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Christian Coelho, Abdelmouhcine Gannoun, Charley Merciecca, Ivan Vlastelic, Christophe Poix, et al.. A 2-year exploratory study to elaborate a methodological protocol for evaluating volcanic terroir influence in Gamay wines in the French PDO Côtes d'Auvergne. *Journal of Food Composition and Analysis*, 2025, 139, pp.107115. 10.1016/j.jfca.2024.107115 . hal-04904952

**HAL Id: hal-04904952**

**<https://hal.science/hal-04904952v1>**

Submitted on 22 Jan 2025

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## A 2-year exploratory study to elaborate a methodological protocol for evaluating volcanic terroir influence in Gamay wines in the French PDO Côtes d'Auvergne

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### ARTICLE INFO

#### Keywords:

Volcanic vineyards  
Soils  
Chemical composition  
Gamay wines  
Sensory perception

### ABSTRACT

The objective of our study is to report a methodology used to determine chemical and sensory attributes of wines that were elaborated from vineyards growing on volcanic-derived soils in the French PDO Côtes d'Auvergne. For that purpose, vineyard soils were sampled in eight different geographical locations and analyzed by their granulometry, hydric stress and multi-elemental composition. Red wines were elaborated during vintages 2020 and 2021 and chemically characterized by classical oenological parameters, anthocyanins and multi-elemental analysis by ICP-MS, and sensorially described by a panel of nineteen experienced tasters. The results show that vineyards could be differentiated into two typologies of soils: four clay-limestone soils (A) and four basaltic soils (B). B soils presented a more heterogeneous granulometry, a higher water retention and a more concentrated mineral content compared to A soils. Volcanic Gamay wines, originating from these B soils, differed by their red yellowish color, lower total acidity, lower anthocyanin contents, higher contents of sodium, lithium and strontium and stronger peppery/smoky flavors compared to non-volcanic wines originating from A soils. The sensory descriptors were discussed as a function of the chemical composition of wines and could reveal the importance of volcanic terroirs in the sensory and chemical composition of wines.

### 1. Introduction

The concept of “terroir” in wine has gained a lot of interest these last decades for marketing and communication purposes for wine consumers, attached to the sense of wine place production (Alonso González and Parga Dans, 2018; Cross et al., 2017). However such a concept emerged in the 12th century by the “corporation des gourmets” in order to guarantee the trueness of wines elaborated in a delimited place for wine traders (Rigaux, 2018). Wine terroir typicity was embodied in France in the 1930s with the origin certifications and spread to other countries, in Europe with the EU regulation 1308/2013 or the USA with the American viticultural areas (Vincent et al., 2019). The International Organization of Vine and Wine proposed in 2010 to define terroir as the “concept that refers to an area in which collective knowledge of the interactions between the identifiable physical and biological

environment and applied vitivincultural practices develops, providing distinctive characteristics” for wine originating from this area (OIV/VITI 333/2010 resolution). The influence of soil, topography, climate, and cultivar on terroir has been described (van Leeuwen et al., 2004; van Leeuwen et al., 2018; Van Leeuwen and Seguin, 2006). Many scientific studies illustrated in different vineyards all over the world how the expression of terroir could affect chemical and sensory attributes in wine (Kontkanen et al., 2005; Prata-Sena et al., 2018; Roullier-Gall et al., 2014); Cornelis (van Leeuwen, 2022; van Leeuwen et al., 2020). All these studies highlighted that for such purpose, investigators should focus their study on identifying quantifiable variables by carrying a multidisciplinary approach. Revealing terroir expression generally requires knowledge of volatile and non-volatile composition that can be found in vine vegetative material, grapes, musts and wines (Arapitsas et al., 2020; Cugnetto et al., 2014), and crossed with unique sensory

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<https://doi.org/10.1016/j.jfca.2024.107115>

Received 22 September 2024; Received in revised form 10 December 2024; Accepted 11 December 2024

Available online 13 December 2024

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attributes gathered in specific area of wine production (Bauer et al., 2011; Douglas et al., 2001). These last decades, the analysis of multi-elemental and stable isotope composition of vineyard soils and monovarietal grapes highly increased our knowledge of mineral nutrition of vines and biomolecular accumulation in grapes and wines as a function of different soil provenance (Blotevogel et al., 2019; Di Paola-Naranjo et al., 2011; Vinciguerra et al., 2016).

Among the viticultural terroirs present in the world that have until now been poorly investigated, volcanic terroirs could confer specific tasting attributes according to wine-tasting professionals (Szabo, 2016). When present in active volcanic areas, humans have adapted vine growing practices over time, constituting a clear testimony of terroir and cultural construction (Acosta and Ferrer, 2014; Frankel, 2014; Szabo, 2016). However, very little evidence has been provided by the scientific community to explain the transfers from volcanic soils to wines (De Lima et al., 2003). Nevertheless, geographical cultivar typicity and geographical authentication of volcanic vineyards have been successfully proposed (Bellanca et al., 2006; Ferioli et al., 2010; Fort et al., 2022; Gonzalez et al., 2021). In this respect, the objectives of our study were to pave the way for understanding the chemical composition and sensory characteristics of volcanic wines. To achieve this goal, we carried out a two-year experimental study in the old volcanic area of Massif Central (France) presenting vineyard soils derived from ancient eruptive and lava flow sites dating from the Miocene geological period. For the study, two typologies of vineyards (volcanic vs non-volcanic) implanted on the PDO Côtes d'Auvergne with Gamay cultivar were analyzed from soils to grapes and wines. All wines were chemically and sensorially analyzed. Descriptive sensory analysis was carried out by a panel of nineteen experienced tasters.

## 2. Materials and methods

### 2.1. Grape variety

The research focused on the red grape variety Gamay. This cultivar has been characterized by conventional phenotyping and genotyping analysis (Maul et al., 2012). Gamay is one of the top ten most planted

cultivars in France with 23187 ha (FranceAgriMer, 2020) and dominates the Auvergne Rhône Alpes vineyards, particularly in the PDO areas like Beaujolais, Côtes du Forez and Côtes d'Auvergne.

### 2.2. Study area

The vineyards for the study were localized in the PDO Côtes d'Auvergne. Four geographical sectors spreading from south to north of the appellation were studied: Boudes (DP), Neschers (DM), Gergovie (DTP) and La Roche Noire (DSV). In each sector, two vineyard typologies (A and B) were chosen for their presence of Gamay grape variety and differentiated soils related to their geological history. Typology A was mainly made of sedimentary clay-limestone soils whereas typology B were soils derived from basaltic rocks deposited from successive eruptive activities in the tertiary and quaternary periods. In Fig. 1, the studied vineyards were reported on the geological cartography support (scale 1:100000) from the pedological map of Val d'Allier (Massif Central, France). GPS coordinates and altitude of the studied vineyards are reported in Table S.I.1.

### 2.3. Climatic conditions

The mean precipitation and the average monthly temperatures during the two vintages of study (2020 & 2021) were calculated from data recorded and compiled by the Aulnat local meteorological station (Météo-France, 2022). Vintage 2020 presented a higher mean annual temperature of 13.6 °C compared to 12.2 °C for vintage 2021 and a lower total annual rainfall of 474 mm compared to 614 mm for vintage 2021 (Figure S.I.1).

