Research Article

Received: 15 September 2023 Revised: 22 November 2023 Accepted article published: 29 November 2023 Published online in Wiley Online Library:

(wileyonlinelibrary.com) DOI 10.1002/jsfa.13179

Assessment of fruity aroma intensity in olive oils from different Spanish regions using a portable electronic nose

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Abstract

BACKGROUND: The organoleptic profile of an olive oil is a fundamental quality parameter obtained by human sensory panels. In this work, a portable electronic nose was employed to predict the fruity aroma intensity of 199 olive oil samples from different Spanish regions and cultivar varieties ('Picual', 'Arbequina', and 'Cornicabra'), with special emphasis in testing the robustness of the predictions *versus* cultivar variety variability. The primary data given by the electronic nose were used to obtain two different feature vectors that were employed to fit ridge and lasso regressions models to two datasets: one consisting of all the samples and another just the cv. Picual samples.

RESULTS: The results obtained showed mean average error (MAE) values below 0.88 in all cases, with an MAE of 0.67 for the 'Picual' model. These MAE values and the similarities in the model parameters fitted for the different data folds are in agreement with the results obtained in previous studies.

CONCLUSION: The large number of samples analyzed and the results obtained show the robustness of the approach and the applicability of the methods. Also, the results suggest that better performance can be obtained when specific models are fitted for particular cultivars. Overall, the proposed methods are capable of providing useful information for a fast screening of the fruity aroma intensity of olive oils.

Keywords: olive oil quality; e-nose; organoleptic assessment; metal oxide semiconductor sensor; feature extraction; multivariate analysis

INTRODUCTION

Owing to its efficacy in the prevention of numerous chronic-degenerative diseases,[1,2] the Mediterranean diet is known for its high utilization of olive oil as the primary source of dietary fat.[3] Countries such as Spain, Italy, and Greece are the world's largest producers.[4]

Based on chemical and sensory qualities, European legislation divides olive oil into three main commercial categories: extra virgin olive oil (EVOO), virgin olive oil (VOO), and lampante olive oil (LOO). The EU rigorously regulates olive oil characteristics and develops methods for analyzing them (EEC Reg 2568/91, EC Reg 640/08).[5] The organoleptic profile of an olive oil, obtained by human sensory panels, is a fundamental quality parameter for its classification.[6]

The quality and the sensory profile of EVOO are closely related; in fact, for a VOO to be considered as EVOO, no sensory defects should be present. The lower quality olive oils are VOO, which is fruity (>0) and also has a few minor defects (≤2.5) deriving from mildly defective olives or incorrect extraction or storage processes, and LOO, which is non-edible and releases an unpleasant smell.[7,8]

Sensory assessment, however, needs many resources and time, as well as specialized panelists, which are not always at the disposal of small and medium-sized enterprises and cooperative societies and should not be used for routine operations.[9,10] In addition, the sensory panel test still has several limitations owing to the nature of the method, such as the inability to evaluate more than a certain number of samples each day and panel misalignment problems.[11,12] It is also important to remember that the global production of VOO is growing at worldwide scale, but the number of recognized sensory panels is limited.[12-14]

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For all these reasons, in recent years, through different ways, many studies have intended to set up an instrumental screening tool that supports the sensory panel.[13,15-17]

Other analytical methods, mainly represented by high-performance liquid chromatographic, vibrational, spectroscopic, and thermal techniques,[12,18-20] give a more precise indication of the chemical composition of the olive oil, but their use is limited by the cost and the time required to generate the results.[21]

Therefore, in order to rapidly determine the olive oil quality, sensor technology such as the electronic nose (e-nose) that can be deployed in the production line is of interest.[18,20,22,23]

For olive oil in particular, e-nose technology has been proven useful for identifying the geographical origin of the oil, the adulterations, the merceological quality and shelf-life changes due to external factors such as storage time, humidity, temperature, and oxidation.[19,23-30]

Multivariate analysis is generally used in e-nose signal analysis because it reduces the high dimensionality of a multivariate problem where variables are partly correlated, allowing the information to be displayed in a smaller dimension.[31] There are many multivariate techniques to choose from; for example, cluster analysis, principal component analysis, partial least squares, linear discriminant analysis.[15,32-35]

In a previous paper, Marchal *et al.*[3] showed that the proposed methods (ridge and lasso regressions) offered promising results for the prediction of fruity aroma intensity in samples from a single producer. The aim of this work is to extend this approach to samples of different producers, geographical regions, and cultivars and verify if the proposed methods still provide useful results in these conditions.

