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Influence of Mineral Fertilizers and Application Methods on Raspberry Composition Cultivated in an Acid Soil

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Abstract

Acid soils are often a limiting factor in the production of most cultivated plants. In practice, the application of inadequate, physiologically acidic fertilizers, urea and NPK, is often encountered, which further worsens the already poor physicochemical properties of such soils. In this study, the influence of different amounts of NPK and urea fertilizers and methods of their application on the chemical properties of a very acidic soil and the accumulation of essential biogenic elements (N, P, K, Ca, Mg, and Al) in raspberry plants (leaves and fruits) was evaluated. The field trial with the raspberry plants was set up on a very acidic soil (pH in KCl 3.6), type Dystric Cambisol, and was monitored for 2 years. The application of NPK and urea mainly increased soil acidity in the second year in all treatments (for 0.10–0.18 pH unit) (except for urea applied in rows). The application of higher amounts of NPK increased the content of available forms of P (for 9.3–30.8 mg/kg) and K (for 57–95 mg/kg) in soil in both years, as well as exchangeable Ca (for 200–510 mg/kg) and Mg in the first year (15–165 mg/kg). The introduction of fertilizers in rows, compared to fertilization of the entire surface, influenced the reduction in mobile Al (especially when applying NPK, from 5.89 to 7.13 mg/100 g), the increase in mineral N and K content in the soil, and the increase in Ca and Mg only when applying urea, i.e., P when applying NPK in rows. In the leaves, the application of fertilizers in rows increased the content of Ca and Mg in the first year and P and K in the second year. In the fruits, the content of all estimated elements was not in correlation with their content in leaves and the fertilizer application, which indicates the influence of other ecological and biological factors on plant nutrition.

Keywords: raspberry; acidic soil; mobile Al; macroelements; NPK fertilizer; urea

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1. Introduction

Very acidic (pH < 4) and heavily compacted soils are not favorable for growing agricultural crops due to negative effects on plant growth and development that can be seen very quickly [1]. Raspberry requires loose, moderately moist and drained soil with a high organic matter, with a pH value of 5.5–6.5 [2]. In Western Serbia, where raspberry cultivation is widespread [3], acid soils of the pseudogley, dystric cambisol, and ranker types are represented to a significant extent [4]. These soils are characterized by limited fertility, such as reduced concentration of macroelements (Ca, Mg, P, and K) and microelements (B, Zn, and Mo), and increased content of mobile Al and available forms of Fe and Mn, which can be restrictive factors in raspberry cultivation [5–7].

In these soils, besides aggravating circumstances due to the natural acidity of the soil, further acidification caused by intensive soil use and by improper fertilization, together with increasing chemical contamination of the soil, can worsen the production conditions for raspberries [8–12]. In practice, improper application of fertilizers and lack of regular control of soil fertility are frequent. The lime fertilizers are rarely applied [11], while inappropriate types or formulations of fertilizers, such as ammonium sulfate and urea [13], are often used, and the applied doses are generalized. Improper fertilization is usually carried out over a long series of years, until plant decay is observed, and raspberry yields are significantly reduced [7].

In acid soils, the growth of plants is limited due to the shortage of biogenic elements P, Ca, Mg, Zn, and Mo and the toxic concentrations of Al, Fe, and Mn [9,14–16]. The biggest problem, however, is Al, which prevents normal root growth and penetration. The branching of the roots and the number of root hairs are reduced, so the absorption of water and nutrients is lower [5]. The negative effect of aluminum is reflected in the reduction in the aerial part of the plant and the dry matter yield [17]. It is considered that the content of mobile Al ions of 6–10 mg/100 g of soil unfavorably affects the growth and development of the majority of crops [18]. Soluble phosphates are prone to binding to Al, causing immobilization of phosphate ions in the soil [19]. Therefore, the positional accessibility of this element is also important for plants, so it is important to introduce phosphorus fertilizers into the zone of the root system [20]. Consequently, the method of fertilizer application, that is, applying it in rows or on the entire surface of the plantation, can significantly affect the nutritional status of the plants and their vitality. In the previous studies, the differential effects of fertilizer application methods on soil properties have not been sufficiently highlighted, which is the object of this study.

Raspberry nutrition in acid soil types is challenging when it comes to obtaining high, stable yields and high-quality crops. Therefore, the aim of this study was to observe the changes in the soil reaction, aluminum mobility, and the content of biogenic elements (N, P, K, Ca, and Mg) in a two-year field experiment with the application of increased doses of fertilization with urea and NPK fertilizers (physiologically acid fertilizers) on an already highly acidic soil (District Cambisol). In particular, the effect of fertilization methods, in rows or the entire planting area, on soil properties was investigated. In addition, the aim was to determine the ratio of elements in the soil–leaf–fruit system, especially of aluminum, which can be a main limiting factor for plant growth.

