

Article

Herbicides Efficacy against Volunteer Oilseed Rape as Influenced by Spray Solution pH

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Abstract: The pH of the spray liquid is one of the factors influencing the efficacy of herbicides. Adjusting the appropriate parameters of the spraying liquid may reduce the consumption of pesticides, which is in line with the currently introduced legal standards and society's requirements. In the greenhouse experiment, the influence of herbicides containing mesotrione, nicosulfuron, rimsulfuron, bromoxynil, and a mixture of nicosulfuron, rimsulfuron, and mesotrione on the efficacy of oilseed rape control was investigated. Oilseed rape (*Brassica napus* L. ssp. *oleifera*), apart from being an important crop, can become a nuisance weed in many fields as form of volunteer plants. Visual assessment, reduction of fresh weight, and chlorophyll fluorescence were performed. Individual herbicides influenced the tested parameters to a different extent. The pH of the spray liquid influenced the efficacy of individual plant protection products. The highest herbicidal efficacy in relation to the tested plants was observed in the case of combinations in which mesotrione was applied at a reduced and increased pH, nicosulfuron applied in an acidic and alkaline environment, rimsulfuron without pH modification, and all treatments in which bromoxynil was applied. The herbicides containing mesotrione and bromoxynil had the greatest impact on the photosystem II activity.

Keywords: herbicides; mesotrione; nicosulfuron; rimsulfuron; bromoxynil; pH adjuster



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1. Introduction

Many factors influence the efficacy of herbicides. These include weather conditions, the composition and growth stage of weeds, the formulation of a plant protection product, the addition of an adjuvant and the application technique [1]. The pH of the spray liquid is also important. It affects the dissolution of herbicides, which in turn is one of the factors determining the uptake of plant protection products by weeds [2]. However, it must be properly adjusted to the applied herbicide, as inappropriate pH may contribute to too fast hydrolysis of the herbicide [3]. Appropriate pH may prevent the integration of the active ingredient with ions contained in water [4].

Winter rape is a very important plant, the oil of which is used in the food industry, biofuel production and animal nutrition [5]. In the case of many crop rotations, however, volunteer of this plant has become a significant problem [6]. They are a threat in many types of crops [7,8]. Rape seeds are characterized by a long life in soil [9]. Additionally, the leaves of this plant are covered with a thick layer of wax, which makes it difficult to moisten the leaves [10].

In terms of the volume of plant protection products used in the world, most of them (about 60%) are herbicides [11]. The use of these substances makes it possible to protect yields to a large extent and keep farmers' income at an appropriate level [12]. At the same time, however, public concerns related to the use of plant protection products and aspects related to environmental protection mobilize to optimize the use of synthetic products in agriculture [13,14]. Therefore, attention should be paid to the need to achieve the highest

possible efficacy of a given treatment, so as to exclude the need for repeated application of herbicides.

Mesotrione control monocotyledonous and dicotyledonous weeds in maize (*Zea mays* L.) [15]. This substance is classified as a triketone, it inhibits 4-hydroxyphenylpyruvate dioxygenase (HPPD). The mentioned enzyme is involved in the synthesis of carotenoids [16]. HPPD inhibition contributes to the disruption of the conversion of tyrosine into plastoquinone and tocopherol. The consequence of the action of substances belonging to this group is the destruction of chlorophyll and bleaching of weeds [17]. Bromoxynil controls dicotyledonous weeds [18]. It is registered in several crops. It belongs to the inhibitors of photosystem II (PSII) [19]. The herbicides belonging to this group bind a protein that should be bound to plastoquinone Qb. This leads to a disturbance in the flow of electrons from plastoquinone Qa to plastoquinone Qb. Consequently, it disrupts the photosynthesis process. Symptoms of the action of PSII inhibitors are chlorosis and necrosis of plants [20]. Nicosulfuron and rimsulfuron are sulfonylurea herbicides. They are intended for post-emergence control of monocotyledonous and selected dicotyledonous weeds in maize cultivation [21]. These substances inhibit the actions of the acetolactate synthase (ALS) enzyme, which is an important element in the synthesis pathway of branched amino acids: leucine, valine and isoleucine [22]. Herbicides belonging to this group initially affect the meristematic tissues, interfering with their growth. Three to four weeks after the application of these herbicides, chlorosis, tissue necrosis, and plant destruction occur [23].

