



Single and combined effects of pre-veraison treatments with 1-naphthaleneacetic and salicylic acids on harvest date and quality potential of Verdejo wine grapes

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ARTICLE INFO

Keywords:

Auxin
Climate change
Maturation
Must
PGR
Vitis vinifera L.

ABSTRACT

The progressive increase in temperature due to climate change is resulting in a shorter vine growing season, with earlier and unbalanced grape maturation which negatively affects the quality of the wine. One of the possible techniques to mitigate these effects is the application of plant growth regulators capable of delaying ripening, making it more balanced at lower temperatures. In this context, the objective of this study was to evaluate the usefulness of 1-naphthaleneacetic acid (NAA, 100 mg/L) and salicylic acid (SA, 1 and 5 mM), applied to clusters before veraison, to delay ripening and improve the composition of white wine grapes. For these, a two-year field trial was carried out in a Verdejo/110R vineyard. The results showed that NAA treatment delayed the harvest date by around 17 days compared to control producing, for a same soluble solids content, musts with higher total acidity and lower polyphenol and yeast assimilable nitrogen contents. SA single treatment did not significantly affect the harvest date and had a different impact on grape composition depending on the dose applied. The combined application of NAA with 5 mM SA produced a delayed harvest by 12 days compared to the single NAA treatment, without affecting the vigour of the plants, maintained the acidity levels in the must and enhanced its polyphenol content. The study provides novel insights into the use of NAA and SA in Verdejo variety, and demonstrates the interest of combined application of both bioregulators to mitigate the adverse effects of climate change on grape quality potential.

1. Introduction

Climate change is affecting wine grape production around the world. The rising of temperatures alters the phenological development of the vineyards [1,2], causing a shorter and earlier grape ripening [3,4]. These changes result in grapes with high sugar content and pH, low acidity and atypical aromatic profile, which leads to unbalanced wines, with high alcohol concentration, low stability and defects in their sensory attributes [5,6].

The adaptation of vineyards to climate change in the short term could be achieved through management practices that allow for a late ripening, where the grapes are subjected to lower daytime temperatures and a greater day-night thermal jump [7]. Several viticultural techniques, including variations in shoot trimming, pruning time, irrigation management and the use of growth regulators, have been implemented to delay berry ripening [8,9]. To date, this topic has been little explored

in Verdejo, a white variety widely recognized for its oenological potential, especially in Spain. In general, scientific studies examining its physiological and agronomic behavior under different viticultural interventions are scarce.

The accumulation of sugars, aroma compounds and anthocyanins during ripening process occurs through a complex interplay of signals of plant hormones such as abscisic acid (ABA) and ethylene [10,11]. The exogenous application of certain growth regulators, such as auxins and gibberellins, can reduce ABA levels in the fruits [12], then producing a ripening delay [13].

Indole-3-acetic acid (IAA), the most prevalent type of auxins, is present in high concentrations in young berries but its content decreases rapidly before veraison, which is a prerequisite for the start of the maturation process [10]. Exogenous applications of 1-naphthalene acetic acid (NAA), an analogous to IAA, to the clusters at pre-veraison stage, have shown to be effective in delaying the increase in berry

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<https://doi.org/10.1016/j.jafr.2025.102527>

Received 16 October 2024; Received in revised form 13 November 2025; Accepted 15 November 2025

Available online 17 November 2025

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size, sugar and anthocyanin accumulation throughout the ripening in climacteric and non-climacteric fruits [14]. With repeated treatments on wine grape varieties, Böttcher et al. [15] have obtained delays in the harvest date of up to 3 weeks.

Salicylic acid (SA) is a phytohormone that has a wide influence on regulating the growth and development of plants. The endogenous concentration of free SA in fruits is high at the beginning of fruit development and then decreases progressively [16]. SA has been described as a berry ripening inhibitor due to its antagonism with ABA and ethylene [17,18]. Kraeva et al. [19] showed that the injection of a 7.2 mM solution of SA into Shiraz grapes before veraison was effective in delaying ripening by 2–4 weeks. García-Pastor et al. [20] obtained similar results spraying SA on Crimson and Magenta grapes at veraison, and observed that using doses up to 5–10 mM could lead to a significant decrease in vine yield. The ability of SA to reduce ethylene production has also been related to improving the postharvest life and quality parameters of table grapes, such as firmness [21], colour, flavour, astringency and bitterness [22].

On the other hand, it is well known that SA can help ameliorate the growth and development of plants under biotic and abiotic stresses [23], which could have significant effects on the yield and quality of the crops. Some studies have demonstrated that SA plays an important role in providing tolerance against heat stress in grapevine leaves [24,25]. In tomato, Osman et al. [26] showed that the foliar application of 4 mM SA enhanced both the growth of vegetative and reproductive organs under heat stress, resulting in a noticeable decrease in sugar accumulation in the fruits and an increase in the final concentrations of free amino acids.

Along with melatonin, auxin and SA are derived from chorismate in plants and interact between them and with other phytohormones to finely regulate the fruit development (Pérez-Llorca et al. [27]). However, little is currently known about how these interactions intervene at functional and molecular levels, to modulate the synthesis of bioactive compounds during ripening.

Based on the different impact of NAA and SA on fruit ripening, and the role of SA in alleviating abiotic stress, it is possible that the combined application of both plant growth regulators can have synergistic effects to improve the grape quality potential in increasingly warm weather conditions. Until now, there are no studies on this subject.

The objective of this work was to evaluate the additive and interaction effects of NAA and SA pre-veraison applications on harvest date and must composition of Verdejo grapes, and their potential usefulness to improve wine quality in the climate change scenario.

