



Università degli Studi della Tuscia di Viterbo
in convenzione con il Consiglio per la ricerca in agricoltura e l'analisi dell'economia
agraria – Centro di ricerca Ingegneria e Trasformazioni agroalimentari

in agreement with the Council for Agricultural Research and Economics, Research Centre for
Engineering and Agro-Food Processing

Corso di Dottorato di Ricerca in

Engineering for Energy and Environment XXXV Ciclo

**NON-DESTRUCTIVE SPECTROSCOPIC TECHNOLOGIES
APPLIED TO SUSTAINABLE FOOD TRANSFORMATION PROCESSES
FOR MONITORING THE HORTICULTURAL SUPPLY CHAIN**

Settore scientifico-disciplinare
(AGR/15)

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A.A. 2021/22 (A.Y. 2021/22)

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1. INTRODUCTION

The realisation of the program activities has been developed with a multidisciplinary approach. The need was to satisfy the three main goals considered: sustainability, digitalisation, and non-destructive control.

1.1 Sustainability

The 2030 Agenda for Sustainable Development, adopted by all United Nations Member States in 2015, provides a shared blueprint for peace and prosperity for people and the planet, now and into the future. At its heart are the 17 Sustainable Development Goals (SDGs), which are an urgent call for action by all countries - developed and developing - in a global partnership. They recognize that ending poverty and other deprivations must go hand-in-hand with strategies that improve health and education, reduce inequality, and spur economic growth – all while tackling climate change and working to preserve our oceans and forests.

In particular, inside the Goal 17 it is declared the promotion “*of the development, transfer, dissemination and diffusion of environmentally sound technologies to developing countries on favourable terms, including on concessional and preferential terms, as mutually agreed*” [1].

In these Goals [2] and targets, they are setting out a supremely ambitious and transformational vision. A world where human habitats are safe, resilient and sustainable and where there is universal access to affordable, reliable and sustainable energy. The world incline towards alternative sources of energy. While wind and hydro energy are also making progress, it is solar energy that has become the favoured one due to its increased affordability. The preservation of foods by drying is the time-honoured and most common method used by humans and the food processing industry.

Dehydration of food is one of the most important achievements in human history, making humans less dependent upon a daily food supply even under adverse environmental conditions [3]. Though in earlier times drying was dependent on the sun, nowadays many types of sophisticated equipment and methods are used to dehydrate foods.

Saving energy and achieving the best quality of dried products have become the most important factors that determine the usefulness and success of operating any drying unit. Heat could be transmitted through the drying unit in three forms: conduction, convection, and radiation. When the temperature of the fresh material increases, the molecular motion gains more energy, and it causes changes in the structure of the material as well as its chemical properties [4].

As indicated by Hii C.L. *et al.* [5], literature data report more than 400 different types of drying plants. Among them, only 100 different types are applied in practice. Design, heat input, operating temperature and pressure, quality specifications of the final dried product are the main variables considered for building dryers. However, such technologies are expensive, and high-energy demanding. In the past, food products have been dried exploiting sun energy. In this process, foods are directly exposed to the sun by placing them on the land or left hanging in the air.

In several Countries, sun drying is still used for domestic food transformation. At industry level, the spoilage reduction, in order to improve the stability of dried products during their storage, is an important input to stimulate a great and rapid evolution in drying technology [6].

During the past few decades, considerable efforts have been made to understand some of the chemical and biochemical changes that occur during dehydration and to develop methods for preventing undesirable quality losses. The main disadvantages of this type of drying are (i) contaminations from the environment, (ii) product losses and contaminations by insects and birds, (iii) floor space requirements, (iv) difficulty in controlling the process, and (v) bad odour [7]. When the climate is not particularly suitable for air drying or higher quality is desired, mechanical air drying is mainly used. However, sun drying is the cheapest method of drying foods. Nowadays, solar, and mechanical air drying are widely used commercially.

Foods can be divided into three broad groups based on the value added through processing by drying. In the case of cereals, legumes, and root crops, very little value is added per kilogram processed. More value per unit mass is added to foods such as vegetables, fruits, and fish; and considerably more to

high-value crops such as spices, herbs, medicinal plants, nuts; bioactive materials; and enzymes.

More than 250,000,000 tons of horticultural products and cereals are dried every year using natural drying [8]. Especially in developing Countries, the products are placed on the ground and turned periodically until completely dry. The newly developed solar drying allows the use of renewable energy sources, minimizing the defects of traditional techniques.

The drying process influences the amount and the organization of water molecules in the food cells producing changes in the product matrix [9]. Modern solar drying techniques try to combine aspects of the natural process with industrial needs, applying the concept of drying for keeping the characteristics of the raw materials and the environment in which they are grown (especially for Developing Countries).

The devices designed to effectively carry out this type of drying use mainly the energy of the sun for their functioning, shielding their deleterious effects and creating a hygienically suitable treatment environment, in tune with the microclimatic conditions of the environment. In African Countries, for example, during the last 10 years, a great effort has been made to improve the horticultural drying process, with the introduction of more efficient solar drying systems [10–14]. Solar drying is an extension of sun drying that uses radiation energy from the sun. Solar drying is a non-polluting process and uses renewable energy. A solar dryer is considered a reliable application of solar energy for food transformation.

The management of the production cycle is influenced both by intrinsic factors linked to the nature of the material to be transformed and by external factors, therefore determined by the context in which the production takes place. The final quality of the product strongly depends on the choices of technological parameters regarding processing and packaging. These choices can favour the conservation of compounds with high nutritional value present in the raw materials that can still be found in the final products, improving their nutritional quality. important, especially in this pandemic era [15-17]. Solar drying has been proven to be adequate to be applied to several crops [18]. It allows the production of products with desirable quality together with minimal environmental impact. Since the process is slow and weather-

dependent, so a wise strategy "fan off-fan on" must be devised considering the following conditions: air temperature, relative humidity, moisture content, and temperature of the material being dried.

Solar dryers are systems capable of exploiting solar radiant energy to heat a flow of air used to dry products. There are types with natural ventilation (which exploit the "chimney" effect) or with forced ventilation, which can be powered by a photovoltaic system or other electrical source [6]. Recently, a very comprehensive review of several types of natural convective and direct type (NCDT) solar dryers has been published [19] with the aim of collecting the most interesting practices of solar drying technology. Authors have classified in a precise way the different types of dryers. They explain the main characteristics of the "direct types" - i.e.: i) direct solar cabinet dryer, ii) modified solar cabinet dryer with natural convection, iii) direct solar drying using the chimney, iv) foldable solar crop dryer; - and the "natural convection type". Particular attention has been made at modelling the process, and at evaluating the performances.

Regarding indirect solar drying technologies, the most exhaustive review has been reported in reference [20] and has been written some years ago. Efforts have been made all over the world around to increase efficiency and drying time thus modern dryers are equipped with fans, efficient collectors, different thermal storage materials, reflectors, auxiliary heat sources, and sun-tracking systems. A recent paper [21] has reported an interesting development of closed, indirect heating, forced convection solar dehydrator with desiccant, which is economical, hygienic, and works off the grid. The whole experimental design has been discussed, paying attention to i) energy balance, ii) quantity of air required, iii) flow of air required, iv) area of the solar heater/collector; v) rates of dehydration.

1.2 Digital agriculture

The evolution of the informatics (IT) and electronics sectors that have occurred in recent years has led to a series of solutions and tools, characterized by a high technological content, which have affected and changed modern society. The agri-food sector has not been exempted from

these transformations, which however have often spread in a disorganized manner at the application level.

The digital agriculture (DA) just defines those production models characterized by the use of new technologies in the primary sector. Moreover, the DA can offer application solutions in line with the current demands of the agri-food sector, where the maximization of income takes place by increasing the value of the product (or its quality) and the economic recognition of practices that respect the environment.

In Italy, in particular, the DA must allow sustainable strengthening (greater remuneration, lower environmental impact, more quality and safety, traceability) of some agri-food subsystems, working together and synergistically from production to processing to the consumer. Today there are many infrastructures and technological solutions potentially at the service of agriculture, but obstacles still exist for the adoption of these technologies by operators, such as: cultural perception, lack of local technical skills and infrastructures, high start-up costs, with, in some cases, a risk of insufficient return on investment.

The development of the DA was made possible, starting in the early 1990s, by the availability of a continuously growing technological structure, divided into various levels [22-26]:

- development of information deriving from satellites (geographical positioning - GPS, GLONASS, GSNN - geographical information - GIS - remote sensing);
 - applications (sensors - remote or proximal - process sensors - RFID);
 - software (proprietary and, especially, open source);
 - high computing capacity in computer products used by the final user;
 - communication technologies (internet, smartphone, augmented and virtual reality, etc.).
- The development and application of digital technologies (electronic, mechatronic, information technology and integrated telecommunications) is of great use for the sustainable strengthening of field productions (machinery and digital agricultural systems), of specific supply chains (vegetables, flowers and cereals) and of agri-food transformations in general.

Then, the digital agriculture revolution must be considered. Historically, agriculture has undergone a series of revolutions that have driven efficiency, yield, and profitability to previously unattainable levels.

Market forecasts for the next decade suggest a ‘digital agricultural revolution’ will be the newest shift which could help ensure agriculture meets the needs of the global population into the future.

Digitalization will change every part of the agri-food chain. Management of resources throughout the system can become highly optimized, individualized, intelligent and anticipatory. It will function in real time in a hyper-connected way, driven by data.

Digitalisation is the defining technological transformation of this era, and, as in other sectors, it will have important impacts on agriculture. Digitalisation refers to the adoption of information communication technologies, including the Internet, mobile technologies, and devices, as well as data analytics, to improve the generation, collection, exchange, aggregation, combination, analysis, access, searchability and presentation of digital content, including the development of services and applications.

The digitalisation process holds the potential to bring about a significant change in how agriculture functions, beyond discrete tools, technologies, or practices, and to offer a path for innovation and new ways of organising production and supply chains. In particular, the agricultural sector is seeing a set of transformative trends due to digitalisation, such as a greater focus on precision agriculture, the internet of things (IoT) and the use of big data to drive production and business efficiencies [27].

Public and private actors in agro-food value chains and the wider agricultural innovation system (AIS) could benefit from the digital transformation of agriculture in multiple ways. For farmers, digital technologies and the insights that are generated from agricultural data could support better decision-making on farms, helping to boost innovation and improve agricultural productivity, sustainability, and resilience.

Digital technologies could also offer opportunities for new sources of efficiency and value creation upstream and downstream of farms, supporting research and innovation, the creation of new services for the sector, and improved traceability and more efficient transactions in value chains [28]. In

addition, policymakers could use digital technologies to improve how policies are designed, implemented and monitored, and design new, better policies for the agriculture sector [29]. Digital adoption in agriculture is commonly associated with potential improvements in societal objectives such as productivity, sustainability, and resilience, contributing to the notion that digital technology adoption should be accelerated as a policy objective.

To some extent, it is conceivable that adoption of digital agriculture can have “knock-on” effects that extend beyond productivity, sustainability, and resilience benefits, in addition to reduced labour supply and quality of life gains.

Currently, a key challenge pertains to the lack of relevant data in many countries, which makes it difficult to establish evidence of linkages between different elements relating to digital adoption and the achievement of agricultural objectives. Better information about the costs and benefits of different technology options is needed for stakeholders to be able to make informed investments and decisions.

For farmers in particular, it is crucial to be able to access reliable evidence-based knowledge about the effects of digital adoption, and the extent to which different tools respond to their different needs. Considering this evidence, policies should not focus on the promotion of specific digital applications, but they should strive to keep in place the appropriate incentives for innovation to enable farmers to choose, among a range of technologies, including digital ones, those that are most appropriate for them. In particular, to enable potential societal gains from agricultural digitalisation, there is a crucial need for the creation and sharing of knowledge on digital technologies and their impacts, while enabling the development of, and the access to, those technologies for all stakeholders [27].

Value chains will become traceable and coordinated at the most detailed level whilst different fields, crops and animals can be accurately managed to their own optimal prescriptions [30]. Integrating multiple sensors into manufacturing systems enables adaptive and flexible automation and increases process adaptivity and quality control.

The design of an optimum configuration must be based on a systematic analysis relative to operational and economic considerations. This design

should include an evaluation of sensor performance, a decision on how many sensors, of which type they should be, and where to locate them, modes of sensor interaction, and the influence on overall performance based on a cost/benefit analysis.

In most real-world applications it is impossible to develop an accurate model to predict sensor performance and therefore, performance must be derived empirically but in a systematic manner [31].

1.3 Near InfraRed Spectroscopy and Aquaphotomics

The analytical methods conventionally applied to determine the products' final quality and monitor the food process kinetics are time-consuming and currently unsuitable for automation [32].

Vibrational spectroscopy in the near-infrared region (NIR) is a non-destructive analytical technique (radiation is low-energy and does not transfer heat to the sample), highly sensitive and low-cost. It allows the rapid and reliable generation of objective data, an advantage compared to the more traditional laboratory methods that require an initial preparation of the sample with the use of chemical reagents instead, an operation requiring experienced and qualified personnel [33].

The fundamental principle on which this technique relies is the fact that each compound has its unique characteristic absorption spectrum. Specific energy intensities, and therefore specific wavelengths, cause transitions between the vibrational levels: consequently, the study of the spectral pattern allows the achievement of qualitative information about the presence of specific functional groups within a sample.

Within the food production and processing industry, the requirements of quality control have received much focus recently.

In parallel, modern near infrared (NIR) technology offers fast and cost-effective analyses that can afford quality control in both the laboratory and factory environments.

Unlike in the past, today the determination of the main composition parameters, useful for describing the quality according to the product category, is carried out with portable instrumentation to be used in field and / or in process.

In order to get an idea of the predictive ability of a model several statistical measures are used. These can be applied to the calibration set (the group of samples that are used to build the model parameters), the cross-validation set (samples temporarily excluded from model development but still ultimately involved in the development of the model), and the independent set (samples that have no input into the development of the model).

Among the main important statistics figures, RPD (Ratio of standard error of Performance to standard Deviation) and RER (Range Error Ratio) cover an important role because of their values are dimensionless, meaning that they can be compared on the same basis between models for different constituents/properties.

In 1999, the AACC International [34] published guidelines containing general information on NIR model development, performance evaluation, and calibration transfer to instruments of like brand, just focused on useful statistics.

Often, the monitoring of a transformation process is related to the control of authenticity and final quality.

The possibility to monitor and analysed each single fruit could represent a step forward for valorising nutritional characteristics of agri-food products and repositioning them on market.

Furthermore, the availability of small and compact instrumentation allowed the easy check of fruit quality along the supply chain, from harvest, to storage, to retail distribution, in order to track the product history and to identify fruit that were not managed according to the product specification.

Predictive models based on the NIR spectra could also be developed in order to monitor the product quality along the supply chain [35].

Last decade was also characterized by a great improvement of mathematic algorithms able to solve problems related to “apparent differences” among samples and sets, that could increase the risks of misunderstanding the significance of analysed and processed data. Furthermore, since 2007 [36], a new approach has taken hold, with a holistic view, where water, and its NIR absorption bands, is the leading actor.

Distinct water configurations, for example dimers, trimers, solvation shells etc., are known to contribute very specifically to its NIR spectrum. They are

very sensitive to the configuration and charges of the solvated molecules or clusters. Therefore, the NIR spectrum of the solvent (i.e. water) has been found to contain significant information about its solutes. Water molecules absorb radiation over the entire range of the electromagnetic spectrum, with intramolecular vibrational transitions occurring in the infrared region.

The absorption of discrete values of NIR energy, due to the interaction between electromagnetic radiation and aqueous matter, results in molecular vibrations in the water molecule (Figure 1) occurring between the hydrogen and oxygen atoms involved in the covalent bond. Such oscillations of interatomic distances may be symmetrical (v1), asymmetrical (v3) or bending (v2) stretching modalities, with the last one requiring less energy to occur compared to the others. [37].

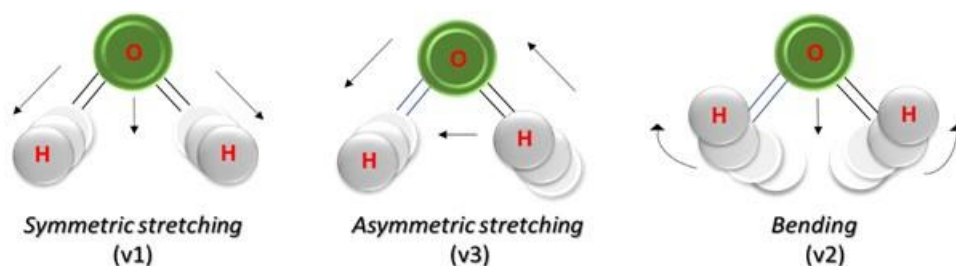


Figure 1: Vibrational modalities of the water molecule hit by near-infrared radiation [37]

Each type of molecule absorbs specific wavelength ranges, and the same is true for the different water species in solution, thus resulting in unique spectral properties that accurately and sensitively reflect the state, the behaviour and the functionality of the entire system [38].

Aquaphotomics relies on these assumptions: it uses the spectral model of water as a holistic biomarker able to provide helpful information on the properties of the studied system [36].

As the name suggests, aquaphotomics is an "omics" science, a biomolecular discipline aimed at representing and systematically interpreting biological complexity. It exploits the interaction between water and light radiation.

The essential principle of this discipline is understanding the role of water, which behaves, at the molecular level, as a collective mirror of matter and energy from which information on the state, behaviour and functionality of any bio-aqueous system results [38].

The added value of aquaphotomics is the use of spectroscopy in the near-infrared region combined with multivariate spectral analysis of water absorption patterns.

The spectrum analysis is possible because water absorbs only a fraction of the energy in the incident infrared radiation, thus making it possible to measure the NIR light coming out of the solution after interacting with matter. When near-infrared radiation hits water molecules, the energy released is converted into vibrational energy in the form of oscillations of interatomic distances and bonding angles.

However, as mentioned above, all the system's components affect the molecular structure of water with subsequent disturbance of the equilibrium. Any variation in the concentration of analytes results in a change in the vibration mode of the water molecules that can be monitored along the spectrum [39].

The spectral pattern of water changes in case of a mutation in the chemical composition of the system or because of a physical or biological perturbation, reflecting accurately and sensitively the transformation at the molecular level that characterises the different species of water constituting the three-dimensional network of hydrogen bonds.

In other words, instead of tracing the individual solutes, it is possible to observe the cumulative effect that these can bring to the water matrix, which changes its spectral model of absorption of NIR radiation, becoming a distinctive and identifying marker for each sample, integrating all the information on its composition at the time of the analysis [40].

Therefore, the aqueous matrix cannot be seen as an inert solvent as it constitutes the scaffolding of the entire biological system, with an interdependent connection with the rest of the constituent components of the biosystem.

For this reason, the objective of this new “omics” science is the possibility of describing in the future the state of any biological system exclusively in terms of the structure of the water molecule [38].

Near infrared (NIR) spectroscopy in combination with chemometrics and Aquaphotomics [36] represents a powerful and rapid analytical tool to also

monitor the dehydration process and to evaluate the changes occurring in food during technological transformation [41, 42].

The drying process influences the organization of water molecules (free water, dimers, trimers, solution and water shells) in the food tissues and results in changes in the water absorbance patterns. When an aqueous system, such as fruit, is measured by NIR spectroscopy, the light absorbance reflects the water vibrations [43].

The Aquaphotomics approach investigates the water–light interactions in biological systems, exploiting the fact that changes in the water matrix reflect, like a mirror, the rest of the molecules the water surrounds [44-45]. In this way, any change of the aqueous system highlighting perturbations can be observed, measured, analysed, and interpreted [45].

According to this holistic approach, the NIR spectra acquired in living systems under various perturbations (temperature, ion concentrations, oxidative stress, illumination, disease and damage) are characterized by 12 water absorption ranges (6–20 nm width each) in the spectral region of the first overtone of water (1300–1600 nm).

Such spectral ranges have been called Water Matrix Coordinates (WAMACs – Table 1) and labelled C_i , $i=1, 12$.

Within the WAMACs, specific water absorbance bands are related to specific water molecular conformations (water species and water molecular structures) [44].

