

Article

Foliar Sulfur Fertilization at Booting Followed by Potassium Fertilization at Flowering Enhances Grain Quality and Processing Performance in Durum Wheat

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Abstract

Improving durum wheat yield and grain quality is a priority in Mediterranean production systems, where sulfur (S) and potassium (K) deficiencies can limit crop performance. This study evaluates the effects of foliar sulfur (SL) applied at booting (BBCH 45) and foliar potassium (KL) applied at flowering (BBCH 65) on agronomic performance, grain composition, technological quality, free asparagine (ASN) accumulation, grain N relative to fertilizer N input, and economic return in two durum wheat cultivars grown under rainfed Mediterranean conditions. Both SL and KL significantly increased grain yield by 0.47 t ha⁻¹ (+11.5%) and 0.36 t ha⁻¹ (+8.5%), respectively, primarily through increased grain number per unit area, while spike density and thousand-kernel weight were unaffected. SL increased grain protein concentration, glutenin accumulation, the grain N-to-applied N ratio, and dough strength, while reducing free ASN by more than 70%, a response consistent with changes in grain nitrogen partitioning. KL complemented these responses by increasing starch yield, modifying starch composition, and further reducing free ASN. SL significantly increased the net nitrogen-adjusted margin, whereas KL showed a positive but non-significant economic response. Overall, stage-specific foliar S and K applications improved grain yield, grain composition, and technological quality, providing a promising strategy for enhancing durum wheat productivity and quality under Mediterranean rainfed conditions.

Keywords: foliar fertilization; grain quality; free asparagine; net nitrogen-adjusted margin; rheology



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1. Introduction

Wheat is a cornerstone of global food security, constituting a significant part of the daily caloric and protein intake for large segments of the population [1,2]. Among wheat species, durum wheat (*Triticum durum* Desf.) is particularly important in Mediterranean regions, where it underpins the production of staple foods such as pasta, couscous, and semolina-based products. Ensuring both high productivity and grain quality in this crop is therefore essential not only for food availability, but also for nutritional value and processing performance.

In recent decades, agricultural intensification has boosted wheat yields, but often at the expense of grain quality. This is particularly evident under nutrient-depleted or

drought-prone conditions, which are common in rainfed Mediterranean systems [3–5]. Continuous cereal cropping, reduced use of nutrient-balanced fertilizers, and declining atmospheric S deposition have led to widespread deficiencies in key nutrients such as K and S, both of which contribute to optimal physiological performance and product quality [6,7].

K plays multifaceted roles in plant metabolism, including enzyme activation, osmotic regulation, photosynthesis, carbohydrate translocation, and nitrogen (N) assimilation, all of which contribute to crop resilience, grain filling, and yield stability [8,9]. S, though required in smaller quantities, is critical for the biosynthesis of S-containing amino acids (cysteine and methionine) and the formation of gluten proteins that determine dough rheology and end-use quality [6,7]. Moreover, S availability strongly affects N assimilation and utilization, with implications for protein content and grain composition [10,11].

An emerging trait of technological and food safety relevance is free asparagine (ASN). ASN is a nitrogen-rich amino acid that accumulates in cereal grains and serves as a precursor of acrylamide, a potentially harmful compound formed during high-temperature processing [12]. While ASN plays physiological roles in plants, such as N storage, transport, and ammonia detoxification, its accumulation in grains is undesirable from a food safety perspective [13,14]. Both K and S nutrition affect ASN metabolism. K supports asparaginase activity, and its deficiency may lead to ASN buildup [15,16]. S is a key regulator of ASN levels through its impact on enzyme activity and N partitioning, with deficiency promoting ASN synthesis and suppressing degradation [17–19]. Balanced foliar fertilization with K and S may thus improve quality while reducing free ASN accumulation. Previous studies have shown that foliar S supply can substantially reduce free ASN accumulation in wheat [19], but the potential contribution of combined S and K nutrition to this response in durum wheat under field conditions remains insufficiently investigated.

Beyond conventional agronomic and compositional traits, the Pasta-Making Index (IP) is a composite indicator integrating protein concentration and rheological properties into a measure of end-use quality [20]. Technological quality traits may also vary substantially among durum wheat genotypes, potentially affecting their response to late-season nutrient supply. In this context, Creso and Svevo represent contrasting technological quality profiles: Creso is appreciated for its reliable agronomic performance and semolina quality, whereas Svevo is characterized by higher protein concentration and a stronger gluten network [21,22]. Evaluating these contrasting cultivars therefore provides an opportunity to determine whether varietal background modulates the agronomic and technological responses to foliar S and K nutrition. Additionally, the net nitrogen-adjusted margin (N_{NaM}) provides a measure of economic performance in relation to N input [23]. However, the integration of technological quality traits, free ASN concentration, and economic indicators within a single field-based assessment of foliar S and K nutrition in durum wheat remains limited.

Despite the well-documented importance of K and S in wheat nutrition, foliar applications, particularly potassium leaf fertilization (KL) and sulfur leaf fertilization (SL) remain underutilized in durum wheat cultivation, especially under Mediterranean rainfed conditions. Previous studies have examined the effects of foliar S or Se nutrition on grain composition and ASN accumulation in common wheat [19,24,25]. Recent work has also explored the combined application of K and S-related compounds on wheat quality traits [26]. However, the effects of foliar S applied at booting, followed by K application at flowering on yield formation, technological quality traits, and free ASN accumulation in durum wheat under rainfed field conditions remain insufficiently documented. The combined and interactive effects of KL and SL on yield and grain quality traits are still poorly understood, despite their complementary roles in N metabolism, protein synthesis, and assimilate partitioning. Moreover, while the individual effects of foliar K and S

have been documented in controlled settings or in bread wheat, field-based evidence for durum wheat under late-season application is limited, especially with respect to quality and processing traits.

To date, there is also limited information on the economic implications of combined foliar S and K supply when evaluated together with technological quality traits and free ASN responses in durum wheat under field conditions. Likewise, the effects of growth stage-specific foliar SL and KL applications on free ASN accumulation remain poorly documented. Finally, the influence of SL and KL on rheological properties, including dough strength and extensibility, has rarely been evaluated in field-grown durum wheat. Within this framework, field-based assessment using alveograph-based indicators, directly related to pasta-making performance, are particularly scarce.

Given that foliar fertilization offers a targeted, late-season strategy to improve nutrient uptake, grain composition, and stress resilience, thereby overcoming limitations of soil-based delivery [27,28], the present study builds on previous research addressing the role of foliar S nutrition in wheat systems [19,24,25] by investigating the complementary effects of S applied at booting and K applied at flowering in durum wheat under Mediterranean rainfed conditions. To account for potential genotype-specific responses, the two commercial durum wheat cultivars Creso and Svevo were selected to represent contrasting technological quality profiles and different breeding backgrounds within the Italian germplasm. Their inclusion allowed us to assess whether genotype modulates the response to foliar S and K application.

Therefore, this study aimed to evaluate the individual and combined effects of foliar S and K application on grain yield, protein concentration, starch content, gluten strength, free ASN, dough rheology, economic performance and the grain N-to-applied N ratio (N_g/N_s) in two durum wheat cultivars grown under Mediterranean rainfed conditions. By integrating agronomic, compositional, technological, and economic responses, the study aimed to provide a comprehensive field-based assessment of stage-specific foliar S and K nutrition.

2. Materials and Methods

2.1. Field Description and Experimental Design

The study was conducted under rainfed conditions over three consecutive growing seasons (GS1: 2021–2022, GS2: 2022–2023, and GS3: 2023–2024) in Alberese, Grosseto, Italy (Lat. 42.68754; Lon. 11.10845; ~6 m a.s.l.). The site was characterized by a Mediterranean climate with warm to hot, dry summers and mild, wet winters [29]. Monthly rainfall and mean air temperature were recorded throughout the three growing seasons (GS1–GS3), from sowing in October to harvest in June (Figure 1).

Total rainfall during the crop cycle amounted to 421.0, 361.0, and 583.4 mm for GS1, GS2, and GS3, respectively (Table 1). Rainfall in GS1 and GS2 was mainly concentrated during autumn (October–December), whereas GS3 showed a more uniform distribution throughout the growing season, with higher precipitation during winter and late spring. Mean monthly temperatures followed the expected seasonal pattern, ranging from 8.3 to 25.1 °C in GS1, 9.1–23.1 °C in GS2, and 10.0–22.5 °C in GS3, with progressively increasing temperatures from late winter to harvest.

