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APPLICATION OF NEAR INFRARED SPECTROSCOPY COMBINED WITH MULTIVARIATE ANALYSIS FOR SCREENING FOLIAR MAIN ESSENTIAL OIL COMPONENTS IN BAY LAUREL

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KEYWORDS

NIRS prediction, *Laurus nobilis* L., 1-8 cineol, PLSR.

ABSTRACT

Ground bay laurel leaf samples (10–15 g) were scanned using Fourier-transform near-infrared (FT-NIR) spectrometer with reflectance mode in the 1000–2500 nm wavelength range. According to the wet chemical analyses, the essential oil content of the samples from different locations varied between 1.77 and 5.30%. The major component of essential oil was 1-8 cineole with a concentration of 43.4–58.1%. The regression coefficients of calibration (R^2_{CAL}) and validation (R^2_{VAL}) for essential oil and 1-8 cineol content with partial least square regression (PLSR) actualized as 0.96–0.98 and 0.98–0.98, respectively. The prediction accuracy of the final NIRS model was reasonable, with acceptable root mean standard errors of prediction (RMSEP) of 0.18% and 0.45%. According to the residual predictive deviation (RPD) index (3.58 and 8.41), the accuracy of the NIRS models was regarded as the best. The PLSR model differentiated bay laurel genotypes very well on the first principal component (PC1), based on the related properties.

INTRODUCTION

Civilization, industrialization, urbanization, intensive agricultural activities and destruction of forests pose an increasingly serious threat to the genetic diversity of many species, which are important to world agriculture and food security. Therefore, the collection and preservation of germplasm via different techniques and the improvement of its utilization are more important because of valuable genes and sustainability. Scientists should know what variations exist in their gene banks to use these genetic resources in breeding programs (Diez et al., 2018). Thus, germplasm characterization is an essential prerequisite (Migicovsky et al., 2019). This poses great opportunities to systematically introduce new species to agricultural systems. The development of a new cultivar or reconstructing an existing one with a gene from a wild form that is well adapted to an ecological condition gives advantages to breeders. The cultivation of medicinal and aromatic plants is a growing trend. Commercial gatherings in natural habitats are a common practice. However, due to increased consumption by humans and the growing demands of the global market, it is recommended to increase the cultivation of medicinal and aromatic plants with domesticated genetic material. It is not only for sustainability but also because it allows better control of biotic and abiotic production conditions, representing a reliable raw material resource (Astutik et al., 2019; Atanasov et al., 2015; Grigoriadou et al., 2020). Agronomists can select accessions from different populations by testing a wide variety of genetic material or limit themselves to evaluating accessions from one group with characteristics in which they are interested (Migicovsky et al., 2019). The proportion and components of the active ingredients of medicinal and aromatic plants are important selection criteria. The popularity of *Laurus nobilis* L. is increasing because of its valuable nutraceutical and pharmaceutical characteristics. It is commonly known as bay leaves, Bay, Sweet Bay, True Laurel or Roman Laurel, belongs to the *Lauraceae* family, and is a native plant in the Mediterranean region and Europe (Bahmanzadegan et al., 2015; Konovalov & Alieva, 2019). The leaves of this plant are used as a remedy in folk medicine in the treatment of arthritis, rheumatic pains, skin

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inflammation, bronchitis and asthma (Batool et al., 2020; Caputo et al., 2017). Several classes of industrially important secondary metabolites were successfully extracted using phytochemical studies. Sangun et al. (2007) determined 25 different compounds, with 1.8-cineole being predominant from the essential oil obtained from the leaves of *Laurus nobilis* L. Ecotypic variation in terms of chemical content was also reported. An inventory study carried out by the Hatay Governorship indicated that there were over 1.5 million bay laurel trees in the Hatay province of Turkey. Selecting with respect to the desired characteristics requires too much time and labor for such a large natural population.

