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Geotraceability of *Falanghina del Sannio* (South Italy): Preliminary mineralogical and isotopic data

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a r t i c l e i n f o

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A B S T R A C T

The present study gathers mineralogical and isotopic geochemical data of 45 soil samples collected from the *La Guardiense* and *Cantina di Solopaca* wineries (Southern Italy) in order to provide useful information for the geotraceability of *Falanghina* white wine *terroir*. Mineralogical analyses show the ubiquitous presence of quartz, frequent and moderate contents of feldspars and phyllosilicates (i.e., illite/mica, kaolinite, and smectite). Rhizosphere soils from *La Guardiense* vineyards show a strontium isotope signature $(^{87}Sr)^{86}Sr$) ranging from 0.708604 to 0.711234 (average 0.710334; $1\sigma = 0.000943$; $n = 7$) for the total fraction and a narrower range (from 0.706907 to 0.708807; average 0.708120; $1\sigma = 0.000641$; $n = 7$) for the bioavailable one. For the rhizosphere soils collected from the vineyards of *Cantina di Solopaca* winery, 87Sr/86Sr ratios range from 0.708268 to 0.712413 (average 0.710145; $1\sigma = 0.001622$; *n* = 8) and from 0.707968 to 0.709157 (average 0.708384; $1\sigma = 0.000409$; $n = 8$) for the total and the bioavailable frac-

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tion, respectively. Actually, the bioavailable Sr fraction shows a narrower range of $87Sr/86Sr$ ratios compared to that of the total fraction, making the former more suitable for any study of wine traceability.

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Specifications Table

1. Value of the Data

- This article provides mineralogical data of soils characterizing some Italian vineyards producing *Falanghina del Sannio DOCG* white wines.
- Oenologists, pedologists, wineries and other stakeholders can benefit of this study for a more sustainable use of soils improving the cultivation of vineyards.
- These data represent a pivotal approach for a proper *Falanghina* white wine *terroir* geotraceability.

2. Background

The traceability of a wine has now become a multidisciplinary topic and the winemakers themselves attempt to frame the phenomenon from different perspectives. Apparently, the geological viewpoint is the one that embraces the whole physical connotations of the *terroir* system thus providing more than just a feeling. Much data is available today to understand the link between a wine and the *terroir* from which it originates [\[1–4\]](#page-9-0). Radiogenic isotope ratios such as $87Sr/86Sr$ are candidates to become excellent geo-tracers suggesting the origin of a wine. They will never carry genetic and/or plant-specific information, but they can guarantee, according to the countless studies so far available, the answer that consumers have been asking producers for a long time: are we sure that the product comes from the area indicated on the label?

The data collected come from an Italian *terroir*, located in the Samnite area of the Campania region, and concern a widespread and famous cultivar: the *Falanghina*. The multidisciplinary

Fig. 1. The seven soil profiles examined from the vineyards of *La Guardiense* winery.

approach adopted allowed to obtain a very large dataset such to be considered as the starting point for the interpretation of the phenomenon of migration of strontium with its isotope ratio from the substrate to the final products, in this case the white wine *Falanghina del Sannio DOCG.*¹

3. Data Description

The 45 soil samples here analyzed were collected from the *La Guardiense* and *Cantina di Solopaca* wineries and underwent a multi-analytical approach (Table S1). *La Guardiense* vineyards are located in the municipalities of Guardia Sanframondi and Castelvenere towns (Benevento, Italy). Seven soil profiles (Fig. 1) were excavated and described in seven different vineyards (V1 through V7). Samples taken from different soil horizons (total No. 23) of each profile studied were accurately described (Table S2). In addition to the soil sampled in each single horizon, bulk samples (V1-B through V7-B), referred to soil taken at a depth of 20–30 cm and showing the absence of vegetative apparatus in the areas surrounding the sampling point, and rhizosphere soil samples (V1-R through V7-R), taken at a maximum depth of 40 cm and close to the root systems of the vines were also analyzed. The *Cantina di Solopaca* vineyards are located in the municipalities of Solopaca and Castelvenere towns (Benevento, Italy). Eight rhizospheric

 $¹$ In the Italian classification system of wine, DOCG "Denominazione di origine controllata e garantita" literally means</sup> controlled and guaranteed designation of origin. Both DOC and DOCG wine classifications are described by European Union law as Protected Designations of Origin (PDO).