### 2.4. Soils characteristics

All vineyard soils were sampled at 30 cm depth with an auger. The soil texture was determined using the standard methodology (Soil-Survey-Staff, 1998) and classified according to the international soil classification system (USDA-NRCS, 1999). Soil samples were collected using a stainless-steel shovel at 0–30 cm depth intervals

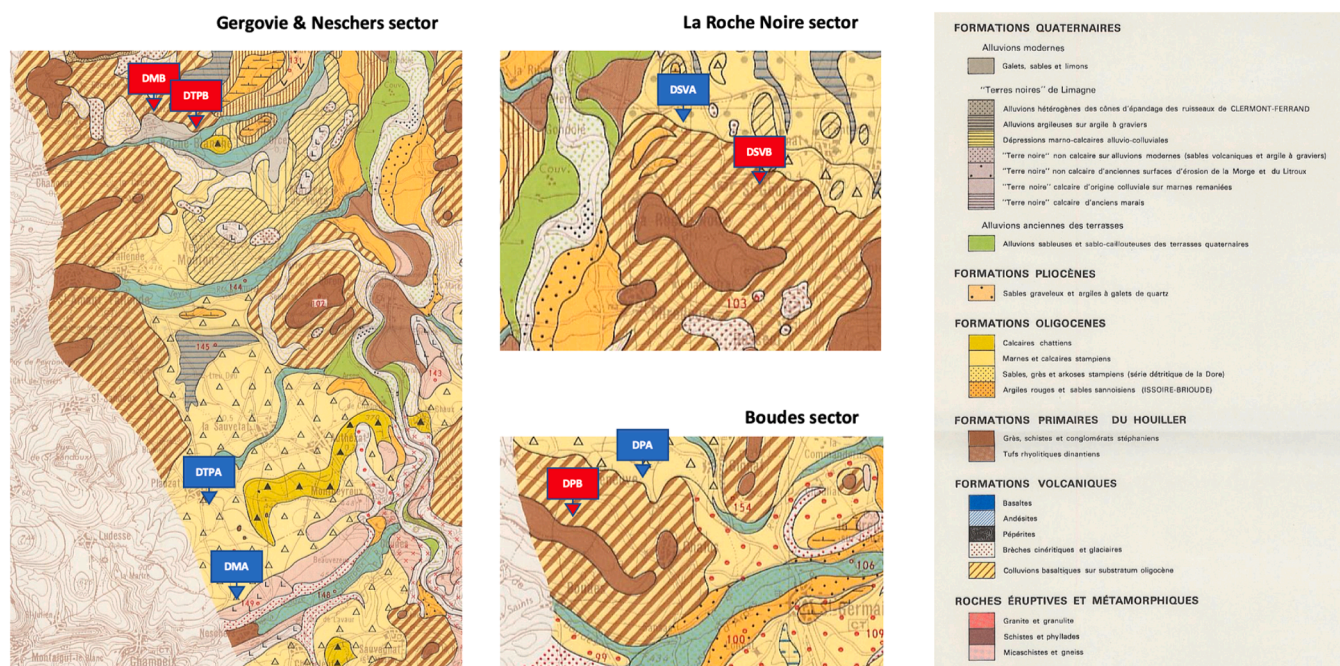


Fig. 1. Pedological map representing the eight vineyard locations in the PDO Côtes d'Auvergne. DMA, DTPA, DPA and DSVA are present on old marly-calcareous Oligocene formations, whereas DMB, DTPB, DPB and DSVB are present on basaltic colluvial deposits originating from volcanic formations that occurred during the Miocene period.



(Givelet et al., 2004) and placed in clean plastic bags. The soil samples were dried in an oven at 105 °C, gently crushed, sieved (0.5 mm) and homogenized. They were calcined by heating at 600 °C for 6 h to eliminate organic matter and then finely crushed before chemical treatment. About 100 mg of soil powder was weighed on a piece of weighing paper, and gently poured into a 15 mL screw cap savillex PFA vessel (Savillex, Eden Prairie, MN, USA) containing 1 mL of 7 M HNO<sub>3</sub>, then dissolved with 2 mL of 29 M HF at 110 °C. Reagent-grade hydrofluoric, nitric, and hydrochloric acids (Fluka, Seelze, Germany) were purified by subboiling distillation in PFA DST-100 systems (Savillex, Eden Prairie, MN, USA). The solution was evaporated to dryness, and then treated several times with a mixture of 6 M HCl and 14 M HNO<sub>3</sub>. After evaporation, the residue was treated again with conc. HNO<sub>3</sub>. Then, the solid residue is taken up with 2 mL of 1 M HNO<sub>3</sub>, and centrifuged, in order to check for the presence of solid residues, especially sparingly soluble fluorides. Any whitish residue observed at that stage was treated with a few hundred µl of a 1 M HNO<sub>3</sub> solution saturated in boric acid (Pin and Gannoun, 2019); this generally ensures a nearly complete dissolution. Then, the resulting solution was combined with the bulk sample solution. Weighted aliquots (i.e. ~ 5 %) were taken and diluted in 0.5 M HNO<sub>3</sub> – 0.05 M HF acid mixture, reaching a dilution factor of about 3500. All trace element measurements were performed using an LMV Agilent 8900 ICP-MS. Counts per second were calibrated using several synthetic solutions (Inorganic Ventures Mix Standard, CMS) ranging from 0.01 to 100 ppb (Inorganic Ventures' high-quality certified reference materials, Christiansburg, Virginia, USA). Calibration curve equations are given in Table S.I.2. The certified rock standard BHVO-2 and BIR-1 were also analyzed as external standards.

## 2.5. Grape technological maturity and vine water status

Gamay grapes were picked up in each of the eight studied vineyards and pneumatically pressed for quality control analysis of technological maturity parameters by OenoFoss™ (Foss, Nanterre, France). Water deficit stress was evaluated in grape musts at maturity by stable isotope ratio in carbon ( $\delta^{13}\text{C}$ ) (Gaudillère et al., 2001; Ramos, 2017); Cornelis (van Leeuwen et al., 2009). Carbon isotopic analyses were performed on a Vario Micro Cube elemental analyzer coupled in a continuous flow mode to an isotope ratio mass spectrometer (IsoPrime, Elementar, Lyon, France). USGS40 L-Glutamic acid ( $\delta^{13}\text{C}_{\text{VPDB}} = -26.39 \pm 0.04 \text{ ‰}$ ) and IAEA-600 Caffeine ( $\delta^{13}\text{C}_{\text{VPDB}} = -27.77 \pm 0.04 \text{ ‰}$ ) were used as internal standards. The  $\delta^{13}\text{C}$  values were reported in parts per thousand (‰) relative to the Vienna Pee Dee Belemnite (VPDB) international reference. Incertitude of  $\delta^{13}\text{C}$  measurements was given at  $\pm 0.15 \text{ ‰}$ .

