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The potential of near infrared (NIR) spectroscopy coupled to principal component analysis (PCA) for product and tanning process control of innovative leathers

Claudia Florio[1]✉, Antonio Medici[1,2]✉, Roberta Aveta[1], Leopoldo Esposito[1], Andrea Favazzi[3], Flavio Belvedere[4], Armando Zarrelli[2] & Maria Sarno[5,6]

The tanning industry faces significant challenges in quality control due to the complex transformations of leather, the anisotropic nature of the raw material, and the increasing adoption of innovative processes and materials in the tanning methods. In this study, it was possible to evaluate the applicability of a Non-Destructive Testing (NDT) technique such as near infrared (NIR) spectroscopy applied to the new generation of tanning products and processes as a potential technique for supporting product and process quality control activities. A microNIR sensor was employed to analyse intermediate leather samples, nano-functionalised finishing products, and tanning waters. By means of Principal Component Analysis (PCA), it was possible to differentiate tanned products with traditional processes and innovative methods. The same chemometric model was applied for process control when using nanostructured substances which helped to assess how effectively the nanostructured agents were incorporated into the final material. These results demonstrated how NIR spectroscopy coupled with chemometric models can provide real-time insights into traditional and innovative tanning processes, optimise resource consumption, and support sustainability in the leather industry.

Keywords Leather characterization, Near infrared spectroscopy, Leather quality control, Tanning process control, Non-destructive testing, Sensors

Considering the numerous and substantial transformations that leather undergoes during the tanning production process, together with the complexity connected to the anisotropy of the matrix of natural origin and the growing use of a wide range of new tanning systems alternative to traditional ones[1,2], and even the use of innovative materials for the functionalization of leather manufactured goods[3–5], it is clear that control of product and tanning process, represents a crucial and challenging issue. A topic that therefore needs to be addressed by deploying technologies that are constantly adapted to changing production scenarios.

The presence of an increasing number of tanning systems and tanning materials generates two major criticalities. First, the absence of in-line monitoring tools limits tanneries to rely on a few experienced operators who visually check the leather at the entry and exit of the process: this limits the fastness of corrective actions and leads to a non-optimised use of chemicals. Secondly, quality control on semi-finished and finished products is still based on destructive laboratory analyses which offer detailed insights but are often time-consuming and impractical for routine or in-line process control slowing down the supply chain, hamper conformity checks (e.g. for the "metal-free" claim) and complicate anti-counterfeiting activities. In this sense, given the complexity of

[1]Comprensorio Olivetti, SSIP - Stazione Sperimentale per l'Industria delle Pelli e delle Materie Concianti – Italian Leather Research Institute, Via Campi Flegrei, 34, Pozzuoli 80078, NA, Italy. [2]Department of Chemical Sciences, University of Napoli Federico II, Complesso Universitario di Monte Sant'Angelo – Via Cintia, 21, Napoli 80126, Italy. [3]Assomac - National Association of Italian Manufacturers of Machinery and Accessories for Footwear, Leather Goods, and Tanneries – Vigevano, Vigevano, PV, Italy. [4]ABCS Srl - Scientific Instruments & Materials Science, Viale Vittorio Veneto, 32, Milano 20144, MI, Italy. [5]Department of Physics "E.R. Caianiello", University of Salerno, Via Giovanni Paolo II, 132, Fisciano 84084, SA, Italy. [6]Centre NANO_MATES, University of Salerno, Via Giovanni Paolo II, 132, Fisciano 84084, SA, Italy. ✉email: c.florio@ssip.it; a.medici@ssip.it

the systems under study, valid support is offered by the use of multi-diagnostic approaches, capable of providing crucial information on the quality characteristics of intermediate and finished products, as well as on process parameters; many of these approaches involve the use of characterization techniques, such as chromatographic, spectroscopic and optical and electronic microscopy techniques which, while satisfying the need to acquire precise information on products and processes, have the disadvantage of requiring on average long execution times. Since not all industries base their production on the entire tanning process (Scheme 1) but only on, for example, retanning and finishing processes, a quick product control at the input on the semi-finished wet hides can be extremely useful in order to limit complex analytical techniques and to be able to perform a quick control on all hides at the input of the process.

The most current solutions include, on the other hand, automated approaches that allow the effectiveness of processes and product quality to be optimally monitored, through tools capable of providing fast and effective responses, with the consequent possibility of saving resources, reducing emissions and environmental pollution[6,7]. Added to these advantages is the possibility of carrying out predictive analysis through the growing use of Non-Destructive Testing (NDT) technologies for in-line control of production.

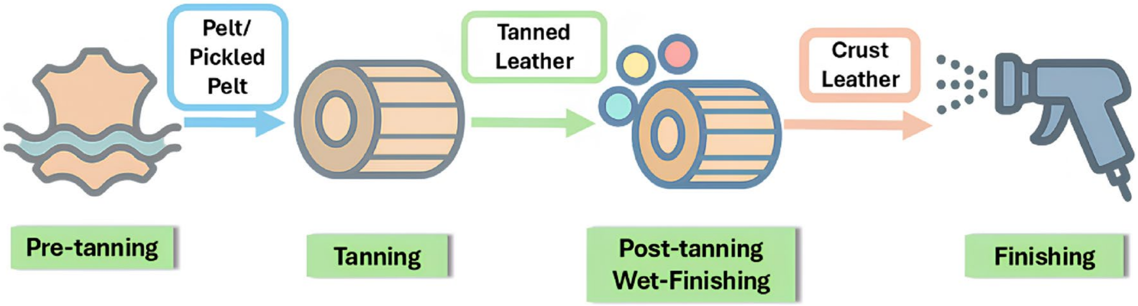
In the tannery process (Scheme 1), starts with the riviera (soaking, liming and depilation to open up the dermis structure), continues with the actual tanning (fixation of the agents - chrome, glutaraldehyde, zeolites or bio-based formulations - to the collagen fibres), passes through the post-tanning, dyeing and fat liquoring stages (where fullness, colour and softness are modulated by means of new chemical baths) and ends in finishing (application of protective and functional coatings), in this sense, these new sensor approaches based on NIR spectroscopy offer a promising route for remotely controlling the quality of both intermediate and finished leather products, allowing the tanning industry and its supply chain to achieve higher standards of quality and functionality. In the tanning stage, on-line NIR spectroscopy may distinguish the spectral fingerprints of chrome, glutaraldehyde, zeolite and bio-based systems while also tracking bath exhaustion and monitoring, for example, the progressive depletion of zeolite clusters, so the liquor is discharged only once the chemicals are fully consumed, thereby reducing pollutant loads at the source. During drying and finishing, NIR spectra detect subtle compositional differences in top-coats, allowing rapid discrimination of surface treatments, verification of metal-free claims, and early correction of over- or under-applied finishes, thus ensuring a consistent appearance and performance in the final leather.