Near-infrared reflectance spectroscopy (NIRS) is a nondestructive, advanced technology that measures the amount of absorbed near-infrared radiation by the chemical structure containing functional hydrogen bonds within a sample. The spectra comprise a spectral fingerprint that is statistically associated with the biochemical composition of a relevant sample (Balan et al., 2019). The use of NIRS in the quality assessment of medicinal and aromatic plants may offer many attributes. A large number of variables that characterize the entire chemical composition of any plant can be predicted in a few minutes from only a small amount of dried and ground samples without spoiling. Sample preparation does not require any reagents; therefore, NIRS is an environmentally friendly system because it generates no chemical waste. Moreover, the accuracy and precision of the measurements are rather high when the application is properly calibrated (Burns & Ciurczak, 2008).

The purpose of this study was to investigate the possibility of using a Fourier transform near infrared (FT-NIR) spectrometer as a selection tool for bay laurel breeding.

MATERIAL AND METHODS

Bay laurel (*Laurus nobilis* L.) individuals were sampled from the native flora of Hatay province located in the eastern Mediterranean coastal region of Turkey (35° 47′–36° 24′ E; 35° 48′–36° 37′ N; 85 m average altitude). The climate is a Mediterranean type characterized by hot and dry summers and mild winters, during which about 67% of the average annual precipitation of 890 mm occurs. The average annual temperature reaches a maximum of 28.8°C in August and a minimum of 8.8°C in January, with an average annual temperature of 12.4°C (Celiktas et al., 2017).

Reference data analyses

Chemically identified individuals were studied in a previous project funded by the Scientific Council of Turkey (TUBITAK) and used for spectra and reference data sources for chemometry. The ecogeographical distribution of individuals was considered throughout selection for the NIRS model. Fresh leaf samples were washed with flowing tap water and rinsed with distilled water to eliminate possible contaminants that could cause inaccurate spectral characterization. They were then oven-dried at 50°C for 24 h and homogenized by particle size reduction (<0.3 mm). Ground leaves were subjected to hydro-distillation using a Clevenger-type glass apparatus for 3 hours for the extraction of essential oils. The oil samples were filter sterilized, dried over anhydrous $CaCl_2$ and then stored in sealed vials at a low temperature (+4°C) before component analysis. The essential oil yield was estimated based on dry leaf mass using the following equation (Boutekedjiret et al., 2003):

$$RHE(\%) = \left(\frac{mHE}{mS}\right) x100$$

Where:

mHE = essential oil mass (g);
mS = dry leave mass (g), and
RHE = essential oil yield (%).

The essential oils were characterized using a Thermo Scientific™ ISQ™ 7000 GC-MS system (Waltham, MA, USA). The separated components were identified based on their relative retention times with the mass spectrum library of the National Institute of Standards and Technology (NIST).

NIRS and multivariate analyses

Dried and ground samples were scanned using a Fourier-transform near-infrared (FT-NIR) benchtop spectrometer (NIRFlex N-500, Büchi Labortechnik AG, Flawil, Switzerland) before extraction. The measurements were performed in glass Petri dishes containing 10–15 gr ground leaf samples. Absorbance data were obtained with an electromagnetic scan using the reflectance mode from 1000 to 2500 nm wavelength (4000–10000.cm^{-1}) with a spectral resolution of 4 cm^{-1}. All spectra were stored as an average of 32 scans, with three repetitions for each sample. Partial least square regression (PLSR) and principal component regression (PCR), which established a linear relationship between the spectral data and reference data obtained through wet chemical analyses, were used to construct calibration models. Principal components (PC_S) were used to create a multivariate discriminant model with the help of the chemometric software NIRCal 5.5 3000 Standart Edition (Büchi Labortechnik AG, Flawil, Switzerland). Different mathematical pre-treatments were employed to correct and reconstruct the spectra during data analysis. In the development of the NIRS models, 66% of the 54 spectra were used for calibration, and the remaining 33% were used for validation.

The calibration was expressed in terms of the coefficient of calibration and validation (R^2_{CAL}, R^2_{VAL}), the standard errors of calibration (SEC) and prediction (SEP). In the validation step, the overall error between the predicted and reference values was assessed using the root mean square error of prediction (RMSEP). Residual predictive deviation (RPD) was calculated by dividing the standard deviation of the reference values used in the validation, or prediction sample set (Std. Dev.)$_y$, by the SEP, as in the following equation (Esbensen et al., 2014).

$$RPD = (Std. Dev.)y/SEP(or\ SECV)$$

Where:

y represents the reference data of the sample set used for validation.