This research will contribute to the comprehensive assessment of the effects of the current common practice of raspberry fertilization in acid soils and could provide recommendations for the development of sustainable strategies of raspberry cultivation in such soils.

2. Materials and Methods

The field experiment was set up in a raspberry plantation in the fourth year of growing (Wilamet variety) in the village of Prijanovići (43°51′2.00″ N, 20°4′9.39″ E) near Požega city (Serbia). The soil was with a very acidic reaction, of the Dystric Cambisol type (WRB, 2014) [20], and the most important agrochemical parameters were determined (Supplementary Table S1). The climate is a mild continental climate. The average air temperature and the sum of annual precipitation during the experimental period of two years were given in Supplementary Table S2. In relation to the multi-year average of the sum of precipitation (749.4 mL), in the first experimental year, the annual sum of precipitation was significantly lower (542.8 mL), while in the second year, it was slightly higher

(786.0 mL). The average temperature during the experimental period of two years was the same (9.8 °C).

The experiment consisted of five treatments with different fertilization and was arranged in a randomized block design with three replications. The fertilization was performed with NPK fertilizer (15% N, 6.54% P, and 12.45% K) and/or with urea. The fertilizers were applied in two ways: on the soil surface of the whole treatment (P) or in the rows (R) with raspberries, avoiding bushes directly. Fertilization was carried out in both years during autumn.

The treatments were as follows:

T1: 1 t/ha NPK, applied on the entire planting area, standard fertilization practice;
T2: 1 t/ha NPK + 400 kg/ha urea applied on the entire planting area, (urea-P);
T3: 1 t/ha NPK + 400 kg/ha urea applied in rows, (urea-R);
T4: 2 t/ha NPK applied on the entire planting area, (NPK-P);
T5: 2 t/ha NPK applied in rows, (NPK-R).

The soil and plant parameters were followed for two years. Soil sampling was performed at the beginning of June in two subsequent years, from the rows near raspberry bushes, from a depth of up to 30 cm, at each treatment. The leaf sampling was conducted at the same time, from the middle of the one-year-old fruiting branches, from several different shrubs. The fruit samples were collected at the beginning of July from several native shoots and different raspberry bushes.

2.1. Soil Samples and Plan Samples Analyses

Soil pH was determined in 1 mol/L KCl (in ratio soil: KCl 1:2.5); hydrolytic acidity (H) and the sum of exchangeable bases (S) were determined by Kappen [21].

The N mineral forms in the soil (NH_4^+-N, NO_3^--N) were determined via the Bremner method [22]; available P and K in soil were determined by using the AL method [23]. Exchangeable soil Ca and Mg were extracted by using the CH_3COONH_4 method and determined with an atomic absorption spectrophotometer.

Exchangeable Al in the soil was determined by the aluminon acetate method as well as in the leaf samples [24]. The total soil N (in soil and plant) was measured with an elemental CNS analyzer, Vario EL III (Hanau, Germany).

In leaf and fruit samples, K, P, Mg, and Ca were determined after being burned to ash, and acid digestion with HCl was performed according to Chapman and Pratt [25]. Phosphorus was measured via the colorimetric ammonium vanadate method, K via flame photometry [23], while Ca and Mg were measured via atomic absorption spectroscopy [25].

2.2. Statistical Analysis

All data were statistically analyzed using analysis of variance (SPSS 22.0), followed by a Duncan's multiple range test ($p < 0.05$), and dependencies between individual soil characteristics and between some soil and plant characteristics were determined using the Pearson correlation coefficient.

3. Results

3.1. Soil Characteristics

Before the experiment was set up, the soil showed an extremely acidic reaction (pH_{KCL}, 3.6) and high hydrolytic acidity (H 14 cmol/100 g) (Supplementary Table S1). The content of adsorbed cations (S) was low (about 3 cmol/100 g), as well as the degree of soil saturation with bases (V). The content of humus, total nitrogen (N), and the C/N value were within optimal limits. The content of available P was very low, 12.0 mg/kg, and the available K was low, 96.2 mg/kg. The content of available Ca and Mg was below the limits of optimal values [26], but the Ca/Mg ratio was favorable (4.35:1).

The results of the chemical properties of the soil in the field experiment, under the influence of the treatment, are shown in Tables 1–3.

Table 1. Soil acidity and soil adsorptive complex.