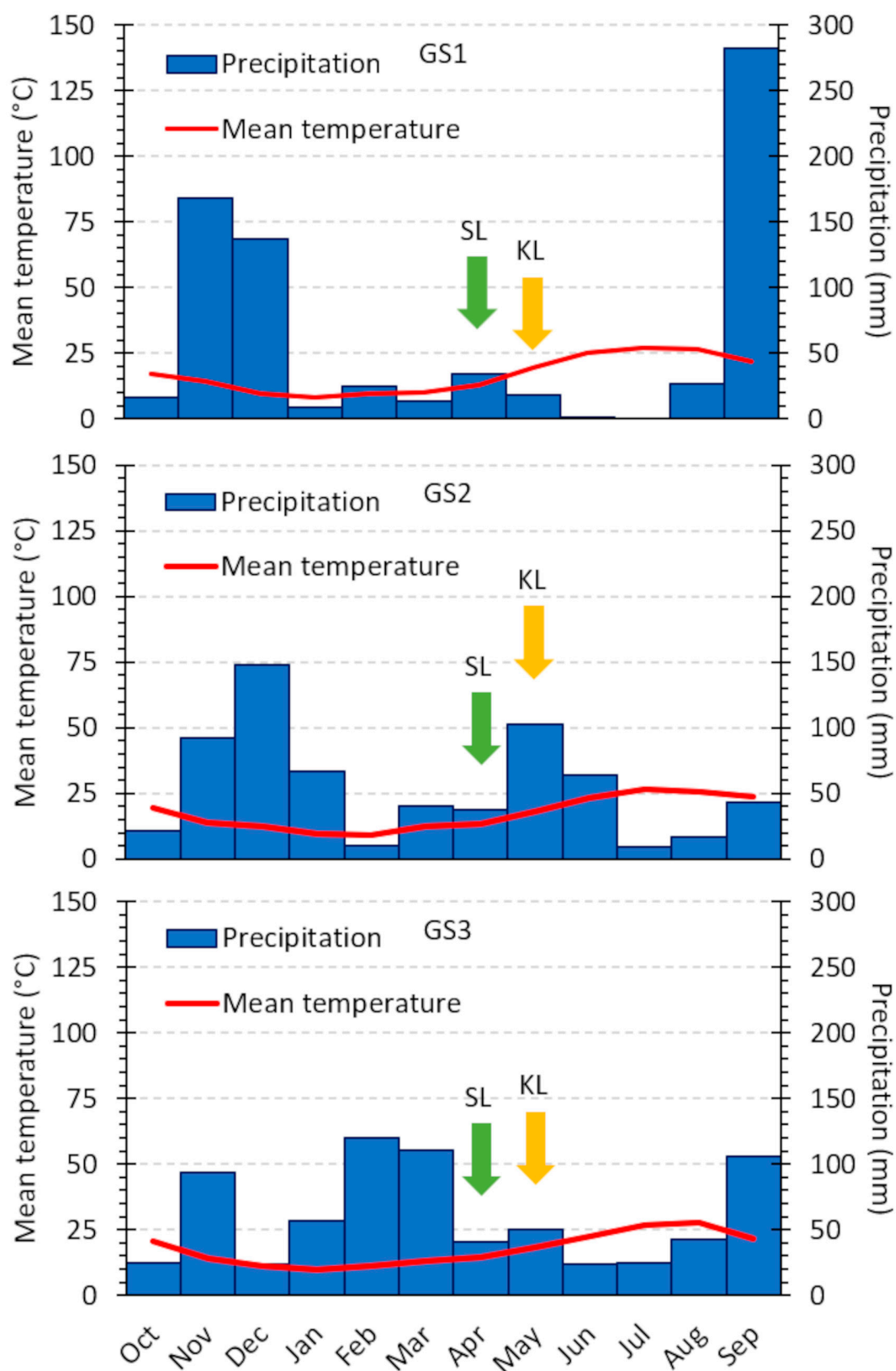


Figure 1. Walter–Lieth climate diagrams showing monthly precipitation and mean air temperature during the three durum wheat growing seasons (GS1–GS3) at the experimental site. Temperature and precipitation are plotted according to the Walter–Lieth scale (1 °C: 2 mm precipitation). Arrows indicate the timing of foliar sulfur application (SL) at booting and foliar potassium application (KL) at flowering.

Table 1. Seasonal rainfall distribution and mean air temperature during the three growing seasons (GS1–GS3), from sowing (October) to harvest (June), for durum wheat.

GS	Total Rainfall (mm)	Autumn Rainfall (Oct–Dec) (mm)	Winter Rainfall (Jan–Mar) (mm)	Spring Rainfall (Apr–Jun) (mm)	Mean Monthly Temperature (°C)	Min Monthly Temperature (°C)	Max Monthly Temperature (°C)
GS1	421.0	321.2	47.0	52.8	14.1	8.3 (Jan)	25.1 (Jun)
GS2	361.0	261.2	47.0	52.8	14.6	9.1 (Feb)	23.1 (Jun)
GS3	583.4	261.2	117.2	205.0	15.1	10.0 (Jan)	22.5 (Jun)

The experimental site had a soil classified as Aquic Haploxeralfs, fine-loamy, mixed, thermic according to the USDA Soil Taxonomy. Soil characterization was carried out on the cultivated topsoil (0–40 cm) at the beginning of each growing season, prior to sowing. The soil texture was classified as a fine sandy loam with no detectable carbonates and a neutral pH (Table 2). The soil showed a low cation exchange capacity (CEC), while electrical conductivity indicated non-saline conditions. According to the FAO classification [30], OC levels were low to borderline medium, Olsen phosphorus (P) was consistently low, and available K fell within the medium range. Based on Kilmer and Neapass (1960) [31], the soil was classified as S-deficient.

Table 2. Selected physicochemical properties of the cultivated topsoil (0–40 cm) at the experimental site in Alberese, Grosseto, Italy.

GS	Sand	Silt	Clay	pH (H ₂ O)	CaCO ₃	OC	Total N	Olsen P	Available K	Available S	CEC	Ca	Mg	Na	K	EC
	%	%	%		%	%	‰	mg kg ^{−1}	mg kg ^{−1}	mg S kg ^{−1}		cmol(+) kg ^{−1}				mS cm ^{−1}
GS1	54	34	12	7.3	0	0.42	0.46	9.7	205	6.2	6.9	3.9	2	0.8	0.5	0.15
GS2	50	36	14	7.2	0	0.38	0.41	7.5	196	6	7.1	4	1.8	0.3	0.48	0.1
GS3	49	34	17	7.3	0	0.57	0.49	6.9	199	6.5	7.6	4	1.9	0.2	0.5	0.12

For each GS and site, the experiment followed a split-plot design within a completely randomized block framework with three replicates. The two durum wheat varieties (Var) were assigned to separate main blocks in the same field and thus considered as independent experimental units across years. Within each Var and year, the factorial combination of SL and KL treatments (2 × 2 levels) was arranged in a split-plot layout (Figure 2). The SL treatment (levels SL_0 and SL_6.4) was allocated to the main plots. Within each SL level, the KL treatment (levels KL_0 and KL_1.17) was randomized as subplots (Figure 2). Each treatment combination was replicated three times for each GS and Var. The fields used for the trials were approximately 10 hectares (ha) in size, and each experimental unit (i.e., each replicate of a treatment combination) occupied one twelfth of the field area (approximately 0.83 ha).

To reflect local agricultural practices and crop rotation cycles, the study was conducted in different fields during each growing season. Faba bean (*Vicia faba* var. minor Beck) was the preceding crop in all GSs.

Two genotypically distinct cultivars of durum wheat (*Triticum durum* Desf.) were evaluated, namely Creso and Svevo. The Creso cultivar, released in 1974, was developed from the progeny of a cross between the short-straw mutant CpB144 of Senatore Cappelli (a traditional Italian durum wheat) and the CIMMYT short-straw line [(Yt54, N10-B) Cp-63] Tc2. The latter is a durum wheat line derived from interspecific crosses between durum and bread wheat (*Triticum aestivum*, L.) genotypes. Creso became one of the most widely cultivated durum wheat varieties in Italy, owing to its high yield potential, short stature, resistance to lodging, and good technological quality for pasta production [21]. However, it is generally considered to have a lower protein content and gluten strength than more

modern cultivars. Svevo, released in 1996, was developed from a cross between the Italian cultivar Zenit and a CIMMYT-derived selection. Svevo is considered a modern, high-performing variety widely adopted in Mediterranean environments, and is renowned for its superior protein concentration and stronger gluten network, rendering it the current reference standard for pasta-making quality in Italy. Notably, Svevo served as the source of the reference genome for *Triticum durum*, which was sequenced and published [22], providing a foundational resource for durum wheat genomics and breeding.

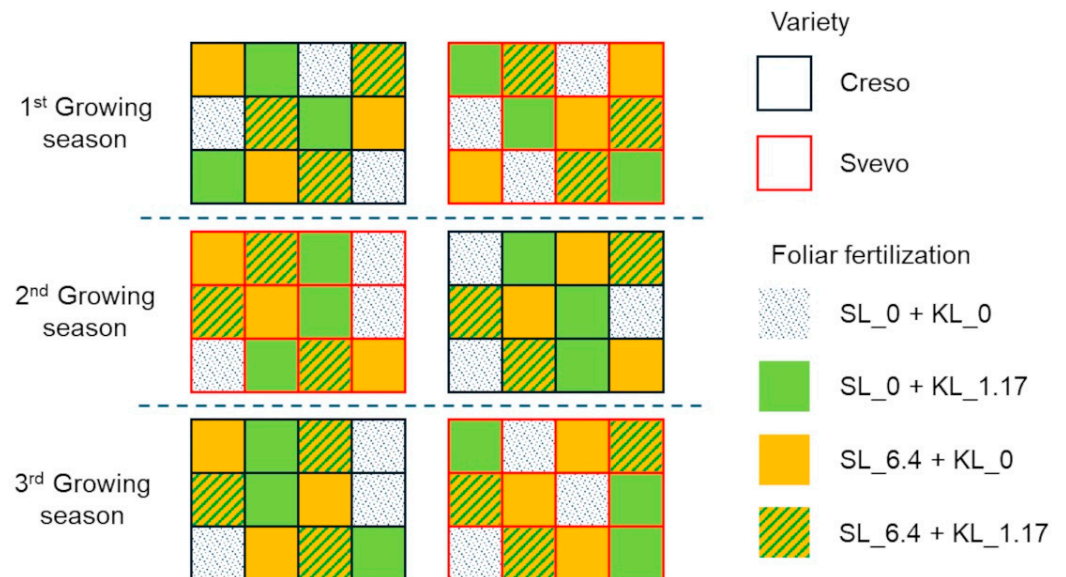


Figure 2. Experimental field design across three consecutive growing seasons. Each square represents a plot, with the colour and pattern indicating the foliar fertilization treatments (SL and KL combinations) and the border colour indicating the durum wheat variety (Creso = black border, Svevo = red border). Fertilization treatments included: SL₀ + KL₀ (dotted), SL₀ + KL_{1.17} (light green), SL_{6.4} + KL₀ (orange), and SL_{6.4} + KL_{1.17} (green-orange striped). Treatments were randomized within each variety block and replicated across years to account for seasonal variability.

