

Evaluation of the influence of temperature and time exposure on Peruvian Maca (*Lepidium meyenii*) flour samples through NIR spectroscopy and chemometrics

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Abstract

In this study, the three most known varieties of the Peruvian Maca (*Lepidium meyenii*), yellow, red, and black, were submitted to heating (from 25 to 200°C) for one hour, during 10-minute intervals and measured by near-infrared (NIR) spectroscopy, after 24 hours resting in a desiccator, to evaluate the changes at each temperature. The study focused on changes related to heating for considering new applications on Maca-enriched food products such as bread, cakes, and cookies. To perform the experiment, 9 portions of 1g of each sample remained in an oven at each temperature (25, 50, 100, 150, 160, 170, 180, 190, and 200°C) for different times (10, 20, 30, 40, 50, and 60 minutes). The multivariate curve resolution with alternating least squares (MCR-ALS) was applied to interpret the results. In general, it was observed that spectral variations start to occur from heating samples from above 100°C. The recovered signals indicated that most spectral variations occurred in regions assigned to N-H vibrations, suggesting that heating influences the protein and/or macamides, which are responsible for most of the properties attributed to the Peruvian Maca. The yellow Maca phenotype was the most susceptible to spectral variations after heating, while the black Maca displayed the slowest spectral transformations. This study may encourage deeper investigations into the use of Maca-derived foods regarding their health-beneficial effects after heating to indicate the limitations of Maca applications.

1. Introduction

Most of the appeal regarding the Peruvian Maca (*Lepidium meyenii* Walp.) is related to medicinal (da Silva Leitão Peres et al. 2020), fertility, and reproductivity effects (Tafari et al. 2021), even though there are lots of controversial claims (Beharry and Heinrich 2018). Popularly, hypocotyls are considered the edible part of the Maca tubers, which are transformed into flour to be commercialized. This is a critical step, and most of the research is devoted to the post-harvest practices relating the drying process to the flour composition (Yábar et al. 2011; Esparza et al. 2015; Pan et al. 2016; Peng et al. 2021) or the effect of specific Maca-derived bioactive products (Geng et al. 2018; Carvalho and Ribeiro 2019; Fu et al. 2021; Korkmaz et al. 2021). Nonetheless, fewer efforts have been made to investigate the quality of the commercialized flour regarding its quality. More recently, our research group investigated different Peruvian Maca flour, focusing on the capability of discrimination according to adulteration (Rodrigues et al. 2021). However, this study should be considered an initial pilot study due to the different possibilities of composition variation influenced by different aspects linked to Maca cultivation (Valqui-Peña et al. 2021).

The concerns regarding Maca quality are supported by allegations over the lack of androgenic activity effectiveness of these food-derived products (Bogani et al. 2006; Beharry and Heinrich 2018). In this sense, besides the importance of evaluating the post-harvest processes, the limits of their applications in which the bioactive substances are preserved must be explored to allow for the optimized consumption of this food and to provide answers regarding the Maca potential. One possibility to explore it is by simulating a process in which Maca flour can be involved, such as in the production of Maca-enriched cakes (Tormena et al. 2017) or the use of the so-called functional foods (Puoci et al. 2013). These food

product preparations demand exposure to temperatures above 100°C during different time periods; therefore, tracking their composition changes is a challenging duty.

Monitoring the effect of processing food, such as cooking, is a task that can be done using different strategies, and a feasible approach can be found by employing non-invasive, non-destructive measurements using spectroscopic techniques, which, although they are less informative than the separation ones, such as chromatography coupled to mass detection systems, they can, however, be easily used as a screening to design further studies better. Near-infrared (NIR) spectroscopy has already been used in studies regarding Peruvian Maca adulteration (Zeng and Zheng 2021; Rodrigues et al. 2021), and geographical origin (Wang et al. 2016), besides NIR through multispectral imaging (Zhang et al. 2020). On the other hand, many applications of NIR coupled with chemometric tools have already been described in previous literature (Ozaki et al. 2007; Beć et al. 2020; Kunze et al. 2021; de Souza Zangirolami et al. 2023).

To evaluate Peruvian Maca sample composition changes over time and temperature, the multivariate curve resolution alternating least squares (MCR-ALS) method can be a suitable tool since it was developed to solve general mixture analysis problems by resolving simultaneously the spectral signals and the respective relative concentrations of the chemical constituents of the analyzed samples (de Juan and Tauler 2016a). In this way, the spectral signals produced at different temperatures can be easily monitored during a period of time in which the sample is exposed to different conditions like temperature changes. In this sense, the influence of temperature and exposure time in flour samples of Peruvian Maca can be investigated using NIR spectroscopy and chemometrics, allowing for the possible screening of a more resistant phenotype and identifying at what temperature the changes in spectra start to occur, and therefore, to check the reasons underlying on the observed spectral changes.

2. Materials and Methods

2.1 Sampling

All the Maca samples were harvested between July and August 2019 and stored in a cooperative of an indigenous community in Santiago De Huari, in the Bolivian Andes. The flour was obtained from a homogenized mixture of Peruvian Maca cultivated around Santiago De Huari. The flour samples were separated as 1 kg from the yellow, 2 kg from the red, and 2 kg from the black phenotype. After processing, the flour was vacuum-packed to be transported to Brazil. The analysis was conducted in the Post-Graduation Program in Food Technology (PPGTA) laboratory at the Federal University of Technology of the Paraná State, Campus Campo Mourão. The samples were stored in a freezer at a temperature of -10°C until their analysis was carried out in mid-2021. It is important to remark that these Maca roots were prepared using traditional post-harvest practices, which consist of exposing them to environmental conditions, i.e., temperatures varying from -20 to 14°C, high UV light exposure, and low precipitation levels.

2.2 Sample preparation and measurements

The Maca flour samples were subdivided into 3 subsamples, each being measured in triplicates. Each sample was fractionated into 1g portions for each of the 9 different temperatures (25, 50, 100, 150, 160, 170, 180, 190, and 200°C), thus totaling 18 samples for each temperature, and the respective times and portions were transferred to Petri dishes to be subjected to drying in an oven with air circulation (CIENLAB and New Lab) for a period of 1 hour in order to dehydrate (remove water/moisture). Every 1g portion sample remained for a time corresponding to 10, 20, 30, 40, 50, and 60 minutes at each temperature. The two experimental factors will be, therefore, temperature and time.

The samples were heated, and each temperature and time was performed in triplicate, totaling 162 (9 temperatures x 6 times x 3 replicates). After heating, the samples were transferred to flasks (amber glass) and stored in desiccators for cooling and stored until the spectra measurement, which was done 24 hours after storage. Each sample was analyzed in triplicate, totaling 486 (162 samples x 3 replicates) spectra.

2.3 Spectra measurements

After 24 hours of cooling inside a desiccator, each portion of the Maca flour sample was placed in a glass tube to be measured by near-infrared spectroscopy, specifically in the region between 900 to 1700 nm, using a *MicroNIR 1700* spectrometer (VIAVI ®). Each spectrum was an average of 50 scans, with 6 nm resolution. The calibration was done using a *spectralon* reflectance sphere (99% reflectance) to calibrate the 100% reflection, and the dark was calibrated by turning off the equipment light source. The measurements were taken with the equipment temperature around 45 degrees, following the manufacturer's guidance.

