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Use of Plant Water Extracts for Weed Control in Durum Wheat (*Triticum turgidum* L. Subsp. *durum* Desf.)

Alessandra Carrubba [1],*, **Andrea Labruzzo** [1], **Andrea Comparato** [1], **Serena Muccilli** [2] **and Alfio Spina** [3]

1   DSAAF—Department of Agriculture, Food and Forest Sciences, University of Palermo, 90128 Palermo (PA), Italy; andrea.labruzzo@unipa.it (A.L.); andreacomparato88@gmail.com (A.C.)

2   CREA—Council for Agricultural Research and Agricultural Economy Analysis – Research Center for Olive, Citrus and Tree Fruits, 95024 Acireale (CT), Italy; serenamuccilli@hotmail.com

3   CREA—Council for Agricultural Research and Agricultural Economy Analysis – Research Center for Cereal and Industrial Crops, 95024 Acireale (CT), Italy; alfio.spina@crea.gov.it

*   Correspondence: alessandra.carrubba@unipa.it; Tel.: +39-091-23862208

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Abstract: The use of plant water extracts to control weeds is gaining attention in environmentally-friendly agriculture, but the study of the effect that such extracts may exert on the yield of durum wheat is still unexplored. In 2014 and 2016, the herbicidal potential of several plant water extracts was field tested on durum wheat (cv Valbelice). In 2014, extracts obtained from *Artemisia arborescens*, *Rhus coriaria*, *Lantana camara*, *Thymus vulgaris*, and *Euphorbia characias* were used, whereas in 2016 only *A. arborescens* and *R. coriaria* were tested as "donor" plants. In both years, weed incidence was evaluated, together with the major yield parameters of wheat. None of the treatments (including chemicals) could eradicate weeds from the field. In 2014, dicots were in general prevailing in plots treated with extracts of *E. characias*, while monocots prevailed after treatments with *L. camara* and *R. coriaria*. In 2016, lower weed biomass and diversity level were found, and only *Avena* and *Phalaris* were detected at harvest time. Treatment with plant water extracts affected grain yields, but it seems likely that those effects are not due to the diverse incidence of weeds in treated and untreated plots, rather to some direct action exerted by allelopathic substances.

1. Introduction

The use of environmentally-friendly methods for weed control is gaining attention in agricultural practice. By one side, the widespread use of synthetically-derived herbicides has caused a number of adverse effects, such as the high persistence of herbicides in the environment and in the food chain, and the development of highly resistant weed populations. Second, there are some special cropping systems, such as those addressed to organic production, where the use of synthetic chemicals is banned.

In this general frame, an increased number of farmers are seeking alternative technical choices for weed management [1]. Many methods have been suggested in time, with contrasting results according to the chosen method, the weed population, and the expected results [2–4]. Among these new techniques, allelopathy plays an increasingly important role [5,6].

The term "allelopathy" indicates a complex of effects exerted by one plant species (the donor) to another one (the acceptor), through the release into the environment of a number of chemical substances, termed allelochemicals. This transmission can occur in several ways: The substances may be released directly and continuously by the living plants in the form of volatile compounds emitted

into the atmosphere, as root exudates into the soil, or as chemicals formed by the microbial degradation of plant residuals [7].

Generally speaking, the allelopathic phenomena may act both on seed germination and on the whole development cycle of the plant, with deterrent effects, e.g., on photosynthesis. The secondary metabolites with allelopathic action belong to many chemical families, the most important being phenols, flavonoids, terpenoids, glucosynolates, benzoxaquinones, and cyanogenic compounds [8].

The possibility to use allelochemicals for environmentally-friendly weed control is not new, being suggested by many authors since the early 2000s [9,10]. Many different methods of use are possible, differentiated by both timing and way of application. In addition, the presowing (or pretransplant) distribution of the "donor" material followed by burying [11], those compounds have been suggested as post-emergence treatments, as usual herbicides.

Studies about this topic may be addressed to two directions: To verify the effects of the plant extracts against the germination of weed seeds, or to test such effects against adult weed plants. Research in the first direction may have a great practical importance, since this action towards soil seed bank could limit the wave of weeds emergence, at least until the crop has developed a good competitiveness. Yet, not many experiments have investigated the application in field conditions of the results from in vitro experiments. Many factors may play a role in modifying these results, such as the interaction with soil organic matter, and micro- and meso-organisms [12]. Although allelopathy is claimed to have a strategic ecological role in natural conditions [13], the possibility to use the allelopathic compounds as a resource for in field weed management strategy is still fairly unexplored.

In biological essays, the most proper operating way to assess allelopathic activity should be the isolation of the one (or few) active molecule(s) which are directly responsible for the given biological activity, and some author [14] advices that only such isolated chemical compound(s) should be termed "allelochemical". Anyway, this field of study is huge, and the starting point is undoubtedly the individuation of effective crop/donor plant combinations. Hence, many researchers all over the world have started to study the efficacy of crude plant extracts or plant parts against many common pests.

In cereal crops, the need for suitable tools for weed management has an outstanding relevance. Indeed, competition with weeds not only is one of the biggest causes of yield losses, but also an issue for quality achievement [15]. Among cereal crops, durum wheat (*Triticum turgidum* L subsp. *durum* Desf.) is used all over the word mainly to produce pasta [16]. This crop reaches an outstanding importance in all Mediterranean countries, where it is cultivated for making high-quality products including, besides pasta, also bulghur, couscous, and bread. Most of the market value of these items relies upon the quality level reached by the harvested grain [17,18], and weed control bears a deep importance in ensuring a high-quality level product [19]. Additionally, the present large demand of organic cereals, associated with the establishment of a public compensation payment system, create a favorable context to promote organic arable farming systems. These systems will face technical problems such as weed control [20], which affect economic viability and may greatly influence cereals quality.

Weed control by means of allelopathic substances was evaluated in soft wheat (*Triticum aestivum* L.), using water extracts obtained from a number of plants including *Sorghum bicolor* (L.) Moench, *Helianthus annuus* L., *Parthenium hysterophorus* L., *Oryza sativa* L., etc., alone, in mixture, or coupled with different chemical herbicides [21–24]. The results from these experiments not only varied according to weed species and conditions (time and rate) of supply, but also according to their various possible combinations.

As far as we know, the effects that crude plant extracts may exert on the yield of durum wheat are still unexplored. This work was carried out in 2014 and 2016, with the goal of evaluating the activity of water extracts obtained by different plant (donor) species towards the weed population of durum wheat (cv Valbelice—acceptor), and to further evaluate the effect exerted by such extracts on crop growth and yield.

2. Materials and Methods

2.1. Preparation and Use of Plant Water Extracts

The donor ("allelopathic") species used for the preparation of water extracts were chosen based on their assessed biological activity and the availability of plant biomass. Five donor plants (*A. arborescens, R. coriaria, L. camara, T. vulgaris,* and *E. characias*) were tested in the first year, whereas in 2016 the field trial was restricted to *A. arborescens* and *R. coriaria*, that had previously proved interesting inhibitory activity of seed germination, coupled with significant effects on some qualitative parameters of durum wheat [25,26].

Artemisia arborescens (Vaill.) L. is a shrub from *Asteraceae*, widespread, and spontaneous in the Mediterranean environments. The essential oil extracted from this species has been the subject of many studies that have assessed its strong biocidal activity towards many micro-organisms and weeds [27,28]. Less information is available about *A. arborescens* water extracts, although their biological activity is demonstrated. Some preliminary studies have assessed their ability in inhibiting in vitro germination of some weeds [29]. Two lignans, ashantin and sesamin, were detected after a phytotoxicity bioassay-guided isolation of *A. arborescens* extracts [30]. Extracts from leaves of *A. arborescens* at very low concentration were found to be responsible for an enhancement of growth in some ornamental plants and, as such, are patented in the US (US 5434122A).