## 2.6. Winemaking steps

During vintage 2020, red wines were elaborated separately in each sector by four different winemakers who applied their usual winemaking practices on Gamay grapes harvested from their limestone and basaltic vineyards. During vintage 2021, Gamay grapes from the eight studied vineyards were harvested and brought to a single winemaker in Châteaugay (Heritage Volcanic, Châteaugay, France) who carried standardized microvinifications in 1 hL thermoregulated stainless-steel tanks. This process involved identical maceration times (10 days) at a constant temperature of 20 °C, and with the same amount of inoculated alcoholic yeast at 20 g/hL (SafEno™ NDA21, Fermentis, Marquette-lez-Lille, France) and lactic acid bacteria at 1 g/hL (Vitalactic® F, Martin Vialatte, Magenta, France). These inoculated microorganisms were recommended for red varieties and conferred to wines an excellent general balance. The resulting wines from each vintage were aged for six months in inert stainless-steel tanks prior to filtration and bottling for chemical and sensory analysis. The implantation of *S. cerevisiae* strain on wine microvinifications was controlled by PCR interdelta analysis (Legras and Karst, 2003). The interdelta profiles indicate identical amplified sequences between inoculated batches and the yeast strain

(Figure S.I.2).

## 2.7. Wine multi-elemental composition

Wine samples from different stages of winemaking protocol were stored in corning Falcon 15 mL conical centrifuge tubes (polypropylene, VWR) and were stored in the freezer at –20 °C. About 4 g of wine sample was weighted into a 15 mL screw cap PFA vessel and gently dried down at 60 °C for 12 h. The organic matter in wine samples was dissolved by adding 2 mL of aqua regia (3:1 HCl/HNO<sub>3</sub>) and 1 mL of ultrapure hydrogen peroxide (Sigma-Aldrich, Saint Louis, USA) for 24 h. Samples were then evaporated to dryness. This sequence was repeated until complete oxidation of the organic matter was attained (Vinciguerra et al., 2016). Samples were taken and diluted in 0.5 M HNO<sub>3</sub> – 0.05 M HF mixture with a dilution factor of about 20. All trace elements measurements were performed using a LMV Agilent 8900 ICP-MS. Counts per second were calibrated using several synthetic solutions (CMS) ranging from 0.01 to 100 ppb. Calibration curve equations are given in Table S.I.2.

## 2.8. Oenological parameters

Wine classical oenological parameters (pH, ethanol, total acidity, tartaric acid, malic acid, lactic acid, volatile acidity, TPI) were analyzed by Lyza 5000Wine® (Anton Paar, Graz, Austria) using a constructor calibrated Fourier transform infrared wine model. Free anthocyanins (ANT) were analyzed by sodium bisulfite discoloration. Total tannins (TAN) were analyzed by acidic hydrolysis according to the Bate-Smith method. Wine color measurements using the CIELab color space, L\* a\* and b\* were recorded on a Konica Minolta CM-5 spectrophotometer (Chiyoda, Japan) with a 1 mm path length optical glass cell. Wine anthocyanin compounds were separated, identified, and quantified on a UHPLC Ultimate 3000 (ThermoFisher, Carlsbad, CA, USA) equipped with a Q-Exactive™ Orbitrap mass spectrometer (ThermoFisher, Sunnyvale, CA, USA) operating in electrospray ionization positive mode (ESI+) and negative mode (ESI-). Red wines were filtered on 0.2 µm polyester membranes (Machery-Nagel, Düren, Germany) and diluted 10 times in methanol prior to injection (5 µL) and separation on a reversed-phase column Zorbax Eclipse XDB-C18 (4.6 × 250 mm; 5 µm particle; Agilent), thermostated at 30 °C. The MS parameters were as follows: dry gas N<sub>2</sub> at 10 mL/min; drying temperature at 350 °C; nebulizer at 65 psi; capillary at + 3.5 kV (positive ion mode) and – 3.5 kV (negative ion mode); target mass at *m/z* 600; scan range from *m/z* 50–1200. The chromatographic elution of wine samples was carried using two solvents: A (water/methanol/formic acid, at 95/4.5/0.5, v/v/v) and B (methanol) at a flow rate of 0.25 mL/min. A gradient elution was performed from 0–10 min, 5 % B; 10–20 min, 5–40 % B; 20–45 min, 40–90 % B; and 45–50 min, 90 % B. Water was first deionized with conventional ion-exchange resins and then further purified to a resistivity of 18.2 MΩ cm with an Aqua System Distribution (Bondoufle, France) device. Methanol (LC-MS grade) and formic acid (>99.5 %) were purchased from Fisher Scientific (NJ, United States). Pure (>98 %) anthocyanin standard compounds (Extrasynthèse, Genay, France) were used for external calibration from 0.1 to 10 mg/L and quantification in red wines according to the calibration curve equations (Table S.I.2).

## 2.9. Wine sensory analysis

The tasting panel consisted of 19 experienced tasters (14 men, and 5 women). The panel was composed of twelve French and seven Canadian experienced tasters. Among them ten were wine professionals, six were winemakers in France and Canada (mixing volcanic and non-volcanic areas) and three were master sommeliers. All panelists had more than 10 years of experience in wine tasting. All wines were tasted by the entire panel knowing that wines could come from volcanic and non-volcanic terroirs, but without any information when glasses were

presented to them during the sensory session. Two annual quantitative descriptive analysis sensory sessions were organized in May 2021 (vintage 2020) and May 2022 (vintage 2021).

The panelists were asked to rate the intensity (from a scale of 0–10) of the red color tint (from yellow to blue) and of flavor attributes (from low to elevated perception for volatile acidity, global acidity, salinity, astringency, lactic, peppery and smoky notes - only for vintage 2021). All wine descriptors were identified from chemical parameters differentiating the two typologies of wines (A vs B) and from preliminary wine tasting before the sensory session. Twenty milliliters of wine samples were served at room temperature in isolated booths. All wine bottles were coded prior to the sensory session and tasters were asked to rinse their glass between each service. The intensity of each individual wine attribute was evaluated by all the sensory panelists and filled on a Woodclap® page.

A second test of quantitative descriptive analysis with a free choice profiling exercise was also carried out for each tasted wine where all the panelists systematically gave a free description for each of the analyzed wines. The free descriptors used by the panel were lemmatized for each wine. The vocabulary terms associated with sensory perceptions were grouped into one descriptor. As an example, the terms cranberry, blueberry, blackcurrant, and red berries were associated with the fruity descriptor. A general representation using a word cloud gathering the sensory descriptors, cited at minimum two times by the panel, of volcanic wines versus non-volcanic wines, is shown in [supplementary information \(Figure S.I.3\)](#).

## 2.10. Statistical analysis

All measurements were carried out in technical triplicate. Results were analyzed by a one-way analysis of variance (ANOVA) and Fisher's least significant different tests, using a p-value < 0.05. To evaluate the chemical composition of the two typologies of soils and the resulting wines, regardless of vintage, principal component analysis was carried out using the chemical parameters concentration as loadings and soils or wines as variables.

For the two studied vintages, each wine was characterized by a set of 45 chemical descriptors and 8 sensory descriptors, representing a matrix of 16 wine samples (53 x 16 wine samples = 848 points). To evaluate the relationships between chemical and sensory parameters a multifactorial analysis was performed on the entire data set. Different Partial Least Square (PLS) models were also carried out on the eight sensory attributes, as the predicted variables using the 45 chemical descriptors, as the observable variables. The multivariate analyses were performed on OriginPro 9.0 software (Originlab, Massachusetts, USA).