2.4 Data Arrangement and Chemometric Analysis

The data was organized according to the structure presented in Table 1 for each Maca phenotype, which was evaluated separately. A $\mathbf{G}_{(r \times v)}$ matrix was organized with " r " rows, representing each spectrum, and " v " columns, representing each variable. Thus, the data matrix was composed of 486 rows (spectra) and 125 columns (wavelength variables), considering that each row displayed in Table 1 was measured in triplicate. In this sense, the effect of each temperature was measured 9 times. The $\mathbf{G}$ matrix was then row-wisely averaged to generate the $\mathbf{D}_{(54 \times 125)}$ matrix, in which each row was the mean of 9 rows (spectra) for each time interval per Maca phenotype. The evaluation of the variance per variable indicated that the spectra extremes were excessively influencing the principal components (PCs), the spectra were cut to include only the spectral region from 932.88 to 1657.62 nm. After this wavelength adjustment, the spectra were processed by MCR-ALS.

Table 1
Spectra acquisition and organization regarding the time
and temperature for each sample

Temperature (°C)	Time (min)					
	10	20	30	40	50	60
25	A1	A1	A1	A1	A1	A1
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
50	A1	A1	A1	A1	A1	A1
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
100	A1	A1	A1	A1	A1	A1
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
150	A1	A1	A1	A1	A1	A1
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
160	A1	A1	A1	A1	A1	A1
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
170	A1	A1	A1	A1	A1	A1
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
180	A1	A1	A1	A1	A1	A1
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
190	A1	A1	A1	A1	A1	A1
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
200	A1	A1	A1	A1	A1	A1

Temperature (°C)	Time (min)					
	10	20	30	40	50	60
	A2	A2	A2	A2	A2	A2
	A3	A3	A3	A3	A3	A3
<i>Total: 162 spectra</i>						

Table 1. Spectra acquisition and organization regarding the time and temperature for each sample

The $\mathbf{D}_{(54 \times 125)}$ matrix was corrected for multiplicative scattering by asymmetric least squares from the PLS Toolbox ® (Eigenvector Research 2023). After that, the MCR-ALS (multivariate curve resolution with alternating least squares) (de Juan et al. 2014; de Juan and Tauler 2016b) was applied through the non-negativity constraints (using the *fnnls* algorithm (Bro and De Jong 1997) implemented to the concentration and spectral profiles, besides normalizing the spectra by the Euclidean norm. The MCR-ALS code was available on the MCR-ALS webpage, whose description and GUI can be found elsewhere (Jaumot et al. 2015). All the data handling was performed using the Matlab R2019a ® computational environment. MCR-ALS methodology was used to resolve the signatures of the different spectral components, that is, to separate the spectral signals and recover the respective concentration profiles (variation of the concentration of each spectral contribution in relation to the heating temperature and to the time of exposure of the sample).

The MCR-ALS bilinear factor decomposition of data matrix $\mathbf{D}$ follows the Eq. (1):

$$\mathbf{D} = \mathbf{CS}^T + \mathbf{E}$$

1

In the equation, the matrix $\mathbf{C}_{(r \times n)}$ describes how the contribution of the n components involved in the process, which are spectroscopically active, changes in the different rows of the data matrix (concentration profiles), while the $\mathbf{S}_{(n \times c)}^T$ matrix describes how the spectral responses of these components change in the columns of the data matrix (pure spectra profiles). The $\mathbf{E}_{(r \times c)}$ matrix gives the residuals with the data variance unexplained by the bilinear model given in Eq. 1 (de Juan et al. 2014).

The data matrix ‘chemical rank’ (mathematical rank in absence of noise) was estimated through principal components analysis (PCA), which suggested the presence of 3 different spectroscopically active components. Initial estimates of these components were obtained from the ‘purest’ samples (rows of the data matrix), implemented in the MCR-ALS interface (Jaumot et al. 2015) similar to the previously proposed method by Windig et al (Windig 1997).

3. Results and Discussions

The results are presented to each Maca phenotype separately to be compared afterwards.

3.1. Yellow Peruvian Maca

As described in the material and methods section, PCA was applied to estimate the number of spectrally active components. Figure 1 presents the NIR (A) raw spectra and (B) the NIR preprocessed spectra obtained from the yellow Maca flour.

Figure 1. (A) Raw and (B) processed NIR spectra obtained from yellow Maca flour.

The MCR-ALS was applied to evaluate the stability of the NIR signal with time/temperature. The NIR spectra and respective relative concentrations resolved by MCR-ALS were normalized (divided by their maximum intensity). The spectra are presented in Fig. 2 – (A), with color-respective concentrations represented by “*” symbols in Fig. 2 – (B). It is noteworthy to mention that the intervals among temperature changes shown in Fig. 2 – B comprise 1 (one) hour of heating, starting with the green-dashed vertical lines (I), indicating 10 minutes of thermal exposition, and ending in the black-dashed vertical lines (I), which marks 60 minutes of thermal exposition. Also, each vertical minor dotted line represents 10 minutes of heating.

Figure 2. MCR-ALS recovery (A) spectra and (B) relative concentrations for the yellow Maca flour heating evaluated by NIR spectroscopy.

Thus, the spectral profiles presented in Fig. 2 – A correspond to the relative concentration changes in Fig. 2 – B. Since portions of the samples were heated separately to avoid the loss of mass effect, it is noticeable that the first spectrum (measured after 10 minutes of heating at each temperature) may appear at all measured temperatures. In Fig. 2 – B, from 25 to 100°C, there is only the blue signal (*), indicating that no changes were observed and only the spectrum represented by the blue line was detected in this temperature range. Then, when the sample is heated at 150°C, the profile represented by the red line starts to appear, increasing intensity after 40 minutes at this temperature. The intensity loss of the blue spectral signal is more important starting at the temperature of 170°C, a temperature at which, from 20 minutes onwards, the predominance of the signal represented by the red color (*) is observed, even though a signal represented by the yellow color (*) starts to take place.

The changes indicated by blue and red symbols are related to vibrational displacements, mainly in the region from 980 to 1100 nm, and a remarkably intense vibration on about 1180–1200 nm, all assigned to the second overtone of N-H primary and secondary stretching vibrations (Workman Jr., Jerry ; Weyer 2012). Also, important changes were detected in the region from 1300 and 1400 nm, assigned to the first overtones of O-H groups vibration, besides the N-H first overtone vibrations of primary amine (Workman Jr., Jerry ; Weyer 2012). These spectral regions can be assigned with changes in functional groups in proteins (Ishigaki and Ozaki 2020; Beć et al. 2020) and amides (Jović et al. 2020) analyzed by NIR spectroscopy. Therefore, heating can influence protein and/or macamide contents related to the Peruvian

Maca so-called benefits. In this sense, there is a need to investigate if these changes can limit the use of this plant as a food when it is processed by heating.

