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Effects of Sustainable Rice Management on the Behavior and Bioefficacy of Bispyribac-Sodium: A Medium-Term Study

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Abstract: The practices (tillage and flooding) used for rice crops are unsustainable, especially in areas characterized by water shortages, such as the Mediterranean region. Therefore, it is necessary to develop sustainable methods in order to ensure the viability of rice production. However, it is essential to understand the effects that alternative management can have on herbicide behavior. In this context, this paper describes the first field experiment conducted to evaluate the medium-term effects of different agricultural practices on the fate of bispyribac sodium (BPS). Thus, the treatments were as follows: tillage and flooding (TF), tillage and sprinkler (TS), and no-tillage and sprinkler (NTS). In addition, “alperujo” compost (AC) from olive mill waste was used in each treatment (TF-AC, TS-AC, and NTS-AC). The AC was applied only once in 2015 when the TS and NTS treatments were implemented. The AC amendment increased the adsorption of BPS and its irreversibility, thereby decreasing the BPS leaching capacity. Furthermore, throughout this study, the BPS persistence was up to 1.85 times greater with sprinklers than in the flooding condition, which could explain the high values of BPS effectiveness (increased by a factor of 1.45 on average) found with sprinklers. Therefore, the implementation of sprinklers in combination with AC can be considered a sustainable strategy for Mediterranean rice production, at least in the medium term, as it reduces BPS water pollution and increases its weed control efficiency.

Keywords: compost; direct seeding; flooding; leaching; sprinkler



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1. Introduction

Weeds can lead to significant rice (*Oryza sativa* L.) yield losses, thereby reducing the viability of growing rice, which is a staple food for a large part of the world’s population [1]. Therefore, weed control in rice fields is crucial to ensuring global food security [2,3]. In this sense, chemical weed control is widely used in rice fields due to different advantages; however, it has a high risk of herbicide pollution, which can have adverse effects on the environment and human health. Thus, one of the main aims of the current “Farm to Fork” strategy of the European Commission is to accelerate the transition of agricultural practices to sustainable food production [4]. Therefore, in order to mitigate or prevent pollution by herbicides, sustainable and innovative agricultural rice-growing practices should be implemented [5,6]. Furthermore, the traditional practices applied to rice growing are unsustainable due to soil degradation processes associated with intensive tillage operations, as well as excessive water consumption because of permanent flooding irrigation [7]. In fact, rice farming accounts for about 40% of the world’s irrigation water [8]. This situation

is further aggravated in Mediterranean countries, where most European rice production takes place, mainly due to the lack of water resources [9]. Accordingly, recent studies have shown that the current drought has led to a significant drop in rice acreage (up to 60%) in Italy and Spain [9,10], which are major European rice producers (they account for 80% of the total rice production in the European Union). It is therefore imperative to develop effective and environmentally responsible rice production [11].

Water-saving irrigation methods have been tested in rice crops (aerobic rice, alternate wetting and drying, drip irrigation, and sprinklers). Thus, several studies have suggested that sprinkler irrigation could be an interesting alternative to flooding irrigation for rice cultivation [12,13], especially in water-stressed regions [14]. Besides saving water, another important advantage of sprinkler irrigation is that it allows the adoption of soil conservation techniques such as direct seeding and the application of organic amendments [12]. Given the deterioration of soil properties associated with the intensive tillage operations used in rice production [15], the combination of agricultural conservation practices and sprinkler irrigation has been considered a promising alternative to conventional management, offering improved resource use efficiency (water and soil) [5,7]. Nevertheless, the adoption of water-saving methods could be disregarded by rice farmers due to the potential yield losses [16]. In fact, Pinto et al. [12] indicated that around 70% of the rice yield could be lost due to inadequate water management under a sprinkler irrigation system in Brazil. Similar results have been reported in different rice-growing regions, suggesting that rice yields under sprinkler irrigation could be improved as crop management practices are further developed and refined [17]. In this sense, the combination of sprinkler irrigation and organic amendments has been proposed as an efficient alternative to flooding in order to ensure the sustainability of rice crops, at least in regions facing water scarcity [5,18]. Hence, the application of organic amendments to soil has a great impact. It improves soil properties and the water-holding capacity, which allows the optimal development of crops and yield enhancement [19,20].

In the Mediterranean area, the olive oil industry produces a large amount of an olive mill waste byproduct known as alperujo. Due to its high organic matter content, it can be used as an organic amendment, especially after a composting process [21]. Thus, different studies have indicated the positive effects of alperujo compost (AC) application on soil properties. It restores soil fertility [22,23], including the fertility in rice soils [18], while also promoting a circular economy. Nevertheless, the implementation of these agricultural practices (water-saving methods, no-tillage methods, and the application of organic amendments) strongly influences the properties of soil, which can alter the environmental fates of herbicides [6,24].

Bispyribac sodium (BPS) is an herbicide that is extensively used in rice fields because it can control a wide range of weeds [5]. However, reports about its environmental consequences are meager [5,25]. Nevertheless, a study showed that the low adsorbed BPS capacity of the soil was likely due to its high water solubility (64 g L^{-1}), as well as its low octanol/water partition coefficient [26]. Furthermore, BPS has relatively high persistence. López-Piñeiro et al. [5] reported $t_{1/2}$ values of up to 86.4 days in a Mediterranean rice ecosystem, which could enhance its potential to contaminate water resources through leaching processes [27]. In fact, in water samples collected in rice production areas, BPS was detected at concentrations above the permitted levels [28], which indicates a serious environmental hazard. In this sense, the use of organic amendments in rice soils has been proposed as a measure to mitigate the risk posed by herbicides [5,6]. Nevertheless, other authors have observed decreases in herbicide efficacy in amended soils, which could affect weed control, indicating a need for additional herbicide applications [29,30]. Similarly, López-Piñeiro et al. [5], using a different rice production system, observed that the application of biochar could reduce the efficacy of BPS, although this effect only persisted under flooding conditions after biochar aging under natural field conditions.