Fast and non-invasive determination of the use of light in photosystem II (PS II) is possible thanks to the measurement of chlorophyll fluorescence [24]. Photosystem II is sensitive to biotic and abiotic stress [25]. Thus, the measurement of fluorescence has become one of the tools for determining plant stress [26,27]. Measurements of chlorophyll fluorescence are used, inter alia, to study plant stress induced by the use of herbicides [28]. The most important parameters determined during chlorophyll fluorescence after darkroom adaptation are F_0 —minimum fluorescence of dark-adapted state, F_v —variable fluorescence, F_m —maximum fluorescence of dark-adapted state, F_v/F_m —maximum quantum yield of PSII photochemistry [29].

The aim of the study was to determine the effect of selected herbicides applied at different pH on the efficacy on oilseed rape and their influence on the plant chlorophyll fluorescence.

2. Materials and Methods

2.1. Materials

The following herbicides were used in the experiment: Kideka 100 SC (mesotrione— $100 \text{ g} \times \text{L}^{-1}$, Nufarm GmbH & Co KG, Linz, Republic of Austria)— $1 \text{ L} \times \text{ha}^{-1}$, Nicogan 040 SC (nicosulfuron— $40 \text{ g} \times \text{L}^{-1}$, Adama Polska Sp. z o. o., Warsaw, Poland)— $1 \text{ L} \times \text{ha}^{-1}$, Rimel 25 SG (rimsulfuron— $250 \text{ g} \times \text{kg}^{-1}$, Innvigo Sp. z o. o., Warsaw, Poland) $60 \text{ g} \times \text{ha}^{-1}$, Arigo 51 WG (mesotrione— $360 \text{ g} \times \text{kg}^{-1}$ + nicosulfuron— $120 \text{ g} \times \text{kg}^{-1}$ + rimsulfuron— $30 \text{ g} \times \text{kg}^{-1}$, DuPont Poland Sp. z o. o., Warsaw, Poland)— $0.33 \text{ kg} \times \text{ha}^{-1}$ + Trend 90 EC (ethoxylated isodecyl alcohol adjuvant, Du Pont, France) at 0.1% v/v concentration, Emblem Pro 385 SC (bromoxynil— $385 \text{ g} \times \text{L}^{-1}$, Nufarm GmbH & Co KG, Linz, Republic of Austria)— $0.65 \text{ L} \times \text{ha}^{-1}$. The spraying liquid was lowered to pH 4 (acid) by adding citric acid ($\text{C}_6\text{H}_8\text{O}_7$, Archem, Lany, Poland) and raised to pH 9 (base) with ammonia solution ($\text{NH}_3 \text{H}_2\text{O}$, TechlandLab, Tarnobrzeg, Poland).

2.2. Methods

Under greenhouse conditions, the influence of selected herbicides applied at variable pH on the efficacy of control of winter oilseed rape (*Brassica napus* L. ssp. *oleifera*) variety Quartz was assessed. Two series of experiments were performed with four replications for each combination in a completely randomized design. Winter oilseed rape seeds were sown in 1 L pots, which were filled with a 1:1 mixture of soil and sand. Soil moisture was determined using the ThetaProbe Soil Moisture Sensor (ThetaProbe, Eijkelkamp, Giesbeek, The Netherlands). Water shortages were replenished with tap water to the level of 65–75%

of soil water capacity. The photoperiod was kept at the level of 16 h day/8 h night. Natural sunlight was supplemented with sodium lamps (HPS) with a capacity of 400 W (Elektro-Valo Oy, Netafim, Avi: 13473, Uusikaupunki, Finland). Air humidity in the greenhouse was maintained at 50–80%. The air temperature was 25 ± 2 °C during the day and 20 ± 2 °C during the night. Seven days after germination, some plants were cut and eight plants were left in each pot.

The treatment was performed when the plants were in the 2–3 true leaf stage with using a laboratory sprayer. The control sample was not sprayed. The output of the spraying liquid was $200 \text{ L} \times \text{ha}^{-1}$ at a pressure of 0.2 MPa. Tee Jet 1102 nozzles (TeeJet Technologies GmbH, Schorndorf, Germany) were used and were positioned 50 cm above the plants.