2. Materials and methods

2.1. Experimental design

The investigation was conducted over two consecutive seasons (2022 and 2023), in a commercial Verdejo/110 Richter vineyard located in La Seca (Valladolid, Spain), within the Rueda Designation of Origin (latitude 41°26'58.9"N 4°52'10.9"W; altitude 731 m). The vineyard was planted with a spacing of 3.0 m × 1.5 m (2222 vines/ha). The vines were pruned in double Guyot and trained in a trellis system, with a load of approximately 35,000 buds/ha. Through drip irrigation, the vineyard received an average water supply of around 30 % of the reference evapotranspiration in each season.

Six different experimental treatments were compared in the study, resulting from a factorial design combining two levels of NAA (0 and 100 mg/L) and three levels of SA (0, 135 and 675 mg/L, corresponding to 0, 1 and 5 mM, respectively). A randomized complete block design with three replications was used in the experiment. The elementary plots consisted of six plants, leaving one border plant between each two elementary plots.

Each experimental treatment consisted of two applications: the first at 52 days after full bloom (DAFB) and the second at the onset of veraison. The full bloom dates were June 3, 2022, and May 26, 2023.

The beginning of the veraison was recorded at 62 DAFB in 2022 and 68 DAFB in 2023.

The treatments were applied to the clusters until fully wet on both sides of the trellis, using manual sprayers. The solutions for applications consisted of aqueous mixtures of NAA (Merck KGaA, Darmstadt, Germany) and/or SA (Labbox labware, Barcelona, Spain) and included a non-ionic surfactant: 0.05 % Agral (Syngenta Agro, Madrid, Spain). The control plants were sprayed with water plus Agral.

To facilitate the penetration of the products, a manual light defoliation was carried out in the cluster area of all plants before the first application in each season.

2.2. Meteorological conditions

Meteorological data recorded in the study area during 2022 and 2023 seasons are shown in Table 1. The precipitations in both 2022 (343 mm) and 2023 (376 mm) were higher than the 10-year average in the site (294 mm). Higher mean and maximum temperatures than the 10-year average were recorded from June to August in 2022 and 2023.

In general, 2022 was a warmer and less rainy year than 2023. The number of days with maximum temperatures above 32 °C and 35 °C was 50 and 26 in 2022; and 39 and 12 in 2023, respectively.

2.3. Ripening monitoring and agronomic controls

The evolution of berry weight and total soluble solid content (TSS) in the grape juice throughout ripening was studied by carrying out a random sampling of 48 berries, collected every week from 52 DAFB until harvest. The treatments were harvested successively as each one of them reached an average TSS of 21.5 °Brix for the three blocks of the field trial.

The number of clusters per shoot, berry weight (BW) and total production per plant were controlled at harvest. BW was obtained from a sample of 100 berries randomly collected from each elementary plot. Vigor was estimated as the mean pruning weight.

2.4. Must composition analysis

The musts for analysis were obtained by crushing the sampled berries with a low-pressure blender (Create, Woods & Go Design, Valencia, Spain).

In the samples collected at harvest, TSS, pH, total acidity (TA), malic and tartaric acid concentrations, yeast assimilable nitrogen (YAN), potassium content and total polyphenol index (TPI) were determined. All measurements were carried out following the OIV methods [28].

The CIELab parameters (L^* , a^* , b^* , C^* , H^*) of the musts were recorded with a JASCO V-530 UV/VIS spectrophotometer, using the D65 Illuminant as a reference [28]. The absorbance at 420 nm (A420), as index of must browning, also was determined.

2.5. Statistical analysis

A factorial analysis of variance and least significant difference (LSD) tests were applied to evaluate the effects of the individual and combined treatments applied to the different variables studied. LSD intervals of the analysis were calculated for a 95 % confidence level.

To study the relationships among grape quality variables, a principal component analysis (PCA) was conducted, taking into account data from different experimental treatments recorded in the field trial.

All data analyses were performed with version 9.2 of SAS statistical software (Statistical Analysis System, version 9.2).

3. Results

Table 2 shows the results of ANOVA of data of all parameters considered in the study. A significant variability was observed among

Table 1

Maximum absolute temperature (Tmax abs) and monthly values of maximum (Tmax), mean (Tmean), minimum temperature (Tmin) (°C), and precipitation (P, in mm), registered in the meteorological station of Rueda, Spain (VA103) during 2022 and 2023. Together with the average values of the previous ten years.

Season	Parameters	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
2023	Tmax abs	17.6	18.0	25.8	30.1	29.9	35.5	36.1	40.3	33.0	33.3	18.3	15.1
	Tmax	8.4	11.5	16.7	21.8	22.1	27.3	31.5	32.3	26.0	22.3	13.9	9.6
	Tmean	3.72	4.2	10.1	13.9	15.0	20.3	23.0	23.6	18.5	15.5	9.5	4.7
	Tmin	−0.5	−1.9	3.8	4.1	8.1	14.2	14.0	14.5	12.3	9.8	5.8	0.4
2022	P	24.0	18.0	25.8	30.1	44.1	35.5	0.0	0.0	58.3	63.7	50.0	26.2
	Tmax abs	17.4	17.3	18.0	23.5	33.4	37.4	40.5	37.5	34.4	28.6	18.9	14.4
	Tmax	10.8	13.7	13.0	16.4	25.1	28.5	34.3	32.6	25.3	22.9	14.2	10.6
	Tmean	3.8	6.2	8.3	10.1	17.8	20.9	25.3	24.2	18.0	16.1	9.3	7.2
Average 2011–2021	Tmin	−1.7	−0.3	4.0	4.1	10.3	12.8	15.5	15.8	11.4	10.4	5.0	4.2
	P	7.9	6.0	60.2	39.6	11.8	5.5	0.0	21.2	6.6	61.5	41.0	81.3
	Tmax abs	14.5	17.0	21.8	24.4	29.7	35.5	36.2	36.7	33.1	27.3	19.7	14.7
	Tmax	9.2	11.7	14.3	17.9	23.1	27.4	31.1	31.0	27.0	21.0	13.0	7.8
	Tmean	3.7	5.6	8.2	11.2	15.2	19.4	22.4	22.0	18.3	13.4	7.6	4.1
	Tmin	−0.6	0.0	3.1	4.8	7.9	11.4	13.4	13.2	10.6	6.7	3.1	1.0
	P	27.0	21.3	32.5	43.9	20.7	19.6	6.9	4.1	19.8	34.4	37.3	29.5