Treatments	pH KCl		H cmol/kg		S cmol/kg		V %		Al mg/100 g	
	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year
Standard fertilization (1 t/ha NPK)	3.50 bc (±0.01)	3.50 b (±0.02)	14.7 b (±0.36)	14.5 b (±0.30)	3.0 b (±0.26)	3.0 c (±0.00)	20.4 c (±0.53)	17.2 b (±0.40)	10.84 a (±1.62)	9.86 b (±0.37)
400 kg/ha urea-P	3.45 c (±0.01)	3.38 c (±0.02)	16.8 a (±0.62)	14.9 b (±0.26)	2.6 c (±0.15)	2.2 d (±0.17)	13.4 e (±0.36)	13.1 d (±0.56)	9.10 a (±0.44)	11.93 a (±0.67)
400 kg/ha urea-R	3.65 a (±0.05)	3.55 a (±0.02)	13.3 c (±0.46)	13.2 c (±0.62)	4.4 a (±0.06)	5.6 b (±0.23)	24.8 b (±0.55)	30.0 a (±0.53)	9.79 a (±0.78)	7.33 c (±0.98)
2 t/ha NPK-P	3.55 b (±0.03)	3.32 d (±0.01)	14.0 c (±0.50)	16.1 a (±0.30)	3.0 b (±0.10)	3.0 c (±0.17)	18.3 d (±0.70)	15.7 c (±0.70)	9.68 a (±1.28)	11.21 a (±0.96)
2 t/ha NPK-R	3.70 a (±0.03)	3.40 c (±0.02)	9.1 d (±0.38)	14.7 b (±0.44)	4.6 a (±0.06)	5.8 a (±0.21)	27.1 a (±0.56)	29.3 a (±0.62)	2.55 b (±0.18)	5.32 d (±0.24)

Means ± SD (n = 3), values followed by the same letter in a column are not significantly different ($p < 0.05$). H—hydrolytic acidity, S—content of adsorbed cations, V—degree of soil saturation with bases, Al—aluminum.

Table 2. The content of mineral forms of N in the soil affected by treatments.

Treatments	NH_4^+ mg/kg		NO_3^- mg/kg		NH_4^++NO_3^- mg/kg	
	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year
Standard fertilization (1 t/ha NPK)	15.1 d (±1.01)	31.5 e (±1.44)	18.2 e (±1.11)	26.3 a (±1.83)	33.3	57.8
400 kg/ha urea-P	23.0 c (±1.81)	38.5 c (±1.93)	54.3 c (±1.11)	23.5 b (±1.40)	77.3	63.0
400 kg/ha urea-R	59.9 a (±4.37)	63.0 a (±2.11)	88.6 a (±1.91)	22.8 b (±0.70)	148.4	87.5
2 t/ha NPK-P	35.0 b (±1.97)	42.0 b (±1.50)	41.3 d (±2.93)	15.8 c (±1.49)	76.3	57.8
2 t/ha NPK-R	58.1 a (±1.85)	35.5 d (±0.98)	65.1 b (±2.09)	23.5 b (±1.40)	123.2	60.4

Means ± SD (n = 3). Values followed by the same letter in a column are not significantly different ($p < 0.05$).

Table 3. The content of available forms of P, K, Ca, and Mg in the soil affected by treatments.

Treatments	P		K		Ca mg/kg		Mg		Ca/Mg	
	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year	1st Year	2nd Year
Standard fertilization (1 t/ha NPK)	14.1 d (±0.65)	37.0 c (±0.98)	119 e (±3.17)	152 c (±4.88)	890 c (±120.0)	890 c (±70.0)	117 c (±5.20)	90 e (±2.00)	4.6:1	06:01
400 kg/ha urea-P	18.5 c (±0.34)	37.8 c (±1.53)	129 d (±2.71)	146 c (±3.73)	780 c (±70.0)	1800 a (±111.4)	97 d (±3.00)	278 b (±6.80)	4.6:1	3.9:1
400 kg/ha urea-R	14.1 d (±0.90)	28.6 d (±1.53)	150 c (±1.80)	201 b (±5.93)	920 c (±72.0)	2010 a (±115.3)	118 c (±4.60)	292 a (±2.10)	5.2:1	4.2:1
2 t/ha NPK-P	23.8 b (±1.47)	52.8 b (±1.71)	176 b (±1.39)	216 b (±13.02)	1130 a (±36.1)	1400 b (±137.5)	158 a (±8.30)	255 c (±5.60)	4.3:1	3.4:1
2 t/ha NPK-R	25.5 a (±0.91)	67.8 a (±2.22)	214 a (±2.83)	233 a (±13.75)	1090 b (±37.9)	1250 b (±132.3)	132 b (±4.40)	205 d (±5.00)	5.0:1	3.8:1

Means ± SD (n = 3). Values followed by the same letter in a column are not significantly different ($p < 0.05$).