Despite the unsustainability of traditional rice crop management (intensive tillage and permanent flooding), particularly in water-stressed regions such as the Mediterranean

Basin, research about the effects of alternative rice management on BPS behavior is limited [5,18,26,30–32], despite its extensive use in rice agroecosystems. In fact, we found only one published study about the effects of the combined use of AC and sustainable agricultural rice management on BPS behavior, which was one of our own previous research publications [18]; however, only short-term effects (≤ 3 years) were determined. Therefore, in order to contribute to the correct decision making of rice farmers to improve the sustainability of rice cultivation, further research studies are required. In addition, the dynamics of herbicides in soils could be time-dependent due to the evolution and transformation of soil and organic amendment properties over time under different rice management systems [5,33]. In this context, reports showed decreases in carbon stocks in agricultural soils after short-term applications of no-tillage management [34,35]. However, significant increases were observed in the medium term [36,37]. Likewise, whereas Li et al. [38], in a short-term study on rice soil, indicated that soil pH and enzyme activities were not affected by different tillage management practices (conventional versus no-tillage management), Roldan et al. [36] showed the highest enzymatic activity values under a medium-term no-tillage implementation. Furthermore, after a 5-year period, López-Fando and Pardo [39] reported that no-tillage soil exhibited strong vertical pH gradients, whereas the pH values were more uniform at different depths under conventional tillage. In fact, Liben et al. [40] concluded that they could observe medium-term positive effects of agricultural conservation practices (no-tillage plus residue retention) on soil properties but these effects were uncertain in the short term. Therefore, due to the impact of these soil properties on the behavior of herbicides, it is necessary to develop medium- and long-term studies to validate the results regarding the behavior of BPS under different agricultural systems. In this context, this research is the first to evaluate the medium-term effects on adsorption–desorption, dissipation, leaching, and efficacy of BPS, after five years of the transition from flooding to sprinkler irrigation, with and without tillage management, in combination with alperujo compost (AC) application. Thus, this information would be invaluable in planning its management and appropriate environmental protection.

As a general objective, this research was the first to evaluate the medium-term effects of sustainable rice management on the behavior and bioefficacy of BPS. For this purpose, the adsorption–desorption, dissipation, leaching, and efficacy of BPS were measured five years after transitioning from flooding to sprinkler irrigation, with and without tillage management, in combination with alperujo compost (AC) application. We hypothesized that (1) the implementation of sprinkler irrigation instead of flooding, especially under no-tillage conditions, would be an effective strategy to reduce the leaching of BPS herbicide in the medium term, although its effectiveness could be affected, and that (2) due to the evolution and transformation of soil properties over time, the medium-term effects of rice management on BPS behavior could be different from those observed in the short term.

2. Materials and Methods

2.1. The Herbicide and the Analytical Procedure

An analytical standard of BPS ($>98\%$ purity) was purchased from Dr Ehrenstorfer, GmbH (Germany) (Figure 1). Its properties were as follows: a molecular weight of 452.4 g mol^{-1} , $\log \text{Pow} = -1.3$, a water solubility of 64 g L^{-1} at 20°C , a vapor pressure of $5.5 \times 10^{-6} \text{ mPa}$ at 20°C , and a pK_a of 3.35 at 25°C [41]. Concentrations of BPS were analyzed using a Waters 600E HPLC (Waters, Milford, MA, USA) system coupled to a Waters 996 diode-array detector (Water, USA). Separation was carried out with a Nova-Pack C18 column ($150 \text{ mm length} \times 4.6 \text{ mm i.d.}$) at 30°C (Waters, Wexford, Ireland). A mixture of 0.1% phosphoric acid ($>85\%$, Panreac, Castellar del Vallès, Spain) and acetonitrile (≥ 99.9 , Fisher Scientific, London, UK) ($55:45 \text{ v/v}$) was used as the mobile phase at a flow rate of 1 mL min^{-1} (isocratic conditions) and the wavelength monitored for UV detection was 248 nm . External calibration curves were obtained by analyzing BPS standard solutions at concentrations ranging from $0.05 \text{ }\mu\text{M}$ to $10 \text{ }\mu\text{M}$. The limits of detection (LOD) and quantification (LQD) were $0.007 \text{ }\mu\text{M}$ and $0.022 \text{ }\mu\text{M}$, respectively.

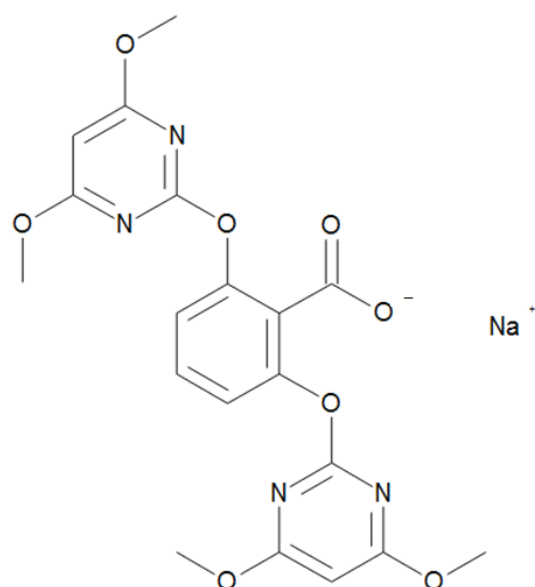


Figure 1. Chemical structure of the herbicide BPS.