Fig. 2. The eight rhizosphere soils examined from the vineyards of *Cantine di Solopaca* winery.

Fig. 3. Representative FTIR spectra of soil sample from *La Guardiense* (V2-R) and *Cantine di Solopaca* (M7-S) wineries.

soils (M1-S through M8-S) from different vineyards were investigated for this winery (Table S1, Fig. 2).

3.1. Mineralogical composition

A preliminary Fourier Transform Infrared (FTIR) spectroscopic analysis of all the examined soils (Table S3, Fig. 3) show the typical absorption bands attributable to the presence of both

Fig. 4. Representative XRPD patterns of a soil profile (V4) from *La Guardiense* winery.

anhydrous and hydrated silicate phases (ca. 3397, 1637, 1002, 641–425 cm⁻¹). Among these, it is possible to distinguish the presence of quartz (ca. 796, 778 and 691 cm−1), with mixtures of phyllosilicate phases such as illite and kaolinite (ca. 3691, 3619, 912, 749 cm−1) [\[5,6\]](#page-9-0). Absorption bands at ca. 1796, 1423, 873, 711 cm⁻¹ are diagnostic for calcium carbonate (i.e., calcite) [\[7\]](#page-9-0). The intensity of these bands is moderate in V4-Ap2, V4-B, M7-S, M4-S and all V5 soil samples, whereas it is particularly strong in V4-Bw, V7–2Ck and V7-B. In the remaining soil samples, calcite is rare or absent. Organic matter can be hardly observed (very weak absorption bands around 2921 and 2856 cm−1) [\[8,9\]](#page-9-0) and mainly occurs in the topsoil samples (bulk and/or rhizosphere ones) and uppermost soil horizons.

X-ray powder diffraction (XRPD) analyses revealed the ubiquitous presence of quartz in all examined soil samples along with frequent and fair contents of feldspars, mainly sanidine and plagioclase (Table S4, Fig. 4). Occurrence of phyllosilicates such as illite/mica, kaolinite, and smectite (e.g., montmorillonite) have been highlighted as well, along with halloysite and vermiculite in V6 soil profile. Traces of phyllosilicates were noticed in samples belonging to the M-series. The presence of carbonate phases is limited to calcite, with quite variable contents as assessed by quali-quantitative evaluations. Samples V6–2CB, M3-S and M6-S clearly show the occurrence of clinopyroxene that was less evident or absent in the remaining soils.

The thermal analyses (thermogravimetry - TG, derivative thermogravimetry - DTG and differential scanning calorimetry - DSC) of soils from *La Guardiense* vineyards allowed the identifica-tion of five thermal ranges (Table S5, [Fig.](#page-5-0) 5). The first one (40–200 \degree C) derives from dehydration processes (endothermic reactions) due to the loss of weakly bound water of hygroscopic materials and/or clay components. The second thermal event (200–380 \degree C) occurs as a partial overlap of residual dehydration processes and the initial decomposition of the organic components (exothermic and subordinately endothermic reactions). In the thermal range between 380 °C and

Fig. 5. Representative TG curves of rhizosphere soils from *La Guardiense* winery.

600 °C an overlap of dehydroxylation phenomena (endothermic reaction) and combustion of the organic substance (exothermic reaction) is observed, sometimes accompanied by the polymorphic α-quartz to β-quartz transition (endothermic reaction at ca. 574 °C). Decomposition of the carbonate phases (endothermic reaction) occurs in the thermal range of 600–900 °C. Above this temperature, up to approximately 1050 °C residual dehydroxylation, combined with polymorphic transitions and sintering phenomena, occasionally occur without significant weight losses.