RESULTS AND DISCUSSION

The essential oil and 1-8 cineole content determined by the chemical and GC-MS analysis of bay laurel leaf samples from different locations and altitudes of Hatay province showed a wide distribution. The essential oil content of the individuals varied between 1.11 and 3.31% (Table 1). The highest value (E1) was obtained from the genotypes collected from the Samandag district of Hatay province, which is located along the Mediterranean coast. Previous investigations have shown that locality and harvesting season had a strong effect on both the concentration and the essential oil composition. Compound 1-8 cineole, with a concentration of about 50%, was found to be the major component in the extracted essential oil in the present study. The highest value for this secondary metabolite was determined in the genotype AY3 from Altınözü district of Hatay province (58.42%; Table 1).

TABLE 1. Foliar essential oil (%) and 1-8 cineol content of investigated bay laurel genotypes.

Property	**n**	**Range**	**Mean**	**Std. Dev.**	**MAD***
Essential Oil (%)	14	1.11–3.31	1.88	0.53	0.38
1-8 cineole (%)	14	45.47–58.42	50.51	4.25	3.38

*Mean absolute deviation.

A quantitative calibration was developed to identify the different chemical structures and separations of active substances to check the raw material using the developed database as a reference. The raw mean NIR reflectance data of the samples are shown in Figure 1.

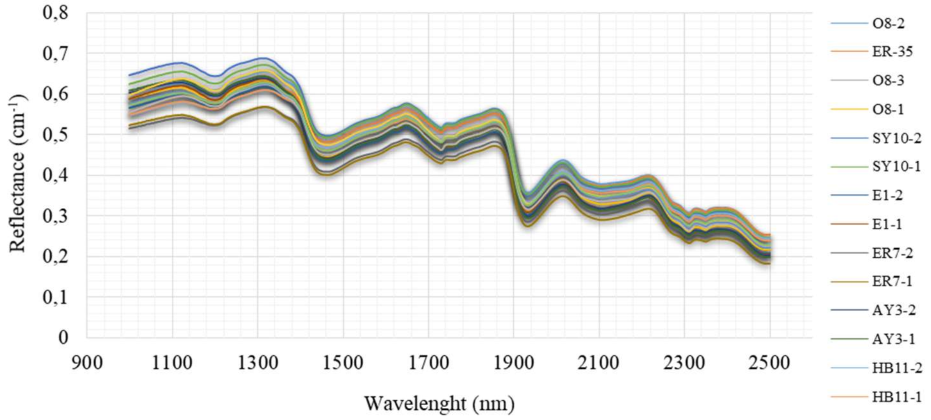


FIGURE 1. The original mean NIR spectra of bay laurel genotypes.

Each line in the figure represents the average reflectance of the three replicates of each sample. The NIRS data were examined using the chemometric software package for the best discrimination of the genotypes based on their essential oil and 1-8 cineol content. The best results in terms of a higher R^2 and a lower SEP prediction error were compared for the cross-validation models of PLSR and PCR. The descriptive statistical data associated with NIR-based calibration and validation are given in Table 2.

TABLE 2. Performance of near-infrared reflectance spectroscopy (NIRS) models developed for some properties of bay laurel.