When a certain perturbation of interest is shown to produce the changes at specific water absorbance bands, and when this is determined consistently and repeatedly throughout the Aquaphotomics analysis, these water absorbance bands (WABS) are considered ‘activated’ by the respective perturbation.

The selected WABS are plotted in spider charts, named ‘aquagrams’, to observe the WASPs (Water absorbance spectral patterns) [46].

Table 1: Coordinates of the molecular matrix of water with their spectroscopic and structural characterisation [44]

WAMACS label	Range (nm)	Assignment
C1	1336-1348	v3, asymmetric stretching vibration of water molecule
C2	1360-1366	Water solvation shell, OH-[H ₂ O] _{1,2,4}
C3	1370-1376	v1 + v3, symmetric and asymmetric stretching vibration of water molecule
C4	1380-1388	Water solvation shell, OH-[H ₂ O] _{1,4} and superoxide of water, O ₂ -[H ₂ O] ₄
C5	1398-1418	Free water molecules without hydrogen bonds and free OH - [S0] or molecules of trapped water
C6	1421-1430	Water hydration, H-OH and O...O bend
C7	1432-1444	Water molecules with one hydrogen bond [S1]
C8	1448-1454	v2 + v3 and water solvation shell, OH-[H ₂ O] _{4,5}
C9	1458-1468	Water molecules with two hydrogen bonds [S2]
C10	1472-1482	Water molecules with three hydrogen bonds [S3]
C11	1482-1495	Water molecules with four hydrogen bonds [S4]
C12	1506-1516	v1, v2 and strongly bound water

2. AIM

The project aims to develop advanced and innovative sensors with a high degree of digital integration and information for the development of advanced systems for the global quality of horticultural products from production to processing, to consumption. For the development of these systems, the NIR sensors on fresh and transformed horticultural products, applied to transformation processes that use renewable energies, will be used in the supply chain.

Dehydration and solar drying processes, implemented with NIR sensors (portable instruments), as well as Arduino sensors for controlling humidity, temperature, weight loss and light intensity, inside and outside the transformation chambers will be considered. NIR sensors of the same type will also allow the monitoring of raw materials and obtained products, and the data collection for the entire duration of the commercial life.

Targets:

- Evaluation of the effectiveness and efficiency of the transformation processes considered
- Verification of the suitability of the chosen digital methodologies for the monitoring of the supply chain
- Evaluation of scalability of the chosen digital solutions
- Development of protocols for data processing and transfer
- Identification of protocols and critical points for the realization of a NOT only documental traceability, transferable to other productions.

3. MATERIALS and METHODS

3.1 Experimental plan

The project activities are linked to the development of advanced and innovative sensors with a high degree of digital integration and information. They have been divided into the following steps, in accordance with the evolution of the research program during the three years to achieve specific objectives.

The first-year experimental plan has been focused on **laboratory tests made by micro plants**.

The standardization of process conditions for the solar drying micro-plant and the evaluation of the influence of climatic conditions on the effectiveness and duration of the process using the solar dehydration plant have been considered.

At the same time the choice of the most suitable sensors by type of detection, optical geometry, etc., has been made.

Further activities have been carried out on designing, programming, and positioning of sensors for data collection; monitoring of transformation processes with continuous sensors for the configuration optimization; data processing for multicomponent calibration curves and qualitative elaboration by aquaphotomics for the construction of aquagrams of immediate interpretation.

During the second-year project, **tests of scale transposition into pilot scale plants** (capacity: 20-40 kg of fresh edible product) have been carried out.

The activity program has been focused on: i) the verification of scale transferability; ii) the development of transformation processes; iii) the verification of plant performance coupled with the adaptation and repositioning of digital sensors, suitably sized; iv) the evaluation of the effectiveness of the actions for an actual transferability of the approach inside the horticultural chain.

Furthermore, the availability on the market of solar dehydration plants, flexibles for dimensions and size, would allow the realisation of the third year scheduled activity.

The confirmation of the results obtained at the pilot level both in terms of product quality and safety and traceability of the supply chain has been obtained during the last year of activities. To reach this goal, **tests on commercial systems already in use** at an Italian SME (capacity: 20-40 kg of fresh edible product) have been carried out. The realisation of this step concerned the identification of an Italian SME equipped with a solar dryer; the inspections and verification of infrastructures and company conditions; the verification of the real transferability of the technologies under study into an industrial reality with a view to "precision factory".

3.1.1 Laboratory tests made by micro plants.

First experiment (without physical sensors)

Micro-plants. The first experiment was carried out using pineapple slices as food matrix.

Parallel transformation tests on pineapple slices using two micro drying plants operating with solar energy were carried out in the field close to the Research Centre for Engineering and Agro-Food Processing in Treviglio, Italy (BG). The two drying systems differed in the air extraction method. The M1 had an air extracting fan that switched on only in case the inner temperature exceeded 35°C; the M2 drying system, on the other hand, had a continuously working fan at the bottom. Both microsystems (45 × 45 × 45 cm) were powered by solar energy, and a dimensioned photovoltaic panel was coupled for moving the fans. Figure 2 shows a representation of the two drying systems.

Samples. Four different pineapple samples from the Dominican Republic were peeled, cut into 10 slices each of 1.5 cm and placed inside the plants following a parallel order.

Physico-chemical analyses. Dry matter content, pH and titratable acidity were measured according to the Official Methods of Analysis of the Association of Official Analytical Chemists [47] on the fresh products, just after cutting ($t = 0$), and at the end of the drying processes, after 56 h ($t = 56$). Water activity (a_w) which is a measure of the energy status of the water in a system, crucial for microbial growth, was determined with AquaLab 4 TE benchtop water

activity meter (METER Group, Inc., Pullman, WA, USA) [48]. Data were analysed using Analysis of Variance (ANOVA) by using SPSS software (version 20.0, SPSS Inc., Chicago, IL, USA). One-way ANOVA was used to compare means of the parameters between $t = 0$ and $t = 56$ samples. The means were separated using a Tukey's HSD test, and their statistical significance was determined at 5% ($p < 0.05$) level.

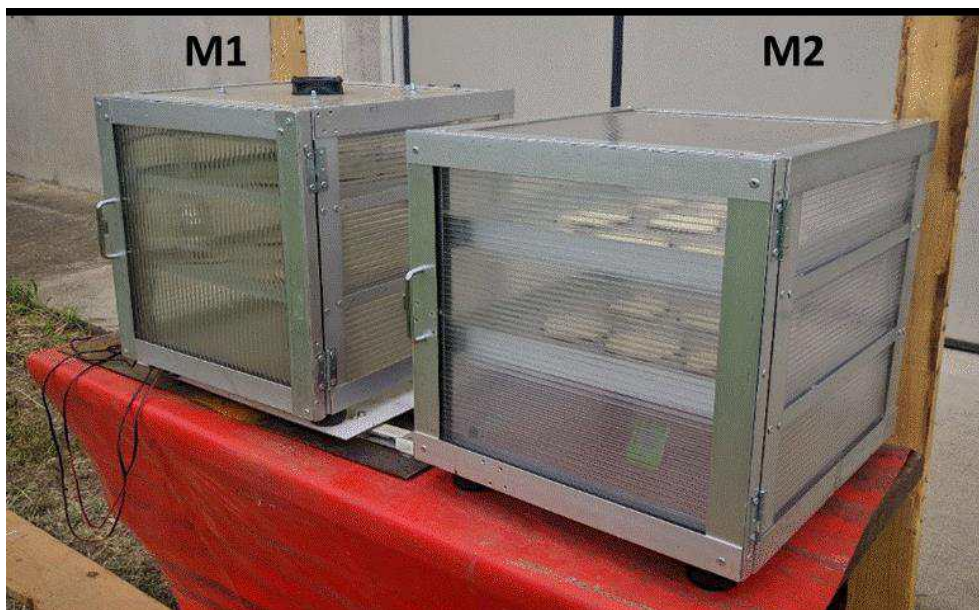


Figure 2 - Representation of the two micro-driers prototypes; M1: drier; M2: dehydrator.

Near InfraRed spectra acquisition and data processing. Near infrared spectra were collected in reflectance mode using the MicroNIR1700TM (VIAVI Solutions Inc., Santa Rosa, CA, USA) portable spectrometer with the MicroNIR Pro ES 1700 software. Spectra acquisition took place on four pineapple slices in the spectral region between 900 and 1600 nm (50 scans; 125 reading points) at three different times: (0) process start ($t = 0$), (1) during the process ($t = 48$) and (2) process end ($t = 56$). For each slice, spectra were acquired in triplicate in four different areas of the surface, for a total of 12 spectra. The process began after stabilizing the plant at the sun. Consequently, the spectra measurements did not undergo the temperature effect because all the process from $t = 0$ to $t = 56$ h was carried out at $47 \pm 1^\circ\text{C}$. Before the analysis, the instrument underwent calibration for the black, in the air, and for the white, on the supplied standard tile.

The spectra were first transformed by converting reflectance to absorbance; then the second derivative Savitzky–Golay filter (second order polynomial fit and 21 points) and multiplicative scatter correction were applied to remove potential scatter effects, according to Tsenkova et al. [46]. The transformed spectra were then normalized by applying the following formula:

$$\frac{A\lambda - \mu\lambda}{\sigma\lambda}$$

where $A\lambda$ is the transformed absorbance, $\mu\lambda$ is the mean value of all spectra and $\sigma\lambda$ is the standard deviation of all spectra at wavelength λ , and properly averaged using MS Excel® to verify the suitability of the aquaphotomics approach in evaluating process efficiency. Aquagrams were calculated from the spectral data. An aquagram is a spider shaped diagram which shows normalized and averaged absorbance at the selected wavelengths according to specific water absorbance bands (1344 nm, 1364 nm, 1372 nm, 1382 nm, 1398 nm, 1410 nm, 1438 nm, 1444 nm, 1464 nm, 1474 nm, 1492 nm and 1518 nm). The spectral data at $t = 0$ and $t = 56$ underwent statistical processing to apply the principal component analysis (PCA, 95% confidence level) and linear discriminant analysis (LDA) using Minitab 17 statistical software (Minitab, Inc., State College, PA, USA). PCA was performed using the correlation matrix to be sure to equally weight all the variables. Afterwards, with the same software, data underwent LDA to classify observations into two or more groups. LDA [49] is one of the mostly used classification procedure which maximizes the variance between categories and minimizes the variance within categories. PCA was firstly carried out using the 12 water absorption bands listed above. To perform explorative results deleting redundant information, PCA was then recalculated using only five WABS (water absorbance bands) selected on the basis of loading values of the first three Principal Components: 1342, 1452, 1488, 1364 and 1412 nm. These WABS were also used to perform LDA. Leave-one-out cross validation was carried out to validate the model. Spectra of $t = 48$ samples were acquired with the main purpose of verifying differences between the two dehydration processes. They were not considered for the PCA and LDA analyses which were applied with the aim to evaluate the power of NIR

spectroscopy for studying the final quality of the dried products subjected to two different dehydration methods.

Second experiment (with sensors implementation)

Sensors implementation. The micro-plants of the previous experiment were implemented with Arduino sensors (ATmega32U4 Arduino Leonardo AVR® ATmega AVR MCU 8 bit; Arduino UNO Rev3 orig. HIGH QUALITY; Yun Rev 2 Arduino – see Figure 3) for the detection of the main physical parameters. In particular, were in continuous monitored:

- the in/out RH%, as percentage ratio between the quantity of vapor contained in a mass of air and the maximum quantity (i.e., at saturation) that the volume of air can contain under the same conditions of temperature and pressure;
- the in/out temperature, expressed as degrees Centigrade ($^{\circ}\text{C}$);
- the light, as the sensor's ability to receive the light signal (millivolts, range between 0 and 1024);
- the fan intensity, as a value within a range between 0 (fan stopped) and 255 (maximum activity = 1800 rpm);
- the weight loss%, as the weight variation detected by 4 load cells placed in correspondence with the four base vertices.

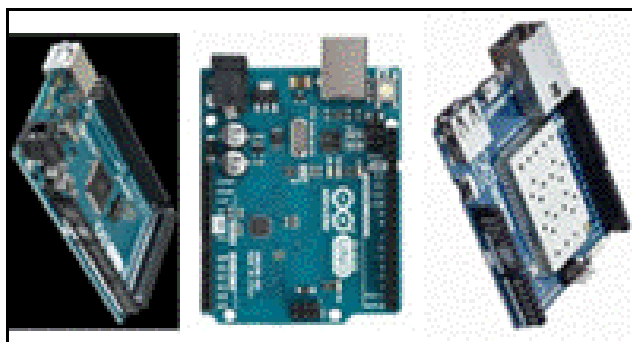


Figure 3 – Arduino sensors

The sensors connection and optimization, and the experiments were carried out at the Research Centre for Engineering and Agro-Food Processing in Treviso. The drying processes were carried out by applying the simultaneous use of sensors and considering the sensors and the site (position) as independent variables, while fan, weight, temperature, and relative humidity (RH) as dependent variables. The processes were monitored up to water activity (a_w) below 0.6, and constant weight (50 hours). The whole dataset was sent to a remote computer connected via Wi-Fi for data collection

and processing. The values of the different variables were standardized to allow reading on the same graph. The values measured by the sensors and stored in an SD-card in the Arduino board, acquired with a frequency of $3 \cdot 10^{-3}$ Hz, were processed with descriptive statistical analysis to verify their trend over time and tests were applied to variance (ANOVA) [50] and Tukey's [51] post-hoc test for observing the significant difference between the means. The analysis of the time series, of the variance and of the significance of the difference between the means of the values was developed through the software R [52].

Samples and determination of water activity at the end point. Fresh eggplant samples were used as horticultural model of raw material to be dried. Samples were cut into slices of 3 mm thickness and arranged side by side in parallel into the two plants. Figure 4 schematically shows the samples arrangement.

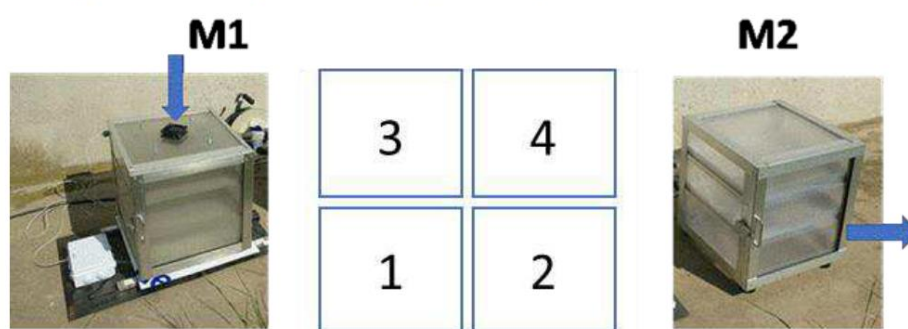


Figure 4 - Samples arrangement inside the two micro-plants.

A second test has been carried out on fruit model (apple slices) to confirm the transferability of the approach to other agro-chains.

Near InfraRed spectra acquisition and data processing. NIR spectra were collected in reflectance mode using the MicroNIR1700 TM (VIAVI Solutions Italia S.r.l., Monza, Italy) portable spectrometer with the MicroNIR Pro ES 1700 software. Spectra were acquired continuously throughout the process by placing the NIR probe over a sample slice, positioned on the intermediate shelf. The probe was thermally insulated to avoid temperature variations. In the eggplant preliminary process, the probe was wrapped in aluminium insulating fabric (Fig. 5a), while for the apple process, a commercial extended probe housing was used (Fig. 5b). For the eggplant

process, spectra were collected for 50 hours, one spectrum every two hours, for a total of 26 spectra; for the apple process, spectra were collected for 61 hours, one spectrum every hour, for a total of 61 spectra. Before the analysis, the instrument underwent calibration for the black, in the air, and for the white on the supplied standard tile.

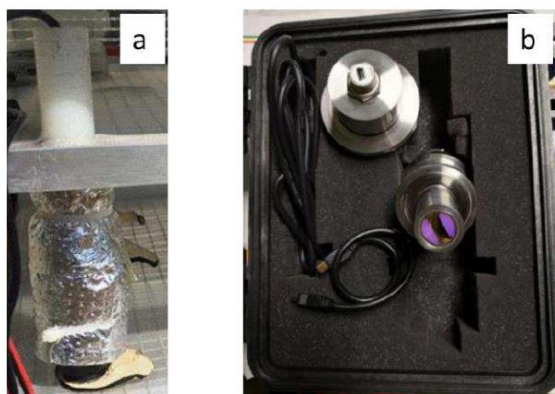


Figure 5 - NIR probe conditioning: aluminium insulating fabric for the eggplant dehydration process (a); commercial extended probe housing for the apple process (b).

In addition to the NIR data processing already reported above, the Moving Block mean Model (MBM) was applied to the data using a block size of 3 for eggplant process and 7 for apple process. Moving Block Models are calibration free, unsupervised methods which allow to monitor the process with no need for prior knowledge of NIR spectra of the analysed matrix. Thus, they are a valuable tool for assessing the endpoints of evolving processes such as powder blending or drying operations. They are based on the summarized variance in blocks of spectra collected over the time course of the process. A block is a predefined number of spectra previously demonstrated to provide enough information about the changes within the evolving process [53].

3.1.2 Scale transposition tests on small-size plants

Small size plant (prototype – pilot scale)

Figure 6 shows the configuration of the prototype of the solar dehydration plant already active and available, without digital control, at CREA.IT Department in Milan. The prototype is food- grade solar devices for drying herbs, vegetables, fruit, fish, meat, and other organic products. It can be placed directly on field in marginal areas close to the cultivation area without

any requirements, allowing for immediate processing of products. The system is built in polycarbonate, a cheap material with a high specific surface area that allows a great recovery of solar energy. Unlike traditional solar dryers, in which solar energy is stored for heating the air which is subsequently introduced into the drying chamber [11], in the described system solar energy is directly stored in the polycarbonate walls and readily used as a source of heat. The plant is equipped with a ventilation system for air circulation with consequent uniform and efficient drying of the product. The air is taken from the outside through the grid on the south side exposed to the sun, heated by the collector, and conveyed to the drying chamber; then the air is expelled by the ventilation unit located at the bottom of the dryer, carrying out a dehydration process called Natural Effect Drying (DEN). The air flow hits the products transversely, reducing the need to turn the products, allowing uniform drying.

The energy required by the ventilation system can be supplied either by a small low-voltage electricity consumption or, alternatively, by a photovoltaic system.

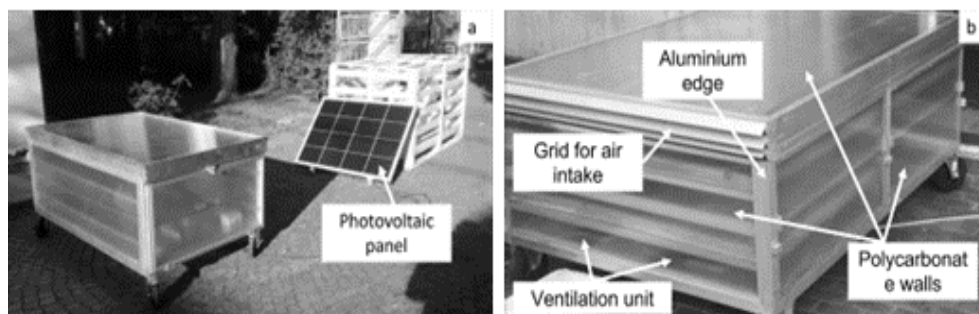


Figure 6 - Solar drier used in the drying experiments (a); details of the drier components (b).

Table 2 reports the technical characteristic of the solar dryer used, while Figure 7 shows the scheme of the air flow inside the plant.

Table 2 - Technical characteristics of the solar dryer.