MATERIALS AND METHODS

Olive oil samples

A total of 199 olive oil samples were used, produced from *Olea europeae* fruits belonging to typical Spanish cultivars during the 2021–2022 crop season from different mills of Spain. Most of the samples were cv. Picual from eastern Andalusia (127 samples), with the rest coming from other regions of the country and being mainly cv. Arbequina (41 samples) and cv. Cornicabra (31 samples). To test each sample's volatile emission by e-nose immediately and to send it to the authorized CM Europa laboratory (Jaén, Spain) for sensory analysis, each sample was separated into representative aliquots and kept in 125 mL dark glass containers. The characterization of 199 olive oil samples is reported in Table 1.

Experimental setup

The compact (92 × 190 × 255 mm^3) and lightweight (2.3 kg) portable olfactory system used for the analyses of the olive oils was the e-nose PEN3 (Airsense Analytics GmbH, Schwerin, Germany). It consists of a sensor array with a maximum flow rate of 600 mL min^{-1} and a gas sampling system, whereas software (Win Muster v. 1.6.2) allows data acquisition, visualization, and analysis.[15] According to the manufacturer's specifications, the integrated sensor array is composed of ten different thermo-regulated (200–500 °C) metal-oxide thick-film sensors (MOS) positioned in a very small stainless steel chamber (volume: 1.8 mL; temperature: 110 °C) and sensitive to different classes of chemical compounds (S1: aromatic organic compounds; S2: nitrogen oxides; S3: ammonia; S4: hydrogen gas; S5: alkanes, aromatic compounds, and non-polar organic compounds; S6: methane; S7: terpenes and sulfur-containing organic compounds; S8: alcohol; S9: inorganic sulfur and organic compounds; S10: methane and aliphatic organic compounds).[22]

As previously described,[22] each measurement process involved two stages: (i) the cleaning (air passes through an activated carbon filter before entering the sensor array); (ii) the measurement (air passes first through an activated carbon filter and through the sample, and finally through a moisture and particle filter before arriving at sensor arrays).

A transducer element (electrode) associated with each sensor may detect the reduction in electrical conductivity caused by an oxygen exchange that occurs when the volatile compounds of the sample interact with the detecting layer of the sensor surface.

A 5 g mass of olive oil was poured into a dark glass 13.5 mL vial for each sample and hermetically sealed with rubber closure; and before the measurement process (two replications), in order to increase the volatile concentration in the headspace, the sample was conditioned to 30 ± 1 °C.

Features extraction

The response curves for all the sensors in the PEN3 device, which show the sensor measurement as a function of time, are the main source of information. Similar to the approach presented in previous work,[3] these curves were employed to obtain a set of features that characterized the curves and summarized their information, as is typical practice in applications that employ e-noses.[3,22,36] This feature extraction process was performed on the raw response curves, as no filtering preprocessing step was required, given the low noise level of the curves. In the previous work,[3] despite using methods that included regularization, the relatively small number of available samples still advocated for a feature vector of limited dimension in order to limit potential overfitting problems. The larger number of samples available for this study allowed us to evaluate the convenience of employing a larger number of features to characterize each response curve, and therefore a larger feature vector. Therefore, four features were extracted:

(1) Peak value of the curve (S*x*M).
(2) Final value of the curve (S*x*F).
(3) Time when the maximum value is obtained (S*x*T).
(4) Ratio between the peak value and the final value (S*x*R).