2.2. Design of the Experiments, Sampling, and Assay

A field experiment was carried out in a rice-growing area located in south-eastern Spain ($38^{\circ}55' \text{ N}$; $6^{\circ}57' \text{ W}$) in a semi-arid Mediterranean climate (with a mean annual temperature of 16.2° C and rainfall of 460 mm). For a long time (>14 years), the experimental area was dedicated to rice (*O. sativa* L.) cropping using traditional practices (permanent flooding irrigation and tillage). Hence, the soil in the experimental area was a Hydric Anthrosol [42]. After the rice harvest in 2014, six different management practices were implemented in a completely randomized design: rice growing under conventional tillage and flooding irrigation without (TF) and with the application of AC (TF-AC), rice growing under conventional tillage and sprinkler irrigation without (TS) and with the application of AC (TS-AC), and rice growing under no-tillage (direct seeding) and sprinkler irrigation without (NTS) and with the application of AC (NTS-AC). Each management practice was carried out in triplicate. Thus, the experimental area had 18 plots in total (180 m^2 for each plot, as shown in Figure 2), which were previously characterized to guarantee soil homogeneity in the different plots. At the beginning of the experiment, the main properties of the original soils were as follows: loam texture (20.8% clay, 50.3% sand, and 28.9% silt), pH of 4.42 ± 0.07 , $12.6 \pm 0.28 \text{ g kg}^{-1}$ of total organic carbon, and $3.50 \pm 0.01 \text{ dS m}^{-1}$ of electrical conductivity. The AC was applied at a rate of 80 Mg ha^{-1} in 2015 and its characterization can be found in Gómez et al. [18]. Briefly, days prior to sowing (at the end of April), a complex fertilizer (9-18-27, Fertiberia, Madrid, Spain) was applied at a rate of 550 kg ha^{-1} in all management conditions. Then, spring moldboard plowing was carried out for the tillage conditions (TF, TF-AC, TS, and TS-AC). Afterward, rice was sown (at a dosage of 160 kg ha^{-1} using seeds of the Sirio variety that were purchased from COPSEMAR, Madrid, Spain) using a Semeato TDNG 320 Disc Seeder (Semeato, Passo Fundo, Brazil) for the sprinkler conditions (TS, TS-AC, NTS, and NTS-AC) and an Amazone za-x-perfect broadcast seed drill (Amazone, Munich, Germany) for the flooding conditions (TF and TF-AC). Rice was sown as the sole crop in early May of each year. In both years, urea (Fertiberia, Madrid, Spain) was applied as a cover fertilizer at dosages of 92 kg N ha^{-1} and 69 kg N ha^{-1} in the tillering (July) and initial panicle (August) stages, respectively. Finally, in all conditions, rice was harvested in September using a Bertolini 124D reaper (Emak, Piano, Italy). Furthermore, the TF and TF-AC conditions were continuously flooded (from May to September), whereas in the TS, TS-AC, NTS, and NTS-AC conditions, the rice was irrigated with a sprinkler system.

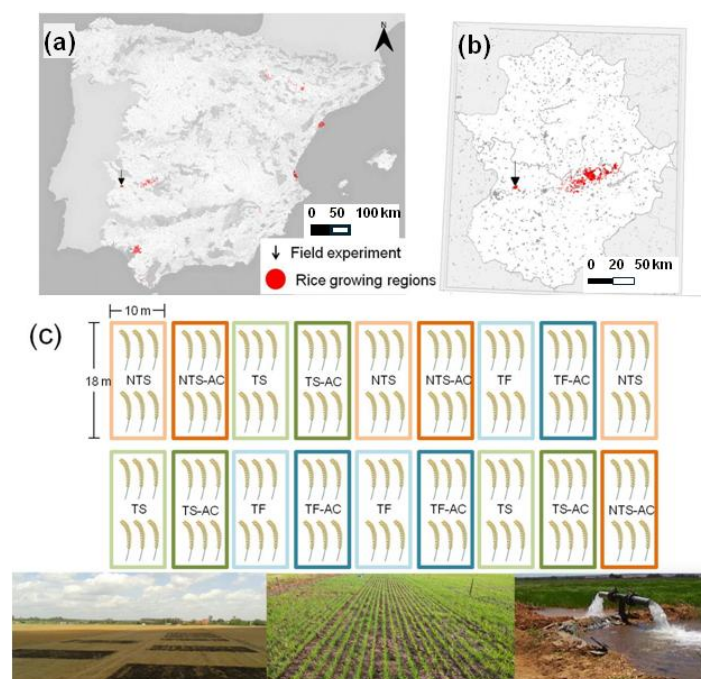


Figure 2. Main rice growing areas in Spain (a) and Extremadura (b). Plot distribution in the study area and different images of the field experiment (c).

Hence, in order to determine the medium-term effects, after the rice harvests (when the different rice management practices were completed) in September 2018 and 2019 (four and five years after the implementation of the management practices, respectively), four subsamples of topsoil (0–20 cm, arable layer) were collected randomly for each of the plots with the help of a manual auger (70 mm diameter) to make a composite sample that was used to determine BPS adsorption–desorption, dissipation, leaching, and efficacy. The soil samples were air-dried and ground and the fraction that passed through a 2 mm sieve was stored at 4 °C until use. The soil properties were analyzed as described by Peña et al. [43] and are compiled in Table 1.

Table 1. Selected soil properties (0–20 cm depth).

	TOC (g kg ^{−1})	WSOC (mg kg ^{−1})	HA (g kg ^{−1})	FA (g kg ^{−1})	pH	EC (dS m ^{−1})
<i>2018</i>						
TF	10.8aB	293abB	0.858aA	0.831bA	5.52aA	1.93bA
TF-AC	20.9cA	716cA	1.48bA	0.971cA	5.92bA	1.52aA
TS	10.2aA	257abA	0.911aA	0.891bA	6.27cA	1.87bA
TS-AC	16.6bA	721cB	1.41bA	1.25eA	6.89eB	2.08cA
NTS	11.1aA	211aA	0.869aA	0.559aA	6.73dB	1.45aA
NTS-AC	16.8bA	392bA	1.53bA	1.05dA	6.94eB	1.79bA
<i>2019</i>						
TF	10.1aA	240aA	0.833abA	0.939bA	5.64aB	2.74bB
TF-AC	21.4eA	562cA	1.44cA	0.996bA	6.11bB	2.65bB
TS	11.6bB	356bB	0.736aA	1.00bA	6.29cA	2.12aA
TS-AC	18.6dB	402bA	1.30cA	1.18cA	6.62eA	3.93cB
NTS	12.3cA	333bB	0.958bA	0.775aB	6.46dA	5.97dB
NTS-AC	21.6eB	357bA	1.96dA	1.04bA	6.61eA	3.73cB
M	***	***	***	***	***	***
Y	***	**	NS	*	***	***
M × Y	**	***	*	NS	***	***

TOC: Total organic carbon; WSOC: Water soluble organic carbon; HA: Humic acid; FA: Fulvic acid; EC: Electrical conductivity. Managements are tillage and flooding irrigation without (TF) and with the application of alperujo compost (TF-AC), conventional tillage and sprinkler irrigation without (TS) and with the application of alperujo compost (TS-AC); no-tillage (direct seeding) and sprinkler irrigation without (NTS) and with the application of alperujo compost (NTS-AC). ANOVA factors are M: management; Y: year; M × Y: interaction management × year. F-values indicate the significance levels * $p < 0.05$; ** $p < 0.01$; and *** $p < 0.001$, respectively, and NS: not significant. Different letters indicate significant differences ($p < 0.05$) between managements in the same year (lower case letters) and between years within the same management (upper case letters).