## 3. Results and discussion

### 3.1. Pedological and geochemical attributes of volcanic soils in the PDO Côtes D'Auvergne

As shown in [Fig. 2](#), the analyzed vineyard soils from the PDO Côtes d'Auvergne presented a clear separation in terms of granulometric and geochemical composition. Soils A contain a dominant clayed material (from 47 % to 61 %) and low content of silty material (from 22 % to 32 %) and sandy (from 7 % to 29 %) materials. Volcanic soils B present a more heterogeneous granulometry compared to soils A with dominating clayed materials for DPB (60 %) and DSVB (52 %), sandy materials for DMB (41 %) and silty materials for DTPB (40 %). DTPB and DMB soils presented the highest values of silty materials among the entire set of analyzed soils (from 33 % to 41 %), revealing specific weathering conditions that occurred during the pedogenesis of such volcanic soils ([Mason and Moore, 1982](#); [Wilson, 2004](#)). As illustrated in [Fig. 2.B](#), the distribution of the bioavailable elements in all the studied vineyard soils enables description of 79 % of the variability along the first two axes of the PCA. Volcanic soils B are clearly separated from clay-limestone soils

A. Among the twenty analyzed elements, in [Fig. 2.C](#), lithium and strontium were the only elements to be statistically more abundant in the volcanic soils (170–305 mg/kg for Li and 531–1156 mg/kg for Sr) compared to soils A (120–145 mg/kg for Li and 356–534 mg/kg for Sr). The multielement mean values analyzed on the different vineyard soils are presented in [Table S.I.3](#). The volcanic soils (DMB and DTPB) presented the highest content of lithium and strontium among all the soils that were analyzed. Such bioavailable elements could be more easily leachable from silty materials than sandy or clayed materials. It was already reported that basalt bedrock weathering is an important source of strontium to soils found on volcanic areas ([Chadwick et al., 2009](#)). Titanium was the dominating bioavailable element found in all the vineyard soils without a geological differentiation, but rather a geographical discrimination. La Roche Noire sector presented the more elevated content of titanium, regardless of the typology of the soils (9755 mg/kg for DSVB and 13819 mg/kg for DSVB). The lowest titanium content in soils was found for DTPA (2853 mg/kg).

### 3.2. Hydric status of volcanic vineyards

The stable isotope ratio ( $\delta^{13}\text{C}$ ) was measured in grape musts at maturity. As shown in [Fig. 3](#), for the two typologies of vineyards (A and B),  $\delta^{13}\text{C}$  values ranged from  $-26.50\text{‰}$  (DPA in 2020) to  $-30.55\text{‰}$  (DPB in 2021).  $\delta^{13}\text{C}$  values were systematically found below  $-26.2\text{‰}$  for the two consecutive vintages, indicating that overall studied vineyards presented no water stress conditions. Vintage 2020 presented higher values of  $\delta^{13}\text{C}$  compared to vintage 2021, regardless of the typology of the vineyard, confirming that  $\delta^{13}\text{C}$  is a good proxy of climatic conditions and water uptake by vines, as the annual precipitation was higher in 2021 compared to 2020 ([Figure S.I.1](#)). Interestingly, regardless of the vintage, the volcanic vineyards presented statistically lower mean values of  $\delta^{13}\text{C}$ :  $-28.15\text{‰}$  in 2020 and  $-29.52\text{‰}$  in 2021 compared to vineyards on sedimentary clay-limestone soils:  $-27.15\text{‰}$  in 2020 and  $-28.30\text{‰}$  in 2021. In our studies, vines growing in ancient volcanic vineyards presented a higher water availability from their growing soils compared to non-volcanic soils. These ancient volcanic soils were predominated by silty materials, particularly in DTPB and DMB soils, contributing to an elevated plant water availability, as previously reported ([Lazcano et al., 2020](#)). Therefore, these studied volcanic vineyards in the PDO Côtes Auvergne could present a better resilience to climatic changes, particularly in the case of drought periods, compared to non-volcanic vineyards.

### 3.3. Wine chemical composition from volcanic vineyards

Oenological parameters of the Gamay wines presented in [Table S.I.4](#) revealed typical values relative to ethanol content, acidity, color, polyphenols, anthocyanins and tannins. When comparing all the parameters associated with volcanic wines (B) vs non-volcanic wines (A), vintage by vintage, it can be noticed that statistical differences ( $p < 0.05$ ) were obtained in 2020 for pH, volatile acidity, tartaric acid, lactic acid, b parameter and hue. No statistical difference was found for the vintage 2021 for any oenological parameters. Nevertheless, the same tendencies were observed for these two consecutive vintages with a higher pH value, a higher volatile acidity, a higher content of lactic acid, a higher tannin concentration and higher b and hue values for the volcanic wines (B) compared to the non-volcanic wines (A).

The individual anthocyanins in the Gamay red wines were further analysed, as presented in [Table S.I.5](#). The major anthocyanin found was malvidin-3-O-glucoside with a mean concentration ranging from  $14.7 \pm 4.9\text{ mg/L}$  (volcanic soils, vintage 2021) to  $32.6 \pm 13.7\text{ mg/L}$  (non-volcanic soils, vintage 2020). No statistical differences in its content were found between volcanic and non-volcanic wines for the two vintages. Nevertheless, the content of each anthocyanin was higher during the vintage 2020 compared to the vintage 2021, surely associated with a lower water availability in vineyards in 2020 compared to 2021. As