Table 2

F-values of factorial analysis of variance of yield, vigour, harvest date and must composition data obtained with 1-naphthalene acetic acid (NAA) and salicylic acid (SA) treatments applied in 2022 and 2023 seasons.

Parameters	Model	Year	NAA	SA	NAA*SA	Block
Harvest date (days after full bloom)	17.40***	2.05	128.30***	1.04	3.29	0.06
Yield and vigour						
Yield	3.10*	0.93	10.96**	2.68	1.52	2.24
Cluster weight	10.90***	80.29**	0.00	0.14	3.19	0.12
100 berry weight	7.36*	16.17***	129.40***	1.60	2.84	2.24
Pruning weight	1.19	0.09	1.85	0.11	0.12	3.56*
Must composition						
pH	7.73***	35.91***	7.85***	2.62	1.78	4.63*
Total acidity	14.19***	14.58***	69.58***	8.35**	5.43*	0.91
Total soluble solids/Acidity	13.76***	14.59***	72.71***	8.27**	2.84	0.41
Tartaric acid	1.59	8.42**	0.40	0.33	1.16	0.45
Malic acid	3.57**	1.23	16.27**	3.00	2.47	0.07
Total polyphenol index	14.24***	67.09***	25.38***	6.13**	4.51*	0.11
Yeast assimilable nitrogen	28.90***	200.41***	4.17*	3.22	9.14***	0.96
Potassium content	3.59**	12.49**	12.96**	0.53	0.86	0.26
Absorbance 420 nm	3.14*	2.55	4.03	3.56*	3.62*	2.07
Colour parameters						
L*	3.16*	4.57*	2.24	3.38*	4.09*	1.78
a*	1.01	0.00	3.97	0.24	0.35	1.45
b*	3.53**	8.27**	6.77*	2.30	1.31	4.20*
H*	2.03	1.03	6.02*	0.51	0.71	3.37*
C*	3.52**	8.04**	7.56*	2.26	0.99	4.17*

F values are significant at * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$.

trial blocks in pruning weight, pH and some colour parameters of the must, such as b*, H* and C*.

Most of the grape composition parameters analysed were highly influenced by the season (Table 2), which would be closely related to the different meteorological conditions of each year. However, there were no significant differences between years in pruning weight (average value of 1.12 kg/plant), yield (6.98 kg/plant) and harvest date (105 DAFB).

The ANOVA revealed that the treatments applied had a significant impact on the studied parameters, except for pruning weight, a* coordinate and concentration of tartaric acid in the must, with different effects depending on the growth regulator used (Table 2). The application of NAA affected harvest date, yield, berry size, pH, TA, TSS/TA ratio, malic acid concentration, TPI, YAN, potassium content and b*, C* and H* coordinates of the must. On the other hand, SA significantly influenced TA, TSS/TA ratio, TPI, L* and A420. Additive effects of NAA and SA on TA and significant interactions NAA x SA in TA and TPI were observed.

3.1. Grape ripening monitoring and harvest date

The evolution of BW throughout the ripening (Fig. 1) reflects that the single application of NAA reduced the berry size versus the control in the first days of the process, but then increased it in the last stage. With this, the application of NAA resulted in a 21 % rise in BW at harvest in 2022 and 29 % in 2023, leading to a rise above 6 % in vine yield in the second season (Table 3).

No significant differences were detected in BW for SA single treatments at 1 mM (SA1) and 5 mM (SA5) versus untreated controls, in any date along the ripening process in the two seasons studied (data not shown). Nonetheless, it was observed that SA sprayings at 5 mM tended to increase BW at harvest when compared to controls (180.3 vs 166.4 g/100 berries), although the differences were not significant at 5 %.

Similar to the NAA treatment, the combination of NAA with SA1 (NSA1) and SA5 (NSA5) resulted in lower BW at the beginning and middle of the ripening curve compared to the control in the two years studied (Fig. 1). Nevertheless, an increase of BW in the last days of grape ripening, which eventually resulted in higher values at harvest, was observed for NSA5 in the two studied years, while in NSA1 was only registered in 2023.

