a new weed control application was needed for a real infested field during high weed season (i.e., May, June, July in Italy). For glyphosate and nonanoic acid, the time needed to reach 90% weed coverage of the ground was less relevant because in these plots there was no weed control. The dose of 1080 g glyphosate per ha$^{-1}$ had no effect on the growth of *C. esculentus* (L.) and *C. arvensis* (L.), which continued their natural growth, whereas *P. annua* (L.) died and was not able to regrow. In the nonanoic acid and control plots, the growth observed naturally occurred in 27 days (i.e., weeds did not die).

Twenty-nine days after the treatment application, the weed dry biomass was similar when flaming and hot foam were applied. This suggests that the weed cover in repetition I at site II was lower, but was made up of larger weeds. Also the lowest weed coverage after glyphosate at site I was made up of the largest weeds, in fact, the weed dry biomass was similar to flaming and hot foam. At site I, in the control and nonanoic acid plots, weed dry biomass was always higher than flaming, glyphosate and hot foam, suggesting that during their growth these weeds, in addition to expanding laterally, had time to grow in size. At site II, the differences in weed dry biomass were less marked than at site I,
suggesting a more homogeneous weed growth, but in any case the control had more time to grow in size.

5. Conclusions

Weed control was effective only when flaming and hot foam were applied, providing respectively 99% and 100% of weed control two days after the treatments. Nonanoic acid at a dose of 11 kg a.i. ha\(^{-1}\) diluted in 5 L of water was not effective at controlling the developed plants of *C. esculentus* (L.), *C. arvensis* (L.) and *P. annua* (L.). Glyphosate at a dose of 1080 g a.i. ha\(^{-1}\) without water dilution only controlled *P. annua* (L.), but had no effect on *C. esculentus* (L.) and *C. arvensis* (L.). Flaming and hot foam controlled these three major species of the weed population effectively together with the other weeds that were observed in the field experiments.

Weed regrowth started sooner after flaming that after hot foam. *P. annua* (L.) did not regrow only after the hot foam and glyphosate application, and there was generally more damage to the weeds’ meristems after hot foam. At site II, a significant time delay was needed after hot foam to recover 10% and 50% of the ground compared to flaming. The time needed to recover 90% of the ground was on average 26–27 days for flaming and hot foam. This time of 26–27 days was estimated to be the time after which a new weed control application was needed after flaming and hot foam for a real infested field during the high weed growth season (e.g., May, June, July in Italy). After 29 days from treatment application, weeds were smaller in size when flaming, glyphosate and hot foam were applied compared with nonanoic acid and the control. From a practical standpoint, hot foam and flaming applications could be repeated once a month in spring and beginning of summer, and less frequently when the weeds growth is slower. Flaming can also be used to control weeds after the emergence/transplant of heat-tolerant crops, whereas hot foam is recommended applied in bands of soil before high-income crop transplant and/or for controlling weeds under vineyard rows, in order to reduce heat production costs compared to the application of the whole ground surface.

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**References**

1. Duke, S.O.; Heap, I. Evolution of weed resistance to herbicides: What have we learned after seventy years. In *Biology, Physiology and Molecular Biology of Weeds*; Jugulam, M., Ed.; CRC Press: Boca Raton, FL, USA, 2017.
2. Heap, I. Global perspective of herbicide-resistant weeds. *Pest Manag. Sci.* 2014, 70, 1306–1315. [CrossRef]
3. Wei, D.; Liping, C.; Zijun, M.; Guangwei, W.; Ruirui, Z. Review of non-chemical weed management for green agriculture. *Int. J. Agric. Biol. Eng.* 2010, 3, 52–60.
4. Craig, I.P.; Hewitt, A.; Terry, H. Rotary atomiser design requirements for optimum pesticide application efficiency. *Crop Prot.* 2014, 66, 34–39. [CrossRef]
5. Matthews, G.A. *Pesticide Application Methods*; Longman (Group UK): Harlow, Essex, UK, 1992.
6. Woods, N.; Craig, I.; Dorr, G.; Young, B. Spray drift of pesticides arising from aerial application in cotton. *J. Environ. Qual.* 2001, 30, 697–701. [CrossRef] [PubMed]
Doğan, M.N.; Öğüt, D.; Müldeler, N.; Boz, Ö.; Brants, I.; Voegler, W. Effect of Water Volume and Water Quality on the Efficacy of Glyphosate on some Important Weed Species in Turkey. In Proceedings of the 25th German Conference on Weed Biology and Weed Control, Braunschweig, Germany, 13–15 March 2012; pp. 229–234.