3.2. Red Peruvian Maca

A similar strategy was used for the NIR spectra obtained from the red Maca flour, selecting the spectral region from 932.88 to 1657.62 nm. Figure 3 presents the NIR (A) raw spectra and the NIR preprocessed spectra (B) obtained from the red Maca flour.

The PCA indicated that there were 3 different spectroscopically active components, which were used to get spectral initial estimates (obtained as previously (Windig 1997)). MCR-ALS results are shown in Fig. 4 (A) for the spectra and (B) color-relative concentrations, respectively.

Figure 3. (A) Raw and (B) processed NIR spectra obtained from red Maca flour.

Like the behavior observed for the heating of the yellow Maca variety, the initial detected signal (represented in blue) started to change at 150°C. However, in this case, the red Maca showed a less pronounced increase in the intensity of the signal represented in red. The main changes are observed at the spectral band around 1120–1140 nm, a region assigned to the 2nd overtone of primary and secondary N-H stretching vibration, besides the 4th overtone for secondary amides (Workman Jr., Jerry ; Weyer 2012). Also, the evolution from the blue profile to the red implies an increasing intensity of the vibration band at 1540 nm, besides the appearance of a band at 1640 nm. This region can be assigned to the 1st overtone for primary and secondary N-H stretching (Workman Jr., Jerry ; Weyer 2012) groups.

Figure 4. MCR-ALS recovery (A) spectra and (B) relative concentrations for the red Maca flour heating, evaluated by NIR spectroscopy.

From 160°C to higher temperatures, the initial spectral profile represented in blue turns more unstable as the heating exposure increases, being converted into the signal illustrated in red. This transformation can be observed from 20 minutes of heating at 160°C and prevailing from 170 to 180°C. From 190°C to 200°C a slight transformation in the spectra can be noticed from 1480 to 1657.62 nm, implying a decrease in the vibrational intensities and a displacement of the band, previously at 1640 nm, to 1600 nm. This is a region assigned to the 3rd overtone of carbonyl (C = O) vibration, besides being the region of the 1st overtone for primary and secondary N-H stretching (Workman Jr., Jerry ; Weyer 2012). All those signals are consistent with changes in the structure of proteins and amides, suggesting that heating can affect the food properties of red Maca derivatives.

3.3. Black Peruvian Maca

In this case, the application of PCA also suggested the presence of 3 major components whose spectra were initially estimated as previously (Windig 1997) to be used afterwards in the MCR-ALS analysis. Figure 5 shows the NIR (A) raw and (B) preprocessed spectra obtained from the black Maca flour.

Figure 5. (A) Raw and (B) processed NIR spectra obtained from the black Maca flour.

The MCR-ALS results are shown in Fig. 6 - (A) for the resolved spectral signals and Fig. 6 - (B) for the respective concentration profiles. Similarly to the other investigated phenotypes, the most remarkable changes start at 150°C, even though slight changes could be noticed at 100°C (by the presence of relatively low concentrations in the profile represented in red).

Figure 6. MCR-ALS recovery (A) spectra and (B) relative concentrations for the black Maca flour heating, evaluated by NIR spectroscopy.

It is notorious that the spectral profiles resulted to be different from one phenotype to another, indicating that the substances responsible for the measured spectral signals are different in each case. However, in the case of the black Maca, the red and yellow spectral profiles seem very similar, and the hypothesis of being the same signal cannot be discarded. Most of the possible differences between red and yellow spectra are concentrated over the region above 1580 nm, which is a region attributed to primary and secondary overtones for the N-H stretching (Workman Jr., Jerry ; Weyer 2012). A slight difference between 1140–1160 nm, attributed to the 4th overtone of secondary amides, can be observed, although its magnitude is less evident.

To compare the effects observed in the analysis of the different types of Maca flour samples, Fig. 7 shows the comparison of the heating effect considering the relative concentration slopes after using the smoothing Savitzky-Golay algorithm (Savitzky and J. E. Golay 2002) and the recalculation of the corresponding spectra profiles. Through this evaluation, the spectra obtained for the Yellow Maca phenotype are the most susceptible to temperature variations, and the heating influences more intensely this Maca from about 100°C compared to the other two studied phenotypes.

Figure 7. smoothed concentrations and respective spectra of the components resolved by MCR-ALS. Yellow Maca phenotype (A) spectra and (B) relative smoothed concentrations. Red Maca phenotype (C) spectra and (D) relative smoothed concentrations. Black Maca phenotype (E) spectra and (F) relative smoothed concentrations.

It is noticeable that the initial spectrum profile for the three phenotypes is quite similar regarding the most important vibrational regions. The red Maca shows heating effects on the spectra after 150°C, although the slopes for the observed transformations suggest that the concentration changes happen faster than for the yellow and black Maca phenotypes, even though the changes in the yellow Maca appear before compared to the red and black Maca phenotypes. Despite these spectral variations in the black Maca profile occurring before any other phenotype, the concentration changes happen slower than for the yellow and red Macas. In this sense, this Maca seems more heat-resistant than the other phenotypes.

4. Conclusions

This work analyzes the influence of heating (temperature) on the NIR spectral signals of flour samples from 3 different phenotypes of the Peruvian Maca plant. By evaluating the spectral regions affected by increasing temperatures, the results suggest that substances such as proteins and/or macamides are

impacted by heating, which is deduced from the changes in the vibrations attributed to N-H bonds in all samples.

Spectral changes can be detected by heating Maca from temperatures above 100°C, being this effect more intense in the yellow Maca phenotype than in red and black Macas. On the other hand, spectral variations on red Maca can be detected only after heating from 150°C to higher temperatures. Nonetheless, the changes in the red Maca occurred slightly faster than in yellow and black phenotypes after starting. Also, the effects occurred slightly slower in the black Maca than in the red and yellow Macas.

In conclusion, this study's results will encourage discussions regarding the use of heating in processing Maca-derived food to confirm the possible Maca expected beneficial effects.

Declarations

Conflicts of Interest: The authors declare no conflict of interest.

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References

1. Beć KB, Grabska J, Huck CW (2020) Near-Infrared Spectroscopy in Bio-Applications. *Molecules* 25:2948. <https://doi.org/10.3390/molecules25122948>