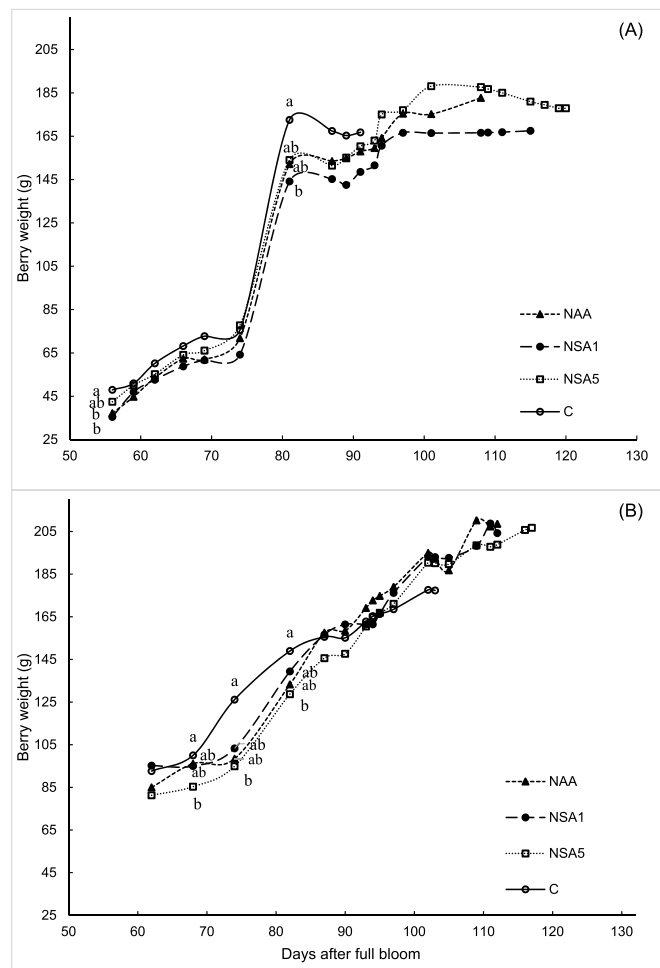


Fig. 1. Evolution of 100-berry weight (BW) in the control, treatments with 100 mg/L of 1-naphthalene acetic acid (NAA), and their combination with salicylic acid at 1 and 5 mM (NSA1 and NSA5) throughout the ripening process in 2022 (A) and 2023 (B) seasons. Means followed by a different letter on each day are significantly different ($p < 0.05$).

Table 3
Main effects of 1-naphthalene acetic acid (NAA) treatment on harvest date, yield, vigour and must composition parameters at harvest in the two seasons studied.

Parameters	2022		2023	
	Control	NAA	Control	NAA
Harvest date (days after full bloom)	93.00 ± 1.7 b	108.00 ± 2.1 a	95.40 ± 6.8 b	111.60 ± 2.2 a
Yield and vigour				
100 berry weight (g)	145.60 ± 38.1 b	175.80 ± 13.9 a	167.10 ± 16.0 b	203.00 ± 9.1 a
Yield (kg/vine)	6.80 ± 1.06 a	7.70 ± 0.80 a	6.45 ± 0.88 b	7.16 ± 0.44 a
Pruning weight (kg/vine)	1.05 ± 0.16	1.16 ± 0.30	1.09 ± 0.14	1.17 ± 0.23
Must composition				
pH	3.47 ± 0.07 b	3.57 ± 0.13 a	3.36 ± 0.08 a	3.39 ± 0.04 a
Total acidity (g/L)	3.43 ± 0.25 b	4.28 ± 0.35 a	3.94 ± 0.53 b	4.75 ± 0.57 a
Total soluble solids/Acidity	6.46 ± 0.50 a	5.09 ± 0.39 b	5.82 ± 0.76 a	4.56 ± 0.59 b
Malic acid (g/L)	1.44 ± 0.41 b	2.39 ± 0.58 a	1.43 ± 0.58 a	1.85 ± 0.69 a
Tartaric acid (g/L)	1.20 ± 0.18 a	1.52 ± 0.40 a	1.84 ± 0.61 a	1.69 ± 0.24 a
Total polyphenol index	8.78 ± 0.24 a	8.36 ± 0.82 b	12.95 ± 0.85 a	9.92 ± 1.50 b
Yeast assimilable nitrogen (mg/L)	147.10 ± 23.10 a	109.80 ± 54.60 b	411.13 ± 18.58 a	363.34 ± 101.84 b
Potassium (g/L)	1.09 ± 0.10 b	1.89 ± 0.24 a	1.18 ± 0.04 a	1.08 ± 0.13 b
Absorbance 420 nm	0.14 ± 0.01 a	0.15 ± 0.03 a	0.15 ± 0.01 a	0.15 ± 0.02 a
Colour parameters				
L*	91.60 ± 1.10 a	90.80 ± 2.00 a	92.20 ± 0.90 a	91.80 ± 1.50 a
a*	−0.29 ± 0.05 b	−0.22 ± 0.07 a	−0.25 ± 0.04 a	−0.25 ± 0.03 a
b*	3.45 ± 0.52 b	4.09 ± 0.90 a	4.84 ± 0.39 a	4.97 ± 0.56 a
H* (°)	94.66 ± 1.80 a	93.32 ± 1.37 b	92.78 ± 0.67 a	93.92 ± 0.83 a
C*	3.46 ± 0.51 b	4.09 ± 0.90 a	4.75 ± 0.41 a	4.97 ± 0.56 a

Within seasons, different letters mean significant differences ($p < 0.05$).

A significantly lower TSS was observed in grapes treated with NAA compared to the controls throughout the entire maturation monitoring in both studied years (Fig. 2). According to the first increase in sugar concentration observed in the curves, the onset of veraison was delayed by 8–12 days in NAA-treated grapes. Nevertheless, the harvest date of NAA treatments, defined as the time when the must reached a TSS of 21.5 °Brix, was delayed finally by 16–17 days, in the two years studied (Table 3).

Grapes from SA1 and SA5 recorded similar TSS values to the untreated control throughout the ripening process and had comparable harvest dates in both seasons (data not shown). The combined treatments NSA1 and NSA5 registered a similar delay in the initial TSS jump-increase as the NAA single treatment (Fig. 2). NSA5 prolonged the maturation process mainly in its last stage, delaying the harvest date by 27 days in 2022 and 21 days in 2023, which represented 12 and 4 days more than the NAA single treatment, respectively.