2.3. Adsorption–Desorption Experiments

For all management conditions, BPS adsorption–desorption isotherms were obtained using a batch equilibration method in accordance with Gómez et al. [18]. Briefly, in triplicate, 5 g soil samples were treated separately with 10 mL BPS solutions at initial concentrations ranging between 0.5 and 20 μM . The BPS adsorption–desorption data were fitted to the Freundlich model. Detailed information from this adsorption–desorption study is presented in the Supplementary Materials (Text S1 and Figure S1).

2.4. Dissipation Experiments

The dissipation of BPS was carried out under two incubation conditions. Thus, flooding was used in the TF and TF-AC conditions, whereas non-flooded conditions were used in the TS, TS-AC, NTS, and NTS-AC conditions, in order to simulate the field conditions. BPS was applied at a rate of $1.63 \mu\text{g g}^{-1}$. Samples were incubated in darkness at $20 \pm 1^\circ\text{C}$ for 49 d. At appropriate time intervals, triplicate samples (for each management condition) were removed and immediately frozen until further analysis. A mixture of distilled water and methanol (≥ 99.9 , Fisher Scientific, UK) (60:40 v/v) was used as an extractant phase. Half-lives ($t_{1/2}$) were calculated on the basis of the experimental data after they were fit to a first-order kinetics equation. Furthermore, simultaneously with the dissipation experiments and using the same conditions, the activity of dehydrogenase (DA) was also determined in accordance with Trevors [44]. Detailed information from this dissipation study is presented in the Supplementary Materials (Text S2 and Figure S2).

2.5. Leaching Experiments

Disturbed soil columns (5 cm inner diameter $\times$ 30 cm length, constructed from PVC) were used to determine the leaching of BPS, in triplicate, for each management condition. Prior to the application of BPS, the columns were saturated using CaCl_2 (0.01 M). Then, the herbicide was applied to the tops of the soil columns at a rate of 0.5 kg ha^{-1} . Daily, 50 mL of CaCl_2 (0.01 M) was applied to each column and the leachates were collected and frozen until further analysis. Detailed information from this leaching study is presented in the Supplementary Materials (Text S3 and Figure S3).

2.6. Bioassays

A laboratory bioassay was conducted according to López-Piñeiro et al. [5] to determine the medium-term effects of AC on the efficacy of BPS in soils that had been subjected to different tillage and water management conditions. Briefly, 50 g samples of the soil were put into pots. The soils under flooding irrigation (TF and TF-AC) were incubated at a soil-to-water ratio of 1:1.25 w/v and those under non-flooding irrigation (TS, TS-AC, NTS, and NTS-AC) were at 80% of field capacity. The pots were placed inside a growth chamber and kept at 25°C with 12 h of daylight for 14 days. Then, for each management condition, weeds were removed by hand and 10 pre-germinated seeds of *Echinochloa crus-galli* L. (one of the main rice weeds in Europe [1]) were put in each pot. After 10 d, BPS was applied to one set of pots at the recommended dosage of 100 g ha^{-1} , leaving another set as controls without herbicide. After 14 d of BPS application, the weights of *E. crus-galli* L. were measured to determine the weed control efficacy (Figure S4).

2.7. Statistical Analyses

Statistical studies (ANOVA, Duncan's test, Pearson's correlation) were done with the use of the IBM's SPSS (vn. 22) software package. A p -value > 0.05 was considered to indicate statistical non-significance.

Statistical analyses were carried out using IBM's SPSS (vn. 22) software package. The data were checked for normality of distribution and homogeneity of variances. Then, they were subjected to a one-way ANOVA considering each variable alone (management and year) to evaluate the statistical differences between the management conditions within each year and in the same management condition in different years. A post hoc Duncan test

was used to further elucidate differences among means ($p < 0.05$). To evaluate the significance and interaction of the tested variables (management $\times$ year), a two-way ANOVA was carried out. Three levels of significance were considered: $p < 0.05$, 0.01, and 0.001. Pearson correlation coefficients were calculated to study possible correlations between different properties. Correlations were considered using two levels of significance: $p < 0.05$ (statistically significant) and $p < 0.01$ (a higher level of statistical significance). Furthermore, the influence of soil properties on BPS behavior was determined via multivariate regression analysis.

3. Results and Discussion

3.1. Adsorption–Desorption Experiments

Adsorption–desorption isotherms of BPS for the different management systems are shown in Figure S5, which shows adequate fitting to the Freundlich model ($R^2 \geq 0.982$, Table 2). Regardless of the management condition and the year of study, the values of n_f (< 1) indicate that the adsorption of BPS was dependent on its initial concentration. Similar results were reported by other authors, who also found values of n_f less than unity in different types of soils, indicating strong competition for adsorption sites [30,45]. The management conditions significantly affected BPS adsorption (Table 2). Thus, for the unamended conditions, after five years of implementation, the results indicated higher soil capacities (K_d) under flooding (TF) than in the sprinkler conditions, regardless of the tillage system (TS or NTS, Table 2). This finding is consistent with previous research, such as that of Gómez et al. [18], who observed that the BPS adsorption capacity was greater under anaerobic management than under aerobic management in Mediterranean rice fields, although this was a short-term effect. Such results could be due to the pH of soils because an increase in pH, as found under sprinkler irrigation (Table 1), can lead to increases in the degree of ionization of BPS (anionic forms), resulting in less adsorption [31]. This is in agreement with the results of Sharma et al. [46], who showed more adsorption of BPS in acidic soils (pH = 5.2) compared to basic soils in rice soils from India, suggesting the importance of soil pH for BPS adsorption. Compared to the unamended management conditions, significant increases in K_d values were observed in the AC-amended conditions, regardless of the irrigation and tillage systems. Hence, the BPS K_d values were 1.10, 1.10, and 1.32 and 1.17, 1.36, and 1.49 times greater in the TF-AC, TS-AC, and NTS-AC conditions than in the corresponding unamended conditions for 2018 and 2019, respectively (Table 2). Higher adsorption of BPS in the AC-amended management conditions could be due to increases in total organic carbon (TOC) (Table 1). In fact, K_d was significantly and positively ($p < 0.01$) correlated with TOC ($r = 0.424$). These results are in agreement with the results observed by Chirukuri and Atmakuru [27], who indicated a positive influence of TOC on BPS adsorption in different soil types. However, Gómez et al. [18] reported no significant differences in BPS adsorption capacity between unamended and AC-amended management conditions in the short term, especially under sprinkler management. These differences could be attributable to greater variations in soil pH after applying AC in the short term than in the medium term. Thus, whereas the short-term application of AC caused increases in soil pH of up to 1.22-fold, in the medium term, these increases were much lower (up to 1.10-fold). Indeed, K_d was significantly and negatively ($p < 0.01$) correlated with pH ($r = -0.711$), in line with previous studies [27,31], due to an increase in repulsive forces as pH increased. These findings show the importance of considering more than short-term effects in order to validate the effects on the behavior of herbicides. Furthermore, according to Green and Hale [47], the solubility of BPS could increase with soil pH, making it more hydrophilic and more available in solution form, thus decreasing its tendency to be adsorbed. Furthermore, after the multivariate analysis (multiple regression), we observed that 90% of the variance in K_d was explained by a combination of pH and TOC variables using the following equation:

$$K_d = 2.873 - (0.448\text{pH}) + (0.031\text{TOC}), R = 0.949 \ (p < 0.001)$$

Table 2. Bispyribac-sodium adsorption–desorption parameters.

	n_f	K_d	R^2	H
<i>2018</i>				
TF	0.929abcA	0.973dB	0.991	386aA
TF-AC	0.979cA	1.067eB	0.988	481abA
TS	0.909abcA	0.486bA	0.984	996bB
TS-AC	0.944bcA	0.537cA	0.989	1894cB
NTS	0.848aA	0.387aA	0.982	316aA
NTS-AC	0.879abB	0.512bcA	0.989	641abA
<i>2019</i>				
TF	0.889bcA	0.776dA	0.994	462abA
TF-AC	0.924cA	0.905eA	0.997	496abA
TS	0.892bcA	0.489aA	0.998	416abA
TS-AC	0.863abA	0.664bB	0.997	872cA
NTS	0.865bA	0.482aB	0.996	330aA
NTS-AC	0.821aA	0.717cB	0.996	625bA
M	*	***		***
Y	*	*		*
M × Y	NS	***		**

The data for n_f , K_d , and hysteresis (H) are mean values. Managements are tillage and flooding irrigation without (TF) and with the application of alperujo compost (TF-AC), conventional tillage and sprinkler irrigation without (TS) and with the application of alperujo compost (TS-AC), and no-tillage (direct seeding) and sprinkler irrigation without (NTS) and with the application of alperujo compost (NTS-AC). ANOVA factors are M: management; Y: year; M × Y: interaction management * year. F-values indicate the significance levels * $p < 0.05$; ** $p < 0.01$; and *** $p < 0.001$, respectively, and NS: not significant. Different letters indicate significant differences ($p < 0.05$) between managements in the same year (lower case letters) and between years within the same management (upper case letters).

This shows that the adsorption capacity of BPS can be predicted well using only the TOC and pH values of soils and is consistent with results reported by López-Piñero et al. [32], who observed that pH and TOC accounted for 93% of the variation in the BPS adsorption capacity, although in their study, the effects of AC were not evaluated.

Similar to adsorption, BPS desorption was significantly ($p < 0.001$) affected by the management conditions (Table 2). Thus, greater hysteresis (H) values (greater irreversibility) were found in the AC-amended conditions than in the unamended conditions, especially under sprinkler irrigation, regardless of the tillage conditions (Table 2). In fact, the H values were 1.24, 1.90, and 2.0 and 1.07, 2.10, and 1.89 times greater in the TF-AC, TS-AC, and NTS-AC conditions than in the corresponding unamended conditions for 2018 and 2019, respectively (Table 2), indicating that more adsorbed BPS could be retained in AC-amended soils under sprinkler irrigation. These results agree with a previous report of higher irreversibility of BPS in amended rice soils [18]. Hence, the decrease in BPS desorption in amended soils could be due to an increase in the number of active sites for BPS adsorption [31]. In particular, in our study, H was significantly and positively ($p < 0.01$) correlated with fulvic acids (FAs) ($r = 0.611$), showing that these humic substances could provide active sites for herbicide adsorption. These results are consistent with those of Fouad et al. [48], who indicated that humic substances provide active adsorption sites for BPS in clay loam soil. Furthermore, our findings are in line with results observed by Alister et al. [49], who indicated that humic substances and pH were the main soil properties governing the adsorption–desorption processes of different herbicides used in rice soils.

3.2. Dissipation Experiments

Figure 3 shows the BPS dissipation curves of the different management conditions, as well as the values of DA recorded throughout the dissipation experiments. In order to calculate the BPS half-lives ($t_{1/2}$), the experimental data were fitted to the first-order kinetics and high correlation coefficients were found ($R^2 \geq 0.809$, Table 3). The BPS $t_{1/2}$ values were significantly ($p < 0.001$) affected by the management conditions (Table 2). Compared to the unamended conditions, the BPS half-life values ranged from 32.3 d

in the TF condition to 60.6 d in the NTS condition in 2018 and from 46.6 d in the TF condition to 86.4 d in the NTS condition in 2019 (Table 2). These values were higher than those obtained by Chirukuri and Atmakuru [27], whose $t_{1/2}$ values for BPS ranged between 5.1 d and 16.2 d, and those of Kalsi and Kaur [50], who reported BPS $t_{1/2}$ values of 4.09–40.96 d, but in line with values reported by Gámiz et al. [30] (21–84 d) and Gómez et al. [18] (27.4–86.5 d). Probable reasons for this variability in BPS dissipation among these studies include differences in the physicochemical properties of the soils and the different conditions of the experiments [30,50]. In this sense, the fast dissipation of BPS was indicated by Reimche et al. [51] in a field study carried out in Southern Brazil. The values of $t_{1/2}$ ranged from 8.9 d to 15.5 d, although these authors only considered rice paddy water without taking soil into account. In fact, our results showed rapid BPS dissipation under anaerobic conditions (TF and TF-AC) compared to aerobic conditions, regardless of the tillage methods (TS, TS-AC, NTS, and NTS-AC). Hence, the $t_{1/2}$ values were higher in the TS and NTS conditions than in the TF condition by factors of 1.84 and 1.88 as well as 1.45 and 1.85 for 2018 and 2019, respectively (Table 3). In accordance with Kalsi and Kaur [50], the higher BPS dissipation under anaerobic conditions may be attributed to a decline in the availability of pores for BPS diffusion after flooding irrigation, which involves more partitioning of BPS in the aqueous phase and thus favors its dissipation. Similar results were found by Gómez et al. [18], who also observed longer persistence of BPS in aerobic incubation conditions.