Size (W × L × H)	97 × 151 × 70 cm
Solar module	80 × 120 cm
Typical drying temp	40 °C
Air volume, nominal	400 m ³ /h max
Nominal capacity	40 kg max
Thermal power *	1.8 KW
Capacity	2.8 m ²

* Power measured at 1000 W/m² of sun radiation

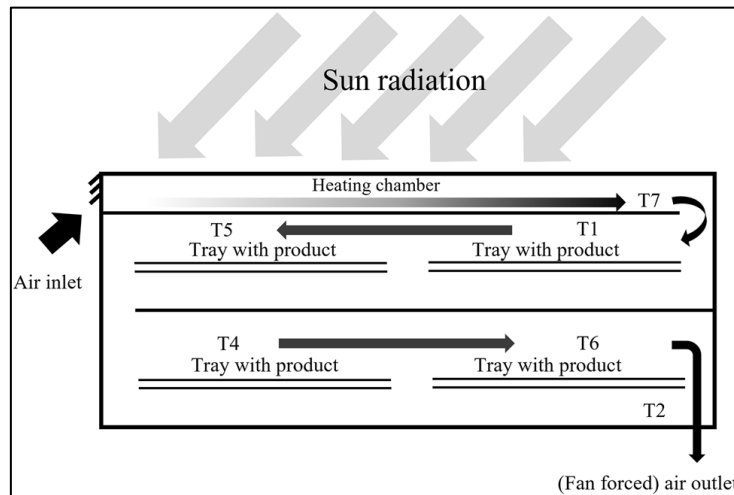


Figure 7 - Scheme of the air flow inside the solar drier.
T1–T7: thermal probes

Two different experiments were carried out using this pilot scale prototype.

Two different matrices were tested: onion slices and melon slices.

Yellow onion (*Allium cepa* L.) bulbs of the breeding line 228/3, grown in Cesenatico countryside in a breeding program for the selection of low pungency onion genotypes were used.

Melon fruits (*Cucumis melo* L. var. *Honey Moon*) came from an Italian farm in the province of Mantova.

To prevent physiological and chemical changes due to respiration process, samples were stored at 4 ± 1 °C before the trials.

Prior the drying process, all not-edible parts of fruits were removed, and the edible weight was recorded.

Edible portions of fruits were mechanically cut into slices of uniform thickness (2 mm) to ensure a uniform drying and a homogenous final product. The slices were placed on the drier trays close to each other without overlapping, using all the available space for the greatest process efficiency, as reported in Figure 8.



Figure 8 - Raw materials ready for drying.

The plant was implemented with Arduino sensors (ATmega32U4 Arduino Leonardo AVR® ATmega AVR MCU 8 bit; Arduino UNO Rev3 orig. HIGH QUALITY; Yun Rev 2 Arduino) for the detection of the main physical parameters.

Were in continuous monitored:

- the in/out RH%, as percentage ratio between the quantity of vapor contained in a mass of air and the maximum quantity (i.e., at saturation) that the volume of air can contain under the same conditions of temperature and pressure;
- the in/out temperature, expressed as degrees Centigrade (° C).

The weight loss, water activity (a_w), and yield were monitored on a portion of product at fixed times during the drying processes. At the same times, samplings for chemical analyses were collected.

As alternative way to monitor RH% and temperature, also data loggers with the possibility to download data at the end of process have been used.

Near Infrared Spectroscopy. NIR spectra were collected in reflectance mode using the MicroNIR On-site W™ (VIAVI Solutions Italia S.r.l., Monza, Italy) portable spectrometer with the MicroNIR Pro ES 1700 software. The instrument is characterized by a miniaturized optics, sub-centimetre in size, with no moving parts, collecting diffuse reflectance measurements with scan times of sub-second and without the need to use optical fibres to interface the optical bench with the sample. The monochromator of the instrument consists of a linear variable filter (LVF), soldered on a 124-element diode array InGaAs detector. Two small tungsten filament radiation sources allow reflectance measurements of samples. A laptop was used to send the proper

commands from the acquisition software to the spectrophotometer and to receive back the spectral data, collected through a USB wired port.

Spectra were acquired continuously throughout the process by placing the NIR probe over a sample slice, positioned on the intermediate shelf. The probe was thermally insulated to avoid temperature variations (Figure 9).

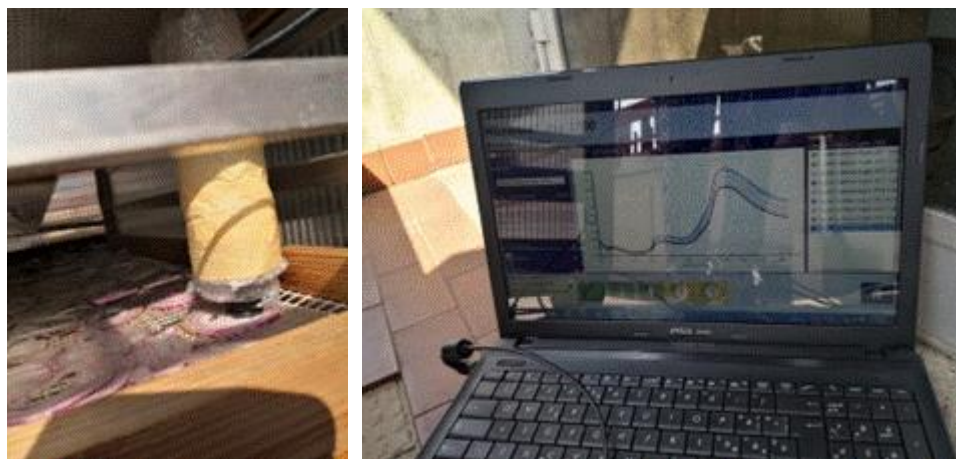


Figure 9 - NIR probe connected with a notebook for data collection during onion slices drying.

Before the analysis, the instrument underwent calibration for the black, in the air, and for the white on the supplied standard tile.

Spectra were collected in continuous mode every ten minutes, respectively for a total of 170 spectra for onion and of 284 spectra for melon slices (from process start to process end).

Data processing. Chemometric analysis of the NIR data was performed using the Unscrambler software package (v 9.7, Camo, Inondhcim, Norway). Several pre-treatments were tested, alone or in combination with each other, including standard normal variate (SNV), normalization, and multiplicative scatter correction (MSC), to reduce the spectral variability due to light scattering; spectral derivatives, such as Norris gap and Savitzky Golay derivatives which allow the resolution of overlapping peaks [54]. Only the best results, in terms of model performances, were retained.

Regressions between chemical data and spectral data were computed by partial least square (PLS) regression through the NIPALS algorithm. PLS

regression is an effective technique based on latent variable extraction techniques used to perform multivariate regression.

Cross-validation was performed in the calibration set using the leave-one-out cross-validation. Calibration models were evaluated on the basis of: i) the coefficient of determination in calibration (R^2_{cal}) and in cross validation (R^2_{CV}); ii) the root mean square error of calibration (RMSEC) and cross validation (RMSECV); iii) the ratio error range (RER), calculated as the ratio of the range of the reference values and the standard error in cross validation (SECV) [55]; iv) the RPD for calibration and cross-validation, a parameter directly linked to R^2 , calculated according to Dardenne [56].

RER and RPD were used to classify the performance of a given NIRS equation.

RER values < 3 indicate low predictive ability models; models with RER between 3 and 10 have low to moderate practical utility, RER values > 10 indicate good practical utility [57], while RER values > 12 indicate high predictability [58]. RPD values greater than 3 are useful for screening, values greater than 5 can be used for quality control, and values greater than 8 for any application. The best PLS models were then used to predict the parameters' profiles along the process.

Aquaphotomics. The spectra were transformed by converting reflectance to absorbance; then the second derivative Savitzky–Golay filter (second order polynomial fit and 21 points) and multiplicative scatter correction were applied to remove potential scatter effects, according to Tsenkova et al. [46]. The transformed spectra were then normalized by applying the following formula:

$$\frac{A\lambda - \mu\lambda}{\sigma\lambda}$$

where $A\lambda$ is the transformed absorbance, $\mu\lambda$ is the mean value of all spectra and $\sigma\lambda$ is the standard deviation of all spectra at wavelength λ , and properly averaged using MS Excel® to verify the suitability of the aquaphotomics approach in evaluating process efficiency. Aquagrams were calculated from the spectral data (see 3.1.1 first experiment).

3.1.3 Tests on commercial systems already in use in Italian SME.

Small-size commercial plant: Italo BASE + (G-teK s.r.l., Carpi, Modena, Italy)

The availability on the market of dehydration plants flexibles for dimensions and size, allowed the realisation of the step 3 of this PhD research activities. This type of plant is already active, without digital control, in companies closed to Milan.

The selected company, Ca' dal Saggia, is located in the Pavia area, and the production of dried fruit and vegetable started about 5 years ago (Figure 10). Basic information on the management of the farm have been collected.



Figure 10 – Products from the Italian SME: Azienda Agricola Ca' dal Saggia.

The supplied solar dryer was produced by another Italian SME: G-teK s.r.l., Carpi, Modena [59]. The plant is the commercial version (Italo BASE+) of the CREA prototype used during the previous experiments (see 3.1.2). Pictures of the Italo BASE + are shown in Figure 11. The dryer is working only using photovoltaic energy for ventilation, without any connection to electricity.



Figure 11 – Italo BASE +: Schematic representation, and at work.

Italo BASE+ was implemented with physical sensors and the NIR system connected with a computer for data collection and processing (see 3.1.2 for details).

Samples. Two experimental tests were carried out on apple slices (*Red Delicious* and *Fuji varieties*; 1.5 mm thickness), for the verification of the real transferability of the studied technologies in industrial realities with a view to "precision factory".

After washing, cleaning, and cutting slices were positioned on dryer trays as shown in Figure 12.



Figure 12 – Fresh apple slices of Red Delicious and Fuji varieties

The same parameters detected during the experiments made using the CREA prototype (see 3.1.2) were collected at fixed times (physico-chemical analyses) or in continuous (physical indices and NIR spectra).

NIR Data processing. A total of 650 spectra have been recorded continuously in both experiments until the end of the processes (55 hours). The duration of drying has been related to the climate conditions, and the total time was calculated to be sure the process end. As reported in 3.1.2, correlation between physico-chemical indices and NIR data was built. At the same time, aquaphotomics was applied to prove its applicability in real situations. To reach the last goal of the project, it was necessary to find a partner able to process the aquaphotomics results in real-time, developing a dedicated platform, named “aquacontrol system”.

The selected company, Qualitade s.r.l. – Cremona, Via Mantova 137, 26100 (Italy), in charge of preparing the cloud space and processing platform, is participating as an active partner in the preparation and implementation of all phases of the aquacontrol system development, with parallel sampling for chemical-physical analysis.

4. RESULTS and DISCUSSION

4.1 Laboratory tests

First experiment (without physical sensors)

Physico-chemical analyses. At $t = 0$ and at the end of the drying processes ($t = 56$),

the physico-chemical parameters between the fresh product and the dried ones showed significant differences ($p < 0.05$). The pH values did not change at the end of the process, supporting the mild stress undergone by the product [60]. As expected, the dry matter content increased significantly after drying, while the water activity, strictly related to the water content, decreased, as shown in Figure 13. Based on titratable acidity values, the M2 process resulted in minor variations in the acidic chemical compounds increasing the titratable substances than the M1 process even though the detected values between the two processes were not statistically different.

Near infrared analysis. Spectra of drying pineapple slices were collected at three

different times ($t=0$; $t=48$; and $t=56$ hours) in both plants (Figure 14).

The raw spectra showed broad and overlapped absorption bands detecting predominant water absorption bands at 960–990 (second overtone -OH stretching), 1150–1190 (Combination of first overtone -OH stretching and OH bending) and 1400–1435 nm (first overtone -OH stretching) [46]. The application of pre-treatments allowed more appropriate assignment of several absorption bands.

Figure 15 reports how the mean spectra for each sampling time appear after the pre-treatments. In particular, the presence of three shifts of the water overtones between time 0 and time 56, with a high variability in the 1300–1600 nm NIR range is highlighted. These findings supported the hypothesis that aquaphotomics could be helpful to discriminate between the considered drying processes.

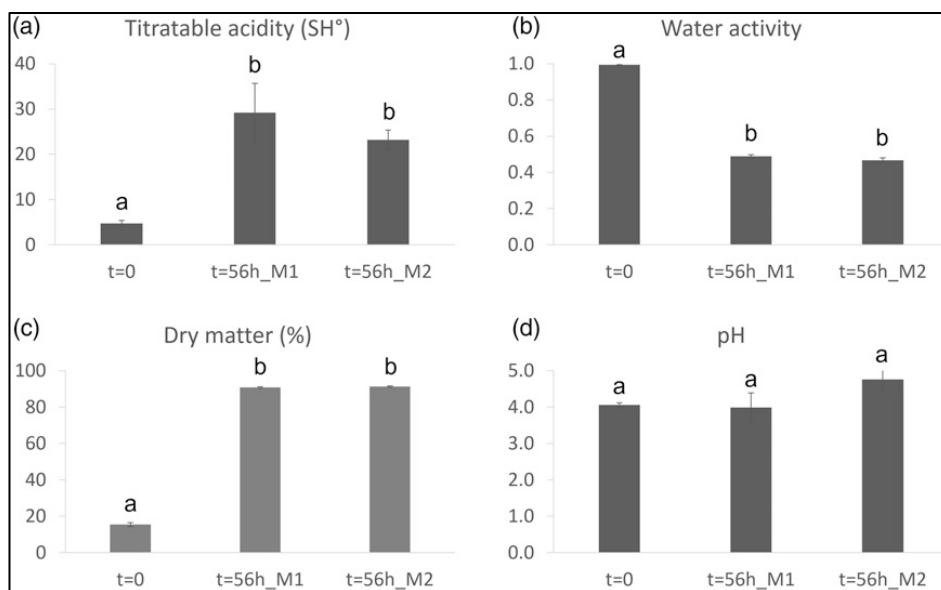


Figure 13 - Physico-chemical data at $t = 0$ and $t = 56$: titratable acidity (a), water activity (b), dry matter (c) and pH (d). M1 = drying plant; M2 = dehydration plant. Same letters = not statistical differences (ANOVA + Tukey's post hoc test ($p < 0.05$)).

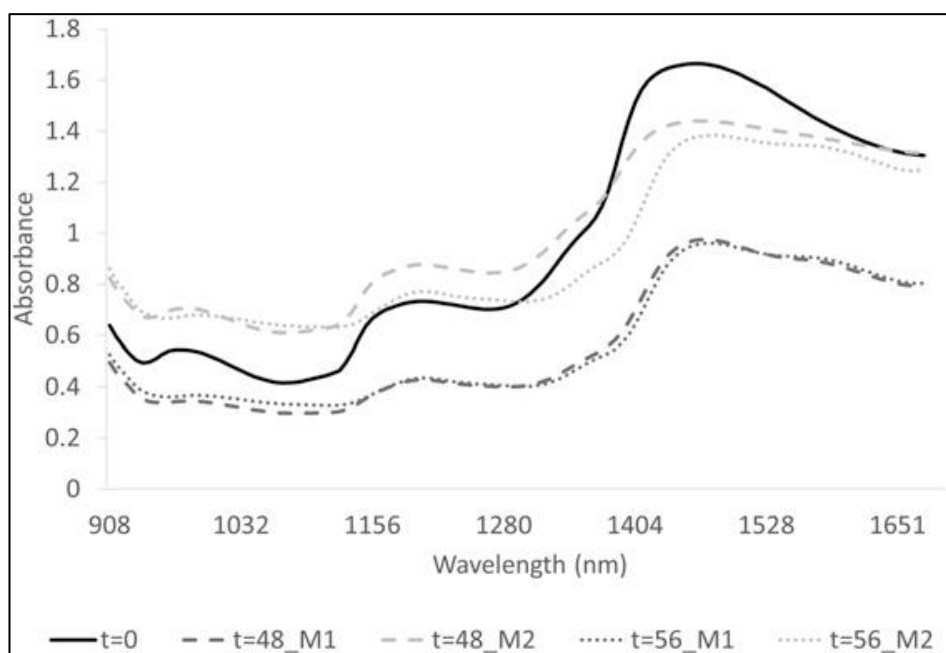


Figure 14 - Raw near infrared spectra collected at three different times (0, 48 and 56 h). $t = 0$ fresh product; M1 = drying plant; M2 = dehydration plant.

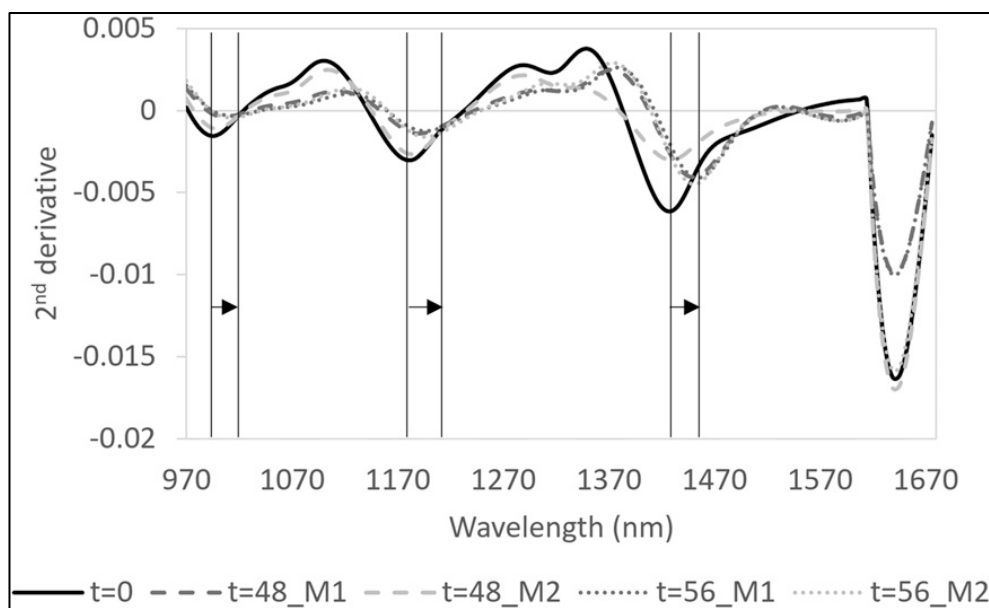


Figure 15 - Second derivative of mean pre-treated near infrared spectra collected at three different times (0, 48 and 56 h). $t = 0$ fresh product; M1 = drying plant; M2 = dehydration plant.

Figure 16 shows the normalized spectra truncated to 1300–1600 nm to focus on the first overtone of the water OH stretching vibration. Based on the common water absorbance bands found in normalized spectra, 12 wavelengths inside the WAMACs

ranges were selected for representation of spectral patterns of water samples using aquagrams: 1342, 1366, 1373, 1410, 1428, 1441, 1453, 1459, 1478, 1490 and 1515 nm. Each wavelength corresponds to absorption of specific water molecular species [10].

The Aquagrams highlighted differences among the various stages of the dehydration process, giving rise to different WASPs at each sampling time and for each dehydration device (Figure 17).

The $t = 48$ M1 aquagram was highly different from the one at the beginning of the process ($t = 0$) and very similar to the $t = 56$ one, meaning that the dehydration process was almost completed, and only minor changes occurred during the last 8 hours. Conversely, for the M2 plant, the 48- and 56-hour profiles did not overlap, indicating that after 48 h, the dehydration of the product was only partial and ended at 56 h. In fact, the $t = 56$ -M2 profile was very close to $t = 56$ -M1.

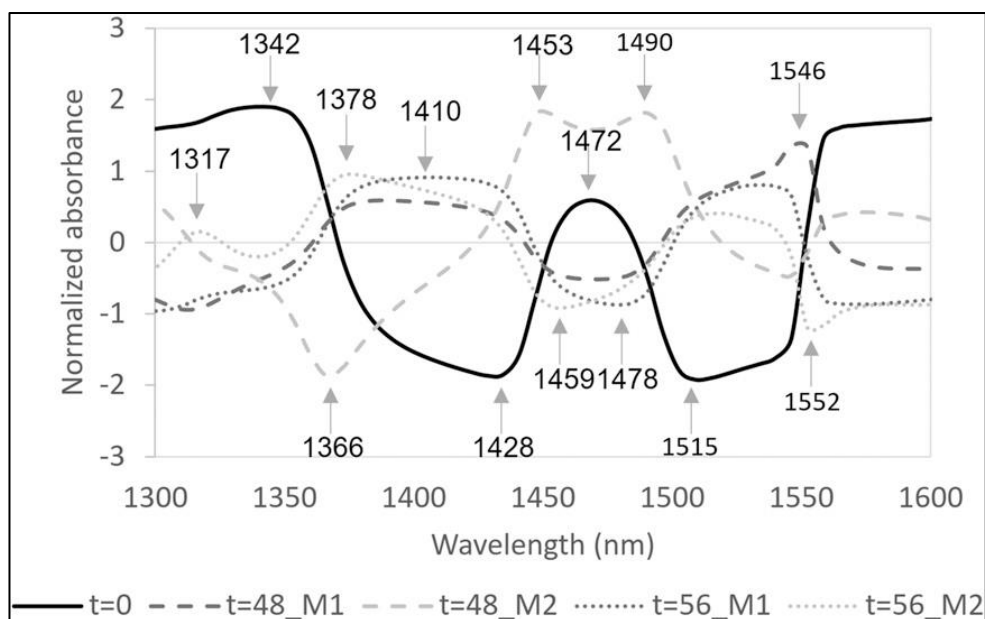


Figure 16 - Normalized near infrared spectra truncated to 1300–1600 nm at three different times (0, 48 and 56 h). $t = 0$ fresh product; M1 = drying plant; M2 = dehydration plant.