3.2. Main effects of NAA on grape composition

Table 3 shows the main effects of NAA application on the composition and colour parameters of the must at harvest. The use of NAA increased TA values (at a constant TSS) in both years studied, which decreased TSS/TA index. Since the concentration of tartaric acid remained unaltered, the rise in TA in NAA-treated plants would be mainly due to the tendency of malic acid concentration to elevate (Table 3).

The grapes from NAA treatments registered a higher pH and potassium content than controls in 2022 while, in 2023, a decrease in potassium concentration occurred in treated grapes without altering pH. On the other hand, NAA treatment resulted in a reduction in TPI of the must in the two years studied (Table 3).

Regarding colour parameters, musts from plants treated with NAA tended to increase the C*, a* and b*, while H* decreased compared to the controls in 2022. In 2023, the same trend remained but with no significant differences at 5 % (Table 3). The L* coordinate and A420 were not affected by NAA treatment in either of the two years.

As a consequence of NAA treatments, a decrease in YAN of the must was consistent in the two seasons studied (Table 3).

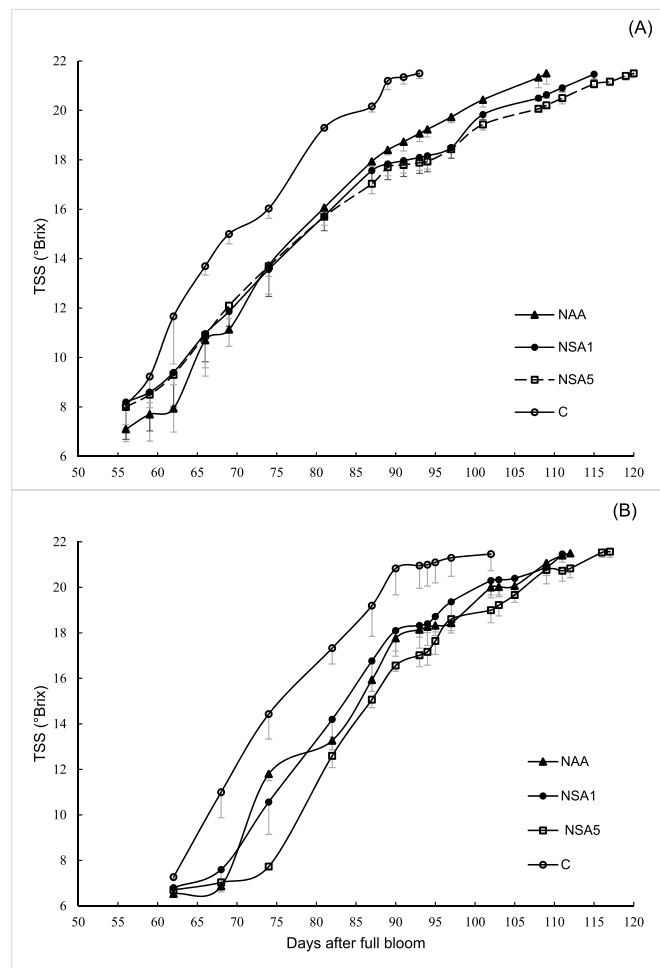


Fig. 2. Evolution of the total soluble solids (TSS) of the must in the control, treatments with 100 mg/L of 1-naphthalene acetic acid (NAA), and their combination with salicylic acid at 1 and 5 mM (NSA1 and NSA5) throughout the ripening process in 2022 (A) and 2023 (B) seasons. Means followed by a different letter on each day are significantly different ($p < 0.05$).

3.3. Main effects of SA and effects of combined treatments on grape composition

Regardless of NAA treatments, the application of SA had different significant effects on TA, pH and TPI values depending on the concentration used (Table 4). In the two years studied, the musts from SA1 exhibited a decline of over 13 % in TA when compared to the controls. As a consequence, the TSS/TA ratio and pH values tended to increase in this treatment, with significant differences in 2022. Nevertheless, SA5 had intermediate TA values that were not significantly different from both control and SA1 in either of the two seasons considered. The concentrations of malic acid in both SA1 and SA5 treatments were lower

than those of controls in 2022. The same trend was maintained in 2023, but without significant differences.

Due to the additive effect between NAA and SA observed in the ANOVA (Table 2), the combined application in NSA1 led to a notable decrease in TA compared to the NAA single treatment in both years (Fig. 3A and B). On the contrary, the values of NSA5 showed a clear trend to increase TA compared to untreated controls, with significant differences in 2023 (3.78 vs 4.46 g/L, $p < 0.05$).

The grapes from SA5 had TPI values comparable to those of the untreated controls in the two years studied, while SA1 had lower values (means represented in Fig. 3). None of the combined treatments exhibited significant variations in TPI when compared to the controls, although NSA5 showed higher values than NSA1 and individual NAA treatment.