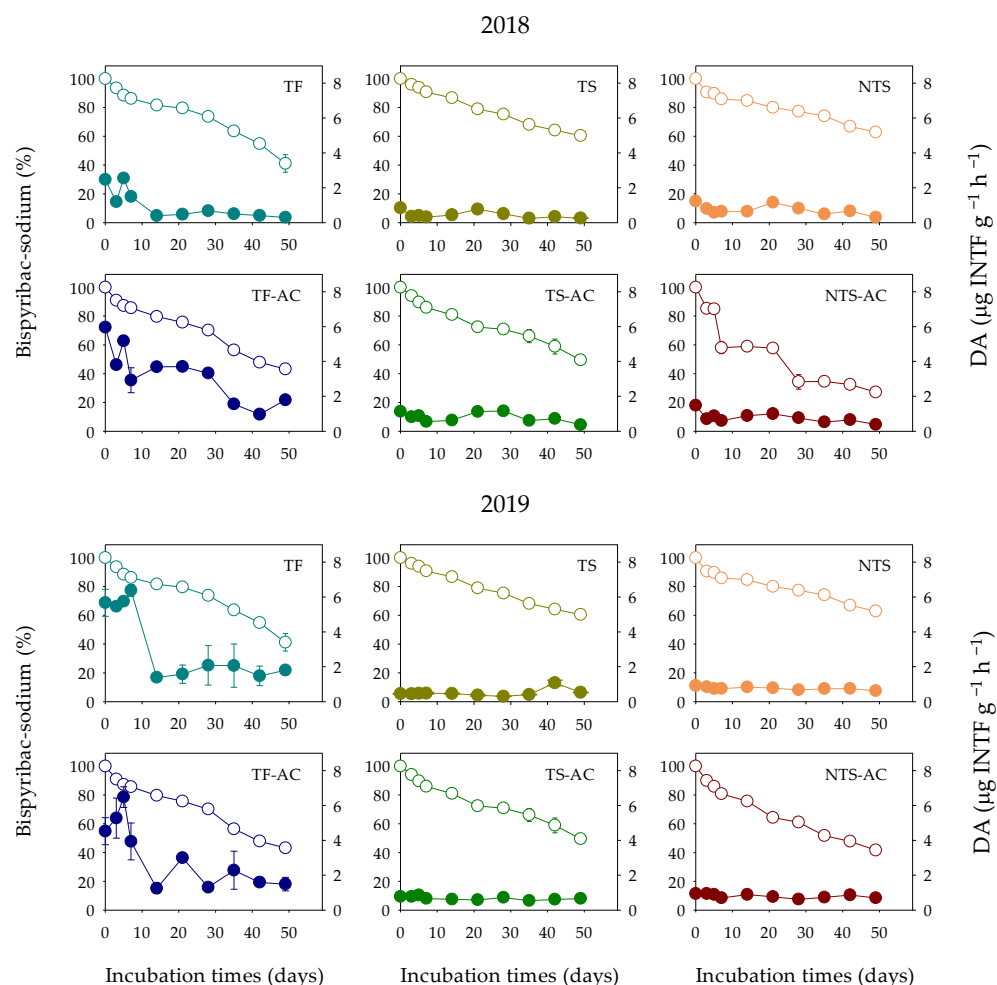


Figure 3. Medium-term effects of different management regimes on Bispyribac-sodium dissipation (○) and dehydrogenase activity (●). Vertical bars representing one standard error of the mean were smaller than the symbols in all cases.

Table 3. Bispyribac-sodium dissipation parameters and dehydrogenase activity .

	$t_{1/2}$ (days)	R^2	DA ($\mu\text{g INTF g}^{-1} \text{ h}^{-1}$)
2018			
TF	32.3bA	0.955	9.77cA
TF-AC	27.5aA	0.902	31.78dA
TS	59.5dA	0.934	5.21aA
TS-AC	38.7cA	0.979	8.58bB
NTS	60.6dA	0.809	8.27bB
NTS-AC	40.4cA	0.949	8.74bA
2019			
TF	46.6aB	0.914	33.65cB
TF-AC	43.2aB	0.955	31.13cA
TS	67.7cB	0.992	4.96aA
TS-AC	55.7bB	0.955	6.81abA
NTS	86.4dB	0.954	7.63bA
NTS-AC	41.3aA	0.986	8.03bA
M	***		***
Y	***		***
M $\times$ Y	***		***

Half-lives: $t_{1/2}$; DA dehydrogenase activity considering all the incubation times in soil conditions. Managements are tillage and flooding irrigation without (TF) and with the application of alperujo compost (TF-AC) and conventional tillage and sprinkler irrigation without (TS) and with the application of alperujo compost (TS-AC); no-tillage (direct seeding) and sprinkler irrigation without (NTS) and with the application of alperujo compost (NTS-AC). ANOVA factors are M: Management; Y: Year; M $\times$ Y: interaction management * year. F-values indicate the significance levels *** $p < 0.001$. Different letters indicate significant differences ($p < 0.05$) between managements in the same year (lower case letters) and between years within the same management (upper case letters).