Prior to sowing, the soil was moldboard plowed to a depth of 35 cm in early September. This primary tillage was followed by surface refinement using a tandem disk harrow to break up soil aggregates in order to prepare a uniform seedbed. A basal dose of 50 kg ha⁻¹ of P₂O₅, supplied as triple superphosphate (46% P₂O₅), was uniformly broadcast and incorporated into the top 5 cm of soil using a tandem disk harrow.

Both cultivars were cultivated in the following GSs: GS1 (sown on 14 October 2021 and harvested on 25 June 2022), GS2 (sown on 12 October 2022 and harvested on 22 June 2023) and GS3 (sown on 28 October 2023 and harvested on 21 June 2024). Durum wheat was sown at a rate of 400 seeds m⁻² with an inter-row spacing of 13 cm and a 2 cm sowing depth.

N fertilization was applied in two split doses. The first dose was applied at tillering (BBCH 25), with 65 kg N ha⁻¹ supplied in the form of ammonium nitrate. The second dose was applied at the onset of stem elongation (BBCH 30), with an additional 65 kg N ha⁻¹ provided as urea.

The SL treatment was applied at booting using a wettable S formulation (Thiovit Jet 80 WG[®], Syngenta, Basel, Switzerland). This timing was selected based on previous field studies demonstrating that S supply at booting supports N–S metabolic coordination and storage protein synthesis during early grain development [24,25,32]. The SL₀ and SL_{6.4} treatments supplied 0 and 6.4 kg S ha⁻¹, corresponding to 0 and 8 kg ha⁻¹ of wettable S formulation, respectively. K treatments (KL₀ and KL_{1.17}) were administered at flowering

using K bicarbonate (Farmacia Agricola S.R.L., Lioni, Avellino, Italy). K bicarbonate was selected because of its practical use in organic farming systems, where it can be applied around flowering as part of crop-protection strategies aimed at limiting fungal infection, while simultaneously providing foliar K. The timing of application reflected the common agronomic use in organic farming systems, where K bicarbonate is typically applied at the onset of flowering to mitigate *Fusarium* infection risk. The application rate was $3 \text{ kg KHCO}_3 \text{ ha}^{-1}$, corresponding to approximately $1.17 \text{ kg K ha}^{-1}$. The selected dose was defined based on the maximum single-application rate considered safe for herbaceous crops and effective according to technical data sheets of similar K bicarbonate products approved for use in organic farming systems in Italy. Fungicide protection was applied uniformly across treatments and no visible disease symptoms were observed during the experimental seasons, therefore, differences between KL treatments were unlikely to result from differential disease control. However, because KHCO_3 supplies both K^+ and HCO_3^- , the experimental treatment does not allow the physiological effects of the two ions to be fully separated. Accordingly, KL should be interpreted as the effect of foliar KHCO_3 supplying $1.17 \text{ kg K ha}^{-1}$ rather than as an experimentally isolated effect of K^+ alone. For both SL and KL treatments, fertilizers were dissolved in 300 L ha^{-1} of water and applied using a field sprayer. To eliminate any confounding irrigation effect from the application of the water, untreated control plots also received 300 L of water ha^{-1} , sprayed in the same manner but without any fertilizer.

Weed and disease management practices were applied uniformly across all plots. Prior to sowing, glyphosate was applied at a rate of 2.3 kg ha^{-1} to control existing vegetation. During the tillering stage, post-emergence weed control was carried out using a selective herbicide formulation containing both 60 g L^{-1} pinoxaden and 15 g L^{-1} cloquintocet-mexyl, applied at a rate of 1 L ha^{-1} . At the booting stage, a foliar fungicide treatment was performed using a product containing bixafen (65 g L^{-1}), fluopyram (65 g L^{-1}), and prothioconazole (130 g L^{-1}), applied at a rate of 1.5 L ha^{-1} . No relevant biotic stress, such as weed pressure, insect infestation, or disease symptoms, was detected during any of the growing seasons. Harvesting was carried out at physiological maturity, once grain moisture declined below 13%.

2.2. Agronomic and Quality Parameters of the Kernels

Within each experimental unit, three 1 m^2 above-ground biomass subsamples were manually collected to assess yield and biomass variability. The number of spikes in each 1 m^2 subsample was counted before threshing to determine spike density (spikes m^{-2}). Grain was then separated from the harvested material using a plot thresher (PLOT 2375, Cicoria, Italy). Following threshing, the total number of grains recovered from each subsample was counted to determine grain number per unit area (grains m^{-2}), while the number of grains per spike was calculated from the ratio between grain number and spike number. GY (t ha^{-1}) was determined by weighing the threshed grain. Thousand kernel weight (TKW, $\text{g } 1000^{-1}$) and test weight (TW, kg hL^{-1}) were measured following the ISO 7971-1 (2009) [33] and ISO 520 (2010) [34] standards, respectively. Wholegrain flour was obtained by milling grain samples through a 0.5 mm screen (Cyclotec 1093, FOSS Tecator, Sweden). Flour samples were sieved to meet the requirements of the Italian “Tipo 2” classification, characterized by an ash content between 0.80% and 0.95%.

Total N content (from 5 mg of flour) was determined using a CHNS analyzer (CHN-S Flash E1112, Thermo-Finnigan, USA), and protein content (PC, %) was calculated by multiplying N content by 5.7 [35]. Total N uptake in grains (Ng, kg ha^{-1}) was then obtained by multiplying the N concentration in grains by the corresponding GY. Total gluten protein composition (Gluten) was determined according to the modified Osborne fractionation

procedure previously described by Guerrini et al. (2020) [32]. Briefly, wholemeal flour obtained from kernels was milled through a 0.5 mm screen and then defatted with hexane prior to protein extraction. Gliadins (Gli) and glutenins (Glu) were sequentially extracted using 70% (*v/v*) ethanol and 0.1 M acetic acid, respectively. Protein fractions were quantified gravimetrically after precipitation and oven drying (105 °C, 5 h). Total Gluten was calculated as the sum of Gli and Glu fractions, and the Gli/Glu ratio was subsequently calculated as an indicator of storage protein partitioning. Results were expressed as a percentage of grain dry matter.

Starch content (SC, %) in wholegrain flour was determined using the Total Starch Assay Kit (K-TSTA, Megazyme, Bray, Ireland), following the procedure described by Cammerata et al. (2021) [36]. Starch yield (SY, t ha⁻¹) was obtained by multiplying GY by SC. Amylose (AM, %) and amylopectin (AP, %) contents, expressed as percentages of SC, were determined using the Megazyme Amylose/Amylopectin Assay Kit (Megazyme Ltd., Bray, Ireland) based on the manufacturer's instructions [37]. The AM/AP ratio was subsequently calculated. Free ASN (μmol g⁻¹) in wholegrain flour was quantified using the enzymatic K-ASNAM kit (Megazyme, Bray, Ireland), with a modification to increase assay sensitivity by using 200 μL of the deproteinized sample instead of 100 μL. The assay is based on the enzymatic conversion of ASN to ammonia, which is then quantified spectrophotometrically [38]. Quantification was performed using a calibration curve corresponding to 0, 0.2, 0.5, 1, 2, 5, and 10 mmol ASN kg⁻¹ of sample. Considering the twofold sample volume used in the assay, the manufacturer-reported limit of quantification (LOQ; 4.9 mg L⁻¹ under standard assay conditions) corresponded to approximately 0.2 mmol ASN kg⁻¹ under the modified conditions. Analytical performance was further monitored by including three randomly selected quality-control samples of known ASN concentration every seven samples analyzed. To normalize free ASN accumulation relative to grain PC, free ASN/PC (μmol g⁻¹ protein) was calculated by dividing the free ASN content (μmol g⁻¹ dry matter) by the PC (g protein g⁻¹ dry matter). This index provides an estimate of the amount of free asparagine remaining per unit of grain protein. This measure reflects the efficiency of nitrogen partitioning between free amino acid storage and protein synthesis. To assess the relationship between N input and N exported through the harvested product, the grain N-to-applied N ratio (Ng/Ns) was calculated as the total amount of N accumulated in harvested grain (Ng; kg ha⁻¹) divided by the amount of N applied to the field (Ns; kg ha⁻¹). This indicator was used to quantify the amount of N contained in the marketable product relative to the external N fertilizer input.

Dough rheological properties were evaluated using a Chopin alveograph [39]. For each test, 250 g of flour was mixed with a 2.5% (*w/w*) NaCl solution for 8 min. No yeast was added. The resulting dough was extruded and allowed to rest for 20 min before testing. The alveograph measured key parameters, including the tenacity (P; mm), the ratio between tenacity and extensibility (P/L), and dough strength (W, 10⁻⁴ J).

The IP, an empirical index, was used to estimate semolina suitability for pasta production. The index was originally developed by the Research Department of the Barilla Pasta Company [20] and later reported by Kovacs et al. (1997) [40]. IP combines gluten content and alveograph parameters to predict the cooking quality of pasta under both low- and high-temperature drying conditions, and was calculated as follows:

$$IP = 4 \times DG + 0.08 \times W + 17 \times \frac{P}{L} - 4 \times \left(\frac{P}{L}\right)^2$$

IP scores range from 0 to 100, with values above 80 considered indicative of high pasta-making quality.