NIR spectroscopy is a consolidated technique analytical for product analysis and controls, which allows investigation of the chemical-physical properties of samples by using the infrared region of the electromagnetic spectrum, in the range 12,500–4000 cm^{-1}, following a simple formula $nm = 10\,000\,000/^{-1}$ can led to 800–2500 nm. Its principles were already known since 1800, but it has progressively spread since the 1990 s, finding applications mainly in the pharmaceutical[8–10], food and agricultural sectors[11–17], where the main advantages of the technique were found in terms of: execution and analysis response speed; non-destructive checks; online control through optical fibres and cameras; ease of use and limited maintenance; high sustainability (not requiring the use of chemical solvents or reagents) and progressively lower costs. To the point that some studies are attempting to implement the technique for medical diagnostic use[18,19].

Concerning the effectiveness of the technique in the aspects of chemical characterization, the most significant absorption bands are attributed to the overtones and to the combinations of fundamental vibrations, particularly relating to the functional groups, such as –CH, –NH, –OH, –SH, with the consequent possibility of using the technique for the qualification and characterization of numerous molecules and macromolecules of different nature[20].

The possibility of using the technique to highlight changes in the response of collagen functional groups under operating conditions experienced in tanning is particularly promising for the sector. Indeed, NIR spectroscopy has been used to study, for example, the influences of pH on the binding interactions of collagen fibre with inorganic salts Al and Zr-based[21]. Specifically, NIR spectroscopy has been shown to have high sensitivity to slight changes in the 1 st N-H harmonic peak of amino groups ($-NH_2$) and the 1 st OH harmonic peak of carboxyl groups (-COOH) in collagen after tanning, providing valuable information regarding the effectiveness of the treatments used.

Although, the obvious use of this approach in product and process control, at the same time, it requires investments, not only on a technological level but also on a knowledge level. Indeed, it must be guaranteed an adequate level of knowledge for the management, processing, and interpretation of the information obtained



Scheme 1. Simplified schematic process of the tanning industry to produce leather from pelt.

from experimental activities. For obtaining usable results, NIR spectroscopy as an analytical technique must be supported by chemometric models and data multivariate statistical analysis throughout the analytical method development. With this combination of chemometric models and adequate multivariate data processing strategies, carried out by expert personnel, it will be possible to compare and NIR spectroscopic data handling in combination with the sample and generate comparative models and reference databases. In other words, to optimize the NIR signal, mathematical transformations are commonly applied where, only by applying multivariate statistical models, such as PCA, it is possible to appreciate and identify groupings or trends in the acquired data and, in definitively, following adequate data training operations, recognize categories of products and materials. In recent years, innovative tanning methods (nanoparticle tanning[22–25], oil tanning[26,27], amino acid tanning[28,29], dialdehyde starch tanning[30,31], zeolite tanning[32–34] have been introduced alongside traditional leather tanning techniques, leading to a wide variety of different products on the market. In some cases, this makes it difficult to easily distinguish between commercial products, necessitating the use of high-impact, destructive, and often very expensive analyses[35,36].

In this work, we aim to demonstrate how a less commonly used technique, NIR, with the right chemometric implementations, can be an easy-to-use, non-destructive, and sometimes even portable tool that can become a common instrument in many analytical laboratories. It can complement more complex and specialized techniques to detect the origin of leather tanning.

In this study, a microNIR sensor was used, coupled with an accessory dedicated to liquid sample acquisition. Through this system, intermediate leather samples, nano-functionalized finishings, chemical products, and tanning waters were studied, highlighting the potential of the technique for monitoring innovative tanning products and processes.

Materials and methods

Spectrophotometer

The OnSite-W microNIR instrument (VIAVI Solutions Inc. USA) was used for all analyses, in diffuse reflectance acquisition mode, range 908–1676 nm, scan count 600 and three replicates per sample. Liquid samples, analysed with the same instrument, were placed in a Suprasil© cuvette with an optical path of 5 mm and were analysed using an adapter, Side-View Vial Holder.

Comparative analysis of leather samples processed with different kinds of tanning agents

To evaluate the effectiveness of the sensor in grouping leather samples tanned by traditional and innovative tanning systems, including samples tanned with next-generation biobased agents, a comparative analysis of semi-finished samples in the wet state was conducted. The analysis included the following intermediate leather samples in their wet state:

Because several of the innovative tanning agents are still at prototype stage and available only in very small batches, some specimens in the first PCA were essentially one-off and cannot yet be reproduced on a larger scale. To reinforce the statistical soundness of the study, a second PCA model was therefore developed with a more representative sampling scheme: three independent hides for each tanning type, each analysed in seven times (twenty-one technical replicates per condition). This expanded dataset focused on the three tanning systems that dominate industrial practice chrome tanning, vegetable (tannin-based) tanning and glutaraldehyde tanning, so the model captures realistic process variability while providing a robust reference framework for classification and quality assessment.

Monitoring of the tanning effectiveness of zeolites

The effectiveness of nanostructured tanning agents, i.e. their ability to be absorbed, was evaluated as a function of different concentrations of zeolites. This evaluation was conducted through two complementary approaches: (*i*) analysis of bovine hides taken from different anatomical regions (Table 2), and (*ii*) analysis of tanning bath solutions collected at both the beginning and end of the tanning process (Table 3).