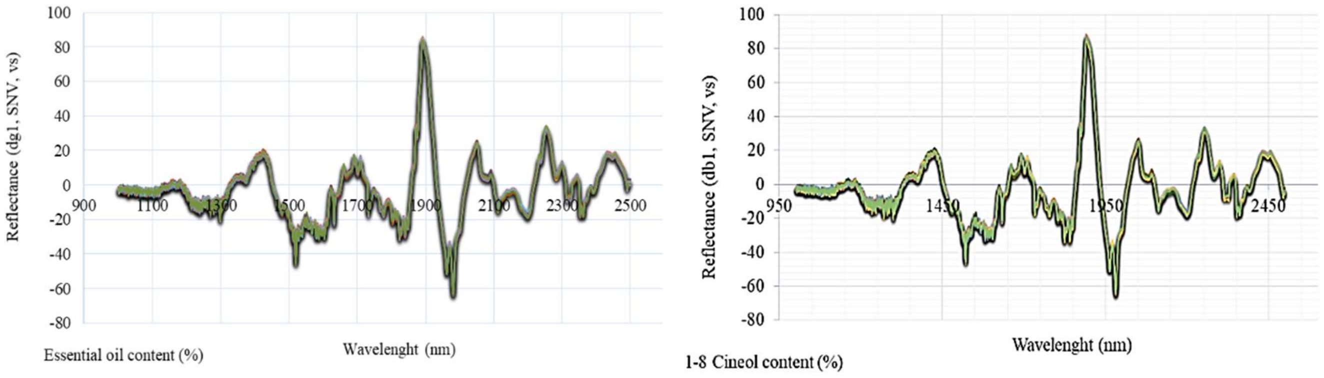
Property	**Method**	**n PCs**	**Pretreatments**	**R^2_{CAL}**	**R^2_{VAL}**	**SEC**	**SEP**	**RMSEP**	**RSD**	**RPD**	**Bias**
Essential Oil (%)	PLSR	3	dg1, SNV, vs	0.961	0.988	0.109	0.109	0.184	0.186	3.586	0.011
	PCR	10	ds2, n01	0.954	0.938	0.112	0.123	0.201	0.204	3.505	−0.022
1-8 cineole (%)	PLSR	2	db1, SNV, vs	0.984	0.984	0.528	0.522	0.451	0.458	8.411	−0.035
	PCR	4	db1, SNV, vs	0.972	0.977	0.718	0.674	0.699	0.710	6.515	−0.041

dg1=1st derivative Savitzky-Golay 9 Points, SNV=standard normal variate, vs= variance scaling, db1=1st derivative BCAP, ds2=second derivative smoothing, n01= normalization between 0 to 1

According to the statistical results, the NIR-based model for predicting the essential oil and 1-8 cineole content from ground leaf samples has rather high coefficients of determination with R^2_{CAL} and R^2_{VAL} for both regression models and properties. The PLSR model produced more acceptable results than PCR, with fewer PCs for both properties (Table 2). PLSR calculations were more up-to-date than the PCR calculations. However, in the PCR-relevant PCs for prediction, the property value was not always useful for the largest spectral variation. However, each PC calculated in the PLSR contained information about the reference values. Therefore, it always shows higher correlations. Wilson et al. (2001) determined a higher coefficient of determination (R^2_{CAL} = 0.95) for the NIR-based prediction of cineol content in *Eucalyptus* oil using the MLR model. However, MLR is reported to be less useful for quantitative calibration, while PLSR or PCR is recommended (Büchi, 2013). Using MLR, only a few wavelengths are used, and the others that are measured are discharged automatically. Due to extreme data reduction, the residual cannot be used for outlier detection during the application.

SEC and SEP, which provide the magnitude of the standard deviation for the calibration set and the independent validation set, were much closer to zero and almost overlapped (Table 2). SEP measures precision, while the RMSEP, which shows the random and systematic errors in the prediction as different from SEP, measures the accuracy of a prediction. These two parameters were found to be very similar to each other in the current experiment. If there is no or low bias, the RMSEP and SEP will be equal or very similar, since the RMSEP is equal to the quadrature addition of SEP and bias expressed by the following formula $RMSEP^2 = SEP^2 + Bias^2$ (Esbensen et al., 2002). Furthermore, the average difference between the NIR-predicted and measured values (in other words, bias values) was actualized to almost zero for all properties and models (Table 2). This difference should be as close to zero as possible for high precision. Milos & Bensa (2017) stated that future predictions at 2 times RMSEP represent a 95% confidence interval for the real values. Consequently, RMSEP is one of the most important and robust figures for judging the quality of a PLSR model (Zhao et al., 2015). Schimleck et al. (2003) reported that the coefficient of determination (R^2) between the measured and NIR estimated values for oil and 1-8 cineol content of *Melaleuca cajuputi* leaves were 0.65 and 0.92, respectively, for the PLSR model. They noted that their NIRS model had higher accuracy, with SEC values of 0.45% and 4.7% for the mentioned properties, respectively. Compared to this statement, the NIR-based model presented in the current study is quite reasonable, with RMSEP values for essential oil and 1-8 cineol content of 0.1847% and 0.4518%, respectively.