2.3. Calculation of the Net Nitrogen-Adjusted Margin (N_{NaM})

Grain market value (GMV, € t⁻¹) was estimated using a standardised reference price derived from the official wholesale prices of the Bologna Commodity Exchange (“Listino settimanale dei prezzi all’ingrosso n. 27 del 10 July 2025”) for national durum wheat production (northern Italy, harvest 2025). This reference price was adopted to allow for consistent comparisons among fertilization treatments across experimental seasons, rather than to reconstruct year-specific profitability conditions for each GS trial. Three commercial quality categories were considered: “fino” (minimum protein 13% and TW 81 kg hL⁻¹), “buono mercantile” (minimum protein 12% and test weight 80 kg hL⁻¹), and “mercantile” (minimum protein 11% and TW 78 kg hL⁻¹). The PC and TW of each plot were classified into ordinal scores (3 = meeting the highest category thresholds as follows: 2 = intermediate category, 1 = minimum category, and 0 = below minimum for both attributes, respectively). For each observation, the minimum score between the PC and TW classifications was used to assign a commercial quality class. The corresponding GMV for each quality category, as reported by the Bologna Commodity Exchange (297.5 € t⁻¹ for “fino” (fine grade), 282.5 € t⁻¹ for “buono mercantile” (good commercial grade), 272.5 € t⁻¹ for “mercantile” (commercial grade), was then multiplied by the GY to estimate gross revenue (GR; € ha⁻¹).

The cost of foliar fertilization (FFC, € ha⁻¹) was estimated by combining the market price of the products with the costs of field applications. Each application of S (Tiovit Jet[®], Syngenta Italia S.p.A., Milan, Italy) was valued at 15.42 € ha⁻¹, whilst each application of K bicarbonate (Farmacia Agricola S.R.L., Lioni, Italy) was valued at 14.64 € ha⁻¹. In addition, an operational cost of 25 € ha⁻¹ was assigned to each field pass, according to local price surveys. The total treatment cost per ha⁻¹ was calculated as the sum of the product price and application cost. The N_{NaM} was calculated as the difference between GR and FFC and should, therefore, be interpreted as a comparative indicator of treatment performance under standardised price conditions rather than as an estimate of actual profitability for the individual experimental years.

2.4. Statistical Analyses

All statistical analyses were carried out in RStudio version 4.3.1 [41]. Data were first checked for compliance with the assumptions of normality and homoscedasticity using the Shapiro–Wilk test and Levene’s test, respectively. When assumptions were not met, a Box–Cox transformation was applied to normalize the distribution of the response variables.

Linear mixed-effects models were then fitted using the lmer function from the lme4 package [42]. The model structure included SL, KL, and Var as fixed factors, together with their two- and three-way interactions. The random effect structure accounted for the hierarchical design of the experiment, with replicates nested within fields, and fields nested within GSs (Years). Growing season (GS) was included in the random-effects structure because the primary objective of the analysis was to estimate the overall effects of SL, KL, and Var across the experimental period, rather than to estimate treatment effects separately for each individual year. This approach accounts for the variability associated with growing seasons and with the hierarchical structure of the field experiments while allowing inference on treatment effects across the range of environmental conditions represented by the study. Accordingly, data from the different GSs were analysed jointly within the mixed-effects framework rather than through separate year-specific analyses. Thus, the general model used for GY was expressed as

$$Y_{ijkl} = \beta_0 + \beta_1 SL_i + \beta_2 KL_j + \beta_3 Var_k + \beta_{ij}(SL \times KL) + \beta_{ik}(SL \times Var) + \beta_{jk}(KL \times Var) + \beta_{ijk}(SL \times KL \times Var) + (1 | Year/Field/Replicate) + \epsilon$$

where Y_{ijkl} is the observed value of the trait, β_0 is the intercept and ϵ is the residual error. β terms represent the fixed effects and their interactions.

The significance of the fixed effects was assessed using Type III analysis of variance with Satterthwaite's approximation for denominator degrees of freedom. The residual variance was obtained from the estimated variance component of the model and expressed as the sum of squares, mean squares, and degrees of freedom. When significant interactions were detected, interaction plots were used to aid interpretation. Post hoc comparisons of treatment means were performed with Tukey's HSD test to assess pairwise differences among factor levels ($p < 0.05$).

Finally, the proportion of variance explained by each source of variation was calculated as the ratio of its sum of squares to the total sum of squares (excluding the residual) in order to quantify the relative contribution of each factor to the variability of the studied traits.

3. Results

3.1. Yield Components and the Physical Grain Characteristics

GY was significantly affected by SL and KL foliar fertilization, whereas no significant effect of Var was detected (Table 3). No significant interactions among factors were observed. On average, SL application increased GY by 0.47 t ha^{-1} compared with the untreated control, while KL application increased GY by 0.36 t ha^{-1} (Table 4).

Table 3. ANOVA results for grain yield (GY), spike number per unit area, grain number (no) per spike, grain number per unit area, thousand kernel weight (TKW) and test weight (TW), considering the effects of wheat variety (Var), leaf sulfur fertilization (SL), and leaf potassium fertilization (KL).

Source of Variability	DF	Sum of Squares					
		GY	Spike Number per Unit Area	Grain Number per Spike	Grain Number per Unit Area	TKW	TW
SL	1	0.013 ***	1.16×10^{-7}	470,612 ***	10,989 **	357	738
KL	1	0.006 *	4.80×10^{-10}	143,916 ***	5,616 *	546	3747
Var	1	0.001	1.01×10^{-6}	14,010	1,023	33,505 *	410,005 **
SL $\times$ KL	1	0.002	2.77×10^{-7}	5959	1,999	173	2469
SL $\times$ Var	1	0.001	5.04×10^{-7}	1431	0.368	45	847
KL $\times$ Var	1	0	3.50×10^{-7}	1275	0.284	38	119
SL $\times$ KL $\times$ Var	1	0.002	7.75×10^{-7}	14,028 *	2.5	897	255
Error	64	0.068	8.51×10^{-6}	137,660.5	0.00003	25,002.77	109,041.6

Significance: * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$.

Table 4. Means ($\pm$ Standard Error) of grain yield (GY), spike number per unit area, grain number (no) per spike, grain number per unit area, thousand kernel weight (TKW) and test weight (TW), across the main factors: wheat variety (Var), leaf sulfur fertilization (SL), and leaf potassium fertilization (KL). Lowercase letters indicate significant differences among levels according to Tukey's HSD test ($p < 0.05$).

Source of Variability	Levels	GY	Spike Number per Unit Area	Grain Number per Spike	Grain Number per Unit Area	TKW	TW
		t ha^{-1}	Spikes m^{-2}	Grains Spike $^{-1}$	Grains m^{-2}	g 1000 Grains $^{-1}$	kg hL $^{-1}$
SL	SL_0	4.14 (0.13) b	406 (12.8)	22.6 (0.3) b	9165 (300) a	45.15 (0.1)	81.28 (0.08)
	SL_6.4	4.61 (0.12) a	392.1 (10.8)	26 (0.2) a	10,179 (276) b	45.2 (0.09)	81.24 (0.08)
KL	KL_0	4.2 (0.11) b	398 (11)	23.4 (0.4) b	9281 (275) a	45.21 (0.1)	81.31 (0.08)
	KL_1.17	4.56 (0.13) a	400 (12.7)	25.3 (0.4) a	10,063 (310) b	45.15 (0.1)	81.24 (0.08)
Var	Creso	4.52 (0.14)	431.7 (12.2)	23.4 (0.4)	10,129 (332)	44.73 (0.07) b	80.45 (0.01) b
	Svevo	4.23 (0.11)	366.3 (8.6)	25.2 (0.4)	9215 (242)	45.62 (0.05) a	82.07 (0.01) a

Spike density was not affected by SL, KL, Var, or their interactions (Table 3). In contrast, both SL and KL significantly increased the number of grains per spike and, consequently, the number of grains per unit area. SL increased the number of grains per spike by 15% and

the number of grains per unit area by 11.1% (Table 4). Similarly, KL application increased the number of grains per spike by about 8.1% and the number of grains per unit area by approximately 8.4% (Table 4).

TKW and TW were primarily influenced by Var (Table 3), with significantly higher TKW and TW found in Svevo (Table 4). Neither SL nor KL significantly affected kernel size and density.

3.2. Grain Protein Composition, Grain N-to-Applied N Ratio, and Free Asparagine

The ANOVA (Table 5) showed that PC was significantly influenced by SL and to a lesser extent by Var. In contrast, neither KL nor any of the interactions was significant. When comparing treatment levels (Table 6), SL led to a significant increase in PC (+6.2%) with respect to the control.

Table 5. ANOVA results for grain protein concentration (PC), gliadin content (Gli), glutenin content (Glu), gliadin-to-glutenin ratio (Gli/Glu), gluten content (Gluten), free asparagine (Free ASN), free asparagine-to-protein concentration ratio (Free ASN/PC), and grain N-to-applied N ratio (Ng/Ns), considering the effects of wheat variety (Var), leaf sulfur fertilization (SL), and leaf potassium fertilization (KL).