Chlorophyll fluorescence measurements were performed on the youngest, fully developed leaves for 3 plants from each replicate, which gave 12 results for combination for each series. For this purpose, a Multi-Mode Chlorophyll Fluorometer (OS5p, Opti-Sciences, Inc., Hudson, NY, USA) with PAR Clip was used. The Fv/Fm protocol was selected. Measurements were made 5 and 7 days after the application of herbicides (5 DAA and 7 DAA). Before the measurements, the leaves were dark-adapted for 30 min. White clips to silence photosynthesis were used. Settings fluorometer protocols were selected according to the OS5p User's Guide: Modulation Source: Red, Modulation Intensity: 15, Saturation Flash Intensity: 29, Detector Gain: 05. The setup parameters were set to the appropriate level before the measurement. The modulation intensity and detector gain values were adjusted so that the set fluorescence signal was stable and was in the range of 150–250 counts. The intensity of the saturation flash was also determined to get the saturation point. During the measurement of 7 DAA, the plants of the combinations in which bromoxynil was used were not tested because they were too damaged. 21 days after herbicide application, a visual assessment of herbicide efficacy was performed using a 0–100% scale (0—no damage, 100—complete plant destruction). Visual estimation is one of the methods of assessing the performance of plant protection products [30]. The efficacy of the herbicides was compared to the control in which the plants were not treated. The fresh weight of the plants was also measured. Data were subjected to ANOVA followed by Tuckey's protected LSD test at the 0.05 probability level.

3. Results

3.1. Efficacy of Herbicides Based on Visual Assessment and Fresh Weight Reduction

According to visual assessment and the reduction of fresh weight (Table 1), the individual active ingredients contributed to the control of rape to a different extent. The effect of the pH of the spray liquid on the efficacy of the treatment was dependent on the applied active ingredient. The highest level of rape control was observed in combinations where bromoxynil was applied in a neutral solution and under conditions of lowered pH. The visual assessment and the measurement of fresh weight reduction indicate that rimsulfuron shows the lowest herbicidal efficacy in relation to oilseed rape, both under conditions of lowered and increased pH of the spray liquid, as well as the mixture of nicosulfuron, rimsulfuron and mesotrione applied in a neutral solution.

In the case of combinations where mesotrione was used, the highest level of control of rape for visual evaluation was under the conditions of the lowered pH of the spray liquid. The results of the fresh weight reduction measurement did not show significant differences for the combinations where mesotrione was used in acidic and basic solution. For the experimental objects where the herbicide containing nicosulfuron was applied, the highest level of control of oilseed rape in the visual assessment was found for the combination in which citric acid was added to the spray liquid composition. The measurement of the fresh weight reduction did not show significant differences within the combinations where nicosulfuron was used in acidic and basic solution. Rimsulfuron showed the highest efficacy in the neutral solution. This was confirmed both by the visual assessment and by the fresh weight reduction assessment. The addition of citric acid and ammonia solution contributed to a significant increase in the efficacy of the herbicide containing nicosulfuron,

rimsulfuron and mesotrione, which was confirmed for the visual assessment and the reduction of fresh weight. Bromoxynil showed the highest efficiency in the conditions of neutral and lowered pH. The addition of ammonia solution to the composition of the spray liquid contributed to a significant decrease in the efficacy of the bromoxynil.

Table 1. Impact of spray solution pH on herbicides efficacy against volunteer oilseed rape.

No.	Treatment	Solution for Each Herbicide	pH	Efficacy (%) Based On	
				Visual Assessment	Fresh Weight Reduction
1	untreated	-	-	0	0
2	mesotrione	neutral	6.1	82.5 efg	72.6 de
3	mesotrione	acidic	4.0	87.5 bc	80.3 cd
4	mesotrione	basic	9.0	85 cde	79.1 cd
5	nicosulfuron	neutral	7.2	80 g	84.1 abcd
6	nicosulfuron	acidic	4.0	89.4 b	88.9 abc
7	nicosulfuron	basic	9.0	86.9 bcd	87.5 abc
8	rimsulfuron	neutral	7.5	85.6 cd	83.7 abcd
9	rimsulfuron	acidic	4.0	73.8 h	55.4 f
10	rimsulfuron	basic	9.0	71.3 h	59.6 f
11	mesotrione + nicosulfuron + rimsulfuron + adjuvant	neutral	6.3	71.9 h	64.6 ef
12	mesotrione + nicosulfuron + rimsulfuron + adjuvant	acidic	4.0	84.4 def	82.3 bcd
13	mesotrione + nicosulfuron + rimsulfuron + adjuvant	basic	9.0	81.9 fg	80.9 cd
14	bromoxynil	neutral	7.3	95.6 a	95.4 a
15	bromoxynil	acidic	4.0	96.6 a	94.2 ab
16	bromoxynil	basic	9.0	87.5 bc	83.3 abcd
LSD (0.05)				2.96	12.18

a–h different letters indicate statistically different mean.