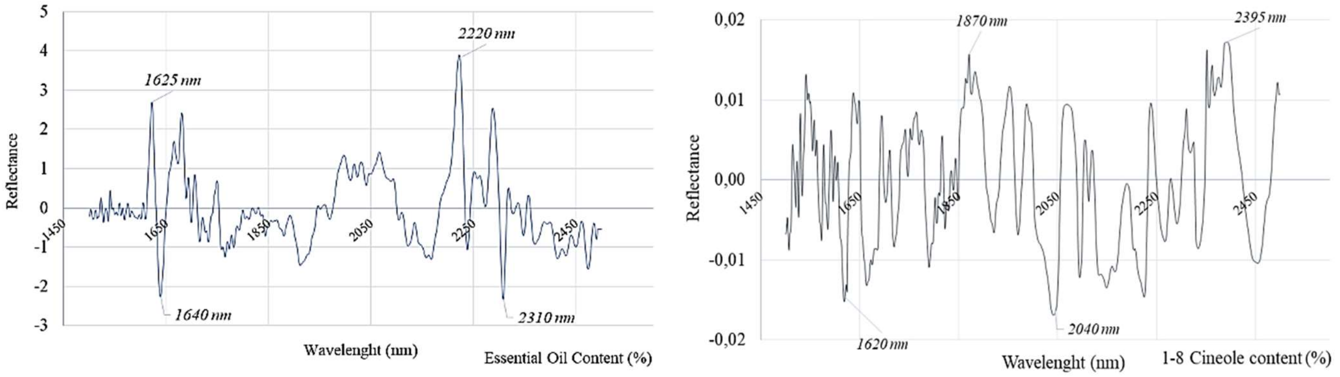
The spectral measurement process can be affected by many factors, leading to interference. Therefore, chemometric pretreatments are needed to optimize the performance of the model (Gautam et al., 2015). Therefore, the math treatments of 1st derivative Savitzky Golay 9 points (dg1) + standard normal variate (SNV) + variance scaling (vs) for essential oil and 1st BCAP (db1) + SNV + vs combination for 1-8 cineol were determined to be the best in the present study and applied to reduce the noise and normalize the spectra in the PLSR model. Yan et al. (2017) noted how pretreatments affected the calibration accuracy for menthe essential oil, showing that some pretreatments and combinations reduced the accuracy compared to non-pretreated data. They also reported the best combination as the 1st derivative + SNV + mc (mean centering) with the lowest RMSEP of 0.097%, while the others were within 0.108–0.152%. The corrected spectra for the properties investigated in Figure 1 are given in Figure 2.



(a) (b)

FIGURE 2. Reconstructed mean spectra for essential oil (a) and 1-8 cineol content (b) of ground bay laurel leaf samples.

The pretreatments never changed the wavelength of the original spectra or the property-related spectrum regions. In general, the longer wavelength region had a larger weight and hence a larger influence on the discriminant model for any property (Tigabu & Odén, 2004). Esbensen (2009) stated that important reflectance bands have higher regression coefficients in multivariate models. Therefore, the highest regression coefficients were found to be around the wavelengths of 2180–2250 nm for essential oil, predominantly 2000–2055 and 2350–2425 but also 1600–1630 and 1845–1900 nm for 1-8 cineol content (Figure 3). Similarly, Schimleck et al. (2003) discovered considerable variation between 1650–1800 nm and 2200–2400 nm for both the essential oil and 1-8 cineol content of *Melaleuca cajuputi*. However, the authors discovered a strong correlation between 1-8 cineol content and the 1768 nm wavelength, while 1696 nm had a strong relationship with essential oil. Similarly, Wilson et al. (2001) also selected the 1756–1758 nm wavelength as the region with the highest correlation in terms of 1-8 cineol content of eucalyptus oil. Important peaks around the mentioned bands are visible in Figure 3, which represents the C-H chemical bonds. However, Nascimento et al. (2015) noted that 2256–2258 nm wavelengths, which had high regression coefficients, were found to be important for the essential oil of *Eremanthus erythropappus* wood powder. Some external and uncertain factors, such as vibration, sample size and temperature, may affect NIR spectra (Zhang et al., 2015). Therefore, these slight differences in view of spectrum bands related to the active substances are quite possible.