Source of Variability	DF	Sum of Squares							
		PC	Gly	Glu	Gly/Glu	Gluten	Free ASN	Free ASN/PC	Ng/Ns
SL	1	0.216 ***	163.61 ***	842.04 ***	0.159 ***	6645.9 ***	15.79 ***	32.31 ***	0.085 ***
KL	1	0	17.39	0.72	0.047 ***	111.7	3.37 ***	5.16 ***	0.019 *
Var	1	0.422 *	22.08	3.09	0.119 ***	170.3	0.53 ***	0.57 *	0.001
SL × KL	1	0.016	0.97	100.5 **	0.114 ***	427.2	0.62 ***	0.19	0.012 *
SL × Var	1	0.01	9.52	58.21 *	0.015 *	440	0.16 *	0.71 **	0
KL × Var	1	0.002	1.07	17.33	0.009	100.7	0.11	0.15	0
SL × KL × Var	1	0.01	18.17	22.88	0.003	323.4	0.01	0.07	0.009
Error	64	0.391	433.64	791.68	0.197	9092.1	1.73	5.71	0.194

Significance: * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$.

Table 6. Means ($\pm$ Standard Error) of grain protein concentration (PC), gliadin content (Gli), glutenin content (Glu), gliadin-to-glutenin ratio (Gli/Glu), gluten content (Gluten), free asparagine (Free ASN), free asparagine-to-protein concentration ratio (Free ASN/PC), and grain N-to-applied N ratio (Ng/Ns) across the main factors of wheat variety (Var), leaf sulfur fertilization (SL), and leaf potassium fertilization (KL). Lowercase letters indicate significant differences among levels according to Tukey's HSD test ($p < 0.05$).

Source of Variability	Levels	PC	Gli	Glu	Gli/Glu	Gluten	Free ASN	Free ASN/PC	Ng/Ns
		%	%	%		%	$\mu\text{mol ASN g}^{-1}$	$\mu\text{mol ASN g}^{-1} \text{ Protein}$	
SL	SL_0	12.99 (0.15) b	4.95 (0.05) b	5.81 (0.06) b	0.853 (0.006) b	10.77 (0.11) b	4.69 (0.16)	32.45 (1.85)	0.63 (0.019)
	SL_6.4	13.8 (0.16) a	5.25 (0.05) a	6.38 (0.05) a	0.824 (0.002) a	11.63 (0.1) a	1.36 (0.1)	8.87 (0.66)	0.74 (0.016)
KL	KL_0	13.4 (0.17)	5.15 (0.05)	6.1 (0.08)	0.846 (0.006) a	11.25 (0.13)	3.48 (0.35)	26.56 (2.78) a	0.66 (0.019)
	KL_1.17	13.39 (0.17)	5.05 (0.06)	6.09 (0.07)	0.83 (0.003) b	11.14 (0.13)	2.57 (0.24)	14.76 (1.45) b	0.71 (0.02)
Var	Creso	12.7 (0.1) b	5.01 (0.05)	6.07 (0.06)	0.826 (0.003) b	11.07 (0.11)	2.78 (0.31)	19.89 (2.49)	0.67 (0.021)
	Svevo	14.09 (0.14) a	5.2 (0.06)	6.12 (0.08)	0.851 (0.006) a	11.32 (0.14)	3.27 (0.3)	21.43 (2.36)	0.7 (0.019)

SL application significantly affected all protein fractions (Table 5). Both Gli and Glu contents increased following SL ($p < 0.001$), with SL explaining 27.4% and 42.9% of the total variance, respectively. Total gluten content also increased significantly ($p < 0.001$), with SL accounting for approximately 31.9% of the observed variation. In contrast, KL and Var had no significant effects on total gluten content. The Gli/Glu ratio was significantly affected

by SL ($p < 0.001$), KL ($p < 0.001$), and Var ($p < 0.001$), with significant SL $\times$ KL and SL $\times$ Var interactions.

At the treatment level (Table 6), SL significantly increased Gli, Glu, and total gluten contents, while reducing the Gli/Glu ratio, indicating a proportionally greater increase in Glu than in Gli. Significant SL $\times$ KL interactions were observed for Glu and the Gli/Glu ratio. Under S deficiency (SL₀), KL slightly increased Glu and significantly reduced the Gli/Glu ratio. Conversely, under SL_{6.4}, Glu remained high irrespective of KL application, whereas the Gli/Glu ratio remained consistently low. Significant SL $\times$ Var interactions were also detected, with Svevo showing a stronger Glu response to SL than Creso. Instead, Creso exhibited the lowest Gli/Glu ratio under SL.

The ANOVA revealed highly significant effects of all main factors, together with significant SL $\times$ KL and SL $\times$ Var interactions for both free ASN content and the free ASN/PC ratio (Table 5). Among the investigated factors, SL accounted for the largest proportion of the total variance in free ASN (70.8%), followed by KL (15.1%), SL $\times$ KL (2.8%), and Var (2.4%). A similar pattern was observed for free ASN/PC, for which SL explained an even greater proportion of the variance (81.3%), followed by KL (13.0%), whereas the contribution of Var and the interaction terms remained comparatively small.

At the treatment level, SL application reduced free ASN concentration by 70.4%, while KL decreased free ASN by approximately 26% (Table 6). When normalized by PC, the effect became even more pronounced. Free ASN/PC declined significantly following SL application (−72.7%), whereas KL reduced the ratio by 44.4% (Table 6). The significant SL $\times$ KL interaction showed that the lowest free ASN content and the lowest free ASN/PC ratio were consistently achieved when S and K were applied together (Figure 3), indicating complementary effects of the two foliar treatments in limiting free ASN accumulation. The significant SL $\times$ Var interaction indicated no varietal differences under SL₀, whereas under SL_{6.4}, Svevo retained significantly higher residual free ASN and a higher free ASN/PC ratio than Creso (Figure 3), indicating a smaller reduction in free ASN in response to SL in Svevo than in Creso.

The ANOVA (Table 5) revealed that the Ng/Ns ratio was significantly affected by both SL and KL fertilization, while no significant effect of Var was detected. On the treatment level (Table 6), the Ng/Ns ratio increased in response to both SL and KL applications, indicating a greater amount of N accumulated in harvested grain relative to the N fertilizer input. The SL $\times$ KL interaction significantly influenced the Ng/Ns ratio, indicating that the effect of KL on Ng/Ns depended on SL treatment. The post hoc analysis (Tukey's HSD) revealed that the Ng/Ns ratio was significantly lower in the absence of both SL and KL fertilization compared to all remaining treatment combinations (Figure 4). In contrast, when at least one foliar treatment was applied, either SL, KL, or both, the Ng/Ns ratio was significantly higher than in the untreated control. No significant differences were detected between these three fertilizer combinations.

3.3. Starch Composition of the Grain

Both SL and KL significantly affected SC, starch yield (SY), AM, and AM/AP, whereas no significant interaction effects among the experimental factors were detected (Table 7). SL significantly reduced SC, while increasing SY (Table 8). In addition, SL significantly increased AM and the AM/AP ratio.

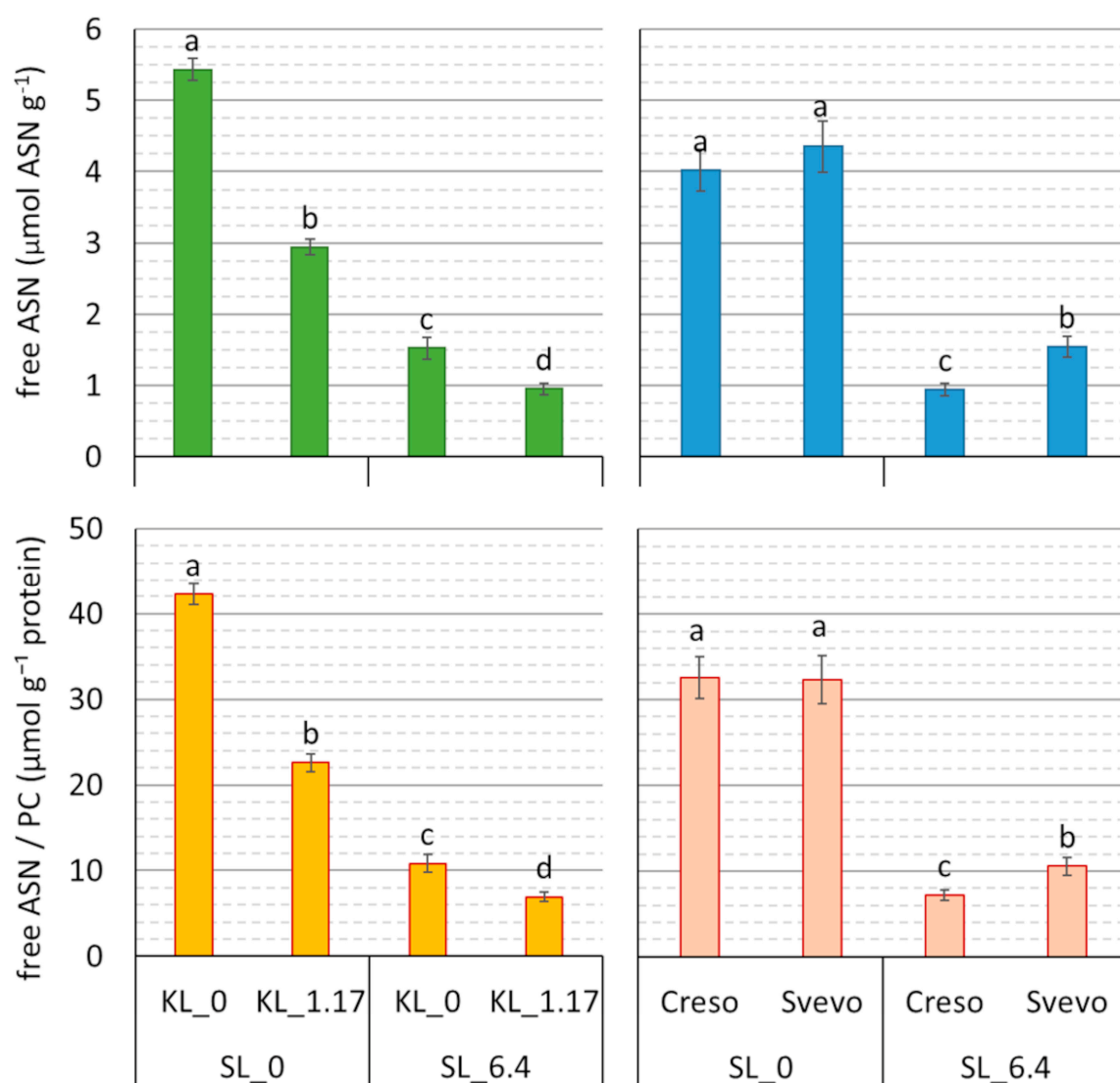


Figure 3. Interaction effects of leaf sulfur (SL), potassium (KL), and cultivar (Var) on free asparagine (ASN) content (**upper panels**) and the free asparagine-to-protein (ASN/PC) ratio (**lower panels**) in durum wheat grain. The (**left panels**) show the significant SL × KL interaction, whereas the (**right panels**) show the significant SL × Var interaction. Values are means ± standard error. Different lowercase letters indicate significant differences among treatment combinations according to Tukey's HSD test ($p < 0.05$).