These samples were collected from both the back and side areas of bovine hides tanned with two different concentrations of zeolites (8% and 13%). A standard wet-white sample (chrome-free and without retanning agents) was included for reference.

To assess the degree of zeolite exhaustion and tanning agent uptake, wastewater was sampled at both the **start** and **end** of the tanning process for each condition. Samples were homogenized by stirring, and an aliquot was transferred to a Suprasil© cuvette for analysis.

Monitoring of nano-functionalized leather finishings

To test transversal approaches for process monitoring and verify the effectiveness of the dispersion of nanostructured materials, functionalizing the finishing, to provide antioxidant, self-cleaning, and antimicrobial properties to leather surface, measurements were carried out using the identified sensor system. The measurements were carried out on different samples of leather finishing products with the addition of different types of nanomaterials and finishing chemicals (Table 4). For measurements it was used an OnSite-W microNIR, equipped with the accessory for liquids (Side-View Vial Holder).

The "Flower Like" nanoparticles, refers to nanoparticles synthesized in a previous work[23] with different functional materials are combined into a single hierarchical morphology. The central "pistil" is composed of titanium dioxide (TiO_2), while the surrounding "petals" consist of silver (Ag) and silica (SiO_2) nanoparticles, all with dimensions in the range of a few tens of nanometres. This architecture mimics the shape of a flower, allowing for a high surface area and a synergistic combination of properties—including antimicrobial activity (from Ag), self-cleaning and photocatalytic effects (from TiO_2), and improved dispersion and mechanical stability (thanks to SiO_2).

Number	Sample	Tanning Process	Description
0	Pikled skin	Untanned leather	Reference untanned
1	Chrome	Chrome-based tanning	Traditional tanning agent
2	Glutaraldehyde	Glutaraldehyde-based tanning	Traditional tanning agent
3	Vegetable	Vegetable based tannin tanning	Traditional tanning agent
4	Carbamoyl sulphonate	Carbamoyl sulphonate-based tanning	Innovative tanning agent
5	Dialdehyde starch	Dialdehyde starch-based tanning	Innovative tanning agent

Table 1. Analysed tanned samples.

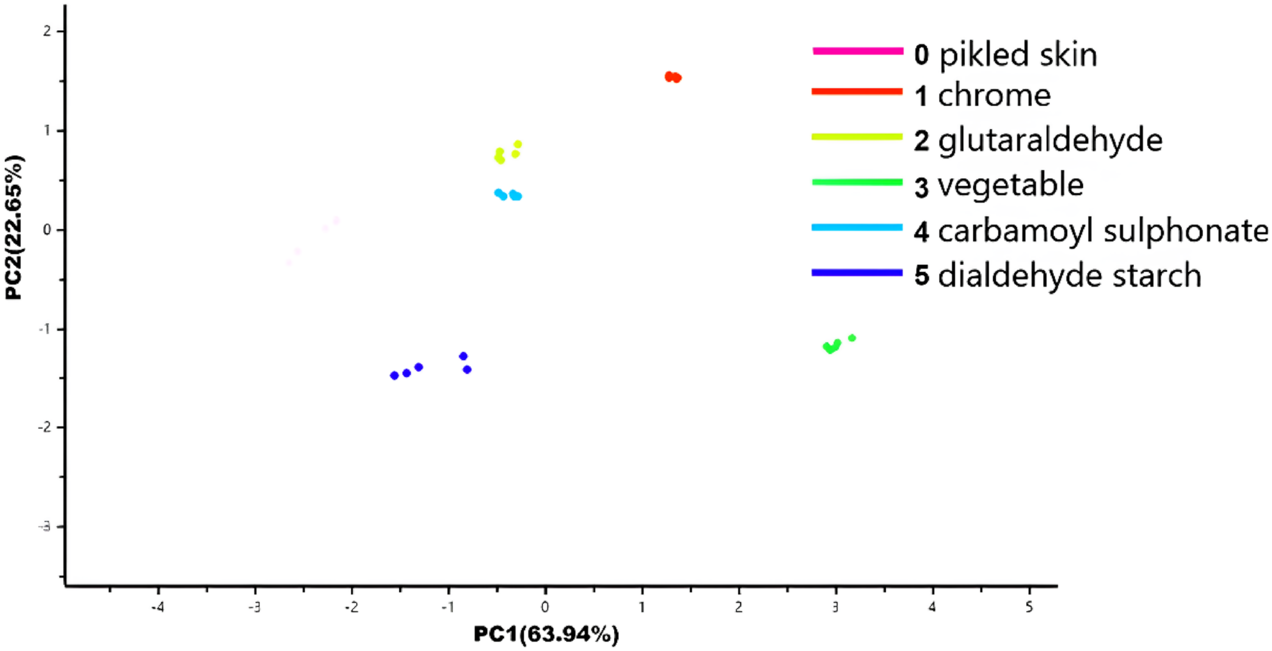


Fig. 1. PCA of leather samples (Table 1) with explained variance (%) processed with different tanning agents. The score plot highlights the chemical and structural variations among the samples as a function of the tanning method applied.

Data analysis

All NIR spectra were acquired with an OnSiteW microNIR instrument in reflectance (R) values. Preprocessing was optimised for solid and liquid matrices Savitzky–Golay first derivative followed by Standard Normal Variate (SNV) scatter correction PCA and chemometric modelling were performed with MicroNIR™ PRO software version 3.x (VIAVI Solutions). The platform supports both PC and tablet deployment, enabling batch processing, model development, real-time prediction and secure data transfer compliant with data-integrity requirements. The number of principal components retained was determined from the scree plot and by requiring a cumulative explained variance of at least 85%.

Results and discussion

Comparative analysis of leather samples processed with different kind of tanning agents

To evaluate the ability of the proposed techniques to properly highlight differences among different typologies of materials, samples with different tanning techniques samples were analysed (Table 1). The first sample (0) is the untanned reference material, the three samples 1–3 were tanned using traditional tanning agents while samples 4 and 5 can be considered examples of innovative tanning processes. The five leather samples processed with different kind of tanning agents were analysed, according to the conditions described in the Sect. 2.1. The spectra of three of the twenty reading are showed in SM1-6.