As an example, the peak value of the curve for sensor 2 is denoted S2M, or the final value of the curve for sensor 3 is

Table 1. Characteristics of the 199 olive oil samples used in the trial

No. of samples	Quality	Medium fruity	Medium defect
138	Extra virgin olive oil	6.14 ± 1.28	—
27	Virgin olive oil	3.48 ± 0.58	2.17 ± 0.31
34	Lampante olive oil	2.34 ± 1.11	3.89 ± 1.70

denoted S3F. This work presents and compares two approaches: (i) 'all_features', which includes the four features extracted (feature vector of dimension 40); (ii) 'reduced_features', which includes the first two features (M and F; feature vector of dimension 20). This latter approach was the one already used in the previous work with good results.[3]

Prediction of fruity aroma

The prediction of the value of fruity aroma constitutes a data regression problem because this is a continuous parameter. Figure 1 shows the sample distribution of the fruity aroma values. As shown in the plot, the values are reasonably spread, so they are susceptible to being treated employing standard regression tools.

In reality, it is possible to utilize a linear model for the prediction of fruitiness, given that the features fundamentally describe the reaction of sensors to the concentration of volatiles. Thus, taking into account the considerations presented in the previous work,[3] two techniques were used: ridge and lasso regressions. The two approaches are fairly similar, in that they both effectively fit a linear model while attempting to solve the problem of optimization with an associated penalty for the size of the linear model's coefficients in the objective function.[37]

The parameters w of a ridge regression model are found minimizing the following expression:

$$J(w) = \left\|y - w^{\mathrm{T}}x\right\|^2 + C\|w\|^2 \quad (1)$$

where C is the coefficient that controls the regularization term. If $C = 0$, then the problem is reduced to a regular least-squares fitting problem.

Similarly, the lasso minimizes the following expression:

$$J(w) = \left\|y - w^{\mathrm{T}}x\right\|^2 + C\|w\|_1 \quad (2)$$

In this case, it is the l_1 norm that penalizes the size of the parameters. This method typically yields solutions that are usually sparse (i.e. contain many coefficients that are exactly zero).

Statistical analysis

Scikit-learn, a machine-learning library for Python,[33] was used for all of the data computations.

RESULTS AND DISCUSSION

Prediction of fruity aroma

The generalization capacity of the models built for prediction of fruity aroma intensity was assessed using fivefold cross-validation. As commented in the previous section, ridge and lasso regressions models were fitted to the 40-feature vector (all_features) and to the 20-feature vector (reduced_features) presented in the previous section for two datasets: one consisting of all the samples and another containing just the 'Picual' samples.

Table 2 shows the mean average error (MAE) and mean maximum error (MME) for both training and testing for all the combinations of data partition, feature sets, and regression techniques. The first comment is that the models that consider just 'Picual' samples offer better results than those obtained using all the samples. This result provides some indication that the use of specific varietal models could be beneficial in terms of model performance. Another point is that, in our previous work, the lasso approach offered better results, both in terms of MAE and MME, than ridge regression did; in contrast, in this study, ridge regression provides better results for the models that consider all the samples, whereas lasso prevails for the 'Picual' models. Some insight into this behavior can be obtained inspecting Figs 2 and 3, which provide the fitted parameters for each of the models, feature sets, and subgroups considered. Although there is strong coherence in the values obtained for the different data folds, the all_features_ridge_picual model (Fig. 2(a)) shows a larger variability in the parameters with low value than that observed in the all_features_ridge_all_samples. This effect may be due to the larger number of samples in the all_samples data partition, which may exert an additional regularization effect that helps in robustifying the models fitted to each data fold.