Rhus coriaria L. is a small shrub from *Anacardiaceae*, and is widely distributed inside wild Sicilian flora. The plant may reach 3 m in height and is considered a noxious weed. Its fruits are red-brownish drupas, that are toxic when consumed fresh, but after drying are largely used in Middle-Eastern cooking to season soups and vegetables. In Sicily, the plant was formerly cultivated in specialized areas named "sommaccheti" (from its local name "Sommacco"), to use in tannery its tannin-rich bark and leaves. This practice is now obsolete, due to the substitution of natural tannins with the analogous synthetically-derived items [31]. In regards to *R. coriaria* chemical composition, available information is mainly focused on fruits (the most commonly used part of the plant), rich in volatile aromatic compounds endowed with strong antioxidant ant antimicrobial activities [31–33]. Less research is available about other parts of the *R. coriaria* plant, although gallotannins and flavonoid derivatives were the most representative compounds in aqueous extracts of sumac leaves [34,35].

Lantana camara L. is a perennial from *Verbenaceae*, native to Central and Southern America and further introduced all over the world especially for ornamental purposes. The plant is strongly invasive, and is considered a noxious weed in many tropical and sub-tropical areas throughout the world [36]. Its essential oil, mainly extracted from the leaves, has shown a strong insecticidal activity, but the allelopathic effect of its leaves proved significant, as well [37]. The leaves contain phenolic compounds, mainly phenolic acids and flavonoids [38,39].

Thymus vulgaris L., belonging to the family *Labiatae*, is a small shrub with woody branches, and is spontaneous in sunny environments of Mediterranean areas. Its essential oil, obtained mainly from the flowering tops, is widely used for its antibacterial, antifungal, and antiviral properties [40,41]. In addition to essential oil, other components have been individuated in thyme, such as phenolic compounds (mostly phenolic acids and flavonoids), that are probably the active agents of many biological activities ascribed to *T. vulgaris* aqueous extracts [42].

Euphorbia characias L. is an evergreen shrub typical of the Mediterranean maquis, sometimes growing higher than 1 m. The plant grows well in dry areas and may tolerate rather long drought periods. All plant parts are toxic, above all because of its whitish latex, which is irritant by contact, but just for this reason in folk medicine it is used to treat warts. In some areas of the Mediterranean, the latex was used for illegal fishing, especially in Sicily where it was used to catch eels in sweet water pools (locally termed "nache") [43]. The toxicity of the latex from *Euphorbiaceae* is well known [44]; in *E. characias*, two toxic lectins [45] and many bioactive compounds such as polycyclic diterpenoids, bicyclic diterpenes, tocopherols, and sterols have been isolated [46].

The extracts that were used for the trials were prepared in the labs of the Department of Agricultural, Food and Forest Sciences of the University of Palermo, using plant material (including both leaves and inflorescences) picked from wild (*A. arborescens, E. characias, R. coriaria*) or cultivated plants (*T. vulgaris, L. camara*) growing near Ciminna (Palermo, Sicily) and Sparacia (Cammarata, Agrigento, Sicily). All plant material was first air dried at room temperature for at least five days.

To obtain water extracts, 1 kg of each dried product was soaked in 10 L of distilled water (weight/volume ratio: 1/10), and put in constant stirring with a speed rotation of 70 rounds/min for at least 10 h. At the end of the extraction process, the mass was filtered through filter paper (Whatman n. 4), and the obtained extracts were refrigerated at 4 °C until used. The dry matter concentration of each extract (Table 1) was measured after desiccation in the stove for 24 h at 105 °C.

Table 1. Concentration of the used extracts (% w/v) ([a]).

Plant species	Concentration (% w/v)
Rhus coriaria	8.75
Artemisia arborescens	18.82
Euphorbia characias	2.27
Lantana camara	6.14
Thymus vulgaris	22.33
([a]) weight/volume percentage	

2.2. Field Management

The field trials were carried out in the experimental farm "Sparacia" (Cammarata, AG, Sicily; 37° 38′ N–13° 46′ E; 415 m a.s.l.), of the Department of Agricultural, Food and Forest Sciences of the University of Palermo. The chosen durum wheat variety was the cv Valbelice (0111 × BC5), obtained in 1992 by the same Department. In both years, the preceding crop was Berseem clover (*Trifolium alexandrinum* L.). Durum wheat was cultivated accordingly to the cropping techniques ordinarily applied in the cereal areas of the site. Hence, soil was prepared by means of a summer work (25–30 cm deep), followed by two shallow harrowings. Sowing was made mechanically on 19 December, 2013 and 22 December, 2015, spreading at a soil depth of about 5 cm, and on rows 30 cm apart, an amount of seed aimed to obtain a seeding density of 350 viable seeds per m^2 (about 200 kg ha^{-1}). At sowing time, 1.5 t ha^{-1} of diammonium phosphate (18/46) were distributed. Next, after the crop had reached the stage of full tillering, 1.1 t ha^{-1} of urea (46) were additionally spread. Eight treatments were tested in the first year (five plant water extracts; one chemical herbicide; two controls), and five treatments (two plant water extracts; one chemical herbicide; two controls) were tested in 2016. The experimental plots were arranged in the field according to a randomized block design with three repetitions; each treatment was applied on nine durum wheat rows 1.70 m in length (size of plots 2.67 × 1.70 m = 4.54 m^2). In order to avoid interference phenomena between the treated plots, an essay area (1.20 × 1.67 m = 2.00 m^2) was delimited within each plot, and all surveys on both weeds and durum wheat were taken therein. Treatments with plant extracts were applied twice, distributing in crop post-emergence, 4 L m^{-2} of each previously prepared extract. In the first year, the first treatment was applied on 13 January, 2014 (i.e., after 25 DAS—days after sowing), when wheat was at the stage of 2–3 true leaves unfolded (Zadoks' scale: Z13) [47], whereas the second was applied on 13 March, 2014 (88 DAS), when the crop was entering the full stem elongation stage (Zadoks' scale: Z31). In the second year, the same crop development stages were detected on 12 February, 2016 and 4 April, 2016, and in those dates the planned treatments were consequently applied.

In the appositely planned plots, chemical weeding was performed only once, contemporarily to the first distribution (in different plots) of water extracts. The chemical herbicide was a mixture of mesosulfuron-methyl 3% + iodosulfuron-methyl-sodium 0.6% + mefenpir-diethyl 9% (ATLANTIS®), distributed by Bayer®for post-emergence weeding against all graminaceous weeds and some important

dicots. In compliance with label recommendation, chemical herbicide was distributed at a supply rate of 1.2 L ha^{-1} (formulated product).

For comparison, a group of plots (further named "untreated") were left undisturbed, i.e., without any weeding operation. Furthermore, in order to verify if the additional amount of water contained in the extracts could have a stimulating effect on plant growth (both on durum wheat and weeds), and to allow separating this effect, if any, an additional control plot was set up, where 4 L m^{-2} of water were spread twice, contemporarily to the distribution of extracts in the other plots.

In either years, durum wheat was harvested in the second half of June. At harvest time, each essay area was harvested separately, and the total obtained biomass (durum wheat and weeds) was sorted by botanical species and weighed. Samples of both durum wheat and weeds were dried in the stove (24 h at 105 °C, until constant weight) to determine their moisture content, in order to convert all biomass measurements in dry matter. In wheat, the number of spikes per unit area (m^2) was measured. Thereafter, wheat biomass was partitioned between grain and straw (g m^{-2}) and the Harvest Index value (%) for each treatment, given by the percent ratio between grain and total biomass (including grain) yield, was calculated. On a representative sample of 30 spikes per plot (including controls), the number of spikelets per spike and number and weight of seeds per spike were counted. On a representative sample of kernels per each plot, thousand kernel weight (TKW; g) was measured, and the moisture level of kernels was assessed after drying in the stove (24 h at 105 °C, until constant weight). For consistency, all weight data were reported as dry matter.