3.2. Results of Chlorophyll Fluorescence Measurements

The highest values of minimum fluorescence of objects adapted to darkness were recorded for combinations in which mesotrione was applied (Figure 1). No significant results were found for the combinations in which the substance was applied under different pH conditions. This tendency was observed five DAA and seven DAA. In the case of combinations where nicosulfuron was applied, the highest values of F_0 during both measurements were demonstrated for combinations where the active ingredient was applied in a neutral solution. Five days after treatments, no differences were observed for F_0 within the combinations where nicosulfuron was used. Seven days after the application of the herbicide, the lowest value of the discussed parameter for the herbicide containing nicosulfuron was recorded in the case of the combination in which the herbicide was applied in a neutral solution. For the experimental objects where the herbicide containing nicosulfuron, rimsulfuron and mesotrione was used, an increase in the value of the F_0 parameter was found, for which citric acid or ammonia solution was added to the composition of the spray liquid. In the case of the herbicide containing bromoxynil, a significant increase in the value of this parameter for the measurement performed 5 days after the treatment was recorded for the combination in which the spray liquid with a reduced pH was applied.

The lowest values of maximum fluorescence of dark-adapted state were recorded for combinations with bromoxynil (Figure 2). The addition of citric acid to the composition of the spray liquid containing this active ingredient contributed to a significant decrease in the F_m value. The use of mesotrione also contributed to a significant reduction of the

Fm parameter. In the case of the measurement carried out 5 days after the application of the plant protection product, the lowest Fm value for the objects on which mesotrione was applied was recorded for the combination in which ammonia solution was added to the composition of the spray liquid. The results of the measurement performed 7 days after the application of the herbicide showed no significant differences within the combinations in which mesotrione was used. In the case of the remaining herbicides, a significant decrease in the value of the Fm parameter was noted compared to the control. However, no significant differences were found within these herbicides and the pH variants for each of them.