The substitutional acidity of the soil (the pH in KCl) in both years of the experiment, in the soil with standard fertilization practice, was 3.50. In the first year, compared to the standard fertilization, there were significant increases in pH values in the treatments; urea-R and NPK-R, from 0.05 to 0.20 pH units. In the second year, pH values decreased compared to the standard fertilization, and the pH values were significantly lower in all treatments, except u in treatment urea-R. In both years, the pH was significantly higher in the treatments with row fertilization compared to surface fertilization, as well as sum of adsorbed basic cations (S). Values of the sum of base have a high correlation with V% and CEC and a medium correlation with hydrolytic acidity (H). The values of H remain high, from 9.1 to 16.8 cmol/kg, with the application of fertilizer.

In all treatments, the concentration of the ammonia ion (NH_4^+) increased in both years, compared to the standard fertilization, while the nitrate ion (NO_3^-) increased only in the first year. In the first year, the content of NH_4^+ and NO_3^- was significantly higher in treatments with fertilizer application in the rows (urea-R and NPK-R treatments). In the second year, the highest content of NH_4^+ ions remained in treatment urea-R, while the other treatments had more uniform values. In this year, the content of NO_3^- ions was significantly lower and also more homogeneous among treatments. Generally, in the first year, the NO_3^- content was higher than the content of NH_4^+, while in the second year, it was the opposite.

The content of available P and K has a similar trend among treatments. A significant increase in the content of available P and K was observed in both years in treatments with double doses of NPK fertilizers (for 10–31 P mg/kg and 57–95 K mg/kg) compared to the standard fertilization, especially in the treatment with application in the rows compared to the surface. Values of P remain in the class of low soil supply, and values of K are in the class of medium to high soil supplies.

The contents of exchangeable Ca and Mg across treatments are highly correlated in both years (r = 0.943 *, $p < 0.05$ and r = 0.922 *, $p < 0.05$). In the first year, compared to the standard fertilization, a significant increase in Ca and Mg was measured in both treatments with double doses of NPK, especially in the NPK-P treatment. In the second year, Ca and Mg values were generally higher in all treatments, more pronounced in treatments with urea (about 2 and 3 times higher compared to the standard fertilization), especially in the urea-R treatment. Overall, in all treatments, P, K, Ca, and Mg concentrations were higher in the second year compared to the first year (except Ca and Mg in standard fertilization). In addition to numerous agroecological factors and processes in the soil, this increase may also be the result of greater solubility of elements due to increased rainfall in the experimental year.

The content of mobile Al in the soil of the standard fertilization was 10 mg/100 g, which can have a harmful effect on plants [18]. In both years of the experiment, potentially toxic concentrations of mobile Al were observed in all treatments, except in the NPK-R treatment in both years, and urea-R treatment in the second year (for 4.54–8.29 and 2.53 mg/100 g less than in standard fertilization). In treatments with surface application of fertilizers, the content of Al increased slightly in the second year.

3.2. Content of Elements in Raspberry Leaves and Fruits

The N content in the leaves in the first year was within the optimal values [27], with small differences compared to the standard fertilization (N in the standard fertilization was 2.87 and 3.05% in the first and second year), except the urea-R treatment (4.30% N, which is above optimum) (Figure 1). In the second year, the N values were optimal only in the standard fertilization, while, in the other treatments, they were significantly lower. Generally, fertilization in rows influenced the higher values of N in the leaves, except for urea in the second year. The N content in leaves has a medium and high correlation with the NO_3^- ion in soil (r = 0.677, $p > 0.05$ and r = 0.917 *, $p < 0.05$, in the first and second year, respectively).