To evaluate the success of treatments against weed population, the weed suppression ability for each treatment (S$_t$) was calculated, applying the formula (modified from [48]):

$$S_t = 100(W_u - W_t)/W_u \tag{1}$$

where W_u is the weight of weed biomass (g m^{-2} of dm) found at harvest time in unrestricted conditions of weed growth (untreated plots), and W_t is the weight of weed biomass measured in every treated plot.

From the first treatment and throughout all crop cycle until harvest, the growth and phytosanitary conditions of wheat were checked by means of periodical field surveys. The presence of weeds and their botanical composition were checked, as well. With this purpose, in each survey, two 50-cm long row segments were randomly chosen in each plot. All plants growing in these lengths (including both wheat and weeds) were counted, and weeds were botanically determined. The number of retrieved species, throughout all crop cycle and in each cropping condition (treated and untreated plots) was used as "richness" index [49].

The degree of diversity inside each plot was evaluated on each survey date through the Shannon–Weiner index H' [49]:

$$H' = \sum_{p=1}^{s} [(p_i) \times \ln(p_i)] \tag{2}$$

where s is the number of retrieved species, p_i is the frequency of the individuals of the species i ($p_i = n_i/N$, being n_i the number of individuals of the species I, and N the total number of individuals of all species).

2.3. Experimental Site and Climatic Details

The trial environment is typical of the inner hilly areas of Sicily (meso-thermo-Mediterranean climate), with a long and dry summer period and a colder winter, with few snow days and irregular rainfall. In the trial area, the average year rainfall is about 500–650 mm, mostly occurring in the autumn-winter period, whereas the spring rainfall amount is about 20% of year rainfall. Summer months are mostly dry, with no more than 10% rainy days, that are mostly torrential. In the first year (Figure 1a), the total rainfall amount reached 390 mm, distributed throughout the whole winter period, mostly between the end of January and the first ten days of February, and in the first ten days of March. As usual in the trial site, the temperatures were fairly high, with minimum values around 2 °C in December, January, and February, and maximum values spanning between 12 and 32 °C at the end of

crop cycle. In 2016 (Figure 1b) the rainfall amount was lower (209 mm from December to June) and lower temperatures were recorded in winter (throughout the second half of January to the first half of February) and in early spring (March).
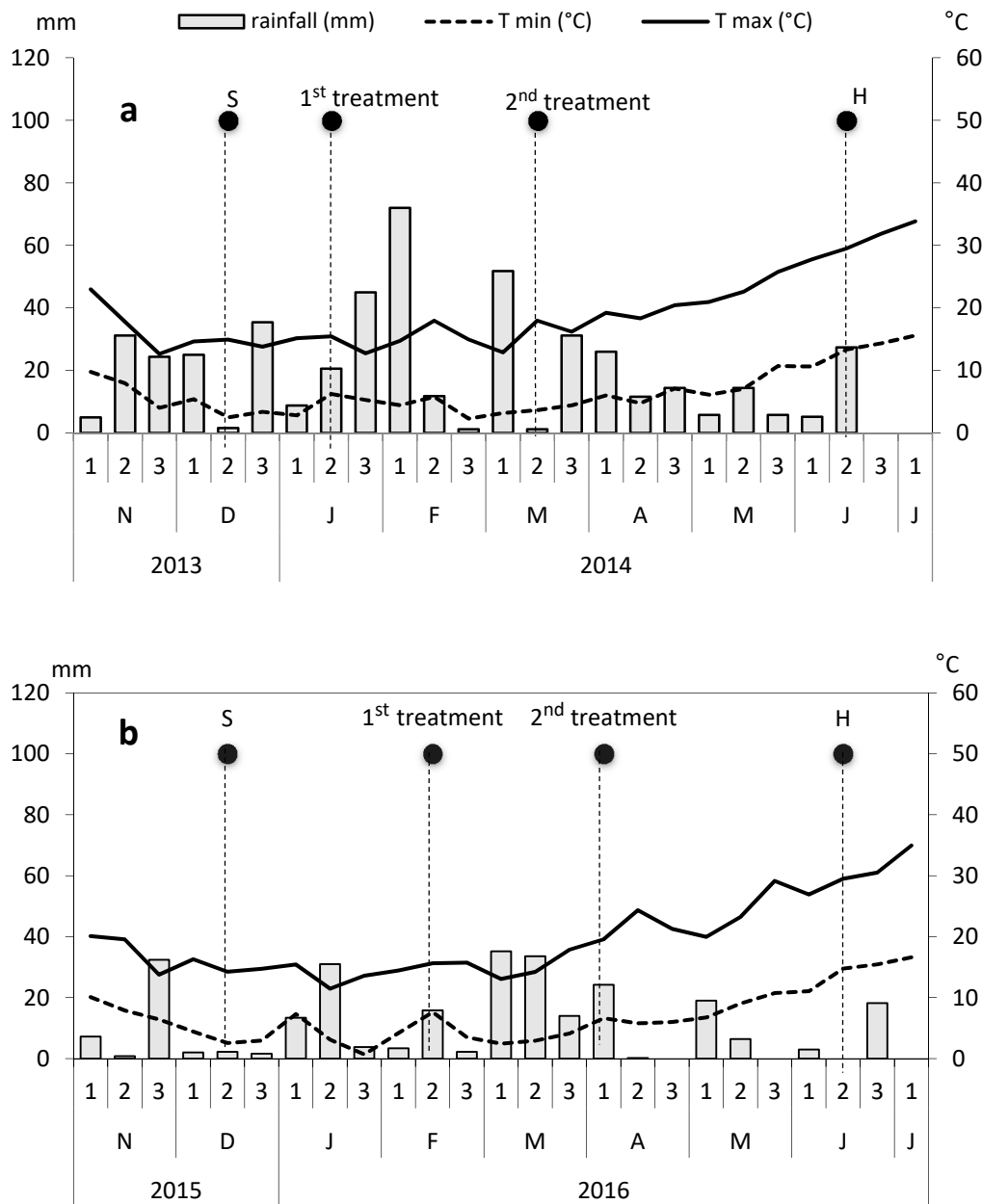
Figure 1. Cumulated rainfall (mm; bars) and mean maximum (°C; solid lines) and minimum (°C; dotted lines) temperatures recorded in 2013–2014 (a) and 2015–2016 (b) at Sparacia (Cammarata, AG, Italy). S: Sowing time; H: Harvest time; first treatment, second treatment: Dates of the two treatments with plant extracts. Data are reported as ten day values from November (N) to July (J) in both trial years.

*2.4. Statistical Data Management*

Field data were managed according to the chosen experimental layout (RCB with three repetitions), using the GLM procedure of the statistical package Minitab v. 17.1.0 (Minitab Inc., State College, PA, USA, 2013). All yield and biomass variables (height of plants, yield, and yield components) measured on wheat were considered as dependent variables, whereas year (Y) and treatments (T) were set as independent variables. All data were submitted to a preliminary individual ANOVA on a per-year

basis; the comparison between years was performed only on the treatments in common (*A. arborescens*, *R. coriaria*, chemical, water, and untreated). In all cases, when the F-test indicated statistical significance at the $p \leq 0.05$ level, Tukey's HSD test was used to evidence the differences among mean values [50]. Shannon Wiener's index was calculated by means of the PAST software [51].