PCA analysis of the acquired samples was carried out. Figure 1 shows that the samples from different tanning systems are well separated after the PCA operation. The different samples and their respective positions are indicated at the side of the graph with different colours.

In Fig. 2 we can see that after a PCA operation, it is possible to estimate the tanning type of new unknown samples, demonstrating the potentiality of chemometric methods based on NIR spectroscopy, after an acquired spectra mathematical manipulation, to differentiate the type of tanning on unknown samples.

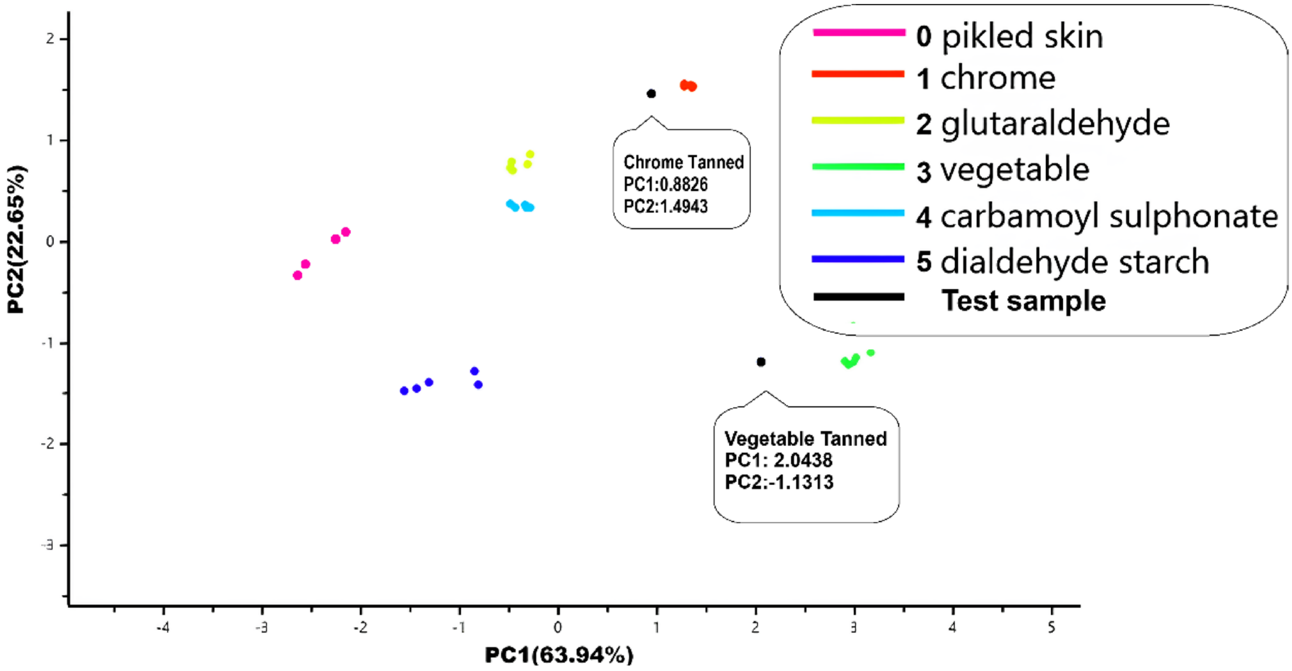


Fig. 2. PCA score plot including two unknown leather samples projected onto the model. Their positions relative to the known clusters support preliminary identification of the tanning agents used.

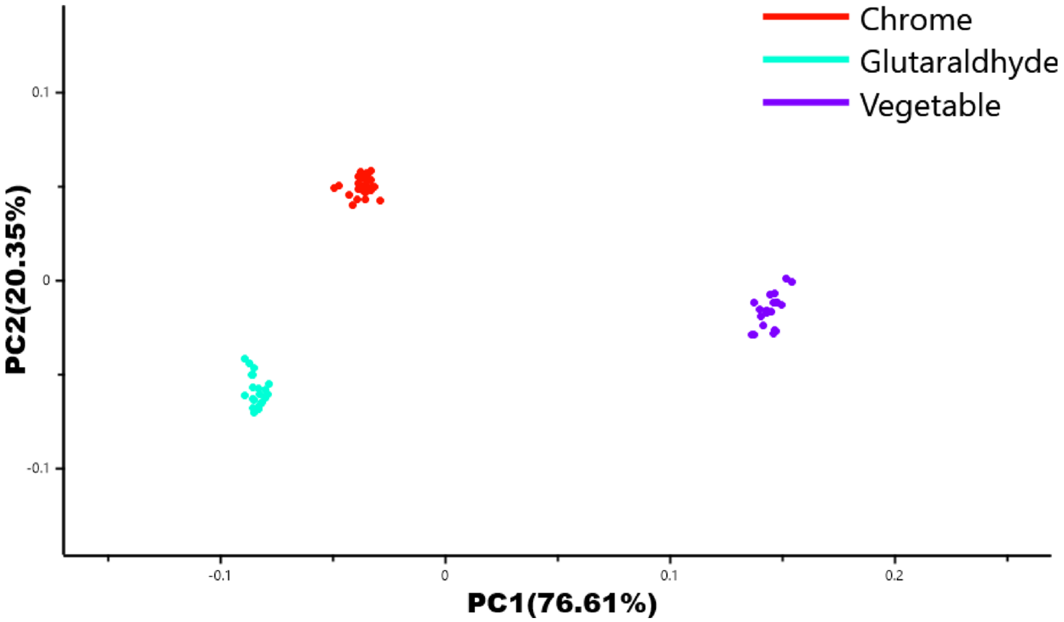


Fig. 3. Illustrates the second PCA model built on 21 spectra for type of leather (three hides per tanning type, each scanned seven times) with explained variance (%).