(a) (b)

FIGURE 3. Regression coefficients of NIR model for essential oil (a) and 1-8 cineol content (b) of ground leaf samples of bay laurel genotypes.

Figure 4 presents the measured versus NIR predicted essential oil and 1-8 cineol content plot after chemometric pretreatments and determination of the optimal number of primary principal components (PCs).

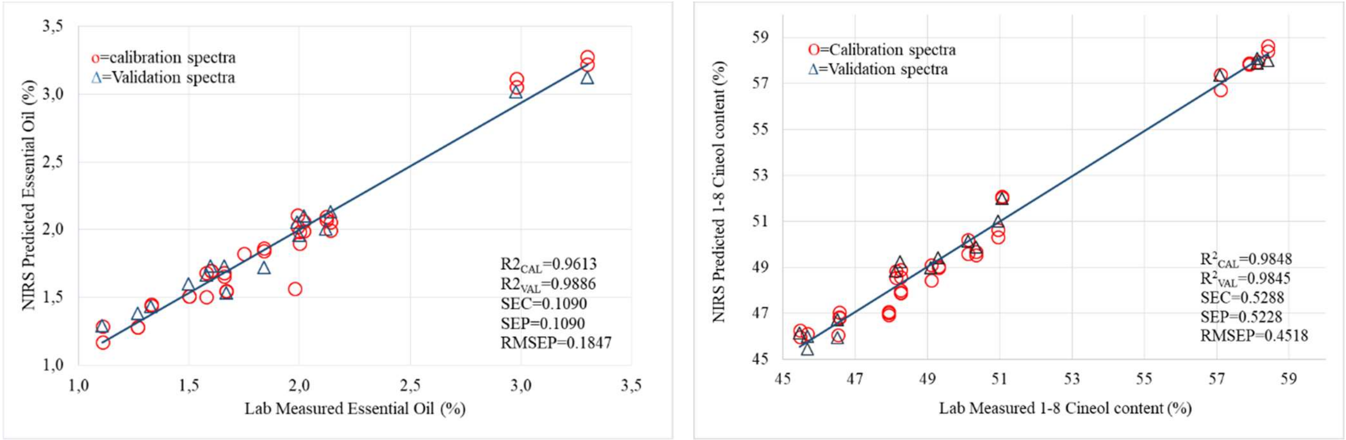
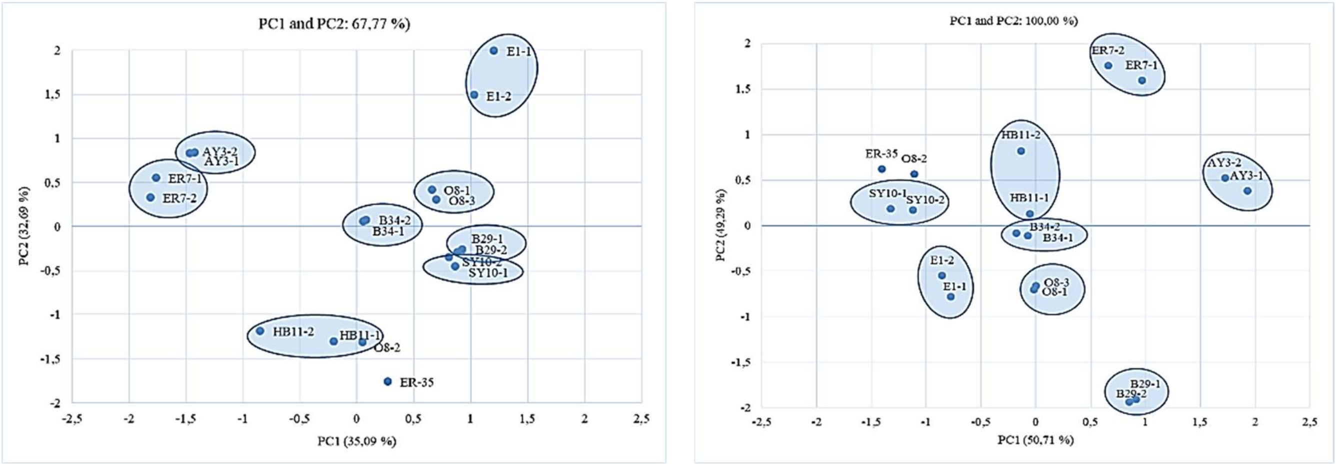