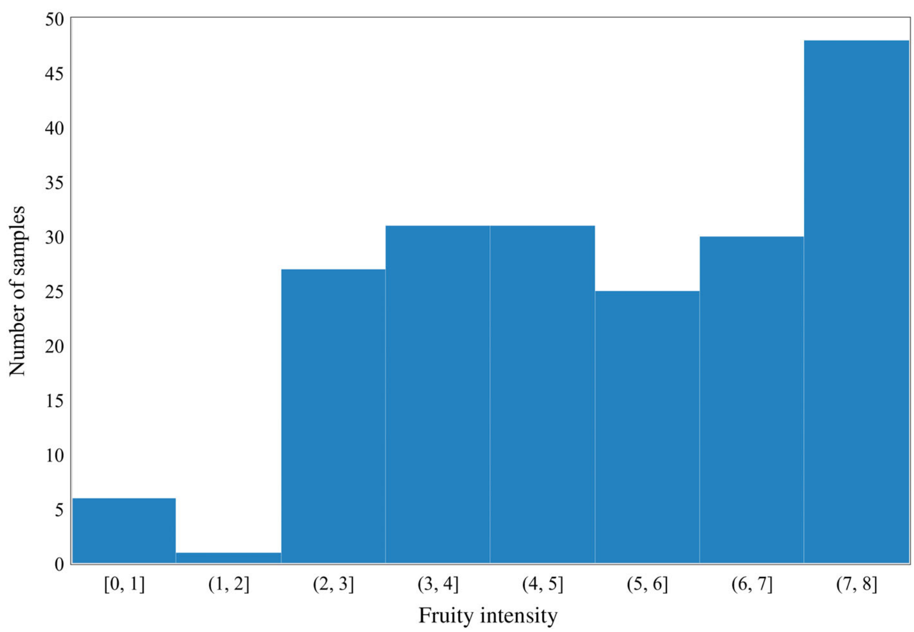

Figure 1. Distribution of fruity intensity for all samples.

Table 2. Results obtained for the prediction of fruity aroma				
	Mean average error		Mean maximum error	
Model	Testing	Training	Testing	Training
All_features_ridge_all_samples	0.82	0.64	2.56	2.68
All_features_lasso_all_samples	0.85	0.76	2.98	2.90
Reduced_features_ridge_all_samples	0.80	0.71	2.82	2.78
Reduced_features_lasso_all_samples	0.88	0.80	3.03	3.04
All_features_ridge_picual	0.75	0.49	2.38	1.76
All_features_lasso_picual	0.67	0.58	2.03	2.00
Reduced_features_ridge_picual	0.72	0.57	2.40	2.12
Reduced_features_lasso_picual	0.73	0.66	2.38	2.42

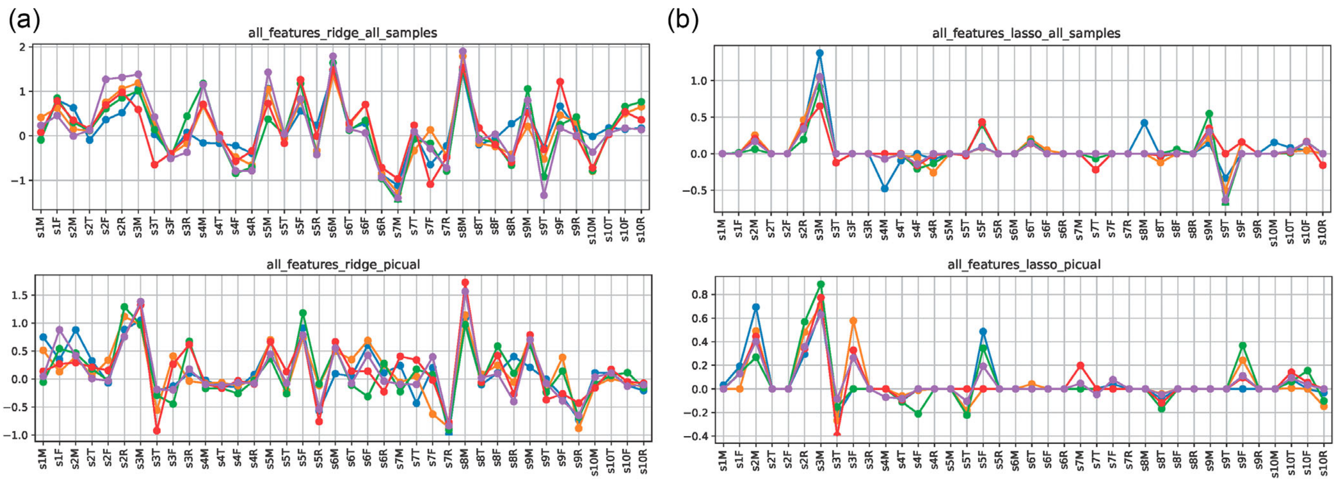

Figure 2. Visualization of the parameters of the ridge classifier (a) and lasso classifier (b) for each of the input features of sample (all samples and 'Picual').