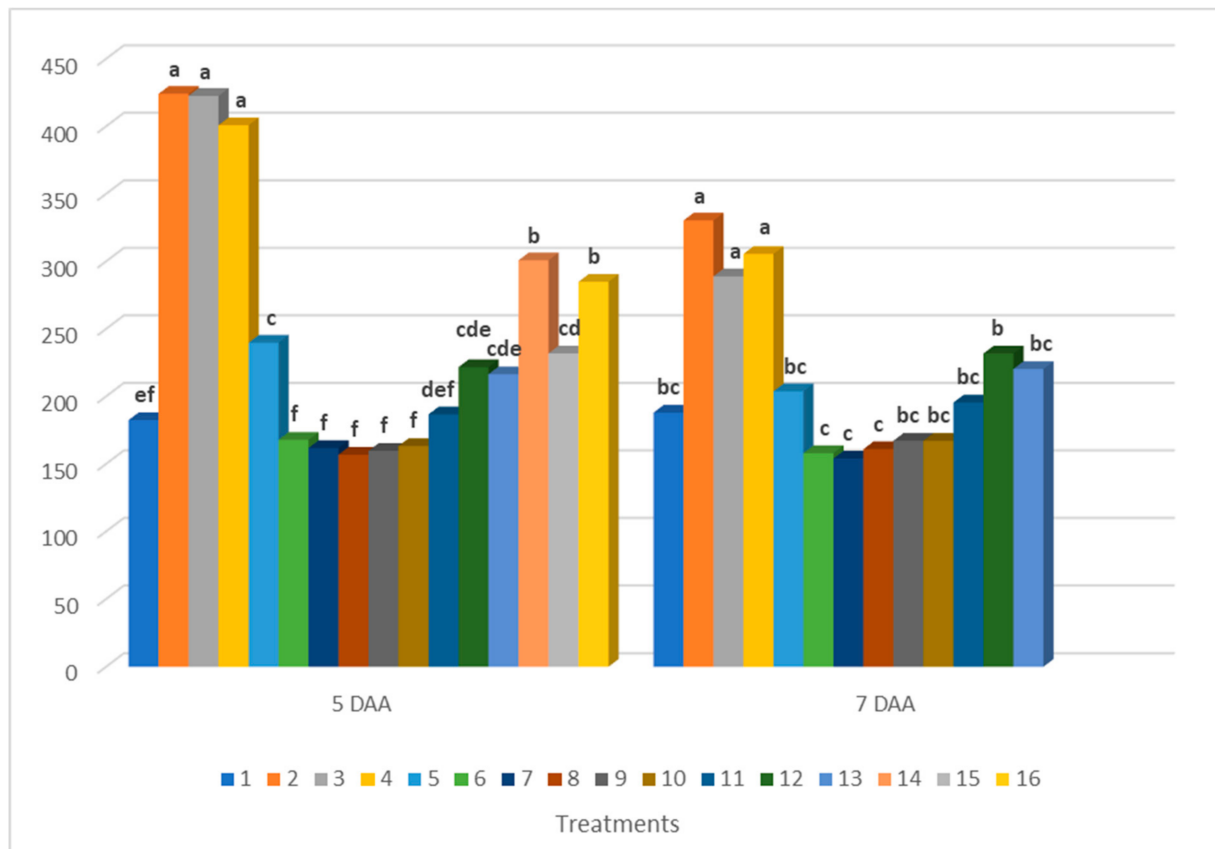


Figure 1. The effect of herbicides on F₀—minimum fluorescence of dark-adapted state (non-nominated units). a–f different letters indicate statistically different mean LSD ($p < 0.05$) 5 DAA = 34.80; LSD ($p < 0.05$) 7 DAA = 44.02. (1—untreated; 2—16 herbicides applied at different pH of the spray solution: 2–4—mesotrione; 5–7—nicosulfuron; 8–10—rimsulfuron; 11–13—mesotrione + nicosulfuron + rimsulfuron + adjuvant; 14–16 bromoxynil; details description of treatments is included in Table 1).

The lowest values of variable fluorescence were found for combinations in which mesotrione and bromoxynil were applied (Figure 3). There were no significant differences within the different spray pH variants for these herbicides. The use of nicosulfuron and rimsulfuron also contributed to a significant decrease in the value of the Fv parameter compared to the control. Within the treatments, no significant differences were found for those substances in which plant protection products were applied at different pH. The change in the pH of the spray liquid also did not contribute to the occurrence of significant differences within the combination, in the case of the five DAA measurements for combinations in which a mixture of nicosulfuron, rimsulfuron and mesotrione was used. The results of the measurement carried out 7 days after the application of the herbicide indicate that the Fv parameter value for the mentioned mixture was decreased to the highest degree for the combination in which citric acid was added to the spray liquid composition.