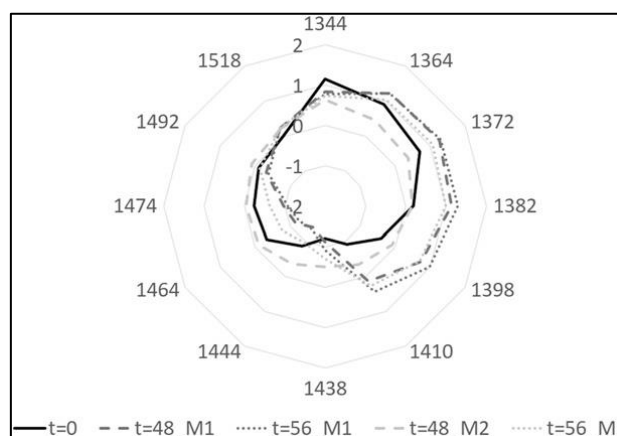


Figure 17 - Aquagrams: average of four slices at three different times (0, 48 and 56 h). $t = 0$ fresh product; M1 = drying plant; M2 = dehydration plant.

The shift of the WASPs from left to right as drying progresses highlighted the involvement of some wavelengths. According to literature data [9, 46] the absorption assignments were also in agreement with the physico-chemical explanation of the phenomena related to the drying process. These results suggest that water molecules were highly organized between the WAMACs C1 to C3 at $t = 0$ and between C2 to C6 for the other evaluated times, indicating more water molecules organized in water shells and less in water clusters.

When drying starts, the fresh product can retain water even in the form of more or less complex clusters, highlighting the presence of free and bound

water molecules. As the process progresses, due to the loss of water and weight, the fruit slices have a tighter tissue mesh, and as evaporation proceeds, the presence of free and trapped water molecules increases, while the absorption contribution of bonded water molecules decreases [7].

These preliminary results suggested continuing the study by applying LDA performed on few wavelengths selected by PCA (1342, 1364, 1412, 1452 and 1488 nm), avoiding the use of redundant information. LDA results are reported in Table 3.

Table 3 - LDA performed on wavelengths selected by PCA:
1342, 1364, 1412, 1452, 1488 nm.

		Actual class		
		T=0h	T=56h M1	T=56h M2
Predicted class	T=0h	46	0	0
	T=56h M1	0	47	8
	T=56h M2	0	1	39
% positive prediction		100	98	83

The obtained percentage of positive prediction confirmed the full discrimination between fresh and dried products, independently from the dryer plant used, and the high discrimination between the two plants with percentages of positive prediction equal to 98 and 83% respectively. Using few selected wavelengths to build up multivariate calibrations could have a positive effect on the instrument costs. Preliminary partial least squares regression was built up to determine titratable acidity content with very promising results (Figure 18), suggesting the possibility of controlling this parameter during the drying process.

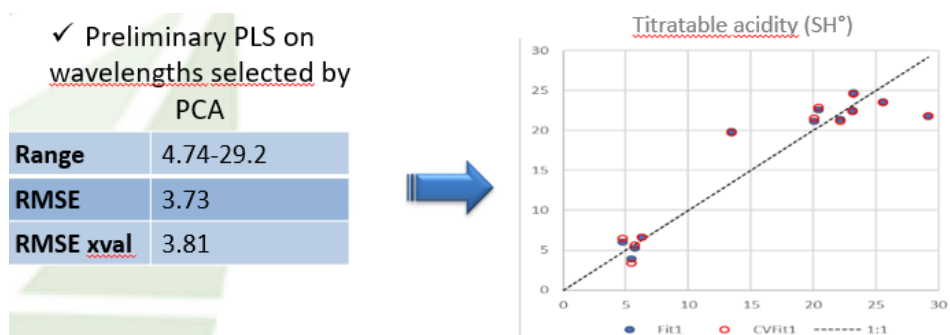


Figure 18 – PLS regression for titratable acidity.

The detected variations in WASPs confirmed the adequacy of the aquaphotomics approach and chemometrics such as PCA and LDA for studying the efficiency of drying processes and suggested its applicability for the continuous monitoring of food drying processes using an adequate NIR probe.

General remarks. M1 plant was the fastest system for drying fruit slices, leading to complete drying already after 48 h, while M2 required 56 h to reach the same degree of drying. However, the system's speed is related to the final quality of the dried product, and it will be verified whether the milder but more prolonged treatment of the M2 plant can have some positive influences in maintaining product freshness. These preliminary results also suggested the applicability of aquaphotomics for the continuous monitoring of food drying processes using an adequate NIR probe. Further experiments for continuous monitoring of the process are planned.

Second experiment (with sensors implementation)

The normalized data, monitored throughout the process, for each variable and relating to the three sites (M1, M2, Ext) are reported in Figure 19.

The diagrams report the trend of the different sensors monitored in each site. Regarding to M1, fan and relative humidity (RH) showed reverse trends, with an increase of RH during the night, when fan was inactive. Temperature trend was, as expected, in agreement with the fan trend.

In the M2 micro-plant, the temperature reached values higher than in M1 during the day, even if the maximum value was always below 48°C.

The difference between the temperature and humidity values recorded in the M1 type were statistically significant compared to the M2 type (ANOVA p-value <0.05).

The temperature reached in M2 with the fan programmed for a constant number of revolutions is more dependent on the external temperature. During the warmest hours of the day, it reaches higher levels than M1 in which the fan is programmed to counteract thermal excesses. The constant activity of

the fan in M2 allowed the reduction of the night-time RH% not contrasted in M1 in which the fan is inactive during the night.

The external monitoring confirmed that the environmental RH was always higher than inside the micro-plants, where the fan could insure forced escape of humid air.

The weight trend, measured by an external balance placed at the bottom of the micro-plants, allowed to follow the weight loss during the whole experiment.

Figure 20 highlights the results showed previously, analysing the collected data sensor by sensor, between sites and between positions (Figure 20a).

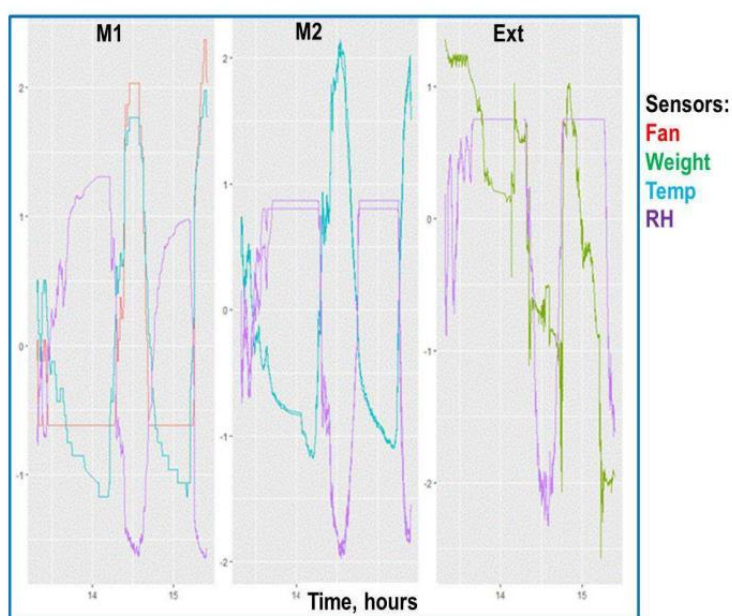


Figure 19 - Variables monitoring between sites. M1: drier; M2: dehydrator; Ext: external environment.

The time interval in which the fan was active in M1 is reported top left; the weight loss trend showed the effect of night humidity, highlighting an increase in the weight measured. The temperature and humidity trends between the two micro-plants, and in comparison, with the external values, are in agreement with the comments reported above.

The two last diagrams reported in Figure 20b, compare the data collected in two different positions within the same plant.

The recorded curves relating to temperature and humidity did not show statistically significant differences between the upper and lower area of the dryer when the fan was active or activated (ANOVA p-value=0.689).

The differences between internal and external values of RH were confirmed by the Tukey's post-hoc test at 0.95 confidence level.

Water activity values, reported in Table 4 as single tray and average value inside each micro-drier system, at the end of the drying process was never found higher than 0.500, proving the safety of the final dried product.

The micro-plant M2 seemed to be able to reach lower a_w values, corresponding to the limits of pollution.

Table 4 - Water activity (a_w) values at the end of drying processes. T1 (M1); T2 (M2); av = mean values; 1-4: samples position

T1.1	T1.2	T1.3	T1.4	T1av	T2.1	T2.2	T2.3	T2.4	T2av
0.4895	0.4872	0.4791	0.5004	0.4890	0.4679	0.4891	0.4989	0.4866	0.4856

The eggplant dehydration process was used as preliminary experimentation to assess

and optimize the dehydration process conditions to be validated in a subsequent experiment with the apple process.

In this first activity, some critical points were identified: a) the positioning of the NIR probe, which must remain in contact with the product throughout the process; b) the working temperature and the NIR probe insulation to avoid that temperature variations could have a detrimental effect on NIR data; c) the sample size; d) the identification of the process end.

Figure 21 shows the system's setup with the chosen configuration of the critical elements: NIR probe positioning over an eggplant slice at the beginning (a) and at the end (b) of the dehydration process.

The drying process lasted 50 hours and it was stopped when the samples reached a constant weight, considering a difference lower than 1% between two consecutive weightings.

At the end of the process, it was verified that the probe was still in the starting position and that it was still in contact with the slice of product.

This ending point corresponded to an a_w value of the dried product of 0.388. This value is well below the microbiological safety threshold identified as 0.6 [61] and indicates that the safety of the product was ensured.

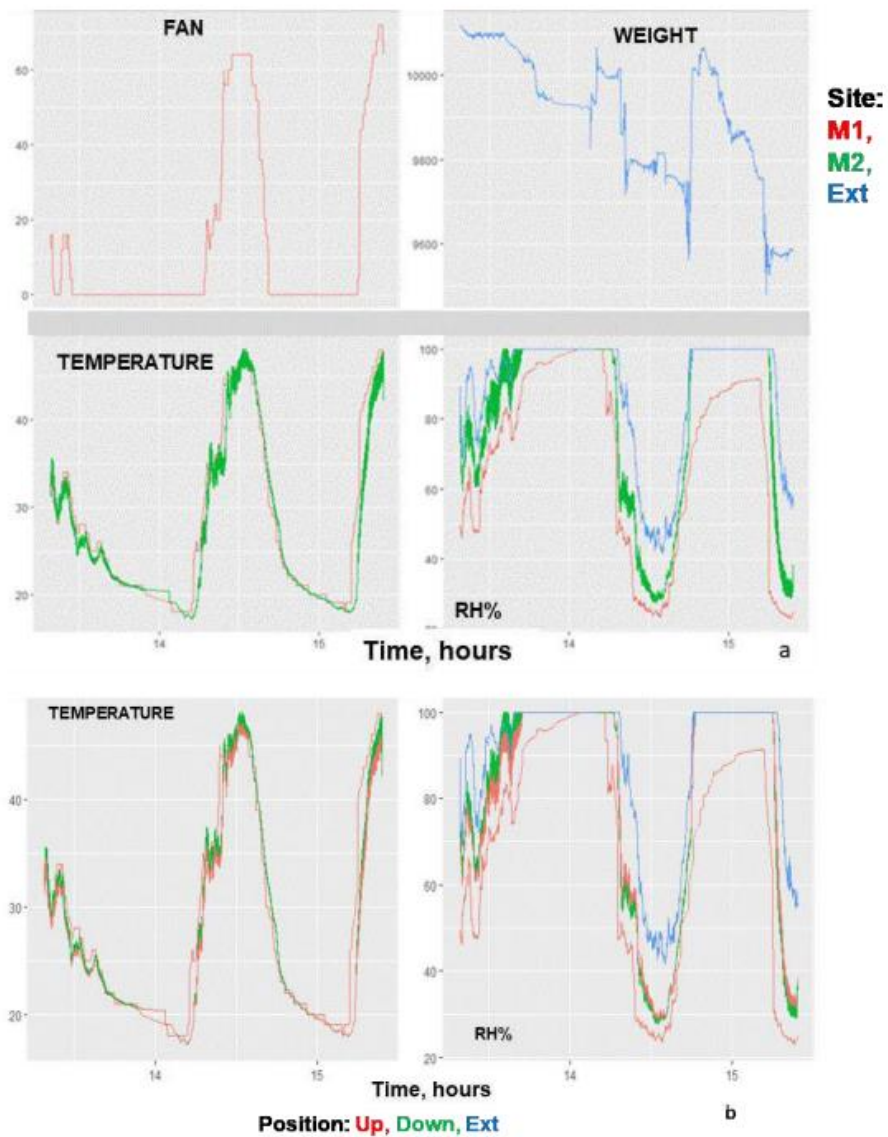


Figure 20 - a) Variables monitoring between sites and between positions. M1: drier; M2: dehydrator; Ext: external environment. b): Up/Down = trays.

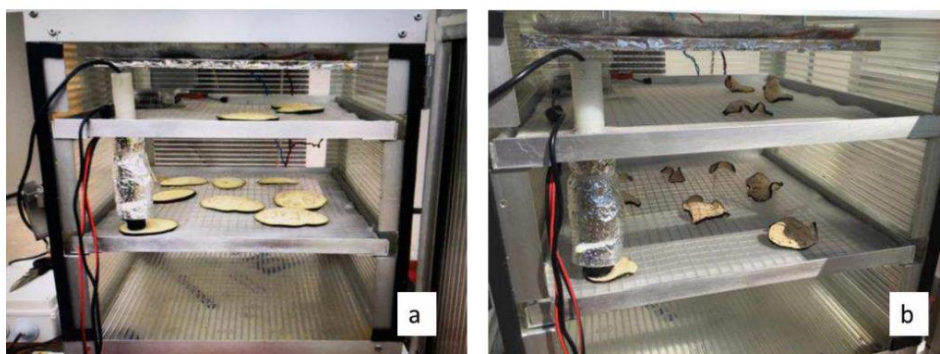


Figure 21 - NIR probe positioning at the beginning (a) and at the end (b) of the dehydration process of eggplants.

Spectra collected during the eggplant dehydration process are shown in Figure 22. The raw spectra (Figure 22a) showed broad and overlapped absorption bands detecting predominant water absorption bands at 960-990 (2nd overtone -OH stretching), 1150-1190 (combination of 1st overtone -OH stretching and OH bending), and 1400-1435 nm (1st overtone -OH stretching) [46]. The application of the 1st derivative calculation as pre-treatment (Figure 22b) allowed better visualization of the changes in the spectral profile during the process. In particular, great variations were recorded in the spectral region between 1300 and 1600 nm, associated to the first overtone of water, indicating the possibility of using the Aquaphotomic approach to monitor the process.

The first two principal components of the PCA performed on the pre-treated spectra explained 99% of the total variance. The trend of the scores defines the positioning of the samples in the multivariate space according to the progress of the process (Figure 23a). PCA score plot loadings confirmed significant variations in the first overtone of the water spectral region (Figure 23b).

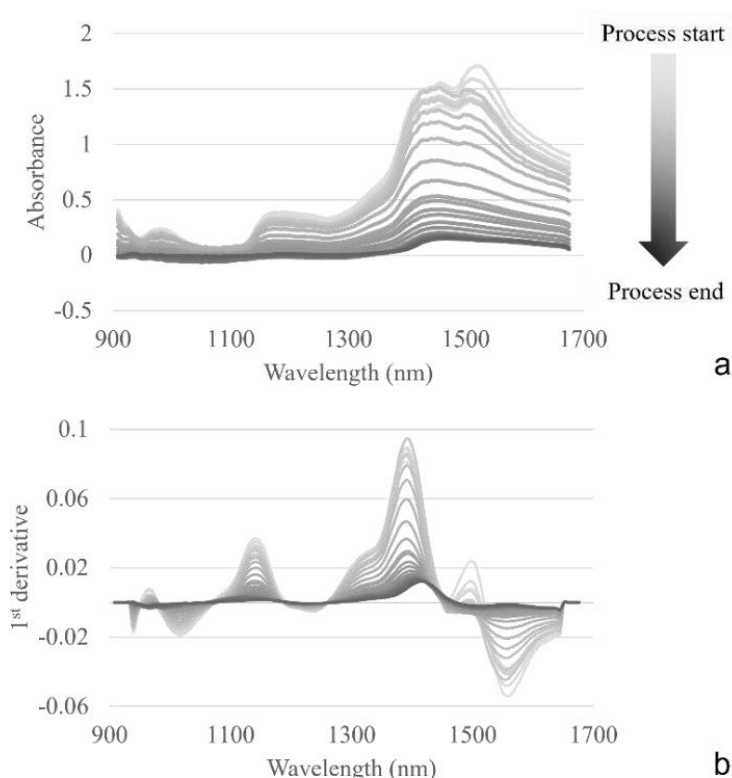


Figure 22 - Raw (a) and 1st derivative (b) NIR spectra of the eggplant dehydration process.

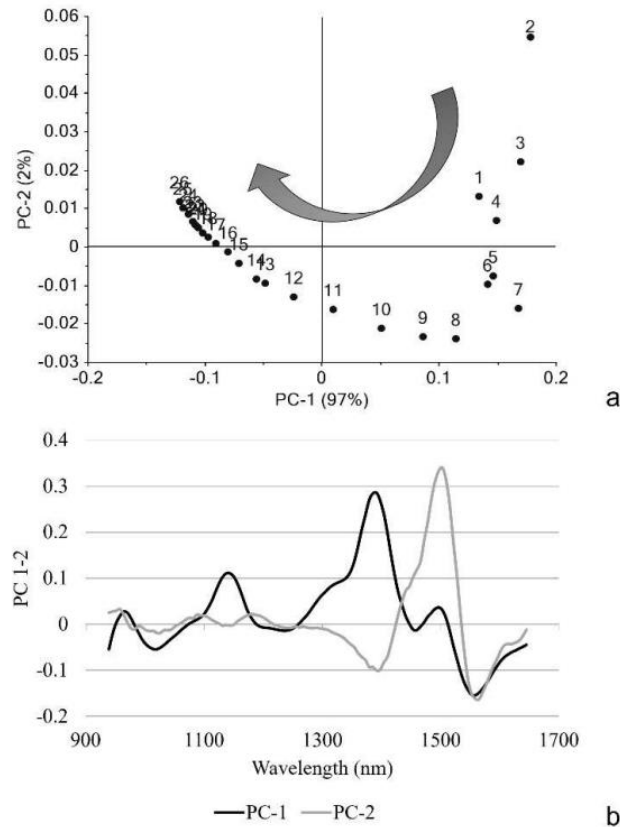


Figure 23 - PCA scores (a) and loadings (b) plot performed on eggplant pre-treated spectra.

Figure 24a provides the results of MBM applied to the acquired data using a block of 3 spectra, which means that the first point represents 1-3 spectra, the second point represents 2-4 spectra and so on. The trajectory of the MBM showed a continuous decrease during the process and tended to a constant value when repeated low values of variability were recorded, greatly helping in identifying the process endpoint. According to the PCA loadings, it can be assumed that the MBM trajectory reflects the modifications of the water component during the process.