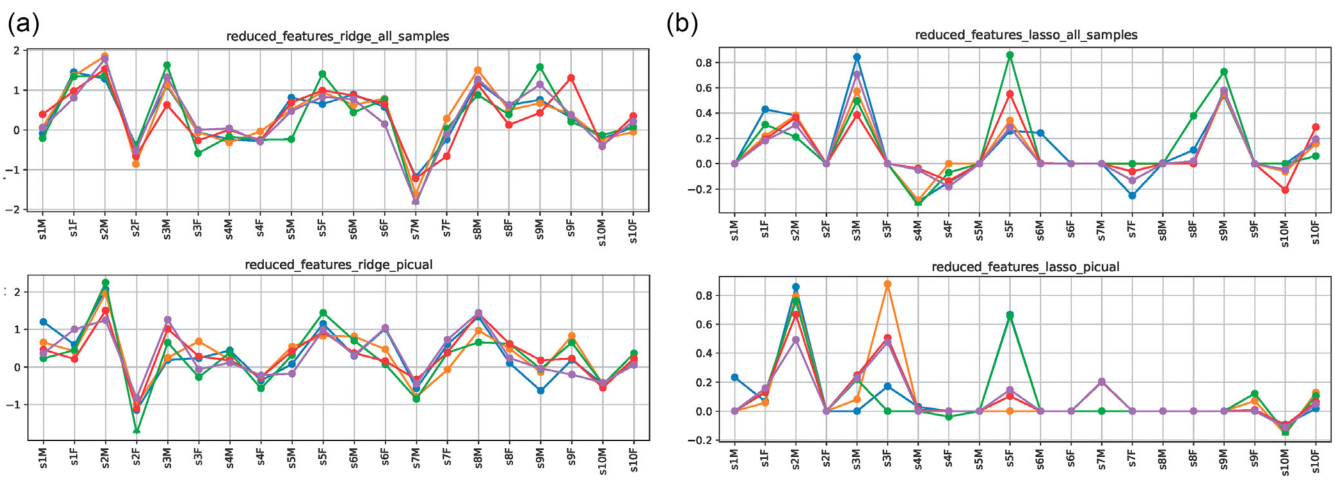

Figure 3. Visualization of the parameters of the ridge classifier (a) and lasso classifier (b) for reduced input features of the sample (all samples and 'Picual').

Another aspect that is worth noting is that the ridge models tend to be more similar for both data partitions than those obtained using the lasso, particularly so for the reduced feature vector. The comparison of the panels of Fig. 3(a) show a similarity of the curves that translates into similar coefficients being assigned to the features for both the all_samples and 'Picual' models. In turn, as shown in Fig. 2(b), the lasso models for the all_features models assign the largest coefficient to S3M for both the 'Picual' and all_samples models, but the 'Picual' model also assigns large values to S2M, to which the all_samples model assigns lower weights. Furthermore, the all_samples model assigns moderate weights to S9M and S9T, to which the