A second PCA model (Fig. 3) was generated on a much larger dataset that included the three tanning chemistries most widely used at industrial scale, thereby satisfying statistical requirements for model robustness. The first two principal components explain 96.96% of the total variance (PC1 = 76.61%, PC2 = 20.35%; see cumulative variance and loading plots in SM7-8) and deliver a clean planar separation of the three tanning classes. Chrome-tanned leathers cluster in the negative PC1/positive PC2 quadrant—an arrangement driven by chromium-related shifts in combination bands around 1150 nm and 1475 nm[37,38]. Glutaraldehyde-tanned leathers, while sharing the attenuation of the N-H overtone at ≈ 1475 nm[37], reside on the negative side of PC2 and are further distinguished by carbonyl features near 1410 nm originating from Schiff-base cross-links with collagen[37]. Vegetable-tanned leathers form a compact group on the positive PC1 axis, their position governed

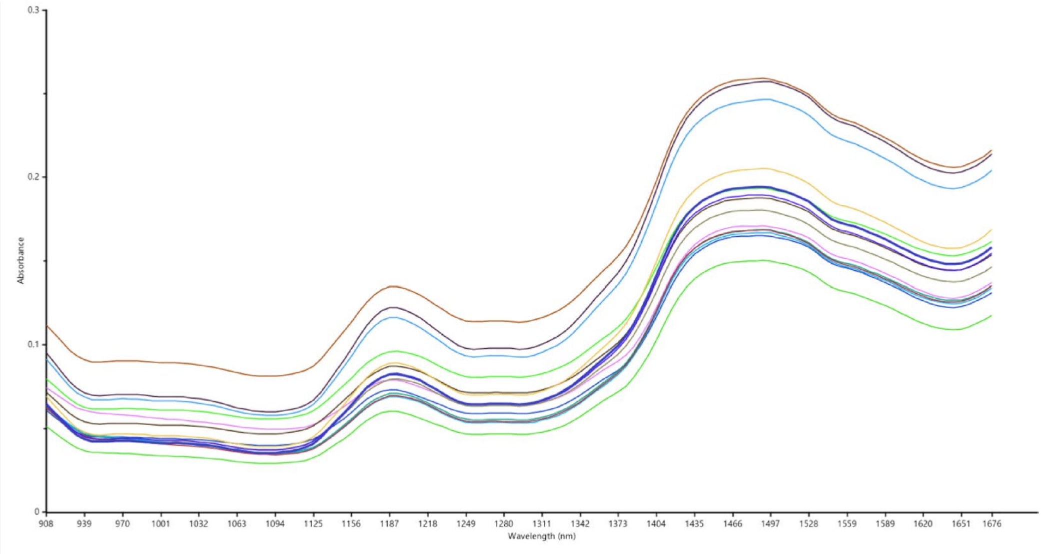


Fig. 4. Untreated NIR spectra of samples 1–4 and the standard, as described in Table 2.

Sample	Zeolite [%]	Position
Sample 1	8	back area
Sample 2	8	side area
Sample 3	13	back area
Sample 4	13	side area
Standard	wet white*	

Table 2. Leather samples analysed after tanning. *Without retanning agents

by strong overtones at about 1215 nm and 1670 nm, signals that may be correlated with the high tannin content of these extracts and their intramolecular hydrogen bonding[39] even though they are seldom reported for leather in the literature.

The three tight, non-overlapping clusters confirm that the microNIR-PCA workflow can unambiguously differentiate the principal industrial tanning systems while accommodating the natural variability introduced by sampling independent hides.

Monitoring of zeolites tanning effectiveness

Tanned leather samples

Figure 4 shows the NIR signals of the five skin samples analysed (Table 2): for each sample three independent signals were obtained, for a total of 15 spectra. As highlighted by visual analysis, NIR spectra are, by their nature, characterized by wide, often overlapping bands, which makes a direct interpretation of these signals very difficult. On the other hand, it is possible to identify two spectral regions of interest: at 1190 nm and a broad band around 1500 nm. These signals are closely related to the overlapping vibrational modes of different functional groups. Specifically, the region around 1190 nm is associated with C–H combination bands, while the 1500 nm region includes several important contributions: absorption at 1475 nm and 1549 nm is attributed to $C=O-NH_2$ and NH_2 groups[37]respectively. Additionally, signals in the 1395–1425 nm range correspond to the first overtone of O–H stretching vibrations from water in the sample. The broader band from 1435 to 1480 nm is typically linked to hydrogen-bonded O–H groups, indicating the presence of bound water or water associated with the collagen structure. The differences in intensity (their distribution along the graph's y-axis) hint at a certain differentiation among the samples. However, it's only by applying multivariate statistical models, like PCA, that we can truly understand and identify the underlying groupings or trends within the collected data.

To optimize the NIR signal, mathematical transformations can be applied. In our case, SNV, and first derivative were applied for each spectrum. SNV normalizes each spectrum individually by subtracting the mean of the spectral points. This reduces variability not directly related to composition but rather due to scaling and offset effects from physical variations in the spectrum. First derivative highlight rapid signal changes and suppress background noise, thereby improving the resolution of absorption bands. An exploratory PCA was performed on the spectra pre-treated in this way, of which the score plot relating to the first two principal components is reported in Fig. 5. In the graph, each spectrum is represented by a dot and the dots of the same colour represent the three replicates, therefore the three scans, performed for each leather. Close points in this graph (Fig. 5) indicate a certain chemical-physical similarity between the samples, while points that are further away in the