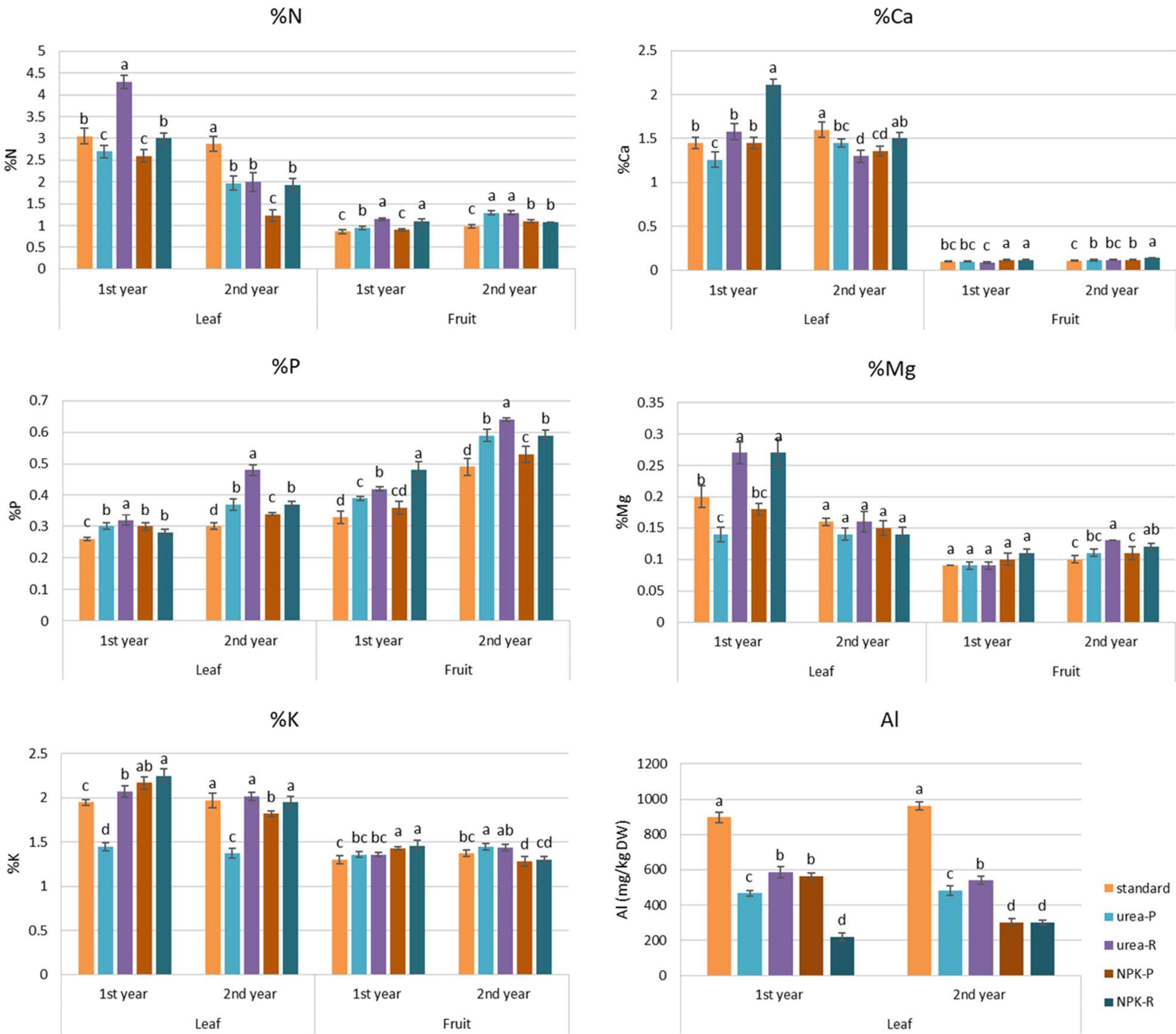

Figure 1. Content of macroelements (N, P, K, Ca, Mg) and Al in raspberry leaves and fruits affected by treatments. Means ± SD (n = 3). Pikes with the same letter are not significantly different ($p < 0.05$).

The N content in raspberry fruits, in all treatments, was lower than the value found previously (1.45%) [27]. The N values in treatments are generally significantly higher compared to the standard fertilization. In the first year, the dynamics of N in the fruit are highly dependent on the content in the soil (r = 0.940 **, $p < 0.01$) and in the leaf (r = 0.725, $p > 0.05$). In the second year, there is only a positive correlation with the concentration of NH_4^--N in the soil (r = 0.694, $p > 0.05$), and the highest N values in the fruit were measured in the treatment with urea.

The P content in raspberry leaves in all treatments, in both years (0.26–0.48%), was below optimal values [27], but significantly higher compared to the standard fertilization. The content of P was the highest in the urea-R treatment, and the values of the other treatments were uniform.

In the fruits, the P content in the first year was below, and in the second year, it was within the previously found values [28]. Compared to the standard fertilization, p-values were significantly higher in all treatments, especially in treatments with fertilization in rows. No correlation was found between the content of P in the soil with P in leaves and fruits, but there was a correlation between P in leaves and fruits in the second year (r = 0.928 *, $p < 0.05$).

The K content in raspberry leaves, in both years (1.37–2.25% K), varied from optimal to above optimal values [27]. The lowest K content in leaves was measured in the urea-

P treatment in both years. The other treatments had a significantly higher K content, compared to standard fertilization, only in the first year. Positive correlations were found in the first year between K in leaves with K in soil ($r = 0.709$, $p > 0.05$; $r = 0.857$ *, $p < 0.05$), Ca ($r = 0.716$, $p > 0.05$), and Mg in leaves ($r = 0.738$, $p > 0.05$; $r = 0.819$, $p > 0.05$). Treatments with the application of fertilizers in the rows mainly showed a significantly higher content of K in the leaves.