2. Beharry S, Heinrich M (2018) Is the hype around the reproductive health claims of maca (*Lepidium meyenii* Walp). justified? *J Ethnopharmacol* 211:126–170. <https://doi.org/10.1016/j.jep.2017.08.003>
3. Bogani P, Simonini F, Iriti M et al (2006) *Lepidium meyenii* (Maca) does not exert direct androgenic activities. *J Ethnopharmacol* 104:415–417. <https://doi.org/10.1016/j.jep.2005.09.028>
4. Bro R, De Jong S (1997) A fast non-negativity-constrained least squares algorithm. *J Chemom* 11:393–401. [https://doi.org/10.1002/\(SICI\)1099-128X\(199709/10\)11:5<393::AID-CEM483>3.0.CO;2-L](https://doi.org/10.1002/(SICI)1099-128X(199709/10)11:5<393::AID-CEM483>3.0.CO;2-L)
5. Carvalho Fv, Ribeiro PR (2019) Structural diversity, biosynthetic aspects, and LC-HRMS data compilation for the identification of bioactive compounds of *Lepidium meyenii*. *Food Res Int* 125:108615. <https://doi.org/10.1016/J.FOODRES.2019.108615>
6. da Silva Leitão Peres N, Cabrera Parra Bortoluzzi L, Medeiros Marques LL et al (2020) Medicinal effects of Peruvian maca (*Lepidium meyenii*): a review. *Food Funct* 11:83–92. <https://doi.org/10.1039/C9FO02732G>
7. de Juan A, Jaumot J, Tauler R (2014) Multivariate Curve Resolution (MCR). Solving the mixture analysis problem. *Anal Methods* 6:4964–4976. <https://doi.org/10.1039/C4AY00571F>
8. de Juan A, Tauler R (2016a) Multivariate Curve Resolution-Alternating Least Squares for Spectroscopic Data. pp 5–51
9. de Juan A, Tauler R (2016b) Data Handling in Science and Technology 30:5–51. <https://doi.org/10.1016/B978-0-444-63638-6.00002-4>. Multivariate Curve Resolution-Alternating Least Squares for Spectroscopic Data
10. de Souza Zangirolami M, Moya Moreira TF, Leimann FV et al (2023) Texture profile and short-NIR spectral vibrations relationship evaluated through Comdim: The case study for animal and vegetable proteins. *Food Control* 143:109290. <https://doi.org/10.1016/j.foodcont.2022.109290>
11. Eigenvector Research Inc (2023) PLS_Toolbox 9.2.1
12. Esparza E, Hadzich A, Kofer W et al (2015) Bioactive maca (*Lepidium meyenii*) alkamides are a result of traditional Andean postharvest drying practices. *Phytochemistry* 116:138–148. <https://doi.org/10.1016/J.PHYTOCHEM.2015.02.030>
13. Fu L, Wei J, Gao Y, Chen R (2021) Antioxidant and antitumoral activities of isolated macamide and macaene fractions from *Lepidium meyenii* (Maca). *Talanta* 221:121635. <https://doi.org/10.1016/J.TALANTA.2020.121635>
14. Geng HC, Yang DS, Chen XL et al (2018) Meyeniihydantoin A–C, three novel hydantoin derivatives from the roots of *Lepidium meyenii* Walp. *Phytochem Lett* 26:208–211. <https://doi.org/10.1016/J.PHYTOL.2018.06.010>
15. Ishigaki M, Ozaki Y (2020) Near-infrared spectroscopy and imaging in protein research. *Vibrational Spectroscopy in Protein Research*. Elsevier, pp 143–176
16. Jaumot J, de Juan A, Tauler R (2015) MCR-ALS GUI 2.0: New features and applications. *Chemometr Intell Lab Syst* 140:1–12. <https://doi.org/10.1016/J.CHEMOLAB.2014.10.003>

17. Jović B, Panić M, Radnović N et al (2020) Investigation of the surface interactions of selected amides with mesoporous silica using FTIR spectroscopy and hyperspectral imaging. *J Mol Struct* 1219:128562. <https://doi.org/10.1016/j.molstruc.2020.128562>
18. Korkmaz IO, Bilici C, Korkmaz S (2021) Sensory, pH, syneresis, water-holding capacity, and microbiological changes in homemade yogurt prepared with maca (*Lepidium meyenii*) powder and propolis extract. *Int J Gastron Food Sci* 23:100291. <https://doi.org/10.1016/J.IJGFS.2020.100291>
19. Kunze DCGC, Pastore TCM, Rocha HS et al (2021) Correction of the moisture variation in wood NIR spectra for species identification using EPO and soft PLS2-DA. *Microchem J* 171:106839. <https://doi.org/10.1016/j.microc.2021.106839>
20. Ozaki Y, McClure WF, Christy A (2007) Near-infrared spectroscopy in food science and technology
21. Pan Y, Zhang J, Li H et al (2016) Simultaneous Analysis of Macamides in Maca (*Lepidium meyenii*) with Different Drying Process by Liquid Chromatography Tandem Mass Spectrometry. *Food Anal Methods* 9:1686–1695. <https://doi.org/10.1007/s12161-015-0346-9>
22. Peng XR, Huang YJ, Liu JH et al (2021) ¹H qNMR-based quantitative analysis of total macamides in five maca (*Lepidium meyenii* Walp.) dried naturally. *J Food Compos Anal* 100:103917. <https://doi.org/10.1016/J.JFCA.2021.103917>
23. Puoci F, Malanchin R, Piangiolo C et al (2013) Maca flour: A powerful ingredient for functionally enhanced bread. *Int Food Res J* 20:1293–1300
24. Rodrigues H, Paulino H, Valderrama P, Março P (2021) The use of Chemometrics to Discriminate Sample Adulteration in Different Levels: the case of Peruvian Maca. *Brazilian J Anal Chem.* <https://doi.org/10.30744/brjac.2179-3425.AR-12-2021>. 8:
25. Savitzky Abraham JE, Golay M (2002) Smoothing and Differentiation of Data by Simplified Least Squares Procedures. *Anal Chem* 36:1627–1639. <https://doi.org/10.1021/ac60214a047>
26. Tafuri S, Cocchia N, Vassetti A et al (2021) *Lepidium meyenii* (Maca) in male reproduction. *Nat Prod Res* 35:4550–4559. <https://doi.org/10.1080/14786419.2019.1698572>
27. Tormena MML, de Medeiros LT, de Lima PC et al (2017) Application of multi-block analysis and mixture design with process variable for development of chocolate cake containing yacon (*Smallanthus sonchifolius*) and maca (*Lepidium meyenii*). *J Sci Food Agric* 97:3559–3567. <https://doi.org/10.1002/jsfa.8211>
28. Valqui-Peña D, Clark D, Gianoli E, Gonzáles WL (2021) Temperature regime influences accessions and effectiveness of germination promoters in the high-Andean crop maca. *Agron J* 113:2557–2566. <https://doi.org/10.1002/agj2.20688>
29. Wang YZ, Zhao YL, Zhang J, Jin H (2016) Study on application of NIR spectral information screening in identification of Maca origin. *Guang Pu Xue Yu Guang Pu Fen Xi* 36:394–400
30. Windig W (1997) Spectral data files for self-modeling curve resolution with examples using the Simplisma approach. *Chemometr Intell Lab Syst* 36:3–16. [https://doi.org/10.1016/S0169-7439\(96\)00061-5](https://doi.org/10.1016/S0169-7439(96)00061-5)

31. Workman J Jr, Weyer L (2012) Practical Guide and Spectral Atlas for Interpretive Near-Infrared Spectroscopy, 2nd edn. CRC Press, Boca Raton
32. Yábar E, Pedreschi R, Chirinos R, Campos D (2011) Glucosinolate content and myrosinase activity evolution in three maca (*Lepidium meyenii* Walp.) ecotypes during preharvest, harvest and postharvest drying. Food Chem 127:1576–1583.
<https://doi.org/10.1016/J.FOODCHEM.2011.02.021>
33. Zeng M-N, Zheng S-Y (2021) Near infrared spectroscopy combined with chemometrics to detect and quantify adulteration of maca powder. J Near Infrared Spectrosc 29:108–115.
<https://doi.org/10.1364/JNIRS.29.000108>
34. Zhang H-R, Liu C-H, Zhang J-K et al (2020) Qualitative Identification and Quantitative Analysis of Maca Adulteration Based on Multispectral Imaging Technology. Spectrosc Spectr Anal 40:152–156.
[https://doi.org/10.3964/j.issn.1000-0593\(2020\)01-0152-05](https://doi.org/10.3964/j.issn.1000-0593(2020)01-0152-05)