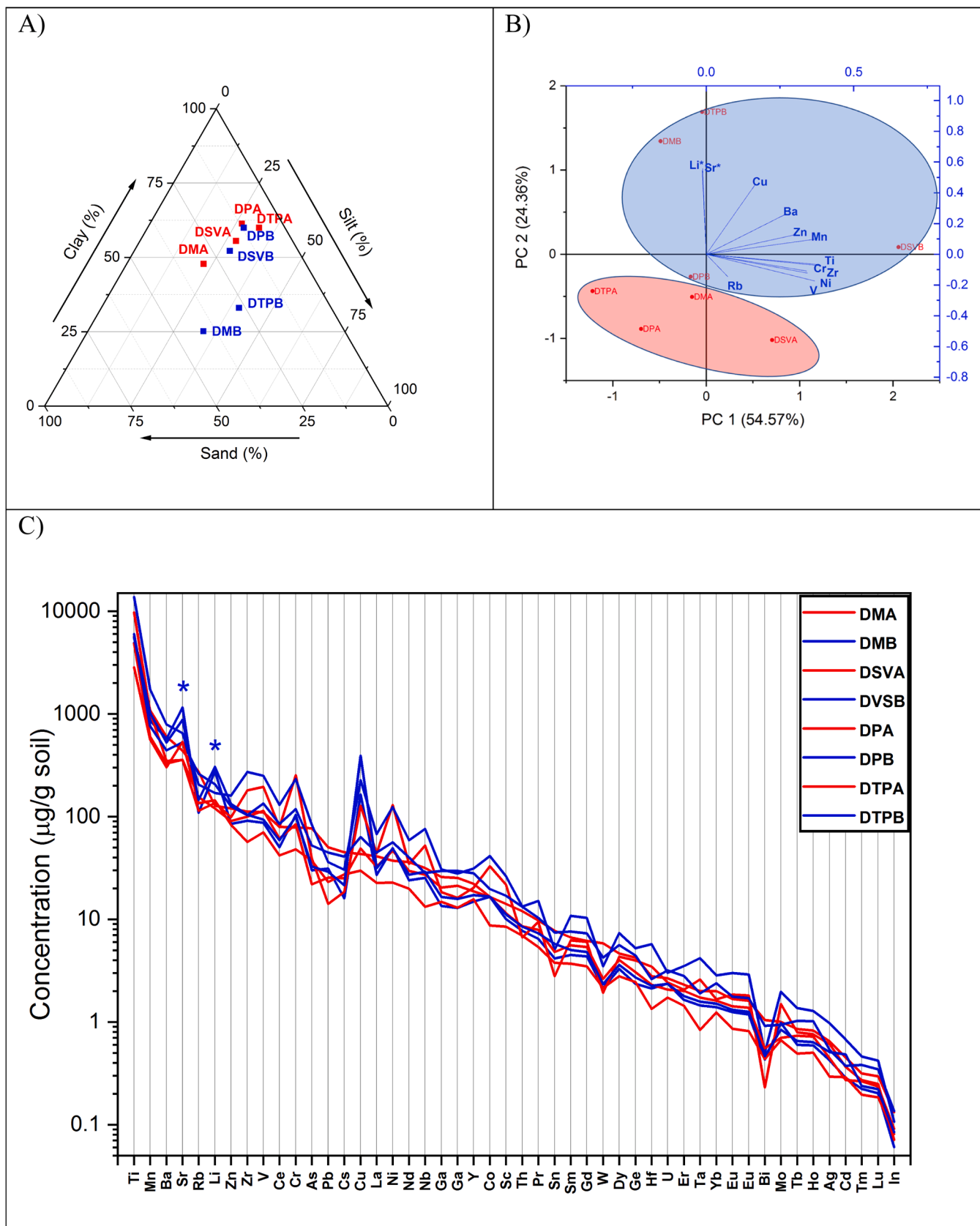


Fig. 2. Granulometry of soils (A); PCA carried out on the 12 major water extractable elements found in soils (B); and multi-elemental analysis of all vineyard soils – Logarithmic scale use for concentration representation. Red for limestone and blue for basaltic (C). The symbol \* means that the average concentrations of the elements concerned are statistically different between the two typologies of vineyards ( $p < 0.05$ ).

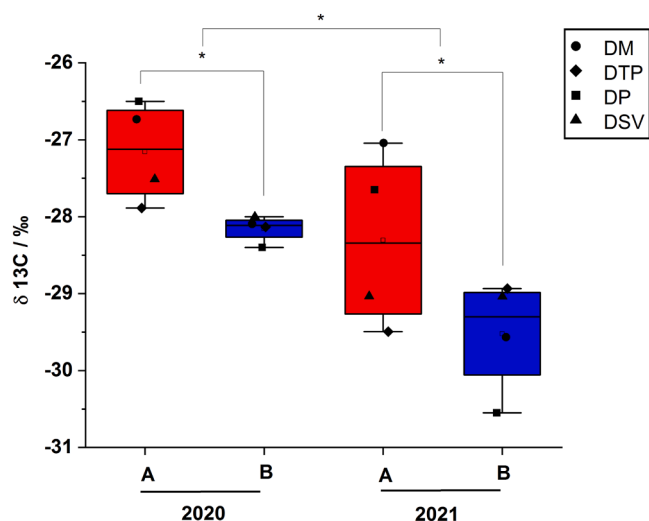


Fig. 3. Histograms representing the stable isotope ratio ( $\delta^{13}\text{C}$ ) values on the two typologies of vineyards (A: non-volcanic and B: volcanic) during the two consecutive vintages (2020 and 2021). The symbols (●, ◆, ■, ▲) represent the four investigated wine sectors: Neschers (DM), Gergovie (DTP), Boudes (DP) and La Roche Noire (DSV).

already shown for red grape varieties, only the early and long-lasting moderate water deficit will enable differentiation of secondary metabolite composition in red wines (Rienth et al., 2021; Savoi et al., 2020). The oenological parameters and individual anthocyanin compound concentrations enabled a global overview of wine characteristics. The differences in the chemical composition between volcanic and non-volcanic wines were used to determine the descriptors to be used for the descriptive sensory analysis.

We further focused our investigation on the inorganic specificities of volcanic wines by analyzing the multi-elemental composition during the two consecutive vintages (2020 and 2021). Fig. 4 depicts the 20 detected elements (among the 27 analyzed that were given in Table S.I.6) in Gamay wines during vintages 2020 and 2021. Among them, magnesium is the most concentrated with contents ranging from 105 mg/kg (DTPA-21) up to 140 mg/kg (DPA-21). Magnesium is, with potassium and calcium (not measured here), the main elements present in wines, accumulating in the solid compartments of grape berries during the maturation grape growing phase (Nistor et al., 2022) and being released

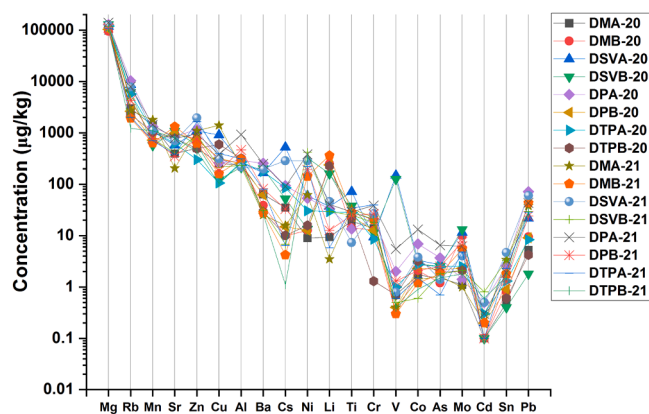


Fig. 4. Multi-elemental analysis of Gamay wines elaborated during vintages 2020 (20) and 2021 (21) from the eight vineyards divided into two typologies (A: non-volcanic and B: volcanic) of four sectors of PDO AOC Côtes d'Auvergne (DM, DSV, DP and DTP) – Logarithmic scale use for concentration representation. Only 20 detected elements were represented among the 27 elements analyzed.

during the maceration phase. Among the 20 elements, four elements (manganese, strontium and lithium in 2020 and zinc in 2021) were statistically different between volcanic and non-volcanic wine in the four studied geographical areas. Even if those elements (Mn, Sr, Li and Zn) appeared to statistically differ in wines elaborated from volcanic and non-volcanic vineyards, it should be emphasized that vintage is an essential driver for the mineral incorporation into grapes and thus in the final composition in wines. No statistical difference was found between the two typologies of wines concerning the other elements analyzed in wines (Table S.I.6). Further analysis on consecutive vintages would be interesting to pursue to draw further conclusions.