3. Results

3.1. Effects of Treatments on Durum Wheat Growth and Yield

In the first trial year, the crop was favored by the satisfactory rainfall amounts, and at harvest time, plants (except for those treated with *A. arborescens* extracts) were higher than 130 cm (Figure 2). Contrastingly, in 2016, the crop was somehow constrained by the weather conditions, and plants always were shorter than 120 cm (Figure 2; Table 2).
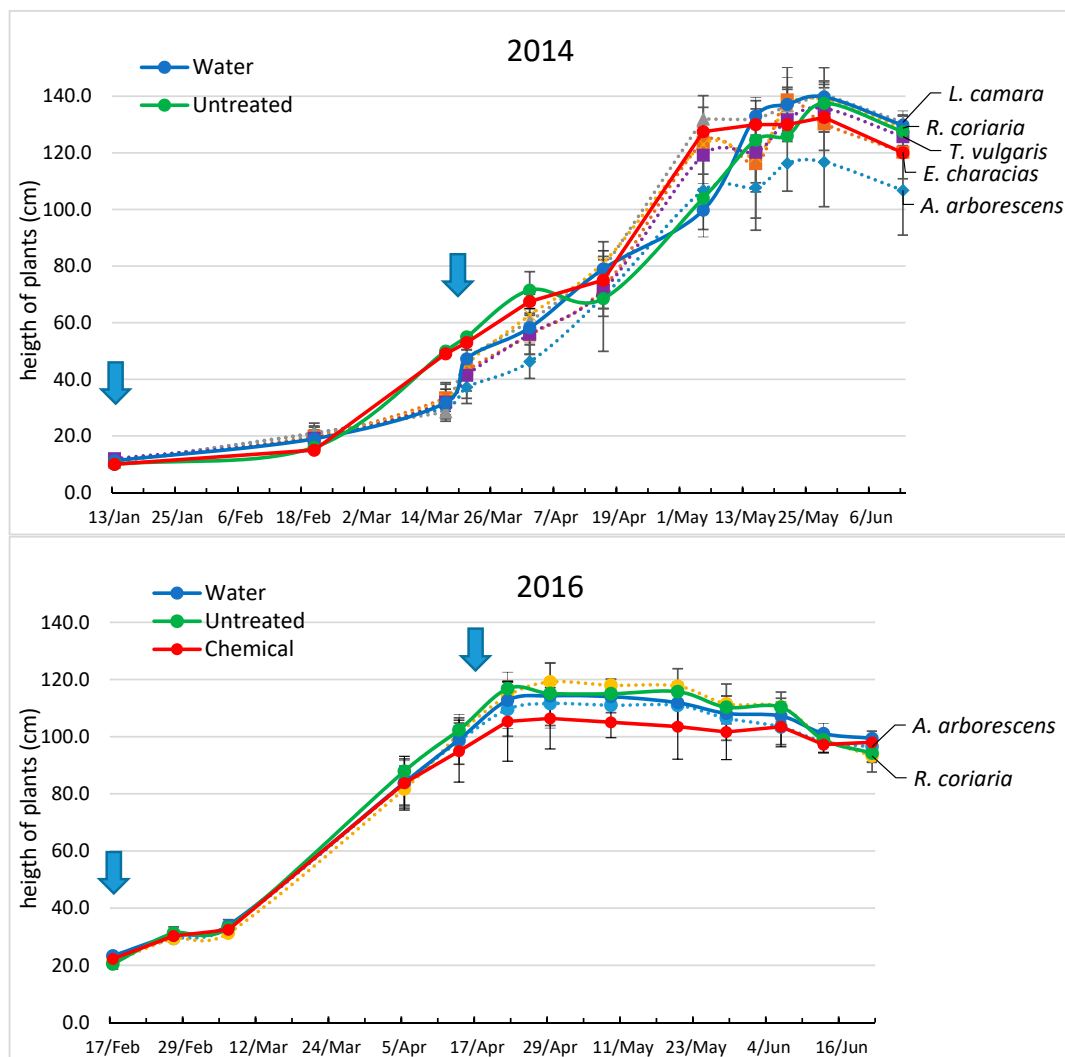
Figure 2. Height of wheat plants measured throughout the trial periods in 2014 and 2016. For each treatment and survey date, each point is the average of five measurements ± standard deviation. Solid lines refer to the controls (water, blue; untreated, green; chemical, red); dotted lines refer to the treatments with water extracts (labels at the last point of the line). In each panel, the arrows indicate the date of the two treatments.

Table 2. Mean effects ± standard deviations of treatment with plant extracts on biometrical and yield traits in durum wheat (cv Valbelice), in comparison with two controls and one chemical weeding, and calculated F values for the treatments. Within each group (2014, 2016, Year, and Treatment), values followed by the same letter are statistically not different ($p \leq 0.05$, Tukey's HSD test).

Variability Source	Plants height at Harvest Time (cm)		Plant Population at Harvest Time (n. plants m^{-2})		Grain yield (g m^{-2})		Spikes (n m^{-2})	Spikelets (n plant^{-1})		Tillers (n plant^{-1})		TKW (g)		HI (% dm)	
	2014														
Water	139.8 ± 0.3	A	81.7 ± 11.7		441.6 ± 56.0	A	293.0 ± 35.0	20.4 ± 0.2	AB	3.58 ± 0.14	A	46.6 ± 0.2	A	51.2 ± 2.1	
Untreated	137.5 ± 5.5	A	110.0 ± 23.3		392.0 ± 59.4	AB	261.5 ± 28.5	19.2 ± 0.5	AC	2.45 ± 0.68	B	43.9 ± 2.0	A	53.2 ± 0.5	
Chemical	132.5 ± 2.5	AB	105.0 ± 5.0		406.5 ± 8.3	AB	284.5 ± 13.5	18.4 ± 0.8	AC	2.71 ± 0.00	AB	42.8 ± 0.6	A	42.2 ± 1.3	
A. arborescens	116.8 ± 14.3	B	98.3 ± 11.7		256.4 ± 62.0	B	268.0 ± 3.0	17.0 ± 1.8	C	2.73 ± 0.15	AB	36.1 ± 2.0	B	41.5 ± 9.4	
R. coriaria	138.8 ± 8.3	A	93.3 ± 6.7		430.3 ± 57.6	AB	312.0 ± 46.0	20.8 ± 0.7	A	3.38 ± 0.74	AB	44.3 ± 3.2	A	43.5 ± 3.7	
E. characias	130.3 ± 7.8	AB	86.7 ± 6.7		383.1 ± 112.3	AB	313.5 ± 14.5	18.2 ± 1.5	AC	3.64 ± 0.25	A	43.5 ± 0.0	A	44.8 ± 0.6	
T. vulgaris	135.8 ± 2.8	AB	100.0 ± 0.0		365.8 ± 61.9	AB	304.5 ± 5.5	17.8 ± 0.5	BC	3.05 ± 0.06	AB	44.7 ± 1.7	A	50.8 ± 11.7	
L. camara	140.8 ± 3.8	A	108.3 ± 1.7		469.8 ± 8.6	A	317.0 ± 5.0	19.5 ± 0.1	AC	2.93 ± 0.09	AB	46.4 ± 2.5	A	46.5 ± 3.8	
2014 F value $_{(7, 16)}$ (a)	3.79 *		2.65 n.s.		3.30 *		2.36 n.s.	5.64 **		2.94 *		9.43 ***		1.87 n.s.	
	2016														
Water	104.6 ± 10.9		64.6 ± 5.5		145.8 ± 105.0		254.0 ± 103.0	16.5 ± 0.8		3.92 ± 1.57		31.8 ± 4.0		25.0 ± 12.5	
Untreated	95.1 ± 8.5		75.7 ± 16.7		167.1 ± 29.1		246.8 ± 39.3	16.3 ± 1.1		3.35 ± 0.84		37.3 ± 1.8		33.6 ± 5.0	
Chemical	100.1 ± 8.1		66.7 ± 5.5		116.5 ± 89.9		213.7 ± 106.9	16.4 ± 1.4		3.27 ± 1.56		32.4 ± 4.9		23.8 ± 17.0	
A. arborescens	95.7 ± 4.8		62.5 ± 14.6		110.6 ± 93.6		234.8 ± 93.0	16.0 ± 0.6		3.53 ± 1.27		33.1 ± 9.7		23.2 ± 16.8	
R. coriaria	103.2 ± 5.3		68.8 ± 6.3		192.6 ± 58.7		275.5 ± 43.6	16.4 ± 1.3		4.09 ± 0.44		37.1 ± 2.9		33.0 ± 2.6	
2016 F value $_{(4, 10)}$ (b)	<1 n.s.		<1 n.s.		<1 n.s.		<1 n.s.	<1 n.s.		<1 n.s.		<1 n.s.		<1 n.s.	
	Year (Y) (c)														
Mean 2014 (c)	133.1 ± 11.0	A	97.7 ± 15.2	A	385.4 ± 82.2	A	283.8 ± 31.1	19.2 ± 1.6	A	2.99 ± 0.61		42.7 ± 4.0	A	46.3 ± 6.4	A
Mean 2016	99.7 ± 7.7	B	67.6 ± 10.3	B	146.5 ± 74.9	B	245.0 ± 73.0	16.3 ± 0.9	B	3.64 ± 1.08		34.3 ± 5.2	B	27.7 ± 11.5	B
Y F value $_{(1,20)}$ (c)	135.25 ***		45.76 ***		92.88 ***		2.94 n.s.	56.91 ***		3.69 n.s.		32.29 ***		29.86 ***	
	Treatment (T) (c)														
Water	122.2 ± 20.5	A	73.1 ± 12.4		293.7 ± 178.6	AB	273.5 ± 72.0	18.4 ± 2.2	A	3.76 ± 1.01		39.2 ± 8.5		38.10 ± 16.44	
Untreated	116.3 ± 24.1	AB	92.8 ± 26.1		279.5 ± 130.1	AB	254.2 ± 31.8	17.8 ± 1.7	AB	2.92 ± 0.87		40.6 ± 4.0		43.40 ± 11.24	
Chemical	116.3 ± 18.5	AB	85.8 ± 21.5		261.5 ± 168.8	AB	249.1 ± 78.4	17.4 ± 1.5	AB	2.94 ± 0.94		37.6 ± 6.5		32.98 ± 14.76	
A. arborescens	106.2 ± 14.9	B	80.4 ± 22.9		183.5 ± 106.9	B	251.4 ± 61.6	16.5 ± 1.3	B	3.25 ± 1.05		34.6 ± 6.5		32.31 ± 15.73	
R. coriaria	121.0 ± 20.4	A	81.0 ± 14.7		311.4 ± 140.2	A	293.8 ± 44.8	18.6 ± 2.6	A	3.69 ± 0.64		40.7 ± 4.8		38.26 ± 6.41	