The simple applications with SA had little effect on must colour parameters and YAN. However, because of the significant $NAA \times SA$ interaction detected on A420 (Table 2), the combined treatment NSA5 recorded a lower A420 than single NAA treatment (interannual average values of 0.12 vs 0.17 , respectively, $p < 0.05$). Similarly, the $NAA \times SA$ interaction on YAN (Table 2) caused the values of grapes from NSA5 were notably lower than those obtained for the single NAA treatment in

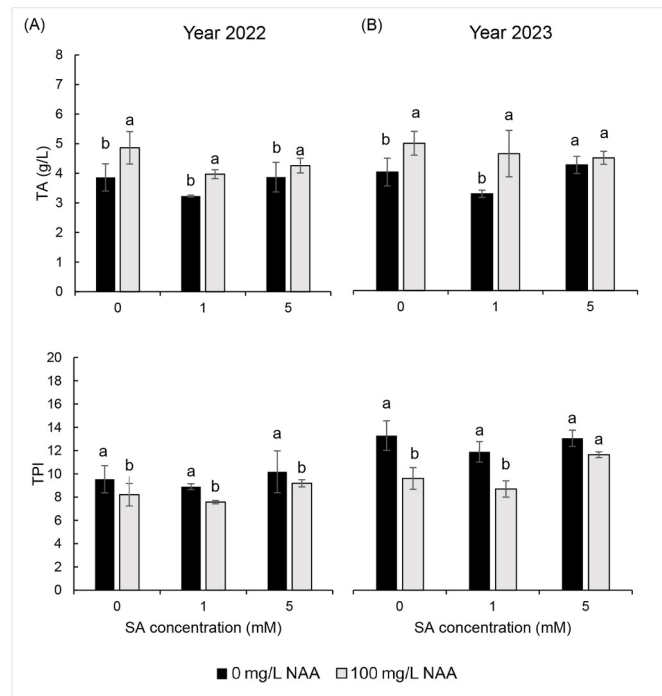


Fig. 3. Mean comparisons of total acidity (TA) and total polyphenol index (TPI) of the must from different experimental treatments, combining 1-naphthalene acetic acid (NAA) and salicylic acid (SA) applications during 2022 (A) and 2023 (B) seasons. For each year and SA dose, means with different letters are significantly different ($p < 0.05$).

Table 4

Significant main effects of treatments with 1 mM (SA1) and 5 mM (SA1 and SA5) of salicylic acid (SA5) on must composition parameters at harvest in the two seasons studied.

Parameters	2022			—	2023		
	Control	SA1	SA5		Control	SA1	SA5
pH	3.51 ± 0.09 b	3.59 ± 0.14 a	3.47 ± 0.06 b		3.38 ± 0.04 a	3.39 ± 0.06 a	3.37 ± 0.09 a
Total acidity (g/L)	4.13 ± 0.59 a	3.59 ± 0.43 b	3.85 ± 0.49 ab		4.48 ± 0.91 a	3.93 ± 0.89 b	4.31 ± 0.21 ab
°Brix/Acidity	5.41 ± 0.80 b	6.16 ± 0.80 a	5.77 ± 0.49 ab		4.94 ± 1.07 b	5.68 ± 1.14 a	5.05 ± 0.30 b
Malic acid (g/L)	2.19 ± 0.79 a	1.72 ± 0.71 b	1.83 ± 0.28 b		1.70 ± 0.44 a	1.50 ± 0.62 a	1.58 ± 0.61 a
Total polyphenol index	8.61 ± 0.47 ab	8.23 ± 0.75 b	8.87 ± 0.31 a		11.48 ± 2.07 b	10.32 ± 1.89 c	12.62 ± 0.86 a

Within seasons, different letters mean significant differences ($p < 0.05$).

2022 (80.6 vs 178.1 mg/L) and 2023 (226.8 vs 400.8 mg/L).

3.4. Principal component analysis

Fig. 4 shows the two first factorial planes of PCA performed with data of grape composition variables affected by experimental treatments (Table 2) from controls, NAA, SA5 and NSA5 base plots of the trial in 2022 and 2023. The total explained variation in each season was 64.7 % and 75.6 %, respectively.

In the graphs in Fig. 4, positive correlations of pH, TA and the concentration of malic acid can be observed with the first principal component, while TPI shows a negative correlation. The relative position of the variables reflects that, in both seasons, TA and concentration of malic acid maintained a positive correlation with BW and a negative correlation with TPI and C*.

Regarding the observations, there is a clearer separation between

plots treated and not treated with NAA (squares vs triangles) than between plots treated and not treated with SA (white vs black figures). In agreement with the results presented above, the NAA treatment shows the highest values of TA and BW (right side of the graphs) while the NSA5 combined treatment (which occupies a more central position) would have, in general, a tendency to decrease BW compared to the NAA single treatment, but with a higher TA and TPI.

4. Discussion

4.1. Grape maturation kinetics and harvest date

In agreement with Böttcher et al. [29], the single application with NAA reduced the grape growth in the initial ripening stage but led to a higher grape size than untreated grapes in the final stage (Fig. 1). A delay in the BW increase during the first ripening stage can be a beneficial factor in producing more resistant grapes, since that avoids the shrinkage that normally might occur later in sensible varieties [30].

The increase in final BW in response to NAA treatment, linked to a greater cell expansion in the grapes [31], produced in our study a significant rise in vine yield. This is a positive outcome for growers that could also contribute to delaying the harvest date.

The detected homogeneity in BW values in plants treated with SA and untreated is not consistent with previous researches conducted on different grape varieties. For instance, Marzouk and Kassem [32] reported BW gains when applying 0.7 mM SA at pea stage and veraison in Thompson Seedless grapes. Alrashdi et al. [33] also found comparable results in El-Bayadi grapes using 4 mM SA in four applications in the growing cycle.

It was observed that the NSA5 treatment produced a more pronounced berry expansion than observed in the NAA single application during the final stages of ripening (Fig. 1), giving larger grapes at harvest. The incorporation of 5 mM SA in combined treatments could enhance the effectiveness of NAA in preventing damages attributable to disorders such as bunch-stem necrosis and berry shrivel [34].