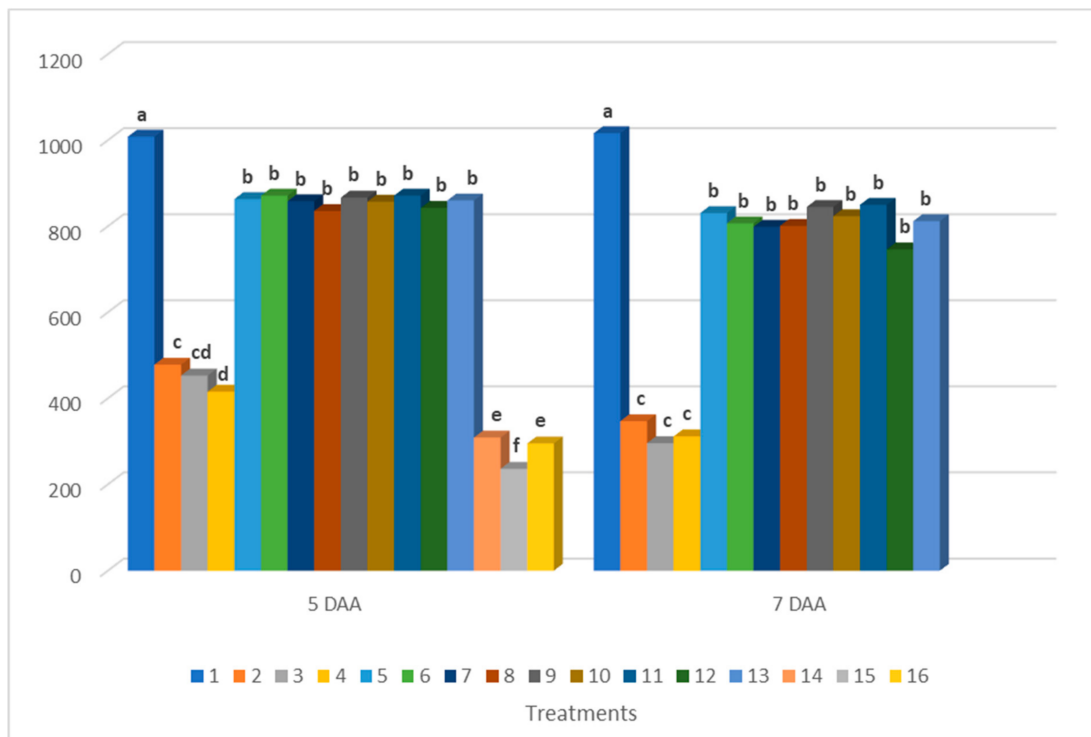


Figure 2. The effect of herbicides on Fm—maximum fluorescence of dark-adapted state (non-nominated units). a–f different letters indicate statistically different mean LSD ($p < 0.05$) 5 DAA = 48.86; LSD ($p < 0.05$) 7 DAA = 67.27. (1—untreated; 2–16 herbicides applied at different pH of the spray solution: 2–4—mesotrione; 5–7—nicosulfuron; 8–10—rimsulfuron; 11–13—mesotrione + nicosulfuron + rimsulfuron + adjuvant; 14–16 bromoxynil; details description of treatments is included in Table 1).

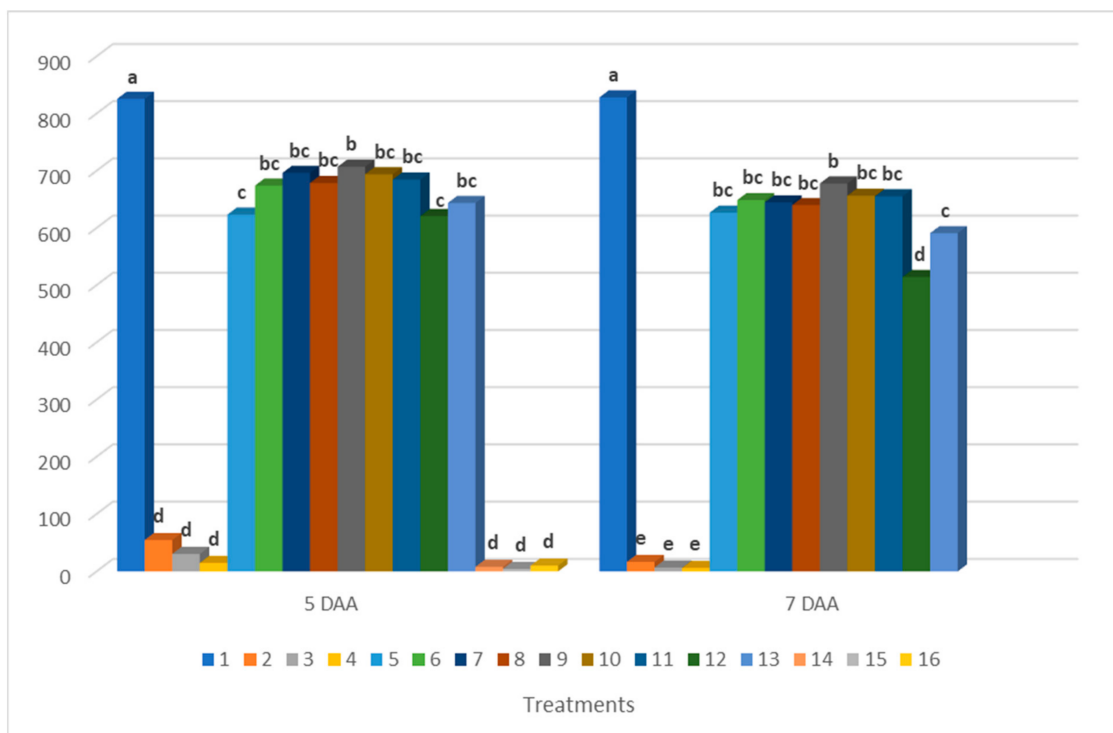


Figure 3. The effect of herbicides on Fv—variable fluorescence (non-nominated units). a–e different letters indicate statistically different mean LSD ($p < 0.05$) 5 DAA = 52.06; LSD ($p < 0.05$) 7 DAA = 50.94. (1—untreated; 2–16 herbicides applied at different pH of the spray solution: 2–4—mesotrione; 5–7—nicosulfuron; 8–10—rimsulfuron; 11–13—mesotrione + nicosulfuron + rimsulfuron + adjuvant; 14–16 bromoxynil; details description of treatments is included in Table 1).