In the fruits, the concentration of K mostly corresponded to the known values [28] and ranged from 1.0 to 1.60%K. The content of K in the fruits, in the first year, in both treatments with NPK, was significantly higher compared to others (by 10% and 12%); however, in the second year, the highest content was measured in the treatments with urea (by 5–6% more, compared to the standard fertilization). The K content in fruits in the first year has a high correlation with Ca and Mg in fruit ($r = 0.918$ *, $p < 0.05$ and $r = 0.804$, $p < 0.05$, respectively).

The content of Ca in raspberry leaves (1.26–2.11%) was mostly optimal [27]. In the first year, a significantly higher Ca content was observed when fertilizing in rows (NPK for 31%, urea 22%). In the second year, the highest content of Ca in leaves was observed in the standard fertilization, and differences between other treatments were smaller. The correlation of the Ca content in leaf and soil was positive in the first year ($r = 0.618$, $p > 0.05$) and negative in the second year ($r = -0.826$, $p > 0.05$), which is similar to fruit ($r = 0.831$, $p > 0.05$ and $r = -0.716$, $p > 0.05$ in the first and second year, respectively).

The measured content of Ca in the fruits is generally somewhat lower than published [28] and ranged from 0.09 to 0.14% Ca. Differences between treatments were relatively small, with the highest values in the NPK-R treatment. There was a correlation between Ca in fruit to soil ($r = 0.784$, $p > 0.05$; $r = 0.704$, $p > 0.05$).

In all treatments, Mg content in raspberry leaves was below the optimum [28]. Dynamics of Mg and Ca in the leaf were similar in the first year ($r = 0.831$, $p > 0.05$), and in the second year, the values of treatment were quite uniform.

The Mg content in the fruits ranged from 0.09 to 0.13%. In contrast to leaves, no significant difference between treatments was found in fruits in the first year. In the second year, a significant increase in Mg was measured in treatments with fertilizer application in rows, compared to surface applications.

The common concentration of Al in plants is about 200 mg/kg, while, in tea plants, they are much higher (2000–5000 mg/kg) [29]. Critical aluminum concentrations in plants vary depending on plant species, solution pH, calcium concentration, and experimental technique [30]. The concentration of Al in the raspberry leaf in the standard fertilization treatment was very high and ranged from 896 to 962 mg/kg of dry matter (Figure 1). In all treatments, compared to the standard fertilization, there was a significant decrease in the Al content in raspberry leaves (by 35–76%). In the first year, the greatest decrease in Al was in the NPK-R variant, followed by urea-P, in accordance with the dynamics of mobile Al in the soil ($r = 0.860$, $p > 0.05$). In the second year, the ratios were similar, except in the NPK-P variant, where there was a significantly lower Al content.

4. Discussion

Inadequate application of mineral fertilizers can have negative effects on the soil, the environment, and human health [31]. The processes of volatilization of NH_3 and denitrification of NO_3^- (release of N_2, N_2O, and NO) pollute the air, leaching of NO_3^- contaminates underground and surface water, and a large amount of active substances can have an effect that imbalances the nutrients in soil, salinization, and acidification [32,33]. It is considered that the soil acidity increases with increasing inputs of nutrients, such as N, P, and C to the soil [34], especially those containing NH_4^+ and SO_4^{2-} ions [35–39]. Urea and NPK fertilizers are physiologically acidic fertilizers, whose regular application affects the gradual acidification of the soil [40]. By the introduction of NPK fertilizers into the soil, basic biogenic nutrients are directly available to plants, unlike urea. Urea is a fertilizer with a prolonged or delayed effect; it contains amide nitrogen, which is transformed in the soil into ammonia and then nitrate forms, which are available to the plants [41–43]. Since different cropping systems differ in the soil acidification characteristics and rates [44], here, we investigated the influence of different nitrogen fertilizers, rates, and modes of application in a raspberry plantation. The key findings of the research are illustrated in Figure 2.

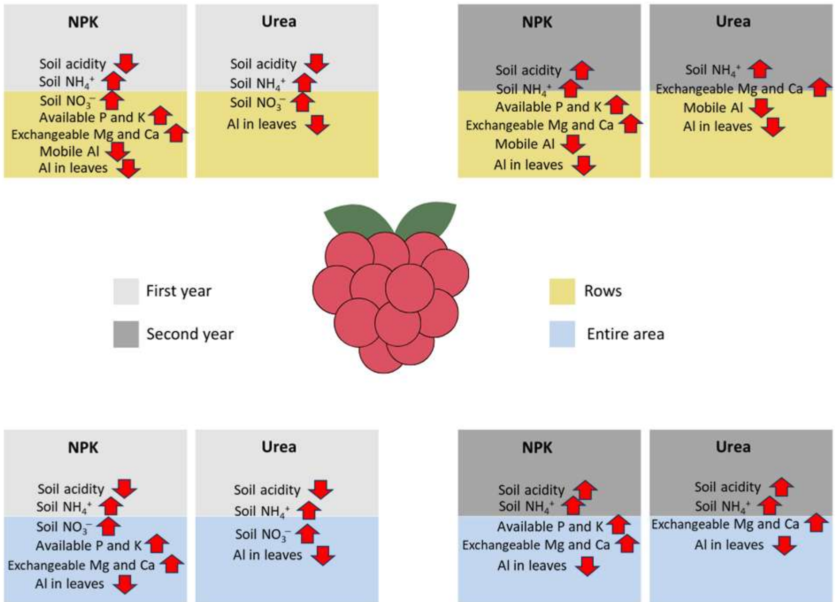

Figure 2. Schematic illustration of the key findings of the research.