Webber, C.L., III; Taylor, M.J.; Shreffler, J.W. Weed control in yellow squash using sequential postdirected applications of pelargonic acid. *HortTechnology* **2014**, *24*, 25–29. [CrossRef]

Melander, B.; Liebman, M.; Davis, A.S.; Gallandt, E.R.; Barberi, P.; Moonen, A.C.; Rasmussen, J.; van der Weide, R.; Vidotto, F. Non-chemical weed management. Thermal weed control. In *Weed Research: Expanding Horizons*; Hatcher, P.E., Froud Williams, R.J., Eds.; John Wiley & Sons Ltd.: Hoboken, NJ, USA, 2017; pp. 259–264.

Senseman, S.A. *Herbicide Handbook*, 9th ed.; Weed Science Society of America: Lawrence, KS, USA, 2007; pp. 379–381.

Ulloa, S.M.; Datta, A.; Knezevic, S.Z. Tolerance of selected weed species to broadcast flaming at different growth stages. *Crop Prot.* **2010**, *29*, 1381–1388. [CrossRef]

Cederlund, H.; Börjesson, E. Hot foam for weed control—Do alkyl polyglucoside surfactants used as foaming agents affect the mobility of organic contaminants in soil? *J. Hazard. Mater.* **2016**, *314*, 312–317. [CrossRef] [PubMed]

Raffaelli, M.; Martelloni, L.; Frasconi, C.; Fontanelli, M.; Peruzzi, A. Development of machines for flaming weed control on hard surfaces. *Appl. Eng. Agric.* **2013**, *29*, 663–673.

Martelloni, L.; Fontanelli, M.; Frasconi, C.; Raffaelli, M.; Peruzzi, A. Cross-flaming application for intra-row weed control in maize. *Appl. Eng. Agric.* **2016**, *32*, 569–578.

Martelloni, L.; Raffaelli, M.; Frasconi, C.; Fontanelli, M.; Peruzzi, A.; D’Onofrio, C. Using flaming as an alternative method to vine suckering. *Agronomy* **2019**, *9*, 147. [CrossRef]

Ascard, J. Effects of flame weeding on weed species at different developmental stages. *Weed Res.* **1995**, *35*, 397–411. [CrossRef]

Rajamannan, A.H.J.; Washington, D.C. U.S. Patent and Trademark Office. Method of Using Hot Air Foam to Kill Vegetation and Pests. U.S. Patent No. 5,575,111, 19 November 1996.

Martelloni, L.; Frasconi, C.; Sportelli, M.; Fontanelli, M.; Raffaelli, M.; Peruzzi, A. The use of different hot foam doses for weed Control. *Agronomy* **2019**, *9*, 490. [CrossRef]

Peerzada, A.M.; Chauhan, B.S. Chapter 2. Thermal Weed Control: History, Mechanisms, and Impacts. In *Non-Chemical Weed Control*; Jabran, K., Chauhan, B.S., Eds.; Elsevier: London, UK, 2018; pp. 9–31.

Kup, F.; Saglam, R. Weed destruction in cotton fields using hot foam method and its comparison to certain other methods. *ARPN J. Agric. Biol. Sci.* **2014**, *9*, 301–307.

De Cauwer, B.; Bogaert, S.; Claerhout, S.; Bulcke, R.; Reheul, D. Efficacy and reduced fuel use for hot water weed control on pavements. *Weed Res.* **2014**, *55*, 195–205. [CrossRef]

Boonen, E.; Beeldens, A.; Fagot, M.; De Cauwer, B.; Reheul, D.; Bulcke, R. Preventive Weed Control on Pavements: Reducing the Environmental Impact of Herbicides Part 1: A Field Survey Study. In Proceedings of the 10th International Conference on Concrete Block Paving, Shanghai, China, 24–26 November 2012; pp. 1–14.