Table 2. *Cont.*

Variability Source	Plants height at Harvest Time (cm)	Plant Population at Harvest Time (n. plants m^{-2})	Grain yield (g m^{-2})	Spikes (n m^{-2})	Spikelets (n plant^{-1})	Tillers (n plant^{-1})	TKW (g)	HI (% dm)
T *F* value $_{(4, 20)}$ (c)	3.83 *	2.16 n.s.	3.20 *	<1 n.s.	4.04 *	1.10 n.s.	2.39 n.s.	1.42 n.s.
Year (Y) × Treatment (T) (c)								
YxT *F* value $_{(4,20)}$ (c)	1.48 n.s.	<1 n.s.	<1 n.s.	<1 n.s.	2.60 n.s.	<1 n.s.	1.80 n.s.	<1 n.s.

(a): Results of univariate ANOVA for 2014 data (DF: 7, 16)
(b): Results of univariate ANOVA for 2016 data (DF: 4, 10)
(c): Means and ANOVA are referred only to treatments in common to both years
Significance of *F* values: *: $P \le 0.05$; **: $P \le 0.01$; ***: $P \le 0.001$; n.s.: not significant

In both years no apparent difference in plant height could be noted among treatments, except for some advantage of untreated and chemically treated plots in the earlier growth stages, that was, however, balanced as growth season was going on. At harvest time in both years, the lowest height values were found on wheat treated with extract of *A. arborescens*, although in 2016 this value was not statistically different from the others (Table 2).

Among the treatments tested in either years, none of the examined biometrical and yield traits (Table 2) showed at ANOVA a significant Year × Treatment interaction. That means, differences among treatments were not significantly affected by the experimental year, and the "year" effect was the same in all tested treatments.

The main effect of both years and treatments were otherwise significant in many cases. Grain yield, number of spikes per plant, number of spikelets per spike, TKW, and HI were all higher in 2014 than in 2016.

Grain yield (Table 2) in the first year was more than twice as in 2016. Concerning the main effect of treatments, those with plant extracts reached the highest and the lowest yield value, respectively for *A. arborescens* (183.5 g m^{-2}) and *R. coriaria* (311.4 g m^{-2}). In the first year, however, the highest yield value (470 g m^{-2} dm) was found in the *L. camara* treatment (tested only in 2014), and the lowest (256.4 g m^{-2} dm) in the *A. arborescens* treatment. The number of spikes per area unit counted at harvest time averaged values between 238 and 287 in 2014 and 214 to 275 in 2016, without showing any significant difference among years or treatments. The number of spikelets per spike exhibited the highest mean value in the plants previously treated with extracts of *R. coriaria*. The number of tillers per plant was higher in the water control and in the treatment with *R. coriaria* extracts; high values were retrieved also in the treatments with *E. characias* and *T. vulgaris*, which however were excluded from pooled ANOVA, being tested only in 2014. The highest mean value of TKW was recorded in the first year (42.7 g), when significant differences showed up between the treatment with *A. arborescens* (36.1 g) and all the other treatments.

The HI (%) showed significant differences only between years, being almost unaffected by treatments. On average, HI ranged between 25% (water control in 2016) and 53.2% (untreated plots in 2014). Values of HI > 50%, demonstrating that more than half of the produced biomass was represented by grain, were obtained in three cases only, all of them in 2014: Water control (51.2%), untreated (53.2%), and *T. vulgaris* (50.8%).