3.2. Strontium isotope signatures

In Table S6 and in [Fig.](#page-6-0) 6 are reported the $87\text{Sr}/86\text{Sr}$ ratios, referred to both total and bioavailable strontium, for the examined soil samples, along with standard errors. Rhizosphere soils from *La Guardiense* winery (V1-R through V7-R) show a strontium isotope signature ranging from 0.708604 to 0.711234 (average 0.710334; $1\sigma = 0.000943$; $n = 7$) for the total fraction and a narrower range (from 0.706907 to 0.708807; average 0.708120; $1\sigma = 0.000641$; $n = 7$) for the bioavailable one. For the rhizosphere soils collected from the vineyards of *Cantina di Solopaca* winery (M1-S through M8-S), ${}^{87}Sr/{}^{86}Sr$ ratios range from 0.708269 to 0.712413 (average 0.710145; $1\sigma = 0.001622$; $n = 8$) and from 0.707968 to 0.709157 (average 0.708384; $1\sigma = 0.000409$; $n = 8$) for the total and the bioavailable fraction, respectively. The average value of the 87Sr/86Sr ratios determined for bulk soils of *La Guardiense* vineyards is slightly higher than those observed for the total strontium fraction of rhizosphere soils, with a value range significantly wider (from 0.708240 to 0.715196; average 0.710774; $1\sigma = 0.002176$; $n = 8$). ⁸⁷Sr/⁸⁶Sr ratios of soil horizons (total fraction) significantly change for each single horizon, generally displaying variable average values: $V1 = 0.710053$; $V2 = 0.714649$; $V3 = 0.712869$; $V4 = 0.708949$; $V5 = 0.710990$; $V6 = 0.709411$; $V7 = 0.708043$. These strontium signatures are systematically higher than ⁸⁷Sr/⁸⁶Sr ratios obtained for the bioavailable fraction (V1 = 0.708304; $V2 = 0.708828$; $V3 = 0.708578$; $V4 = 0.708019$; $V5 = 0.708380$; $V6 = 0.708658$; $V7 = 0.707300$). Furthermore, the value range of the average ${}^{87}Sr/{}^{86}Sr$ ratio for the bioavailable fraction (0.707300 - 0.708828) is much narrower than the range obtained for the total fraction (0.708043 - 0.714649).

Fig. 6. Strontium isotope ratios of the examined soil samples. The analytical uncertainty (2 x standard error) is included in each data symbol.

4. Experimental Design, Materials and Methods

4.1. Characterization of soil samples

Soil samples were collected during the harvesting of wine grapes of the years 2021 and 2023 for *La Guardiense* and *Cantine di Solopaca* wineries, respectively. Sampling procedures and chemical-mineralogical characterizations were carried out according to a protocol widely used in Italy [\[10\]](#page-9-0). A map showing the sampling locations is reported in [Fig.](#page-7-0) 7 (UTM WGS84 coordinates in Table S1). The investigated area belongs to the complex geological and geomorphological context of the Telesina Valley (Campania Region, South Italy), an E-W oriented morphostructural depression filled by Quaternary-aged alluvial fans deposits, interfingered with fluvial and fluviolacustrine sediments of the Calore River [\[11\]](#page-9-0). Middle and Upper Pleistocene pyroclastic layers, i.e., Ignimbrite of Guardia Sanframondi (ca. 560 Kyr B.P.) and Campanian Ignimbrite (ca. 39 Kyr B.P.) [\[12\]](#page-9-0), are interbedded in the alluvial succession.

Collected soil samples have been preliminary air dried and then sieved in order to separate coarse fraction (> 2 mm) from fine-earth fraction. The samples were then powdered using a McCrone micronizing mill (agate cylinders and wet grinding time 10 min). The so-obtained powders have been characterized from a chemical and mineralogical point of view by means of a multi-analytical approach.