#### 3.4. Volcanic wine sensory attributes

The results of the comparative sensory descriptive analysis of Gamay wines are presented in Fig. 5 and Table S.I.7. They reveal a sensorial diversity between the four wine sectors (DTP, DP, DM, DSV) and a difference between the two typologies of soils and the investigated vintages.

To illustrate that point, the red color (taking blue tints) was meanly rated during vintage 2020 from 4.7/10 (DSVB-20) to 7.5/10 (DPA-20), and during vintage 2021 from 4.0/10 (DMB-21) to 7.7/10 (DPA-21). Such parameter was lower in red wines originating from basaltic vineyards compared to clay-limestone for both vintages (except for DSV-21 and DTP-21)). A statistical difference ( $p < 0.05$ ) was found during vintage 2021 for the DP and DM sectors, indicating that the microvinifications carried out during vintage 2021 but not during vintage 2020 enhanced the validity of a yellower red color as a differentiating attribute for volcanic wines from the PDO Côtes d'Auvergne compared to the non-volcanic ones. Such diversity is still present among the flavor wine descriptors.

The global acidity spans, during vintage 2020, from mean values of 4.0/10 (DSVB-20) to 7.6/10 (DMA-20), and during vintage 2021, from 4.9/10 (DPA-21) to 7/10 (DTPA-21). In 2020, red wines from basaltic vineyards presented a lower acidity perception by the panelists compared to wines from clay-limestone vineyards for the four studied sectors (with statistical evidence for DP, DM and DSV). During vintage 2021, such statistically lower acidity perception for wines elaborated from basaltic compared to control vineyards was only present for the DTP sector. For the DP sector, where grapes were harvested at early maturation in 2021, an opposite trend was observed with higher acidity perception for DPB-21 compared to DBPA-21.

The salinity, astringency and lactic characters of wines revealed interesting differences when comparing the typology of vineyards where wines originated, particularly for sectors DM and DTP. Those descriptors were statistically rated to higher values on wines from volcanic vineyards compared to non-volcanic ones for: salinity on DMB-20 (5.9/10) compared to DMA-20 (4.2/10); astringency on DMB-21 (4.9/10) compared to DMA-21 (3.9/10) and lactic on DTPB-20 (5.5/10) compared to DTPA-20 (3.6/10). These descriptors could not at this stage be considered as relevant sensory descriptors of Gamay red wines originating from volcanic vineyards.

Finally, the peppery flavors of Gamay red wines elaborated from volcanic vineyards in the PDO Côtes d'Auvergne were statistically rated higher by the panel compared to wines from non-volcanic vineyards for the DM sector in 2020 (DMB-20 rated at 5.1/10 compared to DMA-20 at 3.3/10) and 2021 (DMB-21 rated at 6.5/10 compared to DMA-21 at 3.7/10), and for the DTP sector in 2021 (DTPB-21 rated at 5.8/10 compared to DTPA-21 at 4.4/10). For smoky flavors, only rated during vintage 2021, three sectors (DTP, DM and DP) presented statistically higher values in wines originating from volcanic vineyards. Curiously, non-volcanic areas from the DSV sector produced wines in 2021 that presented the highest rating for smoky flavor among all sensorially tested wines (DSVA-21 rated at 6.7/10 against DSVB-21 at 3.8/10). The smoky notes in Gamay wines from volcanic vineyards could be more complex to understand than the sole expression of volcanism in wines and require



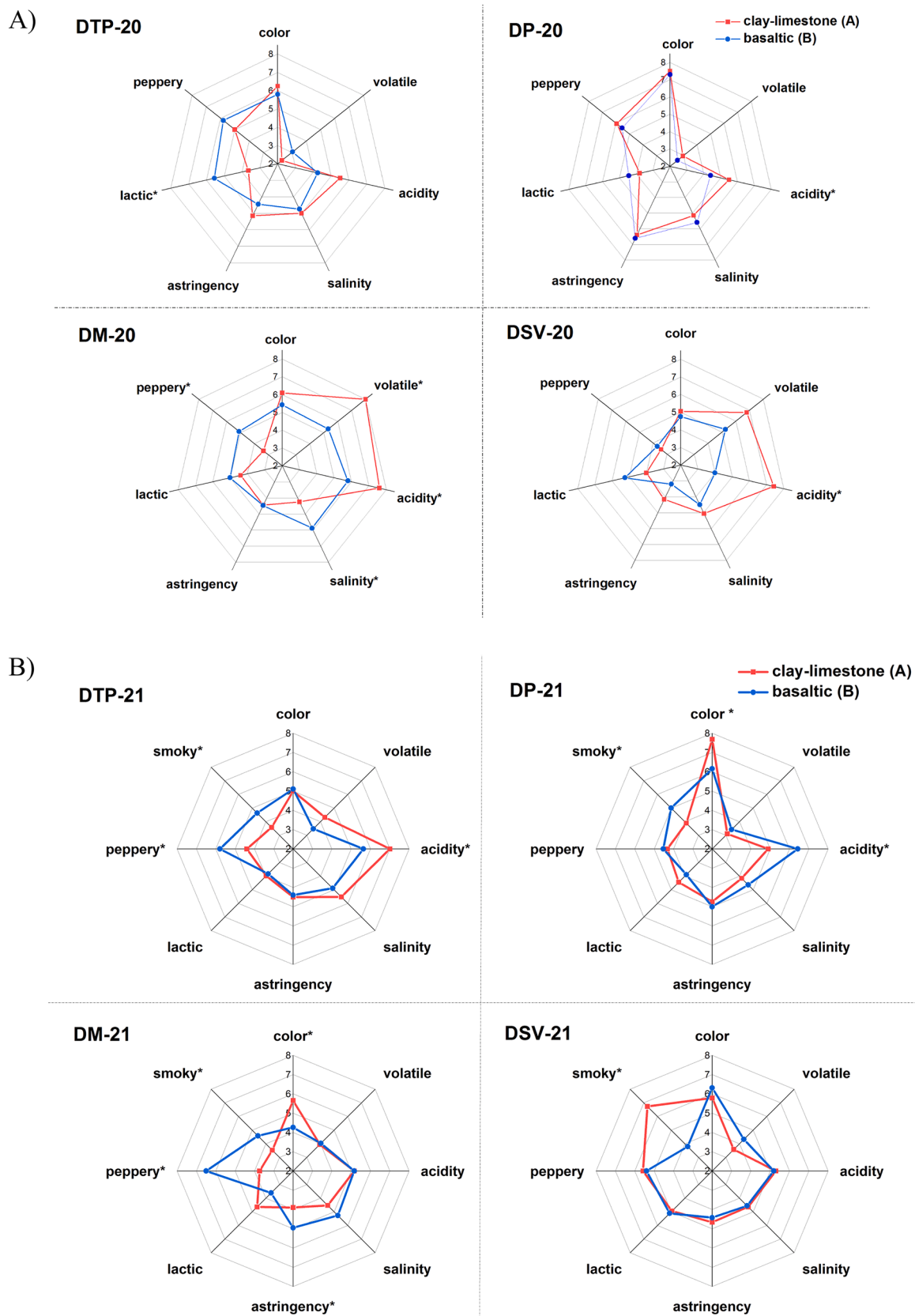


Fig. 5. Radar charts representing mean values of sensory descriptors of Gamay wines originating from the four studied sectors of PDO Côtes d’Auvergne for vintages 2020 (A) and 2021 (B). The two studied typologies of vineyards for both vintages are represented in red for clay-limestone (A) and in blue for basaltic (B). The star symbol (\*) means that the wine descriptor is statistically different ( $p < 0.05$ ) between the two vineyard typologies by wine sector and by vintage.



further investigations for the chemical and sensory conditions to be revealed. Even if peppery and smoky flavors appear as relevant factors for characterizing wines from volcanic areas, it should be noted that meteorological, viticultural and vine material studies should be carried out to evaluate to what extent such sensory descriptor could be expressed from such volcanic vineyards.