Melandar, B.; Holst, N.; Grundy, A.C.; Kempenaar, C.; Riemens, M.M.; Verschwele, A.; Hansson, D. Weed occurrence on pavements in five North European towns. *Weed Res.* **2009**, *49*, 516–525. [CrossRef]

Peruzzi, A.; Martelloni, L.; Frasconi, C.; Fontanelli, M.; Pirchio, M.; Raffaelli, M. Machines for nonchemical intra-row weed control in narrow and wide-row crops: A. review. *J. Agric. Eng.* **2017**, *48*, 57–70. [CrossRef]

Martelloni, L.; Fontanelli, M.; Frasconi, C.; Raffaelli, M.; Pirchio, M.; Peruzzi, A. A combined flamer-cultivator for weed control during the harvesting season of asparagus green spears. *Span. J. Agric. Res.* **2017**, *15*, e0203. [CrossRef]

Hess, M.; Barralis, G.; Bleibholder, H.; Buhr, L.; Eggers, T.H.; Hack, H.; Staus, R. Use of the extended BBCH scale—General for the description of the growth stages of mono- and dicotyledonous weed species. *Weed Res.* **1997**, *37*, 433–441. [CrossRef]

Mankar. ULV Herbicide Spraying System. Available online: [http://agri-flex.com/wp-content/uploads/2017/03/0005.pdf](http://agri-flex.com/wp-content/uploads/2017/03/0005.pdf) (accessed on 24 October 2019).

Weedingtech. Foamstream M1200. Available online: [https://www.weedingtech.com/product/foamstream-m1200](https://www.weedingtech.com/product/foamstream-m1200/) (accessed on 6 November 2019).
29. Weedingtech. Our Technology. Available online: https://www.weedingtech.com/why-foamstream/our-technology/ (accessed on 6 November 2019).

30. Nesbit, M.; Fergusson, M.;Colsa, A.; Ohlendorf, J.; Hayes, C.; Paquel, K.; Schweitzer, J.P. Comparative Study on the Differences between the EU and US Legislation on Emissions in the Automotive Sector; European Parliament: Brussel, Belgium, 2016; Available online: http://www.europarl.europa.eu/RegData/etudes/STUD/2016/587331/IPOL_STU(2016)587331_EN.pdf (accessed on 6 November 2019).

31. IMAGING Crop Response Analyser. Ver. 0.4.. 2018. Available online: http://imaging-crops.dk (accessed on 17 July 2019).

32. Rasmussen, J.; Norremark, M.; Bibby, B.M. Assessment of leaf cover and crop soil cover in weed harrowing research using digital images. Weed Res. 2007, 47, 299–310. [CrossRef]

33. R Core Team. R: A Language and Environment for Statistical Computing; R Foundation for Statistical Computing: Vienna, Austria, 2016; Available online: https://www.R-project.org/ (accessed on 17 July 2019).

34. Kuznetsova, A.; Brockhoff, P.B.; Christensen, R.H.B. lmertest Package: Tests in Linear Mixed Effects Models. J. Stat. Soft. 2017, 82, 1–26. [CrossRef]

35. Wickham, H. Ggplot2: Elegant Graphics for Data Analysis, 3rd ed.; Springer: New York, NY, USA, 2009.

36. Webber, C.L., III; Shreffl, J.W.; Brandenberger, L.P.; Davis, A.R. AXXE® (Pelargonic Acid) and Racer® (Ammonium Nonanoate): Weed Control Comparisons; Vegetable Weed Control Studies; Oklahoma State University, Division of Agricultural Sciences and Natural Resources, Department of Horticulture & Landscape Architecture: Stillwater, OK, USA, 2012; pp. 1–4.

37. Rowley, M.A.; Ransom, C.V.; Reeve, J.R.; Black, B.L. Mulch and organic herbicide combinations for in-row orchard weed suppression. Int. J. Fruit Sci. 2011, 11, 316–331. [CrossRef]

38. Ward, J.S.; Todd, L.M. Nonchemical and herbicide treatments for management of Japanese stiltgrass (Microstegium vimineum). Invasive Plant Sci. Manag. 2012, 5, 9–19. [CrossRef]

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