Regardless of the management conditions, our results showed a clear trend for the effects of AC on BPS dissipation (Table 3). The dissipation rate of BPS increased by factors of 1.17, 1.54, and 1.50 in the TF-AC, TS-AC, and NTS-AC conditions and increased by factors of 1.08, 1.22, and 2.09 compared to the corresponding unamended conditions in 2018 and 2019, respectively. Similar results were observed by García-Jaramillo et al. [52], who found that the dissipation of azimsulfuron, a herbicide that is also widely used in rice cultivation and is a weak acid like BPS, increased with the application of AC but only under flooded conditions. Likewise, Kalsi and Kaur [50] indicated that under field-capacity conditions, the dissipation of BSP increased by 1.86- to 5.96-fold in soils amended with farmyard manure compared to unamended soils. Despite the increase in BPS adsorption capacity in the AC-amended management conditions (Table 2), the highest BPS dissipation in these conditions was probably due to the high TOC and the degree of humification (humic acid, HA). These factors boost biotic degradation and thus hasten the dissipation of herbicides [50]. Indeed, the $t_{1/2}$ values were significantly and negatively ($p < 0.01$) correlated with TOC ($r = -0.433$), HA ($r = -0.485$), and DA ($r = -0.442$). Our results are in agreement with those of Kalsi and Kaur [50], who indicated that high organic matter contents in soils accelerated BPS dissipation due to increased microbial activity. Similar results related to the fast dissipation of different types of herbicides (non-dissociated and dissociated) such as chlorotoluron, flufenacet, and picloram in organic-amended soils were reported by Carpio et al. [53] and Gámiz et al. [33]. However, our results were not consistent with those reported by Gómez et al. [18], who indicated that the persistence of BPS increased with AC application, regardless of the management condition. The variability in the dissipation behavior of BPS in these studies could be attributed to the carbon and/or nitrogen sources of the soil microorganisms. Thus, when a short period of time has elapsed since the AC application (<3 years), soil microorganisms can use the AC as a source of nutrients instead of the herbicide, while in the medium term (4–5 years), soil microorganisms could preferentially use BPS as a source of nutrients. Therefore, the field aging of AC was likely responsible for the shorter persistence of BPS observed in our study. Similar results have been reported [32]

related to faster-dissociated herbicide (such as BPS) dissipation after an aging process for amended soils.

3.3. Leaching Experiments

The cumulative breakthrough curves of BPS for the different management systems are shown in Figure 4. BPS was detected in relatively large amounts in leachates in all management conditions that were studied, where up to 69.1% of the herbicide applied to the columns was recorded. These results are of a similar order to those indicated by Kaur et al. [54], who found values ranging from 62 to 87% for BPS leached in different soils and they are lower than those indicated by Fouad et al. [55], who reported values of about 90% for BPS leached, although these latter authors used soils with $\text{pH} > 8.20$ and higher sand contents. Thus, for the unamended management conditions, the values of the BPS leaching losses ranged from 46.3% in the TF condition to 58.2% in the TS condition in 2018 and from 61.3% in the TF condition to 69.1% in the TS condition in 2019 (Figure 4). Therefore, the medium-term implementation of sprinkler irrigation in rice crops, especially under tillage conditions, promoted BPS leaching losses. These findings could be attributable to the fact that sprinkler irrigation decreased BPS adsorption (Table 2) and enhanced the persistence of the herbicide (Table 3). This behavior showed the importance of the adsorption and dissipation processes to the leaching of herbicides, as indicated in previous studies [5,27,30]. Furthermore, the soil properties could also play a key role in leaching behavior. In fact, in our study, significant negative ($p < 0.05$) correlations were observed between the values of leached BPS and TOC ($r = -0.354$) and HA ($r = -0.388$), confirming that BPS loss by leaching may also depend on organic matter as well as humic substances. These findings could be a reason for the higher leached BPS in the TS conditions compared to the NTS conditions 5 years after their implementation due to the significant increases observed in TOC and HA under no-tillage compared to the tillage conditions (Table 1). Similar results were described by López-Piñeiro et al. [32], who also found a significant negative correlation between BPS leaching and AH in Mediterranean rice fields, although their study was conducted without organic amendment application.

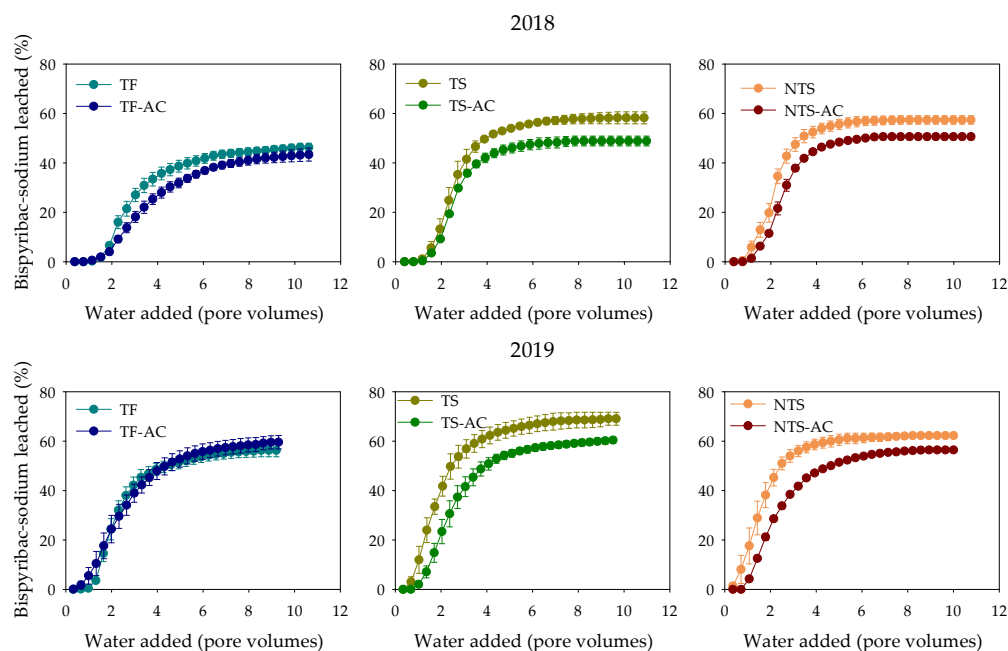


Figure 4. Medium-term effects of different management regimes on cumulative breakthrough curves of bispyribac-sodium. Vertical bars represent one standard error of the mean.