Table 7. ANOVA results for starch content (SC), starch yield per hectare (SY), amylose concentration (AM), and amylose to amylopectin ratio (AM/AP), considering the effects of wheat variety (Var), leaf sulfur fertilization (SL), and leaf potassium fertilization (KL).

Source of Variability	DF	Sum of Squares			
		SC	SY	AM	AM/AP
SL	1	235,689 ***	0.015 **	0.034 ***	0.045 ***
KL	1	198,874 ***	0.012 **	0.025 ***	0.034 ***
Var	1	9992	0.001	0.027 *	0.036 *
SL × KL	1	8150	0.003	0	0
SL × Var	1	1167	0.001	0	0
KL × Var	1	21,430	0.001	0.001	0.001
SL × KL × Var	1	3284	0.003	0.001	0.001
Error	64	439,911.3	0.105	0.021	0.029

Significance: * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$.

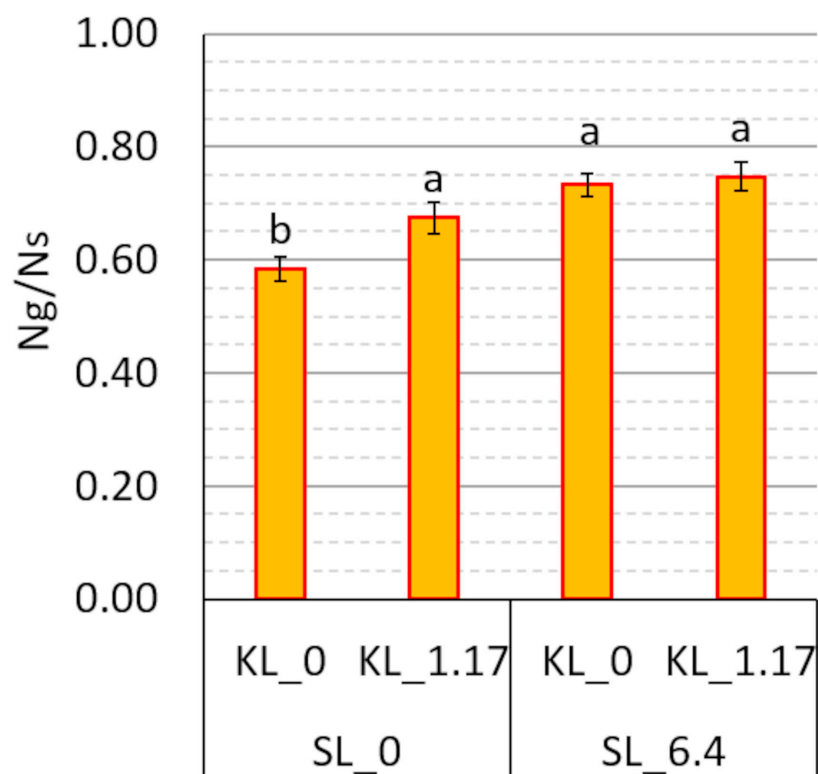


Figure 4. Effect of sulfur (SL) and potassium (KL) foliar fertilization on the grain N-to-applied N ratio (Ng/Ns). Error bars represent the standard error of the mean. Different lowercase letters indicate significant differences between treatments according to Tukey's HSD test ($p < 0.05$).

Table 8. Means ($\pm$ Standard Error) of starch content (SC), starch yield per hectare (SY), amylose concentration (AM), and amylose to amylopectin ratio (AM/AP), across the main factors: wheat variety (Var), leaf sulfur fertilization (SL), and leaf potassium fertilization (KL). Lowercase letters indicate significant differences among levels according to Tukey's HSD test ($p < 0.05$).

Source of Variability	Levels	SC	SY	AM	AM/AP
		%	t ha ⁻¹	%	
SL	SL_0	69.53 (0.13) a	2.88 (0.09) b	25.1 (0.3) b	0.335 (0.006) b
	SL_6.4	68.7 (0.12) b	3.17 (0.08) a	26.2 (0.3) a	0.355 (0.006) a
KL	KL_0	68.74 (0.14) b	2.89 (0.08) b	26.1 (0.3) a	0.354 (0.006) a
	KL_1.17	69.5 (0.11) a	3.17 (0.1) a	25.1 (0.3) b	0.337 (0.006) b
Var	Creso	69.01 (0.16)	3.13 (0.1)	23.8 (0.1) b	0.312 (0.002) b
	Svevo	69.22 (0.13)	2.93 (0.08)	27.4 (0.2) a	0.378 (0.004) a

KL showed the opposite effect on starch composition (Table 8). KL significantly increased SC and SY, and reduced AM and the AM/AP ratio. Var had no significant effect on SC or SY but significantly affected starch composition (Table 7). Svevo exhibited higher amylose concentration and a greater AM/AP ratio than Creso (Table 8). Overall, SL and KL showed contrasting effects on starch composition: SL increased AM and the AM/AP ratio, whereas KL increased SC while reducing AM and the AM/AP ratio.

3.4. Technological Quality and Economic Performance

All main factors (SL, KL, and Var) as well as the SL $\times$ KL interaction significantly affected W (Table 9). The largest share of variance in W was explained by Var, accounting for 34.4%, followed by SL (30%), SL $\times$ KL (22.7%) and KL (3.6%) (Table 10). The combined application of S and K resulted in marked differences in W values depending on the treatment combination (Figure 5). Compared with the control (SL_0 + KL_0), W increased by 8.6% with KL application alone (SL_0 + KL_1.17), by 9.5% when both foliar treatments were applied (SL_6.4 + KL_1.17), and by 12.9% with SL application alone (SL_6.4 + KL_0).

Table 9. ANOVA results for alveograph dough strength (W), tenacity (P), extensibility ratio (P/L), pasta-making index (IP) and net nitrogen-adjusted margin (NNaM). The effects of leaf sulfur fertilization (SL), leaf potassium foliar fertilization (KL), variety (Var), and their interactions, are shown.

Source of Variability	DF	Sum of Squares				
		W	P	P/L	IP	NNaM
SL	1	916,579,187 ***	35.969 ***	3.973 ***	11,206,372 ***	0.139 *
KL	1	109,026,161 ***	0.025	0.045	99	0.041
Var	1	1,052,796,038 ***	11.689 ***	44.022 **	87,315	0
SL × KL	1	694,474,055 ***	0.011	0.047	1,873,469 **	0.042
SL × Var	1	4,820,827	0.941	0.132	439,515	0.008
KL × Var	1	2,708,056	0.07	0.038	106,352	0.002
SL × KL × Var	1	4,384,278	0.149	0.087	223,919	0.037
Error	64	276,000,961	27.948	10.993	6,619,007	0.1272

Significance: * $p < 0.05$; ** $p < 0.01$; *** $p < 0.001$.

Table 10. Means ($\pm$ Standard Error) of dough strength (W), tenacity (P), extensibility ratio (P/L), pasta-making index (IP), and net nitrogen-adjusted margin (NNaM) as affected by leaf sulfur fertilization (SL), leaf potassium foliar fertilization (KL), and variety (Var). Different lowercase letters within the same column indicate significant differences between treatment levels according to Tukey's HSD test ($p < 0.05$).

Source of Variability	Levels	W	P	P/L	IP	NNaM
		10^{-4} J	mm			€ ha ⁻¹
SL	SL_0	229.74 (2.23)	37.82 (0.13) a	2.35 (0.04) a	79.05 (0.45)	1168.17 (34.18) b
	SL_6.4	244.93 (1.67)	36.4 (0.13) b	2.25 (0.04) b	83.91 (0.37)	1272.00 (32.25) a
KL	KL_0	234.53 (2.88)	37.09 (0.17)	2.3 (0.04)	81.47 (0.67)	1189.08 (31.04)
	KL_1.17	240.15 (1.53)	37.13 (0.18)	2.31 (0.04)	81.49 (0.53)	1251.08 (36.65)
Var	Creso	245.42 (1.83) a	36.71 (0.18) b	2.09 (0.02) b	81.92 (0.51)	1229.20 (36.86)
	Svevo	229.26 (1.99) b	37.51 (0.14) a	2.51 (0.02) a	81.05 (0.64)	1210.96 (31.60)

Both P and P/L were significantly affected by SL and Var (Table 9). SL led to a reduction in P and P/L values by 3.8% and 4.3% respectively (Table 10). Varietal differences were also significant, with Svevo exhibiting higher P and P/L than Creso, suggesting a more tenacious and less extensible dough. No significant effects were detected for either KL or any of the interactions.