The elevation of auxin levels in grapes generated by NAA treatment could counteract the developmental control exerted by ethylene, thus delaying the beginning and progression of ripening [35]. The detected delay in onset of veraison by 8–12 days in NAA-treated plants, along with the reduction of the speed of sugar accumulation in the grape throughout the entire maturation, moved the harvest date about 17 days later than that of the untreated controls. These results confirm in Verdejo grapes those reported in other wine grape varieties such as Riesling, Shiraz and Cabernet Sauvignon [15,30,36].

The single application of SA, in either of the two doses employed, did not significantly affect the sugar accumulation throughout the ripening process. These results align with the findings of García-Pastor et al. [20] and Gomes et al. [37], who observed minimal changes in TSS when clusters were treated with SA concentrations ranging from 1 to 4 mM at veraison. Instead, other studies have reported that applying concentrations of 0.5–2 mM resulted in a decrease in TSS of the must [22,38]. The application of SA at low concentrations has been found to enhance photosynthetic pigments and total carbohydrates in leaves of tomato and pepper [39,40], as well as facilitate the translocation of sugars from leaves to fruits [41], which may be indicative of accelerated maturation.

Although the SA single treatments did not modify the harvest date in comparison to the controls, the combined treatments of NAA and 5 mM SA recorded the highest delay in sugar accumulation in grapes among all experimental treatments, with harvest dates up to 12 days later than those of NAA single treatment. The synergism detected highlights the potential interest of including SA in combined treatments to enhance the action of auxins on the delay of ripening. SA can inhibit ethylene biosynthesis by suppressing the activity of the aminocyclopropane-1-carboxylate oxidase enzyme [42]. In combined treatments, this effect could allow the concentration of auxins in grapes to be preserved for a longer time and the delay in maturation to be greater.

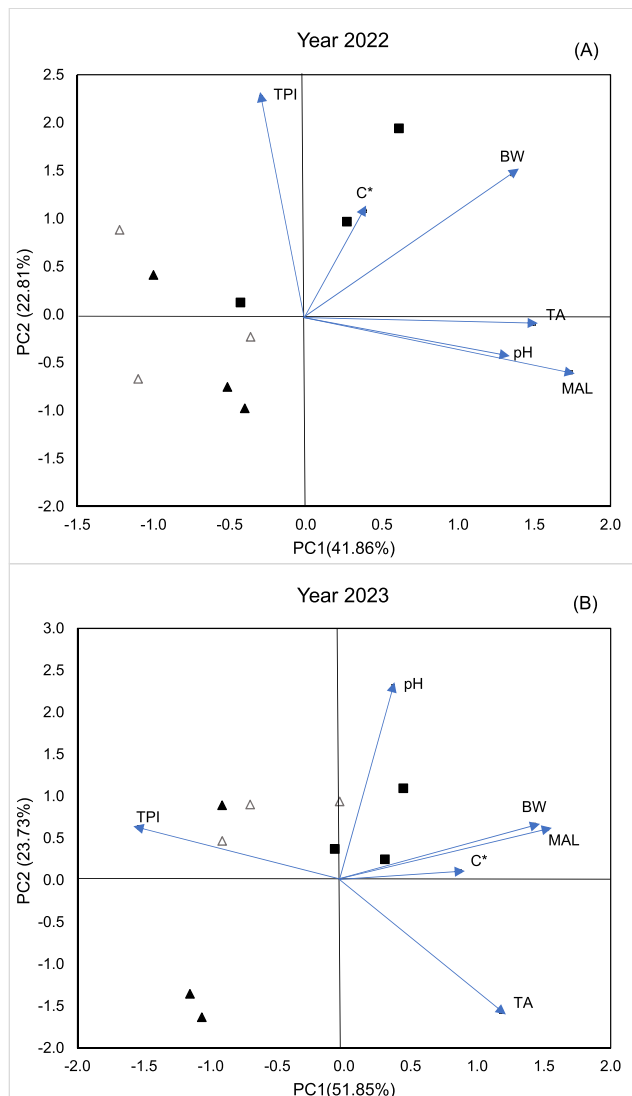


Fig. 4. Principal component analysis of and grape composition data obtained in 2022 (A) and 2023 (B). Variables: BW = berry weight, pH, TA = total acidity of the must, MAL = malic acid concentration, TPI = total polyphenol index and C* = chroma of the must. Observations were classified into four groups considering treated plants with 100 mg/L 1-naphthalenacetic acid versus untreated (squares and triangles, respectively) and, on the other hand, treated plants with 0 and 5 mM salicylic acid and untreated (white and black points, respectively). Data corresponded to variables recorded when the average total soluble solids of the must in each group reached 21.5° Brix.

As discussed below, the observed delay in the harvest date is potentially interesting to mitigate the negative effects of climate change on grapes and wine quality. Moreover, a delayed ripening can facilitate a better organization of harvests, which tend to be concentrated in a shorter period of time as a consequence of global warming.

4.2. Must composition: interannual and interblock variability

The interblock differences detected in field trial (pH, b*, H* and C*), regardless of the effect of the treatments applied, could probably be due to the spatial variation for soil characteristics such as organic matter content, texture, stoniness, or depth explorable by roots.

The mean and maximum temperatures, especially during the ripening process, were higher in 2022 than in 2023 (Table 1), which probably resulted in a decline in TA, TPI and YAN, increasing pH and potassium levels in the must from the first season, compared to the second. Many studies have shown that elevated temperatures during ripening can accelerate malic acid respiration, resulting in a decrease in TA at harvest [43,44]. High temperatures also cause a greater accumulation of potassium within the grapes, which eventually culminates in elevated pH levels in the musts [45,46]. Furthermore, the increase in temperatures can interfere with the biosynthesis pathways of phenolic compounds, leading to reduced accumulation [47]. On the other hand, heat stress can decrease the content of nitrogen and some amino acids in grapes [48,49].