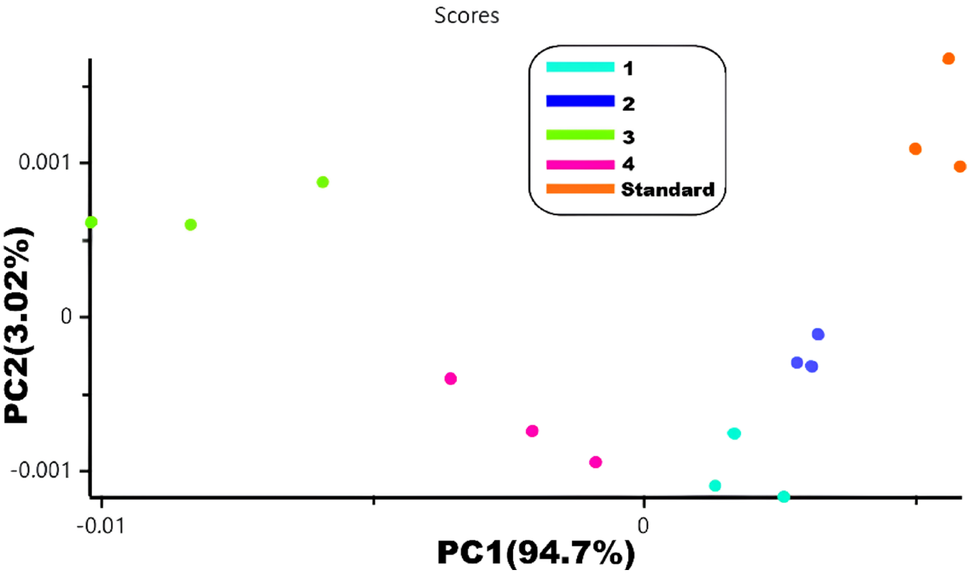


Fig. 5. PCA score plot with explained variance (%) (Principal Components 1 and 2) of the NIR spectra for samples 1, 2, 3, 4, and the standard (as defined in Table 2), after SNV and first derivative pre-treatment.

space defined, in this case by the first two principal components, indicate that the samples are significantly different from each other. The first principal component (PC1) is represented on the abscissa and explains 94.7% of the total variance; PC1 is therefore the most informative principal component in identifying any similarities or differences between samples. So, along PC1 (abscissa axis) it is possible to differentiate the analysed leather samples. In particular, the most marked differences are found between the standard (Orange) and sample 3 (green), respectively at positive and negative values of PC1. Samples 1 (Light Blue) and 2 (Blue) appear rather similar, while sample five, characterized by negative PC1 values, presents some similarities compared to sample number 3. Another important element is that the three replicates, and therefore, the three scans made on each sample fall very close to each other, showing the high repeatability of the measurement by microNIR.

Wastewater samples

Figure 6 shows the spectra relating to the liquid samples, Table 3, already transformed into the first derivative, a pretreatment chosen to optimize the spectral information and compensate for the saturation of the signal recorded in the last portion of the spectrum due to the high-water content. The score plot on the first two principal components highlighted how the samples relating to the starting (yellow points) and the end of the process (red points) differ quite markedly along PC1 (abscissa axis), with the sole exception of the “start 4” sample which overlaps with the end-of-process samples. The standard sample in pink shows characteristics more like the samples relating to the end of the process than to those relating to the beginning, however showing a peculiar pattern that isolates it from the other samples.

To verify the ability of the NIR to diversify the samples at the start and end of the process, a second PCA was performed. It was calculated to model the information relating only to the samples at the end of the process, to characterize this as a target class (Fig. 7). Additionally, the samples relating to the starting of the process were projected onto this model, too. The “start 4” sample was excluded due to its ambiguous behaviour, most likely caused by sampling or experimental error. This second score plot allowed us to outline the blue one as the reference class, relating to the samples at the end of the process, and to project the samples at the start of the process onto it. As highlighted by the graph, the samples in red (start) are systematically rejected by the model built on the blue samples (end), demonstrating the effectiveness of NIR, despite the small number of samples analysed, in discriminating these two classes of samples (PCA loadings SM9).

Monitoring of nano-functionalized leather finishings

In this case, the spectra acquired in transmission (generally preferred mode for liquid samples) were extremely noisy, it was therefore preferred to acquire the samples in diffuse reflectance using glass Petri dishes. The difference of the two acquisition modes is shown in Fig. 8.

Figure 9 shows the raw spectra relating to the samples, transformed into first derivative, and treated by PCA, according to the workflow scheme used. A PCA was carried out for exploratory purposes, to visualize the samples in a two-dimensional graph (score plot) to highlight similarities and differences between the samples. All three replicates acquired for each sample were considered in the calculation. The proximity of the replicas (points of the same colour) highlighted the high repeatability of the instrument, while distinct groupings depending on the sample analysed (points of different colour) seem to suggest the possibility of differentiating and classifying the samples based on the additives used.

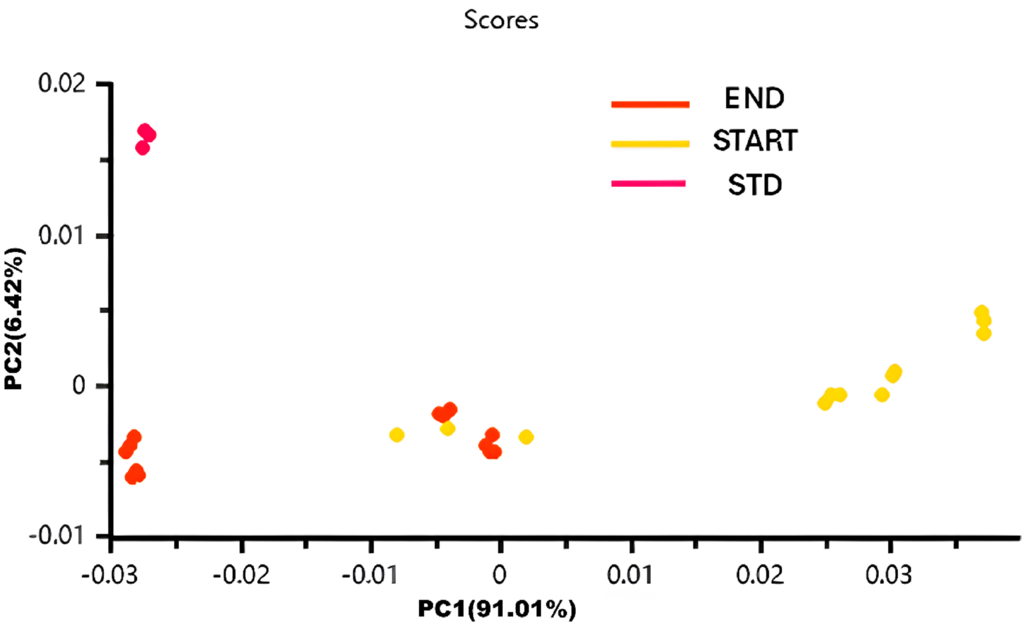


Fig. 6. PCA score plot with explained variance (%) (Principal Components 1 and 2) of the liquid NIR spectra (start and end of process) and the standard, as detailed in Table 3, following first derivative pre-treatment.