In our study, the acidifying effect of NPK and urea can be seen in the second year of their application, where pH was significantly lower in all treatments compared to the pH in standard fertilization (except for urea-R). Further, the application of NPK influenced a stronger acidification effect in soil (due to NH_4^+) than urea. Previously, it was also found that the effect of NH_4^+-N on soil acidification is larger than urea-N, since nitrification of NH_4^+ in NO_3^- results in $2H^+$, while urea transformation results in NO_3^- by hydrolysis and nitrification results in $1H^+$ [45,46]. In addition, the type of fertilizer (NH_4Cl rather than urea) had a higher influence on soil acidification than nitrogen fertilizer rate in the wheat–maize double cropping system [47].

Similarly to our study, the acidifying effect of nitrogen fertilizers was found in previous reports in continuous corn and corn–oat–alfalfa cropping systems [48,49], grassland agroecosystems, and other agricultural crops, such as wheat–maize [35,43,47]. The decrease in soil pH values in the second year varied from 0.10 to 0.18 pH units in the treatments in the raspberry plantation. Previously, the significant decrease in soil pH, from 0.5 to 0.2 units on average over three years, occurred in a grassland agroecosystem under the influence of 360 and 240 kg N/ha, respectively [35]. In a sweet potato–wheat rotation system under different long-term fertilization applying nitrogen fertilizer, the acidification rate reaches 0.121~0.225 units per year, while in a rice and vegetable rotation, long-term rice planting was lower, 0.076 and 0.007 units per year, respectively [44].

In our study, in the first year, in some treatments, the introduction of large amounts of amides, i.e., ammonia ions and potassium ions, into the soil affected the cation exchange reactions on soil colloids and the increase in basic cations in the soil solution, which led to an increase in pH values [25]. In addition, an increase in the sum of basic cations in the soil (S) and the content of NH_4^+ and K^+ was also noticeable, especially in treatments with the application of fertilizers in rows. During the hydrolysis of urea, a hydrolytic basic salt, ammonium carbonate, is formed, which increases the pH value of the soil [43]. These changes in pH were temporary, since, in the process of oxidation of ammonia ions to nitrate, under the influence of nitrifying bacteria, H+ is released into the soil solution, which led to soil acidification.

Our study included a treatment with standard fertilization practice but not an unfertilized control variant, which will be included in future research in order to better assess the absolute effect of fertilization application.

Generally, in our study, changes in the pH value affected the content of mobile Al in soil. The application of fertilizer in rows (especially NPK-R) most effectively reduced the content of mobile Al in the soil, which mainly corresponds to pH values. It was well established that the soil pH is the factor that most significantly influences the Al mobility in soil and bioavailability to plants [15,29]. A very small decrease in the pH value (by 0.1 pH unit) can increase mobile Al twofold in the root zone system [17].

In our study, in all treatments, the Al content in leaves was significantly reduced in both years compared to standard fertilization (up to 76%) but still was very high. The correlation of the Al content in the soil on Al in plant leaves was observed only in the first year. This is in line with a previous report, where the Al reduction in the soil was not proportional to its reduction in the plants [7]. The high concentrations of Al in very acidic soil can place the plant in a state of stress and may be toxic [30,50]. In addition, the Al mainly accumulates in the roots of the plant and can inhibit the root growth within hours [51,52], and in the aerial part of the plant, the Al content can be several times lower [29].

In highly acidic soils, with an increased Al content, phosphorus is bound to poorly soluble compounds (Al- and Fe-phosphates), so its P deficiency usually occurs [53–55]. In our study, by applying a double dose of NPK fertilizer, the content of available P in the soil increased significantly but remained at a low level in all treatments, which possibly caused a low P content in plant leaves. The significant increase in P in leaves and fruits during fertilization was noted in all treatments, but the values were below optimal for plants.