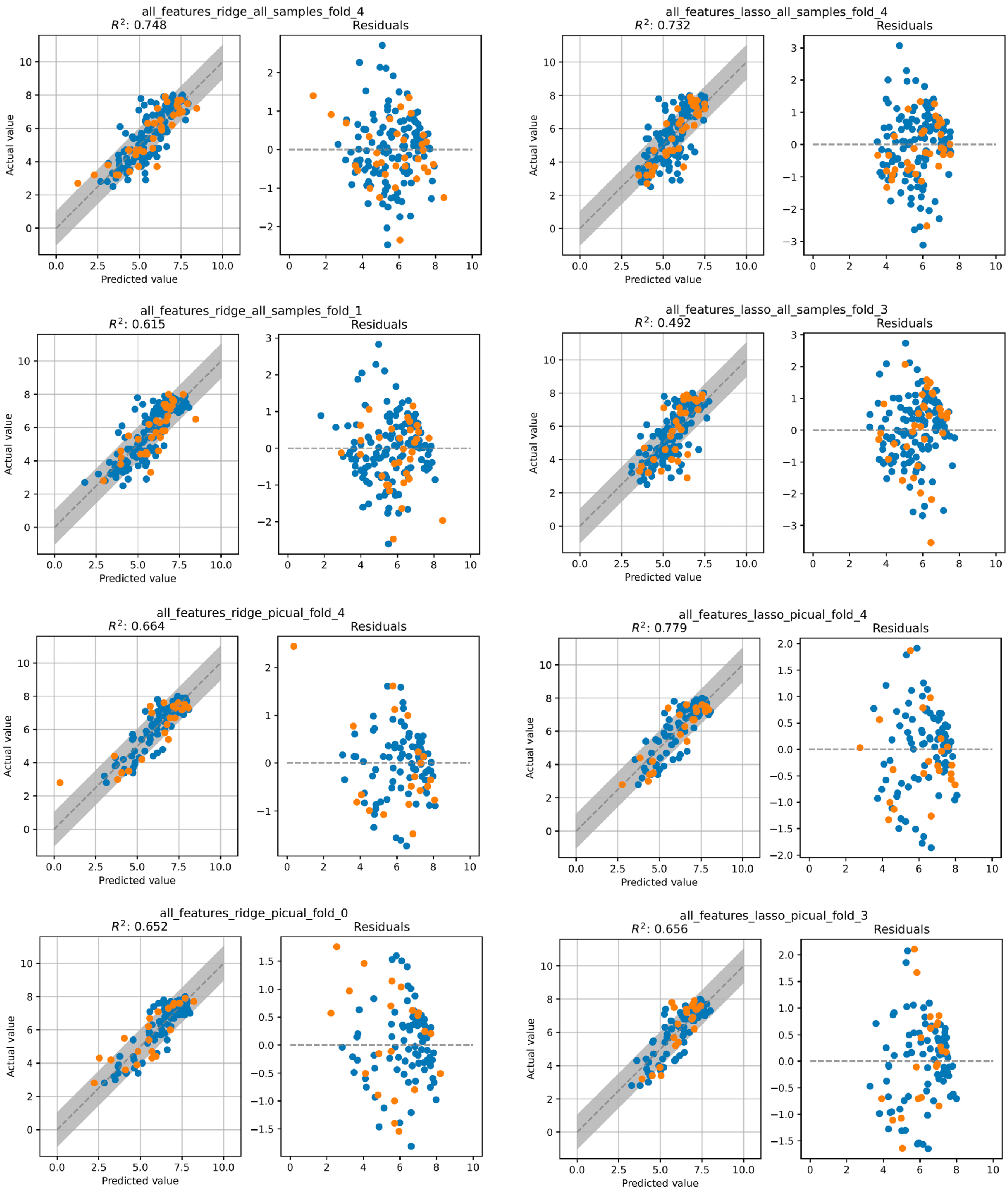

Figure 4. Visualization the results of the testing and training prediction for two of the five iterations for the two models (lasso and ridge) constructed using the 'all features' approach (all samples and 'Picual'). Training points are blue and testing points are orange, and the gray band represents the zone of ±1.

'Picual' model does not assign weight; instead, it provides smaller weights to S9F. The divergence between the model coefficients is more evident for the reduced_features models shown in panels of Fig. 3(b). Here, the largest coefficient for the all_samples models is assigned to S3M, whereas for the 'Picual' models the largest coefficient is S2M.