3.2. Effect of Treatments on Weed Population

The values of total dry biomass (wheat + weeds) recorded at harvest time in treated and untreated plots (Figure 3) were submitted to ANOVA both as cumulated values and sorted between components, i.e., accounting for wheat biomass and weeds biomass, separately (ANOVA results not shown). The factor "year" resulted significant in all analyses, whereas treatments and Y × T interaction were highly significant only on dry matter values of measured weed biomass. Hence, all measured biomass values (wheat, weeds, and the sum of both) were, on average, significantly higher in 2014 than in 2016, but the effect of treatments was significant only on weeds biomass, and such effect was variable according to the year. In both years, although there was no noticeable presence of weeds in the chemically treated plot, neither wheat biomass nor wheat grain yield were significantly higher after chemical weeding. In 2014, the highest weed biomass was retrieved in the untreated plots (255 g m^{-2} dm, sharing 25.7% of total biomass) and in the control plots with water (245 g m^{-2}, i.e., 22% of total mean biomass). In 2016, weed incidence in the control plots was comparatively lower (in the water controls 43.4 g m^{-2}, i.e., 7.8% of total biomass, and in the untreated plots 27.7 g m^{-2}, i.e., 5.2% of total biomass). In 2016, the highest weed biomass was, however, measured in the *A. arborescens* treatments (47.8 g m^{-2}, i.e., 10.2% of total biomass). Except for chemical and controls, the trend of weeds incidence on total biomass in the first year was *R. coriaria* (11.3%) < *E. characias* (15.4%) < *L. camara* (17.4%) < *T. vulgaris* (18.4%) < *A. arborescens* (20.5%). In the second year, when only two water extract treatments were tested, the trend was confirmed as *R. coriaria* (3.9%) < *A. arborescens* (10.2%) (Figure 3).
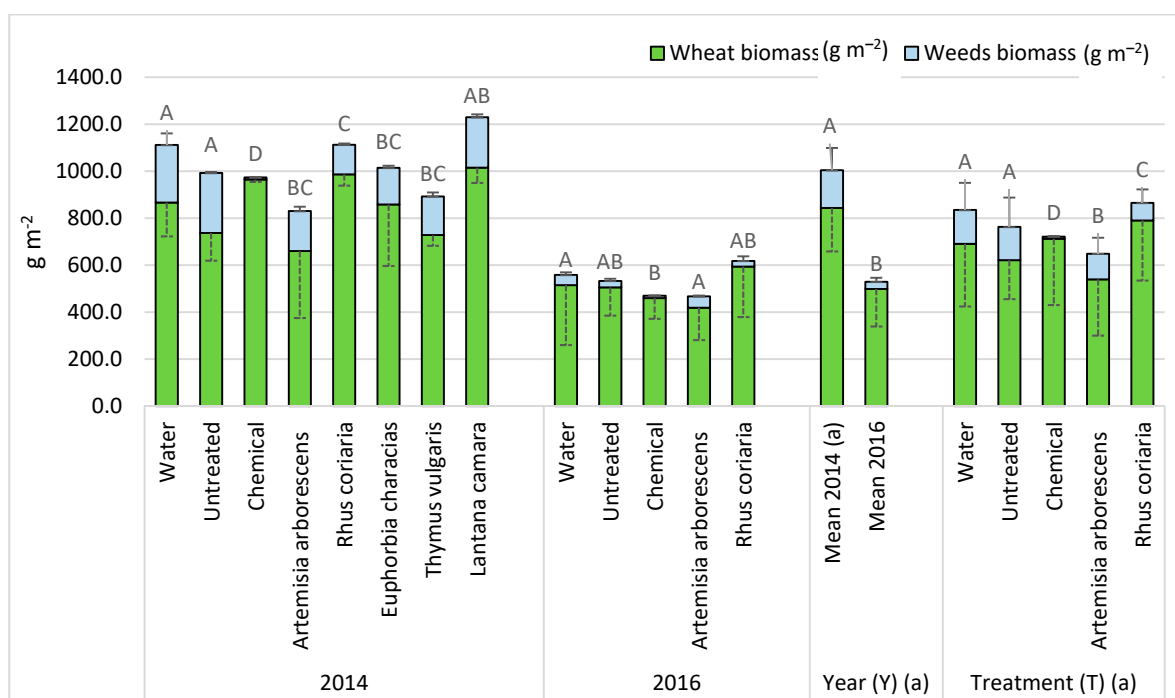
Figure 3. Total biomass (g m^{-2} dm) of wheat and weeds measured at harvest time in treated and untreated plots. Mean values marked with "a" refer only to the treatments common to both years. Error bars indicate the standard deviation of each mean, respectively for wheat (downward dotted lines) and weeds (upward entire lines). In each group (2014, 2016, Year, and Treatment), values of weeds biomass accompanied by the same letter are not different at $p \leq 0.05$ (HSD Tukey's test).

The weed suppression index ($S_t\%$) calculated on data obtained at harvest time (Figure 4) illustrates the overall effect exerted by each treatment compared to the untreated plots. The highest suppression ability was found on the chemically treated plots, that constrained weed incidence of 96.5% in the first year and 65.2% in 2016. In 2014, all treatments with water extracts gained statistically non different values of the S_t index, ranging from 50.8% (*R. coriaria*) to 16.0% (*L. camara*). In 2016, the trend was markedly different, and the most effective treatment (*R. coriaria*) suppressed weeds of 13.4% only, whereas *A. arborescens* appeared to exert a stimulating effect on weed presence, even higher than the effect exerted by water alone.

For a deeper insight of the mechanism underlying the comparison and persistence of weeds, the data retrieved in both years throughout the crop cycle were taken into consideration. Figure 5 shows the time pattern of appearing and duration inside the single plots of the retrieved weed species, as sum of the three repetitions, irrespective of their weight incidence. The botanical composition of the weeds detected at harvest time showed a differentiation among years. In the first year, in all treated plots it was possible to observe how the appearance of wild oat was definitively delayed with respect to the controls. In 2016, this outcome was confirmed for *A. arborescens*, whereas in plots treated with *R. coriaria* extracts, the appearance of *Avena fatua* was almost simultaneous to that recorded on the water control. In 2014, when the weed biomass at harvest time was much higher than in 2016, in rather all plot, irrespective if treated or not, the appearance of weeds was delayed. Contrastingly, in 2016 weed appearance was earlier, but most of weed species disappeared throughout wheat cycle, and weed biomass at harvest time was almost totally composed by *A. fatua*.
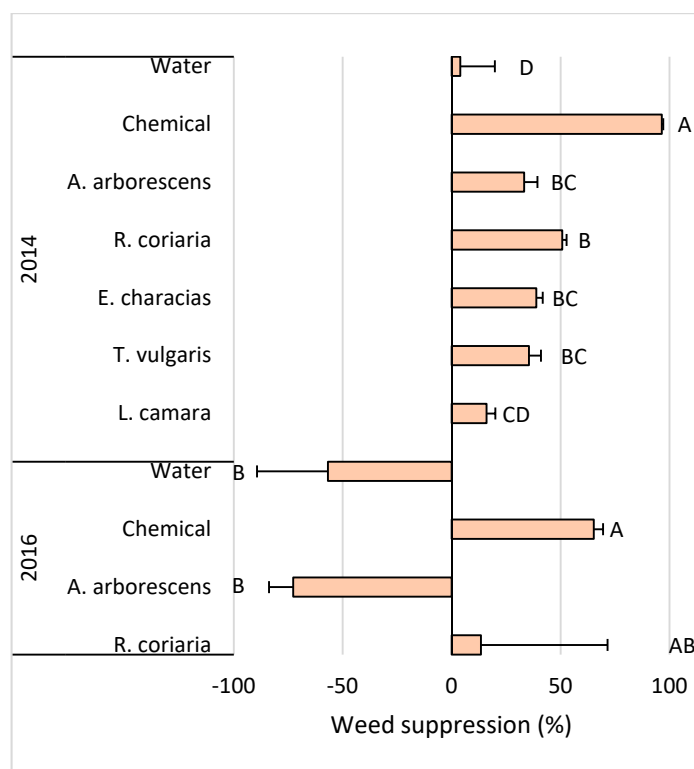
Figure 4. Weed suppression indices (S_t %) calculated in 2014 and 2016 for all treatments, including chemical and water control. Error bars indicate the standard deviation of each mean. In each year, values accompanied by the same letter are not different at $p \leq 0.05$ (HSD Tukey's test).