Based on the water absorbance bands found in pre-treated spectra according to Tsenkova et al. [46], 12 wavelengths were selected for representation of spectral patterns of water samples using Aquagrams: 1342, 1366, 1373, 1410, 1428, 1441, 1453, 1459, 1478, 1490, 1515 nm (Figure 24b). Each wavelength corresponds to the absorption of specific water molecular species [46]. A shift from left to right was observable during the dehydration process, which means moving from a system characterized by the prevalence of bonded

water forms to a situation in which free water molecules prevail due to the evaporation process.

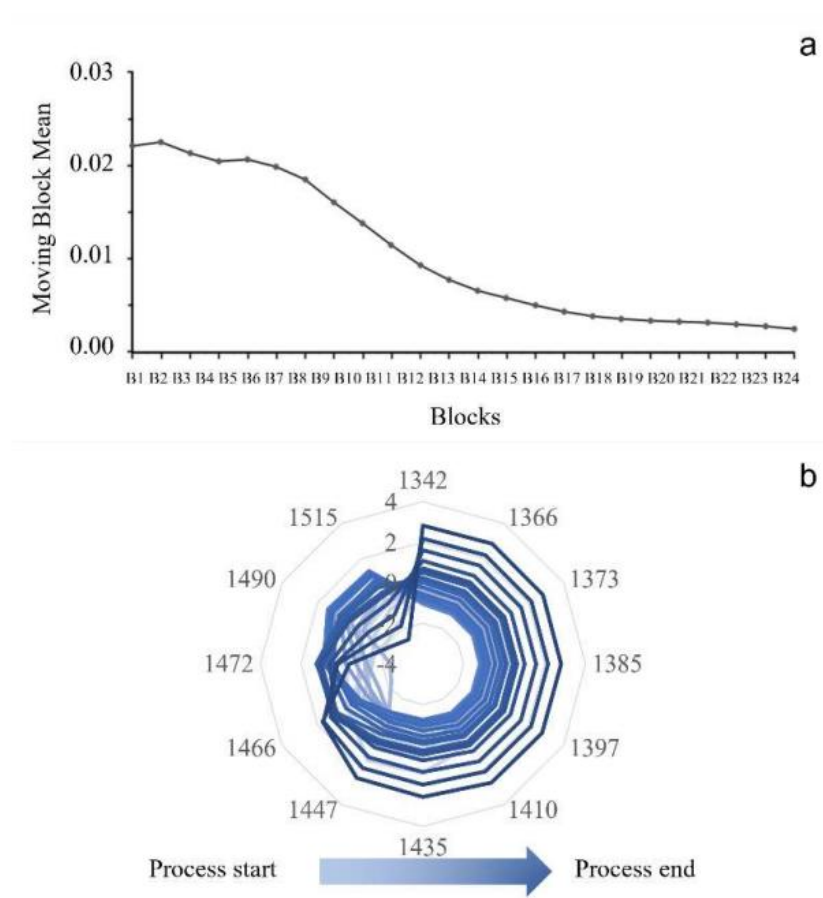


Figure 24 - Eggplant Moving Block mean trajectory (a); Aquagrams (b).

To improve the process and confirm these first results, the apple dehydration process was carried out.

The drying process lasted 61 hours, and it was stopped when the samples reached a constant weight as above specified. At the end of the process, the a_w value of the dried product was equal to 0.329.

For this process, a commercial thermally insulated NIR probe housing was used, and

the number of acquired spectra was increased by increasing the acquisition frequency to one spectrum per hour.

The PCA (Figure 25) on these spectra confirmed the involvement of the water absorption bands even if a different matrix was used.

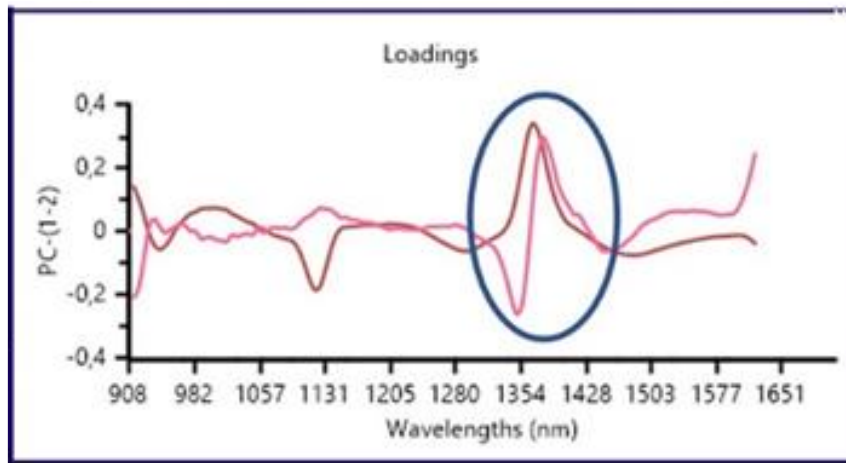


Figure 25 – PCA loadings of apple spectra during the dehydration process.

For this trial, MBM blocks of 7 spectra were used to build up the chart and establish

the endpoint criteria.

Based on MBM trajectory, lower and upper limits of 0.0365 and 0.0368 were tentatively set (Figure 26a), to verify the MBM suitability in identifying the process end.

According to these limits, the spectra corresponding to the block B43 should belong to completely dehydrated samples. The same spectra were represented as yellow lines in the Aquagram of Figure 26b.

These spectra did not correspond to the completely dried samples. They were located in the right side of the Aquagram (Fig. 26b) and characterized by a water pattern with a prevalence of free water molecules.

However, they did not fit with the sample on the far right of the chart. Therefore, by

combining the information deriving from the two approaches, we should identify the

actual end of the process corresponding to a block within the range defined by the end-point criteria beyond the B43 block.

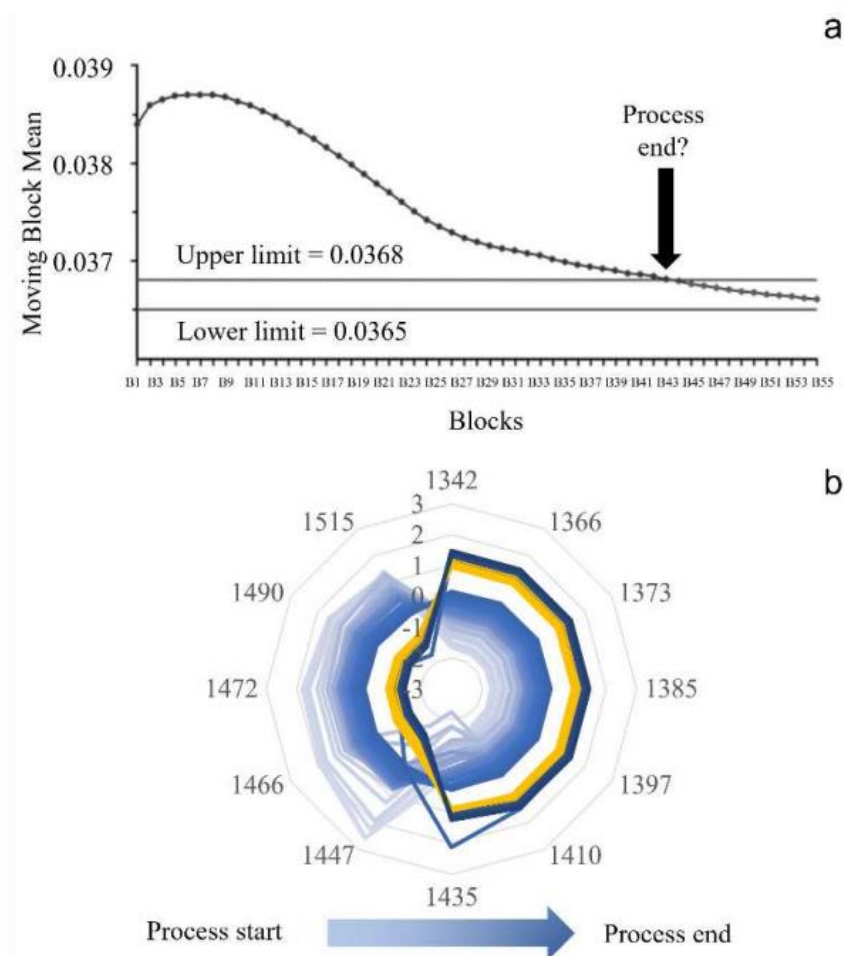


Figure 26 – Apple Moving Block mean trajectory (a); Aquagrams (b).

General remarks. The efficiency of two different transformation processes was proven. Both micro-plants worked to reach a good level of dehydration after 50 hours.

The chosen digital methodologies were suitable to in continuous monitor the dehydration process. The process standardization can be easier by using M1, in which fan worked at controlled temperature, and operating as a mild oven. Conversely, M2 suggested higher probability in maintaining the original nutritional value of the dried products and working as «natural dehydration». In this case, in fact, only non-bound water is removed from the product, ensuring better preservation of the nutritional principles of the fresh product. The choice of their use is strictly related to the final aim of transformation. For example, if the need to standardize the process prevails, the use of M1 is

recommended; if it is possible to accept limited climatic variations that can affect the

duration of the process, M2 is better suited to the production of high quality dehydrated products.

Furthermore, the obtained results suggested the applicability of Aquaphotomics for the continuous monitoring of food drying processes using an adequate NIR probe. PCA loadings confirmed the important contribution of the water absorption in the wavelength range 1300 1600 nm during the dehydration processes independently from the food matrix. The great variations recorded in the spectral region of the first overtone of water, suggested the possibility of using the Aquaphotomic approach to follow the dehydration process. Thus, Aquagrams can help to identify the actual endpoint of the process, due to the detection and the monitoring of the different water forms (free water, dimers, aggregates, clusters, etc.). The combination of information from the two chemometric instruments, MBM and Aquagrams, provided a valuable tool for defining and predicting the end of the dehydration process. The choice of the proper size of blocks is crucial in order to gain a good predictive power of the MBM.

The selection of few wavelengths to study food processes and quality can optimise the transfer of instrumental data to IT platforms and networks.

Further investigations are in progress to check the possibility of transfer the obtained results on a pilot scale for future applicability of these applications. Based on the first year results, also if the standardisation of the process can be easier using the solar drier plant (M1), the dehydration plant (M2) was chosen as useful tool to continue the PhD activities on the basis of the following considerations:

- both procedures are in full agreement with the objectives of the Agenda 2030, assuring a correct use of the alternative energy resources;
- a “natural” process fit the goal of the maintenance of the binding between “tradition” and “innovation”, reproducing the old process of solar drying but in a protected environment;
- the M2 configuration could be easily transferred not only in Italy but also in emerging Countries;

- the availability on the market of dehydration plants, flexibles for dimensions and size;
- a prototype of small size (pilot plant) is also active at CREA.IT Department in Milan;
- this type of plant is already active, without digital control, in cooperatives and farms closed to Milan.

4.2 Scale transposition tests

Two different matrices were tested: onion slices and melon slices.

Physical sensors. Regarding the monitoring of physical parameters by sensors, the in-line control of the “in” temperature and RH% variations confirmed the results obtained by micro-plants, proving the correct protocol transfer into pilot scale level.

Figure 27 showed the trend of the two indices during both drying processes.

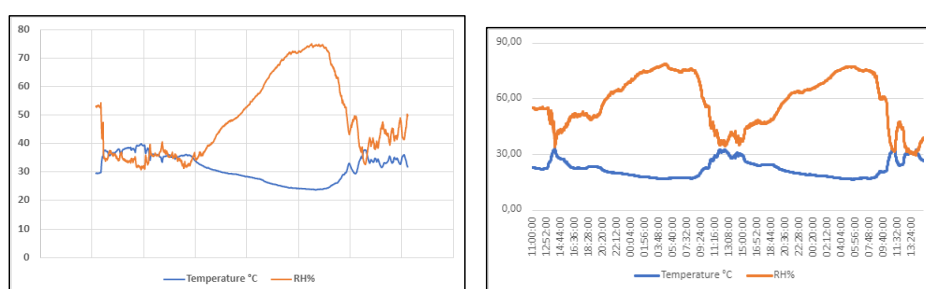


Figure 27 - Continuous monitoring by Arduino sensors of temperature and RH% inside the plant. Left: onion slices; right: melon slices.

The temperature values ranged respectively from 40 to 25°C, and from 33 to 20°C for onion and melon experiments. This finding was due to the weather conditions in the trial days. RH% values highlighted the increase of humidity during the night. For melon, due to the process duration, two RH% increases were recorded (two nights).

The trends of continuous monitoring of external temperature and RH% by using a data logger are reported in Figure 28.

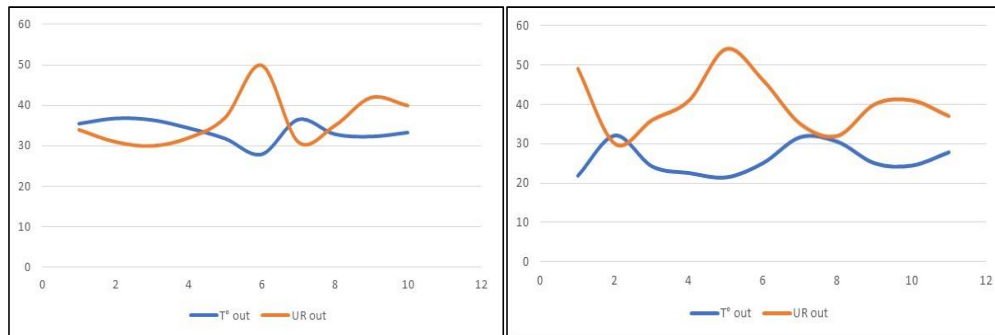


Figure 28 - Continuous monitoring by datalogger of external temperature and RH% (left: onion drying; right melon drying).

It can be noted that the external RH% was lower than the internal one (Figure 27) in particular during the night, conversely the inside temperature was in general higher than the external one, proving the solar power as source of thermal energy.

A great variation of internal relative humidity was highlighted. In both the process marked RH increases were observed between approximately 18-22 hours and 43-47 hours (only for melon), which corresponded to the maximum humidity accumulated overnight.

The trends of the RH measured inside the dryer were very similar to those of the outside but remained significantly lower. It was evident that the external humidity did not affect the processes, proving the efficiency of the solar drying system employed, effective in removing humidity thanks to the ventilation system.

Solar drying is a process highly dependent on the temperature and humidity of the external environment. During the night, the increase in external humidity leads to the partial rehydration of the product.

Physico-chemical analysis. The drying process was stopped when the samples reached a constant weight (29 hours for onion; 47 hours for melon).

The trend of the weight loss and the water activity (a_w) during the drying processes are shown in Figure 29.

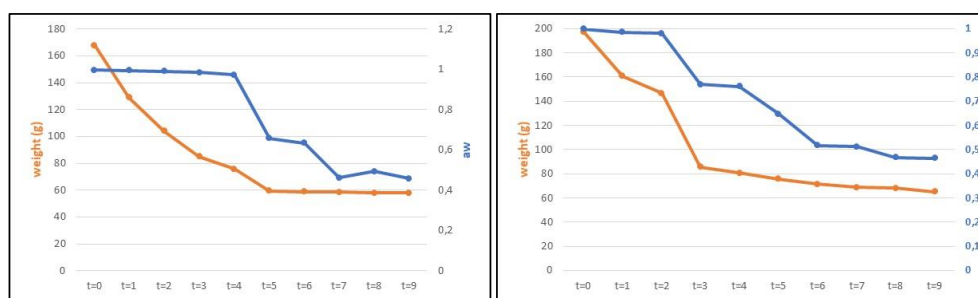


Figure 29 - Weight loss and water activity (a_w) curves during the drying process (left: onion drying; right melon drying).

Product weight, moisture content (MC) and water activity (a_w) values gradually decreased during the drying time until they reached a constant level considered the end of the process. The drying yields were 35% for onion and 33% for melon samples. MC and a_w of onion varied from 90.3 to 16.3% and from 0.9975 to 0.4552, respectively, while MC and a_w of melon varied from 89.4 to 17.8% and from 0.9994 to 0.4666, respectively. At the end of the dehydration process, both products showed MC values of around 16/17%, a value very close to the limit of 15% reported in the literature to be adequate to minimize bacterial, yeast and mould growth [63] and to extend products shelf life. Similarly, the dehydrated products showed a_w values around 0.45, far below the microbiological safety limit set at 0.6, making all the dried products safe in respect to microbial hazard.

Even before involving the spectral data, knowing some statistics of the reference data, in particular the standard deviation and the SEL, it was possible to calculate the maximum R^2 obtainable for each calibration. For onion process, the R^2_{Max} values of MC and a_w reference methods were 0.997 and 0.999, respectively, while for the drying of the melon R^2_{Max} of 0.999 and 0.994 were calculated for MC and a_w methods, respectively. These values would be achievable if no errors were introduced in the spectra or in the model [56]. These results demonstrated the adequacy of the datasets in supporting the construction of predictive models.

NIR analysis. NIR spectra were collected under fluctuating conditions of temperature (T_{in} onion= 29-39°C; T_{in} melon= 17-33°C) and relative humidity, typical conditions occurring during the solar drying processes. The obtained spectra are reported in Figures 30-31.

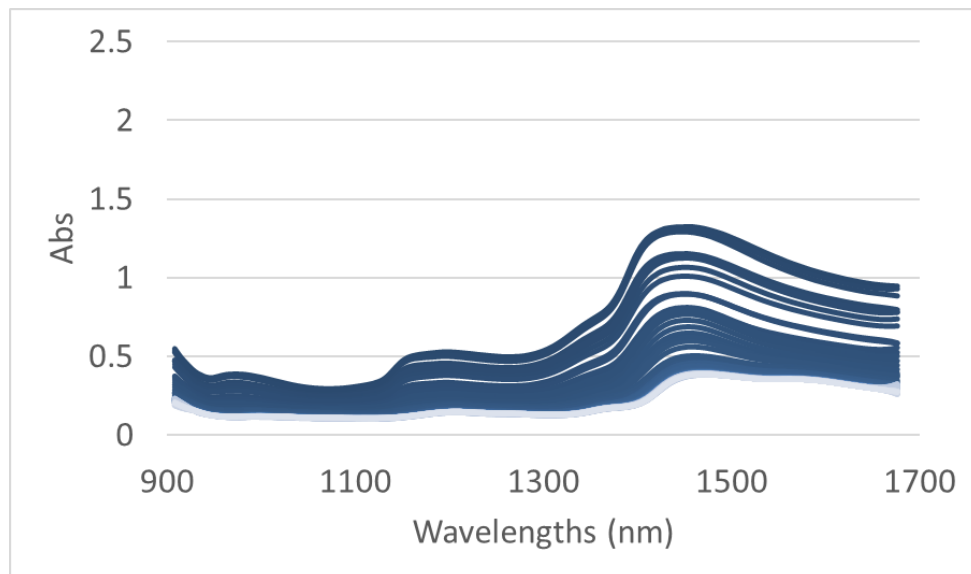


Figure 30 - NIR spectra recorded continuously in real time during the onion drying. Dark lines correspond to the fresh and moist product; light lines correspond to the dry product.

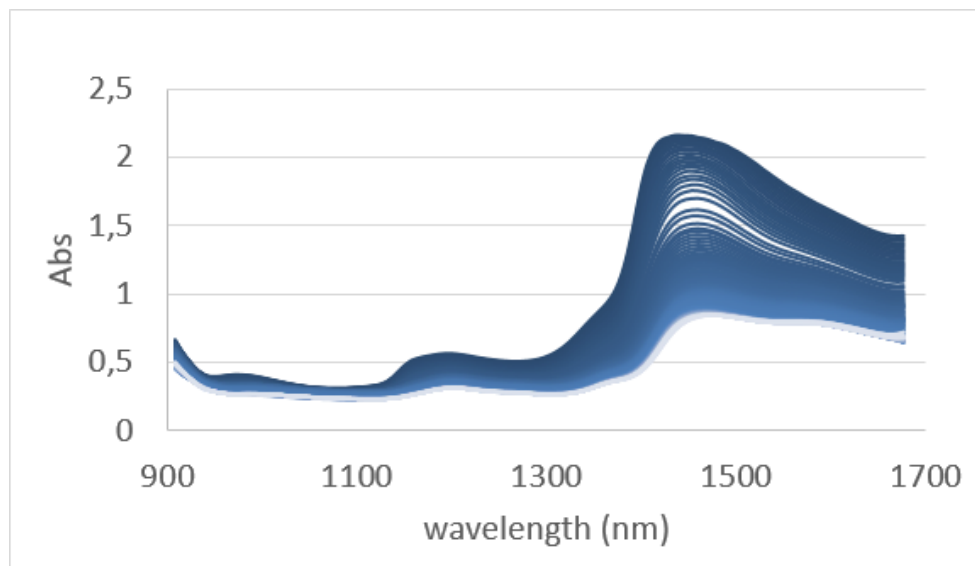


Figure 31 - NIR spectra recorded continuously in real time during the melon drying. Dark lines correspond to the fresh and moist product; light lines correspond to the dry product.