All the investigated soil samples underwent Fourier Transform Infrared spectroscopy (FTIR) and simultaneous thermal analyses (STA). FTIR measurements were performed in Attenuated Total Reflectance mode (ATR) by means of a Bruker ALPHA-R spectrometer in the mid-infrared spectral range (4000–400 cm⁻¹, 64 scans, 4 cm⁻¹ resolution).

The qualitative mineralogical composition of soil samples was determined by X-ray Powder Diffraction (XRPD) using a Malvern Panalytical X'Pert Pro diffractometer (CuKα radiation, 40 kV, 40 mA, 3–70° 2θ scanning interval, RTMS detector, 0.017° 2θ equivalent step size, 120 s per step

Fig. 7. Map showing the sampling locations (UTM WGS84 coordinates reported in TableS1).

equivalent counting time). Malvern Panalytical HighScore Plus 3.0d software was used for phase identification, along with PDF-2 and ICSD database.

STA analyses were carried out using a NETZSCH STA 449 F3 Jupiter able to perform at the same time thermogravimetry (TG) and differential scanning calorimetry (DSC). The instrument was coupled by a transfer line heated at 200 \degree C with a FTIR BRUKER Tensor 27 for the Evolved Gas Analysis (EGA). The samples were heated up to 1050 °C, with a heating rate of 10 °C/min in pure air atmosphere (flow rate 60 mL/min). FTIR and thermal data have been processed using Bruker Opus 7.2 and NETZSCH Proteus 6.1 software, respectively.

4.2. Determination of 87Sr/86Sr ratios

Samples for the Sr-isotope ratio were prepared in an ISO 6 - ISO 5 class clean room where the external air is forced through several HEPA filters. For the dissolution, either Suprapur® or Ultrapur® reagents were used, which were diluted with calculated aliquots of Milli-Q® deionized water when needed.

About 0.05 g of each soil sample were dissolved for the isotopic analysis of the total Sr fraction by adding 0.25 mL of $HNO₃$ (65 %) and 1.5 mL of HF (40 %) in a pre-cleaned PTFE vial. The vials were then kept overnight at 100 \degree C on a hot plate underneath a laminar flow hood equipped with HEPA filter. Then, after addition of 0.75 mL of HNO₃ (65 %), the samples were evaporated to dryness on a hot plate at 120 $^{\circ}$ C, in the same laminar flow hood. After adding 2 mL of HCl 6 N, the samples were completely dried at 120 °C. Two cycles of alternating 0.25 mL of H_2O_2 (30 %) and HNO_3 (65 %) were used to remove organic matter. The final dissolution step consisted in addition of 1 mL of HCl 6 N, and heating at 120 \degree C on hot plate allowing for complete dryness. After, the samples were re-taken in 1 mL of HCl 2.5 N and centrifuged at 4500 rpm for 10 min. Strontium was extracted from the liquid matrix through chromatography,

loading 0.5 mL of sample solution on quartz columns filled with 2 mL of cation exchange resin Dowex AG 50 W X-8 (200–400 mesh), in accordance with the methodology described in [\[13\]](#page-9-0).

Approximately 2 g of soil samples were weighted to acquire the Sr-isotope ratio of the bioavailable Sr fraction. After addition of 15 mL of 1 M ammonium acetate (CH₃COONH₄), samples were left for eight hours at room temperature. Using a sterile syringe and 0.45 μ m filter system to filter the solution, samples were dried on a hot plate at 50 \degree C. The soil residues were treated with an extra 15 mL of 1 M ammonium acetate and left at room temperature for additional eight hours. Following another filtering of the solution using the syringe and filter system, samples were dried again at 50 \degree C. To the filtered and dried solution, 3 mL of 3 M HNO₃ was added, then the beaker was placed in an ultrasonic bath for ten minutes. Once transferred to clean and sealed tubes, the samples were centrifuged for ten minutes at 4500 rpm.