Figures

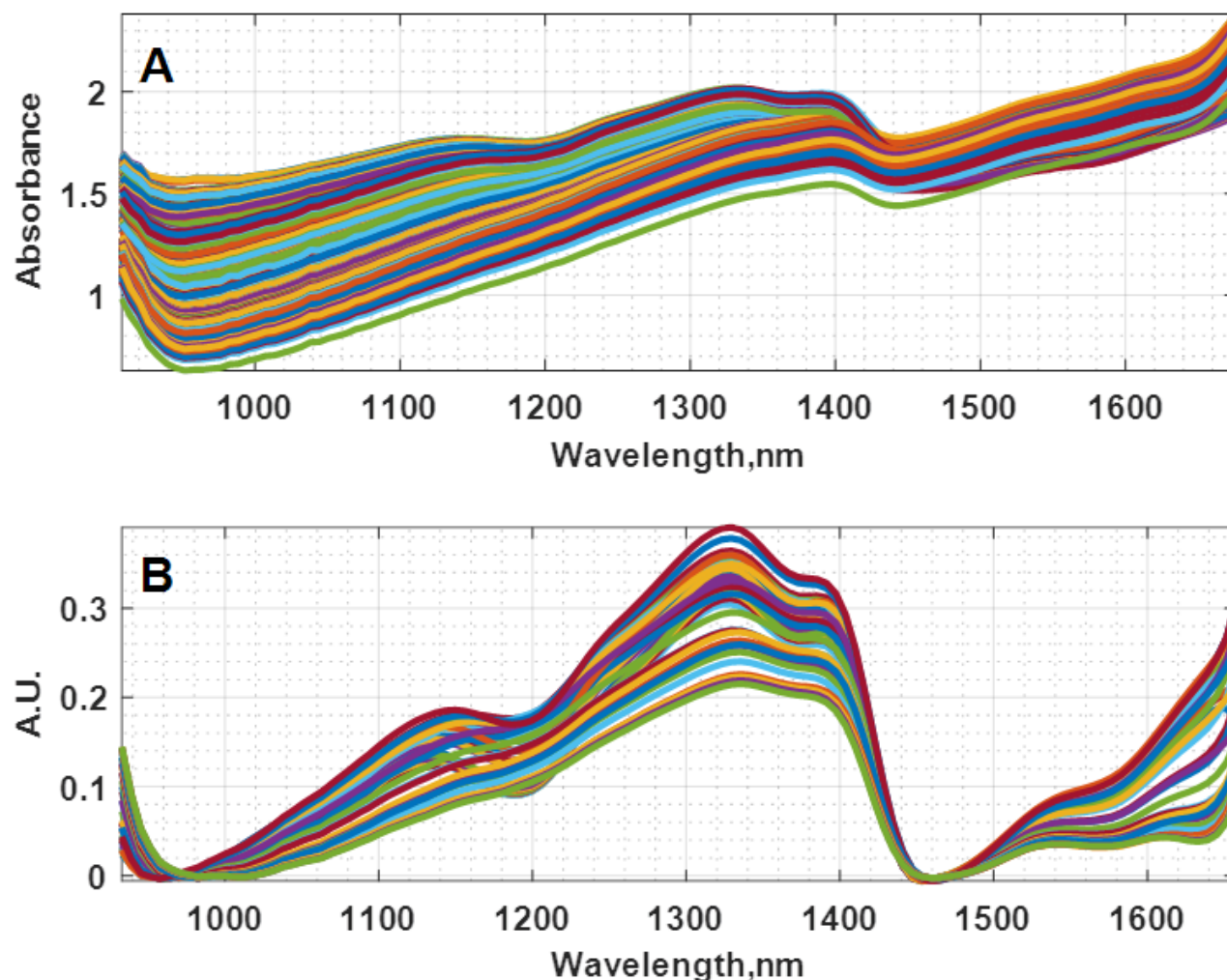


Figure 1

(A) Raw and (B) processed NIR spectra obtained from yellow Maca flour.