The application of high doses of fertilizers significantly increased the content of NH_4^+-N, compared to the standard fertilization (by 4.0–44.8 mg/kg), and NO_3^+—only in the first year (by 23.1–70.4 mg/kg)—mostly with the application of fertilizers in rows. However, the content of NO_3^- in the second year was similar to and lower than the standard fertilization in all varieties. The dynamics of N forms depend on the transformation processes of N compounds, which are the result of a number of factors: soil pH, temperature, moisture, soluble organic carbon, total N, and CEC [56]. The lower content of nitrates in the soil in the second year may be partly the result of more intense precipitation (by 50% higher compared to the previous year) and their easier leaching from the soil [57], as well as a higher uptake by older plants. In very acidic soil conditions, microbiological conditions also become unfavorable, so nitrification processes are reduced due to the reduced number of nitrifiers [58], and their lower activity can also be influenced by the very high content of ammonia ions [59]. On the other hand, NH_4^+ ion from fertilizers can be more intensively fixed to colloidal particles in the initial stages, and later, the binding is weaker and, under favorable conditions, can become more mobile and available to plants [60].

The soil–raspberry and leaf–fruit N ratios were positive only in the first year. In the second year, the leaf N content was lower, which is consistent with the NO_3^--N content in the soil, while the ratio with fruit N was low, which has been established in other studies [28].

The application of a double dose of NPK fertilizer, due to direct K input, influenced the greatest increase in available K in the soil. The application of urea-R also recorded higher

K values, because significant amounts of NH_2^+ and NH_4^+ ions per unit of soil area more strongly displaced K^+ from the soil adsorption complex into the soil solution [26]. There is direct competition between NH_4^+ and K^+ ions during absorption [61]. At high NH_4^+ concentrations in the root medium, especially in combination with low K^+ availability, plants can suffer from NH_4^+ toxicity [62].

Changes in soil K content had different effects on K accumulation in the plant leaf and fruit, depending on the treatment and the year; K was reduced, increased, or unchanged. It is observed that, in the first year in fruits, K, Ca, and Mg have a high correlation. It was reported [63] that the application of high doses of N-fertilizers can lead to an unbalanced ratio between N, P, and K and disturbances in plant nutrition. Some findings indicate that the levels of macro- and microelements in leaves were generally not correlated with their concentration in the soil [64]. In addition, the content of biogenic elements in plant organs is largely a matter of the genetic characteristics of the plant species and even the variety itself.

Increased doses of NPK fertilizers also significantly increase the content of Ca and Mg in the soil in the first year, and in the second year, urea treatments have a greater effect. In contrast, it was previously noted that application of nitrogen fertilizer increased exchangeable Al and decreased Ca^{2+} и Mg^{2+} [45]. Due to the prolonged action of urea, the concentration of NH_4^+ ions increased, which intensified the exchange reactions with Ca^{2+} and Mg^{2+} ions from the adsorption complex in the soil. Based on the results, it can be concluded that the process of establishing a dynamic equilibrium of elements in the soil is slower, so the final effects of fertilizer application are expected only in the following period. While ammonia ions displace basic cations from the adsorption complex into the soil solution, nitrate ions stimulate the uptake and accumulation of Ca and Mg by the plant [65], as was observed in the first year in leaves. Small and uneven variations in Ca and Mg in fruits indicate, similarly to other elements, that their content is largely genetically determined. According to previous reports [66,67], the content of elements in leaves and fruits is mainly influenced by the genetic characteristics of the plant but also to some extent by mineral nutrition and environmental factors.

5. Conclusions

The study demonstrated that the application of higher amounts of N-fertilizers on very acidic soil influenced mild acidification in the second year. Further, a significant increase in ammonia nitrogen and nitrate nitrogen was found only in the first year. Fertilization in rows, compared to surface fertilization, had an effect on the increase in the macronutrient content in soil, for N and K with both types of fertilizers, for Ca and Mg only with urea, and for P only with NPK. On the other hand, the content of mobile Al was mostly reduced by fertilization in rows. The noted increases in biogenic elements in the soil solution were not always correlated with their content in the leaves and fruits of raspberry.

The introduction of higher doses of nitrogen fertilizers induced pronounced differences in soil properties by year, and further acidification with continuous introduction of fertilizers could be expected in the upcoming period, which should be the subject of further research.

In order to avoid the unwanted consequences of the production of this strategically important fruit crop on acid soil, it is necessary to optimize nitrogen management to ensure a sufficient supply of the plant with this element and to reduce the leaching of nitrates and other losses of N.

The balanced application of mineral fertilizers in combination with the introduction of manure and calcination is an effective way to mitigate soil acidification and achieve adequate nutrition with biogenic elements in systems of intensive raspberry production, which should be the subject of further research.

Supplementary Materials: The following supporting information can be downloaded at https://www.mdpi.com/article/10.3390/horticulturae11080914/s1: Table S1: Soil characteristics at the beginning of the experiment; Table S2: Climatic characteristics in the experimental (Požega) area.

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