This outcome is also evidenced in the graphs in Figure 6, where the detected trend of the number of weed species per area unit (species richness) is reported, throughout all survey dates, and Figure 7, which illustrates the trend over time of the calculated Shannon's index in all plots. In 2014, species richness was initially low, and then shifted to higher values until harvest. Appreciable variations were found among treatments and, noticeably, the chemical treatment showed constantly the lowest values. Monocots and dicots where found in rather the same proportions in both controls and in the plots treated with *A. arborescens* and *T. vulgaris*, whereas a sharp prevalence of dicots was found on *E. characias*, and monocots were definitively prevailing in *L. camara* and *R. coriaria*. Among monocots, *Avena fatua* and *Phalaris paradoxa* showed the highest incidence, sharing from 12.7 to 37.4% of total dm weed biomass. Among dicots, wild dill (*Ridolfia segetum*) was certainly the most relevant, found in all plots with highly sized plants, where it represented 23% to 30% of total weed biomass. A significant presence (36%) of *Polygonum aviculare* was found in the plots treated with *E. characias* extracts. In 2016, the opposite trend was evidenced, and weed species number decreased over time. A more simplified weed flora was assessed, and only *Phalaris* and *Avena* were retrieved at harvest time.
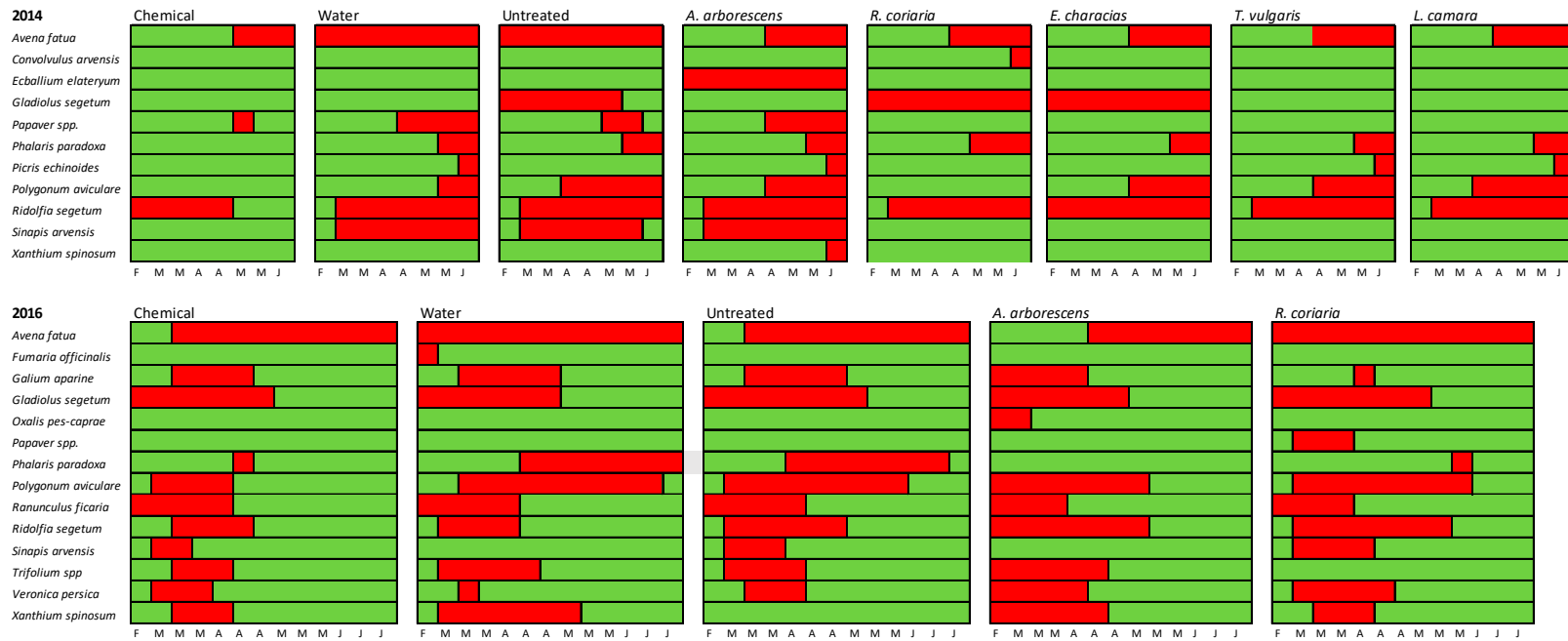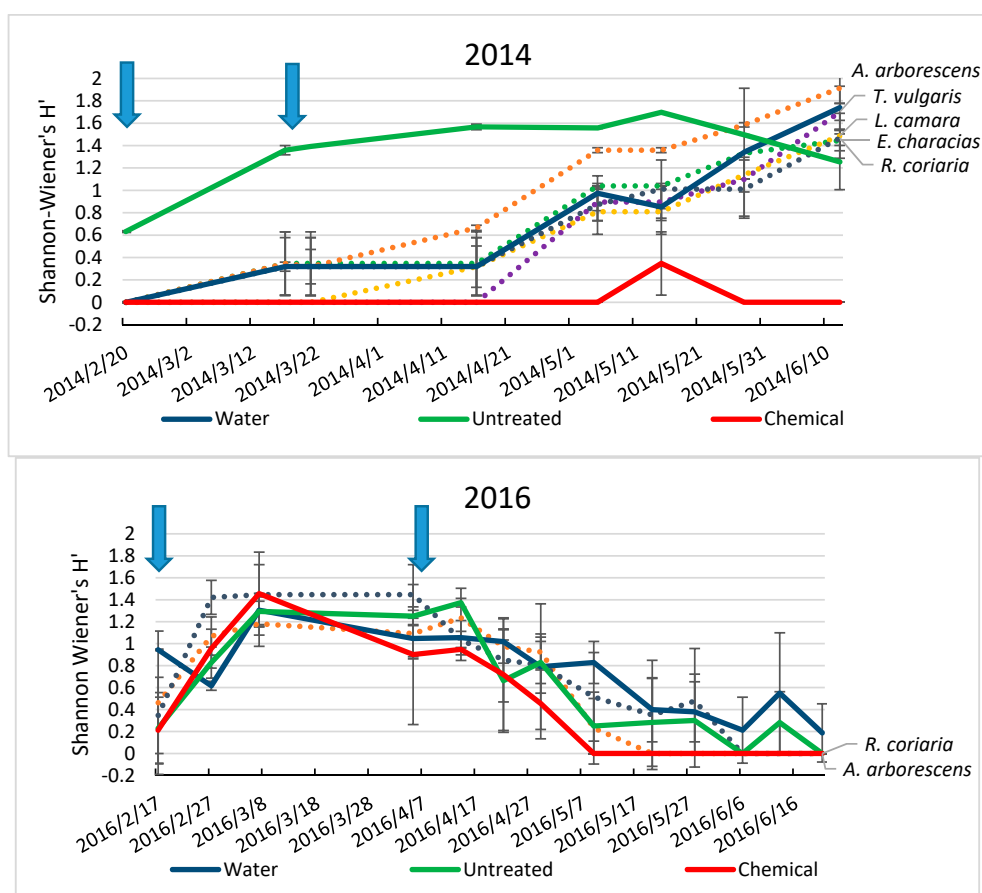
Figure 5. Time pattern of field emergence of weeds in durum wheat treated with five (2014) and two (2016) plant water extracts, compared with an untreated control, a chemical herbicide, and a control with only water. For each weed species, red areas mark the observed presence in the field from February (F) to June (J).