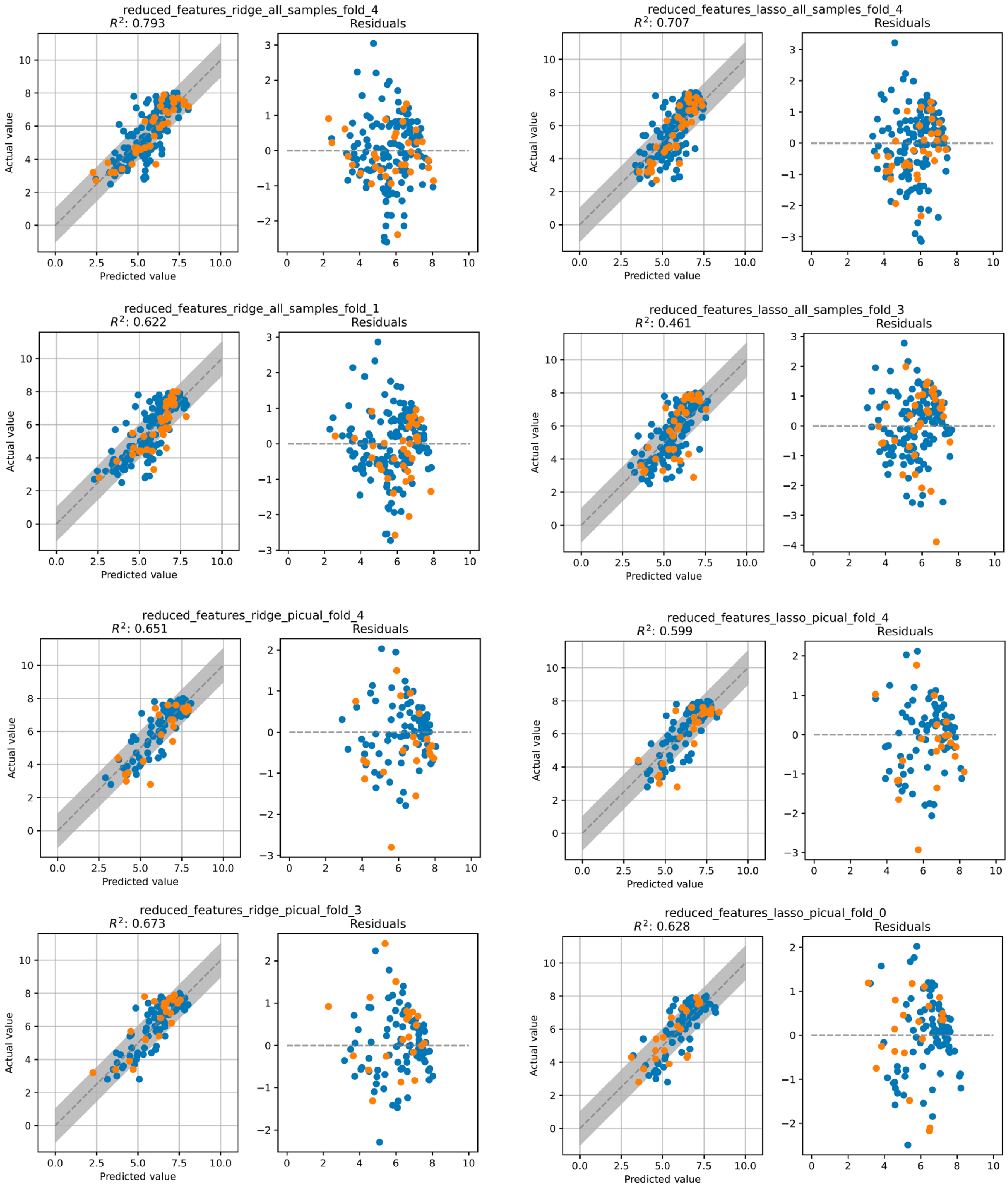

Figure 5. Visualization the results of the testing and training prediction for two of the five iterations for the two models (lasso and ridge) constructed using the 'reduced features' approach (all samples and 'Picual'). Training points are blue and testing points are orange, and the gray band represents the zone of ±1.