The IP was significantly influenced by SL. A significant SL × KL interaction was also detected (Table 9). SL accounted for approximately 48.9% of the total variability in IP, while the SL × KL interaction accounted for 8.1% (Table 10). Post hoc comparisons showed that under SL_0 conditions, the application of KL improved IP from 77.85 to 80.22 (+3.0%). However, under SL_6.4, both KL-treated and untreated plots attained significantly higher IP values.

The ANOVA revealed that SL had a significant effect on NNaM, while no significant effects were observed for KL or Var, or any of the interactions (Table 9). SL accounted for approximately 35.1% of the variance in NNaM, while the remainder was largely attributed to residual variation. At the treatment level (Table 10), SL application increased NNaM by 8.9% relative to SL_0, with a net economic gain of approximately 104 € ha⁻¹. Although KL tended to increase NNaM from KL_0 to KL_1.17 by 5.2% (62 € ha⁻¹), the difference was not statistically significant.

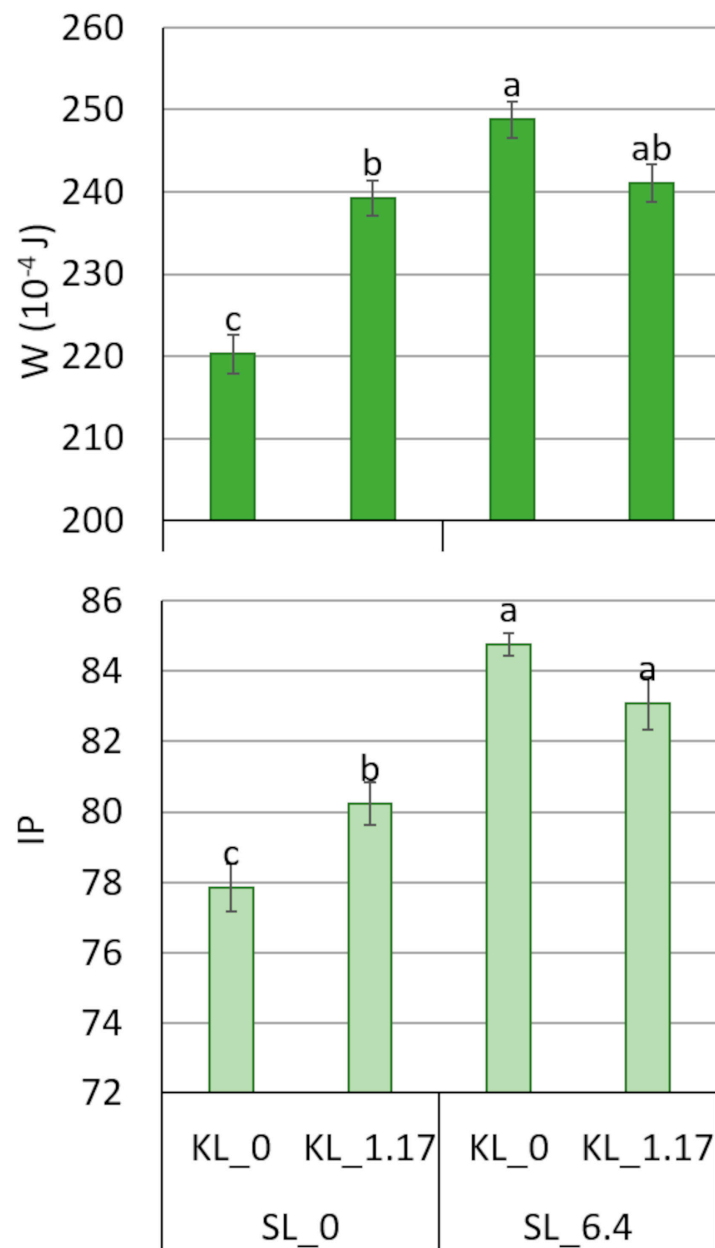


Figure 5. Interaction effects between sulfur (SL) and potassium (KL) leaf fertilization on dough strength (W, **top**) and pasta-making index (IP, **bottom**). Error bars represent standard errors of the mean. Different lowercase letters indicate statistically significant differences among treatment combinations according to Tukey's HSD test ($p < 0.05$).

4. Discussion

4.1. Novelty, Significance, and Broader Implications

The novelty of the present study lies in the integrated assessment of agronomic performance (yield), protein and starch composition, technological quality (rheological properties), free ASN accumulation, and the economic performance in response to foliar S applied at booting, followed by K at flowering, in durum wheat under rainfed Mediterranean conditions. Previous studies have generally focused on individual physiological or technological aspects of S and K fertilization, mainly in bread wheat, and have often examined soil-applied nutrients or applications at earlier developmental stages without considering economic outcomes [19,24,26,32,43]. The present study therefore provides a more comprehensive field-based assessment of the responses to SL and KL in durum wheat.

An additional contribution of this study is the assessment of the distinct and combined responses to S and K supplied at late developmental stages. SL was primarily shown to enhance N assimilation and N allocation towards storage protein synthesis, increasing PC, Gluten accumulation, Glu, N recovery and W, while simultaneously reducing free ASN and the free ASN/PC ratio. KL, although exerting a smaller influence on PC, was associated with changes in storage protein and starch composition and complemented several effects of SL during grain filling.

From a technological perspective, the study showed that the SL and KL administration at booting and flowering, respectively, modified not only the quantity but also the composition of both protein and starch fractions. The preferential accumulation of Glu relative to Gli, together with the contrasting effects on AM and AP partitioning, provided a mechanistic basis for the improvements observed in dough rheology and the PI. Moreover, the reduction in free ASN suggests potential additional quality-related benefit, although acrylamide formation was not directly assessed. The effects of SL and KL on agronomic performance, grain composition, rheological properties, Ng/Ns, and NNaM are discussed in more detail in the following sections.

4.2. Grain Yield and Physical Components of the Kernels

When evaluated across the three growing seasons, both SL and KL significantly increased GY, with no detectable effects of Var or the interactions. The 0.47 t ha^{-1} yield increase with SL and the 0.36 t ha^{-1} increase with KL are consistent with previous reports reporting that S promotes N assimilation, enzyme activity, and protein synthesis, while K enhances photosynthesis, assimilate transport, stomatal regulation, and source–sink relationships during grain filling [7–9,19,43–45]. The positive response to SL was likely enhanced by the absence of basal S fertilization, suggesting that soil S availability was limiting under the present conditions [19,46], whereas the beneficial effect of KL was consistent with previous reports on foliar K fertilization in wheat [26].

The analysis of yield components indicated that the higher GY was primarily associated with a greater number rather than a greater kernel mass. Neither SL nor KL affected spike density, which is determined before the booting and flowering applications, whereas both treatments significantly increased grains per spike and, consequently, grain number per unit area, without affecting TKW or TW. These findings are consistent with an effect of late nutrient supply on grain set around anthesis rather than grain filling, consistent with the greater sensitivity of reproductive processes to S availability and the role of K in supporting assimilate transport, pollen viability and reproductive sink strength [7,44,45,47].

Overall, these results indicated that late SL and KL applications increased GY primarily through a greater grain number per unit area rather than increased individual kernel mass. This interpretation is also consistent with previous studies reporting that grain number was generally more responsive than TKW to late nutrient supply, whereas TKW and TW remained relatively stable and appeared largely under genetic control [19,24,25].

4.3. Grain Protein Composition, Grain N-to-Applied N Ratio, and Free Asparagine

Foliar SL primarily affected traits related to grain N partitioning rather than simply increasing PC. Although PC increased significantly following SL application, the accompanying increase in gluten accumulation, greater Glu relative to Gli, higher Ng/Ns ratio, and lower free ASN concentration was consistent with a greater allocation of grain N towards functional storage proteins. These coordinated responses are compatible with the close physiological coupling between S and N metabolism previously reported in wheat [7,9,43,48]. In contrast, KL had no significant effect on PC but affected traits related to N metabolism and protein partitioning, responses that may be associated with well-

established functions in enzyme activation, assimilate transport, and N remobilization during grain development [8,9]. Unlike our previous studies, which investigated foliar S and Se applications in bread wheat [19,24,25], the present work demonstrated that K administration complemented S by influencing N allocation without altering PC.

The higher Glu concentration and lower Gli/Glu ratio observed after SL application indicated that S modified not only the quantity but also the composition of the gluten proteins. Because Glu are richer in S-containing amino acids than gliadins, adequate S supply preferentially promotes Glu synthesis, thereby improving storage protein composition [7,49]. Similar responses have previously been reported under field conditions [24,32]. Although KL alone had little effect on Gluten, the significant SL $\times$ KL interaction for Glu and the Gli/Glu ratio suggests that the response of storage protein composition to SL could be modulated by KL. Likewise, the significant SL $\times$ Var interaction demonstrated that the magnitude of this response remained genotype dependent.

The coordinated changes in protein composition were accompanied by an increase in Ng/Ns ratio and a marked reduction in free ASN. S was the dominant factor associated with variation in free ASN accumulation, accounting for more than 70% of the treatment effect, whereas K produced a smaller but significant additional reduction. More importantly, the free ASN/PC ratio declined even more markedly, indicating that the reduction in free ASN occurred concurrently with greater storage protein accumulation. This pattern is consistent with a possible shift in N partitioning from the free amino acid pool towards storage proteins. These findings corroborated previous reports showing greater free ASN accumulation under S deficiency and enhanced protein synthesis under adequate S nutrition [17–19,24,50]. K further reduced both free ASN concentration and the ASN/PC ratio, this response may be related to the established role of K in N remobilization and metabolic stability during grain filling [5,15,16].