Since the Shannon index not only takes into account the number of species, but also the total number of individuals, it may be considered as a representation of the degree of botanical diversity inside each plot. In 2014, the diversity index was rather constantly higher in the untreated plots, and constantly lower in the chemically treated ones. All treatments took intermediate values between these two extreme series; a slight advantage of the *R. coriaria* treatments over the other treatments was detectable, but it must be noticed that in the last part of wheat cycle all water extract treatments exhibited high, and similar, values. In 2016, the diversity index showed a decreasing trend from March onward, homogeneous among all treatments (including chemical) and the controls.
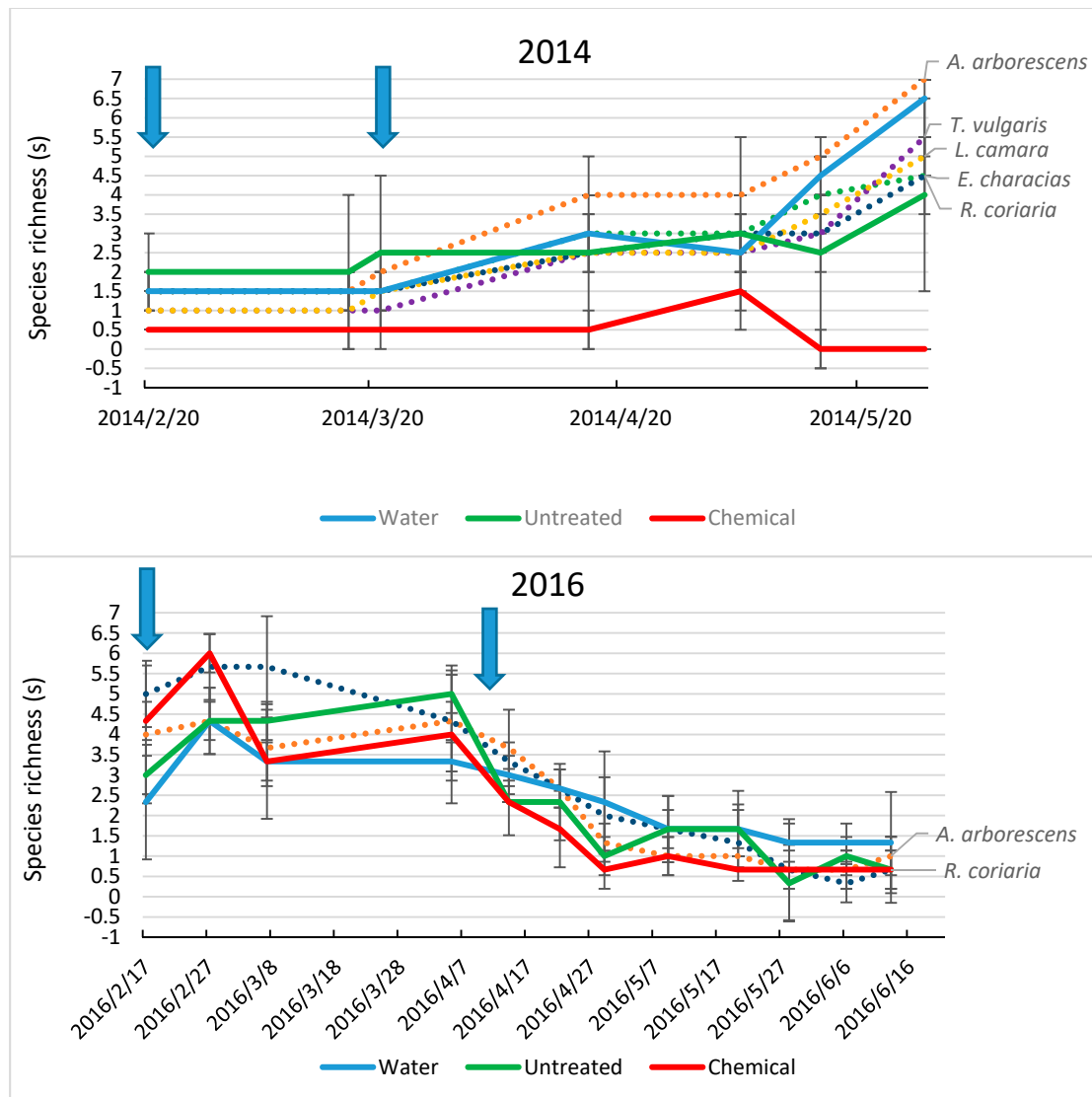
Figure 6. Species richness (n of detected weed species m^{-2}) in durum wheat treated with five (2014) and two (2016) plant water extracts, compared with an untreated control (green line), a chemical herbicide (red line), and a control with only water (blue line). Each value is the mean of three repetitions ± standard deviation. Arrows indicate the date of treatments.

Figure 7. Species diversity (Shannon-Wiener index, *H'*) in 2014 and 2016 in durum wheat treated with five (2014) and two (2016) plant water extracts, compared with an untreated control (green line), a chemical herbicide (red line) and a control with only water (blue line). Each value is the mean of three repetitions ± standard deviation. Arrows indicate the date of treatments.

4. Discussion

This work was aimed to evaluate, in field conditions, the effects on durum wheat of several water plant extracts, applied for weed control. With this purpose, not only the bare conditions of the presence/absence of weeds were accounted for, but also the possible interactions between the supplied extracts and the major growth and yield parameters of the crop.

The effect of treatments on weed population was variable between years. In 2014, dicots were in general prevailing in plots treated with extracts of *E. characias*, while monocots prevailed after treatments with *L. camara* and *R. coriaria*. In 2016, when a generally lower weed biomass was present, also a lower diversity level was found, and only the most competitive weed species (*Avena fatua* and *Phalaris paradoxa*) were detected at harvest time. The marked variability expressed by the *A. arborescens* extract on weeds, as revealed by the opposite directions shown by the calculated suppression index in the two years, may be possibly explained by a toxic effect exerted by this extract against wheat in both years and especially in 2016, when this treatment probably induced a less dense wheat canopy (fewer and shorter plants), which allowed weeds to grow and develop even more than in the untreated control.

In general, none of the tested treatments (including chemicals) was able to eradicate weeds from the field, and weeds were retrieved at harvest time in all plots. Hence, although chemically-treated plots showed in both years the highest suppression ability, some lately-sprouting weeds were found also therein. However, the fact that in both years grain yield was not significantly different between chemically treated plots and untreated ones, demonstrates that, in the chosen wheat genotype, weed control using chemical herbicide does not necessarily result in a significant increase in grain yield.

Total weed biomass did not appear to be a determinant factor in assessing wheat yields, showing on average—opposite to what was expected—the highest values in the most productive year and treatments. Both measurements (grain yield and weed biomass) were, however, significantly different according to the tested treatment. On average, *R. coriaria* always exerted a positive effect on wheat yields, and *A. arborescens* always a negative effect. A possible explanation could be that the retrieved yield differences are a consequence of the distribution of plant extract itself, rather than an effect exerted on weed biomass. An effect of *R. coriaria* extract on several quality parameters of durum wheat has been already assessed by previous experiments [26]. Further research is needed to explore these aspects.

Noticeable differences resulted in the date of appearance of major weeds, whose flush of emergence was generally earlier in 2016 than in 2014. In all treated plots in the first year, the appearance of wild oat (*Avena fatua*) was delayed with respect to the controls, but this trend in 2016 was confirmed only on plots treated with extracts of *A. arborescens*. Since wild oat and *Phalaris* spp. are among the most noxious weeds in wheat, if confirmed by further experiences, this outcome would have a great practical relevance. The delay of weed emergence is claimed to be a major factor in improving yield levels, since a longer time is at the crop's disposal to enhance its competitivity [52].

The competitive ability of the selected durum wheat genotype (cv Valbelice) resulted in a higher yield capacity even in the presence of a significant weed biomass. To explore this aspect, plant traits correlated to crop competitiveness, i.e., plant population, plant height, and tillering [53,54] were taken into consideration. All of them expressed large differences in consequence of the different climatic pattern of the two years (Y factor always highly significant). As such, climatic conditions acted giving wheat a higher competitive ability in the first year (height values always higher; plant population higher). An advantage of taller plants was evident in both years and in all circumstances, since a general trend of higher productivity with higher plants was rather always recognizable. Similarly, the yield disadvantage of shorter plant size, as retrieved in plots treated with *A. arborescens* extracts, was evident as well.

Both tiller number per plant and number of spikes per area unit resulted to be mostly density-dependent, and did not seem associated with reduced weeds.

5. Conclusions

Although certainly preliminary, this work represents a step forward in the study of weed management through allelochemicals. Although the herbicidal effectiveness of the studied extracts under the given experimental conditions was rather limited, water plant extracts confirmed exerting different—and not always predictable—effects on crop yield and development. By one side, it must be stressed that the goal of weeding is no longer the complete eradication of weeds, rather the containment of weeds population beyond an "acceptability" threshold [55–59]. By another side, the occurrence of significant effects of these extracts on crop open the way to a huge field of investigations involving agronomical, physiological, and biochemical issues. Further studies are necessary, using a broader range of crops and allelochemicals, and pointing out in detail doses and methods of application of the supplied compounds.

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