During dehydration, the decrease in moisture content of samples resulted in the decrease in water absorbance intensity in the NIR spectra. The greatest spectral variability was observed in the first 5 hours of the process, then the spectra tended to flatten out and showed less variability. In both reported cases, there was a decrease of the bands at 950-990, related to 2nd overtone of the -OH stretching, at 1150-1200, associated to a combination of 1st overtone of the -OH stretching and OH bending, and at 1440-1450 nm, representative of the 1st overtone of the -OH stretching [64]. The greater spectral variability of the first hours of dehydration reflects the rapid weight

loss of the product recorded in the first 5 hours of the process. This could indicate that a large part of the water in the fruit tissues was in the free or loosely bound form and that the diffusion rate in the tissue was high [65]. The constant trend that was observed in the subsequent phases both in the spectra and in the weight loss can be also related to the tissue shrinkage occurring when most of the water has been removed and which limits the diffusion of water towards the outside of the slice [66]. Both the absorbance at 1450 nm and around 1200 nm are reported to exhibit a linear decrease with decreasing availability of bulk water (water activity). Moreover, the absorption peak around 980 nm is reported to be related to bulk water properties and, being strongly correlated to water activity, it can be used to determine this parameter [62].

Regression models. The regression models for the prediction of MC and a_w values are summarized in Table 5.

Table 5 - Statistical descriptors for NIR calibrations for the prediction of MC and a_w values during drying process.

Samples	Index	Range	LV	R^2_{cal}	RMSEC	R^2_{CV}	RMSECV	RER	RPD_C	RPD_{CV}
Onion	MC	90.3 - 16.3%	7	0.99	0.76	0.94	6.37	11.2	10.0	4.1
	a_w	0.998 - 0.455	7	0.99	0.005	0.95	0.05	6.2	10.0	4.5
Melon	MC	89.4 - 17.8%	5	0.99	1.63	0.99	2.49	28.1	10.0	10.0
	a_w	0.999 - 0.467	6	0.99	0.02	0.97	0.04	13.8	10.0	5.8

PLS regression models showed good ability in predicting MC and a_w of onion and melon slices during drying. The best models were obtained with the application of SNV spectral pretreatment in combination with second derivative Savitzky–Golay transformation (gap size 11 points) and mean centering before multivariate modelling. The removal of uninformative features was crucial in developing good predictive models. The models showed R^2 of 0.99 in calibration and higher than 0.94 in cross validation. The errors in cross validation were very low, ranging between 2.49 and 6.37 for the prediction of MC of melon and onion samples, respectively, and between 0.04 and 0.05 for the prediction of a_w .

The RPD_C ratios were around 10.0 for MC and a_w models. RPD values in cross validation were lower, ranging from 4.1 to 4.5 for MC and a_w of onion, respectively and from 10.0 to 5.8 for MC and a_w of melon, respectively.

According to the classification of Williams [41], the MC models were useful for screening purposes having $RPD_{CV} \geq 3$ and, in agreement with Nie et al. [67], the models can be considered successful ($R^2 > 0.9$ and $RPD > 3$). Furthermore, the RER values for MC and melon aw prediction models were found to be higher than the recommended ($RER > 10$) for screening purposes [55].

The scatter plots of the models are reported in Figures 32-35. Blue dots represent the samples used to build the model while the red ones are the samples used in cross validation. The black line is the bisector 1:1 (y is equal to y measured).

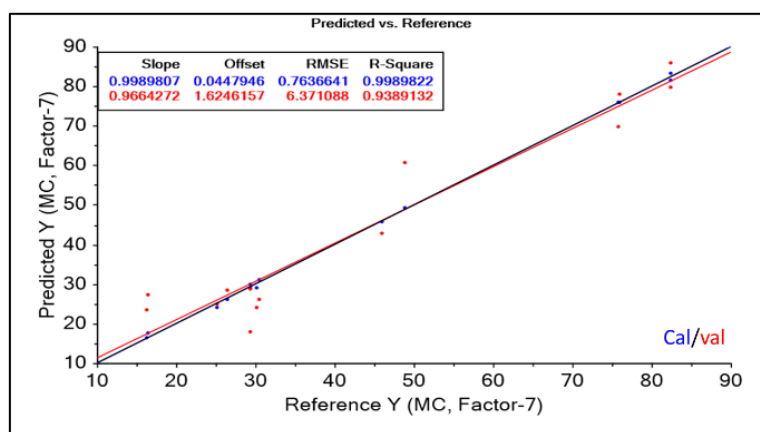


Figure 32 - Scatter plot of regression model for the prediction of MC in onion samples.

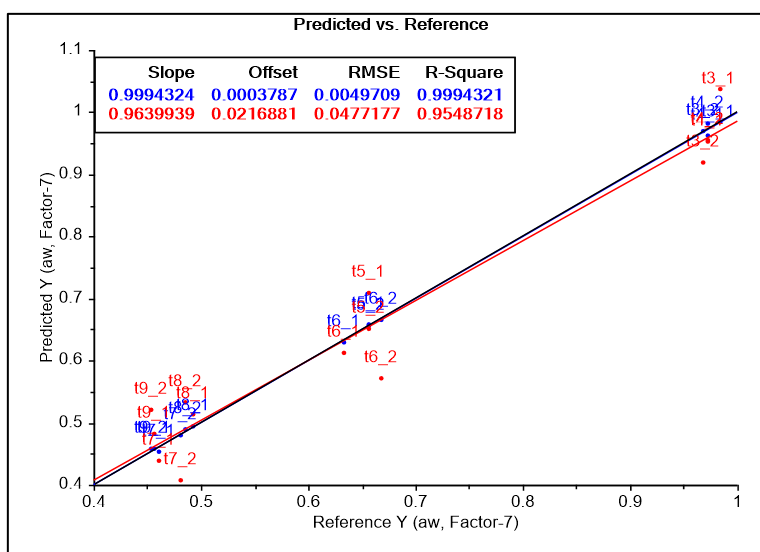


Figure 33 - Scatter plot of regression model for the prediction of a_w in onion samples.

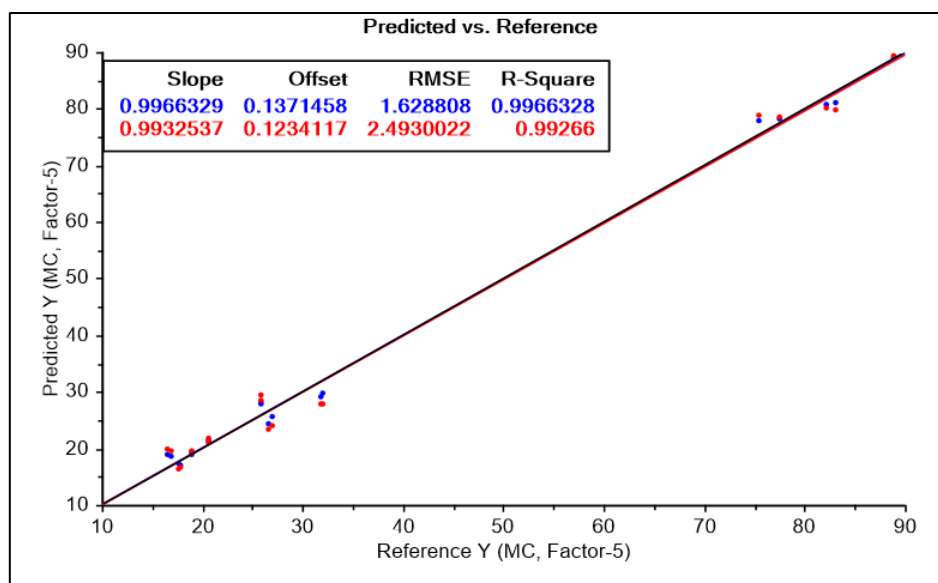


Figure 34 - Scatter plot of regression model for the prediction of MC in melon samples.

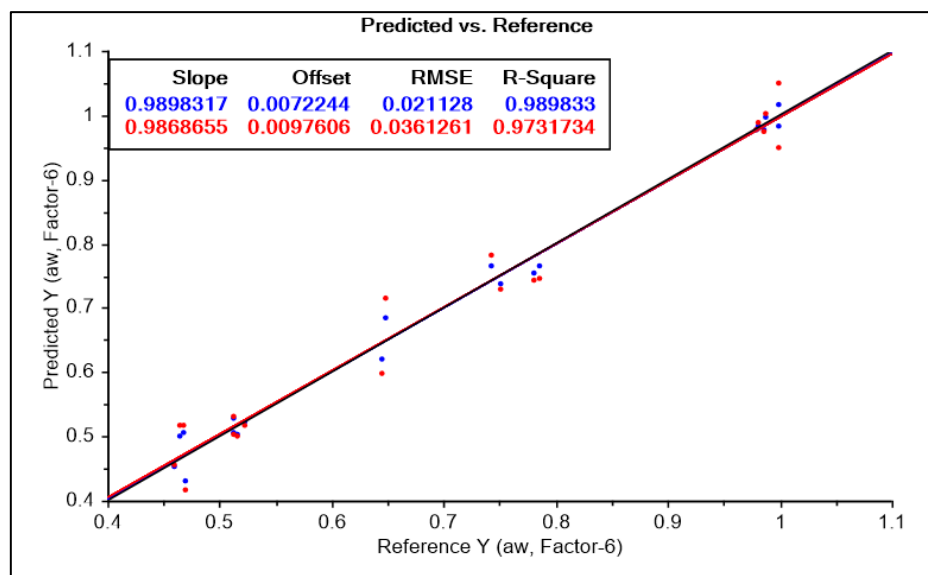


Figure 35 - Scatter plot of regression model for the prediction of a_w in melon samples.

The results of PLS regressions for melon samples were used for the prediction of the MC and a_w profiles along the drying process. Figures 36-37 show the comparison of the analytically determined parameter (green circles) with the predicted profile obtained from the online NIR spectra and the PLS regression model (red circles).

In this case, the moisture content resulting from the NIR spectra provides a good estimation of the data analytically measured, also if the increase of the sampling frequency is necessary to have more robust model.

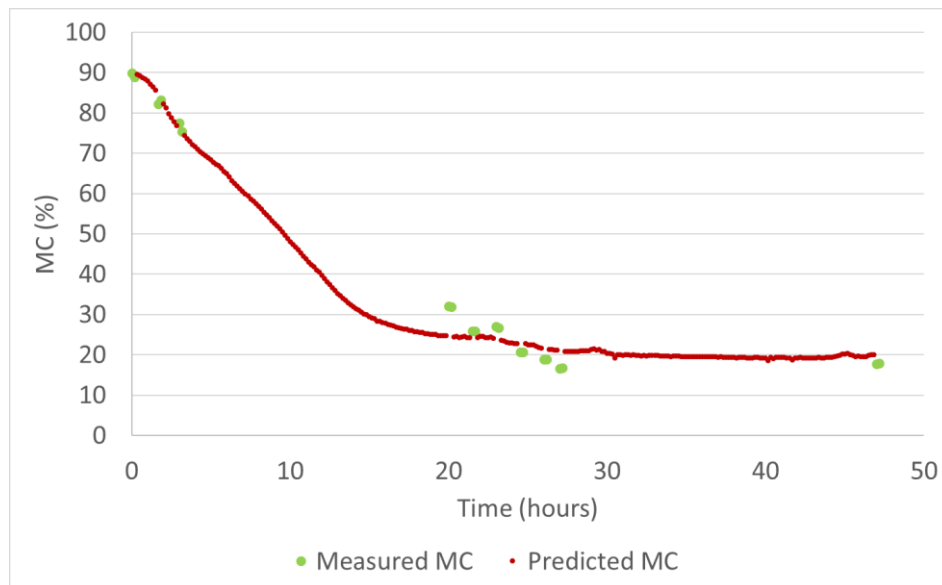


Figure 36 - Trend of MC during the melon drying process predicted applying the NIR (900-1300 nm) regression model.

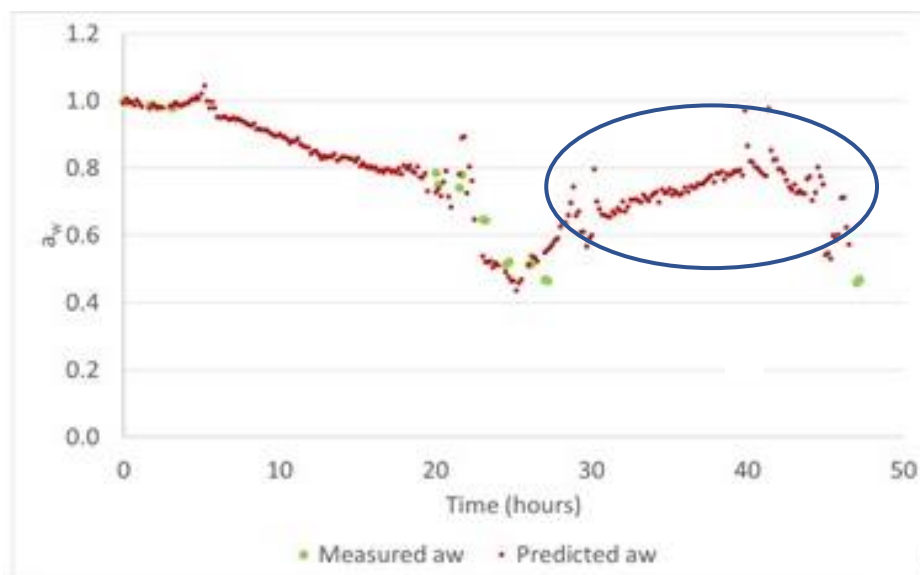


Figure 37 - Trend of a_w during the melon drying process predicted applying the NIR (900-1300 nm) regression model.

The prediction of the a_w trend showed an anomalous trend in the last part of the process, from the 27th to the 45th hour, providing higher estimates than expected. This can be related to the fact that the process was not interrupted at the 27th hour upon reaching an a_w value of 0.45. To be sure that the product was completely dehydrated, the process was extended for a further night. Consequently, during the night hours the product may have undergone a partial natural rehydration due to environmental conditions of high humidity.

Aquaphotomics. The whole data sets of spectra collected in continuous mode every ten minutes, respectively for a total of 170 spectra for onion and of 284 spectra for melon slices (from process start to process end) were processed to obtain the aquagrams. Based on the loadings of PLS regressions, reported in Figure 38, twelve wavelengths inside the WAMACS range (C1-C12), and mainly involved into the water molecules rearrangements, were selected for the onion and melon aquagrams construction: 1348, 1360, 1373, 1385, 1410, 1422, 1441, 1453, 1466, 1478, 1490, 1509.

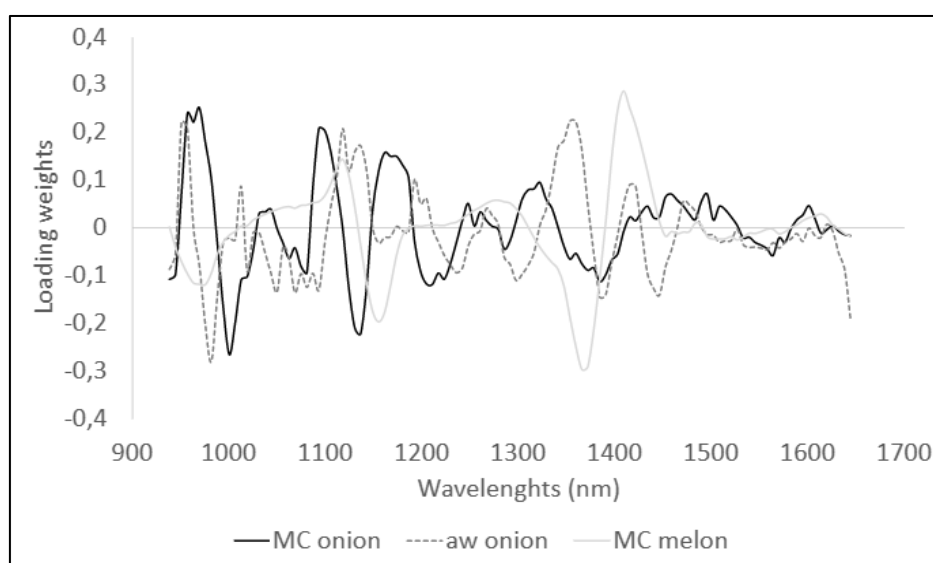


Figure 38 – Loadings plot of involved wavelengths for PLS regressions.

MSC and second derivative Savitzky–Golay filter (second order polynomial fit and 21 points) were applied to the spectra of onion samples, as pre-treatments to remove potential scatter effects, while only MSC was used as pre-treatment in the experiment carried out on melon samples.

Figures 39-40 report the spider charts for onion and melon dehydration processes respectively: selected spectra are displayed to make the graphs easier to read.

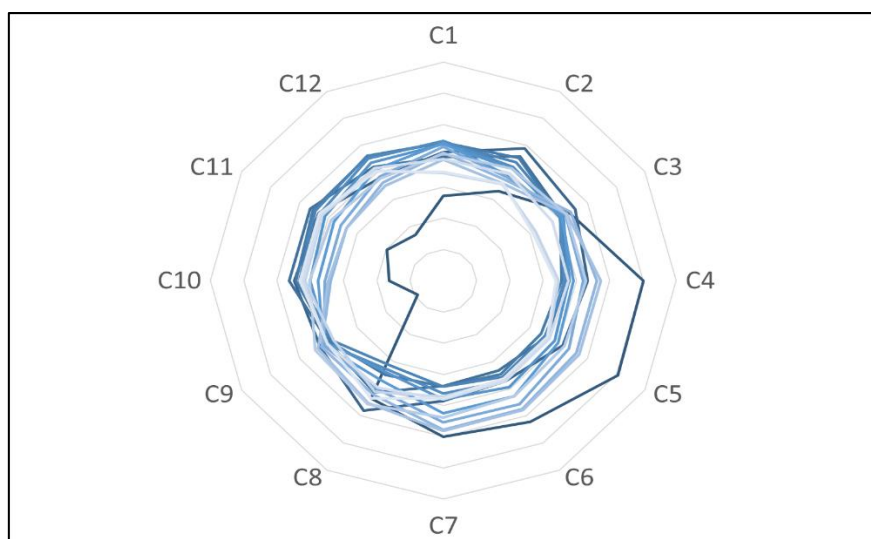


Figure 39 – Aquagram of onion spectra during the dehydration process. Dark blue: process start; light blue: process end.

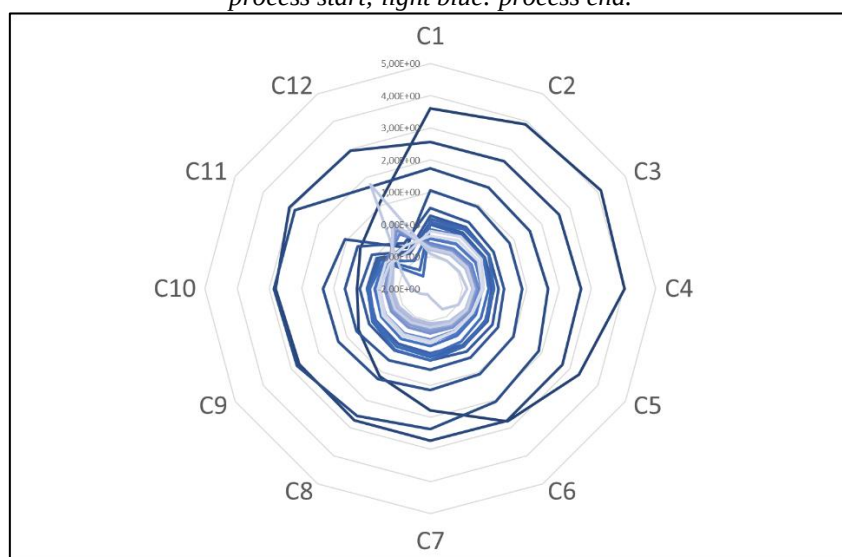


Figure 40 – Aquagram of melon spectra during the dehydration process. Dark blue: process start; light blue: process end.