Overall, the concurrent increase in PC, preferential accumulation of Glu, higher N recovery, and marked reduction in free ASN and the ASN/PC ratio indicated that foliar S, complemented by K, redirected N partitioning away from free amino acid accumulation towards storage protein synthesis. This coordinated response was shown to provide the physiological explanation for the improvements in technological quality discussed in the following section.

4.4. Grain Protein Composition, Nitrogen Use Efficiency and Free Asparagine

Both SL and KL significantly affected starch traits, although their effects on starch composition differed. SL slightly reduced SC while increasing SY, whereas KL increased both SC and SY. The increase in SY observed for both treatments primarily reflected the higher grain yield obtained under foliar fertilization rather than a greater SC per se. Thus, both treatments increased starch yield at the crop level despite their contrasting effects on SC.

The reduction in SC following SL was consistent with the close coordination between C and N metabolism during grain filling. S is known to support N assimilation and protein synthesis, processes that increase the demand for C skeletons required for amino acid biosynthesis and storage protein accumulation [6,7,10,48]. The simultaneous increase in PC and modest decrease in SC observed here may therefore reflect changes in C–N partitioning associated with enhanced protein accumulation. Similar responses have previously been reported following foliar S fertilization in bread wheat [19,24] and are consistent with the well-established negative relationship between grain PC and SC in cereals [51,52].

Interestingly, SL also increased AM and the AM/AP ratio, indicating that SL modified starch composition in addition to total SC. The mechanisms underlying this response cannot be established from the present experiment because starch biosynthetic enzyme activities and associated metabolic pathways were not measured. Therefore, the observed changes in AM and AP should be regarded as treatment-associated responses rather than evidence of a specific regulation of starch biosynthesis.

In contrast, KL increased SC while reducing both AM and the AM/AP ratio. These findings may be interpreted in light of the established role of K in regulating photosynthesis, phloem loading, assimilate transport, and carbohydrate metabolism [8,9,44]. Previous studies have shown that adequate K improves sucrose translocation towards developing grains and enhances the activity of enzymes involved in starch synthesis, thereby promoting carbohydrate deposition. However, because these processes were not measured here, the lower AM proportion observed under KL cannot be directly attributed to a specific alteration of starch biosynthesis.

The absence of significant treatment interactions indicates that the measured starch traits responded largely independently to SL and KL under the conditions of this study. Svevo exhibited a higher AM and a greater AM/AP ratio than Creso, indicating a clear varietal component in starch composition. Overall, these results showed contrasting associations of SL and KL with starch composition. SL was associated with higher protein accumulation, slightly lower SC, and a greater AM proportion, whereas KL was associated with greater carbohydrate accumulation and a lower AM proportion. These contrasting responses suggest that S and K may differently influence C partitioning and starch composition, although the underlying metabolic mechanisms require direct investigation.

4.5. Technological Quality and Economic Performance

The improvements in rheological performance observed in the present study were consistent with the coordinated changes in grain protein and starch composition described above. SL increased W while reducing P and the P/L ratio, responses associated with the preferential accumulation of Glu and the lower Gli/Glu ratio. Since Glu are the principal determinant of W, whereas gliadins mainly contribute to L, the shift in storage protein partitioning is consistent with the improved rheological properties following SL [49,53]. Similar responses have previously been reported in bread wheat [19,24,32], and the present study corroborated the benefits in durum wheat.

Although KL had little effect on total Gluten, its interactions with SL for Glu, the Gli/Glu ratio, and W indicate that the response to KL depended partly on S supply. The contrasting effects of S and K on starch composition may reflect their distinct physiological roles during grain filling. S availability is closely coupled with N assimilation and storage protein synthesis and may therefore indirectly modify carbon allocation between protein and starch, whereas K more directly regulates assimilate transport, enzyme activation and carbohydrate metabolism in developing grains. These distinct effects on carbon partitioning may also have contributed to the observed rheological responses, since starch composition influences water absorption, dough consistency and processing behaviour [54]. Together, these findings indicate that the technological improvements were associated with coordinated changes in both protein and starch composition.

The IP confirmed the dominant role of S in determining overall technological quality, with S explaining nearly half of the treatment effect. K improved IP mainly under S-deficient conditions, mirroring the responses observed for Glu accumulation and the Gli/Glu ratio, which further supported its complementary role. Although Var differences remained evident, particularly between Creso and Svevo, both cultivars responded positively to SL, indicating that these improvements were consistent across contrasting quality backgrounds.

From an economic perspective, SL significantly increased the NNaM, reflecting the combined effects of higher GY, enhanced grain quality, and the relatively low cost of the foliar treatment [7,19,46,48]. Similar economic benefits have previously been reported under S-limited conditions [19,24]. KL increased NNaM by approximately 5.2% ($\approx \text{€}62 \text{ ha}^{-1}$), although this response was not statistically significant. Nevertheless, its positive effects on storage protein composition, starch partitioning, Ng/Ns, and free ASN suggest that its economic value may be greater in production systems with lower soil K availability or where grain quality premiums are available or where reductions in free ASN are valued as a processing-quality attribute. From a practical perspective, foliar S and K applications may therefore represent relatively simple complementary tools for improving both crop performance and grain quality, particularly when integrated into existing foliar treatment operations. Their economic attractiveness will ultimately depend on field nutrient status, treatment costs, the magnitude of the yield response, and the extent to which improvements in grain technological and sanitary quality are reflected in the price received by farmers.

4.6. Limitations and Future Directions

The present study was conducted over three consecutive growing seasons under Mediterranean rainfed conditions, which were characterized by appreciable differences in weather conditions. Such environmental variability may influence crop responses to nutrient supply, particularly under rainfed conditions. In the present analysis, GS was incorporated into the random-effects structure of the mixed model, and the reported effects of SL and KL therefore represent their overall responses across the environmental conditions experienced during the three growing seasons rather than year-specific responses. Although the significant overall effects observed across the experimental period indicate that foliar S and K fertilization can provide agronomic and quality benefits under variable seasonal conditions, the adopted model was not designed to test whether the magnitude of these responses differed significantly among individual growing seasons. Further studies explicitly addressing fertilization $\times$ environment interactions would therefore be valuable to determine the stability of these responses under specific climatic conditions. Moreover, validation across a wider range of environments, soil types, and S and K availability would therefore be valuable before extending these findings to other durum wheat production systems. Likewise, although Creso and Svevo represent contrasting quality types widely cultivated in Italy, additional genotypes must be evaluated to better assess genotype-dependent responses to foliar fertilization.

The physiological mechanisms proposed in this study were inferred from field-based phenotypic responses rather than directly quantified. Future research integrating enzyme activity with transcriptomic, proteomic and metabolomic approaches would improve understanding of how S and K regulate N partitioning, storage protein synthesis, starch metabolism and free ASN accumulation during grain development. Likewise, direct measurements of floret survival, grain set and kernel abortion would further clarify the mechanisms responsible for the increase in grain number following late foliar fertilization.

The reduction in free ASN may also have implications for processing quality, given the established role of ASN as a major precursor of acrylamide formation during high-temperature processing. However, acrylamide formation was not directly assessed in the present study; therefore, the observed reduction in grain ASN should be regarded as an indication of the potential to reduce acrylamide formation rather than evidence of a direct reduction in acrylamide content or associated risk.

Finally, S and K were applied at different developmental stages, with S supplied at booting and K at flowering, reflecting current agronomic practice and the distinct physiological roles of the two nutrients. Consequently, the observed SL $\times$ KL responses should be interpreted as complementary effects of stage-specific nutrient management rather than evidence of a direct S–K interaction. Future studies should compare alternative application timings, including simultaneous and stage-specific applications, together with different nutrient sources, to distinguish the effects of nutrient timing from those of nutrient interactions. Broader economic assessments incorporating market variability, grain quality premiums and environmental incentives would further strengthen the practical relevance of these findings.

A further consideration concerns the use of K bicarbonate as the source of foliar K. Because K bicarbonate supplies both K^+ and HCO_3^- , the experimental design does not allow their individual contributions to the observed responses to be separated. Although the interpretation of KL effects in terms of K nutrition is consistent with the established physiological roles of K in assimilate transport, enzyme activation, and carbohydrate and N metabolism, a contribution of the bicarbonate component cannot be excluded. Therefore, the effects associated with KL should be more precisely interpreted as responses to foliar K bicarbonate supplying $1.17 \text{ kg K ha}^{-1}$. Future studies comparing K bicarbonate with other foliar K sources at equivalent K rates would help distinguish K-specific nutritional effects from those potentially associated with the accompanying anion.

5. Conclusions

Foliar S applied at booting and K applied at flowering improved durum wheat performance under Mediterranean rainfed conditions. Both treatments increased GY, primarily through greater grain number per unit area. SL was also associated with improved grain protein quality, a higher grain N-to-applied N ratio, improved dough rheology, and a marked reduction in free ASN, while KL complemented these responses by modifying starch and protein composition. SL provided the clearest economic benefit, whereas the additional quality improvements associated with KL may increase its value where grain quality premiums are available.

Overall, these findings support stage-specific foliar S and K application as a promising strategy to improve durum wheat productivity and technological quality. The reduction in free ASN represents an additional potential benefit, although its effect on acrylamide formation in processed products remains to be verified. Validation across a wider range of environments, soil nutrient conditions, and genotypes is needed before broader recommendations can be made.

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