### 3.5. Discussion on the chemical and sensory correlations existing between Gamay red wines

Further explorations on the possible correlations existing between wine chemical parameters (multi-elements, oenological parameters, anthocyanins) and sensory descriptors were made by adopting a multifactorial analysis (MFA) approach. Fig. 6A shows the results of the MFA and Figs. 6B, 6C, 6D and 6E depict the observation plots of wine multielement content, anthocyanin content, sensory attributes, and oenological/hydric status parameters, respectively. A total variance of 42.4 % was explained with the entire dataset. Wines are separated along the F1 axis according to their vineyard typology, regardless of the vintage. While vineyard typology has a positive association with many elements and chemical constituents, vintage also associates positively with some of those compounds, which confirms that vineyard typology orients the diversity of chemical composition of wines and that vintage reinforces such distinction.

Gamay wines elaborated from basaltic vineyards appeared to be positively associated with volatile, lactic, smoky, salinity and acidity descriptors and negatively with peppery, astringency and yellow color, suggesting sensory similarities among those categories of wines (Fig. 6D). Even if such classification could not be generalized due to multiple environmental factors occurring during grape growing phases (vintage, grape maturity and wine sector climatic conditions) and

winemaking practices (pre- and post-fermentation, oenological practices, ageing) our methodology suggests that the vintage effect was less dominant than vineyard typology, reinforcing the role of vineyard geology in the expression of sensory characters in wines elaborated in volcanic regions. Further studies are necessary to explore such chemical and sensory diversity in wines from other volcanic regions. For the case of PDO Côtes d'Auvergne wines, the relationships existing between sensory descriptors and wine chemical parameters have been explored by partial least square (PLS) models (N = 16 wines, 45 chemical compounds, 8 sensory attributes). For that purpose, individual PLS models were carried out to predict each sensory attributes using the 45 chemical descriptors as the observable variables. Results of the individual PLS models are given in Table S.I.8. Only four sensory descriptors (color, acidity, astringency and smoky) could be sufficiently predicted with the collected dataset (Table 1), presenting predictive ability ( $Q^2$ Cum) values superior to 0.2. Their observed values are plotted vs PLS predicted values (Figure S.I.4). Concerning the four other studied sensory attributes (volatile, salinity, lactic and peppery) the PLS models were not sufficiently predictive, presenting negative values for  $Q^2$ Cum. Such result indicates that other chemical compounds, such as volatile compounds, could be interestingly analyzed in the future to reinforce sensory interpretation of Gamay wines regarding its chemical composition. However, we could explore the chemical relationships with the color, the acidity, the astringency and the smoky character of Gamay wines, by evaluating the best chemical descriptors (VIP > 1) used for PLS models (Table 1).

Global chemical parameters (L, a, A420, A520, A620, CI, TPI, TAN, ANT) were closely related to red wine color. For the acidity perception, wine parameters such as TA, VA and pH were correlated, but other uncommon chemical parameters associated with wine color such as TPI, TAN, hue, b, alcohol, petunidin-3-O-glucoside, cyanidin-3-O-glucoside,

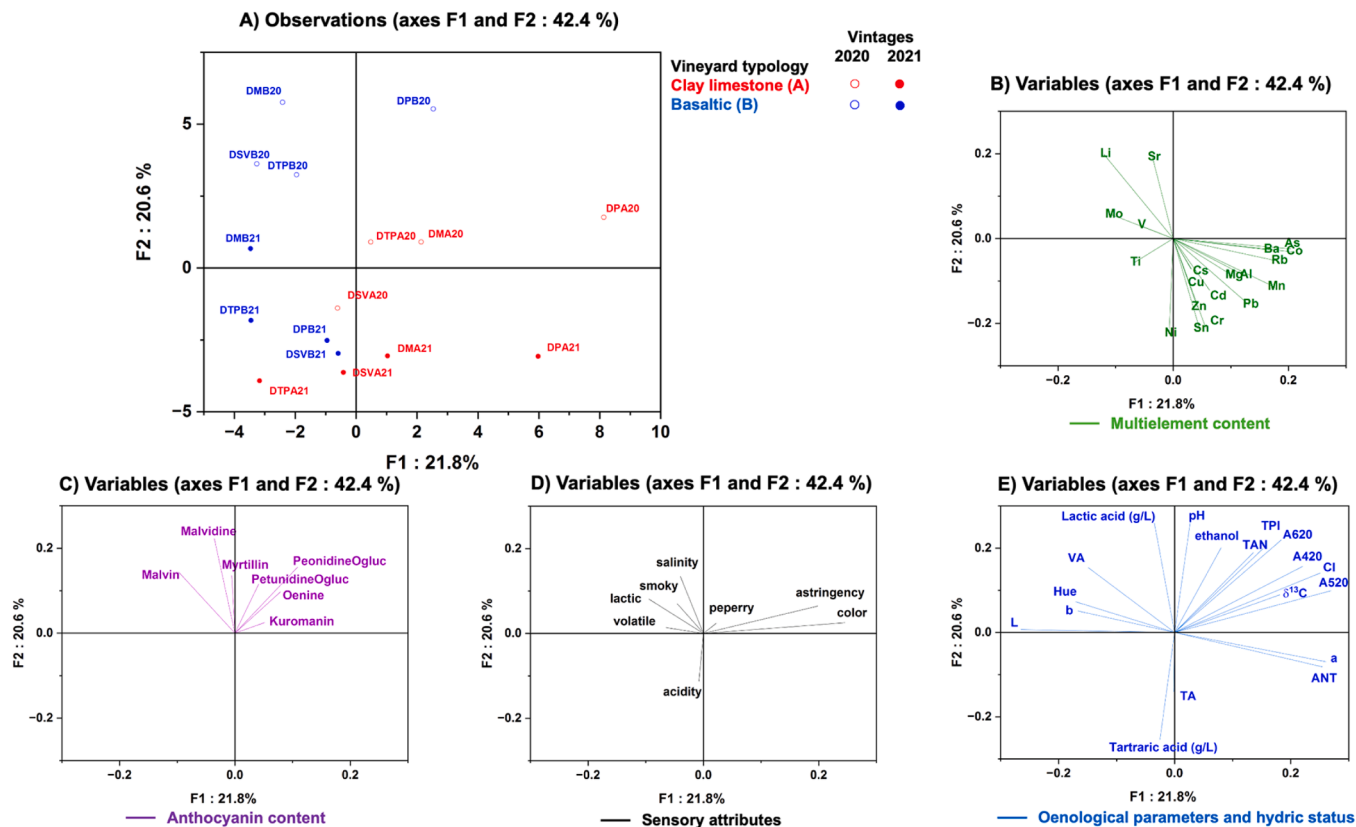


Fig. 6. Multifactorial analysis of Gamay wines from the French PDO Côtes d'Auvergne. (A) Observation biplot of the sixteen wines originating from the two typologies of soil vineyards (A in red and B in blue) for vintages 2020 and 2021, using emptied circle legend and full-colored circle legend, respectively. (B-E) Loading plots of multielement variables, anthocyanin variables, sensory variables and oenological/hydric status variables.