Sample	Details	Zeolite [%]	Position
Start 1	Process start	8	back area
Start 2	Process start	8	side area
Start 3	Process start	13	back area
Start 4	Process start	13	side area
End 1	Process end	8	back area
End 2	Process end	8	side area
End 3	Process end	13	back area
End 4	Process end	13	side area
Standard	Washing liquid		

Table 3. Wastewater samples collected during tanning.

Finishing chemicals containing nanoparticles	
Sample	**Details**
Nano 1	Polyurethane aqueous dispersion
Nano 2	Polyurethane aqueous dispersion + SiO_2 NPs
Nano 3	Polyurethane aqueous dispersion + multifunctional “Flower Like” NPs

Table 4. Analysed liquid finishing chemicals containing nanoparticles.

Conclusion

This study allowed us to highlight the versatility of microNIR in acquiring reliable and robust spectral information, both for solid and liquid samples. In both cases, through the application of PCA, it was possible to demonstrate how NIR can differentiate between the various samples analysed.

The system was ultimately found to be efficient for applications oriented towards the classification of products based on the presence of specific innovative components, through appropriate data training, with the consequent possibility of monitoring the effectiveness of the process, through online diagnosis.

The PCA was applied to the different kinds of studies considered, allowing to distinguish between different tanning or finishing treatments, as well as grouping unknown samples.

Finally, the technique applied to the analysis of the tanning baths made it possible to monitor the exhaustion of the baths and, consequently, to evaluate the absorption efficiency of the chemical products, favouring the chemical, water, and energy-saving efficiency of the tanning processes.

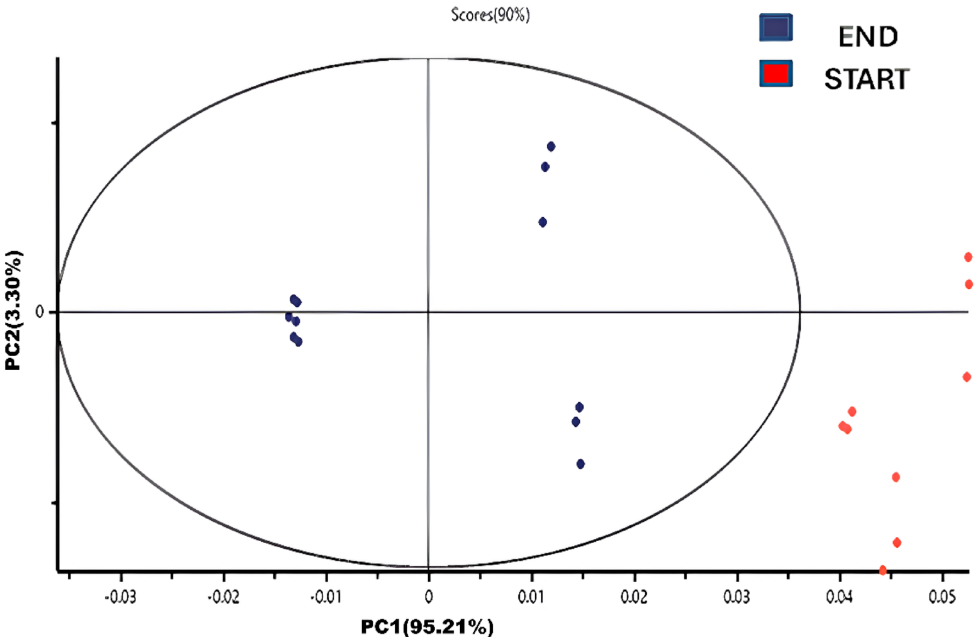


Fig. 7. PCA score plot with explained variance (%) of the end-of-process liquid samples (target class), with projection of the start-of-process samples, based on the NIR model of the data from Table 3.