The lowest values of the maximum photochemical efficiency of PSII were found for the combinations in which mesotrione and bromoxynil were used (Figure 4). In the case of the first of the mentioned substances for the 5 DAA measurements, the addition of citric acid or ammonia solution contributed to a significant decrease in the value of the Fv/Fm parameter compared to the neutral solution, which was confirmed. For the measurement carried out 7 days after the application of the herbicide, no significant differences were found within the different pH variants for the spray liquid containing mesotrione. In the case of combinations where nicosulfuron was applied, the highest decrease in the value of this parameter, which was confirmed, was found for the treatment with neutral solution. The use of rimsulfuron did not reduce the value of the Fv/Fm parameter. For the treatments where the herbicide containing nicosulfuron, rimsulfuron and mesotrione was applied, the addition of citric acid or ammonia solution to the spray liquid composition contributed to a significant reduction in the Fv/Fm parameter. No significant differences were found for the discussed parameter within the combinations in which bromoxynil was applied under conditions of a different pH of the spraying liquid.

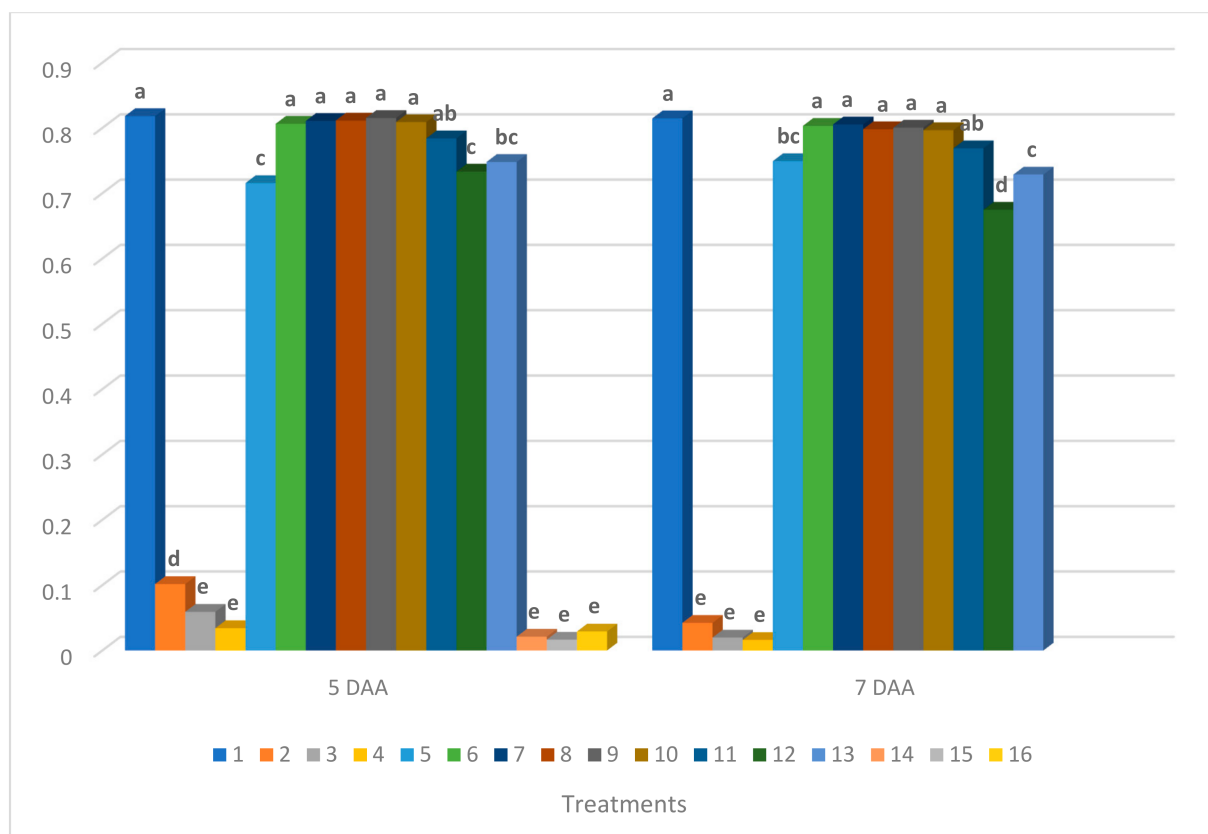


Figure 4. The effect of herbicides on Fv/Fm—maximum photochemical efficiency of PSII (non-nominated units). a–e different letters indicate statistically different mean LSD ($p < 0.05$) 5 DAA = 0.04; LSD ($p < 0.05$) 7 DAA = 0.03. (1—untreated; 2–16 herbicides applied at different pH of the spray solution: 2–4—mesotrione; 5–7—nicosulfuron; 8–10—rimsulfuron; 11–13—mesotrione + nicosulfuron + rimsulfuron + adjuvant; 14–16 bromoxynil; details description of treatments is included in Table 1).