**Table 1**

Results of the Partial Least Square models correlating each wine sensory descriptors with wine chemical parameters, presenting a VIP > 1 used for the predictive modelling.

Sensory descriptor	PLS (models with two components)			Wine chemical descriptors with VIP > 1
	R <sup>2</sup> X	R <sup>2</sup> Y	Q <sup>2</sup> Cum	
Color	0.411	0.898	0.659	L, A420, CI, A520, TPI, a, TAN, ANT, A620
Acidity	0.335	0.877	0.425	TA, VA, petunidin-3-O-glucoside, TPI, cyanidin-3-O-glucoside, TAN, pH, hue, Sr, delphinidin-3-O-glucoside, b, malvidin-3-O-glucoside, alcohol
Astringency	0.382	0.815	0.207	TAN, TPI, A520, CI, b, $\delta^{13}\text{C}$ , L, A620, A420, a, peonidin-o-glucoside, Ba, ANT
Smoky	0.376	0.935	0.248	Tartaric A, Mn, Mg, Cu, Al, Malvidin-3,5-diglucoside, ANT, hue, b

TA: total acidity, VA: volatile acidity, TPI: Total Polyphenol Index, TAN: Tannin content, ANT: Anthocyanin content, L, a, b: Cie Lab parameters, CI: Color Intensity, Tartaric A: Tartaric acid

delphinidin-3-O-glucoside, and malvidin-3-O-glucoside, and with wine elemental composition with strontium (Sr) were also related to the wine acidity perception. Thus, the difference in acidity perception found in volcanic wines could also be intimately related to grape growing conditions by accumulating secondary metabolites and mineral elements. The fact that strontium is an element that has been previously found to be poorly affected by winemaking conditions, but more associated with terroir conditions, reinforces the possible link to the perception of acidity in wines.

Chemical parameters, such as TAN, TPI, A520, CI, L, a, b, A620, A420, and ANT, were found to be correlated with wine astringency. Previous studies already highlighted their use as a proxy for wine astringency perception (Boulet et al., 2016; Josie et al., 2008; Pavez et al., 2022). Other less common parameters, such as the anthocyanin compound peonidin-3-O-glucoside, wine element barium (Ba) and vineyard hydric status with  $\delta^{13}\text{C}$ , were also related to the wine astringency perception. Barium, a major element found in our vineyard soils and still present in resulting wines, appeared to be interestingly correlated to the perception of astringency in wines. Furthermore, peonidin-3-O-glucoside and  $\delta^{13}\text{C}$ , which both distinguished wines according to vintage and vineyard origin, were also related to wine astringency perception.

Finally, wine smoky perception was related to the oenological parameters tartaric acid, malvidin-3,5-O-diglucoside, ANT, hue and b parameter, but also to some elements like Mn, Mg, Cu and Al. Among them, high manganese (Mn) content was already found to be related to less spicy notes in Gewurztraminer wines (Ferretti and Febbroni, 2022). In the present work, wines from volcanic soils contain less Mn than wines from non-volcanic soils (Table S.1.6). It should be noted that manganese, in combination with other trace elements like copper and iron was also found to be a promoter of wine phenolic oxidation leading to evolutive/oxidative notes in wines (Danilewicz, 2016). Those scientific data could partially explain our more intense smoky notes perceived in Gamay wines from volcanic vineyards, containing less manganese, compared to non-volcanic ones. While our study suggests the importance of inorganic constituents in the distinction of wines according to their vineyard typology, other compounds, not been measured in this study such as volatile organic compounds by GC-MS and wine phenolics by LC-MS, could be assessed in the future to gain new insights in the ortho and retro-nasal perceptions of wines elaborated in volcanic regions.

#### 4. Conclusions

During two consecutive vintages, Gamay red wines were elaborated in the French PDO Côtes d'Auvergne on four distinct sectors comparing systematically basaltic and sedimentary vineyards with the objective of unraveling some specificities attributed to volcanic wines. The pedological composition of basaltic soils could be easily distinguished from the composition of non-basaltic soils by its granulometry and its more abundant lithium and strontium. Interestingly, volcanic vineyards present a more moderate hydric status compared to non-volcanic ones. Such pedogeochemical differences in soil vineyards enabled distinguishing their wines. Volcanic wines were characterized by a more yellow-tinted red color and lower anthocyanin content compared to non-volcanic wines. Wine astringency was also affected by such vineyard typology distinction in anthocyanin and barium composition. Other rare elements permitting to differentiation of volcanic and non-volcanic wines (particularly strontium, lithium and manganese during vintage 2020 and zinc during vintage 2021) were closely related to the differences observed in acidity and smoky sensory perception. The four sensory descriptors (color, astringency, acidity and smoky) are interesting sensory descriptors worth investigating in other volcanic regions due to their relationship to wine organic and inorganic components that reflect the geological origin of the vineyard.

#### CRedit authorship contribution statement

**Laurent Rios:** Writing – review & editing. **Christophe Poix:** Writing – review & editing, Formal analysis. **Ivan Vlastelic:** Writing – review & editing, Formal analysis. **Charley Merciecca:** Writing – review & editing, Formal analysis. **Abdelmouhcine Gannoun:** Writing – review & editing, Formal analysis. **Christian Coelho:** Writing – review & editing, Supervision, Methodology, Funding acquisition, Conceptualization. **John Szabo:** Methodology, Funding acquisition. **Benoit Marsan:** Writing – review & editing, Methodology.

#### 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. Coelho et al. report that financial support was provided by Vinora association promoting vitiviculture in volcanic areas in the world, La Région Auvergne-Rhône Alpes and Conseil Départemental du Puy de Dôme

#### Acknowledgements

We thank our financial supporters: Vinora Association which promotes vitiviculture in volcanic areas in the world, La Région Auvergne-Rhône Alpes and Conseil Départemental du Puy de Dôme. We are very grateful to winegrowers and winemakers in the PDO Côtes d'Auvergne who were involved during the winemaking processes. We also thank the French and Canadian panelists for their participation during the two consecutive vintages.

#### Ethical approval

All procedures for sensory evaluation were carried out in accordance with relevant laws and institutional guidelines and were approved by all the academic institutions.

#### Informed consent

Informed consent was obtained from all individual participants, included in the study.

## Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.jfca.2024.107115](https://doi.org/10.1016/j.jfca.2024.107115).

## Data Availability

Data will be made available on request.

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