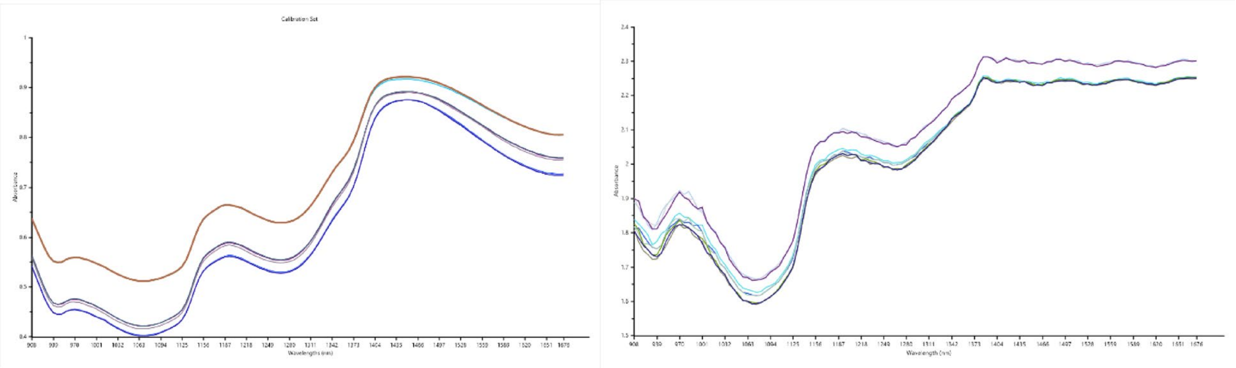
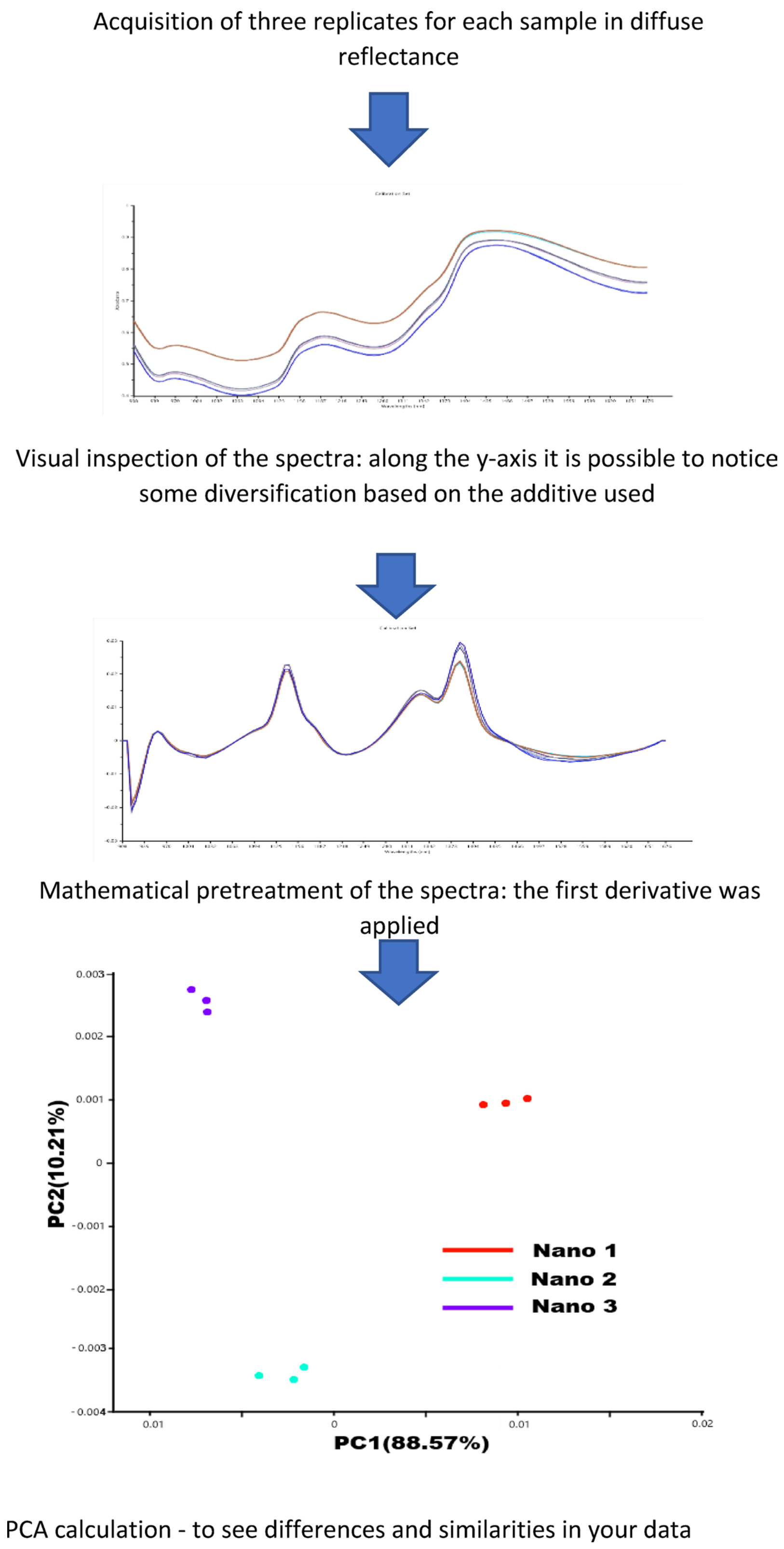


Fig. 8. Raw spectra of samples from Table 4: Diffuse Reflectance vs. Transmission.

Once the technique is calibrated and validated for a specific production line, its industrial deployment could bring multiple benefits. It would enable continuous quality control, reducing in-line dosing errors at each tanning stage; provide rapid, on-site identification of the tanning method applied to semi-finished hides, offering a faster and more cost-effective alternative to laboratory methods such as ICP-MS for metal determination; lower the number of off-spec batches, thereby cutting waste of materials and non-conforming products; and, most importantly, allow real-time monitoring of bath exhaustion, optimising process efficiency and markedly reducing wastewater-treatment costs.



Acquisition of three replicates for each sample in diffuse reflectance

Visual inspection of the spectra: along the y-axis it is possible to notice some diversification based on the additive used

Mathematical pretreatment of the spectra: the first derivative was applied

PCA calculation - to see differences and similarities in your data

Fig. 9. Workflow scheme for the NIR spectral analysis of finishing chemicals with nanostructured additives, illustrating acquisition in diffuse reflectance, visual inspection, first derivative pre-treatment, and PCA calculation with explained variance (%).

Data availability

The datasets used and/or analysed during the current study are available upon reasonable request to Claudia Florio (c.florio@ssip.it).

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Author contributions

C. Florio: Conceptualization, Formal Analysis, Funding Acquisition, Methodology, Project Administration, Resouces, Supervision, Validation, Writing original draft, Writing revision and editing A. Medici: Conceptualization, Data Curation, Formal Analysis, Investigation, Methodology, Supervision, Validation, Visualization, Writing revision and editing; R. Aveta: Formal Analysis, Data Curation, Investigation, Methodology, Visualization; L. Esposito: Data Curation, Formal Analysis, Investigation, Methodology, Validation, Visualization, Writing revision and editing; A. Favazzi: Data Curation, Investigation, Visualization, Writing revision and editing, F. Belvedere: Data Curation, Investigation, Visualization, Writing revision and editing; A. Zarrelli: Data Curation, Methodology, Supervision, Writing revision and editing; M. Sarno, Supervision, Writing revision and editing.

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Declarations

Competing interests

The authors declare no competing interests.

Additional information

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Correspondence and requests for materials should be addressed to C.F. or A.M.