This approach proved to be useful to monitor the whole dehydration process through the changes in water profiles. In both cases, it can be noted the shift caused by water rearrangemnts from blue dark lines (when processes started) to ligh blue lines corresponding to processes end.

Furthermore, each product showed a specific trend, suggesting the peculiarity associated to each horticultural product, suggesting the need to draft one dedicated protocol for each process.

General remarks. These results, obtained using a pilote plant, suggested the possibility of using NIR for monitoring the progress of the dehydration

process also after the scale transposition. The approach used on micro-plant proved to be useful for its application on larger plants.

The development of real-time predictions of MC and a_w could be a useful tool for detecting the end of the dehydration process and for assessing the potential safety of the final product. More relevant information on the model applicability could be obtained by validating them with an external dataset. In some cases, the reduction of the spectral range used in developing NIR models can reduce the noise due to non-contributing wavelengths and could lead to the development of lower-cost systems for the application of the models. The Aquaphotomics approach proved to be useful to monitor the whole dehydration process through the changes in water profiles. The development of a dedicated app would be useful to collect and process the spectra in real time providing an accurate prediction of the parameters of interest.

4.3 Activation of tests on a commercial system

Ca' dal Saggia farm (Figure 41) is a small multifunctional family-run farm. The legal representative of the farm is the agronomist Dr. Andrea Sala. The farm has been established in 2015. The company owns 5 hectares of land, of which 70% is sowed with stable grass and alfalfa. These crops are used to feed the goats with the subsequent production of cheese and meat for family sustenance.

The company strategy provides for high quality food productions obtained in the most sustainable way possible.



Figure 41 – Ca' dal Saggia farm, land, logo, and products.

About 20% of the land is cultivated with vegetables, another 10% with mixed orchards; a small part is woodland.

The farm works in a short supply chain with direct sale for the purpose of acquiring value and safeguarding the flavours of the territory. Inside, a laboratory for the food transformation was set up where jams, juices, syrups, and preserves are produced.

By pursuing a perspective of sustainability, the company uses a synergistic/associative strategy, cultivating plants and varieties typical of the territory associated with crops capable of contrasting the growth of parasites. Very few chemical treatments are carried out and treatments are replaced by *Bacillus thuringensis* against the dorifera attacks for potatoes, lactobacilli and probiotic saccharomycetes in the litters and in the manure to encourage the production of fertilizers, and then Liliaceae for parasites with the production of thiocyanates against bedbugs and aphids. Furthermore, tomato cultivation is made not using the classic rows. The farm has in addition two apiaries near the garden and orchard for the production of honey and for the integrated struggle, making use of bees as a deterrent agent for other insects. Biodiversity is also safeguarded using borders with hedges and flowers capable of attracting bees and butterflies.

Over the past three years, Ca' dal Saggia has expanded the spectrum of action by pushing itself to the consumer and activating a mobile kitchen for agricultural street food "*from farm to fork*". This activity is able to ensure better income and, at the same time, to encourage knowledge of niche products and ancient varieties of the Pavese territory.

Since November 2021, this small farm has been part of the Slow Food "Food Producers Community" together with about other 20 farms of the Oltrepò Pavese.

A real Slow Food protection has not yet been established, but this Community open to producers, transformers, restaurants and schools is a first network aimed at enhancing local niche products - 8-lines corn, Voghera pepper, ancient grains, chestnut honey, Oltrepò woods - in addition to most known oil, wine, cured meats and wheat.

Since 2018, for the diversification of the offer, Dr. Andrea Sala started the production of dried horticultural products, initially using a counter dryer which required a high energy consumption, then purchasing the solar dehydrator "Italo Base+".

The dehydration plant is mainly active from April to October, when the climate conditions are favourable to easy collect solar energy.

Two experimental tests were carried out on apple slices (*Red Delicious* and *Fuji* varieties; 1.5 mm thickness), for the verification of the real transferability of the studied technologies in industrial realities with a view to "precision factory".

Physical sensors. The processes were monitored by using Arduino sensors for the collection of temperature and RH% values for *Red Delicious* variety, and by datalogger for *Fuji* samples. Both processes were monitored for 55 hours. The recorded variations are reported in Figures 42-45.

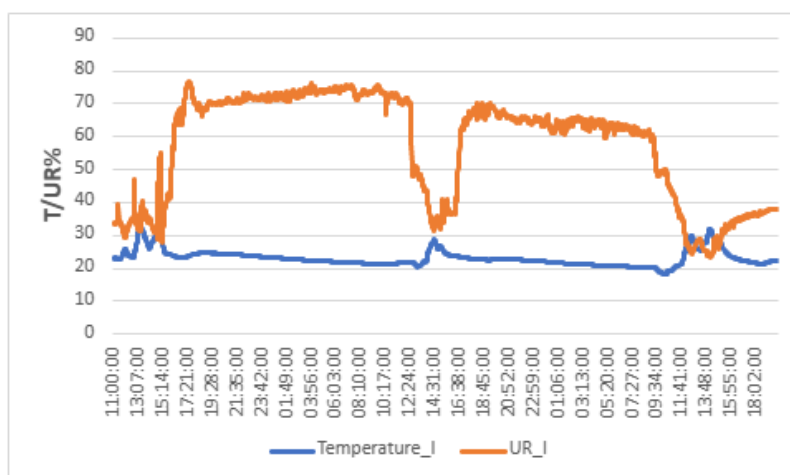


Figure 42 – Continuous monitoring by Arduino sensors of temperature and RH% inside the plant. *Red Delicious* apple.

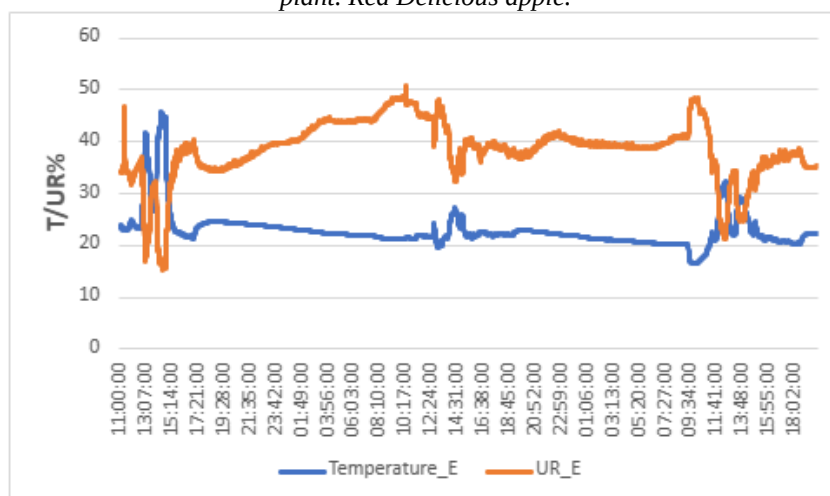


Figure 43 – Continuous monitoring by Arduino sensors of external temperature and RH%. *Red Delicious* apple.

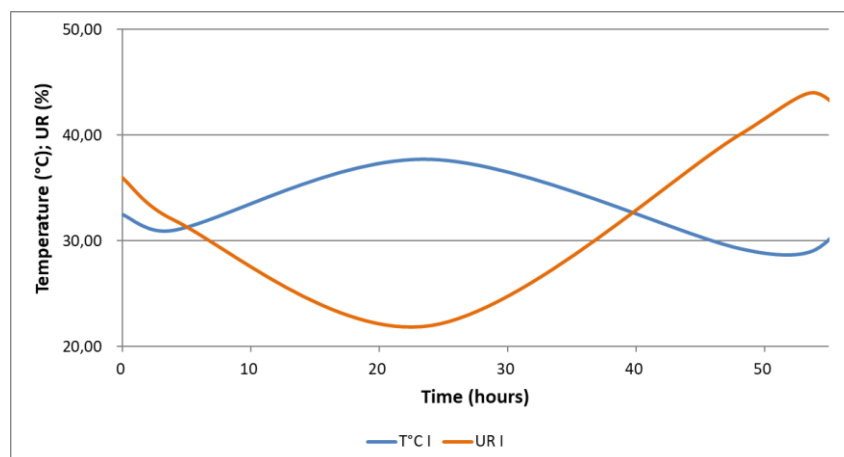


Figure 44 – Continuous monitoring by datalogger of temperature and RH% inside the plant. *Fuji* apple.

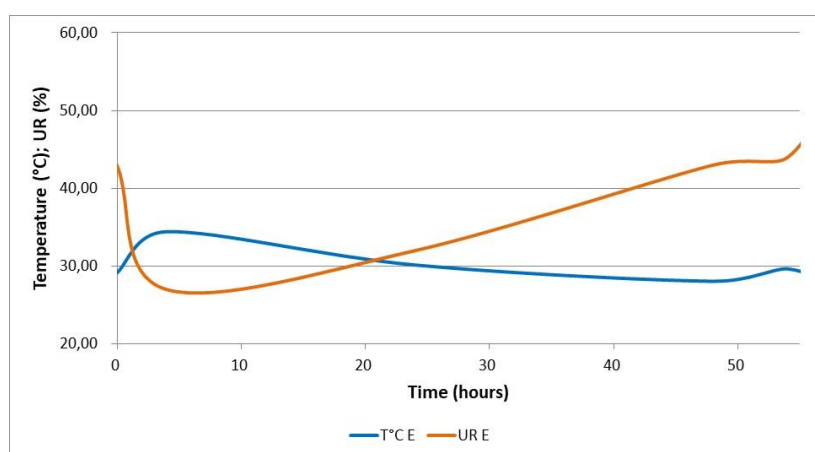


Figure 45 – Continuous monitoring by datalogger of external temperature and RH%. *Fuji* apple.

The two processes were carried out in different days with different climate conditions. During the dehydration processes, the internal temperature varied from 20° to 32°C for *Red Delicious* samples, and from 28° to 38°C for *Fuji* samples.

The trends of RH% confirmed the high fluctuations already recorded in previous experiments. The amount of water vapour stored into the drier increased during the night-time, reaching values around 65% and 45% for *Red Delicious* and *Fuji* samples respectively, and then decreased during the day.

This phenomenon is a peculiarity of solar drying, in fact it is possible to hypothesize that during the exposure to sunlight, the vapour exchange effectiveness present in the solar dryer can be considered equivalent or even better than in conventional system. The alternation of climatic conditions that

cyclically occur in the solar dryer, seems to prevent (or slow) the formation of a surface crust which disturbs the drying process with a negative influence on the quality of the final product.

The external conditions of temperature and RH% seemed to reach similar values during both processes. The trends of external temperature partially influenced the internal ones that reached maximum values of 32°C for *Red Delicious*, and 38°C for *Fuji* samples.

Physico-chemical analyses. The drying processes were stopped when the samples reached a constant weight (55 hours).

Figure 46 shows an example of sample for weighing.



Figure 46 – Examples of aliquots of samples for weighing. Left: *Fuji* apple; right: *Red Delicious* apple.

The trends of the weight loss during the drying processes are shown in Figure 47.

Product weight values gradually decreased during drying until they reached a constant level considered as the end of the process. Both apple varieties showed final values around 40% of the initial weight.

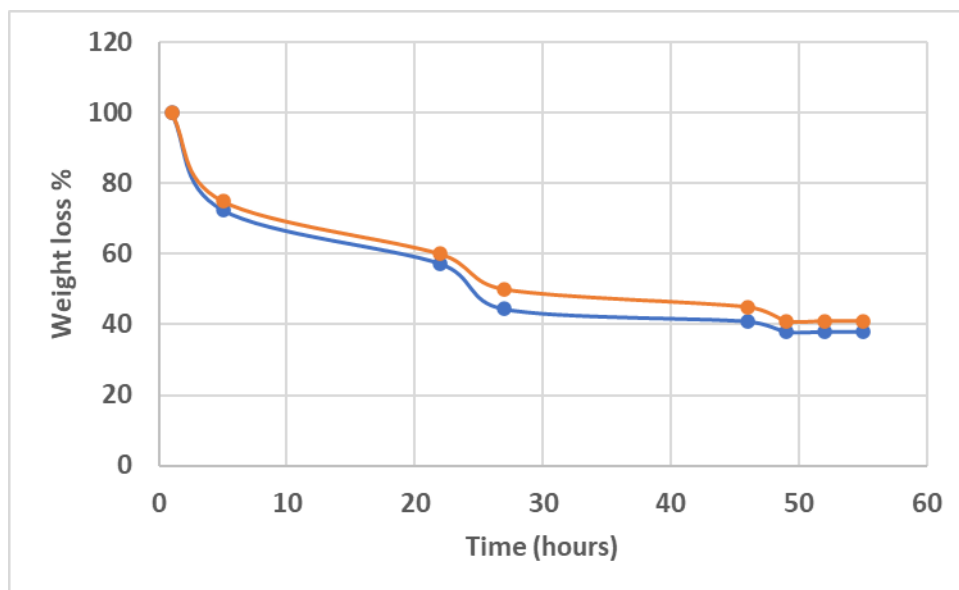


Figure 47 – Weight loss curves during the drying processes. Blue line: Red Delicious; Orange line: Fuji apple samples

Moisture Content (MC), Soluble solid content (SSC) expressed as °Brix, yield and water activity (a_w) values are reported in Table 6.

Table 6 - Moisture Content (MC), Soluble solid content expressed as °Brix, yield and water activity (a_w) values recorded on fresh and/or on dried samples. 1: Red delicious samples; Fuji samples.

Parameters	1	2
°Brix	12.48	11.32
MC% fresh fruit	85.06	84.78
MC% dried slices	9.83	9.65
a_w fresh fruit	0.9997	0.9994
a_w dried slices	0.4217	0.4125
yield%	19.45	21.97

As expected, the content in soluble sugars of *Red Delicious* variety was higher than *Fuji* samples, influencing the sweetness of fruit.

The dehydration processes were able to reduce the MC% of fresh samples, with similar final values around 10%, a value lower than the limit of 15% reported in the literature to be adequate to minimize bacterial, yeast and mould growth [63] and to extend products shelf life.

The drying yields were 19.45% for *Red Delicious* variety and 21.97% for *Fuji* samples. The yield can be influenced by the content of fibers [68]. At the end of the dehydration processes, both products showed a_w values around 0.42,

far below the microbiological safety limit set at 0.6, making all the dried products safe in respect to microbial hazard.

Figures 48-49 report sample tray during the dehydration processes.



Figure 48 – Red Delicious samples during the dehydration process



Figure 49 – Fuji samples during the dehydration process

NIR analysis and Aquaphotomics.

The NIR probe, placed directly over a sample slice, and positioned on the intermediate shelf, collected the NIR spectra every five minutes during the whole process (55 hours). When the *Red Delicious* process ended, the sample slice appeared as shown in Figure 50. This result proved the correct collection of NIR spectra without causing any physical damage to the sample slice. At the same time, this demonstrates that the transfer of the protocol, developed in the previous phases for optimizing the positioning of the sensor, did not

require further modifications resulting directly applicable also in agricultural realities.

Figures 51-52 show the collected spectra during the two trials.



Figure 50 – Red Delicious slice that hosted the NIR probe.

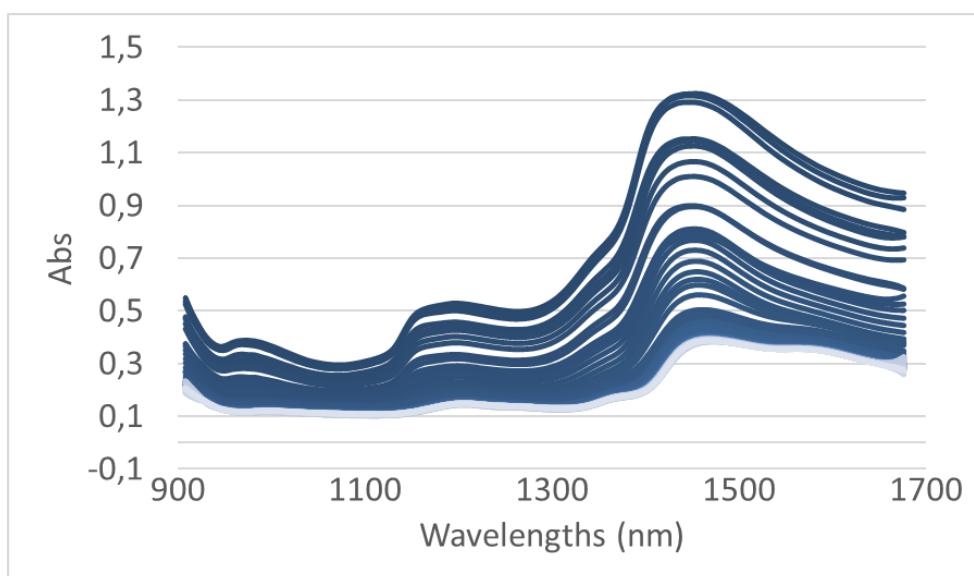


Figure 51 – NIR spectra recorded continuously in real time during the Red Delicious drying. Dark lines correspond to the fresh and moist product; light lines correspond to the dry product.

The raw spectra showed broad and overlapped absorption bands detecting predominant water absorption bands at 960-990 (2nd overtone -OH stretching), 1150-1190 (combination of 1st overtone -OH stretching and OH bending), and 1400-1435 nm (1st overtone -OH stretching) [46], as previously highlighted at micro and pilot scale.

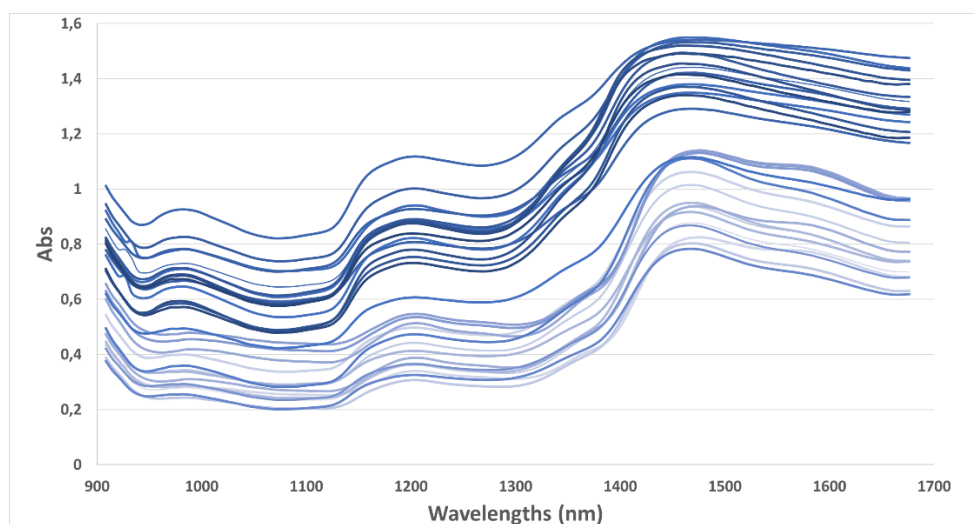


Figure 52 – NIR spectra recorded continuously in real time during the Fuji drying. Dark lines correspond to the fresh and moist product; light lines correspond to the dry product.

The application of the 1st derivative calculation as pre-treatment allowed better visualization of the changes in the spectral profile during the process. This pre-treatment applied to both trials, showed very similar results. Figure 53 show the 1st derivative spectra for *Red Delicious* drying process. In particular, great variations were recorded in the spectral region between 1300 and 1600 nm, associated to the first overtone of water, indicating the possibility of using the Aquaphotomic approach to monitor these processes.