FIGURE 4. Laboratory-measured versus NIRS-predicted values of essential oil and 1-8 cineol content plots for ground leaf samples of bay laurel genotypes.

In the data analysis, two outliers for the essential oil and three outliers for 1-8 cineol content were detected and removed from the calibration spectra. As mentioned above, the average bias between the calibration and validation datasets for both properties was quite low, consequently resulting in a relatively normal linear distribution, and rather high coefficients were observed on the plots. In other words, the samples of both the calibration and validation datasets were similar. Thus, the relative standard deviation (RSD) of validation, which is one of the other detection tools for accuracy, actualized as 0.1864% and 0.4581% for both essential oil and 1-8 cineol content, respectively, in the PLSR model. It refers to the errors after bias and slope corrections (Lequeue et al., 2016) and seems to be quite reasonable for the high accuracy of the prediction and robustness of the model. The RPD is a non-dimensional statistic for the evaluation of a NIR spectroscopy calibration model (Esbensen et al., 2014). However, Williams (2001) emphasized that the use of RPD with R^2 will be more meaningful for evaluating the efficiency of NIRS models. The RPD value in the PLS model was calculated for essential oil and 1-8 cineol content (3.5862 and 8.4115, respectively; Table 2). According to the classification of Chang et al. (2001), when the RPD > 2 and the measured vs. predicted R^2 is between 0.80 and 1.0, it means that NIRS prediction is in the best category. It was foreseen that when the calculated RPD scale was less than 1.5, the calibration could not be used while values of RPD were greater than 3.0, and the model was considered excellent (Sans et al., 2020). Therefore, the NIR-based models generated for the prediction of both essential oil and 1-8 cineol content of bay laurel leaves in the current study were quite reasonable with the appropriate RPD values.

To estimate the number of primary PCs, the software output of PRESS (predicted residual error sum of squares) was considered. At the point where PRESS is at a minimum, the corresponding number of PCs is optimum (Yu et al., 2017). Thus, the primary PCs were determined to be three and two in the PLSR models for both essential oil and 1-8 cineol content, respectively. If too many PCs are selected, in this case, only the calibration spectra will be identified correctly, and the useful spectra in the validation set will be ignored (Büchi, 2013). According to the score plot of PC1 and PC2 (Figure 5), the PLSR model was able to differentiate bay laurel genotypes very well by the first component with respect to related properties, despite the slightly overlapped clusters for essential oil content.



(a) (b)

FIGURE 5. Score plots of essential oil (a) and 1-8 cineol content (%) (b) of ground leaf samples of bay laurel genotypes using FT-NIRS data.

This result is extremely important, as it indicates future predictions of the developed model. Furthermore, we studied independent bay laurel genotypes to test the prediction ability of the model (Figure 6).