Another interesting comparison is between the 'Picual' coefficients obtained in this work and those fitted in our previous work for a single producer. The reduced_features model fitted in this work still provides relevance to sensors 10 and 2 of the e-nose; however, it changes the main feature for sensor 2 from the final value (S2F) to the maximum (S2M). Besides these coefficients, this work assigns some weight to sensors 3 and 5, although with more variability between folds than for sensors 2 and 10.

Finally, Figs 4 and 5 present the predicted *versus* actual values for some of the data folds used in the fivefold cross-validation. As shown in the plots, the data points show healthy distributions, as do the residual plots. These plots, together with the model stability between data folds discussed in the previous paragraphs, show that good generalization capabilities can be expected of the models obtained and the methods proposed.

To our best knowledge, there are no other references in the literature that try to predict the fruity aroma intensity of olive oils using e-noses; however, some other related studies can be found that help to provide some context to the results obtained in this work. For example, Martín-Tornero *et al*.[38] performed a study on the degradation of VOOs in different storage conditions and compared the results provided by panel test and an e-nose to predict the intensity of defect aroma, yielding a root-mean-square error of prediction of 0.65 in cross-validation. Though this result is promising, it should be noted that the number of samples in the validation set was not large ($n = 12$) and that the oil samples were obtained from a single batch of olives; thus, the robustness of the method against olive variability was not studied.

Another relevant paper is Teixeira *et al*.,[39] who employed a custom e-nose to analyze eight brands of commercial olive oil from Portugal and classify them into different categories according to the fruity aroma intensity, reporting an 81% accuracy for the leave-one-out cross-validation. The paper's discussion points out that this performance could probably be hampered by the fact that the oils were of different cultivars. This comment is in agreement with the results presented in this work, where better results are obtained when the prediction model considers only a single cultivar.

Furthermore, Jolayemi *et al*.,[23] analyzed 63 Turkish olive oils using a portable e-nose to discriminate the quality of the samples. Classification models were built with orthogonal partial least-squares discriminant analysis and validated both by cross-validation and external prediction. They reported 82% accuracy in prediction for the e-nose data models.

Another related work is that of Zhou *et al*.,[34] where the possibility of recognizing the addition of vegetable oil to EVOO using flavor fingerprinting, e-nose, and multivariate analysis is assessed. Among the three methods, the e-nose showed the best discrimination effect on mixed oil samples, being able to discern samples with 5% of seed oils. Despite these good results, they report some issues in the reproducibility of the results due to external conditions.

Finally, it is important to emphasize that, as confirmed by Modesti *et al*.,[18] one of the main limitations for the use of the e-nose in the olive oil sector is related to the availability of large sample sets, which are crucial to successfully train and validate the classification and prediction algorithms. This current work presents the study that with the larger number of samples and sources of variability can be found in the literature so far. However, despite the large sample variability, the MAE values included in Table 2 show good results, with values that provide enough accuracy to be useful for the prediction of fruity aroma intensity for screening purposes, particularly so for the 'Picual' model built considering all the features and the lasso regression.

CONCLUSIONS

This work has presented an extension of the approaches proposed in our previous work to a much larger number of samples, showing results that are in agreement with those found in our previous work. The slightly worse values of MAE and MME can be expected, as the variability of the samples considered in this work is much larger than those analyzed in our previous work; however, the MAE values obtained, which are below 0.88 in all the cases, show that the applicability of e-noses for the prediction of fruity aroma for olive oil samples is viable. In particular, the MAE of 0.67 obtained for 'Picual' and the similarity in the models fitted in this work and those in the previous work show the robustness of the approach and the applicability of the methods. These results also indicate that the construction of variety-specific models can benefit the prediction accuracy of the method.

ACKNOWLEDGEMENTS

We thank CM Europa S.L. for their collaboration in this work. This research was partially funded by the Spanish Ministry of Science and Innovation under the project PID2019-110291RB-I00.

CONFLICT OF 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.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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