Compared to the unamended management conditions, significant decreases in BPS leaching losses were observed in the AC-amended conditions, regardless of the irrigation

and tillage systems. Hence, the total leached BPS values were 1.13, 1.19, and 1.13 as well as 1.09, 1.14, and 1.10 times lower in the TF-AC, TS-AC, and NTS-AC conditions than in the corresponding unamended conditions for 2018 and 2019, respectively (Figure 4). These results were consistent with those described above for adsorption–desorption, where AC led to increases in the BPS adsorption capacity, as well as greater irreversibility, which may have helped to reduce the mobility of BPS through the soils. Similar findings related to the leaching behavior of BPS were indicated by Singh and Singh [45] for different soil types. Likewise, Kaur et al. [31] concluded that the application of different organic amendments (rice straw and biochar produced from rice straw) could be interesting agricultural management systems used to control the leaching potential of BPS due to its enhanced adsorption capacity in amended soils. Moreover, these results are in agreement with those of Liu et al. [56], who indicated that the small losses of the herbicide Glyamifop due to leaching in rice soils across China may be explained by the organic matter of soils and its high adsorption capacity, although it is an herbicide with very low water solubility. Furthermore, the leaching findings were also consistent with the decreases in BPS persistence observed in AC-amended soils, which limited the leaching of BPS. In fact, a significant negative ($p < 0.01$) correlation was found between the amount of BPS leached and K_d ($r = -0.549$), while a positive correlation with $t_{1/2}$ ($r = 0.763$) was found. Similar results have been reported by other authors [18,30,32] and could be very important to achieving a reduction in the amount of BPS leached, thereby promoting environmentally responsible rice production.

3.4. Bioassays

The herbicidal activity of BPS under different management systems is shown in Figure 5. The weed control efficacy was significantly affected by the management systems. Compared to the unamended management conditions, the lowest values of BPS efficacy were recorded in the TF condition, whose values were about 60% in both years of this study, whereas with sprinkler systems, values of up to 84% were found (Figure 5). These results could be attributable to the adsorption and persistence behavior of BPS. Thus, decreases in the BPS adsorption capacity when sprinkler irrigation is implemented compared to flooding could favor weed control using BPS [5]. In addition, increases in BPS persistence under sprinkler irrigation can enhance the bioefficacy of this herbicide. Indeed, weed control efficacy showed a significant negative correlation with K_d ($r = -0.689$, $p < 0.01$) and a positive correlation with $t_{1/2}$ ($r = 0.377$, $p < 0.05$), indicating that increases in herbicide persistence might lead to greater effectiveness of these compounds. Independent of the water and tillage management conditions, the medium-term effects of AC on BPS efficacy were not significant ($p > 0.05$), except in the TS-AC condition in 2019, when the BPS efficacy increased to 92%. Our results contrast with those of López-Piñeiro et al. [5], who found significant decreases in BPS in amended soils under different irrigation management conditions, although it was only observed for a short time (≤ 1 year) and biochar was used as an organic amendment. In this sense, many studies have demonstrated reductions in the efficacy of herbicides in biochar-amended soils [29,57]. In fact, Yavari et al. [58] indicated that the weed control of imidazolinones decreased in biochar-amended rice soils. In contrast to biochar, Peña et al. [59] indicated that the bioefficacy of S-metolachlor, a non-dissociated herbicide used for weed control in different crops, was not reduced in different agricultural AC-amended soils, although the organic amendment led to significant increases in herbicide sorption. Therefore, at least in the medium term, five years after the transition from flooding, sprinkler irrigation in combination with AC application could be considered an interesting strategy to improve weed control using BPS as well as promote sustainable rice production, especially in areas characterized by a scarcity of water resources such as the Mediterranean area.

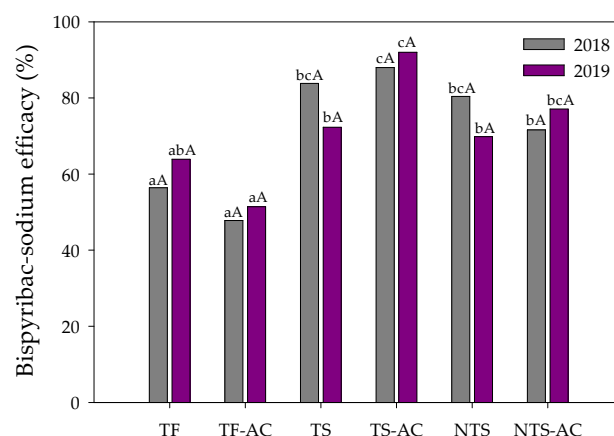


Figure 5. Medium-term effects of different management regimes on bispyribac-sodium efficiency against *Echinochloa crus-galli* L. Beauv. Different letters indicate significant differences ($p < 0.05$) between managements in the same year (lower case letters) and between years within the same management (upper case letters).

4. Conclusions

The environmental fate of BPS was widely affected by the different rice management conditions. After medium-term implementation, the sprinkler irrigation management conditions led to a decline in the BPS adsorption capacity and increases in its persistence compared to the flooding conditions, thereby enhancing the weed control efficiency of BPS. However, the highest BPS leaching losses were observed in the sprinkler management conditions, especially under tillage conditions, and they represent a risk of water contamination. Nevertheless, this threat was counteracted by the AC amendment, despite five years passing since its application, which resulted in a higher herbicide adsorption capacity and greater irreversibility, as well as a lower persistence of BPS. The total organic carbon and humic substances were crucial soil properties that affected the environmental fate of BPS. Therefore, the combined use of AC and water-saving methods such as sprinkler irrigation, especially in a no-tillage system, can be considered a promising alternative to enhance the effectiveness of BPS while reducing its risk of contamination in rice ecosystems, thus enhancing the viability and sustainability of this important crop.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/su16104157/s1>, Texts S1–S3; Figure S1: process of BPS adsorption-desorption experiments; Figure S2: process of BPS dissipation experiments; Figure S3: process of BPS leaching experiments; Figure S4: process of BPS bioassays experiments; Figure S5: bispyribac-sodium adsorption and desorption isotherms. Vertical bars representing one standard error of the mean were smaller than the symbols in all cases. Ce: equilibrium bispyribac-sodium concentration; Cs: amount of bispyribac-sodium adsorbed are presented as Supplementary Materials.

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