4. Discussion

In the experiment, the highest level of damage of rape (85% and more) was observed in combinations in which mesotrione was applied in an acidic and alkaline solution, nicosulfuron with the addition of citric acid or ammonia solution, rimsulfuron without pH modification and bromoxynil in each of the variants of the pH of spray liquid. The greatest reduction in fresh weight was noted in the case of combinations in which nicosulfuron was applied with the addition of citric acid or ammonia solution and bromoxynil without

pH modification and in an acidic solution. In the studies from other Sulewska et al. [31], the herbicides containing nicosulfuron and rimsulfuron as well as mixture of mesotrione and nicosulfuron showed high efficacy on volunteer oilseed rape in maize. In the case of volunteer oilseed rape, the timing of the treatment is important. The method of control must be adapted to the variety of plants previously cultivated in the field. For self-seeding oilseed rape varieties of Clearfield® (CL) technology, low efficacy is observed for selected herbicides registered for use in cereal cultivation, containing substances from the group of ALS inhibitors [32]. According to Idziak and Woźnica [33], the species in question can also be effectively fought by another substance belonging to the 4-HPPD inhibitors—tembotrione. A high level of control of volunteer oilseed rape was found in the case of the application of bromoxynil in the cultivation of winter wheat [34].

The herbicides used in the experiment showed a different reaction in relation to the modification of the pH of the spraying liquid. Green and Hale have shown that the physicochemical properties of the spray liquid, including its pH, should be matched to a specific active ingredient and to the weed species being controlled [35]. In the case of the mesotrione, the increase in efficacy was observed in the case of both lowering and increasing the pH. Similar result observed Sobiech et al. for other herbicides from the group of 4-HPPD inhibitors [36,37]. In the experiment of other scientists, the influence of the pH of the spraying liquid on the efficacy of the herbicide containing nicosulfuron and the plant protection product containing terbutylazine (PSII inhibitor) and mesotrione was determined. It has been shown that the appropriate selection of the pH of the spraying liquid can significantly improve the efficacy of these herbicides [38]. Matocha and Senseman [39] indicate that in the case of the herbicide from the group of ALS inhibitors, the reduced pH contributes to the acceleration of the hydrolysis of the active ingredient compared to the neutral or alkaline pH of the spray liquid, but it does not have to reduce the efficacy of the herbicide.

The study of chlorophyll fluorescence is already used in experiments in which the efficacy of herbicides and their phytotoxicity to the crop are assessed [40–44]. Research indicates that the mechanism of action of the herbicide plays an important role in the level of its effect on plant chlorophyll fluorescence [45,46]. The discussed measurements are also used in research on the effect of activating adjuvants on the efficacy of herbicides [47,48]. However, there are no results on the effect of herbicides applied at changing pH on the plant chlorophyll fluorescence.

Minimum fluorescence (F_0) takes place when all PSII reaction centers are open, Q_a is maximally oxidized [49]. The exposure of plants to stress increases the value of this parameter [50,51]. In the experiment, the highest values of F_0 were recorded for combinations in which mesotrione was used. In the case of three herbicides, significant differences were found for the variable pH within the applied active ingredients. The exposure of plants to stress contributes to the reduction of the F_m parameter [52]. In the conducted experiment, the highest level of decrease in the value of this parameter was found for combinations in which bromoxynil was used. For most herbicides, the change in pH did not have a significant effect on the change in the F_m value. Lowering the value of the F_v parameter indicates a decrease in the quantum yield of PSII [53]. The highest decrease in the F_v value was found for combinations where mesotrione or bromoxynil was used. The F_v/F_m parameter indicates the maximum quantum efficiency of photosystem II [54]. The F_v/F_m value of healthy plants is approximately 0.78–0.84 [55,56]. Under plant stress conditions, a decrease in the F_v/F_m parameter is observed [57]. In the experiment, mesotrione and bromoxynil contributed to the highest reduction of the F_v/F_m value. There were also significant differences in the amount of F_v/F_m for different reaction values within the selected herbicides.

5. Conclusions

One of the conditions for achieving high efficacy of plant protection products is to adjust the optimal parameters of the spraying liquid. The experiment showed that the application of herbicides at the appropriate pH could significantly affect their efficacy.

The pH of the spray liquid should be adapted to the specific plant protection product. Modifications to the properties of the spray liquid can increase the efficacy of herbicides, which will reduce the amount of chemicals that end up in the environment. Individual herbicides affect chlorophyll fluorescence in different ways. Changing the pH of the spray liquid may affect the functioning of photosystem II. This is an additional indicator of the stress of the test plants and therefore of deterioration of the weed condition.

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