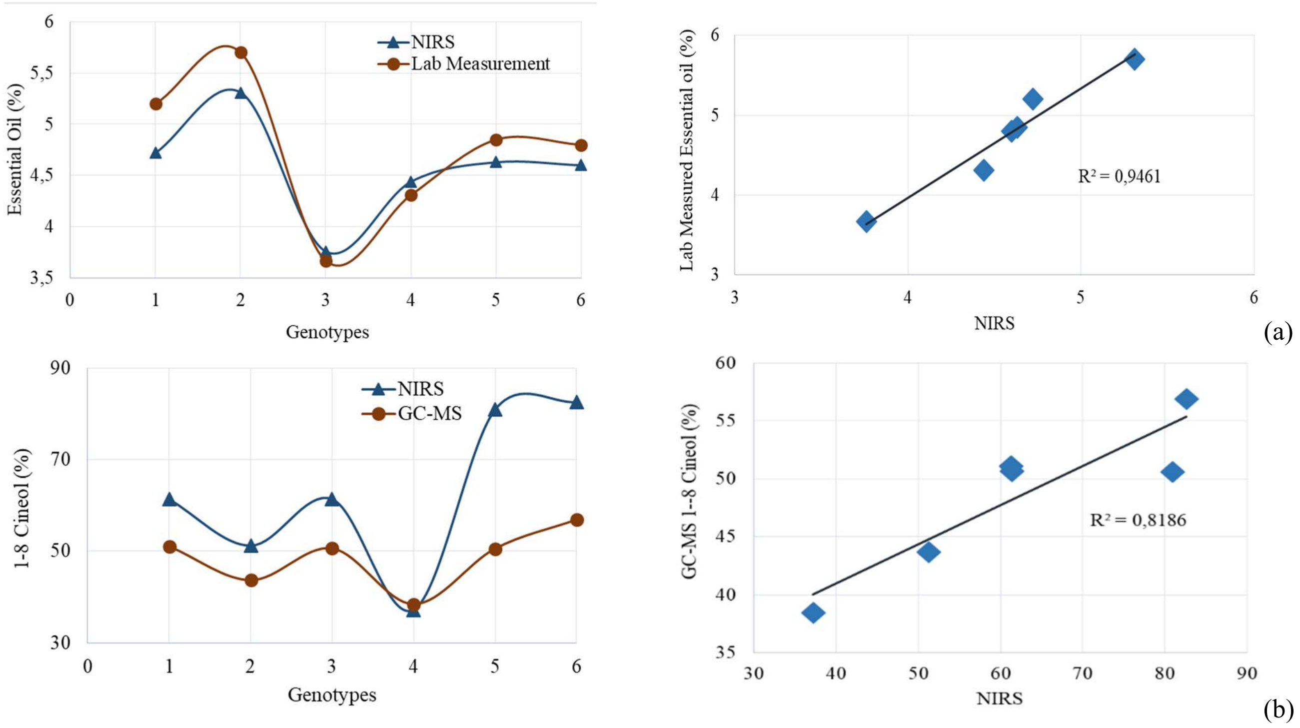


FIGURE 6. The accuracy of the NIRS calibration model developed for essential oil (a) and 1-8 cineol content (b) of ground leaf samples of bay laurel genotypes.

The comparison of the data obtained from both conventional (chemical and GC-MS) and NIRS showed that the accuracy or the functionality of the developed calibration models was sufficient to select an individual within a huge population, as hypothesized. The bias values of 1-8 cineol content were negative in the PLSR model (Table 2), indicating that the NIRS values of 1-8 cineol content of bay laurel genotypes were greater than those from GC-MS, as emphasized by Liu et al. (2008). However, the NIRS values of the essential oil content of the independent samples were slightly lower than the chemical values (Figure 6), while the bias value was positive.

In conclusion, the NIRS model developed for the main substances of bay laurel is rather promising for the efficient screening of a huge population without using reagents. The predicted and conventional data were comparable; therefore, the prediction accuracy was reasonable for both essential oil ($R^2 = 0.94$) and 1-8 cineol content ($R^2 = 0.81$), with highly acceptable RMSEP values of 0.1847% and 0.4518%, respectively. However, NIR spectroscopy allows the evaluation of many other properties in the same application at the same time if enough reference values are available. Thus, versatile selection is possible. This provides time and labor efficiency for both scientists and the industry. Furthermore, the spectral and reference data of any property used in the NIRS model are this type of database and open to continuous development for further studies.

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