To analyze the bioavailable Sr fraction in soil samples, strontium was separated through liquid chromatography using pre-cleaned PTFE columns filled with 0.25 mL of Eichrom Sr-spec cation exchange resin (50–100 mesh) following the protocol for ${}^{87}Sr/{}^{86}Sr$ analysis of geological and biological samples proposed by [\[14\]](#page-10-0).

Eventually, the Sr-bearing eluted solutions were dried on a hot plate under a laminar flow hood. A final step of nitrification was achieved by adding 2–3 drops of concentrated HNO₃. ⁸⁷Sr/⁸⁶Sr ratios were determined using a ThermoScientific® Triton Plus Thermal Ionization Mass Spectrometer (TIMS) equipped with 9 Faraday cups, one fixed in central position and eight movables. During the data collection process, the 2σ (σ = standard deviation) was assessed by replicating the analysis of the NIST SRM 987 international standard following the method proposed by [\[15\]](#page-10-0). Consequently, the ${}^{87}Sr/{}^{86}Sr$ ratios of the samples were adjusted to match the recommended value for NIST SRM 987 (i.e. ${}^{87}Sr/{}^{86}Sr = 0.710248$) [\[16\]](#page-10-0). Each analysis cycle involved acquiring a total of 150 measurements of varying Sr isotope ratios involving all four Sr isotopes besides $87Rb$ to check for mass interference to $87Sr$. To account for in run mass fractionation, the 87 Sr/ 86 Sr ratio was corrected by normalizing the 88 Sr/ 86 Sr ratio to its natural value of 8.37521 through an exponential law.

Limitations

The determination of $87\text{Sr}/86\text{Sr}$ in the investigated samples can be theoretically limited by the available amount of Sr. First, the preparation of a sample with a small amount of Sr (e.g., less than 1 ppm) might be affected by the contamination level of the laboratory (Sr blank) resulting in a $87Sr/86Sr$ measured value different from the original one. However, in such cases, a correction can be made taking into account the blank amount and its $87Sr/86Sr$ value. Second, in extreme cases, the amount of Sr available can be not enough for a complete mass spectrometry run, precluding a precise determination of the 87 Sr $/86$ Sr value.

Ethics Statement

The authors have read and follow the ethical requirements for publication in Data in Brief and confirm that the current work does not involve human subjects, animal experiments, or any data collected from social media platforms.

Data Availability

Supplementary materials for the Data [Article "Geotraceability](https://data.mendeley.com/datasets/yr6pm33ttj/1) of Falanghina del Sannio (South Italy): Preliminary mineralogical and isotopic data" (Original data) (Mendeley Data).

CRediT Author Statement

Mariano Mercurio: Conceptualization, Methodology, Writing – original draft, Supervision, Data curation; **Piergiulio Cappelletti:** Writing – review & editing, Investigation, Formal analysis; **Valeria Di Renzo:** Writing – review & editing, Investigation, Visualization; **Celestino Grifa:** Writing – review & editing, Investigation, Formal analysis; **Carmine Guarino:** Writing – review & editing, Project administration, Funding acquisition, Resources; **Francesco Izzo:** Conceptualization, Writing – original draft, Investigation, Visualization, Formal analysis, Data curation; **Paolo Magliulo:** Writing – review & editing, Investigation, Visualization; **Vincenzo Mercurio:** Writing – review & editing, Conceptualization, Resources; **Piergiorgio Tranfa:** Writing – original draft, Investigation, Visualization; **Daniela Zuzolo:** Writing – review & editing, Investigation, Visualization; **Massimo D'Antonio:** Investigation, Supervision, Validation, Data curation, Writing – original draft; **Alessio Langella:** Conceptualization, Methodology, Writing – original draft, Supervision, Data curation.

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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.

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