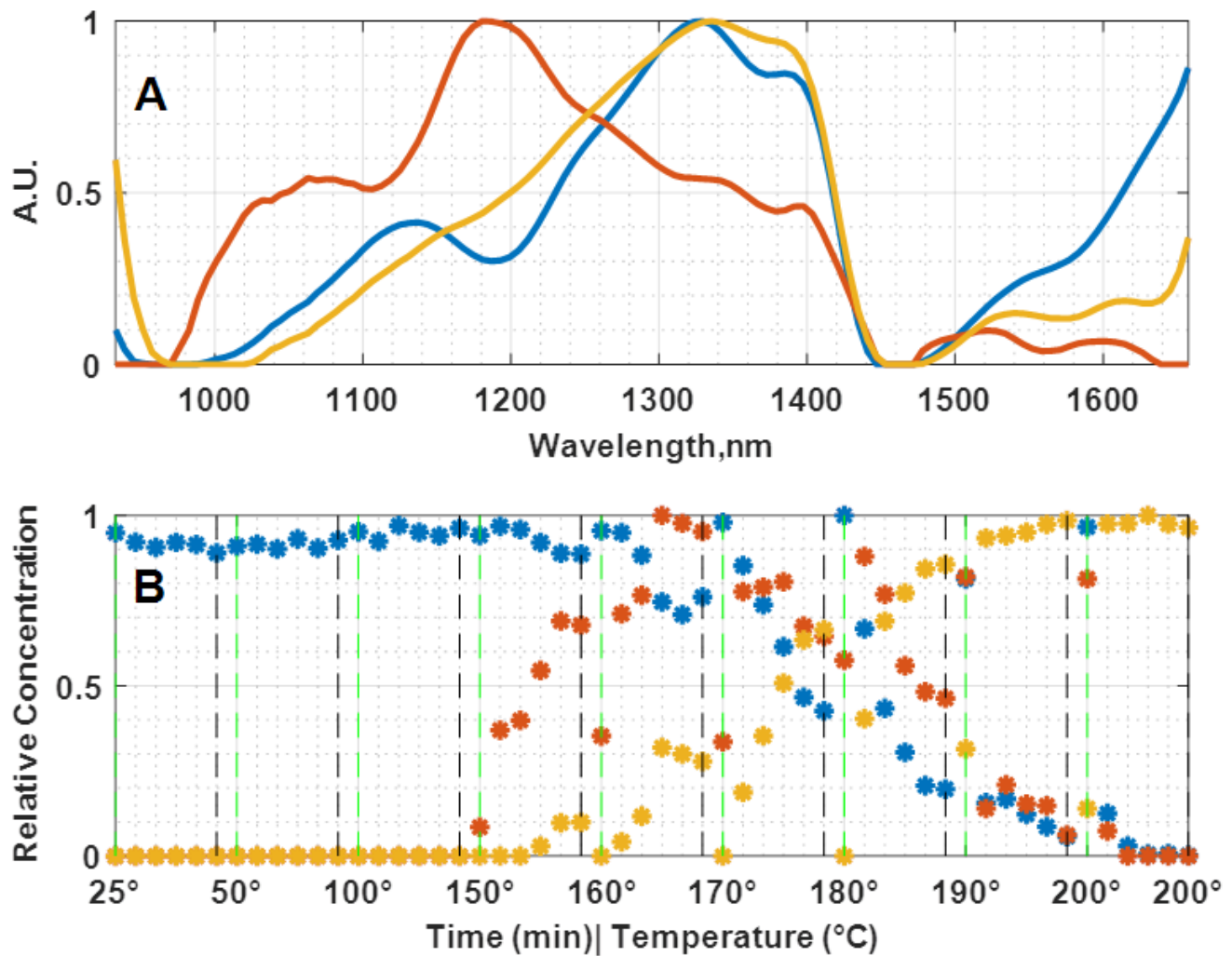


Figure 2

MCR-ALS recovery (A) spectra and (B) relative concentrations for the yellow Maca flour heating evaluated by NIR spectroscopy.

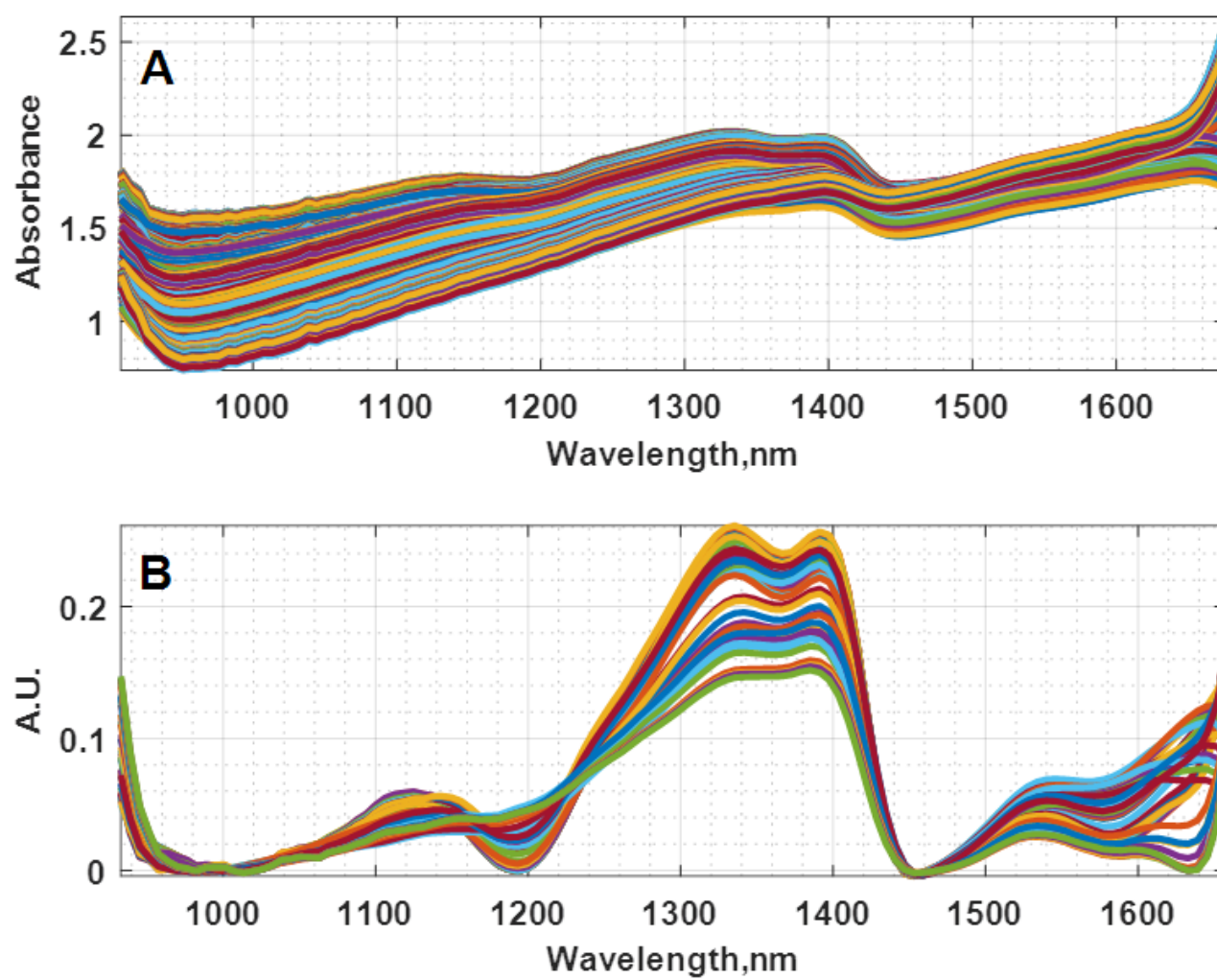


Figure 3

(A) Raw and (B) processed NIR spectra obtained from red Maca flour.