4.3. Must composition: impact of single NAA treatment

The results showed that the NAA treatment was effective in preserving a higher level of acidity in the must, at a fixed TSS. This is a positive factor in mitigating the adverse effects of climate change on grape quality, since acidity is commonly linked to the freshness and aroma of the wine [50]. The increase in TA would be mainly due to a lower degradation of malic acid during ripening. Regardless of the direct effects exerted by the growth regulator, the delay in the onset of veraison observed in the treated plants could have allowed the grapes to be exposed to lower temperatures, resulting in less acidity loss.

Although the increase in TA of must from NAA-treatments was consistent over the two years studied, the pH values were higher than controls in 2022 (Table 3). The impact of NAA treatment on pH might be different depending on the meteorological conditions of the season. The occurrence of higher temperatures in 2022 would lead to a greater accumulation of potassium in treated grapes during ripening process, resulting in a higher pH in the final must [46].

The obtained results on TA and pH align with those of Olego et al. [51] in Tempranillo grapes but disagree with those of Böttcher et al. [15] or Davies et al. [30], who reported no significant variations in TA, malic acid content, and pH between controls and treatments. The impact of NAA treatment on TA might be variable depending on growing conditions, variety, dose and time of application.

The reduction in TPI observed in NAA-treated grapes agrees with previous studies that have reported the application of NAA delays the accumulation of anthocyanins in red grapes throughout the maturation [29,52]. The inverse correlation between BW and TPI found in the PCA (Fig. 4) supports the idea that the reduction in polyphenol content could be due, at least in part, to a larger size of treated grapes which caused a decrease in the skin/pulp ratio.

This TPI decrease in musts from plants treated with NAA can be considered a negative aspect, since the phenolic compounds play a crucial role in white wines by reducing their susceptibility to oxidation and contributing to colour stability [53].

NAA treatment decreased YAN (Table 3), probably as a consequence of the interaction of the auxin on ethylene activity, thereby influencing nitrogen metabolism [54]. The reduction in YAN could be a problem in wine fermentation only if the values are lower than a certain threshold. According to Martínez-Moreno et al. [55], the limit would be around

140–150 mg/L. However, Schreiner et al. [56] indicated that fermentation could be completed even in musts with YAN below 60 mg/L. In the present study, the mean values of YAN were always above these levels.

4.4. Must composition: impact of single SA and combined NAA + SA treatments

As in the present research, the effects of SA treatments on TA and TPI observed in different studies have displayed notable variability depending on variety, concentration of the growth regulator, timing and number of applications used. In general, it has been observed that doses of SA between 1 and 3 mM tend to reduce the acidity of the must, while higher doses maintain or increase it [38,57,58].

The rise in TPI observed in the musts from SA5 treatment would be because the phytohormone acts as an elicitor of phenolics by inhibiting the activity of the polyphenol oxidase enzyme [59,60]. Moreover, SA might regulate key genes or proteins involved in the activation of metabolic pathways for the biosynthesis of phenolic compounds [61]. In fact, different studies have demonstrated that SA exogenous applications can be useful to improve polyphenol contents in grapes [22,37]. The dose used in SA1 was probably not sufficient to produce this effect under the conditions of the field trial.

The increase of TPI (for a constant TSS) achieved with the SA5 treatment could lead to a more balanced grape maturation, contributing to reducing the decoupling between technological and phenolic maturity that typically occurs under high-temperature growing conditions [62].

Our study demonstrates that the NSA5 combined treatment was more effective than single applications of NAA in mitigating the adverse effects of high temperatures on Verdejo grape quality. In addition to delaying the accumulation of sugars throughout maturation, NSA5 maintained the acidity levels to a greater extent, producing musts with a more balanced sugar/acidity ratio. Moreover, this combined treatment tended to compensate for the loss of polyphenols detected in the single application of the auxin, which likely contributed to an interesting decrease in the A420 browning index of the must [63].

As a negative aspect, NSA5 reduced YAN of the must to very low levels, as defined by Martínez-Moreno et al. [55]. This fact should be taken into account in the winemaking process of grapes from combined treatments.

5. Conclusions

Confirming the results obtained by other authors, the pre-veraison treatment of the clusters with 100 mg/L NAA was able to delay harvest date by around 17 days. Our results have demonstrated that this delay could be extended up to 27 days, without modifying the yield and vigour of the vines when applying combined treatments of the auxin with 5 mM SA.

The impact of SA single treatments on the composition of the must was very different depending on the dose used. The 1 mM dose led to a reduction in total acidity, malic acid concentration and TPI, while the 5 mM dose did not modify the acidity and resulted in an increase in polyphenol content of the grapes. In contrast to the NAA single treatment, the combined treatment NSA5 preserved the must acidity and enhanced the polyphenol content of the must, for a fixed TSS. This finding highlights the potential use of NAA and SA combined application as a management tool to alleviate the adverse effects of climate change on grape and wine quality.

CRedit authorship contribution statement

Marie Azuara: Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis. **María Rosa González:** Writing – review & editing, Supervision, Methodology, Investigation,

Funding acquisition, Formal analysis, Conceptualization. **José Manuel Rodríguez-Nogales:** Writing – review & editing, Supervision, Methodology, Investigation, Formal analysis. **Pedro Martín:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

The present work has been carried out within the framework of a collaboration agreement between the University of Valladolid and Bodega Cuatro Rayas S. Coop.

Data availability

Data will be made available on request.

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