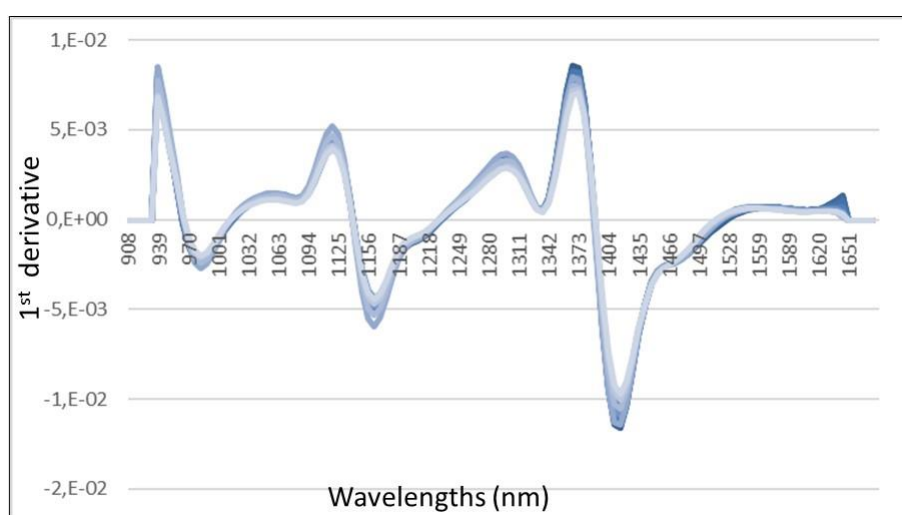


Figure 53 – 1st derivative NIR spectra of the *Red Delicious* dehydration process.

The first two principal components of the PCA performed on the pre-treated spectra explained 98, and 99% of the total variance, for *Red Delicious* and *Fuji* processes respectively.

The trend of the scores defines the positioning of the samples in the multivariate space according to the progress of the processes (Figures 54-55). The loading values obtained from PCA, allowed the selection of 12 wavelengths in the range of WAMCAS (C1-C12: 1300-1600 nm), mainly involved into the water molecules rearrangements, as reported in Table 7, while the related Aquagrams are shown in Figures 56-57.

Table 7 - Wavelengths selected for representation of spectral patterns samples using Aquagrams.

WAMACS	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10	C11	C12
wavelengths	1348	1366	1373	1385	1410	1428	1441	1453	1466	1478	1484	1509

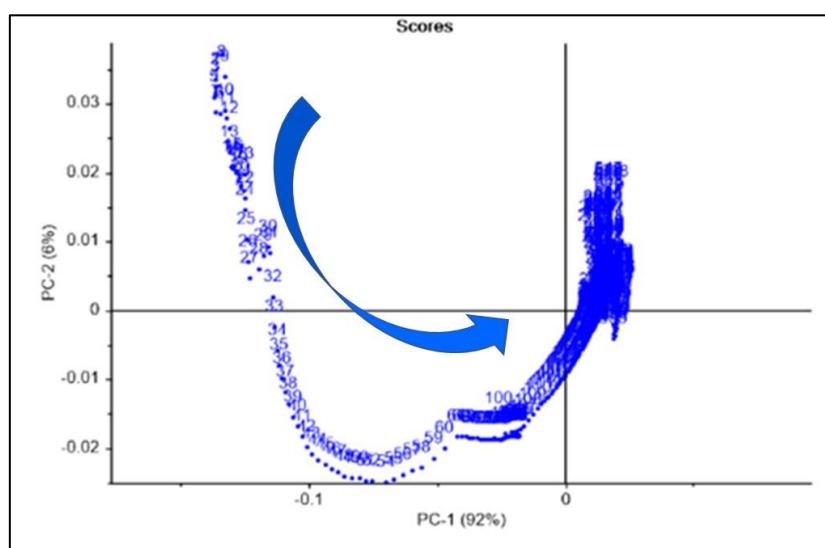


Figure 54 - PCA scores plot performed on Red Delicious pre-treated spectra.

Both the processes have been correctly monitored, highlighting specific shifts of water bands along the drying time.

In particular, an increasing of absorbance was detected over the time into the range C3-C5, which means moving from a system characterized by the prevalence of bonded water forms to a situation in which free water molecules prevail due to the evaporation process and confirming a strong involvement of the 1st overtone -OH stretching (1400-1435 nm).

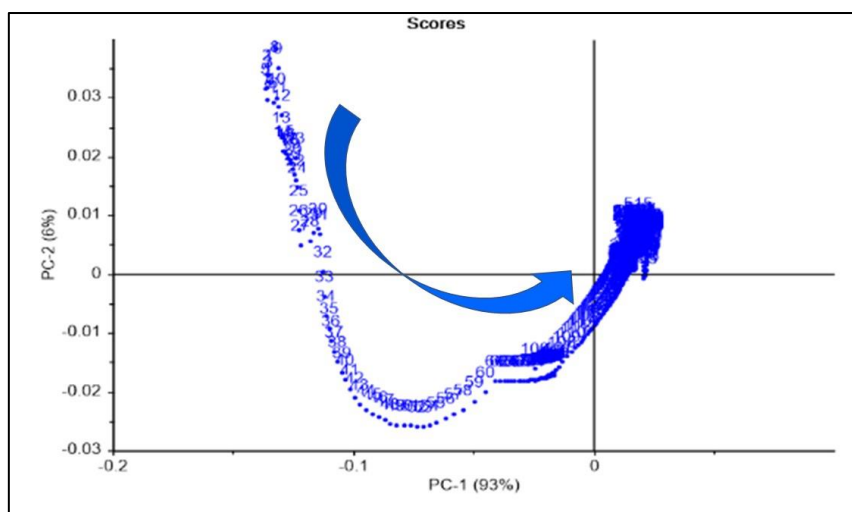


Figure 55 - PCA scores plot performed on Fuji pre-treated spectra.

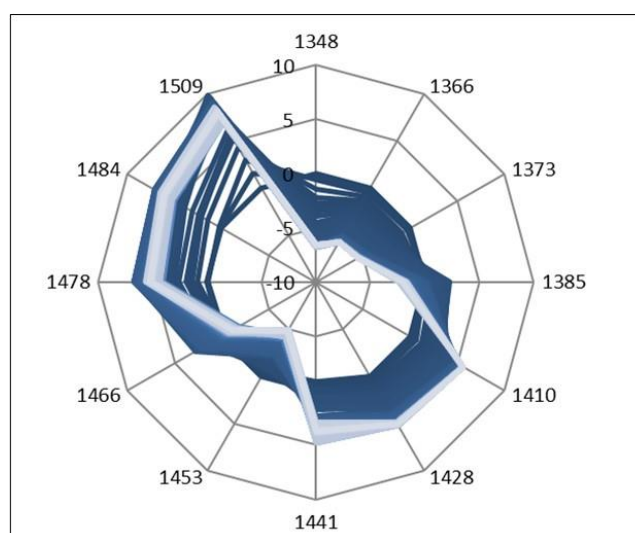


Figure 56 – Aquagram of Red Delicious spectra during the dehydration process. Dark blue: process start; light blue: process end.

Figure 58 provides the results of MBM applied to the acquired data for *Red Delicious* samples using a block of 5 spectra, which means that the first point represents 1-5 spectra, the second point represents 3-6 spectra and so on. The trajectory of the MBM showed a continuous decrease during the process, and tended to a constant value when repeated low values of variability were recorded, helping in identifying the process endpoint. According to the PCA loadings, it can be assumed that the MBM trajectory reflects the modifications of the water component during the process.

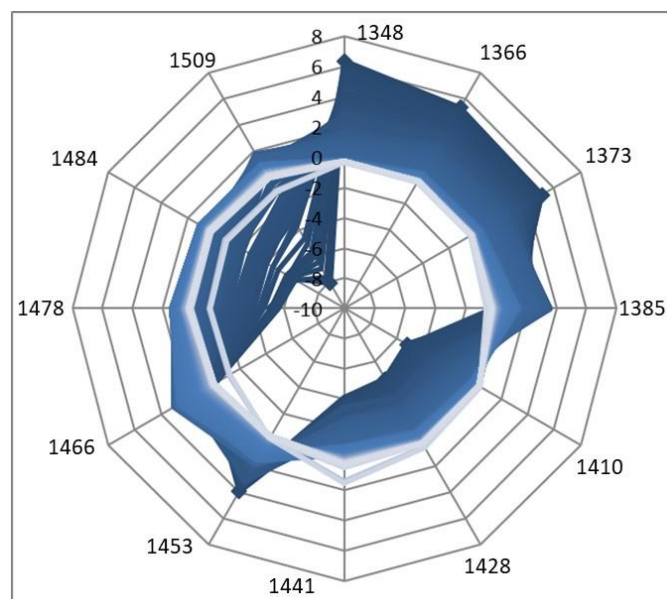


Figure 57 – Aquagram of Fuji spectra during the dehydration process. Dark blue: process start; light blue: process end.

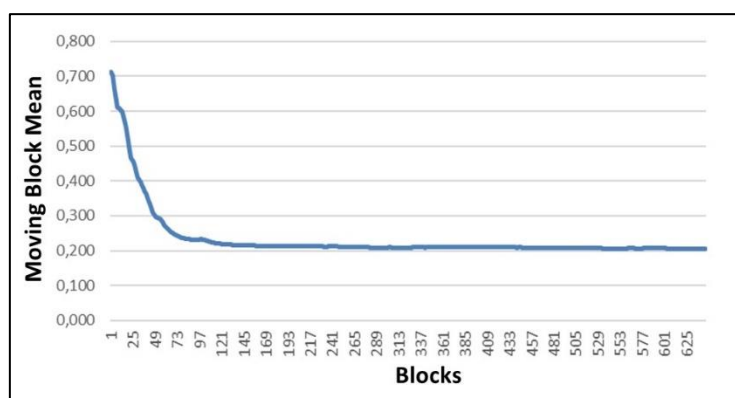


Figure 58 - Red Delicious Moving Block mean trajectory.

Data from *Fuji* drying process were also pre-treated to build up the MBM using the number of spectra in each block equal to 5. Figure 59 shows the obtained results.

It can be noted that, also if the general trend of the MBM showed a gradual decrease, the presence of several spikes was detectable. *Fuji* data were processed using a MBM of 65 spectra per block, with the application of the algorithm that uses the standard deviation (SD), as reported in Figure 60. This permitted the smoothing of the spikes, and results became more simple and accurate.

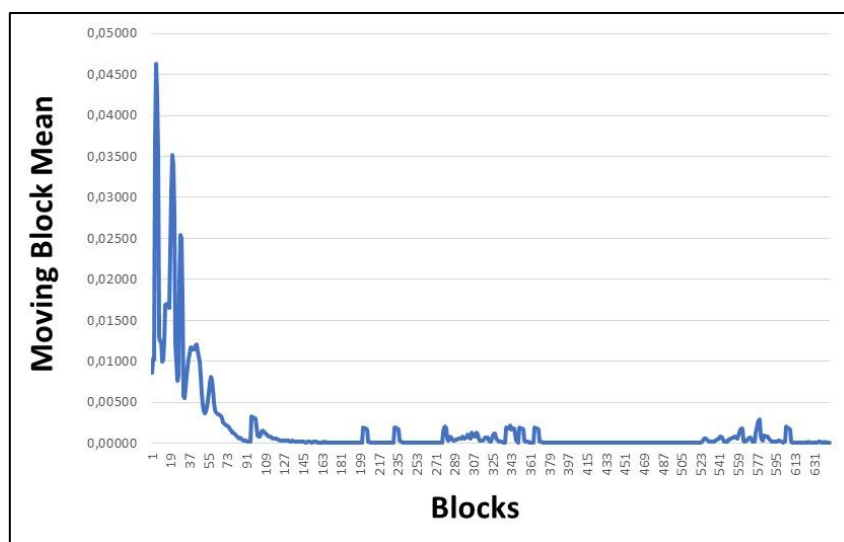


Figure 59 - Fuji Moving Block mean trajectory.

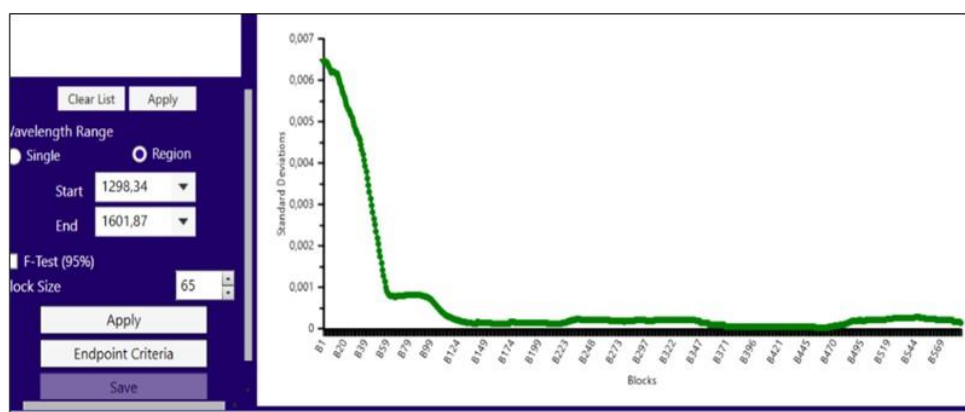


Figure 60 - Fuji Moving Block SD trajectory.

The large amount of data collected made it possible to use this simplification combined with a smoothing action which made it possible to obtain a clearer process profile.

A second observation concerns the curve decrease, that seemed to anticipate the end of the drying process for both the experiments. After the thirtieth hour, the MBM values settled around very small variations, only partially in agreement with the evidence by the weight loss curves and by the Aquagrams which had identified the end of the processes after 55 hours.

Further trials should be carried out for the optimisation of this statistical method and its application at industrial level.

Conversely, the aquaphotomics approach has been considered more reliable and the research was addressed to find a company in charge of preparing the cloud space and processing platform, as an active partner in the preparation

and implementation of all phases of the aqua-control system development, with parallel sampling for chemical-physical analysis.

Qualitade s.r.l. Qualitade s.r.l. is a company located in Cremona, Via Mantova 137, 26100 (Italy). Qualitade is a company of the IT Impresa Group that deals with the integration of IoT systems and related data analysis. The company offers high technological solutions that are easy to use and have high precision. The solutions are continuously updated and designed specifically for the agri-food industry, thanks to the diverse team of food technologists, chemometricians and software engineers. The Quali-Control Department allows to monitor the production during processing and obtain results in seconds. Using a portable NIR spectrometer, it is possible to monitor the process parameters by receiving the results in seconds on smartphones/tablets. Analyses are simultaneously saved, classified and stored within a web portal in the cloud.

A research agreement between Qualitade srl and CREA.IT has been already signed to set up a system that will allow the user to load in a platform web, the spectra gradually collected; this platform performs the following steps:

1. Importing and saving data;
2. Carrying out the appropriate pre-treatments, selected in agreement with the user;
3. Visualization of results via Aquagrams (Spider Plot).

General remarks. The products here studied are stable dried derivates. They have been obtained using the solar drying systems without any energy source except the solar radiation.

These products require a longer processing time rather than the conventional drying methods but allowed to obtain food of good quality associated to no energy cost.

The products obtained by means of the solar drier were characterized by the following reasons:

- they are produced using only solar energy;
- they can be consumed either directly, or as ingredients in other food products;
- they have a long shelf life (Figure 61).



Figure 61 – Red Delicious and Fuji apple dried products.

Drying foods by means of solar exposure using a protected system, can provide significant benefits:

- more intense and consumer accepted product colour;
- improved product hygiene (the system protects the product from animal contamination and weathering);
- no energy required for production.

The scalability and the transferability of the protocols tested at micro and pilot scale were successful.

The control of the physical parameters made by using Arduino sensors and dataloggers allowed the monitoring of the drying processes in real time.

The use of a NIR probe in contact with a product slice, and the collection of spectra every 5 or 10 minutes in continuous, under temperature control, showed to be useful also at industrial level. Among the data processing applied on NIR spectra, the chemometric procedure identified for Aquaphotomics was selected to set up a system, named Aqua-Control, to monitor in real time the progress of the drying process.

5. CONCLUSIONS

The efficiency of two different transformation processes (dryer and dehydration plant) was tested firstly at lab scale. Both micro-plants reached a good level of dehydration after 50 hours.

The chosen digital methodologies were suitable to continuously monitor the dehydration process.

The dehydration plant was chosen as a useful tool for the realisation of PhD activities on the basis of the following considerations:

- both procedures are in full agreement with the objectives of the Agenda 2030, assuring a correct use of the alternative energy resources;
- a “natural” process fit the goal of keeping the binding between “tradition” and “innovation”, reproducing the old process of solar drying but in a protected environment;
- the availability on the market of dehydration plants, flexible for dimensions and size.

The first-year results suggested the applicability of Aquaphotomics for the continuous monitoring of food drying processes using an adequate NIR probe.

PCA loadings confirmed the important contribution of water absorption in the wavelength range 1300–1600 nm during the dehydration processes independently from the food matrix.

Aquagrams can help to identify the actual endpoint of the process, due to the detection and the monitoring of the different water forms (free water, dimers, aggregates, clusters, etc.).

The combination of information from the two chemometric instruments, MBM and Aquagrams, can be useful for defining and predicting the end of the dehydration process. The choice of the proper size of blocks is crucial to gain a good predictive power of the MBM.

The selection of few wavelengths to study food processes and quality can optimise the transfer of instrumental data to IT platforms and networks.

The trials carried out at pilot level proved the use of NIR for monitoring the progress of the dehydration process also after the scale transposition.

The development of real-time predictions of MC and a_w could be a useful tool for detecting the end of the dehydration process and for assessing the potential safety of the final product. More relevant information on the model applicability could be obtained by validating them with an external dataset. In some cases, the reduction of the spectral range used in developing NIR models can reduce the noise due to non-contributing wavelengths and could lead to the development of lower-cost systems for the application of the models.

A large number of samplings can help to have robust models.

The Aquaphotomics approach proved, also at pilot level, to be useful to monitor the whole dehydration process through the changes in water profiles. The scalability and the transferability of the protocols tested at micro and pilot scale proved to be also reliable at industrial level.

The control of the physical parameters made by using Arduino sensors and dataloggers allowed the monitoring of the drying processes in real time.

About the tested chemometric procedures for data processing able to manage in real time the drying process, the Aquaphotomics approach has been considered more reliable than MBM. In fact, it should not need further investigations for its optimisation and application at industrial level.

It should be remembered that it is important to set up dedicated aquagrams for each individual process.

The preparation of annual control charts can help to create a corporate database that allows linking climatic conditions to the realization of food transformations, including their monitoring.

The use of a NIR probe in contact with a product slice, and the collection of spectra every 5 or 10 minutes, under temperature control, showed to be useful also at industrial level. Among the data processing applied to NIR spectra, the chemometric procedure identified for Aquaphotomics was selected to set up a system, named Aqua-Control, to monitor in real time the progress of the drying process.

Furthermore, the Aquaphotomics approach has been applied for its suitability in identifying quality changes during the shelf life of ready-to-eat rocket salad. For this purpose, the shelf life of ready-to-eat rocket salad packed under modified atmospheres was evaluated.

The obtained results, already published [69], proved the transferability of the Aquaphotomics approach to monitor the shelf life of ready-to-eat products, using only non-destructive techniques.

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7. ACKNOWLEDGEMENTS

I thank the Institutions, the groups, and the people who in different ways have collaborated in the realization of this PhD program:

- The University of Viterbo, Dafne department, EEE course: Prof. Andrea Luigi Facci, Prof. Massimo Cecchini, Prof. Danilo Monarca, the secretarial staff;
- The Ministry of Agriculture, Food Sovereignty and Forestry;
- The Council for research in agriculture and economics (CREA);
- The Research Centre Engineering and Food Processing (CREA.IT): Dr. Paolo Menesatti;
- The CREA.IT teams of Milan and Treviglio (BG): Dr. Massimo Brambilla, Dr. Elio Romano, Dr. Maurizio Cutini, Mrs. Annamaria Stellari, Mr. Alex Filisetti, Mr. Ivan Carminati, Dr. Laura Marinoni;
- Dr. Alessandro Cammerata (CREA.IT Roma), Dr. Stefania Barzaghi (Research Centre for Animal Production and Aquaculture – CREA.ZA);
- Dr. Andrea Sala and Ca' dal Saggia farm;
- Dr. Marcello Vanzulli and Qualitade S.r.l.;
- Dr. Roberto Giangiacomo, who has been my professional guide for a long time;
- My family.