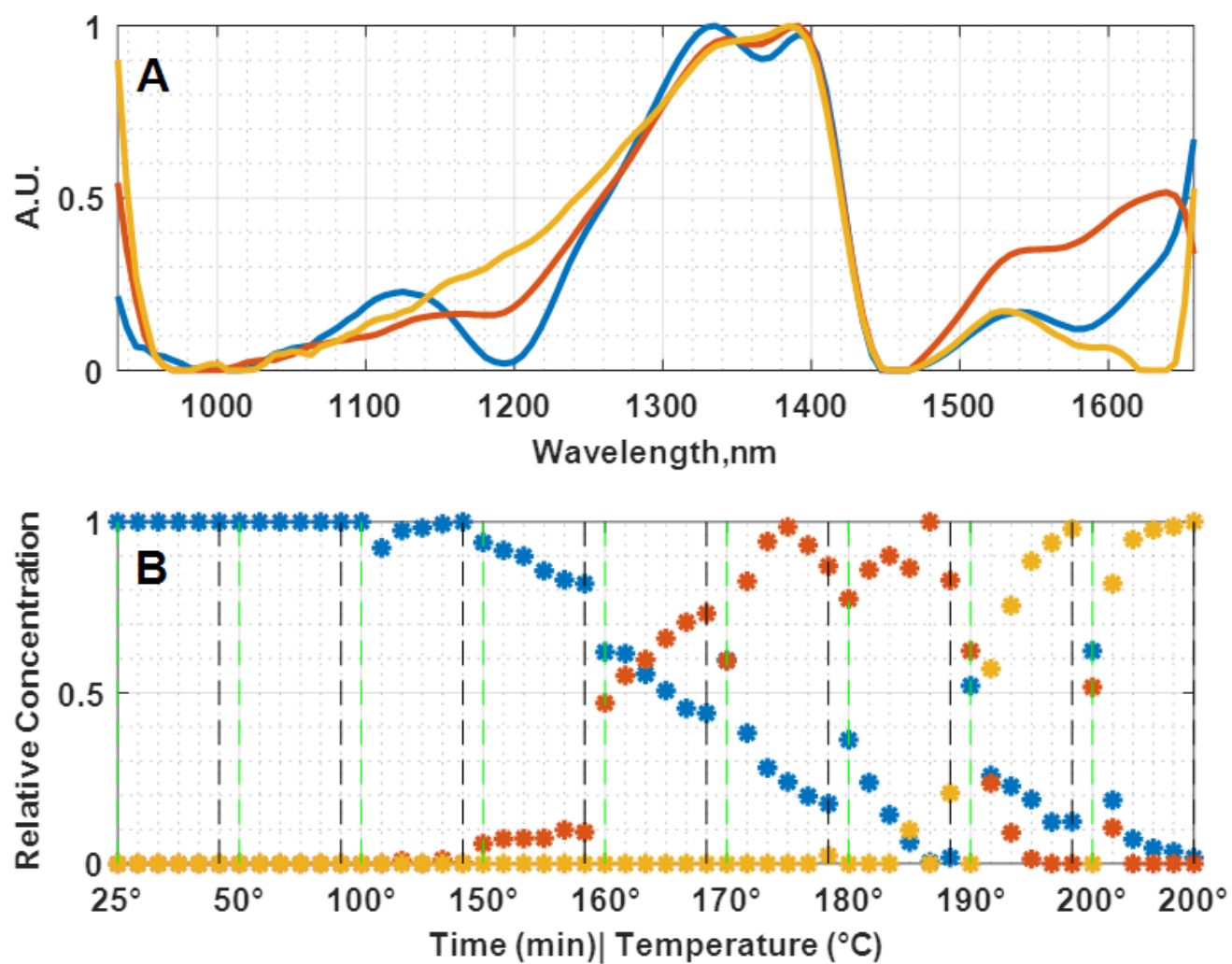


Figure 4

MCR-ALS recovery (A) spectra and (B) relative concentrations for the red Maca flour heating, evaluated by NIR spectroscopy.

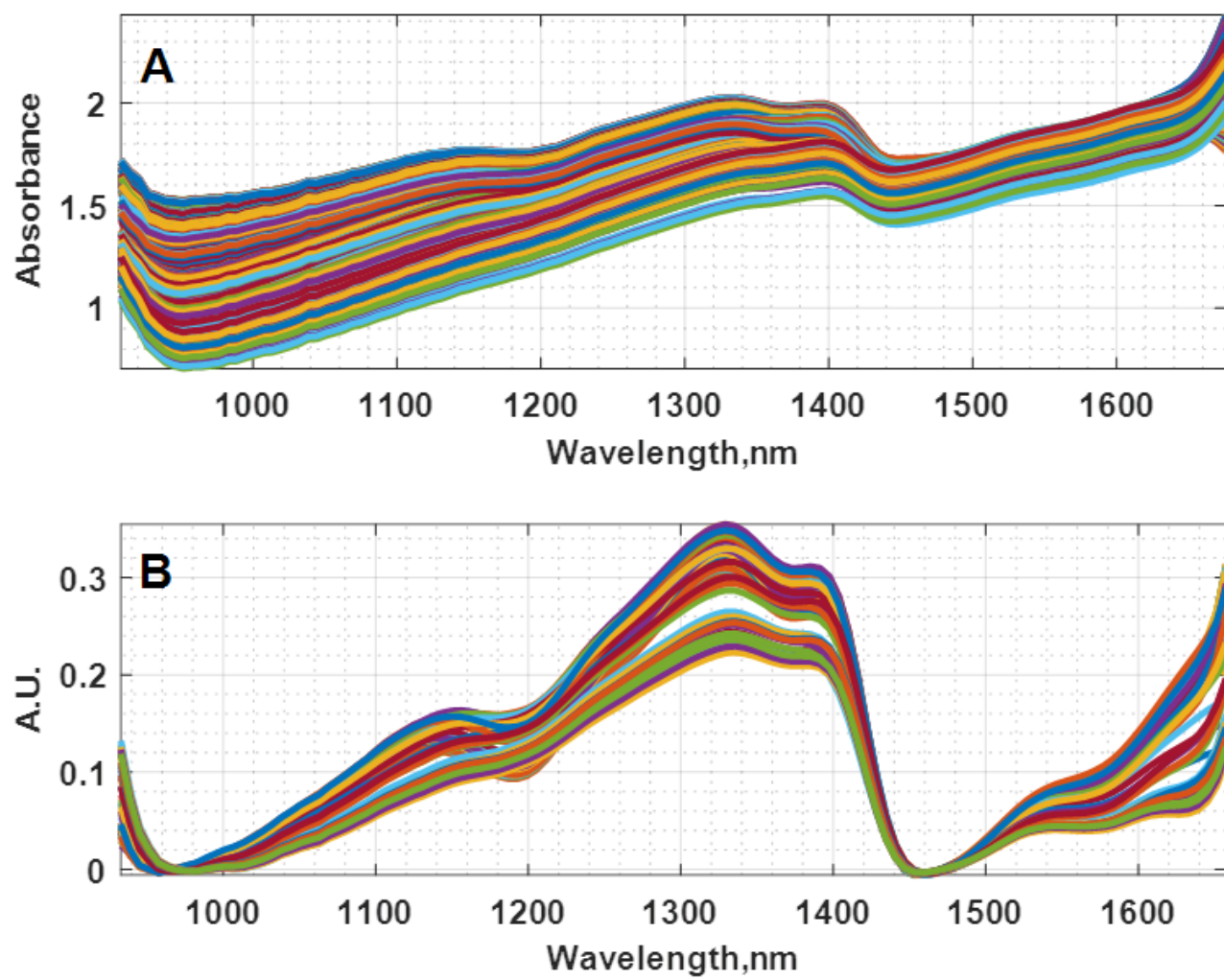


Figure 5

(A) Raw and (B) processed NIR spectra obtained from the black Maca flour.

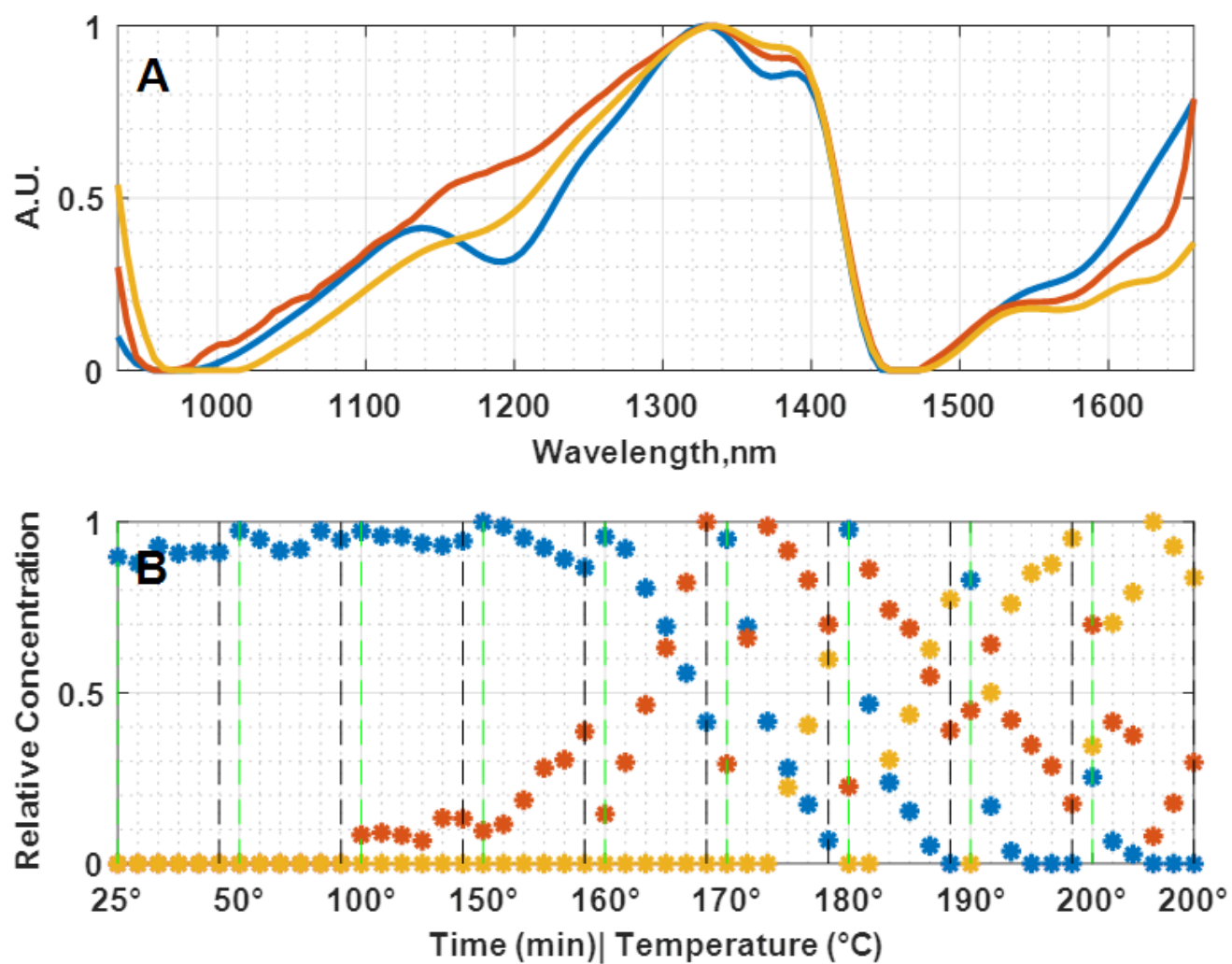


Figure 6

MCR-ALS recovery (A) spectra and (B) relative concentrations for the black Maca flour heating, evaluated by NIR spectroscopy.

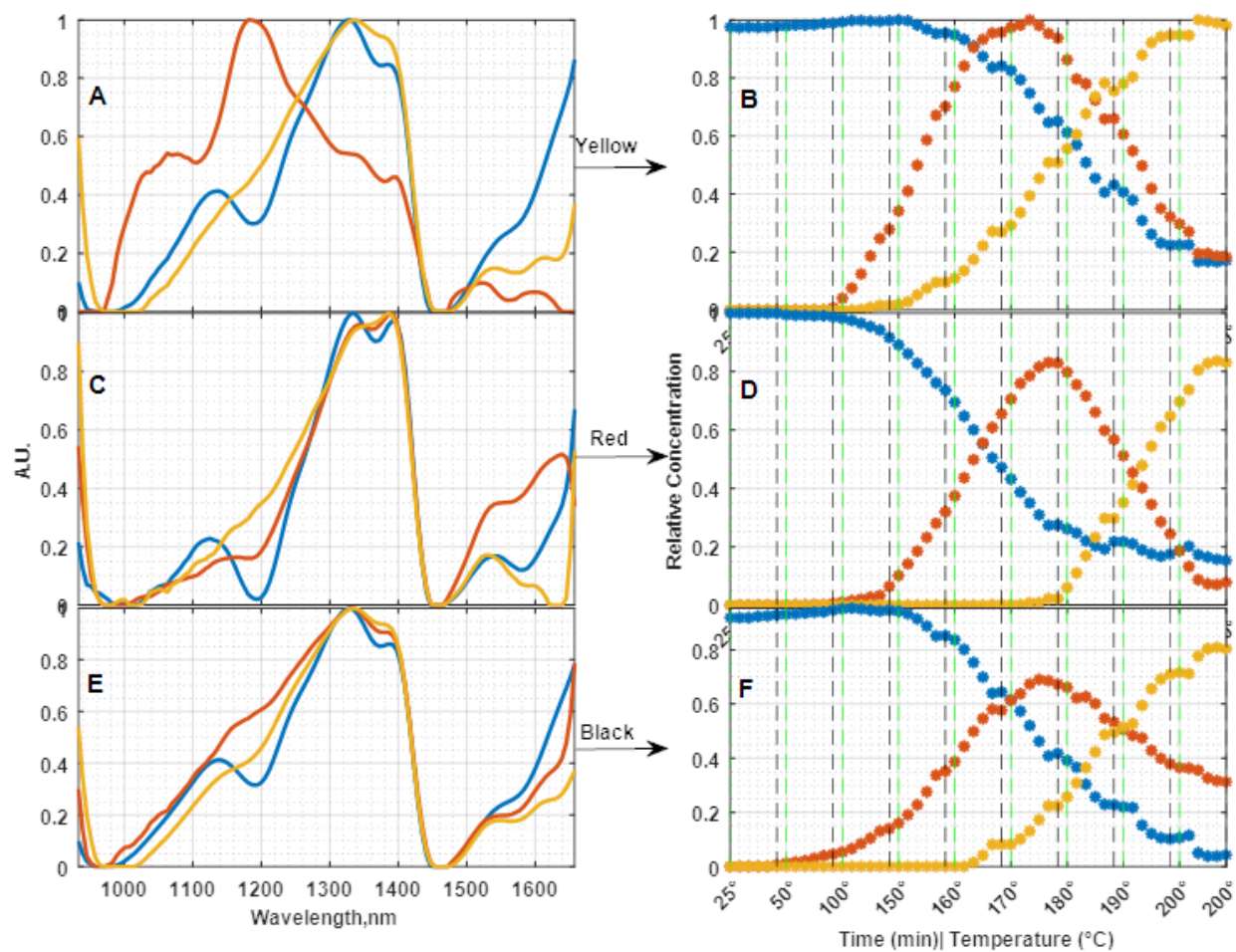


Figure 7

smoothed concentrations and respective spectra of the components resolved by MCR-ALS. Yellow Maca phenotype (A) spectra and (B) relative smoothed concentrations. Red Maca phenotype (C) spectra and (D) relative smoothed concentrations. Black Maca phenotype (E) spectra and (F) relative smoothed concentrations.